ESS

Spiking Report
2006 WeState Carbon Performance Demonstration Test

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Excellence through Combustion Chemistry

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SPIKING REPORT

1.0 Introduction and Background

Background: WeStates Carbon (WeStates) owns and operates an activated carbon re-generation facility located in Parker, AZ. Because wastes, which meet the 40 CFR 261 (RCRA) definition of hazardous waste are managed in this unit, it is subject to the RCRA Regulations (40 CFR 260 through 271). Since this activated carbon re-generation unit does not meet the RCRA (40 CFR 260.10) definitions of an incinerator, a boiler, or an industrial fumace, it is regulated as a (40 CFR 264, Subpart X) Miscellaneous Unit, and is also being held to the HWC MACT requirements found at 40 CFR 63, Subparts A and EEE. This Performance Demonstration Test (PDT) was planned and executed to demonstrate that the unit operates in compliance with all applicable (HWC MACT) Environmental Performance Standards.

Test Project Team: To conduct the PDT in compliance with all applicable regulations, methods, protocols, guidance, & policies, WeStates retained:

1. Focus Environmental, Inc (Focus) to: (a) plan, (b) manage, and (c) report the results of the test,
2. AIRTECH Environmental Services, Inc (AIRTECH) for stack gas sampling services, and
3. Engineered Spiking Solutions, Inc. (***ESS***) for spiking services.

Test Structure, Schedule, & Spiking Requirements: The 2006 WeStates PDT entailed a single, triplicate-run Test Condition (TC) spiking four (4) materials with eight (8) distinct spiking species:

1. Mono-Chlorobenzene (MCB),
2. Perchloroethylene (Perc),
3. Organics Solution [A solution of four organic compounds, Methylene Chloride (CH_2Cl_2), Ethylene Glycol, Toluene, and Naphthalene], &
4. Metals (Pb & Cr^{III}) Solution [A dilute Aqueous solution of Pb $(NO_3)_2$ & Cr $(NO_3)_3 \bullet 9H_2O$].

Table 1 provides a summary of WeStates' Spiking Requirements for this test.

Table 1 Summary of the 2006 WeStates PDT Spiking Requirements:

		Target Spiking Rates, Lb M or S/Hr					
	Test Date →	4/27/2006		4/27/2006		4/28/2006	
	TC # 1, Run # →	Run #1		Run #2		Run #3	
Spiking Materials[1], M ↓	Spiking Species[1], S ↓	Lb M/Hr	Lb S/Hr	Lb M/Hr	Lb S/Hr	Lb M/Hr	Lb S/Hr
MCB	MCB	35	35	35	35	35	35
Perc	Perc	35	35	35	35	35	35
Organics Solution		41		41		0	
	CH_2Cl_2		8		8		-
	Ethylene Glycol		8		8		-
	Toluene		17		17		-
	Naphthalene		8		8		-
Metals Solution		20		20		20	
	Pb		0.10		0.10		0.10
	Cr^{III}		0.35		0.35		0.35

1. Spiking Material (M) refers to the material which is actually spiked, i.e., a metal solution, a TiO_2 and/or metal dispersion, and/or a POHC or a solution of two or more POHCs. Spiking Species (S) refers to that portion of the Spiking Material which is of specific interest in meeting the test objectives, i.e., elemental metal(s), ash, POHC(s), Cl^-, etc.

ESS* Spiking Project Scope:** ***ESS provided the following services in satisfaction of these spiking requirements:

1. All necessary spiking equipment, tools, and supplies,
2. All spiking materials,
3. On-site spiking, and off-site project management, coordination & support services, and
4. This PDT Spiking Report.

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ESS utilized four spiking systems [e. g., for: (1) MCB, (2) Perc, (3) Organics Solution, & (4) Metals Solution] to satisfy the PDT spiking requirements defined in Table 1, above. The on-site aspects of this project were completed during a March, 2006 mobilization.

Spiking Report Organization:

Section 2.0 Provides a Description of the Spiking Methods and Operational & QA Procedures ***ESS*** used to meet these spiking requirements;

Section 3.0 Provides the Species/Material Spiking Rate Results in both Absolute (Lb/Hr) and Relative (% of target) Terms, based on two methods of measuring field spiking rates:
1. The Weight Loss vs. Time Method, and
2. The Mass Flow Meter Method;

Section 4.0 Provides QA/QC Results and a Discussion of these Results in the context of this project; and

Section 5.0 Provides Conclusions related to these QA/QC & Spiking Rate Results.

Spiking Report Attachments:

Attachment I provides the Spiking Plan for the test. The plan identifies the:
1. Spiking species (and spiking materials),
2. The anticipated spiking rate(s) and duration(s),
3. The number and types of spiking pumps, weigh scales, and mass flow meters (MFMs) to be used,
4. The Test Manager's Spiking Orders to ***ESS***. (This is an ISO 9001:2000 QMS related document, which ***ESS***: (a) prepares for each spiking project based on our understanding of the client's spiking requirements and (b) requests that the client's test manager review & approve the document as a means of demonstrating a common understanding of the spiking scope), and
5. Details concerning some of the preparatory efforts (including Work Instructions, Check Lists, Worksheets, & Other Project Preparation Documentation) which ***ESS*** uses to ensure that the defined spiking requirements are consistently met.

Attachment II documents the composition of the spiking materials used during this project.

Attachment III provides a demonstration that the field spiking rate measurement equipment used during this project is accurate as provided in Attachment III documentation of the accuracy of the weighing systems used during the on-site portion of this project. Specifically, ***ESS*** completed (& documented) three separate verifications of the weigh scale calibrations using NIST traceable weight standards:
1. At ***ESS'*** shop prior to mobilization (all scales including spares),
2. At the test site prior to beginning the PDT (for scales selected for use during the test), and
3. At the test site after completing the PDT (for scales used during the test).

Attachment IV provides:
1. The completed Test Manager's Spiking Orders to ***ESS*** and other spiking related information (IV.A),
2. Stack sampling start and stop times (IV.B), and
3. Spiking Log Sheets (field data), spiking rate calculations, and results (IV.C).

Attachment V contains two recently published papers[1], which discuss the effect of measurement uncertainty on the uncertainty in spiking rate results. The first paper (2004 IT3 Paper 103) documents why the method, which ***ESS*** has developed for demonstrating the composition of spiking material composition[2] is at least two (2) orders of magnitude more accurate than the most commonly used method[3] in the spiking industry.

The second paper (2004 IT3 Paper 102) provides a comprehensive comparison of:
1. The measurement uncertainty associated with the two primary methods of measuring and controlling spiking rate [Weight Loss vs. Time & Mass Flow Meters (MFMs)],
2. A summary description of the underlying technology of each measurement method,
3. The resulting operational attributes of each method, and
4. The combined measurement uncertainty accruing from: (a) compositional uncertainty, and (b) spiking rate measurement uncertainty.

This paper will be discussed further at the beginning of Section 2.0.

1. Proceedings of the 2004 International Incineration and Thermal Treatment Technology (2004 IT3) Conference (Papers 103 & 102).
2. The ***Laboratory Standard Method***, i.e., Prepare the spiking material with the same care & attention to accuracy as you would in preparing a "laboratory standard" for calibrating a sensitive analytical instrument.
3. ***Sample & Analyze Method***, i.e., one would take a sample of the finished spiking material and analyze it using analytical methods approved by the environmental agency to which the spiking report would ultimately be submitted.

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2.0 Spiking Method and Operation & QA Procedures

Comparison of Spiking Methods: Historically, two methods to measure and control spiking rate have been used: (1) Weight Loss vs. Time (based on mass measurement technology) and Mass Flow Meter (based on technology which measures mass flow using Coriolis Effects). From the early 1980s until early 2004, ***ESS*** (& staff members) used the Weight Loss vs. Time[4] Method exclusively (See Figure 1). However, for reasons outlined below (and discussed more thoroughly in the previously referenced 2004 IT3 Paper 102), ***ESS*** is currently field implementing a spiking system based on the best attributes of both methods.

The **Weight Loss vs. Time Method**[4] of measuring & controlling spiking rate provides a quick, efficient, and tangible demonstration of accuracy using NIST traceable weight standards[5]. This is due to the mass measurement nature of this technology, e.g., if one places a certified weight standard on the scale, the measured or indicated weight promptly appears on the weight indicator. One can easily obtain a straight forward, quick, and definitive comparison of the "indicated" weight to the "known" weight over the entire operating weight range of interest. ***ESS*** utilizes this approach to demonstrate the accuracy of our spiking rate results with NIST traceability[5]. We believe that demonstrating the accuracy of ones spiking rate data (with traceability to a nationally recognized standard) is essential in the Trial Burn and PDT context in which spiking occurs.

Conversely, it is very difficult to demonstrate the accuracy of **Mass Flow Meters** (MFMs with comparable measurement uncertainty), because of the rate (mass/time, as opposed to mass) measurement basis of the technology. However, the direct and instantaneous measurement of rate provided by MFMs offers an inherent spiking rate control advantage through a feedback control system. Thus, spiking rate can be controlled to a uniform target level &/or the spiking rate can be changed and quickly brought to a new target during, for example, a Trial Burn in which runs at different spiking rates are required.

The *ESS* Spiking Technology: Because of these complimentary characteristics, ***ESS*** has developed spiking technology that incorporates (and benefits from) both methods:

1. MFM provides an instantaneous measurement of spiking rate to ***ESS*** computer based feedback control system, and
2. The Weigh Scale provides spiking rate results with demonstrated accuracy to NIST Standards. Corrections to the control system spiking rate set point are cascaded from the weighing system to the control system, as needed.

Spiking rate data produced by this dual technology system provides both tight control to the client's target spiking rate and spiking rate results, which are demonstratably accurate[5] based on NIST Traceable Standards.

4. With the **Weight Loss vs. Time Method** (as implemented by ***ESS***), a container of spiking material is placed on a highly accurate weigh scale (appropriately sized and calibrated for the test specific weight range), and connected with SS, drip-less, quick-connect fittings to the metering pump, which is similarly connected to the waste feed line. When spiking material is pumped out of the container and into the waste feed line, the mass on the weigh scale will drop. ***ESS'*** computer based control & data acquisition system: (a) records the weight & time at a rate of 1 data set/second, (b) compares the actual rate to the target-spiking rate, and (c) adjusts the spiking rate, as needed.
5. ***ESS*** verifies the calibration of each scale with ***ESS'*** NIST traceable weight standards three separate times for each spiking project: (a) at ***ESS'*** shop prior to mobilizing to the test site, (b) at the test site immediately before testing, and (c) at the test site immediately after testing. ***ESS'*** 50 lb field standards are certified annually by the State of Texas for a maximum absolute uncertainty of ± 0.008 lb (approx. ± 0.016% RU) of NIST Primary Standards. ***ESS'*** standards were last Certified on 8/11/2005 as documented in Attachment III.C. Typically, the measurement uncertainty of ***ESS'*** weigh scales is approximately ±0.01% to ±0.02% of the scale's upper calibration weight, or in terms of the typical 0 to 650 Lb calibration range, ±0.065 to ±0.13 lb. As a result, measurement uncertainty with the weigh scale technology is typically < 0.1% of the material spiked during a given run.

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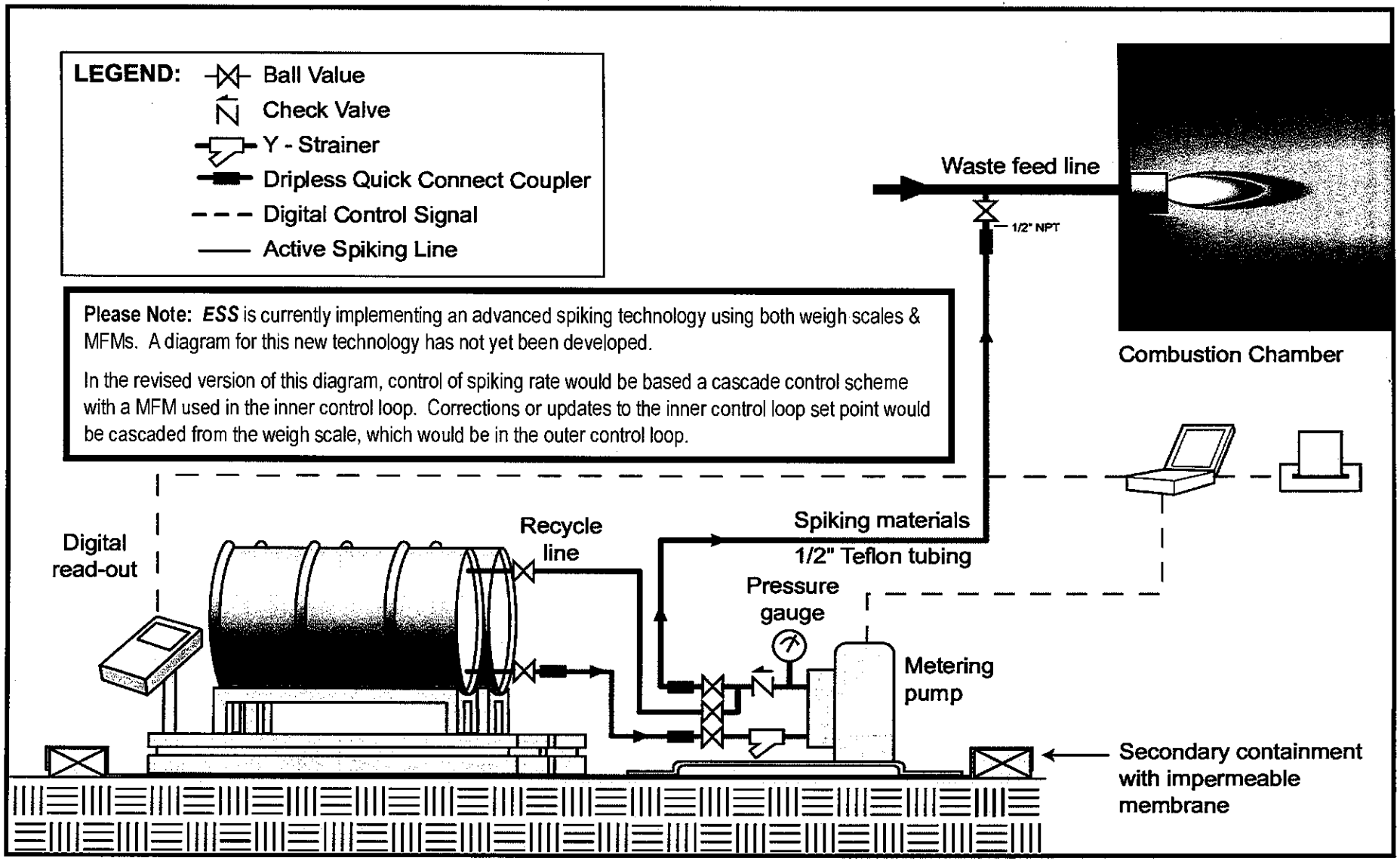
Figure 1 Schematic Diagram: ESS' Spiking Procedure



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QA Program: There are many factors beyond field spiking rate measurement, which can adversely affect the quality/defensibility of the overall spiking rate results. ***ESS*** has invested a substantial effort to identify and address a wide range of preparation and operational concerns through a comprehensive, ISO 9001:2000 Certified Quality Management System (**ISO QMS**[6]).

ESS'* QMS** addresses all aspects of ***ESS' products and services delivery system with emphasis on the two most mission-critical phases of ***ESS'*** Products & Services Delivery System:

1. Off-site preparation measures prior to mobilizing to the test site, and
2. On-site setup & spiking measures immediately prior to, during, & immediately after testing.

Selected aspects of ***ESS'*** QMS are described below.

ESS has developed a number of QA measures to help ensure that client spiking requirements are consistently met. The single most significant measure is based on the observation that all spiking projects can be subdivided into approximately 40 work functions[7]. Each project would require a different subset of these work functions as well as different, project-specific materials, quantities, rates, etc.

While these work functions are individually relatively simple and routine, they must:

1. In aggregate, address all planning, scheduling, execution, & documentation steps associated with meeting the spiking requirements of any given spiking project (i.e., be comprehensive and accurate), and
2. Be consistently completed with strict adherence to prescribed protocols and project-specific details (i.e., completed without error).

In an effort to minimize the possibility of errors & omissions during any given spiking project, ***ESS*** has developed a generic (40+ page) Project Planning, Documenting, & Execution Template (Project Plan Template), which is designed to address all spiking projects, and includes: Protocols & Work Instructions[8] (e.g., SOPs), Work Sheets[8], and numerous Check Lists[9] & Log Sheets and Reports[10].

In the initial phase of every spiking project, ***ESS*** revises this generic template by:

1. Inserting project-specific details into those sections of the template (work functions) which are applicable to that project, and
2. Deleting from the template (or marking as "NA") those sections which are not applicable.

These changes result in the project-specific Project Planning, Execution, & Documentation Package (**Project Plan**) for that project.

The Project Plan addresses:

1. Preparation at ***ESS'*** shop prior to mobilizing to the test site including:
   A. Spiking materials preparation,
   B. Operability & accuracy verification of all equipment selected for a given spiking project at ***ESS'*** shop prior to mobilization,
   C. Identification and completion of all "special" project specific equipment, procedures, materials, training, medical requirements, &/or other special pre-mobilization preparation, &
   D. Project materials, tools, equipment, supplies, etc. checklists, and equipment trailer & truck safety checklists Operability & accuracy verification of all equipment[7] selected for a given spiking project at ***ESS'*** shop prior to mobilization.

6. ***ESS'*** Quality Manual, Rev 1, June 30, 2005.
7. Examples include: (a) weighing out a quantity of spiking material, dispersant, &/or carrier/solvent, (b) mixing a solution of soluble metal salt in an aqueous solution, (c) preparing a dispersion, (d) calibrating or verifying the calibration of a measurement instrument with NIST Traceable Standards, (e) documenting weight &/or rate data, (f) assembling equipment, tools, supplies, etc. for transport to the test site, (g) assembling the equipment into spiking systems at the test site, (h) testing operability of spiking equipment/systems prior to mobilization and at the test site, (i) preparing spiking materials (which are almost always hazardous materials under US DDT & IATA requirements) for common carrier transport, etc.
8. Protocols & Work Instructions (e.g., SOPs), and Work Sheets include: (a) Materials Prep Instructions, (b) Raw Material Weights, & Materials Preparation Work Sheet, (c) Multiple-Packet Preparation Instructions, (d) Equipment Operability Verification & Pre-Mobilization Instructions, (e) Transition Fitting Installation & Use Instructions, (f) Field-Scale Set-Up, Adjustment & Calibration Verification Reports, (g)(i) Pb/Cr^{II} Solution & (ii) Organic Solution Preparation & Composition Calculation Instructions & Work Sheets.
9. Check Lists include: (a) Project Plan Component Transmittal & Acceptance Check List, (b) Overall Project Preparation Check List, (c) Materials Preparation Component Check List, (d) Direct Ship Materials Check List, (e) Materials Preparation Check List, (f) Equipment Operability Verification Check List, (g) Materials Release for Shipment Check List, (h) Multiple-Packet Preparation Check List, (i) Multiple-Packet Information Check List, (j) Pre-Travel Project Preparation Equipment Operations and Maintenance Check List, (k) Pre-Travel Safety Check List, (l) Spiking Plan Transmittal & Acceptance Check List, (m) Overall Test Execution Check List, and (n) Field-Scale Set-Up, Adjustment & Calibration Verification Check List.
10. Log Sheets and Reports include: (a) Shop Scale Calibration & Calibration Verification Reports, (b) Laboratory Scale Calibration & Calibration Verification Reports, (c) Pre-Mobilization, Equipment Operability (@ Test Conditions) Verification & Certification Log Sheets, (d) Tentative & Final Product Release for Shipping Log Sheets, (e) Daily Activity Log Sheets, (f) Pre-Mob and Pre-Test & Post-Test Field Scale Calibration & Calibration Verification Reports, (g) Equipment Operation & Maintenance Log Sheets, (h) Equipment Adjustment Log Sheets, and (i) Spiking Rate Data Log Sheets [(i) 1st Sheet, & (ii) 2nd Sheet.

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2. Mobilizing to the test site;
3. Equipment set-up and operability & accuracy verification at the test-site (including pre-test and post-test scale & MFM calibration verifications);
4. Spiking during the test;
5. Documentation of all QA steps & spiking rate results, &
6. Equipment Decontamination and Demobilizing.

The Project Plan Provides Standard Log Sheets, Worksheets, Check Lists, Instructions, etc. for:

1. Spiking Materials Preparation, which include QA requirements, formal product release protocols, and DOT shipping requirements,
2. Pre-Mobilization, Pre-Test, & Post-Test Weigh Scale and MFM Calibration Verifications,
3. Pre-Travel Project & Safety Check Lists,
4. On-Site preparation of aqueous solutions,
5. Spiking Data Collection, and
6. Daily Spiking Operations.

***ESS* provides all personnel with all necessary:**

1. Detailed, project-specific information,
2. The spiking materials, methods, & equipment,
3. Training for every plausible health & safety related exposure,
4. Training on all spiking functions for which they are responsible, and
5. Feedback from detailed project assessments following every project.

To help ensure that all client spiking requirements are safely & consistently met.

Additionally, "lessons learned" during each project assessment are used to further improve the Generic Template, Equipment Fleet, Methods, Training, etc.

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3.0 Spiking Rate Results

Determining Spiking Rate: ***ESS*** used two methods to measure and control spiking rate during this PDT project:

1. The Weight Loss vs. Time Method, and
2. The MFM Method.

Weight Loss vs. Time Method: With the Weight Loss vs. Time Method, ***ESS*** calculates the average spiking rate utilizing weight loss data:

1. When spiking and stack sampling have both occurred,
2. During the port change in the middle of each run if spiking continued during the port change,
3. During brief interruptions (typically, <20 – 30 minutes) of stack sampling if spiking continued during the interruptions, and
4. During brief spiking interruptions (typically, <20 – 30 minutes), if stack sampling continued during the interruptions.

Not included in these calculations are periods:

1. Prior to the beginning of stack sampling,
2. After stack sampling on a given run is completed,
3. During port changes and/or other brief stack sampling interruptions when spiking was not occurring, or
4. During longer interruptions (typically, >20 – 30 minutes) of either the sampling or spiking functions.

A review of the ***ESS*** Spiking Log Sheets in Attachment IV.C will further clarify these procedures.

MFM Method: The spiking rate with the MFM Method was calculated as the average spiking rate over the same period(s) as described above.

Spiking Rate Results: Tables 2, 3, & 4 provide the Average, Absolute (Lb/Hr) and Relative (% of Target) Spiking Rate Results for TC #1, Runs #1, #2, & #3, respectively, based on both methods of measuring field spiking rate. Table 5 provides a summary of the spiking rate results from Tables 2, 3, & 4.

Inspection of these tables indicates two consistent findings:

1. The spiking rate results are very close to corresponding targets,
2. The two spiking rate measurement methods agree within very close tolerances. This is not surprising, since the MFMs were calibrated to the mated weigh scale (after the scale had been calibrated with NIST Traceable Standards).

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Table 2 Average, Absolute & Relative Spiking Rate Results for TC #1, Run #1 Based on: 1. The Weight Loss vs. Time, & 2. The MFM Methods of Measuring Field-Spiking Rates

TC #/ Run #	Spiking[1]:		Corrections[2] for:				1. Spiking Rate by Weight Loss vs. Time				2. Spiking Rate by Mass Flow Meter			
							Absolute Spiking Rate			Relative SR[5]	Absolute Spiking Rate			Relative SR[5]
	Species[1], S	Material[1], M	Concentration[2]	Purity[2]	Stoichiometry[2]	(Specie)[2]	Lb M/Min[3]	Lb S/Min[4]	Lb S/Hr[4]	% Target	Lb M/Min[3]	Lb S/Min[4]	Lb S/Hr[4]	% Target
TC #1:														
Run #1	MCB	MCB	1.0000	0.999976	1.0000	0.999976	0.5802	0.5802	34.81	99.46	0.5803	0.5803	34.82	99.48
Run #1	Perc	Perc	1.0000	0.99974	1.0000	0.99974	0.5876	0.5872	35.24	100.7	0.5842	0.5840	35.04	100.1
Run #1	Organics Solution:													
Run #1		Methylene Chloride	0.1951	0.9999	1.0000	0.1951	0.6873	0.1341	8.046	100.6	0.6812	0.1329	7.974	99.68
Run #1		Ethylene Glycol	0.1951	0.9998	1.0000	0.1951	0.6873	0.1341	8.046	100.6	0.6812	0.1329	7.974	99.68
Run #1		Toluene	0.4146	0.9999	1.0000	0.4144	0.6873	0.2848	17.09	100.5	0.6812	0.2823	16.94	99.63
Run #1		Naphthalene	0.1951	0.9993	1.0000	0.1950	0.6873	0.1340	8.041	100.5	0.6812	0.1328	7.970	99.63
Run #1	Metals Solution:													
Run #1		Pb	0.007989	1.0000	0.6256	0.004998	0.3350	0.001674	0.1005	100.5	0.3305	0.001652	0.09911	99.11
Run #1		Cr^{III}	0.134842	1.0008	0.1299	0.01753	0.3350	0.005867	0.3520	100.6	0.3305	0.005788	0.34726	99.20

Footnotes:

1. Spiking Material (M) refers to the material, which is actually spiked, i.e., a metal solution, a TiO_2 and/or metal dispersion, and/or a POHC. Spiking Species (S) refers to the portion of the Spiking Material, which is of specific interest in meeting the test objectives, i.e., the elemental metal(s), ash, PDHC(s), Cl^-, etc.
2. Concentration refers to the correction for the concentration of the compound of interest in the Spiking Material assuming compound has 100% purity. If we consider for example TiO_2 in a TiO_2 Dispersion, we would divide the net weight of TiO_2 present in the dispersion assuming 100% purity (i.e., no correction for moisture content) and enter that mass fraction in the Concentration column for Ash. A similar process would apply for a metal compound in an aqueous solution, etc. Purity refers to the assay, or purity of the POHC or metal compound, for example, used to make up the solution to the desired concentration. Stoichiometry refers to the stoichiometric content of the specie of interest in the compound, for example the Cl^- content in Perc or the elemental metal content in a metal compound (***ESS*** uses the Merck Index as our primary source of Stoichiometry Correction).

   (Specie) indicates the specie concentration (usually expressed as Lb Specie/Lb Material, or mass fraction) and is defined as the mathematical product of the three previously described corrections: (Specie) = Concentration x Purity x Stoichiometry. (Specie) is used as an overall correction factor to convert the Spiking Material spiking rate, which is determined from field spiking rate measurements to the corresponding Spiking Specie spiking rate, which is of interest to our clients and their corresponding regulatory agency representative(s).

   Usually, all four of the "correction" terms are expressed as mass fractions. However, Concentration and (Specie) will occasionally be expressed as g S/Lb Material or similar units appropriate for a specific test circumstance.
3. Spiking Material (M) spiking rate without (Specie) correction. Calculated from the Spiking Log Sheets in Attachment IV.C.
4. Spiking Specie (S) spiking rate, i.e., after (Specie) correction.
5. Relative Spiking Rate Expressed as a percent of the corresponding Target Spiking Rate (See Table 1).

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Table 3 Average, Absolute & Relative Spiking Rate Results for TC #1, Run #2 Based on: 1. The Weight Loss vs. Time, & 2. The MFM Methods of Measuring Field-Spiking Rates

TC #/ Run #	Spiking[1]:		Corrections[2] for:				1. Spiking Rate by Weight Loss vs. Time				2. Spiking Rate by Mass Flow Meter			
							Absolute Spiking Rate			Relative SR[5]	Absolute Spiking Rate			Relative SR[5]
	Species[1], S	Material[1], M	Concentration[2]	Purity[2]	Stoichiometry[2]	(Specie)[2]	Lb M/Min[3]	Lb S/Min[4]	Lb S/Hr[4]	% Target	Lb M/Min[3]	Lb S/Min[4]	Lb S/Hr[4]	% Target
TC #1:														
Run #2	MCB	MCB	1.0000	0.999976	1.0000	0.999976	0.5842	0.5842	35.05	100.1	0.5842	0.5842	35.05	100.1
Run #2	Perc	Perc	1.0000	0.99974	1.0000	0.99974	0.5861	0.5859	35.16	100.4	0.5838	0.5836	35.02	100.1
Run #2	Organics Solution:													
Run #2		Methylene Chloride	0.1951	0.9999	1.0000	0.1951	0.6825	0.1332	7.989	99.87	0.6814	0.1329	7.976	99.71
Run #2		Ethylene Glycol	0.1951	0.9998	1.0000	0.1951	0.6825	0.1332	7.989	99.87	0.6814	0.1329	7.976	99.71
Run #2		Toluene	0.4146	0.9999	1.0000	0.4144	0.6825	0.2828	16.97	99.82	0.6814	0.2823	16.94	99.66
Run #2		Naphthalene	0.1951	0.9993	1.0000	0.1950	0.6825	0.1331	7.985	99.81	0.6814	0.1329	7.972	99.65
Run #2	Metals Solution:													
Run #2		Pb	0.007989	1.0000	0.6256	0.004998	0.3360	0.001679	0.1008	100.8	0.3358	0.001678	0.1007	100.7
Run #2		Cr^{III}	0.134842	1.0008	0.1299	0.01753	0.3360	0.005890	0.3534	101.0	0.3358	0.005887	0.3532	100.9

Footnotes:

1. Spiking Material (M) refers to the material, which is actually spiked, i.e., a metal solution, a TiO_2 and/or metal dispersion, and/or a POHC. Spiking Species (S) refers to the portion of the Spiking Material, which is of specific interest in meeting the test objectives, i.e., the elemental metal(s), ash, POHC(s), Cl^-, etc.
2. Concentration refers to the correction for the concentration of the compound of interest in the Spiking Material assuming compound has 100% purity. If we consider for example TiO_2 in a TiO_2 Dispersion, we would divide the net weight of TiO_2 present in the dispersion assuming 100% purity (i.e., no correction for moisture content) and enter that mass fraction in the Concentration column for Ash. A similar process would apply for a metal compound in an aqueous solution, etc. Purity refers to the assay, or purity of the POHC or metal compound, for example, used to make up the solution to the desired concentration. Stoichiometry refers to the stoichiometric content of the specie of interest in the compound, for example the Cl^- content in Perc or the elemental metal content in a metal compound (***ESS*** uses the Merck Index as our primary source of Stoichiometry Correction).

   (Specie) indicates the specie concentration (usually expressed as Lb Specie/Lb Material, or mass fraction) and is defined as the mathematical product of the three previously described corrections: (Specie) = Concentration x Purity x Stoichiometry. (Specie) is used as an overall correction factor to convert the Spiking Material spiking rate, which is determined from field spiking rate measurements to the corresponding Spiking Specie spiking rate, which is of interest to our clients and their corresponding regulatory agency representative(s).

   Usually, all four of the "correction" terms are expressed as mass fractions. However, Concentration and (Specie) will occasionally be expressed as g S/Lb Material or similar units appropriate for a specific test circumstance.
3. Spiking Material (M) spiking rate without (Specie) correction. Calculated from the Spiking Log Sheets in Attachment IV.C.
4. Spiking Specie (S) spiking rate, i.e., after (Specie) correction.
5. Relative Spiking Rate Expressed as a percent of the corresponding Target Spiking Rate (See Table 1).

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Table 4 Average, Absolute & Relative Spiking Rate Results for TC #1, Run #3 Based on: 1. The Weight Loss vs. Time, & 2. The MFM Methods of Measuring Field-Spiking Rates

TC #/ Run #	Spiking[1]:		Corrections[2] for:				1. Spiking Rate by Weight Loss vs. Time				2. Spiking Rate by Mass Flow Meter			
							Absolute Spiking Rate			Relative SR[5]	Absolute Spiking Rate			Relative SR[5]
	Species[1], S	Material[1], M	Concentration[2]	Purity[2]	Stoichiometry[2]	(Specie)[2]	Lb M/Min[3]	Lb S/Min[4]	Lb S/Hr[4]	% Target	Lb M/Min[3]	Lb S/Min[4]	Lb S/Hr[4]	% Target
TC #1:														
Run #3	MCB	MCB	1.0000	0.999976	1.0000	0.999976	0.5867	0.5867	35.20	100.6	0.5841	0.5841	35.05	100.1
Run #3	Perc	Perc	1.0000	0.99974	1.0000	0.99974	0.5830	0.5828	34.97	99.92	0.5809	0.5807	34.84	99.56
Run #3	Organics Solution:													
Run #3		Methylene Chloride	0.1951	0.9999	1.0000	0.1951	0.6838	0.1334	8.005	100.1	0.6788	0.1324	7.946	99.33
Run #3		Ethylene Glycol	0.1951	0.9998	1.0000	0.1951	0.6838	0.1334	8.005	100.1	0.6788	0.1324	7.946	99.33
Run #3		Toluene	0.4146	0.9999	1.0000	0.4144	0.6838	0.2834	17.00	100.0	0.6788	0.2813	16.88	99.28
Run #3		Naphthalene	0.1951	0.9993	1.0000	0.1950	0.6838	0.1333	8.000	100.0	0.6788	0.1324	7.942	99.27
Run #3	Metals Solution:													
Run #3		Pb	0.007989	1.0000	0.6256	0.004998	0.3320	0.001659	0.09957	99.56	0.3313	0.001656	0.09935	99.35
Run #3		Cr^{III}	0.134842	1.0008	0.1299	0.01753	0.3320	0.005820	0.34919	99.77	0.3313	0.005808	0.34846	99.56

Footnotes:

1. Spiking Material (M) refers to the material, which is actually spiked, i.e., a metal solution, a TiO_2 and/or metal dispersion, and/or a POHC. Spiking Species (S) refers to the portion of the Spiking Material, which is of specific interest in meeting the test objectives, i.e., the elemental metal(s), ash, POHC(s), Cl^-, etc.
2. Concentration refers to the correction for the concentration of the compound of interest in the Spiking Material assuming compound has 100% purity. If we consider for example TiO_2 in a TiO_2 Dispersion, we would divide the net weight of TiO_2 present in the dispersion assuming 100% purity (i.e., no correction for moisture content) and enter that mass fraction in the Concentration column for Ash. A similar process would apply for a metal compound in an aqueous solution, etc. Purity refers to the assay, or purity of the POHC or metal compound, for example, used to make up the solution to the desired concentration. Stoichiometry refers to the stoichiometric content of the specie of interest in the compound, for example the Cl^- content in Perc or the elemental metal content in a metal compound (***ESS*** uses the Merck Index as our primary source of Stoichiometry Correction).

   (Specie) indicates the specie concentration (usually expressed as Lb Specie/Lb Material, or mass fraction) and is defined as the mathematical product of the three previously described corrections: (Specie) = Concentration x Purity x Stoichiometry. (Specie) is used as an overall correction factor to convert the Spiking Material spiking rate, which is determined from field spiking rate measurements to the corresponding Spiking Specie spiking rate, which is of interest to our clients and their corresponding regulatory agency representative(s).

   Usually, all four of the "correction" terms are expressed as mass fractions. However, Concentration and (Specie) will occasionally be expressed as g S/Lb Material or similar units appropriate for a specific test circumstance.
3. Spiking Material (M) spiking rate without (Specie) correction. Calculated from the Spiking Log Sheets in Attachment IV.C.
4. Spiking Specie (S) spiking rate, i.e., after (Specie) correction.
5. Relative Spiking Rate Expressed as a percent of the corresponding Target Spiking Rate (See Table 1).

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Table 5 Summary Spiking Rate Results

Spiking Specie, S	Target Spiking Rate, Lb S/Hr	1. Weight Loss vs. Time Method				2. Mass Flow Meter Method				Ave_2/Ave_1 x 100%, %
		Run #1	Run #2	Run #3	$Average_1$	Run #1	Run #2	Run #3	$Average_2$	
MCB	35	99.46	100.1	99.99	99.85	98.57	100.1	100.1	99.59	99.74
Perc	35	100.7	100.4	99.92	100.3	99.85	99.83	100.0	99.89	99.59
CH_2Cl_2	8	100.6	100.2	100.1	100.3	99.68	100.1	100.0	99.93	99.63
Ethyl Glycol	8	100.6	100.2	100.1	100.3	99.68	100.1	100.0	99.93	99.63
Toluene	17	100.5	100.1	100.1	100.2	99.63	100.1	99.95	99.89	99.69
Naphthalene	8	100.6	100.1	100.1	100.3	99.63	100.1	99.95	99.89	99.59
Pb	010	100.5	100.8	99.86	100.4	99.11	100.7	99.95	99.92	99.52
Cr^{III}	0.35	100.6	100.9	99.97	100.5	99.20	100.8	100.1	100.0	99.50
	Average =	100.4	100.4	100.0	100.3	99.42	100.2	100.0	99.87	99.61

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4.0 QA/QC Results and Discussion

Quality Infrastructure: As a part of ***ESS'*** ISO 9001:2000 Quality Management System (**QMS**) and as a means of ensuring that client spiking requirements are met or exceeded on every spiking project, ***ESS*** has developed an extensive and fully integrated quality infrastructure, which includes:

1. A comprehensive, project-specific Project Plan for each spiking project,
2. Ongoing classroom & OJT training;
3. The most extensive fleet of highly accurate & reliable equipment in the spiking industry including: (a) materials preparation equipment, (b) metering pumps, (c) weigh scales, (d) MFMs, (e) computer based spiking rate control & data acquisition equipment, (f) extremely flexible, steam-heated, organic HAP vaporizers, and other spiking equipment plus both general & specialized tools, supplies, & support systems; and
4. Conducting a thorough, critical assessment of each spiking project:
   A. To evaluate the adequacy of ***ESS'*** quality infrastructure to consistently meet or exceed all client spiking requirements,
   B. To confirm adherence of ***ESS*** efforts to the **QMS** requirements, and
   C. To identify & implement refinements to the quality infrastructure based on actual project results[11].

QA/QC Assessment Results: All applicable aspects of ***ESS'*** QMS System and Project Plan were implemented for this project including the post-project (pre-report) QA/QC assessment with the following findings:

1. Each applicable aspect of the Project Plan was initiated by the ***ESS*** Project Manager (PM) & implemented by the Field Services Manager (FSM) &/or PM. Many of the pages of this documentation package contain useful information concerning the details of project planning, preparation, & execution and are provided in appropriate sections of Attachments I, II, III, & IV for convenient reference and project documentation reasons.
2. Spiking materials were prepared to tight compositional tolerances and consistent with the client's requirements. Manufactures' CoAs and related QA documentations are provided in Attachment II for all eight spiking species. ***ESS*** prepared Certificates of Composition (CoCs) for the Metals & Organics Solutions based on this information and included them in Attachment II.
3. ***ESS*** utilized two methods for measuring & controlling field-spiking rates during this project: (a) the Weight Loss vs. Time Method, & (b) the MFM Method. In both cases, all measurements were taken with equipment for which calibrations were recently verified with NIST traceable standards.
4. The equipment required to successfully meet the on-site spiking requirements was: (a) selected, (b) operability verified before mobilization to the test site, (c) set-up & tested at the test site prior to beginning the test, and (d) met all client spiking requirements.
5. ***ESS'*** personal were fully trained, equipped, and able to meet all project requirements.
6. All ***ESS*** activities related to the satisfaction of client's spiking requirements for this project were completed without work related illness, accident, reportable incident, property loss, or mishap of any kind.
7. All client defined spiking requirements were fully satisfied.
8. No significant omission or deficiency in ***ESS'*** Quality Infrastructure was observed.

11. During the 32+ months since ***ESS*** formally implemented the first component of our QMS, ***ESS*** has made at least one improvement to the Quality Infrastructure as a result of the lessons learned from each spiking project completed.

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5.0 Conclusions:

1. As part of its ISO 9001:2000 Quality Management System, ***ESS*** has developed thorough, rigorous, and effective procedures for planning, preparing for, executing, documenting, and reporting the results of every spiking project. These procedures were consistently implemented during the execution of each phase of the March 2006 WeStates Parker, AZ PDT Project.

2. In the preparation of this report, ***ESS*** conducted a thorough assessment of every aspect of ***ESS'*** efforts, which led to the successful completion of the on-site spiking activities and report preparation described herein. This "audit" included review of the:
   (a) Spiking materials preparation,
   (b) Other pre-mobilization preparations,
   (c) Equipment selection, testing, and test performance,
   (d) Demonstrating field spiking rate accuracy through:
      1. Measuring device calibration & calibration verification results,
      2. Certification (with NIST traceability) of measurement device calibration standards, and
      3. All of the calculation steps necessary to produce the spiking rate results.

3. The spiking results reported herein have passed every QA/QC test.

4. As a result of the findings from this review, ***ESS*** believes the spiking rate results presented in Section 3.0 to be true, accurate, and representative of the spiking activities which occurred during the March 2006 WeStates Parker, AZ PDT Project.

W R (Bill) Schofield, PhD, PE — Date
ESS Project Manager

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ATTACHMENTS

Attachment I Original Spiking Plan, Preparatory Work Instructions, & Check Lists:

A. Spiking Plan: Spiking Species, Materials, Rates, & Durations, and Schedule;
B. Test Manager Spiking Orders to ***ESS***; and
C. Preparatory Check Lists, Work Instructions, Worksheets, & Other Project Preparation Documentation.

Attachment I Original Spiking Plan, Preparatory Work Instructions, & Check Lists:

A. Spiking Plan: (1) Spiking Species, Materials, Rates, & Durations, & (2) Test Schedule;

IV.C. Spiking Plan: Spiking Species, Materials, Rates, and Durations

Spiking Specie	Spiking Material	Spiking Rate, Lb/Hr		Pump Assignment[1]	Spiking Duration[2], Hrs	Quantity of Specie/Mat'l Reqr'ed[2]/ Lb S/Lb M/ # Drums M Provided[2],
		As Specie	As Mat'l			
POHCs:						
MCB	MCB	35	35	Neptune 11 gph #3	32	1,120/1500/3-500 [Net] Lb Drums
C_2Cl_4	C_2Cl_4	35	35	LMI 4 gph #10	32	1120/1400/2-700 [Net] Lb Drums
Metals:						
Pb	Pb/Cr^{III} Solution	.1	20	LMI 4 gph #7	32	3.2/640/1-640 [Net] Lb Drum
Cr^{III}	Pb/Cr^{III} Solution	.35	20	LMI 4 gph #7	32	11.2/640/1-640 [Net] Lb Drum
Organic Mixture:						
	Organic Mixture		41	Neptune 18 gph #4	32	1312Lb-2 @ 451[Net] Lb Drum1@ 410 [Net] Lb Drum
Toluene		17			32	
CH_2Cl_2		8			32	
Naphthalene		8			32	
Et Glycol		8			32	

Footnotes: 1. *ESS* will provide two (2) pumps [one (1) 11 gph VS Neptune, & one (1) 18 gph Vs Nepune pumps [including two spare pumps (one for each size pump used)], and four (2) weigh scales with two spares. Spare equipment is provided by *ESS* for enhanced reliability at no charge].

2. Spiking durations based on 12 Hrs on Test Days #1 & #2.

Table 3 Project Definition: Project Schedule, Labor & Travel Requirements, and Cost Detail

Test Day Setup = 0	Project Related Activity:	Travel & Living Costs: Truck Mileage		Days of Travel & Living Expenses					Tech Labor Man-Hours
		W Eq. Trailer	W/O Eq. Trailer	Eq. Trailer	Meals	Hotel	C Phone	Misc	
-4	Assemble Eq, Tools, & Supplies, Test Equipment, & Pack for Transport	0	60	0	0	0	0	1	6
-3	Travel	550	0	1	1	0	1	1	9
-2	Travel	500	0	1	1	1	1	1	9
-1	Travel	500	0	1	1	1	1	1	9
0	Eq Setup & Testing & Prep Pb & Cr^{III} Solutions	10	10	1	2	1	1	1	16*
1	Testing: Run #1	0	20	1	1	1	1	1	10
2	Testing: Run #2	0	20	1	1	1	1	1	10
3	Testing: Run #3	0	20	1	1	1	1	1	10
4	Contingency Test Day, Decon Eq, Disassemble & Pack, & Travel	500	10	1	2	1	1	1	23*
5	Travel	500	0	1	1	1	1	1	9
6	Travel	550	0	1	1	1	1	1	9
7	Re-Stock Equip into Inventory	0	0	1	0	0	0	1	4

* Local Arrangements Have Been Made for Assistance During Equipment Set-Up & Take-Down Periods.

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Attachment I Original Spiking Plan, Preparatory Work Instructions, & Check Lists:
B. Test Manager Spiking Orders to ***ESS***; and

IV.E. Client Test Manager's Spiking Orders[1] to *ESS*:

Section I Initial Spiking Orders[1]:

Spiking:		Spiking Rate, Lb/Hr		Pump Type/Size	Spiking Duration, Hrs	Specie/Mat'l Reqr'ed/ Mat'l Provided, Lb/Lb/# Drums
Specie	Material	As Specie	As Mat'l			
POHCs:						
MCB	MCB	35	35	Neptune #3	32	1,120/1500/3-500 [Net] Lb Drums
C_2Cl_4	C_2Cl_4	35	35	LMI #10	32	1120/1400/2-700 [Net] Lb Drums
Metals:						
Pb	Pb/Cr^{III} Solution	.1	20	LMI #7	32	3.2/640/1-640 [Net] Lb Drum
Cr^{III}	Pb/Cr^{III} Solution	.35	20	LMI #7	32	11.2/640/1-640 [Net] Lb Drum
Organic Mixture:						
	Organic Mixture		41	Neptune #4	32	1312Lb-2 @ 451[Net] Lb Drum1@ 410 [Net] Lb Drum
Toluene		17			32	
CH_2Cl_2		8			32	
Naphthalene		8			32	
Et Glycol		8			32	

Approved by Client/Test Manager: **Date:** / /200

Section II Revised Spiking Orders[2]:

Revision 1:

Approved by Client/Test Manager: **Date:** / /200

Revision 2:

Approved by Client/Test Manager: **Date:** / /200

Revision 3:

Approved by Client/Test Manager: **Date:** / /200

Section III Critique, Suggestions, and Comments[3]:

by Client/Test Manager: **Date:** / /200

Footnotes:
1. Section I contains ***ESS'*** understanding of the spiking requirements (Spiking Orders) for this test. Please review, revise (as necessary), and initial/date to indicate that the Spiking Orders (as revised) are correct.
2. Section II is provided for field revisions to the Spiking Orders by the Client/Test Manager, as needed. Please document the required changes, and initial/date the new orders.
3. Please provide a critique of ***ESS'*** performance on this test, offer suggestions for improving the value of our products and services to you, and/or (if warranted) identify aspect(s) of our products and services with which you are pleased.

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Attachment I Original Spiking Plan, Preparatory Work Instructions, & Check Lists:

C. Preparatory Check Lists, Work Instructions, Worksheets, & Other Project Preparation Documentation.

III. Phases III & IV Prep Plan Transmittal Completeness Checklist & Acceptance Form:			
Project Prep Plan Components by Phase/Sub-Phase:		**Appl/Att'ed?**	**Accepted?**
Project Phase III.A.	Overall Phase III SOP & Checklist	✓	✓
Project Phase III.B.	Materials Preparation Instruction Package	✓	✓
Project Phase III.C.	Equipment Prep Package	✓	✓
Project Phase III.D.	Product Release & Prepare for Shipping Package	✓	✓
Project Phase III.E.	Pre-Travel Checklists	✓	✓
Project Phase IV.	Spiking Plan	✓	✓
Special Test Specific Conditions &/or Requirements:			
Other Information (specify):			
Prep & Approved by *ESS* PM:	**Date:**	**Accepted by *ESS* FSS:** RSD	**Date:** 3/13/2006

III.A. Overall Phase III Preparation SOP & Checklist:	Done?
Materials Purchase &/or Prep:	
Carefully review the "Direct Ship" List & Materials Prep Package (III.B, B.1 thru B.6).	✓
Co-ordinate w ***ESS* PM** & Spiking Coordinator (**SC**) for timely delivery of: (1) "Direct Ship" Materials to the Test Site, (2) Full QA Doc Package (CoA, Shipping Papers, & Invoice with matching Lot #s) to ***ESS***, & (3) Notification of the client's on-site representative that the materials are being shipped w ETA.	✓
Compare the quantity of each raw material required with the corresponding Raw Materials Inventory Sheet. Place orders for raw materials [as necessary] using the spiking materials Purchase Order Preparation SOP [With full QA Doc Package, as applicable].	✓
When all required raw materials, equipment, & manpower are available, prepare these materials using applicable SOPs, forms, &/or work sheets (III.B.3 thru B.6). Record all weights and document completion of each procedure step.	✓
Provide all the materials prep information to the PM and review this QA Package together. If the materials, as prepared, will meet all client requirements, the PM will tentatively release the materials for shipment and will provide information for preparing the materials for shipment (III.D. & D.1). Otherwise further plans will be prepared jointly to modify the materials so that they will meet the client's requirements. Make such revisions and review the results with the PM.	✓
After the material has been tentatively released, prepare the materials for shipment to the test site in accordance with the materials labeling & shipping instructions (III.D.2, 3, & 4). Review preparations with PM & get final product release.	✓
Coordinate client notifications & shipping with the ***ESS* SC**, as appropriate.	✓
Equipment Prep:	
Carefully review the Equipment Prep (III.C) and Spiking Plan (IV) Packages provided herein.	✓
Verify Operability of Assigned Equipment (e.g., verify that each pump, weigh scale, mass flow meter, and computer equipment assigned to this project including spares is operational and is capable of performing the assigned function under the project specific material, through-put, and back pressure conditions).	✓
Assemble & load all required equipment, supplies, tools, documentation, etc. for transport to the test site.	✓
Co-ordinate identification & satisfaction of any special requirements with the *ESS* PM & SC:	
Off-site safety training & documentation.	✓
Special medical monitoring &/or drug screens.	✓
Special equipment such as EP/IS classified equipment. **Bring Pressure Feed System.**	✓
Unusual operating, safety, test conditions, etc.	✓
Pre-Travel Checklist:	
Complete the final project prep checklist (III.E.1).	✓
Complete the Truck & Trailer Inspection & the Pre-Travel Safety Inspection Checklist (III.E.2).	✓
Preparations Were Completed per these Mat'ls & Equip Prep Instructions & SOPs:	

III.B. Checklist: Project Plan, Materials Prep Component:		Applicable?	Accepted?
III.B.1 "Direct Ship" Materials List		✓	✓
III.B.2 Materials Prep Instructions & Checklist		✓	✓
III.B.3 Materials Prep Instructions, Raw Materials Weights		✓	✓
III.B.4 Multi-Packet Materials Prep Instructions		NA	N/A
III.B.5 Applicable Weigh Scale Calibration Verification Log Sheets		✓	✓
III.B.6 DM & Dispersion Prep SOP & Worksheet		NA	N/A
III.B.1 "Direct Ship" Materials List (Get MSDSs from Spiking Mat'ls Tech Info Files & Review.)	Required?	Ordered?	Received?
1.	✓	✓	✓
2.			
3.			
Prepared & Approved by *ESS* PM:	**Date:**	**Accepted by *ESS* FSS:** RSD	**Date:** 3/13/2006

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III.C. Equipment Operability Verification Information Transmittal & Acceptance Form:				
Project Plan Component:			Attached?	Accepted?
III.C.1. Equip Operability Verification SOP			✓	✓
III.C.2. Equip Operability Verification Test Conditions, Checklist, & Certification Form			✓	✓
IV.F. Project Equip Assignments (included with phase IV packet)			✓	✓
Prepped/Approved by PM:	Date:	Accepted by FSS:		Date: 3/14/2006

III.C.1. Equipment Operability Verification & Pre-Mobilization Preparation SOP

*The **purpose** of this SOP is to confirm that: (1) Each piece of equipment assigned to this project is in good operating condition, (2) the pumps have the capability to perform the the assigned spiking function with the project specific spiking material and at the project specific spiking rate & back pressure. [This verification is especially critical for pumps in dispersion duty], and (3) the assigned equipment when combined into a spiking system performs as intended.*

Scale Check-Out: All testing, including equipment testing begins with NIST Traceable Standards.

1. Verify the operability and calibration of each assigned weigh scale & its mated indicator by calibrating the scale/indicator to the maximum weight expected to be seen during this project (III.C.2) using our standard field scale calibration procedure, and ***ESS'*** NIST traceable standards.
2. Then verify the calibration using the weight build up & break down procedure & Logsheet (IV.K.3).
3. If the scale & indicator set is found to be out of calibration, recalibrate it using ***ESS'*** Field Scale Calibration SOP including the Corner Test Procedure (IV.I) & Logsheet (IV.K.2), if necessary.
4. Document the results on the Equipment Operability Checklist & SOP (III.C.2).
5. *If after appropriate calibration, & adjustment, a given scale (& associated indicator) fails to perform to **ESS'** standards (within ± 0.1 Lb for 90+% of the 50 Lb increment calibration verification readings), then[1] (see footnote 1 below).*

Pump Assignments & Operability Verification: The spiking pumps selected for & assigned to this project are identified in Section IV.F of the Spiking Plan. These pumps were selected because:

- The pump materials of construction are chemically compatible with the spiking material,
- The pump has the capacity to:
  - Deliver the assigned spiking material (liquid or dispersion),
  - At the assigned spiking rate (Lb M/Hr)
  - Against the existing the back pressure.

However, ***ESS*** has found that the only method to be certain that a spiking pump will perform as intended is to verify its operability at the project specific test conditions prior to mobilizing to the test site.

Follow the following steps to confirm that each pump identified in III.C.2 is completely functional and actually delivers the flow specified against the back pressure specified.

1. Set-Up and operate ***ESS'*** **Dynamic Test Stand** as follows:
2. Test Material:
   a) Use water as the pumping fluid for pumps assigned to aqueous solution or organic liquid duty, or
   b) Use the actual dispersion for pumps assigned to dispersion duty.
3. Test Conditions:
   a) Back Pressure: Adjust the test stand back pressure setting to match the specified (III.C.2) back pressure value.
   b) Capacity: Adjust the pump rate setting until the specified (target) spiking rate value is achieved.
4. Document the results on the Equipment Operability Verification Checklist (III.C.2).
5. Notify the ESS PM if any equipment does not achieve the required thru-put &/or pressure levels.

Mass Flow Meter Check-Out:

1. Verify that each assigned mass flow meter (MFM) is operational and its accuracy is verified against one or both of the following two methods:
   a) Weigh scale [with NIST Traceable calibrations] based weight gain vs. time method, or
   b) Comparison to our factory calibrated and frequently verified [via method a above] reference MFM.
2. Document the results of these tests on the Equipment Operability Checklist & SOP (III.C.2).

Special Supplies/Equipment/Tools: Based on the Spiking Plan provided to you [plus discussions with the ***ESS*** PM & the client's representative, as needed], prepare a checklist of any *special hardware/supplies/equipment/tools*[2] [e.g., valves, check valves, pressure gauges, tubing, quick connects, spare parts, spill kit supplies, PPE, tools, supplies, etc.] required to successfully meet the clients spiking requirements. Use this checklist to place orders for any items not instock, assemble for packing in the ***ESS*** equipment trailer, and confirmation that each item has actually been packed &/or loaded into the ***ESS*** equipment trailer for transport to the test site.

If problems occur with any aspect of these preparatory efforts which you can not address, notify the PM.

---

[1] *If any equipment fails to meet **ESS'** accuracy & operational reliability requirements, then that equipment:*
   a) Must be removed from the active equipment fleet,
   b) A RED warning tag attached to it, and
   c) That scale/indicator set will not be used for client projects [not readmitted to the active equipment fleet) until it has been validated to be accurate & operationally reliable.

[2] ***ESS*** maintains large array of tote bins in which all standard equipment, tools, supplies, etc. are stored and transported. This check list refers to special items beyond what we take on all ***ESS*** projects.

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III.C.2. Equipment Operability Verification Checklist & Certification

Weigh Scale (ID#)	Calibrate To, Lb	Verified ✓	Standards Used	Corner Test Req'd, ✓	Corner Test Adj'ed, ✓	Count #Δ = ± 0.X Lb 0.0	0.1	0.2	0.3	>0.3	Cert'ed[1] ✓
1. F-1	500	✓	ESS#								✓
2. F-2	600	✓	ESS#								✓
3. F-3	600	✓	ESS#								✓
4. F-4	800	✓	ESS#								✓
5. F-5*	600	✓	ESS#								✓
6. F-6*	800	✓	ESS#								

Footnote: 1. Certify a scale if 90% of its deviations from the weight standards are equal to or less than ± 0.1 Lb (Δ ≤ ± 0.1 Lb).

Equipment Identification: Pump Capacity/Capability/Name (ID#)	Project Specific Conditions: Mat'l for For Actual Test:	Mat'l for For Verifying Op:	P, psig	Rate, Lb/Min	Verify Operability (✓) Tested ✓	Passed ✓	Cert'ed ✓
1. Neptune 11 gph #3	✓	H_2O			✓	✓	✓
2. Neptune 18 gph #4	✓	"			✓	✓	✓
3. LMI #7	✓	"			✓	✓	✓
4. LMI #10	✓	"			✓	✓	✓
5. Neptune #5	✓	"			✓	✓	✓
6. LMI #8	✓	"			✓	✓	✓

Footnote: 1. EP = Explosion Proof &/or Intrinsically Safe. VS = Designed for Pumping High Viscosity Fluids.

MFM Size (ID#)	Operability Verification[1] Method Used (✓): Wt Change vs. Time	Ref MFM	Mat'l	Tested ✓	Meas Uncertainty Demo'ed, ±%	Cert'ed ✓
1. MFM10-1	✓		H_2O	✓		✓
2. MFM10-2	✓		H_2O	✓		✓
3. MFM10-3	✓		H_2O	✓		✓
4. MFM10-4	✓		H_2O	✓		✓
5. MFM10-5*	✓		H_2O	✓		✓
6.			H_2O	✓		✓

Footnote: 1. Verify @ project specific spiking rates.

Spike Manager ©	TC Setups?	Controls?	Data Logging?	Overall System	Cert'ed ✓
System #1	✓	✓	✓	✓	✓
System #2	NA				

* Designated Spare

Certified By *ESS* FSS: [signature] Date: 3/31/2006

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III.E.1. Pre-Travel Project Prep (PPE, Tools, Supplies, & Equipment) Checklist		Done?
Verify that the following project related items are packed &/or have been addressed:		
1.	All PPE required for the project: • ***ESS*** Standard PPE: hard hat, safety glasses, leather gloves, steel toes boots, chemical gloves, tyvek suite, face shield, steel toe rubber boots, half face respirator and both PM & Organic Vapor Canisters; and • Project specific PPE, & Medical Monitoring, if any.	✓
2.	All pumps assigned for this job including spares have been tested, & loaded with spare parts kits.	✓
3.	Assigned scales including spares with their mated indicators have been tested, & loaded with spare parts kits and weight standards.	✓
4.	Assigned Micro Motion Meters including spares & repair kits.	✓
5.	Camile System with Laptop PC & memory stick. Verify that Spike Manager © TC set-ups and Camile/Laptop hardware have been checked.	✓
6.	Extension cords, ground fault protectors, and equipment grounding cabling w clamps.	✓
7.	Drumstands, hoses including spares, drum bung feed & recycle fittings, Tee fitting for tandem scale configuration if needed, chairs and folding table, tarps/tent, tool box, spill pads, magnetic ***ESS*** identification signs, and 5 to 10 gallons of MSO.	✓
8.	If dispersion will be used: (a) dispersion suction & discharge hoses, (b) dedicated dispersion pumps, (c) dispersion pressure feed assembly & air compressor, and (d) dispersion mixer motor & blades,.	✓
9.	Special project specific equipment.	✓
10.	Job specific documentation has been loaded along with SOPs, Spiking Orders, Spiking Plan, Log Sheets, DOT Documentation Kit, ***ESS*** IQ plus Operation Manual, memory stick for backup data files, clipboards, calculator, and test clock.	✓

III.E.2. Pre-Travel Safety Inspection Checklist:		Done?
Pre-Travel Tow Vehicle Checklist: Inspect/check and correct as needed:		
1.	Fluid levels are within safe operating range.	✓
2.	Windshield and side windows are clean.	✓
3.	Tire pressure (per owner's manual) and tire condition/tread depth.	✓
4.	Lights (Head lights, turn signals, brake lights, and reverse lights).	✓
5.	Towing ball for proper size and tightly secured to hitch.	✓
6.	Receiver hitch for unusual wear and hitch pin installation.	✓
Pre-Travel ***ESS*** Equipment Trailer Checklist: Inspect/check and correct as needed:		
1.	Tire pressure (per owner's manual) and tire condition/tread depth.	✓
2.	Wheel lug nuts for tightness.	✓
3.	Coupler/ball for: (a) wear/condition, (b) proper seat, and (c) snug coupler/ball lock.	✓
4.	Safety chains for wear/condition and securely fastened to tow vehicle.	✓
5.	Breakaway battery charge (Pull switch pin & check light).	✓
6.	Even load distribution.	✓
7.	Load secured to E-Tracks (in floor) &/or D-Rings (along the walls).	✓
8.	Doors secured.	✓
9.	Lights (Turn signals and brake lights).	✓
10.	Wheel bearings (Grease before traveling and at 1,000 mile intervals).	✓
Certification of Checklist Completion:		Date: 3/31/2006

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Attachment II Spiking Material Composition Information:

A. Mono-Chlorobenzene (MCB) & Perchloroethylene (Perc)
B. Solution QA Information:
   1. Verification of Weigh Scale Calibration, &
   2. Mass of Raw Material Used in Organic & Metal Solutions;
C. Organic Solution:
   1. Dichloromethane,
   2. Ethylene Glycol,
   3. Toluene &
   4. Naphthalene; and
D. Metals Solution:
   1. Lead Nitrate [$Pb(NO_3)_2$] &
   2. Chromium Nitrate [$Cr(NO_3)_3 \cdot 9H_2O$].

Attachment II Spiking Material Composition Information:

A. Mono-Chlorobenzene (MCB) & Perchloroethylene (Perc)

PPG INDUSTRIES, INC.

CERTIFICATE OF ANALYSIS AND NOTICE OF SHIPMENT

DATE ISSUED 03/15/2006 02:36 P.M.

BY MARK J. SINCLAIR
CUSTOMER QUALITY ASSURANCE DEPT
(304-455-6701)

SHIP TO UNIVAR USA INC
777 BRISBANE
HOUSTON TX 77061

FAX: 713-644-1139

DATE SHIPPED	PPG ORDER NO.	CUSTOMER ORDER NO.	CUSTOMER PRODUCT CODE
03/15/2006 TRUCK	210897-5	HS612336	

FREIGHT	TOTAL WEIGHTS (Bulk Only, Billing Shown If Applicable)			
	GROSS	TARE	NET	BILLING
18396 PPD				

ROUTE PRIJ001 PRIME INCORPORATED

This is to certify that the products shipped below by PPG Industries, Inc. meet or exceed all analysis standards.

PRODUCT DESCRIPTION: MONOCHLOROBENZENE (500 LB DRUM)
501900

LOT NUMBER: A136 QUANTITY: 16

PROPERTY	UNIT OF MEASURE	RESULT	PPG SPECIFICATIONS MINIMUM	MAXIMUM
MCB	%WT	99.9976	99.90	
H2O	%WT	0.0046		0.0200
APHA COLOR	AS IS	10		30

PRODUCT DESCRIPTION: CAU SODA PELS (50 LB BAG)
239870

LOT NUMBER: A296 QUANTITY: 600

PROPERTY	UNIT OF MEASURE	RESULT	PPG SPECIFICATIONS MINIMUM	MAXIMUM
NAOH	%WT AS IS	98.88	96.0	
NA2O	%WT AS IS	76.88	74.4	
NA2CO3	%WT	0.47		1.60
NACL	%WT	0.00		2.20
FE	PPM	1		15

MARK J. SINCLAIR

SIGNATURE

UNIVAR

VOLTAKIS NOW UNIVAR USA
3333 BRIDGELAND DR
COST WISE HS
SHIP WISE HS

777 BRISBANE STREET
HOUSTON TX 77094
713-643-4401

ORDER DATE	03/17/06
ORDER NUMBER	H6324895

CUSTOMER P.O. NUMBER: 032406UNI

CUSTOMER NO: 530389 001

CUSTOMER NO: 530389

SHIP TO:
ESS INC
ATTN: TSR - ALWAYS
VERIFY DELIVERY
ADDRESS
LA PORTE , TX 77571

BILL TO:
ESS INC
1200 HIGHWAY 146 STE #170
LA PORTE , TX 77571-6131

SHIP DATE	SHIP VIA	FREIGHT TERMS	IN. SALES
03/24/06	WILL CALL OBL	COLLECT	EIA0

F.O.B.	DELIVERY CONTACT	DELIVERY PHONE
SHIP POINT	SCOTT	281-471-2071

CREDIT TERMS	OUTSIDE SALES	TAX %	DEPT
NET 30 DAYS	MATT CARTER		04

PRODUCT DESCRIPTION	QTY. SHIPPED	QUANTITY B/O
** ORDER MESSAGES ** MUST DELV ON SHORT TRAILER ** LIFTGATE & PALLET JACK REQUIRED **		
001 MONOCHLOROBENZENE 501900 PPG	3 DR	0
** ** TH ST DR 500 LB DR		

** NOTE: SIGNATURE ON THE RECEIVED BY LINE, BELOW, ALSO ACKNOWLEDGES RECEIPT OF A MATERIAL SAFETY DATA SHEET(S) FOR HAZARDOUS CHEMICALS IN THIS SHIPMENT.

LOT# A 136

PACKING

NO. OF PKGS = TOT NET WT = 1500 TOT GRS WT = 1620

This form is printed on recycled paper and is recyclable

DELIVERED BY	FREIGHT AMT.	TOTAL MDSE.	TOTAL QTY.	RECEIVED BY

The Reilzon Company

PERC CoA

Certificate 1983685 The Dow Chemical Company Page 1

Date: 05/14/2004 Certificate of Analysis

Quality Assurance
CHEMCENTRAL SOUTHWEST LP Fax:
11235 FM529
HOUSTON TX 77007-0000 UNITED STATES

Cust P.O.: 216193 Dlvy Note: 68695647 10

Material: PERCHLOROETHYLENE INDUSTRIAL Spec: 00059009-S
Cust Mtl:
Vehicle: 662
Ship from: THE DOW CHEMICAL COMPANY PLAQUEMINE LA UNITED STATES

This material meets the requirements of the specification.

Feature	Units	Results T1030514	Limits Minimum	Maximum
Water	ppm	14	----	30
Color, Pt-Co	-	5	----	15
Non Volatile Residue	ppm	3	----	10
Alkalinity (as NaOH)	ppm	20	15	30
Perchloroethylene	%	99.974	99.900	----

Typical Properties:
Specific Gravity, 25/25: 1.618 - 1.622
Source of Data:
Non Volatile Residue (results based on quarterly analysis)

Lot No. 3605260420-6

Plant Quality Coordinator

For inquiries please contact Customer Service or local sales.
English: 800-232-2436 French: 800-565-1255

UNIVAR

P.O. BOX 34325
SEATTLE, WA 98124-1325

www.univarusa.com

ORIGINAL INVOICE

UNIVAR PHOENIX
50 SOUTH 45TH AVENUE
PHOENIX AZ 85043-3907
602-272-3272

Scott
ok?

All Total Amt
Price check
Correct
2/13/06
BJN

CUST. NO./SHIP TO

588315 001
ESS INC.
C/O WESTATES
2523 MUTAHAR
ATTN: MONTE MCCUE
PARKER AZ 85344

6.1.1868 1 MB 0.326 69234S11.XRX
588315
ESS INC.
STE# 170
1200 HIGHWAY 146
LA PORTE TX 77571-6156

FREIGHT TERMS	FOB
PPD & ADD SPECIAL	DELIVERED

INVOICE NO.	INV. DATE	ORDER NO.	CUSTOMER P.O. NUMBER	WAREHOUSE LOCATION	ON TIME DELIVERY
PX-920481	01/27/06	678851	012606UNI	LA GARFIELD	ENGLUND EQUIPMENT CO.

SHIP DATE	TAX EXEMPT NO.	SALES REP.	SALES DEPARTMENT	ENTERED BY
01/27/06		PX UNASSIGNED	INDUSTRIAL CHEMICAL	CANDY FITZGERALD

PRODUCT DESCRIPTION	TAX	QUANTITY ORDERED	QUANTITY SHIPPED B.O.	BILLING QTY./ UNIT PRICE	EXTENDED AMOUNT
318120 PERCHLOROETHYLENE 700 LB DR UNIVAR TECH LIQ NUSTL DR	Y	2.00 DR	2.00 DR	1400.00 1.0100 LB	1414.00
600973 FUEL SURCHARGE 1 EA EA TRANSPORTATION ONLY SPCL CHG **** **** NA	Y	1.00 EA	1.00 EA	1.00 35.0000 EA	35.00
644207 UNIVAR PACKAGE DELIVERY 1 EA EA CHARGE SPCL CHG **** **** NA	N	1.00 EA	1.00 EA	1.00 0.0000 EA	0.00
			MERCHANDISE:		1414.00
			SPECIAL CHG:		35.00
			SALES TAX:		117.3[illegible]
TERMS: NET 30			INVOICE TOTAL:		1566.3[illegible]

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Buyer shall handle, use and dispose of the goods as necessary for the safety and protection of personnel, property and environment, and in accordance with Univar USA's Inc. Recommendations and applicable governmental laws and regulations. Buyer shall deliver the most recent product literature to its customers and maintain a written record of such delivery. Buyer shall only sell or release what, in the sole judgment of Buyer, can handle, use and dispose of the goods safely.

Taxes have not been included unless specifically itemized on the invoice. Buyer shall pay all taxes or other charges related to the goods. The terms of this invoice are not modified by any Buyer purchase order [illegible]

Attachment II Spiking Material Composition Information:

B. Solution QA Information:

1. Verification of Weigh Scale Calibration, &
2. Mass of Raw Material Used in Organic & Metal Solutions;

III.B.5 (d) *ESS* Scale Calibration & Calibration Verification Report

ESS Scale #: L- 3 (15 Lb)

Application: Weigh out Pb. Place in large plastic sample bottles for transport to Westate for on-site preparation of Pb Solution.

Calibration Verification: Date: 3/9/2006			Calibration Verification: Date: 3/10/2006		
+ Test Weight, Lb	- Indicated Weight, Lb	= Deviation, Lb	+ Test Weight, Lb	- Indicated Weight, Lb	= Deviation, Lb
Linearity Check Before: ✓			Linearity Check After:		
0.000	0.000	0.000	0.000	0.000	0.000
7.500	7.500	0.000	7.500	7.500	0.000
15.000	15.000	0.000	15.000	15.000	0.000
7.500	7.500	0.000	7.500	7.500	0.000
0.000	0.000	0.000	0.000	0.000	0.000
Span Check Before: If Applicable			Span Check After:		
Calibration Verification Before: ✓			Calibration Verification After: ✓		
5.000	5.000	0.000	5.000	5.000	0.000
10.000	10.000	0.000	10.000	10.000	0.000
15.000	15.000	0.000	15.000	15.000	0.000
10.000	10.000	0.000	10.000	10.000	0.000
5.000	5.000	0.000	5.000	5.000	0.000

Notes, Comments, Data for the Record:

ESS Technician: Scott Weal Date: 3/10/2006

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III.B.5 (a) *ESS* Scale Calibration & Calibration Verification Report

Shop Weigh Scale #: S- 1 | Calibrate & Verify Calibration to: Lbs

Application: 1. Weigh containers @ Lilly prior to CPT.
2. Set weigh head up for computer logging of weight data.

Pre-Test Calibration Verification: Date: 3/8/2006			Post-Test Calibration Verification: Date: 3/8/2006		
Test Load, Lb	- Indicated Wt, Lb	= Difference, ± 0.? Lb	Test Load, Lb	- Indicated Wt, Lb	= Difference, ± 0.? Lb
Build Up in 50 Lb Increments:			Build Up in 50 Lb Increments:		
0.0	0.0	0.0	0.0	0.0	0.0
50.0	50.0	0.0	50.0	50.0	0.0
100.0	100.0	0.0	100.0	100.0	0.0
150.0	150.0	0.0	150.0	150.0	0.0
200.0	200.0	0.0	200.0	200.0	0.0
250.0	249.9	-0.1	250.0	250.0	0.0
300.0	300.0	0.0	299.9	299.9	-0.1
350.0	349.9	-0.1	350.0	350.0	0.0
400.0	399.9	-0.1	399.9	399.9	-0.1
450.0	450.0	0.0	449.9	449.9	-0.1
500.0	499.9	-0.1	499.9	499.9	-0.1
Break Down in 50 Lb Increments:			Break Down in 50 Lb Increments:		
500.0	499.9	-0.1	500.0	499.9	-0.1
450.0	449.9	-0.1	450.0	449.9	-0.1
400.0	399.9	-0.1	400.0	399.9	-0.1
350.0	350.0	0.0	350.0	349.9	-0.1
300.0	299.9	-0.1	300.0	300.0	0.0
250.0	249.9	-0.1	250.0	250.0	0.0
200.0	199.9	-0.1	199.9	199.9	0.1
150.0	149.9	-0.1	150.0	149.9	-0.1
100.0	99.9	-0.1	100.0	100.0	0.0
50.0	49.9	-0.1	50.0	50.0	0.0
0.0	0.0	0.0	0.0	0.0	0.0

Accuracy Assessment & Comments:

ESS Technician: Scott Neal | Date: 3/8/2006

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III.B.2. Materials Preparation Instructions & Checklist:

Material Description:

General Prep Instructions:

ID SOPs/Instructions/Worksheets: Applicable ? =	✓	Att?	Get	Done?	Notes, Comments, &/or instructions:
1. Review all Mat'ls Prep information for this project.	✓		✓	✓	
2. MSDS(s)	✓		✓	✓	Get MSDSs from Mat'l Tech Files & Read.
3. Materials Prep Instructions, Raw Mat'l Weights (III.B.3)	✓	✓	✓	✓	
4. Multi-Packet Mat'ls Prep Instructions (III.B.4)	NA	NA		N/A	See attached detailed instructions.
5. Scale Calibration & Calib. Verification Report (III.B.5)	✓	✓	✓		
6. DM & Dispersion Prep SOP (III.B.6)	NA	NA		N/A	
7. Shipping Instructions (III.D)	✓		✓	✓	
8. Bill of Lading (w go by)	✓		✓	✓	
9. Container Shipping Label &/or go by	✓		✓	✓	
10. Container Warning Label	✓		✓	✓	
11. Other (ID)	✓		✓	✓	Ground Drums During Mixing Process

Approved by: WDS Date: 3/8/2006 Accepted by: Date: Completed per SOP: RSD Date: 3/16/2006

III.B.3. Materials Prep Instructions, Raw Material Weights, & Prep Worksheet:

Batch Or Drum #									
	Raw Mat'l =	Toluene	CH_2Cl_2	Naphthalene	ET Glycol				
	Lot # =	HS026870341	05-01-0117	031411	05-02-0280				
	Purity =	0.9999	0.9999	0.9993	0.9998				
	Use Scale # =	S-1 (1000 Lb)	S-1 (1000 Lb)	S-1 (1000 Lb)	S-1 (1000 Lb)	L- (Lb)	L- (Lb)		
	Calibrated on =	/ /	/ /	/ /	/ /	/ /	/ /		
	Sub-batch =						A*	B*	C*
1	Target Wt (Lb) =	187.00	88.00	88.00	88.00				
	Actual Wt (Lb) =	187	88.00	88.00	88.00				
	Δ (Lb) =	0.00	0.00	0.00	0.00				
2	Target Wt (Lb) =	187.00	88.00	88.00	88.00				
	Actual Wt (Lb) =	187.00	88.00	88.00	88.00				
	Δ (Lb) =	0.00	0.00	0.00	0.00				
3	Target Wt (Lb) =	170.00	80.00	80.00	80.00				
	Actual Wt (Lb) =	170.0	80.0	80.00	80.0				
	Δ (Lb) =	0.00	0.00	0.00	0.00				
		Pb/Cr Solution							
		$[Pb(NO_3)_2]$	$Cr(NO_3)_3 \cdot 9H_2O$						
1	Target Wt (Lb) =	3.517	59.280						
	Actual Wt (Lb) =	3.515	59.280						
	Δ (Lb) =	0.002	0.00						
2	Target Wt (Lb) =								
	Actual Wt (Lb) =								
	Δ (Lb) =								

* Please Note: Batches may be subdivided for convenience as long as the total weigh of all sub-batches matches the total quantity indicated.

III.B.4. Multi-Packet Materials Prep Instructions: See attached detailed instructions.

	✓?
1. Prepare individually labeled, consecutively numbered, pre-weighted (Lb/g ± Lb/g), heat-sealed packets.	
2. Verify the calibration on the weigh scale daily with *ESS'* NIST traceable standards before each work day & record the results in the attached *ESS* Scale Calibration & Calibration Verification Report (III.B.4)	
3. Place the appropriate quantity of material into the packet. Determine the net weight of each packet and record that exact weight on the label and the Packet Weight Log Sheet in the space adjacent to the packet #.	
4. Initial & date each Packet Weight Lo Sheet and Calibration Verification Log Sheet daily to certify the accuracy of the log.	
5. Keep the packets in numerical order and bundle them in convenient sized batches. Pack the batches into container(s) in reverse numerical order. Mark the containers from #1 to # . Attach one Container Shipping Label (front) and three Haz Mat Container Warning Labels per container (front, back, & top, if indicated in the instructions).	
6. Place the Site Contact Information Package (III.D.4.a) on top of the packets inside of Container #1. Securely fasten the container lid(s). Assemble the containers in a tight group just inside the shop door with Container #1 in front. Place the Transporters Information Package on top of Container #1in clear view. Coordinate shipping with the PM and SC.	

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Project F Phase III.D. Spiking Materials Release for Shipment
Project ID: 2006 Westate CPT. Date Prepared: 3/13/2006

III.D. Tentative Spiking Materials Release & Prep for Transport			
Step #	Activity:	Applicable?	Done?
1	Retrieve the spiking materials identified in Tentative Product Release for Shipment (III.D.1) List.	✓	✓
2	Confirm the number, size, type, weight, etc. of the material shipping containers[0].	✓	✓
3	Prepare Shipping Label(s) & Papers, MSDS(s), DOT Required Container Warning Label(s) per Shipment Prep Instructions (III.D.2).	✓	✓
4	Prep Commercial Invoice, Shipping Papers, etc. per attached Non-Domestic Shipping Papers Instructions (III.D.3.).	✓	✓
5	Prep Multi-Packet Shipments per III.D.4.	✓	✓
6	Apply labels, palletize, shrink wrap, & otherwise prep containers for transporter pick-up.	✓	✓
7	Review preparation with PM & confirm final product release for shipment to client.	✓	✓
8	Co-ordinate shipment, delivery, & client notification with Spiking Coordinator.	✓	✓
9	Other (identify):		
Tentative Product Release for Shipment[1] (PM):			
Confirmation of Pre-shipping Preparations[2] (FSS):			
Final Release for Shipping[3] (PM):			

0. Report any non-conformity to the *ESS* PM immediately.
1. By way of my signature, I am confirming that I: (a) have reviewed the available quality information on this product, (b) believe that it meets all client specifications and requirements, and (c) am tentatively releasing it for shipment to the customer subject only to a final review of the preparations for shipping.
2. By way of my signature, I am confirming that I: (a) have reviewed the available quality information on this product, (b) believe that it meets all client specifications and requirements, and (c) have prepared the materials for shipment as described above and the applicable attachments.
3. By way of my signature, I am confirming that I: (a) have reviewed the available quality information on this product including its preparation for shipment, (b) believe that it meets all client specifications and requirements, and (c) am formally releasing it for shipment to the customer.

III.D.1. Tentative[1] Product Release for Shipment:				
Item #:	Product ID:	Type & # Containers:	Location:	Located?
1				
2				
3				
4				
Tentative Product Release by:		Date:		

1. These spiking materials (products) are approved for shipment with respect to quantity & composition. Final release is subject only to confirmation of proper shipping preparations (Per III.D).

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ESS 1200 Hwy 146 South, Suite 170, LaPorte, Texas 77571 (281) 471-2071 Fax (281) 471-2180 *BSPE@ESSpiking.com*

Attachment II Spiking Material Composition Information:

C. Organic Solution:
   1. Dichloromethane,
   2. Ethylene Glycol,
   3. Toluene &
   4. Naphthalene;

ESS Certification of Composition for WeStates Organic Solution

Spiking Material:	Organic Solution
Spiking Application:	POHC Spiking Source for Westate PDT
Production Date:	3/10/2006
Quantity Produced:	1312 Lb Net Weight Solution
Compositions:	[CH_2Cl_2] = 0.1951, [Ethylene Glycol] = 0.0.1951, [Toluene] = 0.4144, & [Naphthalene] = 0.1950.

Based on the information available to me concerning the manufactures' CoAs for CH_2Cl_2, Ethylene Glycol, Toluene, & Naphthalene and the procedures and equipment ***ESS*** used to establish the quantity of each ingredient used to make the final solution, I certify that the CH_2Cl_2, Ethylene Glycol, Toluene, & Naphthalene concentrations provided above are true and accurate to the best of my knowledge and belief.

Signed: _____ _____

W.R. (Bill) Schofield, PhD, PE — Date

ESS Project Manager

III.B.2. Materials Preparation Instructions & Checklist:

Material Description:

General Prep Instructions:

ID SOPs/Instructions/Worksheets: Applicable ? =	✓	Att?	Get	Done?	Notes, Comments, &/or instructions:
1. Review all Mat'ls Prep information for this project.	✓		✓	✓	
2. MSDS(s)	✓		✓	✓	Get MSDSs from Mat'l Tech Files & Read.
3. Materials Prep Instructions, Raw Mat'l Weights (III.B.3)	✓	✓	✓	✓	
4. Multi-Packet Mat'ls Prep instructions (III.B.4)	NA	NA		N/A	See attached detailed instructions.
5. Scale Calibration & Calib. Verification Report (III.B.5)	✓	✓	✓		
6. DM & Dispersion Prep SOP (III.B.6)	NA	NA		N/A	
7. Shipping Instructions (III.D)	✓		✓	✓	
8. Bill of Lading (w go by)	✓		✓	✓	
9. Container Shipping Label &/or go by	✓		✓	✓	
10. Container Warning Label	✓		✓	✓	
11. Other (ID)	✓		✓	✓	Ground Drums During Mixing Process

Approved by: DJPS Date: 3/8/2006 Accepted by: Date: Completed per SOP: RBD Date: 3/16/2006

III.B.3. Materials Prep Instructions, Raw Material Weights, & Prep Worksheet:

Batch Or Drum #	Raw Mat'l =	Toluene	CH_2Cl_2	Naphthalene	ET Glycol				
	Lot # =	HS026870341	05-01-0117	031411	05-02-0280				
	Purity =	0.9999	0.9999	0.9993	0.9998				
	Use Scale # =	S-1 (1000 Lb)	S-1 (1000 Lb)	S-1 (1000 Lb)	S-1 (1000 Lb)	L- (Lb)	L- (Lb)		
	Calibrated on =	/ /	/ /	/ /	/ /	/ /	/ /		
	Sub-batch =						A*	B*	C*
1	Target Wt (Lb) =	187.00	88.00	88.00	88.00				
	Actual Wt (Lb) =	187	88.00	88.00	88.00				
	Δ (Lb) =	0.00	0.00	0.00	0.00				
2	Target Wt (Lb) =	187.00	88.00	88.00	88.00				
	Actual Wt (Lb) =	187.00	88.00	88.00	88.00				
	Δ (Lb) =	0.00	0.00	0.00	0.00				
3	Target Wt (Lb) =	170.00	80.00	80.00	80.00				
	Actual Wt (Lb) =	170.0	80.0	80.00	80.0				
	Δ (Lb) =	0.00	0.00	0.00	0.00				
		$[Pt(NH_3)_2]$	$Cr(NO_3)_3 \cdot 9H_2O$						
	Target Wt (Lb) =	3.517	59.280						
	Actual Wt (Lb) =	3.515	59.280						
	Δ (Lb) =	0.002	0.00						
	Target Wt (Lb) =								
	Actual Wt (Lb) =								
	Δ (Lb) =								

* Please Note: Batches may be subdivided for convenience as long as the total weigh of all sub-batches matches the total quantity indicated.

III.B.4. Multi-Packet Materials Prep Instructions: See attached detailed instructions.

	✓?
1. Prepare individually labeled, consecutively numbered, pre-weighted (Lb/g ± Lb/g), heat-sealed packets.	
2. Verify the calibration on the weigh scale daily with *ESS'* NIST traceable standards before each work day & record the results in the attached *ESS* Scale Calibration & Calibration Verification Report (III.B.4)	
3. Place the appropriate quantity of material into the packet. Determine the net weight of each packet and record that exact weight on the label and the Packet Weight Log Sheet in the space adjacent to the packet #.	
4. Initial & date each Packet Weight Lo Sheet and Calibration Verification Log Sheet daily to certify the accuracy of the log.	
5. Keep the packets in numerical order and bundle them in convenient sized batches. Pack the batches into container(s) in reverse numerical order. Mark the containers from #1 to # . Attach one Container Shipping Label (front) and three Haz Mat Container Warning Labels per container (front, back, & top, if indicated in the instructions).	
6. Place the Site Contact Information Package (III.D.4.a) on top of the packets inside of Container #1. Securely fasten the container lid(s). Assemble the containers in a tight group just inside the shop door with Container #1 in front. Place the Transporters Information Package on top of Container #1in clear view. Coordinate shipping with the PM and SC.	

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● Houston ● Dallas/Fort Worth ● Shreveport/Longview ● Arkansas

Solvents and Chemicals Inc

4704 Shank Road, Pearland TX 77581
Phone: 281-485-5377
Toll Free: 1-800-622-3990
Fax: 281-485-6129

NACD Responsible Distribution Process

CERTIFICATE OF ANALYSIS

(1) CH_2Cl_2 CoA

Date of Shipment:	4/21/2005	Product:	Methylene Chloride
Customer:	AMPAC Chemical	Batch Number:	05-01-0117
Bill of Lading:	241579	Quantity:	1 drum

INSPECTION:	TEST METHOD:	ANALYSIS:
Weight, Lbs / Gal @ 60°F	D1250-80	11.01
Refractive Index @ 25°C		1.4215
Specific Gravity @ 60° F	D-1298-80	1.32
Purity, Wt. %		99.99 ← $[CH_2Cl_2]$
NVR, ppm		<10
Water, ppm		<22
Acidity (as HCL), ppm		None Detected
Appearance		Clear
Color, Pt-Co.		3
Distillation Range °C		
I.B.P.		
D.P.		

Approved by: CRetth Date Approved: 4-21-05

COA blank Chlorinated

● Houston ● Dallas/Fort Worth ● Shreveport/Longview ● Arkansa

Solvents and Chemicals Inc.

4704 Shank Road, Pearland TX 77581
Phone: 281-485-5377
Toll Free: 1-800-622-3990
Fax: 281-485-6129

NACD Responsible Distribution Process

(2)

CERTIFICATE OF ANALYSIS

Ethylene Glycol CoA

Date of Shipment:	4/21/2005	Product:	Ethylene Glycol
Customer:	AMPAC Chemical	Batch Number:	05-02-0280
Bill of Lading:	241579	Quantity:	1 drum

INSPECTION:	TEST METHOD:	ANALYSIS:
WT in Pounds/Gallon		9.31
Refractive Index		1.4303
Specific Gravity @ 60° F	D-1298-80	1.118
Purity, Wt. %		99.98 ← Ethyl Gly
Acidity, Wt. %		<0.002
Water, Wt. %		<0.04
Color		5
Appearance		Clear and Free
Distillation, @°C	D-86-78	
IBP		>196
DP		<199

Approved by: CRitter Date Approved: 4-21-05

COA blank glycols

IS MEMORANDUM ... copy of the BILL OF LADING covering the property named therein and is intended solely for filing of record. ... Not Negotiable ... Shipper's Imprint in Place of Bill of Lading

VED, subject to the classifications and lawfully filed tariffs in effect on the date of issue of the Bill of Lading, the property described below, in apparent good order, except as noted (contents and condition of contents of packages unknown), marked, consigned and destined as indicated below, which said carrier (the word carrier being ... throughout this contract as meaning any person or corporation in possession of the property under the contract) agrees to carry to its usual place of delivery at said destination, if on its route, otherwise to deliver to another ... on the route to said destination. It is mutually agreed, as to each carrier of all or any of said property over all or any portion of said route to destination and as to each party at any time interested in all or any of said property that ... service to be performed hereunder shall be subject to all the terms and conditions of the Uniform Domestic Straight Bill of Lading set forth (1) in Uniform Freight Classification in effect on the date hereof, if this is a rail or a rail-water ... or (2) in the applicable motor carrier classification or tariff if this is a motor carrier shipment. ... Shipper hereby certifies that he is familiar with all the terms and conditions of the said bill of lading, including those on the back hereof, set forth in the classification or tariff which govern the transportation of this shipment, and the said terms and conditions are hereby agreed to by the shipper and accepted for himself and his assigns. ...

Page 1 of 1

ERDINAL DELIVERY SERVICE/CUST REQUEST

VED, subject to the classifications and lawfully filed tariffs in effect on the date of issue of this Bill of Lading.

BILL OF LADING NO. 241579

DATE 4/21/2005

AMPAC CHEMICAL COMPANY, INC
4138 UNIVERSITY BOULEVARD
HOUSTON, TX 77005

SHIP TO: ESS
2020 REPSDORPH, UNIT 32
ATTENTION JAN / SCOTT NEAL 832-364-7048
SEABROOK, TX 77586

ST. ACCT. NO. (4-2688) | CUST. ORDER NO. 4656 | OUR SALES ORDER NO. 241579

NUMBER OF UNITS ORDERED	SHIPPED	DZ MAT		DESCRIPTION AND CLASSIFICATION	WEIGHT Net:	WEIGHT Gross
1 0	590 #	Y	590 # DRUM	DICHLOROMETHANE 4.1,UN1593,PGIII METHYLENE CHLORIDE METHYLENE CHLORIDE LOT# 05-01-0117 RSN	590 Guide Page: 160	627
1 0	510 #		510 # DRUM	NOT REGULATED ETHYLENE GLYCOL (58 GAL/DR) LOT# 05-02-0280 RSN	510 Guide Page: NONE	547
					Total Weights: Net:	1100
					Gross:	1174

Scott Neal

Driver has been given HazMat Guidebook page or appropriate MSDS. Verified and Loaded by: _____

Placards offered _____ DL _____ State: _____ Endorsements: _____

Time In: _____ Time Out: _____ Received by: _____

MSDS AND COA WITH SHIPMENT.

CH_2Cl_2 & ETHYLENE GLYCOL
CHAIN OF CUSTODY

LOT#S CROSS CHECK BETWEEN COAs (ATTACHED) & INVOICE (ATTACHED)

RT. CHARGES TO:
AMPAC CHEMICAL COMPANY, INC.
138 UNIVERSITY BOULEVARD
OUSTON, TEXAS 77005

TTENTION: SONIA

IN THE EVENT OF ANY CHEMICAL EMERGENCIES CONCERNING PRODUCTS IN THIS SHIPMENT CALL TOLL FREE CHEMTREC 1-800-424-9300

s to certify that the above-named materials are properly classified, described, ged, marked and labeled, and are in proper condition for transportation according applicable regulations of the Department of Transportation.

Number of empty drums returned to the plant

t to Section 7 of conditions of applicable bill of lading, if this shipment is to be delivered to the consignee without recourse consignor, the consignor shall sight the following statement. The carrier shall not make delivery of this shipment wit payment of freight and all other lawful charges.

Signature of consignor

CHARGES ARE TO BE: [X] PRE-PAID [ ] COLLECT

ment address of shipper

Received by: Signature: Date: 4/21/05

Per

Company Name: GAMA

UNIVAR

Univar USA
P.O. Box 4579
Houston, Texas 77210-4579
1777 Brisbane Street
Houston, Texas 77061-5044
Phone (713) 641-9454
Fax (713) 644-8369

CERTIFICATE OF ANALYSIS

PRODUCT: TOLUENE PRODUCT CODE: 368900
MANUFACTURER: TAUBER
LOT NUMBER/PACKAGE/DATE: HS026870341 55 GL DRUM 02/17/2006

TEST DESCRIPTION	ANALYSIS RESULTS
COLOR, SAYBOLT	+30
COLOR, PT CO	3
APPEARANCE@ 65-78 DEG F	CLEAR & FREE
RELATIVE DENSITY 15.56/15.56 DEG C	0.8717
API GRAVITY @ 60 DEG F	30.8
DISITILLATION RANGE	0.9
IBP	110.1
DRY POINT	111.0
COPPER CORROSION	PASS (1A)
ACID WASH COLOR	
ACID LAYER	0
OIL LAYER	NO DISCOLORATION
ACIDITY	PASS(NO FREE ACID)
TOLUENE CONTENT,WT.%	99.99
BENZENE,WT.%	0.006
ETHYLBENZENE,WT%	0.025
XYLENE,WT.%	0.017
C 8 AROMATICS,WT%	0.042
NON AROMATICS, WT%	0.042
VOL%	0.059
WATER CONTENT,PPM WT.	138
SULFUR CONTENT,WT	< 1.0
SULFUR DIOXIDE & HYDROGEN SULFIDE	FREE
1,4 DIOXANE, PPM WT.	< 5
NITRATION GRADE QUALITY	COMPLIES

Recochem Inc.
650 Montée de Liesse
Montréal, Québec H4T 1P4
Canada
Tel: (514) 341-3550
Telex: 05-824610
Cables: KUKSON
Telefax: (514) 341-6553

Attention : Michael Yared (4) NAPTHALENE C.A

Customer : Tulstar Products Inc.(Dr.T.Nature)
Product : Refined Naphthalene Crystals
Lot # : 031351, 031411

Bill of Lad. # : RD9878
Cust. P.O. # : 23-182

CERTIFICATE OF ANALYSIS

PROPERTY	TEST RESULT	SPECIFICATION	TEST METHOD
Lot 031351			
Cryst./Solid. Pt.(°C w/c) :	80.11	79.8 min.	N-QI-4.10-01-000
Color (APHA) :	8	50 max.	N-QI-4.10-03-000
GC Analysis (%w/w)			
Naphthalene	99.93	99.0 min.	N-QI-4.10-02-000
Thionaphthene	0.07	0.50 max.	N-QI-4.10-02-000
Lot 031411			
Cryst./Solid. Pt.(°C w/c) :	80.10	79.8 min.	N-QI-4.10-01-000
Color (APHA) :	16	50 max.	N-QI-4.10-03-000
GC Analysis (%w/w)			
Naphthalene	99.93	99.0 min.	N-QI-4.10-02-000
Thionaphthene	0.07	0.50 max.	N-QI-4.10-02-000

Approved : Pascal Giroux

Date : 2003/06/30

Form/Formulaire: N-QI-4.10-00-F03	Rev/Révision: 1	Issue Date d'Émission: 20/03/1997	Approved by/Approuvé par:

Attachment II Spiking Material Composition Information:

D. Metals Solution:

1. Lead Nitrate [$Pb(NO_3)_2$] &
2. Chromium Nitrate [$Cr(NO_3)_3 \cdot 9H_2O$

ESS Certification of Composition for WeStates Pb/CR^{III} Solution

Spiking Material:	Pb/CR^{III} Solution
Spiking Application:	Spiking Source of Pb/CR^{III} (SVM) for 2006 WeStates PDT
Production Date:	3/26/2006
Quantity Produced:	440.0 Lb Net Weight Solution
Composition:	[Pb] = 0.004998 [Cr^{III}] = 0.01753

Based on the information available to me concerning the manufactures' CoAs for $Pb(NO_3)_2$ & $Cr(NO_3)_3 \cdot 9H_2O$ and the procedures and equipment ***ESS*** used to establish the quantity of each ingredient used to make the final solution, I certify that the Pb concentrations provided above are true and accurate to the best of my knowledge and belief.

Signed: _____ _____

W.R. (Bill) Schofield, PhD, PE — Date

ESS Project Manager

IV.J.2 Pb Solution Preparation, Data Collection, & Composition Calculation SOP, & Worksheet

Pertinent Background Project and Technical Information:

1. **The Specified Lead (Pb):**
   A. **Spiking rate = 0.1 Lb Pb/Hr**, and
   B. **Spiking duration = 22 Hrs**.
   C. Thus, the total quantity of Pb required is **2.2 Lb Pb**.
2. *ESS* has provided the lead as Lead Nitrate, $Pb(NO_3)_2$, which will be used by *ESS'* Tech to prepare an aqueous solution on-site.
3. Technical Data for $Pb(NO_3)_2$:
   A. Pure **$Pb(NO_3)_2$ is 62.56 wt% Pb** (Merck Index, 13th Edition);
   B. Solubility: (1) One part $Pb(NO_3)_2$ is soluble in 2 parts cold H_2O, & (2) Solubility increases with increasing temperature (Merck Index); &
   C. The specific production lot[2] of $Pb(NO_3)_2$* which was used for this project (Loba Chemie Lot #V0580031) has a **100.0 wt% purity**[2] (Loba CoA, Also, see the footnote below for an explanation of the * symbol as used herein).
4. With this information, we can calculate the quantities of lead nitrate needed as follows:
   A. Spiking Rate: **0.1 Lb Pb/Hr = 0.1598 Lb $Pb(NO_3)_2$*/Hr**; and
   B. The quantity of $Pb(NO_3)_2$* required is 3.517 Lb $Pb(NO_3)_2$*/22 Hrs.

Pre-Mobilization Preparation:

5. Modify *ESS'* On-Site Pb Solution Prep SOP based on the project specific requirements.
6. Assemble, load, and transport all standard equipment/supplies required for this project plus:
   A. Prepare an appropriate shipping container w 3.517 Lb $Pb(NO_3)_2$* labeled w "$Pb(NO_3)_2$*", the actual net weight, the date weighed, and your initials, and ship the container to the test site.
   B. One large, clean, plastic laboratory funnel,
   C. A copy of this procedure, and
   D. Arrange for a suitable Pb solution prep drum (i.e., lined steel or heavy-duty plastic, closed-top) to be available on-site.

On-Site Solution Preparation:

7. Preparation:
   A. Prepare secondary containment for the field spiking equipment.
   B. Retrieve the $Pb(NO_3)_2$* Solution Drum.
   C. Retrieve PPE, i.e., latex gloves, tyvex smock, apron, or suit, and face shield.
   D. Retrieve the $Pb(NO_3)_2$* container.
   E. Record the exact net weight of $Pb(NO_3)_2$* from the container label here: 3.515 Lb $Pb(NO_3)_2$*.
   F. Verify weigh scale calibration, if this has not already been done.

Note: Solu Prep Procedure [redacted] must be completed within secondary containment.

8. Weigh the empty drum, and record the drum tare weight here: 77.2 Lb (drum tare weight).
9. Add approximately 100 Lb of water to the empty drum.
10. After donning PPE (See [redacted] above), carefully add water to the $Pb(NO_3)_2$* container until it is approximately ¾ full. Tightly secure the lid, and thoroughly mix the contents.
11. Using the funnel, carefully pour the $Pb(NO_3)_2$* solution from the container into the 100 Lb of water in the solution drum. Repeat [redacted] as necessary to ensure that all of the $Pb(NO_3)_2$* is dissolved. Rinse the container and funnel thoroughly with water, pouring all of the rinse water into the solution drum.
12. Add water to the solution drum until the gross weight of the drum plus the solution is approximately 440 Lb.
13. Agitate the $Pb(NO_3)_2$* solution in the drum with the folding prop mixer to ensure that the solution is thoroughly mixed and of uniform composition. Wipe up any water from the outside of the drum and the top & sides of the scale.

Calculation of the Solution Spiking Rate:

14. Weigh the drum and $Pb(NO_3)_2$* solution, to get: 477.2 Lb (gross weight of solution plus drum.
15. Subtract the drum tare weight (from [redacted] above.) from this (gross) weight, to get: 400.0 Lb (net weight of solution).
16. To get the weight of Pb now in the solution, multiply the exact weight of the $Pb(NO_3)_2$* (from [redacted] above) as follows: 3.515 Lb $Pb(NO_3)_2$* X (0.6256 Lb Pb/1.0 Lb $Pb(NO_3)_2$) X (1.0 Lb $Pb(NO_3)_2$/1.0 Lb $Pb(NO_3)_2$* =), to get: 2.1990 LbPb.
17. Divide the weight of Pb (g Pb from [redacted]) by the net weight of solution (from [redacted]) to get the Pb concentration in the solution: 0.004998 LbPb/Lb Solution.
18. Calculate the target solution spiking rate by dividing the target Pb spiking rate of 0.1 Lb Pb/Hr (from Step 1.A.) by the solution concentration (i.e., Lb Pb/Lb Solu, from [redacted]), to: 20.01 Lb Solu/Hr (should be very close to 20.0 Lb Solu/Hr).
19. Divide the hourly spiking rate by 60 Min/Hr to get the "target pounds of solution per minute" spiking rate: 0.3335 Lb Solu/Min (Should be very close to 0.3330 Lb Solu/Min).

QA Checks:

20. Complete a careful QA/QC check on each step above, and call the *ESS* PM (@ the *ESS* office or 713-452-5714) for a joint review of all figures, and calculations. Please signify that the QA/QC Check was satisfactorily completed with your initials:
    ESS Tech/PM: _____.
    FSH DJD

1 The $Pb(NO_3)_2$* to be used in this test has an actual purity of 1.000 %. The use of the symbol * in the chemical formula $Pb(NO_3)_2$* indicates the actual [less than 100% purity] spiking material.

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IV.J.4 Cr^{III} Solution Preparation, Data Collection, & Composition Calculation SOP, & Worksheet

Pertinent Background Technical and Project Information:

1. The Specified Chromium (Cr^{III}):
   A. Spiking rate = 0.35 Lb Cr^{III}/Hr,
   B. Spiking duration = 22 Hrs, and
   C. Thus, the total quantity of CrIII required is = 22 Hr x 0.35Lb Cr/Hr = 7.7 Lb Cr
2. *ESS* is providing the Cr^{III} as Chromium Nitrate (III), $Cr(NO_3)_3 \cdot 9H_2O$.
3. Technical Data for $Cr(NO_3)_3 \cdot 9H_2O$:
   A. Pure $Cr(NO_3)_3 \cdot 9H_2O$ is 12.99 wt% Cr (Merck Index);
   B. Solubility Data: $Cr(NO_3)_3 \cdot 9H_2O$ is: (1) "soluble" (Merck Index & Perry's), (2) "very soluble" (CRC Handbook), and (3) 208 g of $Cr(NO_3)_3 \cdot 9H_2O$ is soluble in 100ml H_2O (Lange's Handbook); and
   C. The specific production lot of $Cr(NO_3)_3 \cdot 9H_2O$* from which we were supplied for this project has a 100.08 wt% purity* (ProChem CoA, Also, see the footnote below for an explanation of the * symbol as used herein).
4. With this information, we can calculate the:
   A. $Cr(NO_3)_3 \cdot 9H_2O$* spiking rate = (0.35 Lb Cr/Hr)(1.0 Lb $Cr(NO_3)_3 \cdot 9H_2O$/0.1299 Lb Cr)(1.0 Lb $Cr(NO_3)_3 \cdot 9H_2O$*/100.08 Lb $Cr(NO_3)_3 \cdot 9H_2O$) = 2.697 Lb $Cr(NO_3)_3 \cdot 9H_2O$* /Hr; and
   B. Quantity of $Cr(NO_3)_3 \cdot 9H_2O$* required = 59.32 Lb $Cr(NO_3)_3 \cdot 9H_2O$*/22 Hrs.

Required Pre-Mobilization Preparation:

5. Modify the general *ESS* On-Site Metal Solution Prep SOP for the project specific Cr^{III} requirements.
6. Assemble, load, and transport all standard equipment/supplies required for this project plus:
   A. Prepare an appropriate shipping container w 59.32 Lb $Cr(NO_3)_3 \cdot 9H_2O$* labeled w "$Cr(NO_3)_3 \cdot 9H_2O$*", the actual net weight, the date weighed, and your initials, and ship the container to the test site.
   B. One large, clean, plastic laboratory funnel,
   C. A copy of this procedure, and
   D. Arrange a suitable Cr^{III} Solution Prep Drum (i.e., lined steel or heavy-duty plastic, closed-top) to be available on-site.

On-Site Solution Preparation:

7. Preparation:
   A. Prepare secondary containment.
   B. Retrieve the Cr Solution Prep Drum.
   C. Retrieve PPE, i.e., latex gloves, tyvex smock, apron, or suit, and face shield.
   D. Retrieve the $Cr(NO_3)_3 \cdot 9H_2O$* container.
   E. Record the exact weight of $Cr(NO_3)_3 \cdot 9H_2O$* from its container label here: 59.32 Lb.
   F. Verify weigh scale calibration, if this has not already been done.

NOTE: Sol Prep Procedure [redacted] must be completed within secondary containment.

8. Weigh the empty drum, and record that weight here: 37.2 Lb (tare weight).
9. Add approximately 100 Lb of water to the empty drum.
10. After donning PPE (See [redacted]. above), carefully add water to the $Cr(NO_3)_3 \cdot 9H_2O$* container until it is approximately ¾ full. Tightly secure the lid, and thoroughly mix the contents.
11. Using the funnel, carefully pour the $Cr(NO_3)_3 \cdot 9H_2O$* solution from the container into the 100 Lb of water in the drum. Repeat [redacted] as necessary to ensure that all of the salt is dissolved. Rinse the container and funnel thoroughly with water pouring all of the rinse water into the solution drum.
12. Add water to the solution drum until the net weight of the solution is approximately 440Lb.
13. Agitate the $Cr(NO_3)_3 \cdot 9H_2O$* solution with the folding prop mixer to ensure that the solution is thoroughly mixed and of uniform composition. Wipe up any water from the outside of the drum and the top & sides of the scale.

Calculation of the Solution Spiking Rate:

14. Weigh the drum and $Cr(NO_3)_3 \cdot 9H_2O$* solution, and record that weight here: 477.2 Lb (gross weight).
15. Subtract the empty drum weight (from [redacted] above.) from this (gross) weight to get: 440.0 Lb (net weight of solution).
16. Multiply the exact weight of the $Cr(NO_3)_3 \cdot 9H_2O$* (from [redacted] above) as follows: 59.32 Lb $Cr(NO_3)_3 \cdot 9H_2O$* x (0.1299 Lb Cr/1.0 lb $Cr(NO_3)_3 \cdot 9H_2O$)(1.0008 Lb $Cr(NO_3)_3 \cdot 9H_2O$/1.0 lb $Cr(NO_3)_3 \cdot 9H_2O$*=) to get: 7.712 Lb Cr.
17. Divide the weight of Cr^{III} (from [redacted]) by the net weight of solution (from [redacted]) to get 0.01753 Lb Cr/Lb solution.
18. Calculate the hourly spiking rate by dividing 0.35lb Cr^{III}/Hr by the solution concentration (from [redacted]) to get: 19.97 Lb solution/Hr (This number should be very close to 20 Lb sol/Hr).
19. Divide the hourly spiking rate by 60 Min/Hr to get the target spiking rate, and record the result here: 0.3328 Lb solution/Min.

QA Checks:

21. Complete a careful QA/QC check on each step above, and call the *ESS* PM (@ the *ESS* office or 713-542-5714) for a joint review of all figures, and calculations. Please signify that the QA/QC Check was satisfactorily completed with your initials:
    ESS Tech/PM: _____
    ESS PM RDW

* The symbol * in the chemical formula $Cr(NO_3)_3 \cdot 9H2O$* indicates the actual 100.08 % purity Chromium Nitrate which will be used on this test.

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III.B.2. Materials Preparation Instructions & Checklist:

Material Description:

General Prep Instructions:

ID SDPs/Instructions/Worksheets: Applicable ? =	✓	Att?	Get	Done?	Notes, Comments, &/or Instructions:
1. Review all Mat'ls Prep Information for this project.	✓		✓	✓	
2. MSDS(s)	✓		✓	✓	Get MSDSs from Mat'l Tech Files & Read.
3. Materials Prep Instructions, Raw Mat'l Weights (III.B.3)	✓	✓	✓	✓	
4. Multi-Packet Mat'ls Prep Instructions (III.B.4)	NA	NA		N/A	See attached detailed instructions.
5. Scale Calibration & Calib. Verification Report (III.B.5)	✓	✓	✓		
6. DM & Dispersion Prep SDP (III.B.6)	NA	NA		N/A	
7. Shipping Instructions (III.D)	✓		✓	✓	
8. Bill of Lading (w go by)	✓		✓	✓	
9. Container Shipping Label &/or go by	✓		✓	✓	
10. Container Warning Label	✓		✓	✓	
11. Other (ID)	✓		✓	✓	Ground Drums During Mixing Process

Approved by: LJPS Date: 3/8/2006 Accepted by: Date: Completed per SOP: RBD Date: 3/16/2006

III.B.3. Materials Prep Instructions, Raw Material Weights, & Prep Worksheet:

Batch Or Drum #	Raw Mat'l =	Toluene	CH_2Cl_2	Naphthalene	ET Glycol				
	Lot # =	HS026870341	05-01-0117	031411	05-02-0280				
	Purity =	0.9999	0.9999	0.9993	0.9998				
	Use Scale # =	S-1 (1000 Lb)	S-1 (1000 Lb)	S-1 (1000 Lb)	S-1 (1000 Lb)	L- (Lb)	L- (Lb)		
	Calibrated on =	/ /	/ /	/ /	/ /	/ /	/ /		
	Sub-batch =						A*	B*	C*
1	Target Wt (Lb) =	187.00	88.00	88.00	88.00	*	*	*	*
	Actual Wt (Lb) =	187	88.00	88.00	88.00				
	Δ (Lb) =	0.00	0.00	0.00	0.00				
2	Target Wt (Lb) =	187.00	88.00	88.00	88.00				
	Actual Wt (Lb) =	187.00	88.00	88.00	88.00				
	Δ (Lb) =	0.00	0.00	0.00	0.00				
3	Target Wt (Lb) =	170.00	80.00	80.00	80.00				
	Actual Wt (Lb) =	170.0	80.0	80.00	80.0				
	Δ (Lb) =	0.00	0.00	0.00	0.00				
		Pb	$Cl(NaO)_2 9H_2O$						
	Target Wt (Lb) =	3.517	59.280						
	Actual Wt (Lb) =	3.515	59.280						
	Δ (Lb) =	0.002	0.00						
	Target Wt (Lb) =								
	Actual Wt (Lb) =								
	Δ (Lb) =								

* Please Note: Batches may be subdivided for convenience as long as the total weigh of all sub-batches matches the total quantity indicated.

III.B.4. Multi-Packet Materials Prep Instructions: See attached detailed instructions.

	✓?
1. Prepare individually labeled, consecutively numbered, pre-weighted (Lb/g ± Lb/g), heat-sealed packets.	
2. Verify the calibration on the weigh scale daily with *ESS'* NIST traceable standards before each work day & record the results in the attached *ESS* Scale Calibration & Calibration Verification Report (III.B.4)	
3. Place the appropriate quantity of material into the packet. Determine the net weight of each packet and record that exact weight on the label and the Packet Weight Log Sheet in the space adjacent to the packet #.	
4. Initial & date each Packet Weight Lo Sheet and Calibration Verification Log Sheet daily to certify the accuracy of the log.	
5. Keep the packets in numerical order and bundle them in convenient sized batches. Pack the batches into container(s) in reverse numerical order. Mark the containers from #1 to # . Attach one Container Shipping Label (front) and three Haz Mat Container Warning Labels per container (front, back, & top, if indicated in the instructions).	
6. Place the Site Contact Information Package (III.D.4.a) on top of the packets inside of Container #1. Securely fasten the container lid(s). Assemble the containers in a tight group just inside the shop door with Container #1 in front. Place the Transporters Information Package on top of Container #1in clear view. Coordinate shipping with the PM and SC.	

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LOBA CoA FOR $Pb(NO_3)_2$
100.1% OR 1.001

Ref: LOBA/COA/V

August 19, 2004

CERTIFICATE OF ANALYSIS

Product Name	:	LEAD NITRATE PURE
Code no	:	4377
Batch no	:	V0580
Mol. Formula	:	$Pb(NO_3)_2$
Mol. Weight	:	331.21

Sr. No	Tests	Specification	Results
1	Description	White crystalline powder	White crystalline powder
2	Assay (min)	99%	100.1%
3	Chloride (Cl)	< 0.005%	< 0.005%
4	Copper (Cu)	< 0.001%	< 0.001%
5	Iron (Fe)	< 0.001%	< 0.001%

This above product complies as per the specification of LOBA CHEMIE.

For LOBA CHEMIE PVT. LTD.

Note: This is document has been produced electronically and it is valid without signature.

PACKING LIST

Exporter LOBA CHEMIE PVT. LTD 80 BABU GENU ROAD MBAI - 400 002 INDIA CHAIN OF CUSTODY DOCUMENTATION SUPPLIER TO ESS	Invoice No. & Date EXP/056 Dt. 09/09/2004
	Exporter's Ref.
	Buyer's Order No. & Date PRO/178/04 Dt. 01/09/2004
	Other Reference(s)
Consignee ENGINEERED SPIKING SOLUTIONS, INC(ESS) 1200 HWY 146 SOUTH SUITE 170 LAPORTE, TX 77571 USA	Buyer (If other than consignee)
	Country of Origin of Goods INDIA
	Country of Final Distination U.S.A.
Pre-Carriage by	Place of receipt by Pre-Carrier J.N.P.T.(INDIA)
Vessel/Flight No.	Port of Loading J.N.P.T.(INDIA)
Port of Discharge HOUSTON	Final Destination HOUSTON
	Terms of Delivery and Payment

Mark & Nos./ Container No.	No. & Kind of Packages	Despcription of Goods	Quantity	Nett Wt. (Kgs.)	Gross Wt. (Kgs.)
ENGINEERED SPIKING SOLUTIONS, INC,(ESS) LAPORTE, HOUSTON USA	48 PKGS	OF LABORATORY CHEMICALS			
DRUM NO.1TO20		BATCH NO			
SODIUM DICHROMATE (DIHYDRATE)		V1695041	20x25KG	500.000	540.000
DRUM NO.21TO28					
~~CHROMIUM (III) OXIDE GREEN~~		~~V1697041~~	~~8x25KG~~	~~200.000~~	~~216.000~~
DRUM NO.29TO40					
LEAD NITRATE PURE		V0580031	12x25KG	300.000	312.000
DRUM NO.41TO48					
LEAD MONOXIDE (LITHARGE)		V1026041	8x25KG	200.000	208.000
TOTAL NET WEIGHT : 1,200.000 KGS. TOTAL GROSS WEIGHT : 1,276.000 KGS.					
		Total Weight		1200.000	1276.000

Declaration :
We declare that this Invoice shows the actual price of the goods described and that particulars are true and correct

FOR LOBA CHEMIE PRIVATE LTD.
Signature & Date
09/09/2004
DIRECTOR

Att II A(2), 3/13

ProChem inc.
"We react"

CoA

Cerificate of Analysis

Product name: Chromium (III) Nitrate Hydrate, 99+%

Chemical Formula: Cr(NO3)3.XH2O ⇒ EXPRESSED BY ESS AS $Cr(NO_3)_3 \cdot 9H_2O$

LOT#1031390

Quantity: 100 lbs.

CAS#: 13548-38-4

Specifications		Results
Chromium	12.7% min.	13.0%
H2O Insolubles	0.010% max	0.001%
Chloride	0.05% max	0.001%
Sulfate(SO4)	0.01% max	0.001%
Iron(Fe)	0.020%max	0.004%

$$\text{PURITY} = \frac{0.1300}{0.1299} \times 100\% = 100.08\% = 1.0008$$

0.1299 IS MASS FRACTION Cr IN $Cr(NO_3)_3 \cdot 9H_2O$.

826 Roosevelt Rd. • Rockford, IL 61109 • (815) 398-1788 • Fax (815) 398-1810

High Purity Inorganics • Research Biochemicals • Custom Synthesis

PROCHEM, INC.
826 ROOSEVELT RD.
ROCKFORD, IL 61109-2025
(815) 398-1788

INVOICE Att II A(2), 5/13

Invoice # : 00013999

Bill To:

Engineered Spiking Solutions
1200 Hwy. 146 S.
Street 170
LaPorte, TX 77571

Ship To:

Eli Lily & Company
1650 Lilly Road
Lafayette, IN 47909
Attn: Miguel Gonzales

SALESPERSON	YOUR ORDER NO.	SHIP VIA	COL	PPD	SHIP DATE	TERMS	DATE	PG.
	7665	OVERNITE	X		9/14/05	2% 10 Net 30	9/14/05	1

QTY.	ITEM NO.	DESCRIPTION	PRICE	UNIT	DISC %	EXTENDED PRICE	TX
100	1476	Chromium Nitrate, 99+%, Hydrate, Lot #1031390		LB			

SALE AMOUNT	0.00
FREIGHT	0.00
SALES TAX	0.00
TOTAL AMOUNT	0.00
PAID TODAY	0.00
BALANCE DUE	0.00

08082125-42

Attachment III Documentation of Accuracy of the Field Spiking Rate Measuring Equipment Used during this Project:

A. Pre-Mob, Pre-Test, and Post-Test Calibration Verification Reports, and
B. Current Certification of Weight Standards with NIST Traceability.

Attachment III Documentation of Accuracy of the Field Spiking Rate Measuring Equipment Used during this Project:

A. Pre-Mob, Pre-Test, and Post-Test Calibration Verification Reports,

III.B.5 (a) *ESS* Scale Calibration & Calibration Verification Report

Shop Weigh Scale #: P-1 | Calibrate & Verify Calibration to: 500 Lbs

Application: D Kwdo

Pre-Test Calibration Verification: Date 3/17/2006			**Post-Test** Calibration Verification: Date 3/17/2006		
Test Load, Lb	- Indicated Wt, Lb	= Difference, ± 0.? Lb	Test Load, Lb	- Indicated Wt, Lb	= Difference, ± 0.? Lb
Build Up in 50 Lb Increments:			Build Up in 50 Lb Increments:		
0.0	0.0	0.0	0.0		
50.0	50.0	0.0	50.0		
100.0	100.0	0.0	100.0		
150.0	149.9	-0.1	150.0		
200.0	199.9	-0.1	200.0		
250.0	249.9	-0.1	250.0		
300.0	299.9	-0.1	300.0		
350.0	350.0	0.0	350.0		
400.0	400.0	-0.1	400.0		
450.0	449.9	-0.1	450.0		
500.0	499.9	-0.1	500.0		
Break Down in 50 Lb Increments:			Break Down in 50 Lb Increments:		
500.0	499.9	-0.1	500.0		
450.0	449.9	-0.1	450.0		
400.0	399.9	-0.1	400.0		
350.0	349.9	-0.1	350.0		
300.0	299.9	-0.1	300.0		
250.0	249.9	-0.1	250.0		
200.0	199.9	-0.1	200.0		
150.0	149.9	-0.1	150.0		
100.0	99.9	-0.1	100.0		
50.0	49.9	-0.1	50.0		
0.0	-0.1	-0.1	0.0		

Accuracy Assessment & Comments:

ESS Technician: [signature] | Date: 3/17/2006

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III.B.5 (a) *ESS* Scale Calibration & Calibration Verification Report

Shop Weigh Scale #: F-2 | Calibrate & Verify Calibration to: 500 **Lbs**

Application: Pickup

Pre-Test Calibration Verification: Date: 3/17/2006			**Post-Test** Calibration Verification: Date: / /200		
Test Load, Lb	- Indicated Wt, Lb	= Difference, ± 0.? Lb	Test Load, Lb	- Indicated Wt, Lb	= Difference, ± 0.? Lb
Build Up in 50 Lb Increments:			Build Up in 50 Lb Increments:		
0.0	0.0		0.0		
50.0	50.0		50.0		
100.0	100.0		100.0		
150.0	150.1		150.0		
200.0	200.0		200.0		
250.0	250.1		250.0		
300.0	300.0		300.0		
350.0	350.1		350.0		
400.0	400.1		400.0		
450.0	450.1		450.0		
500.0	500.1		500.0		
Break Down in 50 Lb Increments:			Break Down in 50 Lb Increments:		
500.0	500.1		500.0		
450.0	450.1		450.0		
400.0	400.0		400.0		
350.0	350.0		350.0		
300.0	300.1		300.0		
250.0	250.0		250.0		
200.0	200.1		200.0		
150.0	150.1		150.0		
100.0	100.0		100.0		
50.0	50.0		50.0		
0.0	0.0		0.0		

Accuracy Assessment & Comments:

ESS Technician: [signature] | Date: 3/17/2006

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III.B.5 (a) *ESS* Scale Calibration & Calibration Verification Report

Shop Weigh Scale #: F-3 | Calibrate & Verify Calibration to: 200 Lbs

Application: Pecuda

Pre-Test Calibration Verification: Date: 3/17/2006			**Post-Test** Calibration Verification: Date: / /200		
Test Load, Lb	- Indicated Wt, Lb	= Difference, ± 0.? Lb	Test Load, Lb	- Indicated Wt, Lb	= Difference, ± 0.? Lb
Build Up in 50 Lb Increments:			Build Up in 50 Lb Increments:		
0.0	0.0		0.0		
50.0	50.0		50.0		
100.0	100.0		100.0		
150.0	150.0		150.0		
200.0	200.0		200.0		
250.0	250.0		250.0		
300.0	300.0		300.0		
350.0	350.0		350.0		
400.0	400.0		400.0		
450.0	450.0		450.0		
500.0	500.1		500.0		
550.0	550.0				
600.0	600.1				
650.0	650.1				
700.0	700.0				
750.0	750.1				
800.0	800.0				
Break Down in 50 Lb Increments:			Break Down in 50 Lb Increments:		
800.0	800.0				
750.0	750.1				
700.0	700.1				
650.0	650.1				
600.0	600.0				
550.0	550.1				
500.0	500.1		500.0		
450.0	450.0		450.0		
400.0	400.0		400.0		
350.0	350.0		350.0		
300.0	300.0		300.0		
250.0	250.0		250.0		
200.0	200.0		200.0		
150.0	150.0		150.0		
100.0	100.0		100.0		
50.0	50.0		50.0		
0.0	0.0		0.0		

Accuracy Assessment & Comments:

ESS Technician: [signature] | Date: 3/17/2006

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III.B.5 (a) *ESS* Scale Calibration & Calibration Verification Report

Shop Weigh Scale #: F-5 | Calibrate & Verify Calibration to: 500 Lbs

Application: Jacuzzi

Pre-Test Calibration Verification: Date: 3/7/2006			**Post-Test** Calibration Verification: Date: / /200		
Test Load, Lb	- Indicated Wt, Lb	= Difference, ± 0.? Lb	Test Load, Lb	- Indicated Wt, Lb	= Difference, ± 0.? Lb
Build Up in 50 Lb Increments:			Build Up in 50 Lb Increments:		
0.0	0.0		0.0		
50.0	49.9		50.0		
100.0	99.9		100.0		
150.0	150.0		150.0		
200.0	199.9		200.0		
250.0	250.0		250.0		
300.0	300.0		300.0		
350.0	350.0		350.0		
400.0	399.9		400.0		
450.0	450.0		450.0		
500.0	500.0		500.0		
Break Down in 50 Lb Increments:			Break Down in 50 Lb Increments:		
500.0	500.0		500.0		
450.0	449.9		450.0		
400.0	399.9		400.0		
350.0	350.0		350.0		
300.0	300.0		300.0		
250.0	249.9		250.0		
200.0	199.9		200.0		
150.0	150.0		150.0		
100.0	100.0		100.0		
50.0	50.0		50.0		
0.0	0.0		0.0		

Accuracy Assessment & Comments:

ESS Technician: [signature] | Date: 3/7/2006

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III.B.5 (a) *ESS* Scale Calibration & Calibration Verification Report

Shop Weigh Scale #: F-1 | Calibrate & Verify Calibration to: 500 Lbs

Application: NCB

Pre-Test Calibration Verification: Date: 3/27/2006			**Post-Test** Calibration Verification: Date: 3/31/2006		
Test Load, Lb	- Indicated Wt, Lb	= Difference, ± 0.? Lb	Test Load, Lb	- Indicated Wt, Lb	= Difference, ± 0.? Lb
Build Up in 50 Lb Increments:			Build Up in 50 Lb Increments:		
0.0	0.0	0.0	0.0	0.0	0.0
50.0	50.0	0.0	50.0	50.0	0.0
100.0	100.0	0.0	100.0	100.0	0.0
150.0	150.0	0.0	150.0	150.0	0.0
200.0	200.0	0.0	200.0	200.0	0.0
250.0	249.9	-0.1	250.0	250.0	0.0
300.0	299.9	-0.1	300.0	300.0	0.0
350.0	349.9	-0.1	350.0	350.0	0.0
400.0	399.9	-0.1	400.0	400.0	0.0
450.0	450.0	0.0	450.0	450.1	0.1
500.0	500.0	0.0	500.0	500.1	0.1
Break Down in 50 Lb Increments:			Break Down in 50 Lb Increments:		
500.0	500.0	0.0	500.0	500.1	0.1
450.0	449.9	-0.1	450.0	450.1	0.1
400.0	399.9	-0.1	400.0	400.0	0.0
350.0	349.9	-0.1	350.0	350.0	0.0
300.0	299.9	-0.1	300.0	300.0	0.0
250.0	249.9	-0.1	250.0	250.0	0.0
200.0	199.9	-0.1	200.0	200.0	0.0
150.0	149.9	-0.1	150.0	150.0	0.0
100.0	99.9	-0.1	100.0	100.0	0.0
50.0	49.9	-0.1	50.0	50.0	0.0
0.0	-0.1	-0.1	0.0	0.0	0.0

Accuracy Assessment & Comments:

ESS Technician: Scott West | Date: 3/31/2006

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III.B.5 (a) *ESS* Scale Calibration & Calibration Verification Report

Shop Weigh Scale #: F-2 | Calibrate & Verify Calibration to: 500 Lbs

Application: Olamic Solution

Pre-Test Calibration Verification: Date: 3/27/2006			**Post-Test** Calibration Verification: Date: 3/31/2006		
Test Load, Lb	- Indicated Wt, Lb	= Difference, ± 0.? Lb	Test Load, Lb	- Indicated Wt, Lb	= Difference, ± 0.? Lb
Build Up in 50 Lb Increments:			Build Up in 50 Lb Increments:		
0.0	0.0	0.0	0.0	0.0	0.0
50.0	50.0	0.0	50.0	50.0	0.0
100.0	100.0	0.0	100.0	100.0	0.0
150.0	150.0	0.0	150.0	150.0	0.0
200.0	200.1	0.1	200.0	200.1	0.1
250.0	250.0	0.0	250.0	250.0	0.0
300.0	300.0	0.0	300.0	300.0	0.0
350.0	350.0	0.0	350.0	350.0	0.0
400.0	400.1	0.1	400.0	400.0	0.0
450.0	450.0	0.0	450.0	450.0	0.0
500.0	500.0	0.0	500.0	500.0	0.0
Break Down in 50 Lb Increments:			Break Down in 50 Lb Increments:		
500.0	500.0	0.0	500.0	500.0	0.0
450.0	450.0	0.0	450.0	450.0	0.0
400.0	400.1	0.1	400.0	400.1	0.1
350.0	350.0	0.0	350.0	350.0	0.0
300.0	300.0	0.0	300.0	300.0	0.0
250.0	250.0	0.0	250.0	250.0	0.0
200.0	200.1	0.1	200.0	200.1	0.1
150.0	150.0	0.0	150.0	150.0	0.0
100.0	100.0	0.0	100.0	100.0	0.0
50.0	50.0	0.0	50.0	50.0	0.0
0.0	0.0	0.0	0.0	0.0	0.0

Accuracy Assessment & Comments:

ESS Technician: [illegible] | Date: 3/31/2006

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III.B.5 (a) *ESS* Scale Calibration & Calibration Verification Report

Shop Weigh Scale #: P-3 | Calibrate & Verify Calibration to: 800 Lbs

Application: PCAC

Pre-Test Calibration Verification: Date: 3/27/2006			Post-Test Calibration Verification: Date: 3/27/2006		
Test Load, Lb	- Indicated Wt, Lb	= Difference, ± 0.? Lb	Test Load, Lb	- Indicated Wt, Lb	= Difference, ± 0.? Lb
Build Up in 50 Lb Increments:			Build Up in 50 Lb Increments:		
0.0	0.0		0.0	0.0	
50.0	50.0		50.0	50.0	
100.0	100.0		100.0	100.1	
150.0	150.0		150.0	150.0	
200.0	200.0		200.0	200.0	
250.0	250.0		250.0	250.0	
300.0	300.0		300.0	300.1	
350.0	350.1		350.0	350.1	
400.0	400.1		400.0	400.1	
450.0	450.0		450.0	450.1	
500.0	500.0		500.0	500.1	
550.0	550.1		550.0	550.1	
600.0	600.1		600.0	600.2	
650.0	650.1		650.0	650.1	
700.0	700.1		700.0	700.1	
750.0	750.1		750.0	750.1	
800.0	800.1		800.0	800.1	
Break Down in 50 Lb Increments:			Break Down in 50 Lb Increments:		
800.0	800.1		800.0	800.1	
750.0	750.1		750.0	750.1	
700.0	700.1		700.0	700.1	
650.0	650.1		650.0	650.1	
600.0	600.1		600.0	600.1	
550.0	550.1		550.0	550.1	
500.0	500.1		500.0	500.1	
450.0	450.1		450.0	450.1	
400.0	400.1		400.0	400.1	
350.0	350.0		350.0	350.0	
300.0	300.0		300.0	300.1	
250.0	250.0		250.0	250.0	
200.0	200.0		200.0	200.0	
150.0	150.0		150.0	150.1	
100.0	100.0		100.0	100.1	
50.0	49.9		50.0	50.1	
0.0	0.0		0.0	0.1	

Accuracy Assessment & Comments:

ESS Technician: [signature] | Date: 3/27/2006

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III.B.5 (a) *ESS* Scale Calibration & Calibration Verification Report

Shop Weigh Scale #: E-5 | Calibrate & Verify Calibration to: 500 Lbs

Application: Pb/Cu" Solution

Pre-Test Calibration Verification: Date: 3/7/2006			**Post-Test** Calibration Verification: Date: 3/7/2006		
Test Load, Lb	- Indicated Wt, Lb	= Difference, ± 0.? Lb	Test Load, Lb	- Indicated Wt, Lb	= Difference, ± 0.? Lb
Build Up in 50 Lb Increments:			Build Up in 50 Lb Increments:		
0.0	0.0		0.0	0.0	
50.0	50.0		50.0	50.0	
100.0	100.0		100.0	100.0	
150.0	150.1		150.0	150.0	
200.0	200.0		200.0	199.9	
250.0	250.0		250.0	250.0	
300.0	300.0		300.0	300.0	
350.0	350.0		350.0	349.9	
400.0	400.0		400.0	399.9	
450.0	449.9		450.0	450.0	
500.0	499.9		500.0	500.0	
Break Down in 50 Lb Increments:			Break Down in 50 Lb Increments:		
500.0	500.0		500.0	500.0	
450.0	450.0		450.0	449.9	
400.0	399.9		400.0	399.9	
350.0	349.9		350.0	350.0	
300.0	300.0		300.0	300.0	
250.0	250.0		250.0	250.0	
200.0	200.1		200.0	200.0	
150.0	150.0		150.0	150.0	
100.0	100.0		100.0	100.0	
50.0	50.0		50.0	50.0	
0.0	0.0		0.0	0.0	

Accuracy Assessment & Comments:

ESS Technician: [signature] | Date: 3/3/2006

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Attachment III Documentation of Accuracy of the Field Spiking Rate Measuring Equipment Used during this Project:
B. Current Certification of Weight Standards with NIST Traceability.

Test No: G000001191

TEXAS DEPARTMENT OF AGRICULTURE
SUSAN COMBS, COMMISSIONER
REGULATORY DIVISION
CONSUMER PROGRAM
METROLOGY LABORATORY

REPORT OF TEST

ENGINEERING SPIKING SOLUTIONS
1200 Hwy. 146 South, Ste. 170
La Porte, Texas 77571

Test Date: 08/11/2005
Phone Number: 281-471-2071
County: Harris
Region: 3

Total Pounds Sealed:	Weights Sealed	Weights Rejected	Measures Sealed:	Measures Rejected:
1250.00000	25	0	0	0

This is to certify that the physical standards described below were on this day compared to the standards of the State of Texas which are directly traceable to standards of the National Institute of Standards and Technology.

test_description	*# sealed*	*# rejected*	*test_description*	*# sealed*	*# rejected*
50 lbs.	25	0			

*See attachment

Metrologist

5/05
TDA C279E

TEXAS DEPARTMENT OF AGRICULTURE
SUSAN COMBS, COMMISSIONER
TEXAS METROLOGY LABORATORY
CERTIFICATE OF CALIBRATION

For

Cast Iron Test Weights

Test Completed
08/11/2005

Test Number
G-000001191

Submitted by
Engineered Spiking Solutions
1200 Highway 146 South, Suite 170
La Porte, Texas 77571

The standards described below have been compared to the standards of the State of Texas (N.I.S.T. Test # 40093, 251996) and were found to have the following mass corrections:

Temperature Range: 15°C - 30°C
Humidity Range: 30% - 60%
SOP Used: Mod. Sub., SOP-8

State Standards Cal Date: 12/2004
State Standards Cal Due Date: 12/2005

Nominal Value	Serial / ID #	As Found Mass Correction (Milligram)	As Left Mass Correction (Milligram)	Expanded Uncertainty (Milligram)	Tolerance Class	Tolerance (Milligram)
50 LB	ESS7	1527.000000	457.000000	157	F	2300
50 LB	ESS16	4227.000000	327.000000	157	F	2300
50 LB	ESS24	4387.000000	567.000000	157	F	2300
50 LB	ESS19	6417.000000	557.000000	157	F	2300
50 LB	ESS25	3697.000000	487.000000	157	F	2300
50 LB	ESS22	3757.000000	347.000000	157	F	2300
50 LB	ESS20	1297.000000	537.000000	157	F	2300
50 LB	ESS18	2767.000000	597.000000	157	F	2300
50 LB	ESS13	6497.000000	537.000000	157	F	2300
50 LB	ESS23	3767.000000	517.000000	157	F	2300
50 LB	ESS9	1497.000000	507.000000	157	F	2300
50 LB	ESS5	2787.000000	357.000000	157	F	2300
50 LB	ESS10	2097.000000	357.000000	157	F	2300
50 LB	ESS1	1857.000000	347.000000	157	F	2300
50 LB	ESS11	3337.000000	387.000000	157	F	2300
50 LB	ESS17	2587.000000	467.000000	157	F	2300
50 LB	ESS15	2217.000000	577.000000	157	F	2300
50 LB	ESS3	3117.000000	547.000000	157	F	2300
50 LB	ESS12	5330.000000	497.000000	157	F	2300
50 LB	ESS2	1767.000000	557.000000	157	F	2300
50 LB	ESS14	1757.000000	577.000000	157	F	2300
50 LB	ESS8	3337.000000	607.000000	157	F	2300
50 LB	ESS21	5067.000000	637.000000	157	F	2300
50 LB	ESS6	1727.000000	297.000000	157	F	2300
50 LB	ESS4	6737.000000	427.000000	157	F	2300

The effect of air bouyancy has been considered negligible.
The expanded uncertainty given here is in compliance with NIST Technical Note 1297 ("Guidelines for Evaluating and Expressing the Uncertaity of NIST Measurement Results") with a coverage factor of two, representing a 95% confidence level. This report is not to be used to claim product endorsement by the Texas Department of Agriculture or any agency of the U.S. Government. This document shall not be reproduced, except in full, without the written approval of the Texas Department of Agriculture Metrology Laboratory.

S:\Giddings Metrology Lab\Spreadsheet Programs\SOP-8.doc

Laboratory Metrologist

Attachment IV Field Spiking Data

A. Executed Test Manager Spiking Orders to ***ESS*** and other operations logs,
B. Stack Sampling Start/Stop Times, and
C. Field Spiking Log Sheets (Field Data) and Spiking Rate Calculations for:
   1. Test Condition #1, Run #1:
      (a) Mono-Chlorobenzene,
      (b) Perchloroethylene,
      (c) Organics Solution, &
      (d) Metals Solution,
   2. Test Condition #1, Run #2:
      (a) Mono-Chlorobenzene,
      (b) Perchloroethylene,
      (c) Organics Solution, &
      (d) Metals Solution, and
   3. Test Condition #1, Run #3:
      (a) Mono-Chlorobenzene,
      (b) Perchloroethylene,
      (c) Organics Solution, &
      (d) Metals Solution.

Attachment IV Field Spiking Data

A. Executed Test Manager Spiking Orders to ***ESS*** and other operations logs

IV.E. Client Test Manager's Spiking Orders[1] to *ESS*:

Section I Initial Spiking Orders[1]:

Spiking:		Spiking Rate, Lb/Hr		Pump Type/Size	Spiking Duration, Hrs	Specie/Mat'l Reqr'ed/ Mat'l Provided, Lb/Lb/# Drums
Specie	Material	As Specie	As Mat'l			
POHCs:						
MCB	MCB	35	35	Neptune #3	32	1,120/1500/3-500 [Net] Lb Drums
C_2Cl_4	C_2Cl_4	35	35	LMI #10	32	1120/1400/2-700 [Net] Lb Drums
Metals:						
Pb	Pb/Cr^{III} Solution	.1	20	LMI #7	32	3.2/640/1-640 [Net] Lb Drum
Cr^{III}	Pb/Cr^{III} Solution	.35	20	LMI #7	32	11.2/640/1-640 [Net] Lb Drum
Organic Mixture:						
	Organic Mixture		41	Neptune #4	32	1312Lb-2 @ 451[Net] Lb Drum1@ 410 [Net] Lb Drum
Toluene		17			32	
CH_2Cl_2		8			32	
Naphthalene		8			32	
Et Glycol		8			32	

Approved by Client/Test Manager: [signature] **Date:** 3 /27/200[illegible]

Section II Revised Spiking Orders[2]:

Revision 1:

Approved by Client/Test Manager: **Date:** / /200

Revision 2:

Approved by Client/Test Manager: **Date:** / /200

Revision 3:

Approved by Client/Test Manager: **Date:** / /200

Section III Critique, Suggestions, and Comments[3]:

by Client/Test Manager: **Date:** / /200

Footnotes:
1. Section I contains *ESS'* understanding of the spiking requirements (Spiking Orders) for this test. Please review, revise (as necessary), and initial/date to indicate that the Spiking Orders (as revised) are correct.
2. Section II is provided for field revisions to the Spiking Orders by the Client/Test Manager, as needed. Please document the required changes, and initial/date the new orders.
3. Please provide a critique of *ESS'* performance on this test, offer suggestions for improving the value of our products and services to you, and/or (if warranted) identify aspect(s) of our products and services with which you are pleased.

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ESS 1200 Hwy 146 South, Suite 170, LaPorte, Texas 77571 (281) 471-2071 Fax (281) 471-2180 *BSPE@ESSpiking.com*

IV. Spiking Plan Transmittal Checklist:			
Spiking Plan Component:		**Applicable & Att'ed?**	**Received & Accepted?**
Gen Info Re:	A. Test ID & Site Contact Information.	✓	✓
Phase IV Proj Exec.	B. Overall Test Execution SOP & Checklist.	✓	✓
Spiking Plan:	C. Spiking Mat'ls, Species, Rates, & Durations, plus Quantities of Mat'ls & Equip Required.	✓	✓
	D. Test Schedule.	✓	✓
	E. Spiking Orders.	✓	✓
	F. Pump Assignments.	✓	✓
	G. (1) Terms & Conditions, and (2) Support Requirements.	✓	✓
	H. Project Rate Schedule	✓	✓
	I. Field Scale Set-Up, Adjustment, Calibration, and Calibration Verification SOP & Checklist.	✓	✓
	J. On-Site Solution Preparation SOPs, and & Documentation Worksheets:	✓	✓
	(1) Pb, Cr^{III} Solution	✓	✓
	K. Other Project Execution Related Log Sheets, Checklists, & Worksheets:	✓	✓
	(1) Daily Operation	✓	✓
	(2) ***ESS*** Field Scale Corner Test Report	✓	✓
	(3) Pre- & Post-Test Calib. Verification Report	✓	✓
	(4) Eq Operation & Maintenance	✓	✓
	(5) Spiking Log Sheets:	✓	✓
	(a) 1st Sheet per run	✓	✓
	(b) 2nd Sheets per run	✓	✓
Prepared & Approved by:	Date:	Received & Accepted by: RJD	Date: 3/16/2006
Footnotes:			

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IV.A. General Project ID & Site Contact Information:	
1. Test Type:	CPT
2. Test Dates	Week of 3/27/2006 (Mob on 24th & Spike on 28th through 31st)
3. Test Location	Parker, Az @ US Filter (See maps, etc.)
4. Contact Name & #	Drew Boyard (928) 669-5758
5. Other Information	

IV.B. Project Phase IV: Test Execution SOP & Checklist	
Test Day = -1: Travel to the Test Site:	✓?
Safely drive to the test site obeying all traffic laws and applicable DOT requirements including the DOT Time Log/limits. Stop for coffee/coke &/or rest, as needed. Plan to arrive in the vicinity of the test facility on the day prior to the equipment set-up day to get a good nights rest.	✓
Test Day = 0 (Mob or Equipment Set-Up Day):	
Arrive at the gate early wearing PPE and ***ESS*** logo apparel, as appropriate. Place magnetic ***ESS*** signs on truck doors. Check in at the gate. Receive any site provided safety or other training.	✓
Make contact with the client representative & clarify any uncerlainties about the test schedule, spiking rates, management of contaminated materials, & establish the method of communications.	✓
Check into the unit control room, obtain required permits, and synchronize ***Spike Manager*** © clock.	✓
Locate all spiking materials; confirm lot numbers, drum counts, condition of containers, etc. When there are multiple drums of a given material (say N drums), mark each drum numerically from 1 to N and then use the drums in numerical order.	✓
Confirm availability of: (a) required utilities, (b) a flat, level, hard surfaced work area, and (c) reasonable access to the spiking injection point.	✓
Ask for fork lift or other assistance, as needed, to off load equipment and relocate to the spiking area. *Use **ESS'** dolly &/or hand truck, &/or request assistance from operator/test manager, as needed, to protect your back from stains.*	✓
Set-up secondary containment (if not already available). Lay down impermeable barrier to protect the work surface from possible contamination, even if secondary containment is available. *Only open spiking material containers when the containers are inside the secondary containment area.*	✓
Set-up and verify calibration of weigh scales. Set-up the spiking pumps, MFMs, drums on the drum dollies, and make connections from the drum, through the pump and to the injection point. Prime the pump in recirculation mode. Verify ***Spike Manager*** © operability.	✓
After obtaining agreement with the site operations, *test operability of the complete spiking system by pumping all spiking materials into the injection point using **Spike Manager** © with the most demanding project specific TC.* Thoroughly document the equipment assignments.	✓
Thoroughly agitate all dispersion drums.	✓
When all necessary preparations have been satisfactorily completed, *review all Log Sheet documentation for clarity, completeness, and accuracy.*	✓
If the Client's Spiking Orders to ***ESS*** have not already been signed, *have the Test Manager review & approve the spiking rates, durations, etc. with revisions if appropriate. Please keep the **ESS** PM informed of any revisions as soon as reasonably practical after they are made.*	✓
Check out with the test manager, from the control room (closing out any safety permits), and at the gate. Remove the magnetic ***ESS*** signs from the truck doors.	✓
Call in a status report to the ***ESS*** office daily. Leave voice mail message if no problems have surfaced. Contact the SC &/or PM if problems have surfaced, especially if you need assistance.	✓

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Each Test Day (Test Day = 1, 2, ...): Spiking	✓?
Arrive at or before the set start time each day. Wear all appropriate PPE and ***ESS*** logo apparel as appropriate. Put ***ESS*** signs on truck doors. Check in at the gate.	✓
At the unit control room confirm clock synchronization daily, check in and obtain all required work permits. *Observe all client safety/operational requirements.*	✓
Quickly verify that all equipment remains in working order. Thoroughly agitate all dispersion drums.	✓
Maintain close contact with the test manager. Start spiking sufficiently early that the unit will be conditioned before stack sampling is scheduled to begin. *Obtain & record the same stack testing start & stop times as the Test Manager.*	✓
Record the spiking data – if manually, no less often than 1 data point/10 Minutes per spiking system. Record all times military time [e.g., 00:00 to 24:00 Hours]. *Use the same Test Condition & Run numbering system as tha Test Manager.* Stay outside and in the spiking area whenever a run is in progress or is about to start. Document the spiking material drum # being used on each spiking system at the beginning of each run. Record any changes in equipment assignments.	✓
Keep your work area neat, clean, & orderly. *Frequently inspect the spiking area & all lines for leaks/drips, and clean any indication of even a minor leak immediately.*	✓
To insure that we cover the entire sampling period, continue spiking for 5 or 10 minutes after the declared sampling stop time or until you see the last sampling probe has been removed from the stack, or you are able to confirm that all sampling has finished by some other sure method.	✓
After all spiking has been completed for the day, *review all log sheets for completeness, accuracy, dates, signatures, etc. Police up the spiking area before leaving the area. Double check all valves. Inspect for leaks, drips, etc. and clean them up immediately.*	✓
Confirm the schedule & test plan for the next day. Check out with the Test Manager, the control room (closing out all work permits), & gate. Remove the ***ESS*** signs from the truck.	✓
*Call in a status report to the **ESS** office daily. Leave voice mail message if no problems have surfaced. Contact the SC &/or PM as needed if problems have surfaced, especially if you need assistance.*	✓
Demob Day: After all testing is completed, decontaminate & pack equipment, & travel. Check in as usual.	✓?
Thoroughly decontaminate all equipment by pumping MSO (for organics & dispersions) and water (for aqueous solutions) through the pumps & hoses. Wipe down all equipment to remove any evidence of leaked/spilled spiking material.	✓
Load the equipment, tools, etc. into the ***ESS*** equipment trailer *taking special care to avoid damage to the electronic equipment, electrical wiring, & weigh cells.* Police up the spiking work area thoroughly. *Leave your work area clean and orderly.* *Collect any potentially contaminated items and dispose of them per client's directions.* Record how each piece of equipment performed. ID its application, spiking rate, line back pressure, Identify any equipment maintenance & supplies restocking required.	✓
Request that the Test Manager critique ESS' performance before leaving the site. Check out with the Test Manager, the control room (closing out all work permits), & gate. Remove the ***ESS*** signs from the truck.	✓
Drive back to the *ESS* shop with care to observe defensive driving practices, DOT hour limits. Stop for rest, coffee, and sleep, as needed/required. *Call in a status report to the **ESS** office daily. Leave voice mail message if no problems have surfaced. Contact the SC &/or PM as needed if problems have surfaced, especially if you need assistance.*	✓
Re-stock the equipment at the ***ESS*** shop. Let the SC know about any supplies which need to be ordered. Complete &/or schedule equipment maintenance, as needed.	✓
Provide a complete spiking log package to the ***ESS*** PM with the pages in order (e.g., TC #1, Runs #1,#2, & #3, TC #2, Runs # 1, etc.) by spiking material (i.e., all MCB sheets together & in order, all Cal Verification Logs together & in order) with a briefing of events, problems, ideas, & suggestions.	✓
File all equipment log sheets into their respective equipment specific files	✓
Spiking Technician's Confirming Signature: [signature]	Date: 3/21/2006

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Engineered Spiking Solutions, Inc

ESS Standard Operating Procedure: Tie-In of *ESS'* Spiking Material Delivery Line to Owner's Process

Purpose: The interface of *ESS'* equipment and our client's process is a sensitive step with potential operational, safety, and liability concerns. The purpose of this SOP is to define the physical interface between *ESS'* and our client's (owner's) process equipment and the respective responsibilities for safely managing the injection of *ESS'* spiking materials into the owner's process line.

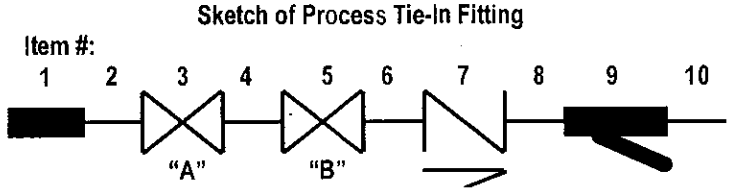
Process Tie-In Fitting: *ESS* has prepared this SOP and a Process Tie-In Fitting [as described in the table & sketch below] to provide:

1. A clear line of demarcation between the parties' areas of responsibility and control.
2. A clearly defined, convenient, and safe means of: (a) connecting *ESS'* spiking material delivery line to, (b) controlling the spiking material flow into, and (c) disconnecting the delivery line from the owner's process line.

The Process Tie-In Fitting is made up of five (5) ½" nipples NPT E/E, two (2) ball valves, one (1) check valve, one (1) Y-strainer, and one (1) quick-connect, dripless coupler assembled in the following order:

Item #	Description:	Controlled by:
1	quick-connect, dripless coupler	*ESS*
2	½" threaded nipple	*ESS*
3	ball valve "A"	*ESS*
4	***½" threaded nipple***	***Interface***
5	ball valve "B"	**Owner**
6	½" threaded nipple	**Owner**
7	check valve	**Owner**
8	½" threaded nipple	**Owner**
9	Y-strainer	**Owner**
10	½" threaded nipple	**Owner**

Sketch of Process Tie-In Fitting



Procedure:

#	Action By:	Action:	When:
1.	*ESS*	Provides this SOP to **Owner**.	Pre-Mob
2.	*ESS*	Provides Process Tie-In Fitting to **Owner**.	Mob Day
3.	**Owner**	Installs Process Tie-In Fitting at agreed injection point in owner's process[1].	Mob Day
4.	*ESS*	Connects spiking material delivery line to quick connect coupler.	Mob Day
5.	**Owner**	Opens Valve "B".	Mob Day
6.	**Owner**	May close valve "B" when spiking is discontinued &/or when necessary for safety.	Thru-out Test
7.	*ESS*	Opens valve "A" after starting spiking pump & closes valve "A" prior to stopping pump.	Thru-out Test
8.	*ESS*	Flushes delivery line & Process Tie-In Fitting. Disconnects delivery line from fitting.	Demob Day
9.	**Owner**	Disconnects the Process Tie-In Fitting from the process & returns it to *ESS*.	Demob Day
Footnote: 1. The ½" nipple (Item #10) on the Process Tie-In Fitting is connected to the process.			

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Attachment IV Field Spiking Data

B. Stack Sampling Start/Stop Times, & Run Durations

TC #/ Run#	Test Date	Sampling Start/Stop Times, & Run Durations			
		Start	End	Run Duration, Min	
TC #1/					
Run #1	3/28/2006	12:10	16:44	274	
Run #2	3/29/2006	11:15	17:00	345	
Run #3a	3/30/2006	11:50	12:39	49	318
Run #3b	3/30/2006	15:30	19:59	269	

Attachment IV Field Spiking Data

C. Field Spiking Log Sheets (Field Data) and Spiking Rate Calculations for:

1. Test Condition #1, Run #1:
   - (a) Mono-Chlorobenzene,
   - (b) Perchloroethylene,
   - (c) Organics Solution, &
   - (d) Metals Solution.

Proje ın: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet) MCB 35

Data ID	Date: 3/28/2006	TC#: 1	Run#: 1	Spiking Material (ID): MCB				Page 1 of 2
Equipment ID:	*Spiking Manager* © #: 2	Pump ID	LMI#	Neptune# 3	Moyno# —	Weigh Scale #: F- 1		MFM# 10-1
Spiking Data File Name: MCB TCIR1			Weather Conditions: warm					

Notes:

0.5833

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Spiking Rate Calculations: Short-Term Average ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	Cum Run Average $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run $Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations (START 12:10 STOP 16:44)
0	07:54	721.85							
1	07:55	721.60							
2	09:05	718.4							
3	09:11	716.15	2.25	6 MIN	0.375				
4	09:21	711.15	5.0	10 MIN	0.50				
5	09:31	705.50	5.65	10 MIN	0.565				
6	09:50	689.35	16.15	22 MIN	0.598				
7	10:20	671.9	17.45	30 MIN	0.5816				
8	10:42	663.8	8.1	14 MIN	0.578				
9	11:02	652.3	11.5	20 MIN	0.575				
10	11:23	640.1	12.2	21 MIN	0.58				
11	11:36	632.6	7.5	13 MIN	0.58				
12	11:52	623.4	9.2	16 MIN	0.58				0.4
13	12:04	616.4	7	12 MIN	0.583				7.5
14	12:13	611.25	5.15	9 MIN	0.57				12.6
15	12:23	605.40	5.85	10 MIN	0.585				18.4
16	12:33	599.7	5.7	10 MIN	0.57	12.69	23 MIN	0.55	24.1
17	12:43	593.9	5.8	10 MIN	0.58	18.49	33 MIN	0.56	29.9
18	12:53	588.2	5.7	10 MIN	0.57	24.19	43 MIN	0.56	35.6
19	13:03	582.4	5.8	10 MIN	0.58	29.99	53 MIN	0.57	41.4
20	13:13	576.6	5.8	10 MIN	0.58	35.79	63 MIN	0.57	47.2
21	13:23	570.9	5.7	10 MIN	0.57	41.49	73 MIN	0.57	52.9
22	13:33	564.5	6.4	10 MIN	0.64	47.89	83 MIN	0.57	59.3
23	13:43	558.7	5.8	10 MIN	0.58	53.69	93 MIN	0.57	65.1
24	13:53	552.8	5.9	10 MIN	0.59	59.59	103 MIN	0.58	71.0
25	14:03	546.9	5.9	10 MIN	0.59	65.49	113 MIN	0.58	76.9
26	14:13	541.1	5.8	10 MIN	0.58	71.29	123 MIN	0.58	82.7
27	14:23	535.4	5.7	10 MIN	0.57	76.99	133 MIN	0.58	88.4
28	14:33	529.5	5.9	10 MIN	0.59	82.88	143 MIN	0.58	94.3
29	14:43	523.5	5.9	10 MIN	0.59	88.68	153 MIN	0.58	100.2

ESS Spiking Technician Signature: [signature] Date: 3/28/2006

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Proje[illegible]n: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID | Date: 3/28/2006 | TC#: 1 | Run#: 1 | Spiking Material (ID): MCB

Page 2 of 2

START 12:10
STOP 16:44

Spiking Rate Data:		Spiking Rate Calculations:						
		Short-Term Average			Cum Run Average			
Time (T) 00:00	Mass (M), Lb	ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	$\Sigma\Delta M$	$\Sigma\Delta T_i$	$Run\ Ave_i = \Sigma_i\Delta M_i/\Sigma_i\Delta T_i$	Comments/Observations
14:53	522.7	5.8	10 MIN	0.58	88.60	153 MIN	0.58	105.8
14:53	517.9	5.0	10 MIN	0.58	94.48	163 MIN	0.58	111.8
15:03	512.0	5.9	10 MIN	0.59	100.38	173 MIN	0.58	117.7
15:03	506.3	5.7	10 MIN	0.57	106.08	183 MIN	0.58	123.4
15:13	500.5	5.8	10 MIN	0.58	111.88	193	0.58	129.2
15:13	494.75				117.68	203	0.58	134.9
15:23	489.45	5.3		0.58	123.48	213	0.58	140.7
15:23	483.10	5.15	10 MIN	0.585	129.33	223	0.58	146.5
16:03	477.3	5.8	10 MIN	0.58	135.13	233	0.58	152.3
16:03	471.6	5.7	10 MIN	0.57	140.83	243	0.58	158.0
16:13	465.8	5.8	10 MIN	0.58	146.63	253	0.579	163.8
16:13	460.1	5.7	10 MIN	0.57	152.33	263	0.579	169.5
16:23	454.4	5.8	10 MIN	0.58	158.13	273	0.578	175.2
16:23	448.5	5.9	10 MIN	0.59				181.0

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Date: 3/28/2006

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Project ID: Westlake PDT TC# 1 Run# 1 Date 3/28/2006
Material: MCB Based on Sample F-1 Rates

∴Start Time 12:10
∴End Time 16:44
Δ Time = 16:44 - 12:10 = 274 Min

∴Start Mass 611.25 + (3 min @ 0.515 lb/min = 1.545 lb) = 612.795

∴End Mass 454.4 - (1 min @ 0.59 lb/min = 0.59 lb) = 453.81

∴Start Mass

∴End Mass

Δ Mass = 612.795 - 453.81 = 158.985 Lb

Spiking Rate = 158.985 Lb / 274 Min = 0.5802 Lb/Min X 60 = 34.81 Lb/Hr

Target Spiking Rate for Test Condition 35.00

Spiking Rate 34.81 Lb ÷ Target Spiking Rate 35.00 Lb = 99.47 % of Target Rate

---

Project ID: Westlake PDT TC# 1 Run# 1 Date 3/28/2006
Material: Based on MCB 10-1 Rates

∴Start Time 12:10
∴End Time 16:44
Δ Time = 16:44 - 12:10 = 274 Min

∴Start Mass 12.6 (3 min @ 0.51 lb/min = 1.53 lb) = 11.07 lb

∴End Mass 169.5 + (1 min @ 0.57 lb/min = 0.57 lb) = 170.07 lb

∴Start Mass

∴End Mass

Δ Mass = 170.07 - 11.07 = 159.0 Lb

Spiking Rate = 159.0 Lb / 274 Min = 0.5803 Lb/Min X 60 = 34.82 Lb/Hr

Target Spiking Rate for Test Condition 35.0 lb/Hr

Spiking Rate 34.82 Lb ÷ Target Spiking Rate 35.0 Lb = 99.48 % of Target Rate

Proje an: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 3/28/2006	TC#: 1	Run#: 1	Spiking Material (ID): Perc		Page 1 of 2
Equipment ID:	Spiking Manager © #: 2	Pump ID —	LMI#	Neptune#	Moyno# —	Weigh Scale #: F- 3	MFM# 10-2
Spiking Data File Name: Perc TC1R1			Weather Conditions: Warm			

Notes:

35 lb/hr 0.583 lb/min

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Spiking Rate Calculations: Short-Term Average ΔMi	ΔTi	Ratei = ΔMi/ΔTi	Cum Run Average ΣiΔMi	ΣiΔTi	Run Avei = ΣΔMi/ ΣiΔTi	Comments/Observations (Start 12:10, Stop 16:44)
0		659.9							
1	09:03	639.8							
2	09:09	634.8	4	6 MIN	0.667				
3	09:19	629.0	5.8	10 MIN	0.58				
4	09:29	623.2	5.8	10 MIN	0.58				
5	09:56	608.0	15.2	27 MIN	0.56				
6	10:26	591.2	16.8	30 MIN	0.56				
7	10:40	582.9	8.3	14 MIN	0.59				
8	11:00	571.4	11.5	20 MIN	0.575				
9	11:21	559.3	12.1	21 MIN	0.576				
10	11:34	551.7	7.6	13 MIN	0.585				
11	11:50	542.7	9	16 MIN	0.56				10.1
12	12:02	535.7	7	12 MIN	0.58				17.1
13	12:11	530.5	5.2	9 MIN	0.577				22.3
14	12:21	524.9	5.6	10 MIN	0.56				27.9
15	12:31	519.1	5.8	10 MIN	0.58	11.977	21 MIN	0.57	33.6
16	12:41	513.3	5.8	10 MIN	0.58	17.77	31 MIN	0.573	39.4
17	12:51	507.5	5.8	10 MIN	0.58	23.57	41 MIN	0.575	45.2
18	13:01	501.6	5.9	10 MIN	0.59	29.47	51 MIN	0.577	51.0
19	13:11	496.0	5.6	10 MIN	0.56	35.07	61 MIN	0.57	56.6
20	13:21	490.4	5.6	10 MIN	0.56	40.67	71 MIN	0.57	62.5
21	13:31	484.6	5.8	10 MIN	0.58	46.47	81 MIN	0.57	68.1
22	13:41	478.2	6.4	10 MIN	0.64	52.87	91 MIN	0.58	73.9
23	13:51	472.6	5.6	10 MIN	0.56	58.47	101 MIN	0.58	79.3
24	14:01	466.9	5.7	10 MIN	0.57	64.17	111 MIN	0.58	85.9
25	14:11	461.2	5.7	10 MIN	0.57	69.87	121 MIN	0.58	91.6
26	14:21	455.4	5.8	10 MIN	0.58	75.67	131 MIN	0.58	97.3
27	14:31	450.0	5.4	10 MIN	0.54	81.07	141 MIN	0.575	103.1
28									
29									

ESS Spiking Technician Signature: [signature] Date: 3/28/2006

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Proje(ın: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID	Date: 3/28/2006	TC#: 1	Run#: 1	Spiking Material (ID): Perc	Page 2 of 2

START 12:10
STOP 16:44

Spiking Rate Data:		Spiking Rate Calculations:						
		Short-Term Average			Cum Run Average			
Time (T) 00:00	Mass (M), Lb	ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	$\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	$Run\ Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
	450.0				51.07			
14:41	444.4	5.6	10 MIN	0.56	86.67	151 MIN	0.57	104.4
14:51	438.8	5.6	10 MIN	0.56	92.27	161 MIN	0.57	114.0
15:01	433.3	5.5	10 MIN	0.55	97.77	171 MIN	0.57	119.5
15:11	426.4	6.9	10 MIN	0.69	104.67	181 MIN	0.58	126.4
15:21	420.4	6.0	10 MIN	0.60	110.67	191 A	0.58	132.3
15:31	414.8	5.6	10 MIN	0.56	116.27	201	0.58	138.0
15:41	409.2	5.6	10 MIN	0.56		211 MIN		143.5
15:51	403.6	5.6	10 MIN	0.56	127.47	221 MIN	0.57	149.1
16:01	397.6	6.0	10 MIN	0.60	133.47	231 MIN	0.58	154.7
16:11	391.1	6.5	10 MIN	0.65	139.97	241 MIN	0.58	160.7
16:21	384.7	6.4	10 MIN	0.64	146.37	251 MIN	0.583	167.2
16:31	378.2	6.5	10 MIN	0.65	152.87	261	0.585	173.8
16:41	371.8	6.4	10 MIN	0.64	159.27	271	0.5877	180.1
16:46	368.9	2.9	5 MIN	0.58				183.0

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Project ID: Westate PDT TC# 1 Run# 1 Date 3/28/2006
Material: Perc Based on Scale F-3 Rates

∴Start Time 12:10
∴End Time 16:44
Δ Time = 16:44 - 12:10 = 274 Min

∴Start Mass 530.5 + (1 min @ 0.56 lb/min = 0.56 lb) = 531.06

∴End Mass 368.9 + (2 min @ 0.58 lb/min = 1.16 lb) = 370.06

∴Start Mass

∴End Mass

Δ Mass = 531.06 - 370.06 = 161.00 Lb

Spiking Rate = 161.00 Lb / 274 Min = 0.5876 Lb / Min X 60 = 35.26 Lb / Hr

Target Spiking Rate for Test Condition 35.00

Spiking Rate 35.26 Lb ÷ Target Spiking Rate 35.00 Lb = 100.7 % of Target Rate

---

Project ID: Westate PDT TC# 1 Run# 1 Date 3/28/2006
Material: Perc Based on MFM 10-2 Rates

∴Start Time 12:10
∴End Time 16:44
Δ Time = 16:44 - 12:10 = 274 Min

∴Start Mass 22.3 - (1 min @ 0.52 lb/min = 0.52 lb) = 21.78

∴End Mass 183.0 - (2 min @ 0.58 lb/min = 1.16 lb) = 181.84

∴Start Mass

∴End Mass

Δ Mass = 181.84 - 21.78 = 160.06 Lb

Spiking Rate = 160.06 Lb / 274 Min = 0.5842 Lb / Min X 60 = 35.05 Lb / Hr

Target Spiking Rate for Test Condition 35.0

Spiking Rate 35.05 Lb ÷ Target Spiking Rate 35.0 Lb = 100.1 % of Target Rate

Proje ıan: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 3/28/2006	TC#: 1	Run#: 1	Spiking Material (ID): Organic Solution			Page 1 of 1
Equipment ID:	*Spiking Manager* © #:	Pump ID	LMI#	Neptune# 4	Moyno#	Weigh Scale #: F- 2	MFM# 10-5
Spiking Data File Name: Organic TCRI			Weather Conditions: Warm				

Notes:

4/16 1/Hr 0.6833

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Spiking Rate Calculations: Short-Term Average ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	Cum Run Average $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run $Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Start 12:50 Stop 16:44 Comments/Observations	
0	09:02	553.0								
1	09:08	549.1	3.9	6MIN	0.65					
2	09:18	542.4		10MIN	0.67					
3	09:28	535.8	6.6	10MIN	0.66					
4	09:55	517.6	18.2	27	0.674					
5	10:25	497.5	20.1	30MIN	0.67					
6	10:39	487.9	9.6	14MIN	0.70					
7	10:59	473.6	14.1	20MIN	0.705					
8	11:20	458.9	14.7	21MIN	0.70					
9	11:33	450.4	8.5	13MIN	0.65					
10	11:49	439.3	11.1	16MIN	0.69					
11	12:08	430.5		12MIN	0.73				12:05	144.7
12	12:10	424.5	6.0	9MIN	0.666				12:15	150.8
13	12:20	418.0	6.5	10MIN	0.65				12:25	157.3
14	12:30	411.1	6.9	10MIN	0.69	13.4	20MIN	0.67	12:30	160.7
15	12:40	404.4	6.7	10MIN	0.67	20.1	30MIN	0.67	12:40	167.4
16	12:50	397.7	6.7	10MIN	0.67	26.8	40MIN	0.67	12:50	
17	13:00	391.0	6.7	10MIN	0.67	33.5	50MIN	0.67	13:00	180.9
18	13:10	383.8	7.2	10MIN	0.72	40.7	60MIN	0.678	13:10	188.1
19	13:20	377.0	6.8	10MIN	0.68	47.5	70MIN	0.679	13:20	194.9
20	13:30	370.2	6.8	10MIN	0.68	54.3	80MIN	0.68	13:30	201.7
21	13:40	363.1	7.1	10MIN	0.71	61.4	90MIN	0.68	13:40	208.3
22	13:50	356.2	6.9	10MIN	0.69	68.3	100MIN	0.683	13:50	215.2
23	14:00	349.3	6.9	10MIN	0.69	75.2	110MIN	0.683	14:00	220.2
24	14:10	342.6	6.7	10MIN	0.67	81.9	120MIN	0.683	14:10	229.0
25	14:20	335.8	6.8	10MIN	0.68	88.7	130MIN	0.683	14:20	235.9
26	14:30	329.0	6.8	10MIN	0.68	95.5	140MIN	0.682	14:30	242.7
27										
28										
29										

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Proje an: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID	Date: 3/28/2006	TC#: 1	Run#: 1	Spiking Material (ID): Organic Solution	Page 2 of 2

START 12:10
STOP 16:44

Spiking Rate Data: Time (T) 00:00	Mass (M), Lb	Spiking Rate Calculations: Short-Term Average ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	Cum Run Average $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run $Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations	
14:30	329.0				95.5	140	0.682	14:30	242.7
14:40	322.1	6.9	10 MIN	0.69	102.4	150	0.682	14:40	249.2
14:50	315.4	6.7	10 MIN	0.67	109.1	160	0.68	14:50	256.5
15:00	308.5	6.9	10 MIN	0.69	116.0	170	0.68	15:00	263.3
15:10	301.6	6.9	10 MIN	0.69	122.9	180	0.68	15:10	270.1
15:20	294.8	6.8	10 MIN	0.68	129.7	190	0.68	15:20	276.9
15:30	288.1	6.7	10 MIN	0.67	136.4	200	0.68	15:30	283.8
15:40	281.1	7.0	10 MIN	0.70	143.4	210	0.68	15:40	290.5
15:50	274.3	6.8	10 MIN	0.68	150.2	220	0.68	15:50	297.4
16:00	267.5	6.8	10 MIN	0.68	157	230	0.68	16:00	304.2
16:10	260.7	6.8	10 MIN	0.68	163.8	240	0.6825	16:10	311.1
16:20	253.9	6.8	10 MIN	0.68	170.6	250	0.6825	16:20	317.9
16:30	247.1	6.8	10 MIN	0.68	177.4	260	0.6823	16:30	324.7
16:40	240.2	6.9	10 MIN	0.69	184.3	270	0.6825	16:40	331.6
16:50	233.3	6.9	10 MIN	0.69	191.2	280	0.6829	16:45	335.8

ESS Spiking Technician Signature: [signature] Date: 3/28/2006

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Project ID: WESTATES PDT TC# 1 Run# 1 Date 3/28/2006
Material: Organic Solution Based on Table F-2 Rates

∴Start Time 12:10
∴End Time 16:44
Δ Time = 16:44 - 12:10 = 274 Min

∴Start Mass 424.5

∴End Mass 233.34 (6 min @ 0.69 lb/min = 4.14 lb) = 237.44

∴Start Mass

∴End Mass

Δ Mass = 424.5 - 237.44 = 187.06 Lb

Spiking Rate = 187.06 Lb / 274 Min = 0.6873 Lb / Min X 60 = 40.96 Lb / Hr

Target Spiking Rate for Test Condition 41.00

Spiking Rate 40.96 Lb ÷ Target Spiking Rate 41.00 Lb = 99.90 % of Target Rate

---

Project ID: Westate PDT TC# 1 Run# 1 Date 3/28/2006
Material: Organic Solution Based on Mix 10-5 Rates

∴Start Time 12:10
∴End Time 16:44
Δ Time = 16:44 - 12:10 = 274 Min

∴Start Mass 144.7 + (5 min @ 0.61 lb/min = 3.05 lb) = 147.75

∴End Mass 335.1 - (1 min @ 0.70 lb/min = 0.70 lb) = 334.4

∴Start Mass

∴End Mass

Δ Mass = 334.4 - 147.75 = 186.65 Lb

Spiking Rate = 186.65 Lb / 274 Min = 0.6812 Lb / Min X 60 = 40.87 Lb / Hr

Target Spiking Rate for Test Condition 41.00

Spiking Rate 40.87 Lb ÷ Target Spiking Rate 41.00 Lb = 99.69 % of Target Rate

Projec n: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 3/28/2006	TC#: 1	Run#: 1	Spiking Material (ID): Pb/Cr III Solution				Page 1 of 2
Equipment ID:	Spiking Manager © #: 2	Pump ID	LMI#	Neptune#	Moyno# —	Weigh Scale #: F- 5		MFM# 10-4
Spiking Data File Name: Pb/Cr III TC1 R1			Weather Conditions: warm					

Notes:

20 lb/gal 0.3333 lb/min

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Spiking Rate Calculations: Short-Term Average ΔMi	ΔTi	Rate$_i$ = ΔMi/ΔTi	Cum Run Average ΣiΔMi	ΣiΔTi	Run Ave$_i$ = ΣiΔMi/ ΣiΔTi	Comments/Observations
									START 12:10 STOP 16:44
0	07:56	544.2							
1	09:04	542.6							
2	09:10	541.0	1.6	6 min	0.2667				
3	09:20	537.5	3.5	10 min	0.35				
4	09:30	533.6	3.9	10 min	0.39				
5	09:57	524.2	9.4	27 min	0.348				
6	10:27	514.1	10.1	30 min	0.336				
7	10:41	508.9	5.2	14 min	0.37				
8	11:01	501.9	7.0	20 min	0.35				
9	11:22	493.2	8.7	21 min	0.41				
10	11:35	490.1	3.1	13 min	0.23				
11	11:51	483.8	6.3	16 min	0.39				
12	12:03	478.9	4.9	12 min	0.40				12:06 93.6
13	12:12	475.4	3.5	9 min	0.38				12:16 97.7
14	12:22	472.2	3.2	10 min	0.32				12:26 100.4
15	12:32	468.7	3.5	10 min	0.35	7.6	22 min	0.336	12:32 102.4
16	12:42	464.9	3.8	10 min	0.38	11.2	32 min	0.35	12:42 106.0
17	12:52	460.4	4.5	10 min	0.45	15.7	42 min	0.374	12:52 110.4
18	13:02	457.0	3.4	10 min	0.34	19.1	52 min	0.36	13:02 113.9
19	13:12	454.0	3.0	10 min	0.30	22.1	62 min	0.35	13:12 116.8
20	13:22	451.1	2.9	10 min	0.29	25.0	72 min	0.347	13:22 119.7
21	13:32	448.2	2.9	10 min	0.29	27.9	82 min	0.34	13:32 122.6
22	13:42	445.1	3.1	10 min	0.31	31.0	92 min	0.337	13:42 125.3
23	13:52	442.1	3.0	10 min	0.30	34.0	102 min	0.333	13:52 128.2
24	14:02	439.0	3.1	10 min	0.31	37.1	112 min	0.33	14:02 131.2
25	14:12	436.0	3.0	10 min	0.30	40.1	122 min	0.328	14:12 134.1
26	14:22	432.5	3.5	10 min	0.35	43.6	132 min	0.33	14:22 137.7
27	14:32	428.7	3.8	10 min	0.38	47.4	142 min	0.333	14:22 141.4
28									
29									

ESS Spiking Technician Signature: [signature] Date: 3/28/2006

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IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID	Date: 3/28/2006	TC#: 1	Run#: 6	Spiking Material (ID): Pb/Cr Solution	Page 2 of 2

Spiking Rate Data:		Spiking Rate Calculations:						
		Short-Term Average			Cum Run Average			STOP 16:44
Time (T) 00:00	Mass (M), Lb	ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	$\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	$Run\ Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
	428.9				47.4			
14:42	425.3	3.4	10 MIN	0.34	50.8	152 MIN	0.334	14:42 144.9
14:52	422.1	3.2	10 MIN	0.32	54	162	0.333	14:52 148.2
15:02	418.7	3.4	10 MIN	0.34	57.4	172	0.333	15:02 151.5
15:12	415.1	3.6	10 MIN	0.34	61.0	182	0.335	15:12 155.2
15:22	411.9	3.2	10 MIN	0.32	64.2	192	0.333	15:22 158.5
15:32	408.5	3.4	10 MIN	0.34	67.6	202	0.333	15:32 161.8
15:42	405.3	3.2	10 MIN	0.32	70.9	212	0.334	15:42 165.0
15:52	402.0	3.3	10 MIN	0.33	74.2	222	0.334	15:52 168.3
16:02	398.5	3.5	10 MIN	0.35	77.7	232	0.335	16:02 171.8
16:12	395.0	3.5	10 MIN	0.35	81.2	242	0.335	16:12 175.3
16:22	391.2	3.8	10 MIN	0.38	85.0	252	0.337	16:22 179.0
16:32	387.9	3.3	10 MIN	0.33	88.3	262	0.337	16:32 182.3
16:42	384.9	3.0	10 MIN	0.30	91.3	272	0.335	16:42 185.0
16:47	383.4	1.5	5 MIN	0.30				16:47 186.5

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Project ID: Westate PDT
Material: Pb/Cr III Solution

TC# 1 Run# 1 Date 3/28/2006
Based on Scale F-5 Rates

∴Start Time 12:10
∴End Time 16:44
Δ Time = 16:44 - 12:10 = 274 Min

∴Start Mass 475.4 + (2 min @ 0.35 lb/min = 0.70 lb) = 476.1

∴End Mass 386.9 - (2 min @ 0.30 lb/min = 0.60 lb) = 384.3

∴Start Mass

∴End Mass

Δ Mass = 476.1 - 384.3 = 91.8 Lb

Spiking Rate = 91.8 Lb / 274 Min = 0.3350 Lb/Min X 60 = 20.10 Lb/Hr

Target Spiking Rate for Test Condition 20.00

Spiking Rate 20.10 Lb ÷ Target Spiking Rate 20.00 Lb = 100.5 % of Target Rate

---

Project ID: Westate PDT
Material: Pb/Cr III Solution

TC# 1 Run# 1 Date 3/28/2006
Based on LCF-4/10-4 Rates

∴Start Time 12:10
∴End Time 16:44
Δ Time = 16:44 - 12:10 = 274 Min

∴Start Mass 93.6 + (4 min @ 0.36 lb/min = 1.44 lb) = 95.04

∴End Mass 185.0 + (2 min @ 0.30 lb/min = 0.60 lb) = 185.6

∴Start Mass

∴End Mass

Δ Mass = 185.6 - 95.04 = 90.56 Lb

Spiking Rate = 90.56 Lb / 274 Min = 0.3305 Lb/Min X 60 = 19.83 Lb/Hr

Target Spiking Rate for Test Condition 20.00

Spiking Rate 19.83 Lb ÷ Target Spiking Rate 20.00 Lb = 99.15 % of Target Rate

Attachment IV Field Spiking Data

C. Field Spiking Log Sheets (Field Data) and Spiking Rate Calculations for:

2. Test Condition #1 Run #2:
   - (a) Mono-Chlorobenzene,
   - (b) Perchloroethylene,
   - (c) Organics Solution, &
   - (d) Metals Solution.

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 3/29/2006	TC#: 1	Run#: 2	Spiking Material (ID): MCB			Page 1 of 2
Equipment ID:	*Spiking Manager* © #: 2	Pump ID	LMI# —	Neptune# 3	Moyno# —	Weigh Scale #: F- 1	MFM# 0-1
Spiking Data File Name: MCB TC1R2			Weather Conditions:				

Notes:

3516/hr 0.5853

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Spiking Rate Calculations: Short-Term Average ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	Cum Run Average $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run $Ave_i = \Sigma \Delta M_i / \Sigma_i \Delta T_i$	Start 11:15 STOP 17:00 Comments/Observations
0	09:24	719.5							
1	09:35	713.2	6.3	19 MIN	0.33				
2	10:05	707.1	6.1	20 MIN	0.33				
3	10:27	692.7	14.4	22 MIN	0.65				
4	10:37	687.2	5.2						9.2
5	10:47	681.0	6.2	10 MIN	0.62				15.4
6	10:57	675.8	5.2	10 MIN	0.52				20.6
7	11:07	669.5	6.3	10 MIN	0.63				26.8
8	11:17	663.4	6.1	10 MIN	0.61		2 MIN		33.0
9	11:29	655.2	8.2	12 MIN	0.68	9.42	14 MIN	0.67	41.2
10	11:37	651.5	3.7	8 MIN	0.46	13.12	22 MIN	0.596	44.9
11	11:47	646.9	4.6	10 MIN	0.46	17.72	32 MIN	0.554	49.5
12	11:57	641.6	5.3	10 MIN	0.53	23.02	42 MIN	0.55	54.8
13	12:07	635.4	6.2	10 MIN	0.62	29.22	52 MIN	0.56	61.0
14	12:17	629.2	6.2	10 MIN	0.62	35.42	62 MIN	0.57	67.2
15	12:27	622.8	6.4	10 MIN	0.64	41.82	72 MIN	0.58	73.4
16	12:37	616.9	5.9	10 MIN	0.59	47.72	82 MIN	0.58	79.3
17	12:47	610.6	6.3	10 MIN	0.63	54.02	92	0.58	85.7
18	12:57	605.1	5.5	10 MIN	0.55	59.52	102	0.583	91.0
19	13:07	599.6	5.5	10 M	0.55	65.02	112	0.58	97.5
20	13:17	594.0	5.6	10 M	0.56	70.62	122	0.58	103.0
21	13:27	587.5	6.5	10 M	0.65	77.12	132	0.584	109.6
22	13:42	578.2							
23	13:47	575.1	12.4	10 M	0.62	89.52	152	0.588	121.0
24	13:57	568.8	6.3	10 M	0.63	95.82	162	0.59	127.4
25	14:07	562.7	6.1	10 M	0.61	101.92	172	0.59	133.5
26	14:17	556.4	6.3	10 M	0.63	108.22	182	0.59	139.8
27	14:27	551.1	5.3	10 M	0.53	113.52	192	0.59	145.1
28									
29									

ESS Spiking Technician Signature: [signature] Date: 3/29/2006

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Proje... lan: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID	Date: 3/29/2006	TC#: 1	Run#: 2	Spiking Material (ID): MCB	Page 2 of 2

Spiking Rate Data:		Spiking Rate Calculations:						
		Short-Term Average			Cum Run Average			
Time (T) 00:00	Mass (M), Lb	ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	$\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	$Run\ Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
14:27	551.1							STOP 17:00
14:37	545.8	5.3	10 MIN	0.53	113.52	192		
14:47	540.2	5.6	10 MIN	0.56	118.82	202	0.588	150.4
14:57	534.5	5.7	10 MIN	0.57	124.42	212	0.587	156.0
15:07	529.1	5.4	10 MIN	0.54	130.12	222	0.586	161.7
15:17	523.9	5.2	10 MIN	0.52	135.52	232	0.584	167.1
15:27	518.0	5.9	10 MIN	0.59	140.72	242	0.581	172.3
15:37	512.2	5.8	10 MIN	0.58	146.62	252	0.58	178.2
15:47	506.4	5.8	10 MIN	0.58		271		184.0
15:57	500.5	5.9	10 MIN	0.58	158.22	272	0.581	189.9
16:07	494.3	6.2	10 MIN	0.62	164.12	282	0.581	195.7
16:17	488.5	5.8	10 MIN	0.58	170.32	292	0.583	201.9
16:27	482.4	6.1	10 MIN	0.61	176.12	302	0.583	207.7
16:37	476.4	6.0	10 MIN	0.60	182.22	312	0.584	213.8
16:47	470.7	5.7	10 MIN	0.57	188.22	322	0.584	219.8
16:57	464.8	5.9	10 MIN	0.59	193.92	332	0.584	225.8
17:07	459.0	5.8	10 MIN	0.58		342		231.5
						352		237.5

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Date: 3/29/2006

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Project ID: Westate PDT TC# 1 Run# 2 Date 3/29/2006
Material: MCB Based on Scale F-1 Rates

∴Start Time 11:15
∴End Time 17:00
Δ Time = 17:00 - 11:15 = 345 Min

∴Start Mass 663.4 + (2 min @ 0.61 lb/min = 1.22 lb) = 664.62

∴End Mass 464.8 - (3 min @ 0.58 lb/min = 1.74 lb) = 463.06

∴Start Mass

∴End Mass

Δ Mass = 664.62 - 463.06 = 201.56 Lb

Spiking Rate = 201.56 Lb / 345 Min = 0.5842 Lb/Min X 60 = 35.05 Lb/Hr

Target Spiking Rate for Test Condition 35.00

Spiking Rate 35.05 Lb ÷ Target Spiking Rate 35.00 Lb = 100.1 % of Target Rate

---

Project ID: Westate PDT TC# 1 Run# 2 Date 3/29/2006
Material: MCB Based on MFW Rates

∴Start Time 11:15
∴End Time 17:00
Δ Time = 17:00 - 11:15 = 345 Min

∴Start Mass 33.0 - (2 min @ 0.62 lb/min = 1.24 lb) = 31.76 lb

∴End Mass 464.8 + (3 min @ 0.58 lb/min = 1.74 lb) = 233.3 lb

∴Start Mass

∴End Mass

Δ Mass = 233.3 - 31.76 = 201.54 Lb

Spiking Rate = 201.54 Lb / 345 Min = 0.5842 Lb/Min X 60 = 35.05 Lb/Hr

Target Spiking Rate for Test Condition 35.00

Spiking Rate 35.05 Lb ÷ Target Spiking Rate 35.00 Lb = 100.1 % of Target Rate

Proje lan: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 3/29/2006	TC#: 1	Run#: 2	Spiking Material (ID): Pure				Page 1 of 2
Equipment ID:	Spiking Manager © #: 2	Pump ID –	LMI#	Neptune#	Moyno# –		Weigh Scale #: F- 3	MFM# 10-2
Spiking Data File Name: Pure TC1 R2			Weather Conditions:					

Notes:

35 lb/hr 0.583

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Short-Term Average: ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	Cum Run Average: $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	$Run\ Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
									Start 11:15 Stop 17:00
0	09:23	772.0							
1	09:43	758.2	13.8	20min	0.69				
2	10:03	746.7	11.5	20min	0.575				
3	10:24	733.7	13	22min	0.59				
4	10:35	727.7	6	10m	0.60				
5	10:45	721.8	5.9	10min	0.59				
6	10:55	716.1	5.7	10min	0.57				8.1
7	11:05	710.2	5.9	10min	0.59				14.0
8	11:15	704.6	5.6	10min	0.56				19.6
9	11:25	699.0	5.6	10min	0.56	5.6	10min	0.56	25.2
10	11:35	692.8	6.2	10min	0.62	11.8	20min	0.59	31.4
11	11:45	687.0	5.8	10min	0.58	17.6	30min	0.58	37.2
12	11:55	681.1	5.9	10min	0.59	23.5	40	0.587	43.1
13	12:05	674.8	6.3	10min	0.63	29.8	50min	0.59	49.4
14	12:15	669.2	5.6	10min	0.56	35.4	60min	0.59	55.0
15	12:25	663.5	5.7	10min	0.57	41.1	70min	0.587	60.8
16	12:35	657.5	6.0	10min	0.60	47.1	80min	0.583	66.8
17	12:45	651.3	6.2	10min	0.62	53.3	90	0.59	73.0
18	12:55	645.6	5.7	10min	0.57	59.4	100	0.59	78.0
19	13:05	639.9	5.7	10m	0.57	65.1	110	0.59	83.7
20	13:15	634.4	5.5	10m	0.55	70.6	120	0.588	89.2
21	13:25	628.8	5.6	10m	0.56	76.2	130	0.586	94.8
22	13:40	620.3							
23	13:45	617.8	11.4	20min	0.57	87.6	150	0.584	106.2
24	13:55	611.8	5.6	10m	0.56	93.2	160	0.582	111.8
25	14:05	606.0	5.8	10m	0.58	99	170	0.582	117.6
26	14:15	600.3	5.7	10m	0.57	104.7	180	0.58	123.3
27	14:25	594.2	6.1	10m	0.1	110.8	190	0.583	129.4
28									
29									

ESS Spiking Technician Signature: [signature] Date: 3/29/2006

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Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID | Date 3/29/2006 | TC#: 1 | Run#: 2 | Spiking Material (ID): Pere | Page 2 of 2

START 11:15
STOP 17:00

Spiking Rate Data:		Spiking Rate Calculations:						
		Short-Term Average			Cum Run Average			
Time (T) 00:00	Mass (M), Lb	ΔM_i	ΔT_i	Rate$_i$ = $\Delta M_i/\Delta T_i$	$\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run Ave$_i$ = $\Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
14:25	594.2				110.8	190		129.4
14:35	587.7	6.5	10 min	0.65	117.3	200	0.586	135.9
14:45	581.9	5.8	10 m	0.58	123.1	210	0.586	141.7
14:55	576.3	5.6	10 m	0.56	128.7	220	0.585	147.3
15:05	570.2	6.1	10 m	0.61	134.8	230	0.586	153.4
15:15	564.4	5.8	10 m	0.58	140.6	240	0.586	159.2
15:25	558.4	6.0	10 m	0.60	146.6	250	0.586	165.3
15:35	552.5	5.9	10 m			260		171.1
15:45	546.5	6.0	10 m	0.60	152.5	270	0.587	177.1
15:55	540.7	5.8	10 m	0.58	158.3	280	0.586	182.9
16:05	534.6	6.1	10 MIN	0.61	170.4	290	0.587	189.0
16:15			10 MIN			300		
16:25	522.8	11.8	10 MIN	0.59	182.2	310	0.587	200.9
16:35	516.4	6.4	10 MIN	0.64	188.6	320	0.589	207.0
16:45	510.8	5.6	10 MIN	0.56	194.2	330	0.588	212.6
16:55	505.2	5.6	10 MIN	0.56	199.8	340	0.587	218.2
17:05	499.6	5.6	10 MIN	0.56		350		223.8

ESS Spiking Technician Signature: [signature]

Date: 3/29/2006

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Project ID: Westate PDT
Material: Pere

TC# 1 Run# 2 Date 3/29/2006
Based on Scale F-3 Rates

∴Start Time 11:15
∴End Time 17:00
Δ Time = 17:00 - 11:15 = 345 Min

∴Start Mass 704.6

∴End Mass 505.2 - (5 min @ 0.56 lb/min = 2.8 lb) = 502.4

∴Start Mass

∴End Mass

Δ Mass = 704.6 - 502.4 = 202.2 Lb

Spiking Rate = 202.2 Lb / 345 Min = 0.5861 Lb / Min X 60 = 35.17 Lb / Hr

Target Spiking Rate for Test Condition 35.00

Spiking Rate 35.17 Lb ÷ Target Spiking Rate 35.00 Lb = 100.4 % of Target Rate

---

Project ID: Westate PDT
Material: Pere

TC# 1 Run# 2 Date 3/29/2006
Based on Flow 10-2 Rates

∴Start Time 11:15
∴End Time 17:00
Δ Time = 17:00 - 11:15 = 345 Min

∴Start Mass 19.6 lb

∴End Mass 223.2 - (5 min @ 0.56 lb/min = 2.8 lb) = 221.0

∴Start Mass

∴End Mass

Δ Mass = 221.0 - 19.6 = 201.4 Lb

Spiking Rate = 201.4 Lb / 345 Min = 0.5838 Lb / Min X 60 = 35.03 Lb / Hr

Target Spiking Rate for Test Condition 35.00

Spiking Rate 35.03 Lb ÷ Target Spiking Rate 35.00 Lb = 100.1 % of Target Rate

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 3/29/2006	TC#: 1	Run#: 2	Spiking Material (ID): Organic Solution	Page 1 of 2
Equipment ID:	Spiking Manager © #:	Pump ID –	LMI# —	Neptune# 4 / Moyno# — / Weigh Scale #: F- 2	MFM# 0.5
Spiking Data File Name: Organic FG1R2			Weather Conditions: Clear		

Notes:

41 lb/hr 0.683

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Spiking Rate Calculations: Short-Term Average ΔMi	ΔTi	Rate$_i$ = ΔMi/ΔTi	Cum Run Average ΣiΔMi	ΣiΔTi	Run Ave$_i$ = ΣiΔMi/ ΣiΔTi	Comments/Observations (START 11:15 STOP 17:00)
0	09:20	509.9							
1	09:42	494.0	15.9	22 MIN	0.72				14.0
2	10:02	480.2	13.8	20 MIN	0.69				28.0
3	10:24	464.8	15.4	22 min	0.70				42.9
4	10:34	458.9	5.9	10 min	0.59	12.55			48.9
5	10:44	452.0	6.9	10 MIN	0.69				55.8
6	10:54	444.9	7.1	10 MIN	0.71				63.3
7	11:04	437.3	7.6	10 min	0.76				70.6
8	11:14	430.4	6.9	10 min	0.69				77.6
9	11:24	423.9	6.5	10 min	0.65	5.85	9 MIN	0.65	84.1
10	11:34	417.2	6.7	10 MIN	0.67	12.55	19 MIN	0.66	90.9
11	11:44	409.8	7.4	10 MIN	0.74	19.95	29 MIN	0.687	98.2
12	11:54	403.2	6.6	10 MIN			39 MIN		104.9
13	12:04	397.2	6.0	10 MIN	0.60	32.55	49 MIN	0.664	111.1
14	12:14	391.0	6.2	10 MIN	0.62	38.75	59 MIN	0.66	117.2
15	12:24	384.4	6.6	10 MIN	0.66	45.35	69 min	0.66	123.7
16	12:34	376.6	7.8	10 MIN	0.78	53.15	79 MIN	0.672	131.5
17	12:44	367.9	8.7	10 MIN	0.87	61.85	89	0.69	140.0
18	12:54	361.0	6.9	10 MIN	0.69	68.75	99	0.69	147.1
19	13:04	354.1	6.9	10 m	0.69	75.65	109	0.69	153.9
20	13:14	347.4	6.7	10 m	0.67	82.35	119	0.69	160.7
21	13:24	340.5	6.9	10 m	0.69	89.25	129	0.69	167.6
22	13:34	330.4							177.8
23	13:44	327.0	13.5	20 m	0.675	102.75	149	0.68	181.2
24	13:54	320.2	6.8	10 m	0.68	109.55	159	0.68	187.9
25	14:04	313.2	7.0	10 m	0.70	116.55	169	0.689	194.7
26	14:14	306.9	6.3	10 m	0.63	122.85	179	0.686	201.1
27	14:24	300.7	6.2	10 m	0.62	129.05	189	0.683	207.2
28									
29									

ESS Spiking Technician Signature: Scott [illegible] Date: 3/29/2006

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Project Plan: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID | Date: 3/29/2006 | TC#: 1 | Run#: 2 | Spiking Material (ID): Organic Solution | Page 2 of 2

START 11:15
STOP 17:00

Spiking Rate Data:		Spiking Rate Calculations:						
		Short-Term Average			Cum Run Average			
Time (T) 00:00	Mass (M), Lb	ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	$\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	$Run\ Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
14:24	300.7				129.05			
14:34	293.8	6.9	10min	0.69	135.95	199	0.683	214.1
14:44	286.9	6.9	10m	0.69	142.85	209	0.683	221.1
14:54	280.0	6.9	10m	0.69	149.75	219	0.683	227.9
15:04	272.9	7.1	10m	0.71	156.85	229	0.684	235.0
15:14	266.0	6.9	10m	0.69	163.75	239	0.685	241.8
15:24	259.1	6.9	10m	0.69	170.65	249	0.685	248.7
15:34	251.7	7.4	10m	0.74		259		255.8
15:44	245.2	6.5	10m	0.65	184.25	269	0.684	262.6
15:54	238.0	7.2	10m	0.72	191.45	279	0.686	270.0
16:04	231.3	6.7	10m	0.67	198.15	289	0.685	276.6
16:14	217.		10MIN			299		290.4
16:24	217.3	14	20MIN	0.7	212.15	309	0.686	297.4
16:34	210.4	6.9	10MIN	0.69	219.05	319	0.686	297.4
16:44	204.0	6.4	10MIN	0.64	225.45	329	0.685	303.6
16:54	198.4	5.6	10MIN	0.56	231.05	339	0.6815	309.3
17:04	191.5	6.9	10MIN	0.69	237.95	349	0.6818	316.1

ESS Spiking Technician Signature: Scott Neal

Date: 3/29/2006

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Project ID: Westate PDT TC# 1 Run# 2 Date 3/29/2006
Material: Organic Solution Based on Scale F-2 Rates

∴Start Time 11:15
∴End Time 17:00
Δ Time = 17:00 - 11:15 = 345 Min

∴Start Mass 430.4 - (1 min @ 0.69 lb/min = 0.69 lb) = 429.71

∴End Mass 198.4 - (1 min @ 0.69 lb/min = 4.14 lb) = 194.26

∴Start Mass

∴End Mass

Δ Mass = 429.71 - 194.26 = 235.45 Lb

Spiking Rate = 235.45 Lb / 345 Min = 0.6825 Lb/Min X 60 = 40.95 Lb/Hr

Target Spiking Rate for Test Condition 41.0

Spiking Rate 40.95 Lb ÷ Target Spiking Rate 41.0 Lb = 0.997 % of Target Rate

---

Project ID: Westate PDT TC# 1 Run# 2 Date 3/29/2006
Material: Organic Solution Based on MPM 10-9 Rates

∴Start Time 11:15
∴End Time 17:00
Δ Time = 17:00 - 11:15 = 345 Min

∴Start Mass 77.6 + (1 min @ 0.70 lb/min = 0.70 lb) = 78.3

∴End Mass 319.3 + (1 min @ 0.68 lb/min = 4.08 lb) = 313.38

∴Start Mass

∴End Mass

Δ Mass = 313.38 - 78.3 = 235.08 Lb

Spiking Rate = 235.08 Lb / 345 Min = 0.6814 Lb/Min X 60 = 40.88 Lb/Hr

Target Spiking Rate for Test Condition 41.0

Spiking Rate 40.88 Lb ÷ Target Spiking Rate 41.0 Lb = 0.997 % of Target Rate

Proje an: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 3/29/2006	TC#: 1	Run#: 2	Spiking Material (ID): Pb/Cu Solution				Page 1 of 2
Equipment ID:	Spiking Manager © #: 2	Pump ID	LMI#	Neptune#	Moyno# —	Weigh Scale #: F-5		MFM# 04
Spiking Data File Name: Pb/Cu TC1 R2			Weather Conditions: Clear					

Notes:

20 lb/hr 0.3333 lb/min

START 11:15
END 17:00

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Short-Term Average: ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	Cum Run Average: $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run $Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
0	09:24	325.8							
1	09:44	320.8	5.0	20 min	0.25				5.0
2	10:04	314.5	6.3	20 min	0.315				10.9
3	10:26	308.9	5.6	22 min	0.25				17.4
4	10:36	304.9	4	10 min	0.4				21.3
5	10:46	300.6	4.3	10 min	0.43				25.4
6	10:56	297.0	3.6	10 min	0.36				29.1
7	11:06	293.5	3.5	10 min	0.35				32.7
8	11:16	289.8	3.7	10 min	0.37		1 min		36.3
9	11:26	285.5	4.3	12 min	0.35	4.67	13 min	0.35	40.6
10	11:36	283.2	2.3	8 min	0.29	6.97	21 min	0.331	43.1
11	11:46	280.2	3.0	10 min	0.30	9.97	31 min	0.32	46.0
12	11:56	276.8	3.4	10 min	0.34	13.37	41 min	0.326	49.4
13	12:06	273.3	3.5	10 min	0.35	16.87	51 min	0.33	52.9
14	12:16	269.5	3.8	10 min	0.38	20.67	61 min	0.338	56.6
15	12:26	265.6	3.9	10 min	0.39	24.57	71 min	0.34	60.4
16	12:36	261.6	4.0	10 min	0.40	28.57	81 min	0.35	64.4
17	12:46	257.8	3.8	10 min	0.38	32.37	91	0.35	68.4
18	12:56	255.2	2.6	10 min	0.26	34.97	101	0.34	71.1
19	13:06	252.5	2.7	10 min	0.27	37.67	111	0.339	73.8
20	13:16	249.7	2.8	10 min	0.28	40.47	121	0.334	75.5
21	13:26	246.7	3.0	10 min	0.3	43.47	131	0.331	79.5
22	13:41	242.1							84.2
23	13:46	240.5	6.2	20 min	0.31	49.67	151	0.328	85.7
24	13:56	237.3	3.2	10 min	0.32	52.87	161	0.328	90.0
25	14:06	234.1	3.2	10 min	0.32	56.07	171	0.327	92.2
26	14:16	231.0	3.1	10 min	0.31	59.17	181	0.326	95.3
27	14:26	227.6	3.4	10 min	0.34	62.57	191	0.327	98.7
28									
29									

ESS Spiking Technician Signature: [signature] Date: 3/29/2006

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Proje ıan: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID	Date: 3/29/2006	TC#: 1	Run#: 2	Spiking Material (ID): Pb/Cl Solution	Page 2 of 2

Spiking Rate Data:		Spiking Rate Calculations:						
		Short-Term Average			Cum Run Average			
Time (T) 00:00	Mass (M), Lb	ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	$\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	$Run\ Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
14:26	277.6				62.57	191		
14:36	274.1	3.5	10min	0.35	66.07	201	0.323	102.3
14:46	270.7	3.4	10m	0.34	69.07	211	0.324	105.7
14:56	266.9	3.8	10m	0.38	71.87	221	0.325	109.3
15:06	263.5	3.4	10m	0.34	75.27	231	0.325	113.0
15:16	259.8	3.7	10m	0.37	78.97	241	0.327	116.7
15:26	255.0	3.8	10m	0.38	82.77	251	0.329	120.4
15:36	252.0	4.0	10m			261		124.5
15:46	248.2	3.8	10m	0.38	90.57	271	0.334	128.2
15:56	244.1	4.1	10m	0.41	94.67	281	0.336	132.1
16:06	240.0	4.1	10m	0.41	98.77	291	0.339	135.9
16:16	23					301		143.2
16:26	233.1	6.9	10m	0.69	105.67	311	0.339	143.2
16:36	230.0	3.1	10m	0.31	108.77	321	0.338	146.1
16:46	228.2	1.8	10m	0.18	110.57	331	0.334	148.0
16:56	226.0	2.2	10m	0.22	112.77	341	0.3307	150.0
17:06	221.6	4.4	10m	0.44	117.17	351	0.3338	154.5

ESS Spiking Technician Signature: Scott Wise

Date: 3/29/2006

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Project ID: Westate PDT TC# 1 Run# 2 Date 3/29/2006
Material: Pb/Cr III Solution Based on Scale 8-5 Rates

∴ Start Time 11:15
∴ End Time 17:00
Δ Time = 17:00 - 11:15 = 345 Min

∴ Start Mass 339.8 + (1 min @ 0.37 lb/min = 0.37 lb) = 340.17

∴ End Mass 226.0 + (4 min @ 0.44 lb/min = 1.76 lb) = 224.24

∴ Start Mass

∴ End Mass

Δ Mass = 340.17 - 224.24 = 115.93 Lb

Spiking Rate = 115.93 Lb / 345 Min = 0.3360 Lb/Min X 60 = 20.16 Lb/Hr

Target Spiking Rate for Test Condition 20.00

Spiking Rate 20.16 Lb ÷ Target Spiking Rate 20.00 Lb = 100.8 % of Target Rate

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Project ID: Westate PDT TC# 1 Run# 2 Date 3/29/2006
Material: Pb/Cr III Solution Based on MEM 10-4 Rates

∴ Start Time 11:15
∴ End Time 17:00
Δ Time = 17:00 - 11:15 = 345 Min

∴ Start Mass 36.3 - (1 min @ 0.36 lb/min = 0.36 lb) = 35.94

∴ End Mass 150.0 + (4 min @ 0.45 lb/min = 1.8 lb) = 151.8

∴ Start Mass

∴ End Mass

Δ Mass = 151.8 - 35.94 = 115.86 Lb

Spiking Rate = 115.86 Lb / 345 Min = 0.3358 Lb/Min X 60 = 20.15 Lb/Hr

Target Spiking Rate for Test Condition 20.00

Spiking Rate 20.15 Lb ÷ Target Spiking Rate 20.00 Lb = 100.7 % of Target Rate

Attachment IV Field Spiking Data

C. Field Spiking Log Sheets (Field Data) and Spiking Rate Calculations for:

3. Test Condition #1, Run #3:
   - (a) Mono-Chlorobenzene,
   - (b) Perchloroethylene,
   - (c) Organics Solution, &
   - (d) Metals Solution.

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 2/30/2006	TC#: 1	Run#: 3	Spiking Material (ID): MCB				Page 1 of 2
Equipment ID:	*Spiking Manager* © #: 1		Pump ID	LMI# –	Neptune# 3	Moyno# –	Weigh Scale #: F- 1	MFM# ND-1
Spiking Data File Name: MCB TC1R3				Weather Conditions:				

Notes:

35 lb/ha 0.5833 lb/min

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Spiking Rate Calculations: Short-Term Average ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	Cum Run Average $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run $Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
									START 11:50 STOP 12:39 START 13:30 END 14:59
0	10:20	608.6							
1	10:45	593.7	14.9	25 MIN	0.596				
2	11:06	581.3	12.4	21 MIN	0.59				
3	11:11	575.9	5.4	9 MIN	0.60				11.3
4	11:36	564.1	11.8	20 MIN	0.59				23.1
5	11:45	558.2	5.9	10 MIN	0.59				29.0
6	11:55	552.3	5.9	10 MIN	0.59	2.55	5 MIN	0.59	34.9
7	12:09	544.0					19 MIN		
8	12:15	540.4	11.9	20 MIN	0.595	14.25	25 MIN	0.594	46.8
9	12:35	528.8		20 MIN					
10	12:39	523.3	17.1	34 MIN	0.50	31.95	49 MIN	0.65	63.9
11	15:07	434.6	7.6						
12	15:20	437.0	8.6	13 MIN	0.58				155.2
13	15:29	426.6	5.4	9 MIN	0.59				160.6
14	15:39	420.8	5.8	10 MIN	0.58	37.17	58 MIN	0.64	166.4
15	15:49	416.9	3.9	10 MIN	0.59	43.07	68 MIN	0.63	172.3
16	15:59	409.2	5.7	10 MIN	0.59	48.77	78 MIN	0.62	178.0
17	16:09	403.6	5.6	10 MIN	0.58	54.37	88 MIN	0.617	183.6
18	16:19	398.0	5.6	10 MIN	0.58	59.97	98 MIN	0.61	189.3
19	16:29	392.2	5.8	10 MIN	0.58	65.77	108 MIN	0.608	195.1
20	16:39	386.5	5.7	10 MIN	0.57	71.47	118 MIN	0.605	200.8
21	16:49	380.8	5.7	10 MIN	0.57	77.17	128 MIN	0.60	206.5
22	16:59	375.0	5.8	10 MIN	0.58	82.97	138 MIN	0.60	212.2
23	17:09	369.2	5.8	10 MIN	0.58	88.77	148 MIN	0.599	218.0
24	17:19	363.4	5.8	10 MIN	0.58	94.57	158 MIN	0.598	223.7
25	17:29	357.5	5.9	10 MIN	0.59	100.47	168 MIN	0.59	229.6
26	17:39	351.9	5.6	10 MIN	0.56	106.07	178 MIN	0.596	235.2
27	17:49	345.7	6.2	10 MIN	0.62	112.27	188 MIN	0.597	241.4
28	17:59	340.3	5.4	10 MIN	0.54	117.67	198 MIN	0.594	246.8
29	18:09	334.9	5.4	10 MIN	0.54	123.07	208 MIN	0.591	252.2

ESS Spiking Technician Signature: Scott Neal

Date: 3/30/2006

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Projec. ..n: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID	Date: 3/30/2006	TC#: 1	Run#: 3	Spiking Material (ID): MCB	Page 2 of 2

Spiking Rate Data:		Spiking Rate Calculations:						
		Short-Term Average			Cum Run Average			STOP 19:59
Time (T) 00:00	Mass (M), Lb	ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	$\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	$Run\ Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
18:09	334.9				123.07	208		252.2
18:19	324.1	5.8	10 min	0.58	128.87	218	0.59	258.0
18:29	328.2	5.9	10 min	0.59	1	228		263.9
18:39	317.4	5.8	10 min	0.58	140.57	238	0.59	269.6
18:49	311.6	5.8	10 min	0.58	146.37	248	0.59	275.4
18:59	305.8	5.8	10 min	0.58	152.17	258	0.589	281.2
19:09	300.0	5.8	10 min	0.58	157.97	268	0.589	287.0
19:19	294.3	5.7	10 min	0.57	163.67	278	0.58	292.8
19:29	288.6	5.7	10 min	0.57	169.37	288	0.588	298.5
19:39	282.8	5.8	10 min	0.58	175.17	298	0.588	304.2
19:49	277.1	5.7	10 min	0.57	180.87	308	0.588	310.0
19:59	271.4	5.7	10 min	0.57	186.57	318	0.588	315.7

ESS Spiking Technician Signature: [signature] Date: 3/30/2006

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Project ID: Westate PDT TC# 1 Run# 3 Date 3/30/2006
Material: MCB Based on SCALE F-1 Rates

∴Start Time 11:50 / 15:30
∴End Time 12:39 / 19:59
Δ Time = (12:39 - 11:50) + (19:59 - 15:30) = 318 Min

∴Start Mass 552.3 + (5 min @ 0.59 lb/min = 2.95 lb) = 555.25

∴End Mass 523.3

∴Start Mass 426.6 - (1 min @ 0.58 lb/min = 0.58 lb) = 426.02

∴End Mass 271.4

Δ Mass = (555.25 - 523.3) + (426.02 - 271.4) = 126.57 Lb
126.57

Spiking Rate = 126.57 Lb / 318 Min = 0.5967 Lb/Min X 60 = 35.20 Lb/Hr

Target Spiking Rate for Test Condition 35.0

Spiking Rate 35.20 Lb ÷ Target Spiking Rate 35.0 Lb = 100.6 % of Target Rate

---

Project ID: Westate PDT TC# 1 Run# 3 Date 3/30/2006
Material: MCB Based on MFM 10-1 Rates

∴Start Time 11:50 / 15:30
∴End Time 12:39 / 19:59
Δ Time = (12:39 - 11:50) + (19:59 - 15:30) = 318 Min

∴Start Mass 29.0 + (5 min @ 0.59 lb/min = 2.95 lb) = 31.95

∴End Mass 63.9

∴Start Mass 160.6 + (1 min @ 0.58 lb/min = 0.58 lb) = 161.8

∴End Mass 315.7

Δ Mass = (315.7 - 161.8) + (63.9 - 31.95) = 185.85 Lb

Spiking Rate = 185.85 Lb / 318 Min = 0.5844 Lb/Min X 60 = 35.07 Lb/Hr

Target Spiking Rate for Test Condition 35.0

Spiking Rate 35.07 Lb ÷ Target Spiking Rate 35.0 Lb = 100.2 % of Target Rate

Project: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 7/30/2006	TC#: 1	Run#: 3	Spiking Material (ID): Perc				Page 1 of 2
Equipment ID:	Spiking Manager © #: 2	Pump ID —	LMI#	Neptune#	Moyno# —		Weigh Scale #: F- 3	MFM# 10.2
Spiking Data File Name: Perc TC1R3			Weather Conditions: Warm					

Notes:

35 lb/hr 0.583 lb/hr-N

START 11:50
STOP 12:39
START 15:30 END 19:59

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Short-Term Average: ΔM_i	ΔT_i	Rate$_i$ = $\Delta M_i/\Delta T_i$	Cum Run Average: $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run Ave$_i$ = $\Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
0	10:18	490.3							
1	10:43	475.2	15.1	25	0.604				
2	11:03	463.3	11.9	20	0.595				
3	11:13	457.4	5.9	10	0.59				
4	11:23	446.1	11.3	20	0.565				3.6
5	11:43	440.5	5.6	10 min	0.56				10.2
6	11:53	435.0	5.5	10 min	0.55	1.65	3 min	0.55	15.8
7	12:03	426.9							
8	12:13	423.1	11.9	20 min	0.595	13.55	23 min	0.589	27.7
9	12:33	411.7							
10	12:39	406.1	17			30.55	49	0.623	44.6
11	15:05	327.5							123.2
12	15:17	321.1	6.4	12	0.53				129.6
13	15:27	314.9	6.2	10	0.62				135.8
14	15:37	309.5	5.4	10 min	0.54	3.78	7 m	0.54	141.2
15	15:47	302.2	6.3	10 m	0.63	10.08	17	0.59	147.5
16	15:57	297.8	5.4	10 m	0.54	46.03	76	0.605	153.8
17	16:07	292.5	5.3	10 m	0.53	51.33	86	0.596	159.2
18	16:17	286.8	5.7	10 min	0.53	57.03	96	0.59	164.5
19	16:27	281.2	5.6	10 min	0.56	62.63	106	0.59	170.2
20	16:37	275.8	5.4	10 m	0.54	68.03	116	0.586	175.6
21	16:47	270.1	5.7	10 m	0.57		126		181.2
22	16:57	264.7	5.4	10 m	0.54	79.13	136	0.582	186.7
23	17:07	258.9	5.8	10 m	0.58	84.93	146	0.58	192.3
24	17:17	253.1	5.8	10 m	0.53	90.13	156	0.57	198.2
25	17:27	247.3	5.8	10 m	0.58	96.53	166	0.581	203.9
26	17:37	241.6	5.7	10 m	0.57	102.23	176	0.580	209.7
27	17:47	235.8	5.8	10 m	0.57	108.03	186	0.580	215.5
28	17:57	230.0	5.8	10 m	0.58		196		221.3
29	18:07	223.7	6.3	10 m	0.63	120.13	206	0.583	227.5

ESS Spiking Technician Signature: Scott [illegible]

Date: 7/30/2006

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IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID	Date: 3/30/2006	TC#: 1	Run#: 3	Spiking Material (ID): Pere

Page 2 of 2

Spiking Rate Data: Time (T) 00:00	Mass (M), Lb	Spiking Rate Calculations: Short-Term Average ΔM_i	ΔT_i	Rate$_i$ = $\Delta M_i/\Delta T_i$	Cum Run Average $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run Ave$_i$ = $\Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
								Start 19:59
18:07	223.7							
18:17	217.8	5.9	10 min	0.59	120.13	206	0.583	227.5
18:27	212.0	5.8	10 min	0.58	126.03	216	0.583	233.4
18:37	206.1	5.9	10 min	0.59	131.83	226	0.5833	239.2
18:47	200.2	5.9	10 min	0.59	137.73	236	0.5836	245.0
18:57	194.4	5.8	10 min	0.58	143.63	246	0.5838	251.0
19:07	188.6	5.8	10 min	0.58	149.43	256	0.5837	256.8
19:17	182.8	5.8	10 min	0.58	155.23	266	0.5835	262.6
19:27	177.0	5.8	10 min	0.58	161.03	276	0.5834	268.4
19:37	171.1	5.9	10 min	0.59	166.93	286	0.5833	274.1
19:47	165.3	5.8	10 min	0.58	172.73	296	0.5835	280.0
19:57	159.5	5.8	10 min	0.58	178.53	306	0.5834	285.8
20:07	153.7	5.8	10 min	0.58	184.33	316	0.5833	291.6
					190.13	326	0.5832	297.3

ESS Spiking Technician Signature: Scott Neal

Date: 3/30/2006

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Project ID: Westinghouse PDT TC# 1 Run# 3 Date 3/30/2006
Material: PCRC Based on Sample #3 Rates

∴Start Time 11:50 / 15:30
∴End Time 12:39 / 19:59
Δ Time = 12:39-11:50 = 49 / 19:59-15:30 = 269 = 318 Min

∴Start Mass 435.0 + (3 mins @ 0.55 lb/min = 1.65 lb) = 436.65

∴End Mass 406.1

∴Start Mass 314.9 − (3 min @ 0.54 lb/min = 1.62 lb) = 313.28

∴End Mass 159.5 − (2 min @ 0.58 lb/min = 1.01 lb) = 158.44

Δ Mass = (436.65-406.1) + (313.28 - 158.44) = 185.39 Lb

Spiking Rate = 185.39 Lb / 318 Min = 0.5830 Lb/Min X 60 = 34.98 Lb/Hr

Target Spiking Rate for Test Condition 35.00

Spiking Rate 34.98 Lb ÷ Target Spiking Rate 35.00 Lb = 99.94 % of Target Rate

---

Project ID: Westinghouse PDT TC# 1 Run# 3 Date 3/30/2006
Material: PCRC Based on HPM 10-2 Rates

∴Start Time 11:50 / 15:30
∴End Time 12:39 / 19:59
Δ Time = (19:59 - 15:30) + (12:39 - 11:50) = 318 Min

∴Start Mass 10.2 + (7 min @ 0.55 lb/min = 3.85 lb) = 14.05

∴End Mass 44.6

∴Start Mass 135.8 + (3 min @ 0.54 lb/min = 1.62 lb) = 137.42

∴End Mass 291.6

Δ Mass = (291.6 - 135.8) + (44.6 - 14.05) = 184.73 Lb

Spiking Rate = 184.73 Lb / 318 Min = 0.5809 Lb/Min X 60 = 34.85 Lb/Hr

Target Spiking Rate for Test Condition 35.0

Spiking Rate 34.85 Lb ÷ Target Spiking Rate 35.0 Lb = 99.58 % of Target Rate

Projec ,n: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 3/30/2006	TC#: 1	Run#: 3	Spiking Material (ID): Organic Solution				Page 1 of 2
Equipment ID:	*Spiking Manager* © #:		Pump ID	LMI# —	Neptune# 4	Moyno# —	Weigh Scale #: F- 2	MFM# 103
Spiking Data File Name: Organic TC1 R3				Weather Conditions: warm				

Notes:

4/16/HW 0.68 16/MIN

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Short-Term Average: ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	Cum Run Average: $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run $Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	START 11:50 STOP 12:39 RESTART 15:30 Comments/Observations
0	10:17	508.8							
1	10:[illegible]	493.1	15.7	25	0.628				15.6
2	11:02	480.3	12.8	20	0.64				28.4
3	11:12	473.1	7.2	10	0.72				35.7
4	11:32	459.1	14	20	0.70				49.7
5	11:42	452.2	6.9	10 MIN	0.69				56.7
6	11:52	446.8	5.4	10 MIN	0.54	1.08	2 MIN	0.54	62.2
7	12:06	438.2		14 MIN					70.8
8	12:12	434.6	12.2	20 MIN	0.61	13.28	22 MIN	0.603	74.4
9	12:32	429.0							
10	12:39	406.0				41.28	49 MIN	0.85	[illegible]
11	15:04	360.4							100.6
12	15:16	352.7	7.7	12 MIN	0.64				
13	15:26	344.4	8.3						8.1
14	15:36	335.6	8.8	10 MIN	0.88	5.28	6 MIN	0.88	18.9
15	15:46	330.4	5.2	10 MIN	0.52	10.48	16 MIN	0.655	22.1
16	15:56	325.0	5.4	10 MIN	0.54	59.76	75	0.77	27.5
17	16:06	319.3	5.7	10 M	0.57	62.46	85	0.74	33.2
18	16:16	313.8	5.5	10 M	0.55	68.95	95	0.72	38.7
19	16:26	308.2	5.6	10 M	0.56	74.55	105	0.71	44.3
20	16:36	302.6	5.6	10 M	0.56	80.15	115	0.696	49.8
21	16:46	297.0	5.6	10 M	0.56		125		55.5
22	16:56	291.5	5.5	10 M	0.55	91.25	135	0.675	61.0
23	17:06	285.2	6.3	10 M	0.63	97.55	145	0.672	67.3
24	17:16	278.8	6.4	10 M	0.64	103.95	155	0.67	73.6
25	17:26	272.4	6.4	10 M	0.64	110.35	165	0.668	80.0
26	17:36	265.7	6.7	10 M	0.67	117.05	175	0.6688	86.8
27	17:46	158.3	7.4	10 M	0.74	124.45	185	0.672	93.5
28	17:56	251.7	6.6	10 M	0.66	130.95	195	0.6772	100.1
29	18:06	244.3	7.4	10 M	0.74	138.45	205	0.675	107.5

Test Stopped Due to Drum Change

ESS Spiking Technician Signature: Scott Neal

Date: 3/30/2006

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Projec ..n: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.b Spiking Log (2nd & subsequent sheets for each run) 19:59

Data ID	Date: 3/30/2006	TC#: 1	Run#: 3	Spiking Material (ID): Organic Solution	Page 2 of 2

Spiking Rate Data:		Spiking Rate Calculations:						
		Short-Term Average			Cum Run Average			
Time (T) 00:00	Mass (M), Lb	ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	$\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	$Run\ Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
18:06	244.3				138.45	205	0.675	107.5
18:16	237.2	7.1	10 MIN	0.71	145.55	215	0.676	114.6
18:26	229.9	7.3	10 MIN	0.73	152.85	225	0.679	121.8
18:36	222.9	7.0	10 MIN	0.70	159.85	235	0.68	128.8
18:46	216.0	6.9	10 MIN	0.69	166.75	245	0.6806	135.8
18:56	209.1	6.9	10 MIN	0.69	173.65	255	0.6809	142.7
19:06	202.1	7.0	10 MIN	0.70	180.65	265	0.6817	149.7
19:16	195.2	6.9	10 MIN	0.69	187.55	275	0.6820	156.6
19:26	188.4	6.8	10 MIN	0.68	194.35	285	0.682	163.4
19:36	181.5	6.9	10 MIN	0.69	201.25	295	0.682	170.3
19:46	174.6	6.9	10 MIN	0.69	208.15	305	0.682	177.1
19:56	167.8	6.8	10 MIN	0.68	214.95	315	0.682	183.9
20:06	161.9	6.9	10 MIN	0.69	221.85	325	0.6826	190.8

STOP 19:16
STRT 19:59

ESS Spiking Technician Signature: [signature] Date: 3/30/2006

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Project ID: Westlake PDT TC# 1 Run# 3 Date 3/30/2006
Material: Organic Solution Based on Scale 8-2 Rates

∴Start Time 11:50/15:30
∴End Time 12:39/19:59
Δ Time = (12:39-11:50)+(19:59-15:30) = 318 Min

∴Start Mass 446.8+(2min @ 0.54lb/min = 1.08lb) = 447.88

∴End Mass 406.0

∴Start Mass 344.4 - (4min @ 0.88lb/min = 3.52lb) = 340.88

∴End Mass 167.8-(3min @ 0.69lb/min 2.07lb) = 165.3

Δ Mass = (447.88-406.0)+(340.88-165.3) = 217.46 Lb

Spiking Rate = 217.46 Lb / 318 Min = 0.6838 Lb/Min X 60 = 41.03 Lb/Hr

Target Spiking Rate for Test Condition 41.0

Spiking Rate 41.03 Lb ÷ Target Spiking Rate 41.0 Lb = 100.1 % of Target Rate

---

Project ID: Westlake PDT TC# 1 Run# 3 Date 3/30/2006
Material: Organic Solution Based on ACFM 10-5 Rates

∴Start Time 11:50/15:30
∴End Time 12:39/19:59
Δ Time = (12:39-11:50)+(19:59-15:30) = 318 Min

∴Start Mass 62.2+(2min @ 0.55lb/min = 1.1lb) = 61.1

∴End Mass 102.6

∴Start Mass 8.14(4min @ 0.87lb/min = 3.48lb) = 11.58

∴End Mass 183.9+(3min @ 0.69lb/min = 2.07lb) = 185.97

Δ Mass = (185.97-11.58)+(102.6-61.1) = 215.89 Lb

Spiking Rate = 215.89 Lb / 318 Min = 0.6788 Lb/Min X 60 = 40.73 Lb/Hr

Target Spiking Rate for Test Condition 41.0

Spiking Rate 40.73 Lb ÷ Target Spiking Rate 41.0 Lb = 0.994 % of Target Rate

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 3/30/2006	TC#: 1	Run#: 3	Spiking Material (ID): Pb/Cu Solution			Page 1 of 2
Equipment ID:	Spiking Manager © #:		Pump ID	LMI# —	Neptune#	Moyno# —	Weigh Scale #: F- 5 / MFM# 10-4
Spiking Data File Name: Pb/Cu TC1R3				Weather Conditions:			

Notes:

2016 / Hr 0.3333 lb/min

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Spiking Rate Calculations: Short-Term Average ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	Cum Run Average $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run $Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
0	10:19	428.3							
1	10:26	428.3	1.0	25 min	0.32				13.0
2	11:00	412.4							14.9
3	11:14	410.3	3.1	9 min	0.34				23.0
4	11:34	403.5	6.8	20 min	0.34				29.7
5	11:44	400.0	3.5	10 min	0.35				33.2
6	11:54	396.9	3.1	10 min	0.31	1.24	4 min	0.31	36.2
7	12:07	392.1							41.0
8	12:14	390.0	6.9	20 min	0.31	8.14	24 min	0.339	43.1
9	12:34	384.2							
10	12:39	383.9	6.1	25 min	0.244	14.24	49 min	0.29	49.3
11	15:06	347.2							
12	15:19	342.1							
13	15:28	338.5	4.1	10 min	0.41				90.6
14	15:38	334.5	4.0	10 min	0.40	3.2	8 min		95.5
15	15:48	331.0	3.9	10 min	0.35	30.24	67 min		102.1
16	15:58	327.5	3.5	10 min	0.36	34.14	77 min	0.317	105.6
17	16:08	323.9	3.6	10 min	0.36	38.04	87 min	0.327	109.1
18	16:18	320.2	3.7	10 min	0.37	41.74	97 min	0.327	112.8
19	16:28	316.7	3.5	10 min	0.35	45.24	107 min	0.329	116.3
20	16:38	313.1	3.6	10 min	0.36	48.84	117 min	0.332	123.6
21	16:48	309.4	3.7	10 min	0.37	52.54	127 min	0.334	127.2
22	16:58	305.8	3.6	10 min	0.36	56.14	137 min	0.336	131.0
23	17:08	301.9	3.9	10 min	0.39	60.04	147 min	0.340	135.7
24	17:18	297.8	4.1	10 min	0.41	64.14	157 min	0.3448	139.8
25	17:28	294.2	3.6	10 min	0.36	67.74	167 min	0.345	143.1
26	17:38	290.9	3.3	10 min	0.33	71.04	177 min	0.345	146.4
27	17:48	287.5	3.4	10 min	0.34	74.44	187 min	0.344	149.5
28	17:58	285.3	2.2	10 min	0.22	76.64	197 min	0.339	151.7
29	18:08	282.5	2.8	10 min	0.28	79.44	207 min	0.335	150.4

ESS Spiking Technician Signature: [signature] Date: 3/30/2006

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IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID	Date: 3/30/2006	TC#: 1	Run#: 3	Spiking Material (ID): Pb/Cl Solution	Page 2 of 2

END 19:59

Spiking Rate Data:		Spiking Rate Calculations:						
		Short-Term Average			Cum Run Average			
Time (T) 00:00	Mass (M), Lb	ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	$\Sigma_i \Delta M_i$	$\Sigma \Delta T_i$	$Run\ Ave_i = \Sigma_i \Delta M_i / \Sigma \Delta T_i$	Comments/Observations
18:07	272.5				69.44	207 min	0.335	152.4
18:17	279.3	3.2	10 min	0.32	72.64	217 min	0.345	153.6
18:28	276.4	2.9	10 min	0.29	75.54	227 min	0.333	158.5
18:38	278.2	3.6	10 min	0.36	79.14	237 min	0.334	160.1
18:48	279.5	3.3	10 min	0.33	82.44	247 min	0.338	163.4
18:58	280.2	3.3	10 min	0.33	85.74	257 min	0.336	166.7
19:08	283.5	3.2	10 min	0.32	88.94	267 min	0.332	169.9
19:18	284.8	3.2	10 min	0.32	92.14	277 min	0.333	173.1
19:28	286.5	3.3	10 min	0.33	95.44	287 min	0.333	176.2
19:38	288.3	3.2	10 min	0.32	98.64	297 min	0.332	179.5
19:48	290.0	3.3	10 min	0.33	101.94	307 min	0.332	182.8
19:58	291.7	3.3	10 min	0.33	105.24	317 min	0.332	186.1
20:08	293.5	3.2	10 min	0.32	108.44	327 min	0.332	189.3

ESS Spiking Technician Signature: Scott Wise

Date: 3/30/2006

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Project ID: Westate PDT TC# 1 / Run# 3 Date 3/30/2006
Material: Pb/Cr III Solution Based on Scale F5 Rates

∴Start Time 11:50/15:30
∴End Time 12:39/19:59
Δ Time = (19:59-15:30)+(12:39-11:50) = 318 Min

∴Start Mass 396.9+(4min @ 0.31lb/min = 1.24lb) = 398.14

∴End Mass 383.9

∴Start Mass 335.5-(2min @ 0.40lb/min = 0.80lb) = 337.7

∴End Mass 246.7-(1min @ 0.33 lb/min = 0.33lb) = 246.37

Δ Mass = (398.14-383.9)+(337.7-246.37) = 105.57 Lb

Spiking Rate = 105.57 Lb / 318 Min = 0.3320 Lb/Min X 60 = 19.92 Lb/Hr

Target Spiking Rate for Test Condition 20.00

Spiking Rate 19.92 Lb ÷ Target Spiking Rate 20.0 Lb = 99.6 % of Target Rate

---

Project ID: Westate PDT TC# 1 Run# 3 Date 3/30/2006
Material: Pb/Cr III Solution Based on MPM 10-4 Rates

∴Start Time 11:50/15:30
∴End Time 12:39/19:59
Δ Time = (19:59-15:30)+(12:39-11:50) = 318 Min

∴Start Mass 36.2-(4min @ 0.30lb/min = 1.2lb) 35.0

∴End Mass 49.3

∴Start Mass 94.6+(2min @ 0.39lb/min = 0.78lb) = 95.38

∴End Mass 186.1+(1min @ 0.32lb/min = 0.32lb) = 186.42

Δ Mass (186.42-95.38)+(49.3-35.0) = 105.34 Lb

Spiking Rate = 105.34 Lb / 318 Min = 0.3313 Lb/Min X 60 = 19.88 Lb/Hr

Target Spiking Rate for Test Condition 20.00

Spiking Rate 19.88 Lb ÷ Target Spiking Rate 20.0 Lb = 99.4 % of Target Rate

Attachment IV Field Spiking Data

A. Executed Test Manager Spiking Orders to ***ESS*** and other operations logs,
B. Stack Sampling Start/Stop Times, and
C. Field Spiking Log Sheets (Field Data) and Spiking Rate Calculations for:
   1. Test Condition #1, Run #1:
      (a) Mono-Chlorobenzene,
      (b) Perchloroethylene,
      (c) Organics Solution, &
      (d) Metals Solution,
   2. Test Condition #1, Run #2:
      (a) Mono-Chlorobenzene,
      (b) Perchloroethylene,
      (c) Organics Solution, &
      (d) Metals Solution, and
   3. Test Condition #1, Run #3:
      (a) Mono-Chlorobenzene,
      (b) Perchloroethylene,
      (c) Organics Solution, &
      (d) Metals Solution.

Attachment IV Field Spiking Data

A. Executed Test Manager Spiking Orders to ***ESS*** and other operations logs

IV.E. Client Test Manager's Spiking Orders[1] to *ESS*:

Section I Initial Spiking Orders[1]:

Spiking:		Spiking Rate, Lb/Hr		Pump Type/Size	Spiking Duration, Hrs	Specie/Mat'l Reqr'ed/ Mat'l Provided, Lb/Lb/# Drums
Specie	Material	As Specie	As Mat'l			
POHCs:						
MCB	MCB	35	35	Neptune #3	32	1,120/1500/3-500 [Net] Lb Drums
C_2Cl_4	C_2Cl_4	35	35	LMI #10	32	1120/1400/2-700 [Net] Lb Drums
Metals:						
Pb	Pb/Cr^{III} Solution	.1	20	LMI #7	32	3.2/640/1-640 [Net] Lb Drum
Cr^{III}	Pb/Cr^{III} Solution	.35	20	LMI #7	32	11.2/640/1-640 [Net] Lb Drum
Organic Mixture:						
	Organic Mixture		41	Neptune #4	32	1312Lb-2 @ 451[Net] Lb Drum1@ 410 [Net] Lb Drum
Toluene		17			32	
CH_2Cl_2		8			32	
Naphthalene		8			32	
Et Glycol		8			32	

Approved by Client/Test Manager: [signature] **Date:** 3 /27/200[illegible]

Section II Revised Spiking Orders[2]:

Revision 1:

Approved by Client/Test Manager: **Date:** / /200

Revision 2:

Approved by Client/Test Manager: **Date:** / /200

Revision 3:

Approved by Client/Test Manager: **Date:** / /200

Section III Critique, Suggestions, and Comments[3]:

by Client/Test Manager: **Date:** / /200

Footnotes:
1. Section I contains *ESS'* understanding of the spiking requirements (Spiking Orders) for this test. Please review, revise (as necessary), and initial/date to indicate that the Spiking Orders (as revised) are correct.
2. Section II is provided for field revisions to the Spiking Orders by the Client/Test Manager, as needed. Please document the required changes, and initial/date the new orders.
3. Please provide a critique of *ESS'* performance on this test, offer suggestions for improving the value of our products and services to you, and/or (if warranted) identify aspect(s) of our products and services with which you are pleased.

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IV. Spiking Plan Transmittal Checklist:			
Spiking Plan Component:		**Applicable & Att'ed?**	**Received & Accepted?**
Gen Info Re:	A. Test ID & Site Contact Information.	✓	✓
Phase IV Proj Exec.	B. Overall Test Execution SOP & Checklist.	✓	✓
Spiking Plan:	C. Spiking Mat'ls, Species, Rates, & Durations, plus Quantities of Mat'ls & Equip Required.	✓	✓
	D. Test Schedule.	✓	✓
	E. Spiking Orders.	✓	✓
	F. Pump Assignments.	✓	✓
	G. (1) Terms & Conditions, and (2) Support Requirements.	✓	✓
	H. Project Rate Schedule	✓	✓
	I. Field Scale Set-Up, Adjustment, Calibration, and Calibration Verification SOP & Checklist.	✓	✓
	J. On-Site Solution Preparation SOPs, and & Documentation Worksheets:	✓	✓
	(1) Pb, Cr^{III} Solution	✓	✓
	K. Other Project Execution Related Log Sheets, Checklists, & Worksheets:	✓	✓
	(1) Daily Operation	✓	✓
	(2) ***ESS*** Field Scale Corner Test Report	✓	✓
	(3) Pre- & Post-Test Calib. Verification Report	✓	✓
	(4) Eq Operation & Maintenance	✓	✓
	(5) Spiking Log Sheets:	✓	✓
	(a) 1st Sheet per run	✓	✓
	(b) 2nd Sheets per run	✓	✓
Prepared & Approved by:	Date:	Received & Accepted by: RJD	Date: 3/16/2006
Footnotes:			

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IV.A. General Project ID & Site Contact Information:	
1. Test Type:	CPT
2. Test Dates	Week of 3/27/2006 (Mob on 24th & Spike on 28th through 31st)
3. Test Location	Parker, Az @ US Filter (See maps, etc.)
4. Contact Name & #	Drew Boyard (928) 669-5758
5. Other Information	

IV.B. Project Phase IV: Test Execution SOP & Checklist	
Test Day = -1: Travel to the Test Site:	✓?
Safely drive to the test site obeying all traffic laws and applicable DOT requirements including the DOT Time Log/limits. Stop for coffee/coke &/or rest, as needed. Plan to arrive in the vicinity of the test facility on the day prior to the equipment set-up day to get a good nights rest.	✓
Test Day = 0 (Mob or Equipment Set-Up Day):	
Arrive at the gate early wearing PPE and ***ESS*** logo apparel, as appropriate. Place magnetic ***ESS*** signs on truck doors. Check in at the gate. Receive any site provided safety or other training.	✓
Make contact with the client representative & clarify any uncerlainties about the test schedule, spiking rates, management of contaminated materials, & establish the method of communications.	✓
Check into the unit control room, obtain required permits, and synchronize ***Spike Manager*** © clock.	✓
Locate all spiking materials; confirm lot numbers, drum counts, condition of containers, etc. When there are multiple drums of a given material (say N drums), mark each drum numerically from 1 to N and then use the drums in numerical order.	✓
Confirm availability of: (a) required utilities, (b) a flat, level, hard surfaced work area, and (c) reasonable access to the spiking injection point.	✓
Ask for fork lift or other assistance, as needed, to off load equipment and relocate to the spiking area. *Use **ESS'** dolly &/or hand truck, &/or request assistance from operator/test manager, as needed, to protect your back from stains.*	✓
Set-up secondary containment (if not already available). Lay down impermeable barrier to protect the work surface from possible contamination, even if secondary containment is available. *Only open spiking material containers when the containers are inside the secondary containment area.*	✓
Set-up and verify calibration of weigh scales. Set-up the spiking pumps, MFMs, drums on the drum dollies, and make connections from the drum, through the pump and to the injection point. Prime the pump in recirculation mode. Verify ***Spike Manager*** © operability.	✓
After obtaining agreement with the site operations, *test operability of the complete spiking system by pumping all spiking materials into the injection point using **Spike Manager** © with the most demanding project specific TC.* Thoroughly document the equipment assignments.	✓
Thoroughly agitate all dispersion drums.	✓
When all necessary preparations have been satisfactorily completed, *review all Log Sheet documentation for clarity, completeness, and accuracy.*	✓
If the Client's Spiking Orders to ***ESS*** have not already been signed, *have the Test Manager review & approve the spiking rates, durations, etc. with revisions if appropriate. Please keep the **ESS** PM informed of any revisions as soon as reasonably practical after they are made.*	✓
Check out with the test manager, from the control room (closing out any safety permits), and at the gate. Remove the magnetic ***ESS*** signs from the truck doors.	✓
Call in a status report to the ***ESS*** office daily. Leave voice mail message if no problems have surfaced. Contact the SC &/or PM if problems have surfaced, especially if you need assistance.	✓

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Each Test Day (Test Day = 1, 2, ...): Spiking	✓?
Arrive at or before the set start time each day. Wear all appropriate PPE and ***ESS*** logo apparel as appropriate. Put ***ESS*** signs on truck doors. Check in at the gate.	✓
At the unit control room confirm clock synchronization daily, check in and obtain all required work permits. *Observe all client safety/operational requirements.*	✓
Quickly verify that all equipment remains in working order. Thoroughly agitate all dispersion drums.	✓
Maintain close contact with the test manager. Start spiking sufficiently early that the unit will be conditioned before stack sampling is scheduled to begin. *Obtain & record the same stack testing start & stop times as the Test Manager.*	✓
Record the spiking data – if manually, no less often than 1 data point/10 Minutes per spiking system. Record all times military time [e.g., 00:00 to 24:00 Hours]. *Use the same Test Condition & Run numbering system as tha Test Manager.* Stay outside and in the spiking area whenever a run is in progress or is about to start. Document the spiking material drum # being used on each spiking system at the beginning of each run. Record any changes in equipment assignments.	✓
Keep your work area neat, clean, & orderly. *Frequently inspect the spiking area & all lines for leaks/drips, and clean any indication of even a minor leak immediately.*	✓
To insure that we cover the entire sampling period, continue spiking for 5 or 10 minutes after the declared sampling stop time or until you see the last sampling probe has been removed from the stack, or you are able to confirm that all sampling has finished by some other sure method.	✓
After all spiking has been completed for the day, *review all log sheets for completeness, accuracy, dates, signatures, etc. Police up the spiking area before leaving the area. Double check all valves. Inspect for leaks, drips, etc. and clean them up immediately.*	✓
Confirm the schedule & test plan for the next day. Check out with the Test Manager, the control room (closing out all work permits), & gate. Remove the ***ESS*** signs from the truck.	✓
*Call in a status report to the **ESS** office daily. Leave voice mail message if no problems have surfaced. Contact the SC &/or PM as needed if problems have surfaced, especially if you need assistance.*	✓
Demob Day: After all testing is completed, decontaminate & pack equipment, & travel. Check in as usual.	✓?
Thoroughly decontaminate all equipment by pumping MSO (for organics & dispersions) and water (for aqueous solutions) through the pumps & hoses. Wipe down all equipment to remove any evidence of leaked/spilled spiking material.	✓
Load the equipment, tools, etc. into the ***ESS*** equipment trailer *taking special care to avoid damage to the electronic equipment, electrical wiring, & weigh cells.* Police up the spiking work area thoroughly. *Leave your work area clean and orderly.* *Collect any potentially contaminated items and dispose of them per client's directions.* Record how each piece of equipment performed. ID its application, spiking rate, line back pressure, Identify any equipment maintenance & supplies restocking required.	✓
Request that the Test Manager critique ESS' performance before leaving the site. Check out with the Test Manager, the control room (closing out all work permits), & gate. Remove the ***ESS*** signs from the truck.	✓
Drive back to the *ESS* shop with care to observe defensive driving practices, DOT hour limits. Stop for rest, coffee, and sleep, as needed/required. *Call in a status report to the **ESS** office daily. Leave voice mail message if no problems have surfaced. Contact the SC &/or PM as needed if problems have surfaced, especially if you need assistance.*	✓
Re-stock the equipment at the ***ESS*** shop. Let the SC know about any supplies which need to be ordered. Complete &/or schedule equipment maintenance, as needed.	✓
Provide a complete spiking log package to the ***ESS*** PM with the pages in order (e.g., TC #1, Runs #1,#2, & #3, TC #2, Runs # 1, etc.) by spiking material (i.e., all MCB sheets together & in order, all Cal Verification Logs together & in order) with a briefing of events, problems, ideas, & suggestions.	✓
File all equipment log sheets into their respective equipment specific files	✓
Spiking Technician's Confirming Signature: [signature]	Date: 3/21/2006

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Engineered Spiking Solutions, Inc

ESS Standard Operating Procedure: Tie-In of *ESS'* Spiking Material Delivery Line to Owner's Process

Purpose: The interface of *ESS'* equipment and our client's process is a sensitive step with potential operational, safety, and liability concerns. The purpose of this SOP is to define the physical interface between *ESS'* and our client's (owner's) process equipment and the respective responsibilities for safely managing the injection of *ESS'* spiking materials into the owner's process line.

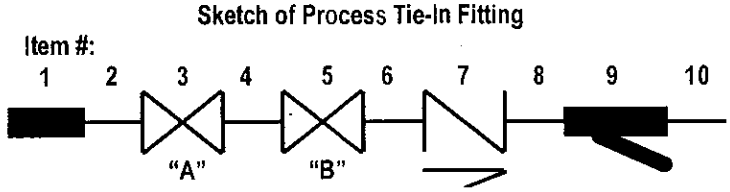
Process Tie-In Fitting: *ESS* has prepared this SOP and a Process Tie-In Fitting [as described in the table & sketch below] to provide:

1. A clear line of demarcation between the parties' areas of responsibility and control.
2. A clearly defined, convenient, and safe means of: (a) connecting *ESS'* spiking material delivery line to, (b) controlling the spiking material flow into, and (c) disconnecting the delivery line from the owner's process line.

The Process Tie-In Fitting is made up of five (5) ½" nipples NPT E/E, two (2) ball valves, one (1) check valve, one (1) Y-strainer, and one (1) quick-connect, dripless coupler assembled in the following order:

Item #	Description:	Controlled by:
1	quick-connect, dripless coupler	*ESS*
2	½" threaded nipple	*ESS*
3	ball valve "A"	*ESS*
4	***½" threaded nipple***	***Interface***
5	ball valve "B"	**Owner**
6	½" threaded nipple	**Owner**
7	check valve	**Owner**
8	½" threaded nipple	**Owner**
9	Y-strainer	**Owner**
10	½" threaded nipple	**Owner**

Sketch of Process Tie-In Fitting



Procedure:

#	Action By:	Action:	When:
1.	*ESS*	Provides this SOP to **Owner**.	Pre-Mob
2.	*ESS*	Provides Process Tie-In Fitting to **Owner**.	Mob Day
3.	**Owner**	Installs Process Tie-In Fitting at agreed injection point in owner's process[1].	Mob Day
4.	*ESS*	Connects spiking material delivery line to quick connect coupler.	Mob Day
5.	**Owner**	Opens Valve "B".	Mob Day
6.	**Owner**	May close valve "B" when spiking is discontinued &/or when necessary for safety.	Thru-out Test
7.	*ESS*	Opens valve "A" after starting spiking pump & closes valve "A" prior to stopping pump.	Thru-out Test
8.	*ESS*	Flushes delivery line & Process Tie-In Fitting. Disconnects delivery line from fitting.	Demob Day
9.	**Owner**	Disconnects the Process Tie-In Fitting from the process & returns it to *ESS*.	Demob Day
Footnote: 1. The ½" nipple (Item #10) on the Process Tie-In Fitting is connected to the process.			

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Attachment IV Field Spiking Data

B. Stack Sampling Start/Stop Times, & Run Durations

TC #/ Run#	Test Date	Sampling Start/Stop Times, & Run Durations			
		Start	End	Run Duration, Min	
TC #1/					
Run #1	3/28/2006	12:10	16:44	274	
Run #2	3/29/2006	11:15	17:00	345	
Run #3a	3/30/2006	11:50	12:39	49	318
Run #3b	3/30/2006	15:30	19:59	269	

Attachment IV Field Spiking Data

C. Field Spiking Log Sheets (Field Data) and Spiking Rate Calculations for:

1. Test Condition #1, Run #1:
   - (a) Mono-Chlorobenzene,
   - (b) Perchloroethylene,
   - (c) Organics Solution, &
   - (d) Metals Solution.

Proje ın: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet) MCB 35

Data ID	Date: 3/28/2006	TC#: 1	Run#: 1	Spiking Material (ID): MCB				Page 1 of 2
Equipment ID:	*Spiking Manager* © #: 2	Pump ID	LMI#	Neptune# 3	Moyno# —	Weigh Scale #: F- 1		MFM# 10-1
Spiking Data File Name: MCB TCIR1			Weather Conditions: warm					

Notes:

0.5833

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Spiking Rate Calculations: Short-Term Average ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	Cum Run Average $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run $Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations (START 12:10 STOP 16:44)
0	07:54	721.85							
1	07:55	721.60							
2	09:05	718.4							
3	09:11	716.15	2.25	6 MIN	0.375				
4	09:21	711.15	5.0	10 MIN	0.50				
5	09:31	705.50	5.65	10 MIN	0.565				
6	09:50	689.35	16.15	22 MIN	0.598				
7	10:20	671.9	17.45	30 MIN	0.5816				
8	10:42	663.8	8.1	14 MIN	0.578				
9	11:02	652.3	11.5	20 MIN	0.575				
10	11:23	640.1	12.2	21 MIN	0.58				
11	11:36	632.6	7.5	13 MIN	0.58				
12	11:52	623.4	9.2	16 MIN	0.58				0.4
13	12:04	616.4	7	12 MIN	0.583				7.5
14	12:13	611.25	5.15	9 MIN	0.57				12.6
15	12:23	605.40	5.85	10 MIN	0.585				18.4
16	12:33	599.7	5.7	10 MIN	0.57	12.69	23 MIN	0.55	24.1
17	12:43	593.9	5.8	10 MIN	0.58	18.49	33 MIN	0.56	29.9
18	12:53	588.2	5.7	10 MIN	0.57	24.19	43 MIN	0.56	35.6
19	13:03	582.4	5.8	10 MIN	0.58	29.99	53 MIN	0.57	41.4
20	13:13	576.6	5.8	10 MIN	0.58	35.79	63 MIN	0.57	47.2
21	13:23	570.9	5.7	10 MIN	0.57	41.49	73 MIN	0.57	52.9
22	13:33	564.5	6.4	10 MIN	0.64	47.89	83 MIN	0.57	59.3
23	13:43	558.7	5.8	10 MIN	0.58	53.69	93 MIN	0.57	65.1
24	13:53	552.8	5.9	10 MIN	0.59	59.59	103 MIN	0.58	71.0
25	14:03	546.9	5.9	10 MIN	0.59	65.49	113 MIN	0.58	76.9
26	14:13	541.1	5.8	10 MIN	0.58	71.29	123 MIN	0.58	82.7
27	14:23	535.4	5.7	10 MIN	0.57	76.99	133 MIN	0.58	88.4
28	14:33	529.5	5.9	10 MIN	0.59	82.88	143 MIN	0.58	94.3
29	14:43	523.5	5.9	10 MIN	0.59	88.68	153 MIN	0.58	100.2

ESS Spiking Technician Signature: [signature] Date: 3/28/2006

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Proje[illegible]n: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID | Date: 3/28/2006 | TC#: 1 | Run#: 1 | Spiking Material (ID): MCB

Page 2 of 2

START 12:10
STOP 16:44

Spiking Rate Data:		Spiking Rate Calculations:						
		Short-Term Average			Cum Run Average			
Time (T) 00:00	Mass (M), Lb	ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	$\Sigma\Delta M$	$\Sigma\Delta T_i$	$Run\ Ave_i = \Sigma_i\Delta M_i/\Sigma_i\Delta T_i$	Comments/Observations
14:53	522.7	5.8	10 MIN	0.58	88.60	153 MIN	0.58	105.8
14:53	517.9	5.0	10 MIN	0.58	94.48	163 MIN	0.58	111.8
15:03	512.0	5.9	10 MIN	0.59	100.38	173 MIN	0.58	117.7
15:03	506.3	5.7	10 MIN	0.57	106.08	183 MIN	0.58	123.4
15:13	500.5	5.8	10 MIN	0.58	111.88	193	0.58	129.2
15:13	494.75				117.68	203	0.58	134.9
15:23	489.45	5.3		0.58	123.48	213	0.58	140.7
15:23	483.10	5.15	10 MIN	0.585	129.33	223	0.58	146.5
16:03	477.3	5.8	10 MIN	0.58	135.13	233	0.58	152.3
16:03	471.6	5.7	10 MIN	0.57	140.83	243	0.58	158.0
16:13	465.8	5.8	10 MIN	0.58	146.63	253	0.579	163.8
16:13	460.1	5.7	10 MIN	0.57	152.33	263	0.579	169.5
16:23	454.4	5.8	10 MIN	0.58	158.13	273	0.578	175.2
16:23	448.5	5.9	10 MIN	0.59				181.0

ESS Spiking Technician Signature: [signature]

Date: 3/28/2006

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Project ID: Westlake PDT TC# 1 Run# 1 Date 3/28/2006
Material: MCB Based on Sample F-1 Rates

∴Start Time 12:10
∴End Time 16:44
Δ Time = 16:44 - 12:10 = 274 Min

∴Start Mass 611.25 + (3 min @ 0.515 lb/min = 1.545 lb) = 612.795

∴End Mass 454.4 - (1 min @ 0.59 lb/min = 0.59 lb) = 453.81

∴Start Mass

∴End Mass

Δ Mass = 612.795 - 453.81 = 158.985 Lb

Spiking Rate = 158.985 Lb / 274 Min = 0.5802 Lb/Min X 60 = 34.81 Lb/Hr

Target Spiking Rate for Test Condition 35.00

Spiking Rate 34.81 Lb ÷ Target Spiking Rate 35.00 Lb = 99.47 % of Target Rate

---

Project ID: Westlake PDT TC# 1 Run# 1 Date 3/28/2006
Material: Based on MCB 10-1 Rates

∴Start Time 12:10
∴End Time 16:44
Δ Time = 16:44 - 12:10 = 274 Min

∴Start Mass 12.6 (3 min @ 0.51 lb/min = 1.53 lb) = 11.07 lb

∴End Mass 169.5 + (1 min @ 0.57 lb/min = 0.57 lb) = 170.07 lb

∴Start Mass

∴End Mass

Δ Mass = 170.07 - 11.07 = 159.0 Lb

Spiking Rate = 159.0 Lb / 274 Min = 0.5803 Lb/Min X 60 = 34.82 Lb/Hr

Target Spiking Rate for Test Condition 35.0 lb/Hr

Spiking Rate 34.82 Lb ÷ Target Spiking Rate 35.0 Lb = 99.48 % of Target Rate

Proje an: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 3/28/2006	TC#: 1	Run#: 1	Spiking Material (ID): Perc		Page 1 of 2
Equipment ID:	Spiking Manager © #: 2	Pump ID —	LMI#	Neptune#	Moyno# —	Weigh Scale #: F- 3	MFM# 10-2
Spiking Data File Name: Perc TC1R1			Weather Conditions: Warm			

Notes:

35 lb/hr 0.583 lb/min

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Spiking Rate Calculations: Short-Term Average ΔMi	ΔTi	Ratei = ΔMi/ΔTi	Cum Run Average ΣiΔMi	ΣiΔTi	Run Avei = ΣΔMi/ ΣiΔTi	Comments/Observations (Start 12:10, Stop 16:44)
0		659.9							
1	09:03	639.8							
2	09:09	634.8	4	6 MIN	0.667				
3	09:19	629.0	5.8	10 MIN	0.58				
4	09:29	623.2	5.8	10 MIN	0.58				
5	09:56	608.0	15.2	27 MIN	0.56				
6	10:26	591.2	16.8	30 MIN	0.56				
7	10:40	582.9	8.3	14 MIN	0.59				
8	11:00	571.4	11.5	20 MIN	0.575				
9	11:21	559.3	12.1	21 MIN	0.576				
10	11:34	551.7	7.6	13 MIN	0.585				
11	11:50	542.7	9	16 MIN	0.56				10.1
12	12:02	535.7	7	12 MIN	0.58				17.1
13	12:11	530.5	5.2	9 MIN	0.577				22.3
14	12:21	524.9	5.6	10 MIN	0.56				27.9
15	12:31	519.1	5.8	10 MIN	0.58	11.977	21 MIN	0.57	33.6
16	12:41	513.3	5.8	10 MIN	0.58	17.77	31 MIN	0.573	39.4
17	12:51	507.5	5.8	10 MIN	0.58	23.57	41 MIN	0.575	45.2
18	13:01	501.6	5.9	10 MIN	0.59	29.47	51 MIN	0.577	51.0
19	13:11	496.0	5.6	10 MIN	0.56	35.07	61 MIN	0.57	56.6
20	13:21	490.4	5.6	10 MIN	0.56	40.67	71 MIN	0.57	62.5
21	13:31	484.6	5.8	10 MIN	0.58	46.47	81 MIN	0.57	68.1
22	13:41	478.2	6.4	10 MIN	0.64	52.87	91 MIN	0.58	73.9
23	13:51	472.6	5.6	10 MIN	0.56	58.47	101 MIN	0.58	79.3
24	14:01	466.9	5.7	10 MIN	0.57	64.17	111 MIN	0.58	85.9
25	14:11	461.2	5.7	10 MIN	0.57	69.87	121 MIN	0.58	91.6
26	14:21	455.4	5.8	10 MIN	0.58	75.67	131 MIN	0.58	97.3
27	14:31	450.0	5.4	10 MIN	0.54	81.07	141 MIN	0.575	103.1
28									
29									

ESS Spiking Technician Signature: [signature] Date: 3/28/2006

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Proje(ın: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID	Date: 3/28/2006	TC#: 1	Run#: 1	Spiking Material (ID): Perc	Page 2 of 2

START 12:10
STOP 16:44

Spiking Rate Data:		Spiking Rate Calculations:						
		Short-Term Average			Cum Run Average			
Time (T) 00:00	Mass (M), Lb	ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	$\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	$Run\ Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
	450.0				51.07			
14:41	444.4	5.6	10 MIN	0.56	86.67	151 MIN	0.57	104.4
14:51	438.8	5.6	10 MIN	0.56	92.27	161 MIN	0.57	114.0
15:01	433.3	5.5	10 MIN	0.55	97.77	171 MIN	0.57	119.5
15:11	426.4	6.9	10 MIN	0.69	104.67	181 MIN	0.58	126.4
15:21	420.4	6.0	10 MIN	0.60	110.67	191 A	0.58	132.3
15:31	414.8	5.6	10 MIN	0.56	116.27	201	0.58	138.0
15:41	409.2	5.6	10 MIN	0.56		211 MIN		143.5
15:51	403.6	5.6	10 MIN	0.56	127.47	221 MIN	0.57	149.1
16:01	397.6	6.0	10 MIN	0.60	133.47	231 MIN	0.58	154.7
16:11	391.1	6.5	10 MIN	0.65	139.97	241 MIN	0.58	160.7
16:21	384.7	6.4	10 MIN	0.64	146.37	251 MIN	0.583	167.2
16:31	378.2	6.5	10 MIN	0.65	152.87	261	0.585	173.8
16:41	371.8	6.4	10 MIN	0.64	159.27	271	0.5877	180.1
16:46	368.9	2.9	5 MIN	0.58				183.0

ESS Spiking Technician Signature: [signature] Date: 3/28/2006

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Project ID: Westate PDT TC# 1 Run# 1 Date 3/28/2006
Material: Perc Based on Scale F-3 Rates

∴Start Time 12:10
∴End Time 16:44
Δ Time = 16:44 - 12:10 = 274 Min

∴Start Mass 530.5 + (1 min @ 0.56 lb/min = 0.56 lb) = 531.06

∴End Mass 368.9 + (2 min @ 0.58 lb/min = 1.16 lb) = 370.06

∴Start Mass

∴End Mass

Δ Mass = 531.06 - 370.06 = 161.00 Lb

Spiking Rate = 161.00 Lb / 274 Min = 0.5876 Lb / Min X 60 = 35.26 Lb / Hr

Target Spiking Rate for Test Condition 35.00

Spiking Rate 35.26 Lb ÷ Target Spiking Rate 35.00 Lb = 100.7 % of Target Rate

---

Project ID: Westate PDT TC# 1 Run# 1 Date 3/28/2006
Material: Perc Based on MFM 10-2 Rates

∴Start Time 12:10
∴End Time 16:44
Δ Time = 16:44 - 12:10 = 274 Min

∴Start Mass 22.3 - (1 min @ 0.52 lb/min = 0.52 lb) = 21.78

∴End Mass 183.0 - (2 min @ 0.58 lb/min = 1.16 lb) = 181.84

∴Start Mass

∴End Mass

Δ Mass = 181.84 - 21.78 = 160.06 Lb

Spiking Rate = 160.06 Lb / 274 Min = 0.5842 Lb / Min X 60 = 35.05 Lb / Hr

Target Spiking Rate for Test Condition 35.0

Spiking Rate 35.05 Lb ÷ Target Spiking Rate 35.0 Lb = 100.1 % of Target Rate

Proje ıan: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 3/28/2006	TC#: 1	Run#: 1	Spiking Material (ID): Organic Solution			Page 1 of 1
Equipment ID:	*Spiking Manager* © #:	Pump ID	LMI#	Neptune# 4	Moyno#	Weigh Scale #: F- 2	MFM# 10-5
Spiking Data File Name: Organic TCRI			Weather Conditions: Warm				

Notes:

4/16 1/Hr 0.6833

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Spiking Rate Calculations: Short-Term Average ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	Cum Run Average $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run $Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Start 12:50 Stop 16:44 Comments/Observations	
0	09:02	553.0								
1	09:08	549.1	3.9	6MIN	0.65					
2	09:18	542.4		10MIN	0.67					
3	09:28	535.8	6.6	10MIN	0.66					
4	09:55	517.6	18.2	27	0.674					
5	10:25	497.5	20.1	30MIN	0.67					
6	10:39	487.9	9.6	14MIN	0.70					
7	10:59	473.6	14.1	20MIN	0.705					
8	11:20	458.9	14.7	21MIN	0.70					
9	11:33	450.4	8.5	13MIN	0.65					
10	11:49	439.3	11.1	16MIN	0.69					
11	12:08	430.5		12MIN	0.73				12:05	144.7
12	12:10	424.5	6.0	9MIN	0.666				12:15	150.8
13	12:20	418.0	6.5	10MIN	0.65				12:25	157.3
14	12:30	411.1	6.9	10MIN	0.69	13.4	20MIN	0.67	12:30	160.7
15	12:40	404.4	6.7	10MIN	0.67	20.1	30MIN	0.67	12:40	167.4
16	12:50	397.7	6.7	10MIN	0.67	26.8	40MIN	0.67	12:50	
17	13:00	391.0	6.7	10MIN	0.67	33.5	50MIN	0.67	13:00	180.9
18	13:10	383.8	7.2	10MIN	0.72	40.7	60MIN	0.678	13:10	188.1
19	13:20	377.0	6.8	10MIN	0.68	47.5	70MIN	0.679	13:20	194.9
20	13:30	370.2	6.8	10MIN	0.68	54.3	80MIN	0.68	13:30	201.7
21	13:40	363.1	7.1	10MIN	0.71	61.4	90MIN	0.68	13:40	208.3
22	13:50	356.2	6.9	10MIN	0.69	68.3	100MIN	0.683	13:50	215.2
23	14:00	349.3	6.9	10MIN	0.69	75.2	110MIN	0.683	14:00	220.2
24	14:10	342.6	6.7	10MIN	0.67	81.9	120MIN	0.683	14:10	229.0
25	14:20	335.8	6.8	10MIN	0.68	88.7	130MIN	0.683	14:20	235.9
26	14:30	329.0	6.8	10MIN	0.68	95.5	140MIN	0.682	14:30	242.7
27										
28										
29										

ESS Spiking Technician Signature: Scott Neal — Date: 3/28/2006

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Proje an: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID	Date: 3/28/2006	TC#: 1	Run#: 1	Spiking Material (ID): Organic Solution	Page 2 of 2

START 12:10
STOP 16:44

Spiking Rate Data: Time (T) 00:00	Mass (M), Lb	Spiking Rate Calculations: Short-Term Average ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	Cum Run Average $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run $Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations	
14:30	329.0				95.5	140	0.682	14:30	242.7
14:40	322.1	6.9	10 MIN	0.69	102.4	150	0.682	14:40	249.2
14:50	315.4	6.7	10 MIN	0.67	109.1	160	0.68	14:50	256.5
15:00	308.5	6.9	10 MIN	0.69	116.0	170	0.68	15:00	263.3
15:10	301.6	6.9	10 MIN	0.69	122.9	180	0.68	15:10	270.1
15:20	294.8	6.8	10 MIN	0.68	129.7	190	0.68	15:20	276.9
15:30	288.1	6.7	10 MIN	0.67	136.4	200	0.68	15:30	283.8
15:40	281.1	7.0	10 MIN	0.70	143.4	210	0.68	15:40	290.5
15:50	274.3	6.8	10 MIN	0.68	150.2	220	0.68	15:50	297.4
16:00	267.5	6.8	10 MIN	0.68	157	230	0.68	16:00	304.2
16:10	260.7	6.8	10 MIN	0.68	163.8	240	0.6825	16:10	311.1
16:20	253.9	6.8	10 MIN	0.68	170.6	250	0.6825	16:20	317.9
16:30	247.1	6.8	10 MIN	0.68	177.4	260	0.6823	16:30	324.7
16:40	240.2	6.9	10 MIN	0.69	184.3	270	0.6825	16:40	331.6
16:50	233.3	6.9	10 MIN	0.69	191.2	280	0.6829	16:45	335.8

ESS Spiking Technician Signature: [signature] Date: 3/28/2006

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Project ID: WESTATES PDT TC# 1 Run# 1 Date 3/28/2006
Material: Organic Solution Based on Table F-2 Rates

∴Start Time 12:10
∴End Time 16:44
Δ Time = 16:44 - 12:10 = 274 Min

∴Start Mass 424.5

∴End Mass 233.34 (6 min @ 0.69 lb/min = 4.14 lb) = 237.44

∴Start Mass

∴End Mass

Δ Mass = 424.5 - 237.44 = 187.06 Lb

Spiking Rate = 187.06 Lb / 274 Min = 0.6873 Lb / Min X 60 = 40.96 Lb / Hr

Target Spiking Rate for Test Condition 41.00

Spiking Rate 40.96 Lb ÷ Target Spiking Rate 41.00 Lb = 99.90 % of Target Rate

---

Project ID: Westate PDT TC# 1 Run# 1 Date 3/28/2006
Material: Organic Solution Based on Mix 10-5 Rates

∴Start Time 12:10
∴End Time 16:44
Δ Time = 16:44 - 12:10 = 274 Min

∴Start Mass 144.7 + (5 min @ 0.61 lb/min = 3.05 lb) = 147.75

∴End Mass 335.1 - (1 min @ 0.70 lb/min = 0.70 lb) = 334.4

∴Start Mass

∴End Mass

Δ Mass = 334.4 - 147.75 = 186.65 Lb

Spiking Rate = 186.65 Lb / 274 Min = 0.6812 Lb / Min X 60 = 40.87 Lb / Hr

Target Spiking Rate for Test Condition 41.00

Spiking Rate 40.87 Lb ÷ Target Spiking Rate 41.00 Lb = 99.69 % of Target Rate

Projec n: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 3/28/2006	TC#: 1	Run#: 1	Spiking Material (ID): Pb/Cr III Solution				Page 1 of 2
Equipment ID:	Spiking Manager © #: 2	Pump ID	LMI#	Neptune#	Moyno# —	Weigh Scale #: F- 5		MFM# 10-4
Spiking Data File Name: Pb/Cr III TC1 R1			Weather Conditions: warm					

Notes:

20 lb/gal 0.3333 lb/min

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Spiking Rate Calculations: Short-Term Average ΔMi	ΔTi	Rate$_i$ = ΔMi/ΔTi	Cum Run Average ΣiΔMi	ΣiΔTi	Run Ave$_i$ = ΣiΔMi/ ΣiΔTi	Comments/Observations
									START 12:10 STOP 16:44
0	07:56	544.2							
1	09:04	542.6							
2	09:10	541.0	1.6	6 min	0.2667				
3	09:20	537.5	3.5	10 min	0.35				
4	09:30	533.6	3.9	10 min	0.39				
5	09:57	524.2	9.4	27 min	0.348				
6	10:27	514.1	10.1	30 min	0.336				
7	10:41	508.9	5.2	14 min	0.37				
8	11:01	501.9	7.0	20 min	0.35				
9	11:22	493.2	8.7	21 min	0.41				
10	11:35	490.1	3.1	13 min	0.23				
11	11:51	483.8	6.3	16 min	0.39				
12	12:03	478.9	4.9	12 min	0.40				12:06 93.6
13	12:12	475.4	3.5	9 min	0.38				12:16 97.7
14	12:22	472.2	3.2	10 min	0.32				12:26 100.4
15	12:32	468.7	3.5	10 min	0.35	7.6	22 min	0.336	12:32 102.4
16	12:42	464.9	3.8	10 min	0.38	11.2	32 min	0.35	12:42 106.0
17	12:52	460.4	4.5	10 min	0.45	15.7	42 min	0.374	12:52 110.4
18	13:02	457.0	3.4	10 min	0.34	19.1	52 min	0.36	13:02 113.9
19	13:12	454.0	3.0	10 min	0.30	22.1	62 min	0.35	13:12 116.8
20	13:22	451.1	2.9	10 min	0.29	25.0	72 min	0.347	13:22 119.7
21	13:32	448.2	2.9	10 min	0.29	27.9	82 min	0.34	13:32 122.6
22	13:42	445.1	3.1	10 min	0.31	31.0	92 min	0.337	13:42 125.3
23	13:52	442.1	3.0	10 min	0.30	34.0	102 min	0.333	13:52 128.2
24	14:02	439.0	3.1	10 min	0.31	37.1	112 min	0.33	14:02 131.2
25	14:12	436.0	3.0	10 min	0.30	40.1	122 min	0.328	14:12 134.1
26	14:22	432.5	3.5	10 min	0.35	43.6	132 min	0.33	14:22 137.7
27	14:32	428.7	3.8	10 min	0.38	47.4	142 min	0.333	14:22 141.4
28									
29									

ESS Spiking Technician Signature: [signature] Date: 3/28/2006

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IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID	Date: 3/28/2006	TC#: 1	Run#: 6	Spiking Material (ID): Pb/Cr Solution	Page 2 of 2

Spiking Rate Data:		Spiking Rate Calculations:						
		Short-Term Average			Cum Run Average			STOP 16:44
Time (T) 00:00	Mass (M), Lb	ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	$\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	$Run\ Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
	428.9				47.4			
14:42	425.3	3.4	10 MIN	0.34	50.8	152 MIN	0.334	14:42 144.9
14:52	422.1	3.2	10 MIN	0.32	54	162	0.333	14:52 148.2
15:02	418.7	3.4	10 MIN	0.34	57.4	172	0.333	15:02 151.5
15:12	415.1	3.6	10 MIN	0.34	61.0	182	0.335	15:12 155.2
15:22	411.9	3.2	10 MIN	0.32	64.2	192	0.333	15:22 158.5
15:32	408.5	3.4	10 MIN	0.34	67.6	202	0.333	15:32 161.8
15:42	405.3	3.2	10 MIN	0.32	70.9	212	0.334	15:42 165.0
15:52	402.0	3.3	10 MIN	0.33	74.2	222	0.334	15:52 168.3
16:02	398.5	3.5	10 MIN	0.35	77.7	232	0.335	16:02 171.8
16:12	395.0	3.5	10 MIN	0.35	81.2	242	0.335	16:12 175.3
16:22	391.2	3.8	10 MIN	0.38	85.0	252	0.337	16:22 179.0
16:32	387.9	3.3	10 MIN	0.33	88.3	262	0.337	16:32 182.3
16:42	384.9	3.0	10 MIN	0.30	91.3	272	0.335	16:42 185.0
16:47	383.4	1.5	5 MIN	0.30				16:47 186.5

ESS Spiking Technician Signature: [signature] Date: 3/28/2006

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Project ID: Westate PDT
Material: Pb/Cr III Solution

TC# 1 Run# 1 Date 3/28/2006
Based on Scale F-5 Rates

∴Start Time 12:10
∴End Time 16:44
Δ Time = 16:44 - 12:10 = 274 Min

∴Start Mass 475.4 + (2 min @ 0.35 lb/min = 0.70 lb) = 476.1

∴End Mass 386.9 - (2 min @ 0.30 lb/min = 0.60 lb) = 384.3

∴Start Mass

∴End Mass

Δ Mass = 476.1 - 384.3 = 91.8 Lb

Spiking Rate = 91.8 Lb / 274 Min = 0.3350 Lb/Min X 60 = 20.10 Lb/Hr

Target Spiking Rate for Test Condition 20.00

Spiking Rate 20.10 Lb ÷ Target Spiking Rate 20.00 Lb = 100.5 % of Target Rate

---

Project ID: Westate PDT
Material: Pb/Cr III Solution

TC# 1 Run# 1 Date 3/28/2006
Based on LCF-4/10-4 Rates

∴Start Time 12:10
∴End Time 16:44
Δ Time = 16:44 - 12:10 = 274 Min

∴Start Mass 93.6 + (4 min @ 0.36 lb/min = 1.44 lb) = 95.04

∴End Mass 185.0 + (2 min @ 0.30 lb/min = 0.60 lb) = 185.6

∴Start Mass

∴End Mass

Δ Mass = 185.6 - 95.04 = 90.56 Lb

Spiking Rate = 90.56 Lb / 274 Min = 0.3305 Lb/Min X 60 = 19.83 Lb/Hr

Target Spiking Rate for Test Condition 20.00

Spiking Rate 19.83 Lb ÷ Target Spiking Rate 20.00 Lb = 99.15 % of Target Rate

Attachment IV Field Spiking Data

C. Field Spiking Log Sheets (Field Data) and Spiking Rate Calculations for:

2. Test Condition #1 Run #2:
   - (a) Mono-Chlorobenzene,
   - (b) Perchloroethylene,
   - (c) Organics Solution, &
   - (d) Metals Solution.

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 3/29/2006	TC#: 1	Run#: 2	Spiking Material (ID): MCB			Page 1 of 2
Equipment ID:	*Spiking Manager* © #: 2	Pump ID	LMI# —	Neptune# 3	Moyno# —	Weigh Scale #: F- 1	MFM# 0-1
Spiking Data File Name: MCB TC1R2			Weather Conditions:				

Notes:

3516/hr 0.5853

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Spiking Rate Calculations: Short-Term Average ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	Cum Run Average $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run $Ave_i = \Sigma \Delta M_i / \Sigma_i \Delta T_i$	Start 11:15 STOP 17:00 Comments/Observations
0	09:24	719.5							
1	09:35	713.2	6.3	19 MIN	0.33				
2	10:05	707.1	6.1	20 MIN	0.33				
3	10:27	692.7	14.4	22 MIN	0.65				
4	10:37	687.2	5.2						9.2
5	10:47	681.0	6.2	10 MIN	0.62				15.4
6	10:57	675.8	5.2	10 MIN	0.52				20.6
7	11:07	669.5	6.3	10 MIN	0.63				26.8
8	11:17	663.4	6.1	10 MIN	0.61		2 MIN		33.0
9	11:29	655.2	8.2	12 MIN	0.68	9.42	14 MIN	0.67	41.2
10	11:37	651.5	3.7	8 MIN	0.46	13.12	22 MIN	0.596	44.9
11	11:47	646.9	4.6	10 MIN	0.46	17.72	32 MIN	0.554	49.5
12	11:57	641.6	5.3	10 MIN	0.53	23.02	42 MIN	0.55	54.8
13	12:07	635.4	6.2	10 MIN	0.62	29.22	52 MIN	0.56	61.0
14	12:17	629.2	6.2	10 MIN	0.62	35.42	62 MIN	0.57	67.2
15	12:27	622.8	6.4	10 MIN	0.64	41.82	72 MIN	0.58	73.4
16	12:37	616.9	5.9	10 MIN	0.59	47.72	82 MIN	0.58	79.3
17	12:47	610.6	6.3	10 MIN	0.63	54.02	92	0.58	85.7
18	12:57	605.1	5.5	10 MIN	0.55	59.52	102	0.583	91.0
19	13:07	599.6	5.5	10 M	0.55	65.02	112	0.58	97.5
20	13:17	594.0	5.6	10 M	0.56	70.62	122	0.58	103.0
21	13:27	587.5	6.5	10 M	0.65	77.12	132	0.584	109.6
22	13:42	578.2							
23	13:47	575.1	12.4	10 M	0.62	89.52	152	0.588	121.0
24	13:57	568.8	6.3	10 M	0.63	95.82	162	0.59	127.4
25	14:07	562.7	6.1	10 M	0.61	101.92	172	0.59	133.5
26	14:17	556.4	6.3	10 M	0.63	108.22	182	0.59	139.8
27	14:27	551.1	5.3	10 M	0.53	113.52	192	0.59	145.1
28									
29									

ESS Spiking Technician Signature: [signature] Date: 3/29/2006

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Proje... lan: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID	Date: 3/29/2006	TC#: 1	Run#: 2	Spiking Material (ID): MCB	Page 2 of 2

Spiking Rate Data:		Spiking Rate Calculations:						
		Short-Term Average			Cum Run Average			
Time (T) 00:00	Mass (M), Lb	ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	$\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	$Run\ Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
14:27	551.1							STOP 17:00
14:37	545.8	5.3	10 MIN	0.53	113.52	192		
14:47	540.2	5.6	10 MIN	0.56	118.82	202	0.588	150.4
14:57	534.5	5.7	10 MIN	0.57	124.42	212	0.587	156.0
15:07	529.1	5.4	10 MIN	0.54	130.12	222	0.586	161.7
15:17	523.9	5.2	10 MIN	0.52	135.52	232	0.584	167.1
15:27	518.0	5.9	10 MIN	0.59	140.72	242	0.581	172.3
15:37	512.2	5.8	10 MIN	0.58	146.62	252	0.58	178.2
15:47	506.4	5.8	10 MIN	0.58		271		184.0
15:57	500.5	5.9	10 MIN	0.58	158.22	272	0.581	189.9
16:07	494.3	6.2	10 MIN	0.62	164.12	282	0.581	195.7
16:17	488.5	5.8	10 MIN	0.58	170.32	292	0.583	201.9
16:27	482.4	6.1	10 MIN	0.61	176.12	302	0.583	207.7
16:37	476.4	6.0	10 MIN	0.60	182.22	312	0.584	213.8
16:47	470.7	5.7	10 MIN	0.57	188.22	322	0.584	219.8
16:57	464.8	5.9	10 MIN	0.59	193.92	332	0.584	225.8
17:07	459.0	5.8	10 MIN	0.58		342		231.5
						352		237.5

ESS Spiking Technician Signature: [signature]

Date: 3/29/2006

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Project ID: Westate PDT TC# 1 Run# 2 Date 3/29/2006
Material: MCB Based on Scale F-1 Rates

∴Start Time 11:15
∴End Time 17:00
Δ Time = 17:00 - 11:15 = 345 Min

∴Start Mass 663.4 + (2 min @ 0.61 lb/min = 1.22 lb) = 664.62

∴End Mass 464.8 - (3 min @ 0.58 lb/min = 1.74 lb) = 463.06

∴Start Mass

∴End Mass

Δ Mass = 664.62 - 463.06 = 201.56 Lb

Spiking Rate = 201.56 Lb / 345 Min = 0.5842 Lb/Min X 60 = 35.05 Lb/Hr

Target Spiking Rate for Test Condition 35.00

Spiking Rate 35.05 Lb ÷ Target Spiking Rate 35.00 Lb = 100.1 % of Target Rate

---

Project ID: Westate PDT TC# 1 Run# 2 Date 3/29/2006
Material: MCB Based on MFW Rates

∴Start Time 11:15
∴End Time 17:00
Δ Time = 17:00 - 11:15 = 345 Min

∴Start Mass 33.0 - (2 min @ 0.62 lb/min = 1.24 lb) = 31.76 lb

∴End Mass 464.8 + (3 min @ 0.58 lb/min = 1.74 lb) = 233.3 lb

∴Start Mass

∴End Mass

Δ Mass = 233.3 - 31.76 = 201.54 Lb

Spiking Rate = 201.54 Lb / 345 Min = 0.5842 Lb/Min X 60 = 35.05 Lb/Hr

Target Spiking Rate for Test Condition 35.00

Spiking Rate 35.05 Lb ÷ Target Spiking Rate 35.00 Lb = 100.1 % of Target Rate

Proje lan: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 3/29/2006	TC#: 1	Run#: 2	Spiking Material (ID): Pure				Page 1 of 2
Equipment ID:	Spiking Manager © #: 2	Pump ID –	LMI#	Neptune#	Moyno# –		Weigh Scale #: F- 3	MFM# 10-2
Spiking Data File Name: Pure TC1 R2			Weather Conditions:					

Notes:

35 lb/hr 0.583

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Short-Term Average: ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	Cum Run Average: $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	$Run\ Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
									Start 11:15 Stop 17:00
0	09:23	772.0							
1	09:43	758.2	13.8	20min	0.69				
2	10:03	746.7	11.5	20min	0.575				
3	10:24	733.7	13	22min	0.59				
4	10:35	727.7	6	10m	0.60				
5	10:45	721.8	5.9	10min	0.59				
6	10:55	716.1	5.7	10min	0.57				8.1
7	11:05	710.2	5.9	10min	0.59				14.0
8	11:15	704.6	5.6	10min	0.56				19.6
9	11:25	699.0	5.6	10min	0.56	5.6	10min	0.56	25.2
10	11:35	692.8	6.2	10min	0.62	11.8	20min	0.59	31.4
11	11:45	687.0	5.8	10min	0.58	17.6	30min	0.58	37.2
12	11:55	681.1	5.9	10min	0.59	23.5	40	0.587	43.1
13	12:05	674.8	6.3	10min	0.63	29.8	50min	0.59	49.4
14	12:15	669.2	5.6	10min	0.56	35.4	60min	0.59	55.0
15	12:25	663.5	5.7	10min	0.57	41.1	70min	0.587	60.8
16	12:35	657.5	6.0	10min	0.60	47.1	80min	0.583	66.8
17	12:45	651.3	6.2	10min	0.62	53.3	90	0.59	73.0
18	12:55	645.6	5.7	10min	0.57	59.4	100	0.59	78.0
19	13:05	639.9	5.7	10m	0.57	65.1	110	0.59	83.7
20	13:15	634.4	5.5	10m	0.55	70.6	120	0.588	89.2
21	13:25	628.8	5.6	10m	0.56	76.2	130	0.586	94.8
22	13:40	620.3							
23	13:45	617.8	11.4	20min	0.57	87.6	150	0.584	106.2
24	13:55	611.8	5.6	10m	0.56	93.2	160	0.582	111.8
25	14:05	606.0	5.8	10m	0.58	99	170	0.582	117.6
26	14:15	600.3	5.7	10m	0.57	104.7	180	0.58	123.3
27	14:25	594.2	6.1	10m	0.1	110.8	190	0.583	129.4
28									
29									

ESS Spiking Technician Signature: [signature] Date: 3/29/2006

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Project: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID | Date 3/29/2006 | TC#: 1 | Run#: 2 | Spiking Material (ID): Pere | Page 2 of 2

START 11:15
STOP 17:00

Spiking Rate Data:		Spiking Rate Calculations:						
		Short-Term Average			Cum Run Average			
Time (T) 00:00	Mass (M), Lb	ΔM_i	ΔT_i	Rate$_i$ = $\Delta M_i/\Delta T_i$	$\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run Ave$_i$ = $\Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
14:25	594.2				110.8	190		129.4
14:35	587.7	6.5	10 min	0.65	117.3	200	0.586	135.9
14:45	581.9	5.8	10 m	0.58	123.1	210	0.586	141.7
14:55	576.3	5.6	10 m	0.56	128.7	220	0.585	147.3
15:05	570.2	6.1	10 m	0.61	134.8	230	0.586	153.4
15:15	564.4	5.8	10 m	0.58	140.6	240	0.586	159.2
15:25	558.4	6.0	10 m	0.60	146.6	250	0.586	165.3
15:35	552.5	5.9	10 m			260		171.1
15:45	546.5	6.0	10 m	0.60	152.5	270	0.587	177.1
15:55	540.7	5.8	10 m	0.58	158.3	280	0.586	182.9
16:05	534.6	6.1	10 MIN	0.61	170.4	290	0.587	189.0
16:15			10 MIN			300		
16:25	522.8	11.8	10 MIN	0.59	182.2	310	0.587	200.9
16:35	516.4	6.4	10 MIN	0.64	188.6	320	0.589	207.0
16:45	510.8	5.6	10 MIN	0.56	194.2	330	0.588	212.6
16:55	505.2	5.6	10 MIN	0.56	199.8	340	0.587	218.2
17:05	499.6	5.6	10 MIN	0.56		350		223.8

ESS Spiking Technician Signature: [signature]

Date: 3/29/2006

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Project ID: Westate PDT
Material: Pere

TC# 1 Run# 2 Date 3/29/2006
Based on Scale F-3 Rates

∴Start Time 11:15
∴End Time 17:00
Δ Time = 17:00 - 11:15 = 345 Min

∴Start Mass 704.6

∴End Mass 505.2 - (5 min @ 0.56 lb/min = 2.8 lb) = 502.4

∴Start Mass

∴End Mass

Δ Mass = 704.6 - 502.4 = 202.2 Lb

Spiking Rate = 202.2 Lb / 345 Min = 0.5861 Lb / Min X 60 = 35.17 Lb / Hr

Target Spiking Rate for Test Condition 35.00

Spiking Rate 35.17 Lb ÷ Target Spiking Rate 35.00 Lb = 100.4 % of Target Rate

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Project ID: Westate PDT
Material: Pere

TC# 1 Run# 2 Date 3/29/2006
Based on Flow 10-2 Rates

∴Start Time 11:15
∴End Time 17:00
Δ Time = 17:00 - 11:15 = 345 Min

∴Start Mass 19.6 lb

∴End Mass 223.2 - (5 min @ 0.56 lb/min = 2.8 lb) = 221.0

∴Start Mass

∴End Mass

Δ Mass = 221.0 - 19.6 = 201.4 Lb

Spiking Rate = 201.4 Lb / 345 Min = 0.5838 Lb / Min X 60 = 35.03 Lb / Hr

Target Spiking Rate for Test Condition 35.00

Spiking Rate 35.03 Lb ÷ Target Spiking Rate 35.00 Lb = 100.1 % of Target Rate

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 3/29/2006	TC#: 1	Run#: 2	Spiking Material (ID): Organic Solution	Page 1 of 2
Equipment ID:	Spiking Manager © #:	Pump ID –	LMI# —	Neptune# 4 / Moyno# — / Weigh Scale #: F- 2	MFM# 0.5
Spiking Data File Name: Organic FG1R2			Weather Conditions: Clear		

Notes:

41 lb/hr 0.683

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Spiking Rate Calculations: Short-Term Average ΔMi	ΔTi	Rate$_i$ = ΔMi/ΔTi	Cum Run Average ΣiΔMi	ΣiΔTi	Run Ave$_i$ = ΣiΔMi/ ΣiΔTi	Comments/Observations (START 11:15 STOP 17:00)
0	09:20	509.9							
1	09:42	494.0	15.9	22 MIN	0.72				14.0
2	10:02	480.2	13.8	20 MIN	0.69				28.0
3	10:24	464.8	15.4	22 min	0.70				42.9
4	10:34	458.9	5.9	10 min	0.59	12.55			48.9
5	10:44	452.0	6.9	10 MIN	0.69				55.8
6	10:54	444.9	7.1	10 MIN	0.71				63.3
7	11:04	437.3	7.6	10 min	0.76				70.6
8	11:14	430.4	6.9	10 min	0.69				77.6
9	11:24	423.9	6.5	10 min	0.65	5.85	9 MIN	0.65	84.1
10	11:34	417.2	6.7	10 MIN	0.67	12.55	19 MIN	0.66	90.9
11	11:44	409.8	7.4	10 MIN	0.74	19.95	29 MIN	0.687	98.2
12	11:54	403.2	6.6	10 MIN			39 MIN		104.9
13	12:04	397.2	6.0	10 MIN	0.60	32.55	49 MIN	0.664	111.1
14	12:14	391.0	6.2	10 MIN	0.62	38.75	59 MIN	0.66	117.2
15	12:24	384.4	6.6	10 MIN	0.66	45.35	69 min	0.66	123.7
16	12:34	376.6	7.8	10 MIN	0.78	53.15	79 MIN	0.672	131.5
17	12:44	367.9	8.7	10 MIN	0.87	61.85	89	0.69	140.0
18	12:54	361.0	6.9	10 MIN	0.69	68.75	99	0.69	147.1
19	13:04	354.1	6.9	10 m	0.69	75.65	109	0.69	153.9
20	13:14	347.4	6.7	10 m	0.67	82.35	119	0.69	160.7
21	13:24	340.5	6.9	10 m	0.69	89.25	129	0.69	167.6
22	13:34	330.4							177.8
23	13:44	327.0	13.5	20 m	0.675	102.75	149	0.68	181.2
24	13:54	320.2	6.8	10 m	0.68	109.55	159	0.68	187.9
25	14:04	313.2	7.0	10 m	0.70	116.55	169	0.689	194.7
26	14:14	306.9	6.3	10 m	0.63	122.85	179	0.686	201.1
27	14:24	300.7	6.2	10 m	0.62	129.05	189	0.683	207.2
28									
29									

ESS Spiking Technician Signature: Scott [illegible] Date: 3/29/2006

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Project Plan: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID | Date: 3/29/2006 | TC#: 1 | Run#: 2 | Spiking Material (ID): Organic Solution | Page 2 of 2

START 11:15
STOP 17:00

Spiking Rate Data:		Spiking Rate Calculations:						
		Short-Term Average			Cum Run Average			
Time (T) 00:00	Mass (M), Lb	ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	$\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	$Run\ Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
14:24	300.7				129.05			
14:34	293.8	6.9	10min	0.69	135.95	199	0.683	214.1
14:44	286.9	6.9	10m	0.69	142.85	209	0.683	221.1
14:54	280.0	6.9	10m	0.69	149.75	219	0.683	227.9
15:04	272.9	7.1	10m	0.71	156.85	229	0.684	235.0
15:14	266.0	6.9	10m	0.69	163.75	239	0.685	241.8
15:24	259.1	6.9	10m	0.69	170.65	249	0.685	248.7
15:34	251.7	7.4	10m	0.74		259		255.8
15:44	245.2	6.5	10m	0.65	184.25	269	0.684	262.6
15:54	238.0	7.2	10m	0.72	191.45	279	0.686	270.0
16:04	231.3	6.7	10m	0.67	198.15	289	0.685	276.6
16:14	217.		10MIN			299		290.4
16:24	217.3	14	20MIN	0.7	212.15	309	0.686	297.4
16:34	210.4	6.9	10MIN	0.69	219.05	319	0.686	297.4
16:44	204.0	6.4	10MIN	0.64	225.45	329	0.685	303.6
16:54	198.4	5.6	10MIN	0.56	231.05	339	0.6815	309.3
17:04	191.5	6.9	10MIN	0.69	237.95	349	0.6818	316.1

ESS Spiking Technician Signature: Scott Neal

Date: 3/29/2006

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Project ID: Westate PDT TC# 1 Run# 2 Date 3/29/2006
Material: Organic Solution Based on Scale F-2 Rates

∴Start Time 11:15
∴End Time 17:00
Δ Time = 17:00 - 11:15 = 345 Min

∴Start Mass 430.4 - (1 min @ 0.69 lb/min = 0.69 lb) = 429.71

∴End Mass 198.4 - (1 min @ 0.69 lb/min = 4.14 lb) = 194.26

∴Start Mass

∴End Mass

Δ Mass = 429.71 - 194.26 = 235.45 Lb

Spiking Rate = 235.45 Lb / 345 Min = 0.6825 Lb/Min X 60 = 40.95 Lb/Hr

Target Spiking Rate for Test Condition 41.0

Spiking Rate 40.95 Lb ÷ Target Spiking Rate 41.0 Lb = 0.997 % of Target Rate

---

Project ID: Westate PDT TC# 1 Run# 2 Date 3/29/2006
Material: Organic Solution Based on MPM 10-9 Rates

∴Start Time 11:15
∴End Time 17:00
Δ Time = 17:00 - 11:15 = 345 Min

∴Start Mass 77.6 + (1 min @ 0.70 lb/min = 0.70 lb) = 78.3

∴End Mass 319.3 + (1 min @ 0.68 lb/min = 4.08 lb) = 313.38

∴Start Mass

∴End Mass

Δ Mass = 313.38 - 78.3 = 235.08 Lb

Spiking Rate = 235.08 Lb / 345 Min = 0.6814 Lb/Min X 60 = 40.88 Lb/Hr

Target Spiking Rate for Test Condition 41.0

Spiking Rate 40.88 Lb ÷ Target Spiking Rate 41.0 Lb = 0.997 % of Target Rate

Proje an: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 3/29/2006	TC#: 1	Run#: 2	Spiking Material (ID): Pb/Cu Solution				Page 1 of 2
Equipment ID:	Spiking Manager © #: 2	Pump ID	LMI#	Neptune#	Moyno# —	Weigh Scale #: F-5		MFM# 04
Spiking Data File Name: Pb/Cu TC1 R2			Weather Conditions: Clear					

Notes:

20 lb/hr 0.3333 lb/min

START 11:15
END 17:00

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Short-Term Average: ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	Cum Run Average: $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run $Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
0	09:24	325.8							
1	09:44	320.8	5.0	20 min	0.25				5.0
2	10:04	314.5	6.3	20 min	0.315				10.9
3	10:26	308.9	5.6	22 min	0.25				17.4
4	10:36	304.9	4	10 min	0.4				21.3
5	10:46	300.6	4.3	10 min	0.43				25.4
6	10:56	297.0	3.6	10 min	0.36				29.1
7	11:06	293.5	3.5	10 min	0.35				32.7
8	11:16	289.8	3.7	10 min	0.37		1 min		36.3
9	11:26	285.5	4.3	12 min	0.35	4.67	13 min	0.35	40.6
10	11:36	283.2	2.3	8 min	0.29	6.97	21 min	0.331	43.1
11	11:46	280.2	3.0	10 min	0.30	9.97	31 min	0.32	46.0
12	11:56	276.8	3.4	10 min	0.34	13.37	41 min	0.326	49.4
13	12:06	273.3	3.5	10 min	0.35	16.87	51 min	0.33	52.9
14	12:16	269.5	3.8	10 min	0.38	20.67	61 min	0.338	56.6
15	12:26	265.6	3.9	10 min	0.39	24.57	71 min	0.34	60.4
16	12:36	261.6	4.0	10 min	0.40	28.57	81 min	0.35	64.4
17	12:46	257.8	3.8	10 min	0.38	32.37	91	0.35	68.4
18	12:56	255.2	2.6	10 min	0.26	34.97	101	0.34	71.1
19	13:06	252.5	2.7	10 min	0.27	37.67	111	0.339	73.8
20	13:16	249.7	2.8	10 min	0.28	40.47	121	0.334	75.5
21	13:26	246.7	3.0	10 min	0.3	43.47	131	0.331	79.5
22	13:41	242.1							84.2
23	13:46	240.5	6.2	20 min	0.31	49.67	151	0.328	85.7
24	13:56	237.3	3.2	10 min	0.32	52.87	161	0.328	90.0
25	14:06	234.1	3.2	10 min	0.32	56.07	171	0.327	92.2
26	14:16	231.0	3.1	10 min	0.31	59.17	181	0.326	95.3
27	14:26	227.6	3.4	10 min	0.34	62.57	191	0.327	98.7
28									
29									

ESS Spiking Technician Signature: [signature] Date: 3/29/2006

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Proje ıan: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID	Date: 3/29/2006	TC#: 1	Run#: 2	Spiking Material (ID): Pb/Cl Solution	Page 2 of 2

Spiking Rate Data:		Spiking Rate Calculations:						
		Short-Term Average			Cum Run Average			
Time (T) 00:00	Mass (M), Lb	ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	$\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	$Run\ Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
14:26	277.6				62.57	191		
14:36	274.1	3.5	10min	0.35	66.07	201	0.323	102.3
14:46	270.7	3.4	10m	0.34	69.07	211	0.324	105.7
14:56	266.9	3.8	10m	0.38	71.87	221	0.325	109.3
15:06	263.5	3.4	10m	0.34	75.27	231	0.325	113.0
15:16	259.8	3.7	10m	0.37	78.97	241	0.327	116.7
15:26	255.0	3.8	10m	0.38	82.77	251	0.329	120.4
15:36	252.0	4.0	10m			261		124.5
15:46	248.2	3.8	10m	0.38	90.57	271	0.334	128.2
15:56	244.1	4.1	10m	0.41	94.67	281	0.336	132.1
16:06	240.0	4.1	10m	0.41	98.77	291	0.339	135.9
16:16	23					301		143.2
16:26	233.1	6.9	10m	0.69	105.67	311	0.339	143.2
16:36	230.0	3.1	10m	0.31	108.77	321	0.338	146.1
16:46	228.2	1.8	10m	0.18	110.57	331	0.334	148.0
16:56	226.0	2.2	10m	0.22	112.77	341	0.3307	150.0
17:06	221.6	4.4	10m	0.44	117.17	351	0.3338	154.5

ESS Spiking Technician Signature: Scott Wise

Date: 3/29/2006

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Project ID: Westate PDT TC# 1 Run# 2 Date 3/29/2006
Material: Pb/Cr III Solution Based on Scale 8-5 Rates

∴ Start Time 11:15
∴ End Time 17:00
Δ Time = 17:00 - 11:15 = 345 Min

∴ Start Mass 339.8 + (1 min @ 0.37 lb/min = 0.37 lb) = 340.17

∴ End Mass 226.0 + (4 min @ 0.44 lb/min = 1.76 lb) = 224.24

∴ Start Mass

∴ End Mass

Δ Mass = 340.17 - 224.24 = 115.93 Lb

Spiking Rate = 115.93 Lb / 345 Min = 0.3360 Lb/Min X 60 = 20.16 Lb/Hr

Target Spiking Rate for Test Condition 20.00

Spiking Rate 20.16 Lb ÷ Target Spiking Rate 20.00 Lb = 100.8 % of Target Rate

---

Project ID: Westate PDT TC# 1 Run# 2 Date 3/29/2006
Material: Pb/Cr III Solution Based on MEM 10-4 Rates

∴ Start Time 11:15
∴ End Time 17:00
Δ Time = 17:00 - 11:15 = 345 Min

∴ Start Mass 36.3 - (1 min @ 0.36 lb/min = 0.36 lb) = 35.94

∴ End Mass 150.0 + (4 min @ 0.45 lb/min = 1.8 lb) = 151.8

∴ Start Mass

∴ End Mass

Δ Mass = 151.8 - 35.94 = 115.86 Lb

Spiking Rate = 115.86 Lb / 345 Min = 0.3358 Lb/Min X 60 = 20.15 Lb/Hr

Target Spiking Rate for Test Condition 20.00

Spiking Rate 20.15 Lb ÷ Target Spiking Rate 20.00 Lb = 100.7 % of Target Rate

Attachment IV Field Spiking Data

C. Field Spiking Log Sheets (Field Data) and Spiking Rate Calculations for:

3. Test Condition #1, Run #3:
   - (a) Mono-Chlorobenzene,
   - (b) Perchloroethylene,
   - (c) Organics Solution, &
   - (d) Metals Solution.

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 2/30/2006	TC#: 1	Run#: 3	Spiking Material (ID): MCB				Page 1 of 2
Equipment ID:	*Spiking Manager* © #: 1		Pump ID	LMI# –	Neptune# 3	Moyno# –	Weigh Scale #: F- 1	MFM# ND-1
Spiking Data File Name: MCB TC1R3				Weather Conditions:				

Notes:

35 lb/ha 0.5833 lb/min

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Spiking Rate Calculations: Short-Term Average ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	Cum Run Average $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run $Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
									START 11:50 STOP 12:39 START 13:30 END 14:59
0	10:20	608.6							
1	10:45	593.7	14.9	25 MIN	0.596				
2	11:06	581.3	12.4	21 MIN	0.59				
3	11:11	575.9	5.4	9 MIN	0.60				11.3
4	11:36	564.1	11.8	20 MIN	0.59				23.1
5	11:45	558.2	5.9	10 MIN	0.59				29.0
6	11:55	552.3	5.9	10 MIN	0.59	2.55	5 MIN	0.59	34.9
7	12:09	544.0					19 MIN		
8	12:15	540.4	11.9	20 MIN	0.595	14.25	25 MIN	0.594	46.8
9	12:35	528.8		20 MIN					
10	12:39	523.3	17.1	34 MIN	0.50	31.95	49 MIN	0.65	63.9
11	15:07	434.6	7.6						
12	15:20	437.0	8.6	13 MIN	0.58				155.2
13	15:29	426.6	5.4	9 MIN	0.59				160.6
14	15:39	420.8	5.8	10 MIN	0.58	37.17	58 MIN	0.64	166.4
15	15:49	416.9	3.9	10 MIN	0.59	43.07	68 MIN	0.63	172.3
16	15:59	409.2	5.7	10 MIN	0.59	48.77	78 MIN	0.62	178.0
17	16:09	403.6	5.6	10 MIN	0.58	54.37	88 MIN	0.617	183.6
18	16:19	398.0	5.6	10 MIN	0.58	59.97	98 MIN	0.61	189.3
19	16:29	392.2	5.8	10 MIN	0.58	65.77	108 MIN	0.608	195.1
20	16:39	386.5	5.7	10 MIN	0.57	71.47	118 MIN	0.605	200.8
21	16:49	380.8	5.7	10 MIN	0.57	77.17	128 MIN	0.60	206.5
22	16:59	375.0	5.8	10 MIN	0.58	82.97	138 MIN	0.60	212.2
23	17:09	369.2	5.8	10 MIN	0.58	88.77	148 MIN	0.599	218.0
24	17:19	363.4	5.8	10 MIN	0.58	94.57	158 MIN	0.598	223.7
25	17:29	357.5	5.9	10 MIN	0.59	100.47	168 MIN	0.59	229.6
26	17:39	351.9	5.6	10 MIN	0.56	106.07	178 MIN	0.596	235.2
27	17:49	345.7	6.2	10 MIN	0.62	112.27	188 MIN	0.597	241.4
28	17:59	340.3	5.4	10 MIN	0.54	117.67	198 MIN	0.594	246.8
29	18:09	334.9	5.4	10 MIN	0.54	123.07	208 MIN	0.591	252.2

ESS Spiking Technician Signature: Scott Neal

Date: 3/30/2006

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Projec. ..n: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID	Date: 3/30/2006	TC#: 1	Run#: 3	Spiking Material (ID): MCB	Page 2 of 2

Spiking Rate Data:		Spiking Rate Calculations:						
		Short-Term Average			Cum Run Average			STOP 19:59
Time (T) 00:00	Mass (M), Lb	ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	$\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	$Run\ Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
18:09	334.9				123.07	208		252.2
18:19	324.1	5.8	10 min	0.58	128.87	218	0.59	258.0
18:29	328.2	5.9	10 min	0.59	1	228		263.9
18:39	317.4	5.8	10 min	0.58	140.57	238	0.59	269.6
18:49	311.6	5.8	10 min	0.58	146.37	248	0.59	275.4
18:59	305.8	5.8	10 min	0.58	152.17	258	0.589	281.2
19:09	300.0	5.8	10 min	0.58	157.97	268	0.589	287.0
19:19	294.3	5.7	10 min	0.57	163.67	278	0.58	292.8
19:29	288.6	5.7	10 min	0.57	169.37	288	0.588	298.5
19:39	282.8	5.8	10 min	0.58	175.17	298	0.588	304.2
19:49	277.1	5.7	10 min	0.57	180.87	308	0.588	310.0
19:59	271.4	5.7	10 min	0.57	186.57	318	0.588	315.7

ESS Spiking Technician Signature: [signature] Date: 3/30/2006

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Project ID: Westate PDT TC# 1 Run# 3 Date 3/30/2006
Material: MCB Based on SCALE F-1 Rates

∴Start Time 11:50 / 15:30
∴End Time 12:39 / 19:59
Δ Time = (12:39 - 11:50) + (19:59 - 15:30) = 318 Min

∴Start Mass 552.3 + (5 min @ 0.59 lb/min = 2.95 lb) = 555.25

∴End Mass 523.3

∴Start Mass 426.6 - (1 min @ 0.58 lb/min = 0.58 lb) = 426.02

∴End Mass 271.4

Δ Mass = (555.25 - 523.3) + (426.02 - 271.4) = 126.57 Lb
126.57

Spiking Rate = 126.57 Lb / 318 Min = 0.5967 Lb/Min X 60 = 35.20 Lb/Hr

Target Spiking Rate for Test Condition 35.0

Spiking Rate 35.20 Lb ÷ Target Spiking Rate 35.0 Lb = 100.6 % of Target Rate

---

Project ID: Westate PDT TC# 1 Run# 3 Date 3/30/2006
Material: MCB Based on MFM 10-1 Rates

∴Start Time 11:50 / 15:30
∴End Time 12:39 / 19:59
Δ Time = (12:39 - 11:50) + (19:59 - 15:30) = 318 Min

∴Start Mass 29.0 + (5 min @ 0.59 lb/min = 2.95 lb) = 31.95

∴End Mass 63.9

∴Start Mass 160.6 + (1 min @ 0.58 lb/min = 0.58 lb) = 161.8

∴End Mass 315.7

Δ Mass = (315.7 - 161.8) + (63.9 - 31.95) = 185.85 Lb

Spiking Rate = 185.85 Lb / 318 Min = 0.5844 Lb/Min X 60 = 35.07 Lb/Hr

Target Spiking Rate for Test Condition 35.0

Spiking Rate 35.07 Lb ÷ Target Spiking Rate 35.0 Lb = 100.2 % of Target Rate

Project: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 7/30/2006	TC#: 1	Run#: 3	Spiking Material (ID): Perc				Page 1 of 2
Equipment ID:	Spiking Manager © #: 2	Pump ID —	LMI#	Neptune#	Moyno# —		Weigh Scale #: F- 3	MFM# 10.2
Spiking Data File Name: Perc TC1R3			Weather Conditions: Warm					

Notes:

35 lb/hr 0.583 lb/hr-N

START 11:50
STOP 12:39
START 15:30 END 19:59

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Short-Term Average: ΔM_i	ΔT_i	Rate$_i$ = $\Delta M_i/\Delta T_i$	Cum Run Average: $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run Ave$_i$ = $\Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
0	10:18	490.3							
1	10:43	475.2	15.1	25	0.604				
2	11:03	463.3	11.9	20	0.595				
3	11:13	457.4	5.9	10	0.59				
4	11:23	446.1	11.3	20	0.565				3.6
5	11:43	440.5	5.6	10 min	0.56				10.2
6	11:53	435.0	5.5	10 min	0.55	1.65	3 min	0.55	15.8
7	12:03	426.9							
8	12:13	423.1	11.9	20 min	0.595	13.55	23 min	0.589	27.7
9	12:33	411.7							
10	12:39	406.1	17			30.55	49	0.623	44.6
11	15:05	327.5							123.2
12	15:17	321.1	6.4	12	0.53				129.6
13	15:27	314.9	6.2	10	0.62				135.8
14	15:37	309.5	5.4	10 min	0.54	3.78	7 m	0.54	141.2
15	15:47	302.2	6.3	10 m	0.63	10.08	17	0.59	147.5
16	15:57	297.8	5.4	10 m	0.54	46.03	76	0.605	153.8
17	16:07	292.5	5.3	10 m	0.53	51.33	86	0.596	159.2
18	16:17	286.8	5.7	10 min	0.53	57.03	96	0.59	164.5
19	16:27	281.2	5.6	10 min	0.56	62.63	106	0.59	170.2
20	16:37	275.8	5.4	10 m	0.54	68.03	116	0.586	175.6
21	16:47	270.1	5.7	10 m	0.57		126		181.2
22	16:57	264.7	5.4	10 m	0.54	79.13	136	0.582	186.7
23	17:07	258.9	5.8	10 m	0.58	84.93	146	0.58	192.3
24	17:17	253.1	5.8	10 m	0.53	90.13	156	0.57	198.2
25	17:27	247.3	5.8	10 m	0.58	96.53	166	0.581	203.9
26	17:37	241.6	5.7	10 m	0.57	102.23	176	0.580	209.7
27	17:47	235.8	5.8	10 m	0.57	108.03	186	0.580	215.5
28	17:57	230.0	5.8	10 m	0.58		196		221.3
29	18:07	223.7	6.3	10 m	0.63	120.13	206	0.583	227.5

ESS Spiking Technician Signature: Scott [illegible]

Date: 7/30/2006

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IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID	Date: 3/30/2006	TC#: 1	Run#: 3	Spiking Material (ID): Pere

Page 2 of 2

Spiking Rate Data: Time (T) 00:00	Mass (M), Lb	Spiking Rate Calculations: Short-Term Average ΔM_i	ΔT_i	Rate$_i$ = $\Delta M_i/\Delta T_i$	Cum Run Average $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run Ave$_i$ = $\Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
								Start 19:59
18:07	223.7							
18:17	217.8	5.9	10 min	0.59	120.13	206	0.583	227.5
18:27	212.0	5.8	10 min	0.58	126.03	216	0.583	233.4
18:37	206.1	5.9	10 min	0.59	131.83	226	0.5833	239.2
18:47	200.2	5.9	10 min	0.59	137.73	236	0.5836	245.0
18:57	194.4	5.8	10 min	0.58	143.63	246	0.5838	251.0
19:07	188.6	5.8	10 min	0.58	149.43	256	0.5837	256.8
19:17	182.8	5.8	10 min	0.58	155.23	266	0.5835	262.6
19:27	177.0	5.8	10 min	0.58	161.03	276	0.5834	268.4
19:37	171.1	5.9	10 min	0.59	166.93	286	0.5833	274.1
19:47	165.3	5.8	10 min	0.58	172.73	296	0.5835	280.0
19:57	159.5	5.8	10 min	0.58	178.53	306	0.5834	285.8
20:07	153.7	5.8	10 min	0.58	184.33	316	0.5833	291.6
					190.13	326	0.5832	297.3

ESS Spiking Technician Signature: Scott Neal

Date: 3/30/2006

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Project ID: Westinghouse PDT TC# 1 Run# 3 Date 3/30/2006
Material: PCRC Based on Sample #3 Rates

∴Start Time 11:50 / 15:30
∴End Time 12:39 / 19:59
Δ Time = 12:39-11:50 = 49 / 19:59-15:30 = 269 = 318 Min

∴Start Mass 435.0 + (3 mins @ 0.55 lb/min = 1.65 lb) = 436.65

∴End Mass 406.1

∴Start Mass 314.9 − (3 min @ 0.54 lb/min = 1.62 lb) = 313.28

∴End Mass 159.5 − (2 min @ 0.58 lb/min = 1.01 lb) = 158.44

Δ Mass = (436.65-406.1) + (313.28 - 158.44) = 185.39 Lb

Spiking Rate = 185.39 Lb / 318 Min = 0.5830 Lb/Min X 60 = 34.98 Lb/Hr

Target Spiking Rate for Test Condition 35.00

Spiking Rate 34.98 Lb ÷ Target Spiking Rate 35.00 Lb = 99.94 % of Target Rate

---

Project ID: Westinghouse PDT TC# 1 Run# 3 Date 3/30/2006
Material: PCRC Based on HPM 10-2 Rates

∴Start Time 11:50 / 15:30
∴End Time 12:39 / 19:59
Δ Time = (19:59 - 15:30) + (12:39 - 11:50) = 318 Min

∴Start Mass 10.2 + (7 min @ 0.55 lb/min = 3.85 lb) = 14.05

∴End Mass 44.6

∴Start Mass 135.8 + (3 min @ 0.54 lb/min = 1.62 lb) = 137.42

∴End Mass 291.6

Δ Mass = (291.6 - 135.8) + (44.6 - 14.05) = 184.73 Lb

Spiking Rate = 184.73 Lb / 318 Min = 0.5809 Lb/Min X 60 = 34.85 Lb/Hr

Target Spiking Rate for Test Condition 35.0

Spiking Rate 34.85 Lb ÷ Target Spiking Rate 35.0 Lb = 99.58 % of Target Rate

Projec ,n: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 3/30/2006	TC#: 1	Run#: 3	Spiking Material (ID): Organic Solution				Page 1 of 2
Equipment ID:	*Spiking Manager* © #:		Pump ID	LMI# —	Neptune# 4	Moyno# —	Weigh Scale #: F- 2	MFM# 103
Spiking Data File Name: Organic TC1 R3				Weather Conditions: warm				

Notes:

4/16/HW 0.68 16/MIN

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Short-Term Average: ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	Cum Run Average: $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run $Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	START 11:50 STOP 12:39 RESTART 15:30 Comments/Observations
0	10:17	508.8							
1	10:[illegible]	493.1	15.7	25	0.628				15.6
2	11:02	480.3	12.8	20	0.64				28.4
3	11:12	473.1	7.2	10	0.72				35.7
4	11:32	459.1	14	20	0.70				49.7
5	11:42	452.2	6.9	10 MIN	0.69				56.7
6	11:52	446.8	5.4	10 MIN	0.54	1.08	2 MIN	0.54	62.2
7	12:06	438.2		14 MIN					70.8
8	12:12	434.6	12.2	20 MIN	0.61	13.28	22 MIN	0.603	74.4
9	12:32	429.0							
10	12:39	406.0				41.28	49 MIN	0.85	[illegible]
11	15:04	360.4							100.6
12	15:16	352.7	7.7	12 MIN	0.64				
13	15:26	344.4	8.3						8.1
14	15:36	335.6	8.8	10 MIN	0.88	5.28	6 MIN	0.88	18.9
15	15:46	330.4	5.2	10 MIN	0.52	10.48	16 MIN	0.655	22.1
16	15:56	325.0	5.4	10 MIN	0.54	59.76	75	0.77	27.5
17	16:06	319.3	5.7	10 M	0.57	62.46	85	0.74	33.2
18	16:16	313.8	5.5	10 M	0.55	68.95	95	0.72	38.7
19	16:26	308.2	5.6	10 M	0.56	74.55	105	0.71	44.3
20	16:36	302.6	5.6	10 M	0.56	80.15	115	0.696	49.8
21	16:46	297.0	5.6	10 M	0.56		125		55.5
22	16:56	291.5	5.5	10 M	0.55	91.25	135	0.675	61.0
23	17:06	285.2	6.3	10 M	0.63	97.55	145	0.672	67.3
24	17:16	278.8	6.4	10 M	0.64	103.95	155	0.67	73.6
25	17:26	272.4	6.4	10 M	0.64	110.35	165	0.668	80.0
26	17:36	265.7	6.7	10 M	0.67	117.05	175	0.6688	86.8
27	17:46	158.3	7.4	10 M	0.74	124.45	185	0.672	93.5
28	17:56	251.7	6.6	10 M	0.66	130.95	195	0.6772	100.1
29	18:06	244.3	7.4	10 M	0.74	138.45	205	0.675	107.5

Test Stopped Due to Drum Change

ESS Spiking Technician Signature: Scott Neal

Date: 3/30/2006

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Projec ..n: Phase IV.K. Field Spiking Log Sheets:
Project ID: 2006 Westate CPT. Date Prepared: 3/8/2006

IV.K.5.b Spiking Log (2nd & subsequent sheets for each run) 19:59

Data ID	Date: 3/30/2006	TC#: 1	Run#: 3	Spiking Material (ID): Organic Solution	Page 2 of 2

Spiking Rate Data:		Spiking Rate Calculations:						
		Short-Term Average			Cum Run Average			
Time (T) 00:00	Mass (M), Lb	ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	$\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	$Run\ Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
18:06	244.3				138.45	205	0.675	107.5
18:16	237.2	7.1	10 MIN	0.71	145.55	215	0.676	114.6
18:26	229.9	7.3	10 MIN	0.73	152.85	225	0.679	121.8
18:36	222.9	7.0	10 MIN	0.70	159.85	235	0.68	128.8
18:46	216.0	6.9	10 MIN	0.69	166.75	245	0.6806	135.8
18:56	209.1	6.9	10 MIN	0.69	173.65	255	0.6809	142.7
19:06	202.1	7.0	10 MIN	0.70	180.65	265	0.6817	149.7
19:16	195.2	6.9	10 MIN	0.69	187.55	275	0.6820	156.6
19:26	188.4	6.8	10 MIN	0.68	194.35	285	0.682	163.4
19:36	181.5	6.9	10 MIN	0.69	201.25	295	0.682	170.3
19:46	174.6	6.9	10 MIN	0.69	208.15	305	0.682	177.1
19:56	167.8	6.8	10 MIN	0.68	214.95	315	0.682	183.9
20:06	161.9	6.9	10 MIN	0.69	221.85	325	0.6826	190.8

STOP 19:16
STRT 19:59

ESS Spiking Technician Signature: [signature] Date: 3/30/2006

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Project ID: Westlake PDT TC# 1 Run# 3 Date 3/30/2006
Material: Organic Solution Based on Scale 8-2 Rates

∴Start Time 11:50/15:30
∴End Time 12:39/19:59
Δ Time = (12:39-11:50)+(19:59-15:30) = 318 Min

∴Start Mass 446.8+(2min @ 0.54lb/min = 1.08lb) = 447.88

∴End Mass 406.0

∴Start Mass 344.4 - (4min @ 0.88lb/min = 3.52lb) = 340.88

∴End Mass 167.8-(3min @ 0.69lb/min 2.07lb) = 165.3

Δ Mass = (447.88-406.0)+(340.88-165.3) = 217.46 Lb

Spiking Rate = 217.46 Lb / 318 Min = 0.6838 Lb/Min X 60 = 41.03 Lb/Hr

Target Spiking Rate for Test Condition 41.0

Spiking Rate 41.03 Lb ÷ Target Spiking Rate 41.0 Lb = 100.1 % of Target Rate

---

Project ID: Westlake PDT TC# 1 Run# 3 Date 3/30/2006
Material: Organic Solution Based on ACFM 10-5 Rates

∴Start Time 11:50/15:30
∴End Time 12:39/19:59
Δ Time = (12:39-11:50)+(19:59-15:30) = 318 Min

∴Start Mass 62.2+(2min @ 0.55lb/min = 1.1lb) = 61.1

∴End Mass 102.6

∴Start Mass 8.14(4min @ 0.87lb/min = 3.48lb) = 11.58

∴End Mass 183.9+(3min @ 0.69lb/min = 2.07lb) = 185.97

Δ Mass = (185.97-11.58)+(102.6-61.1) = 215.89 Lb

Spiking Rate = 215.89 Lb / 318 Min = 0.6788 Lb/Min X 60 = 40.73 Lb/Hr

Target Spiking Rate for Test Condition 41.0

Spiking Rate 40.73 Lb ÷ Target Spiking Rate 41.0 Lb = 0.994 % of Target Rate

IV.K.5.a Spiking Log & Run Identification Sheet (1st sheet)

Data ID	Date: 3/30/2006	TC#: 1	Run#: 3	Spiking Material (ID): Pb/Cu Solution			Page 1 of 2
Equipment ID:	Spiking Manager © #:		Pump ID	LMI# —	Neptune#	Moyno# —	Weigh Scale #: F- 5 / MFM# 10-4
Spiking Data File Name: Pb/Cu TC1R3				Weather Conditions:			

Notes:

2016 / Hr 0.3333 lb/min

i	Spiking Rate Data: Time (T), 00:00	Mass (M), Lb	Spiking Rate Calculations: Short-Term Average ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	Cum Run Average $\Sigma_i \Delta M_i$	$\Sigma_i \Delta T_i$	Run $Ave_i = \Sigma_i \Delta M_i / \Sigma_i \Delta T_i$	Comments/Observations
0	10:19	428.3							
1	10:26	428.3	1.0	25 min	0.32				13.0
2	11:00	412.4							14.9
3	11:14	410.3	3.1	9 min	0.34				23.0
4	11:34	403.5	6.8	20 min	0.34				29.7
5	11:44	400.0	3.5	10 min	0.35				33.2
6	11:54	396.9	3.1	10 min	0.31	1.24	4 min	0.31	36.2
7	12:07	392.1							41.0
8	12:14	390.0	6.9	20 min	0.31	8.14	24 min	0.339	43.1
9	12:34	384.2							
10	12:39	383.9	6.1	25 min	0.244	14.24	49 min	0.29	49.3
11	15:06	347.2							
12	15:19	342.1							
13	15:28	338.5	4.1	10 min	0.41				90.6
14	15:38	334.5	4.0	10 min	0.40	3.2	8 min		95.5
15	15:48	331.0	3.9	10 min	0.35	30.24	67 min		102.1
16	15:58	327.5	3.5	10 min	0.36	34.14	77 min	0.317	105.6
17	16:08	323.9	3.6	10 min	0.36	38.04	87 min	0.327	109.1
18	16:18	320.2	3.7	10 min	0.37	41.74	97 min	0.327	112.8
19	16:28	316.7	3.5	10 min	0.35	45.24	107 min	0.329	116.3
20	16:38	313.1	3.6	10 min	0.36	48.84	117 min	0.332	123.6
21	16:48	309.4	3.7	10 min	0.37	52.54	127 min	0.334	127.2
22	16:58	305.8	3.6	10 min	0.36	56.14	137 min	0.336	131.0
23	17:08	301.9	3.9	10 min	0.39	60.04	147 min	0.340	135.7
24	17:18	297.8	4.1	10 min	0.41	64.14	157 min	0.3448	139.8
25	17:28	294.2	3.6	10 min	0.36	67.74	167 min	0.345	143.1
26	17:38	290.9	3.3	10 min	0.33	71.04	177 min	0.345	146.4
27	17:48	287.5	3.4	10 min	0.34	74.44	187 min	0.344	149.5
28	17:58	285.3	2.2	10 min	0.22	76.64	197 min	0.339	151.7
29	18:08	282.5	2.8	10 min	0.28	79.44	207 min	0.335	150.4

ESS Spiking Technician Signature: [signature] Date: 3/30/2006

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IV.K.5.b Spiking Log (2nd & subsequent sheets for each run)

Data ID	Date: 3/30/2006	TC#: 1	Run#: 3	Spiking Material (ID): Pb/Cl Solution	Page 2 of 2

END 19:59

Spiking Rate Data:		Spiking Rate Calculations:						
		Short-Term Average			Cum Run Average			
Time (T) 00:00	Mass (M), Lb	ΔM_i	ΔT_i	$Rate_i = \Delta M_i/\Delta T_i$	$\Sigma_i \Delta M_i$	$\Sigma \Delta T_i$	$Run\ Ave_i = \Sigma_i \Delta M_i / \Sigma \Delta T_i$	Comments/Observations
18:07	272.5				69.44	207 min	0.335	152.4
18:17	279.3	3.2	10 min	0.32	72.64	217 min	0.345	153.6
18:28	276.4	2.9	10 min	0.29	75.54	227 min	0.333	158.5
18:38	278.2	3.6	10 min	0.36	79.14	237 min	0.334	160.1
18:48	279.5	3.3	10 min	0.33	82.44	247 min	0.338	163.4
18:58	280.2	3.3	10 min	0.33	85.74	257 min	0.336	166.7
19:08	283.5	3.2	10 min	0.32	88.94	267 min	0.332	169.9
19:18	284.8	3.2	10 min	0.32	92.14	277 min	0.333	173.1
19:28	286.5	3.3	10 min	0.33	95.44	287 min	0.333	176.2
19:38	288.3	3.2	10 min	0.32	98.64	297 min	0.332	179.5
19:48	290.0	3.3	10 min	0.33	101.94	307 min	0.332	182.8
19:58	291.7	3.3	10 min	0.33	105.24	317 min	0.332	186.1
20:08	293.5	3.2	10 min	0.32	108.44	327 min	0.332	189.3

ESS Spiking Technician Signature: Scott Wise

Date: 3/30/2006

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Project ID: Westate PDT TC# 1 / Run# 3 Date 3/30/2006
Material: Pb/Cr III Solution Based on Scale F5 Rates

∴Start Time 11:50/15:30
∴End Time 12:39/19:59
Δ Time = (19:59-15:30)+(12:39-11:50) = 318 Min

∴Start Mass 396.9+(4min @ 0.31lb/min = 1.24lb) = 398.14

∴End Mass 383.9

∴Start Mass 335.5-(2min @ 0.40lb/min = 0.80lb) = 337.7

∴End Mass 246.7-(1min @ 0.33 lb/min = 0.33lb) = 246.37

Δ Mass = (398.14-383.9)+(337.7-246.37) = 105.57 Lb

Spiking Rate = 105.57 Lb / 318 Min = 0.3320 Lb/Min X 60 = 19.92 Lb/Hr

Target Spiking Rate for Test Condition 20.00

Spiking Rate 19.92 Lb ÷ Target Spiking Rate 20.0 Lb = 99.6 % of Target Rate

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Project ID: Westate PDT TC# 1 Run# 3 Date 3/30/2006
Material: Pb/Cr III Solution Based on MPM 10-4 Rates

∴Start Time 11:50/15:30
∴End Time 12:39/19:59
Δ Time = (19:59-15:30)+(12:39-11:50) = 318 Min

∴Start Mass 36.2-(4min @ 0.30lb/min = 1.2lb) 35.0

∴End Mass 49.3

∴Start Mass 94.6+(2min @ 0.39lb/min = 0.78lb) = 95.38

∴End Mass 186.1+(1min @ 0.32lb/min = 0.32lb) = 186.42

Δ Mass (186.42-95.38)+(49.3-35.0) = 105.34 Lb

Spiking Rate = 105.34 Lb / 318 Min = 0.3313 Lb/Min X 60 = 19.88 Lb/Hr

Target Spiking Rate for Test Condition 20.00

Spiking Rate 19.88 Lb ÷ Target Spiking Rate 20.0 Lb = 99.4 % of Target Rate

Attachment V Effect of Measurement Uncertainty on Spiking Rate Uncertainty

A. Schofield, PhD, PE, Bill, Anthony R Eicher, and Sean O'Brien, The Effect of Measurement Uncertainty on Material Composition and Spiking Rate Uncertainties, 2004 IT3 Conference Proceedings, Paper #103, Phoenix, AZ, May, 2004
B. Schofield, PhD, PE, Bill, Anthony R Eicher, and Sean O'Brien, The Effect of Measurement Uncertainty on Field Spiking Rate and Overall Specie Spiking Rate Uncertainties, 2004 IT3 Conference Proceedings, Paper #102, Phoenix, AZ, May, 2004

Attachment V Effect of Measurement Uncertainty on Spiking Rate Uncertainty

A. Schofield, PhD, PE, Bill, Anthony R Eicher, and Sean O'Brien, The Effect of Measurement Uncertainty on Material Composition and Spiking Rate Uncertainties, 2004 IT3 Conference Proceedings, Paper #103, Phoenix, AZ, May, 2004
B. Schofield, PhD, PE, Bill, Anthony R Eicher, and Sean O'Brien, The Effect of Measurement Uncertainty on Field Spiking Rate and Overall Specie Spiking Rate Uncertainties, 2004 IT3 Conference Proceedings, Paper #102, Phoenix, AZ, May, 2004

Attachment V. Effect of Measurement Uncertainty on Spiking Rate Uncertainty

A. Schofield, PhD, PE, Bill, Anthony R Eicher, and Sean O'Brien, The Effect of Measurement Uncertainty on Material Composition and Spiking Rate Uncertainties, 2004 IT3 Conference Proceedings, Paper #103, Phoenix, AZ, May, 2004

THE EFFECT OF MEASUREMENT UNCERTAINTY ON SPIKING MATERIAL COMPOSITION AND SPIKING RATE UNCERTAINTIES

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ABSTRACT

It is not unusual for an agency, a client, or even a supplier of spiking materials to assert without justification that the only way to "know" the composition of a spiking material is through sampling and analysis of that spiking material. While this approach [which will be identified herein as the *sample and analyze method*] offers the advantage of determining composition independently of the spiking material supplier, it suffers the disadvantage of large measurement uncertainties resulting from inherent limitations in the analytical methods employed. However, there is another, fundamentally different, approach which is based on long standing principles of analytical chemistry and provides spiking material compositions with significantly smaller uncertainties. Conceptually, this approach is analogous to an analytical chemist preparing a laboratory standard for calibrating a sensitive analytical instrument. This approach, which will be identified herein as the *laboratory standard method* for preparing spiking materials, provides very accurate spiking specie concentrations.

Estimates of compositional uncertainty with the *laboratory standard method* developed herein are based on: (1) the test-specific details of a Case Study (e.g., a 2003 TB conducted at a private, US based HWC Unit), and (2) a series of worst case assumptions related to both the magnitude and direction of individual measurement uncertainties to produce the largest cumulative compositional uncertainty. Conversely, the assumption was made that no blatant operator mistakes were made since: (1) all measurements affecting composition were straight forward weight measurements using non-interpretive digital indicators, (2) the material preparation procedures were simple, and clear, (3) the documentation and record keeping procedures used were thorough, comprehensive, and consistently followed; and (4) the preparation procedure had built-in cross checks which included the utilization of two independent measurement observers and data recorders for most measurements. The premises on which the uncertainty analysis is based are explicitly identified, and rationales provided for their validity in the HWC Spiking context. Also, due to the small magnitude of all first-order uncertainties, second order uncertainties were ignored. Using first-order uncertainties developed for the case study example, the validity of this assumption was demonstrated.

The compositional uncertainty with the *laboratory standard method* is smaller (e.g., on the order of ± 0.1%) than is possible with the *sample and analyze method* using commercially available analytical methods. Depending on the method, matrix, and specie, the measurement uncertainty with commercially available analytical methods could vary from ± 5% up to ± 50%, assuming no uncertainty associated with sample collection and preparation.

This paper: (1) describes the spiking material preparation procedures used, (2) develops the apparent concentration of each spiking specie using the *laboratory standard method*, (3) describes the calculation procedures used to estimate uncertainty and presents the resulting estimates of uncertainty of spiking specie concentrations, (4) presents the impact of compositional uncertainty on spiking rate uncertainty, (5) estimates measurement uncertainty for the analytical methods most likely used to analyze spiking material composition in a HWC testing context, (6) presents the resulting impact on spiking rate, (7) presents a comparison of the composition and spiking rate uncertainties based on the *laboratory standard method* to those based on the *sample and analyze method*, and (8) proposes an approach for verifying spiking material composition independently of the material preparation firm should that be required in a regulatorily sensitive circumstance.

INTRODUCTION AND BACKGROUND

It is not unusual for an agency, a client, or even a supplier of spiking materials to assert that the only way to "know" the composition of a spiking material (e.g., a metal solution, an organic solution, a dispersion, and/or a "neat" POHC) is through sampling and analysis of that spiking material. While this approach [which will be identified herein as the *sample and analyze method*] offers the advantage of determining composition independently of the spiking material supplier, it suffers the disadvantage of large measurement uncertainties resulting from inherent limitations in the analytical methods employed. However, there is another, fundamentally different, approach which is based on long standing principles of analytical chemistry and provides spiking material compositions with significantly smaller uncertainties.

Conceptually, this approach is analogous to an analytical chemist preparing a laboratory standard for calibrating a sensitive analytical instrument and requires that one:

1. Know, with as much accuracy as possible, the purity of each reagent used in the preparation of a laboratory standard; especially as it relates to the chemical specie to be analyzed;

2. Use highly accurate, and carefully maintained measuring devices which are calibrated prior to use with NIST traceable standards; and

3. Maintain careful records for each step in the preparation of the laboratory standard.

This approach, which will be identified herein as the *laboratory standard method* for preparing spiking materials, provides very accurate spiking specie concentrations. The concentration uncertainty with this approach is smaller (e.g., on the order of ± 0.1%) than is possible with commercially available analytical methods (i.e., which, depending on the method, matrix, and specie could vary from ± 5% up to ± 50% without consideration of possible sample collection and preparation uncertainties).

Large concentration uncertainties are especially likely in spiking applications in which the use of SW846 and similar "low [analyte concentration] level" methods is required. As a result of large dilutions, these methods are generally not suitable for obtaining highly accurate analyses of the high analyte concentrations frequently encountered with spiking materials. Further, the *laboratory standard method* is expected to have smaller uncertainties than commercially available analytical methods which have been designed for analysis of samples with high analyte concentrations, due to the very large magnitude of the uncertainty advantage compared to low level methods, and since all of the analytical method uncertainties remain with high level methods except those associated with sample dilutions.

Estimates of compositional uncertainty with the *laboratory standard method* developed herein are based on: (1) the test-specific details of a Case Study (e.g., a 2003 TB conducted at a private, US based HWC

Unit), and (2) a series of worst case assumptions related to both the magnitude and direction of individual measurement uncertainties to produce the largest possible cumulative compositional uncertainty. Conversely, due to fact that: (1) all measurements are made with an absolute measurement method [based on the most fundamental parameter, e.g., gravity = mass, paraphrased from Reference (4)], (2) the use of non-interpretive digital indicators for all measurements, (3) the simplicity and clarity of the material preparation procedures used, (4) the use of thorough record keeping for each procedural step and measurement, (5) the experience and training of the personnel weighing the ingredients and preparing the finished spiking materials, and (6) built-in procedural cross checks including the utilization of two independent measurement observers and data recorders for most measurements; blatant operator mistakes are assumed to not be present. Also, due to the small magnitude of all first-order uncertainties, second order uncertainties were ignored.

Please note that a significant number of calculations are made in this uncertainty analysis. To avoid rounding errors and to retain the integrity of the uncertainty estimates developed herein, a relatively large number of significant figures are carried through the calculations and presented in the tables. The authors are not claiming the accuracy &/or precision in these figures that would normally be implied by the standard significant figures rules.

Engineered Spiking Solutions, Inc. (*ESS*) was retained to provide spiking materials as well as all necessary spiking equipment, and services for a Trial Burn (TB) which was conducted on a confidential, non-commercial, HWC Unit during 2003. The spiking materials used were: (1) a TiO_2 Dispersion (@ a nominal 25wt% Total Ash), and (2) a Naphthalene in Toluene Solution (@ a nominal 27wt% Naphthalene). The *laboratory standard method* was used in the case study with excellent results.

This paper: (1) describes the spiking material preparation procedures used, (2) develops the apparent concentration of each spiking specie using the *laboratory standard method*, (3) describes the calculation procedures used to estimate uncertainty and presents the resulting estimates of uncertainty of spiking specie concentrations, (4) presents the impact of compositional uncertainty on spiking rate uncertainty, (5) estimates measurement uncertainty for the analytical methods most likely used to analyze spiking material composition in a HWC testing context, (6) presents the resulting impact on spiking rate, (7) presents a comparison of the composition and spiking rate uncertainties based on the *laboratory standard method* to those based on the *sample and analyze method*, and (8) proposes an approach for verifying spiking material composition independently of the material preparation firm should that be required in a regulatorily sensitive circumstance.

Description of the "Case Study" Trial Burn

The Case Study TB consisted of two Test Conditions (TC) which were defined as follows: (1) TC #1: Maximum Waste Feed, and (2) TC #2: Minimum Temperature (DRE). The spiking materials[i] consisted of a 27% Naphthalene in Toluene Solution [Nap Sol] and a 25% TiO_2 Dispersion. The testing/spiking schedule is summarized as follows:

Test Condition	Date Conducted	Spiking With:	
		Nap Sol	Dispersion
TC #1	2003	✓	✓
TC #2	2003	✓	

The spiking function for this TB involved three spiking species[i] (e.g., Total Ash, Naphthalene, and Toluene) which were contained in two spiking materials[i] (e.g., TiO_2 Dispersion and Naphthalene in

Toluene Solution). The dispersion was used as an ash surrogate with ash contributions from both the TiO_2 (primary) and the proprietary dispersing agent (secondary). The Naphthalene in Toluene Solution spiking material contained both POHCs, e.g., Naphthalene and Toluene.

Conceptual Basis for the *Laboratory Standard Method* to Demonstrating Spiking Material Composition

The *laboratory standard method* for preparing and demonstrating the composition of spiking materials is analogous in concept to the approach employed by analytical chemists to prepare a laboratory standard for use in calibrating sensitive analytical instruments:

1. Make every effort to know, with as much accuracy as possible, the purity of each ingredient used in the preparation of a spiking material; especially as it relates to the specie being spiked, e.g. Naphthalene, Toluene, or Total Ash.

2. Have and carefully maintain a range (e.g., 1 Lb, 10 Lb, 50 Lb, 300 Lb, and 1,000 Lb) of highly accurate (Measurement Uncertainty = ± 0.01% of Full Scale Capacity, or better) weigh scales for accurately determining the quantity of each ingredient used. Use the most accurate (smallest) scale practical for a given application. Calibrate each scale with NIST traceable weight standards prior to each use.

3. Carefully record every spiking materials preparation step to facilitate documentation of the resulting spiking material composition, and QA audits.

Premises [with Supporting Rationales] on which this Uncertainty Analysis Was Based

The following premises were used as a basis for the Uncertainty Analysis provided herein. A rationale which demonstrates the validity of each premise in the HWC Testing/Spiking context is also provided:

1. **Premise:** No Chemical reactions will occur between the raw materials used to prepare a spiking material.

   Rationale: The two spiking materials used in the Case Study Trial Burn (e.g., Naphthalene in a Toluene solution, & TiO_2 in a mineral oil based dispersion) are typical of spiking materials in general, in that they well known in terms of chemistry and have been successfully used many times over a period of more than a decade. Naphthalene does not react chemically with toluene. Similarly TiO_2 and the proprietary dispersing agent are both chemically inert and furthermore would revert back to the same ash producing Ti & Si oxides in the combustion chamber if chemical reactions were to occur. Other systems, such as metal nitrates in an aqueous solution, will produce a weak nitric acid which could very well react with an unlined steel drum, will require special containers (lined or plastic drums) to ensure that such reactions do not occur.

2. **Premise:** Precipitation of spiking species out of solutions will not occur.

   Rationale: The solubility of the vast majority of spiking species (Naphthalene in Toluene, and metal salts in aqueous solutions) is well known (Merck Index, Perry's, etc.), and have been successfully used many times over a period of more than a decade. Solutions are never prepared at ≥90% of saturation (where cost has an impact, such as a Naphthalene in Toluene solution) and usually <50% of saturation (where there

is essentially no cost impact, such as aqueous solutions). Whenever there is any uncertainty, metal salts are not combined into the same solution as a means of ensuring that common ion and similar solubility effects do not bring composition in doubt.

3. **Premise:** Vapor Losses will have negligible impact on composition, or can be easily corrected for.

Rationale: All solutions are prepared in closed top drums which are kept sealed except when solute is added and mixed. Almost all spiking material solutions are aqueous solutions prepared at concentrations which are far from saturation. Thus, mixing times and associated vapor losses from the closed top drums are modest. For solutions prepared with higher vapor pressure solvents (e.g., Toluene), the quantity of vapor losses can be determined (by weight loss) and, if necessary, corrections made.

Measurement Uncertainty with Weight Measurements

All spiking material quantity measurements [which could affect the composition of the spiking materials discussed in this paper] were made using two weigh scales: (1) a 50 Lb bench scale, Model #: CQ25R33 manufactured by Ohaus Corporation, and (2) a 1,000 Lb floor scale, Model: Survivor FB2424-1000 manufactured by Rice Lake Weighing Systems. Selected (accuracy related) specifications for both of these scales are provided in Table I.

Table I Weighing Equipment Specifications

Specification	Units	Weigh Scale Manufacturer	
		Ohaus	Rice Lake
Capacity @ Full Scale (FS)	Lb (Kg)	50 (25)	1,000 (500)
Divisions[1](d)/FS			
NTEP[2]	d/FS	5,000	5,000
Non-NTEP[2]	d/FS	10,000	10,000
Lb/Division (%FS/d)			
NTEP[2]	Lb/d (%FS/d)	0.01 (0.01%)	0.02 (0.02%)
Non-NTEP[2]	Lb/d (%FS/d)	0.005 (0.005%)	0.01 (0.01%)
Non-Linearity	0.03% FS	NA	0.03% FS
Hysterises	0.02% FS	NA	0.02% FS

Footnotes:
1. The number of divisions/FS is an indication of scale sensitivity. For example, a division is the smallest weight increment discernable by the weighing system according to a given set of accuracy, calibration frequency, and environmental condition requirements.
2. NTEP is a quasi governmental organization established to regulate weights and measures used for commercial purposes. NTEP certified equipment has a conservative classification to properly reflect how measuring equipment may be used in commerce [i.e., infrequently calibrated, handled roughly, operated in a wide range of environmental conditions] while still providing acceptable accuracy. For the purposes of weighing ingredients for spiking materials with very frequent equipment maintenance & calibrations, and in controlled conditions of temperatures and humidity, the Non-NTEP division count is generally considered to be representative of scale accuracy. This observation has been confirmed by extensive pre-use and post-used calibration verifications with NIST traceable standards which consistently demonstrated deviations from the standards of $\leq$ 0.01% or equivalently d/FS $\geq$ 10,000.

PREPARATION OF SPIKING MATERIALS: TiO_2 DISPERSION

This section provides: (1) a description of the TiO_2 Dispersion preparation procedure, (2) the calculation procedure for determining Total Ash concentration and the calculated results, (3) the calculation procedure for estimating uncertainty in the apparent Total Ash concentration and the calculated results, and (4) the Certification of Composition for the TiO_2 Dispersion.

TiO_2 Dispersion Preparation Procedure (Summarized)

1. Setup and calibrate the 50 Lb ± 0.01 Lb, and 1,000 Lb ± 0.1 Lb weigh scales using NIST Traceable Weight Standards;

2. Add Mineral Seal Oil (MSO) to the Dispersion Matrix (DM) blend tank. Weigh each drum before and after the MSO transfer with the 1,000 Lb weigh scale. Record the drum gross and tare weights;

3. Weigh out the dispersion agent (DA) in four (4) batches on the 50 Lb ± 0.01 Lb weigh scale. Record the tare and gross weights;

4. Slowly add the dispersion agent to the blend tank and mix with maximum shear;

5. Weigh out the activator in five (5) batches on the 50 Lb ± 0.01 Lb weigh scale. Record the tare and gross weights;

6. Slowly add the activator to the blend tank and mix with maximum shear until the dispersing system is fully developed;

7. Drum off the DM per the Drum Weight Schedule provided. Weigh each numbered DM drum (Drum # 1-6 for the TiO_2 Dispersion) before and after adding DM and record the tare and gross weights;

8. Prepare six (6) batches (numbered 1-6) of TiO_2 for addition to the corresponding six (6) numbered drums of the TiO_2 Dispersion being prepared. Determine the quantity of TiO_2 in each batch on the basis of 0.3228 Lb TiO_2/Lb DM in the corresponding DM drum. Prepare each batch of TiO_2 in three sub-batches, (numbered as 1A, 1B, 1C; 2A, 2B,.....6B, 6C), record each tare and gross weight;

9. Slowly add each TiO_2 sub-batch to the corresponding drum of DM and shear thoroughly; and

10. Tightly seal, label and prepare each drum for shipment to the test site.

Calculation of Dispersion Matrix (DM) Composition

The DM composition data (e.g., the weights developed in procedure steps 2, 3, & 5 above) were compiled and summarized in Table II. Table II provides the DM composition information on weight (Lb), and weight percent (wt%) bases with corresponding uncertainty estimates. The term "indicated" as used herein refers to the apparent weight or weight percent of a substance as "indicated" on the digital readout devices [indicators] employed in this work.

Table II Composition of Dispersion Matrix

Constituent[1]	DM[1] #1 Composition By:					
	Weight, Lb			Weight Per Cent, Wt%		
	Target	Indicated	Uncertainty[2]	Target	Indicated	Uncertainty
MSO[1]	4,862.00	4,832.70	± 2.60[2]	94.16	94.12	± 0.0506
DA[1]	146.64	146.94	± 0.08[2]	2.84	2.8618	± 0.0016
Activator	154.91	154.98	± 0.10[2]	3.00	3.0183	± 0.0019
Total	5,163.55	5,134.62	± 2.78	100.00	100.00	± 0.0541

Footnotes:
1. **DM** = Dispersion Matrix, **MSO** = Mineral Seal Oil, & **DA** = Dispersing Agent.
2. Basis: A. Obtaining the total MSO weight involved a total of 26 individual weight measurements (e.g., gross and tare weights for 13 drums), each with an estimated measurement uncertainty of ± 0.1 Lb/weight measurement.
   B. Obtaining the total DA weight involved a total of 8 individual weight measurements (i.e., tare and gross weights for four batches of DA), each with an estimated measurement uncertainty of ± 0.01 Lb/weight measurement.
   C. Obtaining the total Activator weight involved a total of 10 individual weight measurements (i.e., tare and gross weights for five batches of activator), each with an uncertainty of ± 0.01 Lb/weight measurement.
   D. Each weight measurement is assumed to have measurement uncertainties in the direction which would produce the largest cumulative positive or negative uncertainty.

Calculation of the Total Ash Drum Concentrations

The TiO_2 Dispersion composition data from procedure steps 7 & 8 above, and Table II are summarized in Table III. Additionally, measured values for ash concentration (mass fraction ash) in the TiO_2 and the dispersion agent were provided by their respective manufacturers. These values were used to calculate the total ash content (expressed as Lb ash/Drum, and wt% ash) for each drum of finished TiO_2 Dispersion.

Estimated Uncertainty in Total Ash Concentration

The uncertainty in the total ash concentration (wt%) in a given drum is comprised of four primary components of uncertainty which were estimated as follows:

1. **The measurement uncertainty in determining the net weight of Dispersion Matrix (DM) per drum:** This measurement uncertainty is estimated as the sum of the uncertainties in the two [tare and gross] weight measurements obtained in preparation procedure (step 7 above) and is calculated as follows:

   - **DM Uncertainty** = 2 [weigh measurements] x ± 0.1 Lb DM [the uncertainty associated with each measurement].

   Thus, the net weight of the DM present in Drum #1, for example, [see Table III, column (4)], is estimated to be:

   - **DM/Drum** = 283.97 Lb ± 0.2 Lb DM/Drum.

Table III TiO_2 Dispersion, Total Ash Concentration

Drum #	Indicated Weight TiO_2 Disp Lb/Dr	DM Wt, Lb/Dr		Net Weight, Lb/Drum						Total Ash, Wt %
		Target	Indicated	TiO_2			DA		Total Ash	
				Target	Indicated	Ash[2]	Indicated	Ash[3]		
(1)[1]	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)
1	375.53	283.9	283.9	91.63	91.63	90.44	8.12	4.30	94.74	25.23[4]
2	375.53	283.9	283.9	91.63	91.63	90.44	8.12	4.30	94.74	25.23[4]
3	375.53	283.9	283.9	91.63	91.63	90.44	8.12	4.30	94.74	25.23[4]
4	375.53	283.9	283.9	91.63	91.63	90.44	8.12	4.30	94.74	25.23[4]
5	376.72	283.9	284.8	91.92	91.92	90.73	8.15	4.32	95.05	25.23[4]
6	374.87	283.9	283.4	91.47	91.47	90.28	8.11	4.30	94.58	25.23[4]
Ave	375.62	283.9	283.97	91.65	91.65	90.46	8.12	4.303	94.77	25.23

Footnotes:
1. The bracketed numbers, i.e. (1), (2) ...(11), in this row signify the Column numbers which are used in the calculation explanations to the right.
2. The TiO_2 is 98.7 wt% ash based on manufacturer's CoA.
3. The DA is 52.96 wt% ash based on manufacturer's CoA.
4. These values range from 25.2283% for Drums #1, 2, 3, & 4; to 25.2309% for Drum #5; and 25.2301% for Drum #6, for an average of 25.2290 wt % and a range of -0.0007 wt % to + 0.0019 wt %.
5. Mass fraction of DA in DM from Table II.

Information Sources:
1. Values provided in Columns (4) and (6) are based on measured weights.
2. Values provided in Columns (3) and (5) are targets provided in the detailed dispersion preparation SOP.

Calculations:
1. Weight TiO_2 Disp (Column 2) = Columns (4) + (6)
2. "Ash" content of TiO_2 (Column 7) = Column (6) x 0.987[2].
3. DA content (Column 8) = Column (4) x 0.0286[5]
4. Ash content of DA (Column 9) = Column (8) x 0.5296[3].
5. Total Ash (Column 10) = Columns (7) + (9).
6. Total Ash, wt % (Column 11) = Column (10) ÷ Column (2) x 100%.

2. **The measurement uncertainty in the ash contribution from TiO_2:** This uncertainty is estimated as the sum of:

(a) the weight measurement uncertainty in the quantity of TiO_2 added to each drum is estimated as follows:

± Ash (Lb ash/Drum) = [two weight measurements per TiO_2 sub-batch] x 3 [three sub-batches/drum] x ± 0.01 Lb [the uncertainty per weight measurement] x 0.987 [the mass fraction of TiO_2 which is ash]
= ± 0.0592 Lb ash/Drum), and

(b) the uncertainty in the Lb ash/drum due to uncertainty in the % ash in TiO_2 measurement. This uncertainty is estimated to be 98.7% ± 0.1% ash (or 0.987 ± 0.001 expressed as a mass fraction) in the ash content measurement times 91.65 Lb TiO_2/Drum [from Table III, column (6)], or:

± Ash (Lb ash/Drum) = (91.65 Lb TiO_2/Drum) x (± 0.001 Lb ash/Lb TiO_2)
= ± 0.09165 Lb ash/Drum.

The total estimated uncertainty in the mass of ash per drum from the TiO_2 is then ± 0.1509 Lb ash/Drum (e.g., ± 0.0592 Lb ash/Drum ± 0.09165 Lb ash/Drum = ± 0.1509 Lb ash/Drum).

Note: Consistent with the assumption that second-order uncertainties can be ignored, these two first-order uncertainties are simply added. The validity of this assumption is demonstrated below.

3. **The measurement uncertainty in the weight of TiO_2 per drum:** Following the logic [and the TiO_2 related math] of step 2(a) above, the weight measurement uncertainty in the quantity of TiO_2 added to each drum is estimated as follows:

   ± TiO_2 (Lb ash/Drum) = 2 [two weight measurements per TiO_2 sub-batch]
   x 3 [three sub-batches/drum]
   x ± 0.01 Lb [the uncertainty per weight measurement]

   Then, following the format of step 1 above:

   TiO_2/Drum = 91.65 Lb ± 0.06 Lb TiO_2/Drum.

4. **The uncertainty in the ash contribution from the dispersing agent:** Following the logic of step 2 above, this uncertainty is estimated as the sum of:

   (a) the uncertainty in the DA content per drum (± 0.08 Lb DA [from the fourth column from the left in Table II] divided by the number of DM drums produced in this DM lot [19 drums]) x (0.5296 Lb ash/Lb DA), or:

   ± Ash (Lb ash/Drum) = (± 0.08 Lb DA/19 Drums) x (0.5296 Lb ash/Lb DA)
   = ± 0.0022 Lb ash/Drum, and

   (b) the uncertainty in the weight loss on ignition measurement which was estimated at 52.96 % ± 0.1 % (or 0.5296 ± 0.001 expressed as a mass fraction) times 8.12 Lb DA/Drum [From Table III, column (8)]), or:

   ± Ash (Lb ash/Drum) = (± 0.001 Lb ash/Lb DA) x (8.12 Lb DA/Drum)
   = ± 0.00812 Lb ash/Drum.

   The total ash contribution from DA is ± 0.0103 Lb ash/Drum (± 0.0022 Lb ± 0.00812 Lb ash/Drum).

Uncertainty in the Total Ash Content per Drum:

The uncertainty in the total ash content per drum is then:

Ash Content Uncertainty from TiO_2	= ± 0.1509	Lb ash/Drum
Ash Content Uncertainty from DA	= ± 0.0103	Lb ash/Drum
Total Ash Content Uncertainty	= ± 0.1612	Lb ash/Drum

The uncertainty in ash concentration (expressed on a wt% basis) is estimated as follows (Drum #1 is used as an example):

$$\text{Uncertainty in wt\% Ash} = \frac{\pm\ 0.1612 \text{ Lb ash/Drum} \times 100\%}{(283.97 \pm 0.2 \text{ Lb DM/Drum}) + (91.65 \pm 0.06 \text{ Lb } TiO_2\text{/Drum})}$$

Note that wt% uncertainty is maximized when the DM weight is assumed to be the indicated weight minus the measurement uncertainty. [The smaller DM weight will minimize the denominator which in turn maximizes the wt% uncertainty.] Therefore, the maximum:

Positive Uncertainty in wt% Ash $= \frac{+0.1612 \text{ Lb ash X } 100\%}{(283.77\text{Lb} + 91.71^{ii} \text{ Lb})} = +0.0429$ wt % ash.

Negative Uncertainty in wt% Ash $= \frac{-0.1612 \text{ Lb ash X } 100\%}{(283.77\text{Lb} + 91.59^{iii} \text{ Lb})} = -0.0429$ wt % ash.

Thus, the TiO_2 Dispersion is 25.23% ± 0.0429% which was revised upward to 25.23% ± 0.045% to compensate for the minor drum to drum ash concentration difference described in Table III, footnote 4.

Certification of Composition for the TiO_2 Dispersion:

Based on this information, a Certification of Composition (CoC) for the TiO_2 Dispersion was prepared (See Fig. 1 for a highly abbreviated version of the TiO_2 Dispersion CoC).

Fig. 1 CERTIFICATE OF COMPOSITION: TiO_2 DISPERSION (Highly Abbreviated Format)

Product:	**TiO_2 DISPERSION**
Composition:	**Total Ash: 25.23 wt %[1]**

CERTIFICATION OF COMPOSITION:
I hereby certify that the composition information provided above and in the footnote is true and accurate to the best of my knowledge and belief.

Signed: _____ _____
W.R. (Bill) Schofield, PhD, PE **Date**
***ESS* Project Manager**

Footnotes:
[1] Based on an analysis of:
(a) the measurement uncertainty of weigh scales used to produce this material,
(b) the raw material composition information provided by the manufacturers, and
(c) the procedures which *ESS* used to produce this material;
I have concluded that the composition of this TiO_2 dispersion is 25.23% ± 0.045 wt% ash.

Demonstrating the Validity of the Assumption that Second-Order Uncertainties Can Be Ignored

This analysis of measurement uncertainty is partially based on the assumption that second-order uncertainties can be ignored. Using the first-order uncertainties calculated above, we can demonstrate the validity of this assumption. For example, we demonstrated above that the uncertainty in the quantity of ash from TiO_2: (1) due to weight measurement uncertainties was ± **0.0592 Lb ash/Drum**, and (2) due to ash concentration measurement uncertainty was ± **0.09165 Lb ash/Drum.** We will now calculate the second-order ash content uncertainty due to both TiO_2 weight measurement uncertainty and ash concentration uncertainty as follows:

± Ash (Lb ash/Drum) = [(± 0.0592 Lb ash/Drum)/(0.987 Lb ash/Lb TiO_2)] x (± 0.001 Lb Ash/Lb TiO_2)

= [± 0.0600 Lb TiO_2/Drum] x (± 0.001 Lb Ash/Lb TiO_2)

= ± 0.00006 Lb Ash/Drum

Obviously, an uncertainty of 6 parts in 100,000 parts is not significant even in the HWC Testing context. Similarly insignificant results would occur with other second-order uncertainties, simply due to the very small first-order uncertainties present.

PREPARATION OF SPIKING MATERIALS: NAPHTHALENE IN TOLUENE SOLUTION

This section provides: (1) a description of the Naphthalene in Toluene Solution preparation procedure, (2) the calculation procedure for determining Naphthalene and Toluene Concentrations and the calculated results, (3) the calculation procedure for estimating uncertainties in the apparent Naphthalene and Toluene Concentrations and the calculated results, and (4) the Certification of Composition for the Naphthalene in Toluene Solution.

Naphthalene in Toluene Solution Preparation Procedure (Summarized):

1. Setup and Calibrate the 50.00 Lb ± 0.01 Lb, and 1,000.0 Lb ± 0.1 Lb weigh scales using NIST Traceable Weight Standards;

2. Number fourteen (14) closed top "DOT" drums as Drum #1 through Drum #14;

3. Prepare fourteen (14) numbered batches (numbered I through 14) of 100.71 Lb of Naphthalene Flake. Weigh each batch on the 50.00 Lb bench scale as four sub-batches in sealed containers which are numbered as 1A, 1B, 1C, and 1D; through 14A, 14B, 14C, and 14D. Weigh each container before (tare weight) and after (gross weight) adding the Naphthalene and record the weights;

4. Weigh each drum and record the tare weight;

5. Add each Naphthalene sub-batch to the corresponding numbered closed top drum;

6. Weigh each drum after adding the Naphthalene and record the weight;

7. Add 272.3 Lb of Toluene to each drum and record the weight;

8. Mix the Naphthalene and Toluene contents of each drum thoroughly; and

9. Tightly seal, label and prepare each drum for shipment to the test site.

Calculation of Naphthalene Concentrations:

Table IV below provides the measured or indicated[iv] weights of each batch of Naphthalene, and the Toluene added to each drum; the estimated measurement uncertainty associated with each weigh scale reading (indication of weight); the Naphthalene purity (per the Manufacturer's Certificate of Analysis for the lot of Naphthalene used); and the calculated apparent or indicated Naphthalene concentration (wt%, assuming all weight measurements are accurate), as well as the cumulative Naphthalene concentrations uncertainty (based on the cumulative uncertainties assuming that each measurement was made with the maximum [error] measurement uncertainty and with the direction of each measurement uncertainty [error] which would result in largest increased[v] or decreased[v] concentrations, respectively, e.g., which would result in the maximum cumulative uncertainty).

Table IV Composition of Naphthalene in Toluene Solution by Drum

Nap Batch # & Drum #	Indicated Nap Weight, Lb/Batch	Scale Uncertainty[1], ± Lb	Indicated Toluene Weight, Lb/Drum	Scale Uncertainty[1], ± Lb	Nap Purity Correction, Mass Fraction	Wt% Naphthalene		
						Indicated	Min[2]	Max[2]
1	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
2	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
3	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
4	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
5	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
6	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
7	100.71	± 0.01	272.5	± 0.1	0.9985	26.944	26.914	26.974
8	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
9	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
10	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
11	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
12	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
13	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
14	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
Average	100.71	± 0.01	272.31	± 0.1	0.9985	26.958	26.928	26.988

Footnotes:

1. Estimated measurement uncertainty for a single weight measurement on the weigh scale used.
2. The following assumptions were made in estimating the maximum Naphthalene concentration for a given drum:
   All four Naphthalene tare Weights were assumed to be smaller by the scale measurement uncertainty,
   All four Naphthalene gross weights were assumed larger,
   The Toluene tare weight (drum + Naphthalene) was assumed larger, and
   The Toluene gross weight was assumed smaller.

In Toto, these worst case assumptions result in the Naphthalene weight being 0.08 Lb larger than indicated weight and the Toluene weight being 0.2 Lb smaller than the indicated weights. These assumptions resulted in the maximum Naphthalene concentration. The opposite assumptions would produce the minimum Naphthalene concentration. See Table V for further explanation.

Estimated Uncertainty in the Average Naphthalene Concentration:

Table V below describes the computational method and information used to estimate the concentration uncertainty for Naphthalene in the Naphthalene in Toluene Solution.

TABLE V Calculation of the Minimum and Maximum Naphthalene Drum Concentrations Based on Worst Case Cumulative Measurement Uncertainty Assumptions

Drum #	Weight, Lb						Naphthalene Concentration, Wt %		
	Naphthalene (Purity Corrected[1])			Toluene					
	Indicated[1]	Min[3]	Max[3]	Indicated[2]	Min[4]	Max[4]	Indicated[5]	Min[6]	Max[7]
1-6, & 8-14	100.559	100.479	100.639	272.3	272.1	272.5	26.959	26.929	26.989
7	100.559	100.479	100.639	272.5	272.3	272.7	26.944	26.914	26.974
Ave	100.559	100.479	100.639	272.31	272.11	272.51	26.958	26.928	26.988
Range	100.559	100.479	100.639	272.3 -272.5	272.1 -272.3	272.5 -272.7	26.944-26.959	26.914-26.929	26.974-26.989

Summary Results: Maximum Naphthalene Concentration Range = 26.914 to 26.989 wt %
Deviation from Indicated Average Concentration = - 0.044 to + 0.031 wt % Naphthalene
[Known] Naphthalene Concentration = 26.958 ± 0.044 wt % = 26.96 wt % ± 0.045 wt % Nap.

Footnotes:

1. Indicated Naphthalene Weight [from second column from left in Table IV] x 0.9985 [Nap Purity, Mass Fraction].
2. Indicated Toluene Weight from fourth column from left in Table IV.
3. Max Nap Weights (wt} = Indicated Nap wt + (4 [sub-batches]) x {(+0.01 [positive deviation[8] of gross wt]) – (-0.01 Lb [negative deviation[8] of tare wt])} = Indicated Nap wt + 0.08 Lb.
4. Max Toluene Weight = Indicated Toluene weight + (+0.1 Lb [positive[8] deviation of gross weight]) – (-0.01 Lb [negative[8] deviation of tare weight]) = Indicated Toluene wt + 0.2 Lb.
5. Indicated Naphthalene wt % = [(Indicated Nap[9] weight)/(Indicated Nap[10] weight + Indicated Toluene[10] weight)] x 100%.
6. Min Naphthalene wt % = [Min Nap[9] wt/(Min Nap[10] wt + Max Toluene[10] wt)] x 100%.
7. Max Naphthalene wt % = [Max Nap[9] wt/(Max Nap[10] wt + Min Toluene[10] wt}] x 100%.
8. Reverse wt deviations for minimum Nap and Toluene wts.
9. Purity Corrected.
10. Not Purity Corrected.

Estimated Toluene Concentration Uncertainty

Table VI below describes the computational method and information used to estimate the concentration uncertainty for Toluene in the Naphthalene in Toluene Solution.

TABLE VI Calculation of the Minimum and Maximum Toluene Drum Concentrations Based on Worst Case Cumulative Measurement Uncertainty Assumptions

Drum #	Weight, Lb						Toluene Concentration, Wt %		
	Naphthalene (w/o Purity Corrected[1])			Toluene (with Purity Corrected[2])					
	Indicated[1]	Min[3]	Max[3]	Indicated[2]	Min[4]	Max[4]	Indicated[5]	Min[6]	Max[7]
1-6, & 8-14	100.71	100.63	100.79	272.11	271.91	272.31	72.95	72.92	72.98
7	100.71	100.63	100.79	272.31	272.11	272.51	72.964	72.934	72.994
Ave	100.71	100.63	100.79	272.1243	271.9243	272.3243	72.951	72.921	72.981
Range	100.71	100.63	100.79	272.11 -272.31	271.91 -272.11	272.31 -272.51	72.95 –72.964	72.92- 72.934	72.98– 72.994

Summary Results: Maximum Toluene Concentration Range = 72.92 to 72.994 wt %.
Deviation from Indicated Average Concentration = - 0.031 to + 0.043 wt %.
[Known] Toluene Concentration = 72.951 wt % ± 0.043 wt % = 72.95 wt % ± 0.045 wt %.

Footnotes:

1. Indicated Naphthalene Weight [from second column from left in Table III, without purity correction].
2. Indicated Toluene Weight from fourth column from left in Table III x 0.9993 [Toluene purity, mass fraction, see attached manufacturer's CoA].
3. Max Nap Weights (wt) = Indicated Nap wt + (4 [sub-batches]) x {(+0.01 [positive deviation[8] of gross wt]) – (-0.01 Lb [negative deviation[8] of tare wt])} = Indicated Nap wt + 0.08 Lb.
4. Max Toluene Weight = Indicated Toluene weight + (+0.1 Lb [positive[8] deviation of gross weight]) – (-0.01 Lb [negative[8] deviation of tare weight]) = Indicated Toluene wt + 0.2 Lb.
5. Indicated Toluene wt % = [(Indicated Toluene[9] weight)/(Indicated Toluene[10] weight + Indicated Nap[10] weight)] x 100%.
6. Min Toluene wt % = [Min Toluene[9] wt/(Max Nap[10] wt + Min Toluene[10] wt)] x 100%.
7. Max Toluene wt % = [Max Toluene[9] wt/(Min Nap[10] wt + Max Toluene[10] wt)] x 100%.
8. Reverse wt deviations for minimum Nap and Toluene wts.
9. Purity Corrected.
10. Not Purity Corrected

Certificate of Composition Naphthalene in Toluene Solution

Based on the information provided herein, the Certificate of Composition (CoC) for the Naphthalene in Toluene Solution was prepared (See Fig. 2 for a highly abbreviated version of the Naphthalene in Toluene Solution CoC).

Fig. 2 CERTIFICATE OF COMPOSITION: NAPHTHALENE IN TOLUENE SOLUTION (Highly Abbreviated Format)

Product:	**Naphthalene in Toluene Solution**
Composition:	**Naphthalene[1]: 26.96 wt %** **Toluene[1]: 72.95 wt %**

CERTIFICATION OF COMPOSITION:
I hereby certify that the composition information provided above and in the footnote is true and accurate to the best of my knowledge and belief.

Signed: _____ _____
W.R. (Bill) Schofield, PhD, PE **Date**
***ESS* Project Manager**

Footnotes:
[1] Based on an analysis of:
(a) the measurement uncertainty of the weigh scales used to produce this material,
(b) the Naphthalene and Toluene manufacturers' Certifications of Analysis, and
(c) the procedures which were used to produce this material,
I have concluded that the composition of the Naphthalene in Toluene Solution is:
(a) Naphthalene = 26.96 wt % ± 0.045 wt %, and
(b) Toluene = 72.95 wt % ± 0.045 wt %.

IMPACT OF COMPOSITION UNCERTAINTY ON ABSOLUTE AND RELATIVE SPIKING RATE UNCERTAINTY

The impact of compositional uncertainty discussed above on the Species (S) spiking rate uncertainty was calculated on two bases:

1. Absolute Species (S) Spiking Rate Uncertainty, ± Lb S/Hr, and

2. Relative Species Spiking Rate Uncertainty [uncertainty expressed as a % of the indicated spiking rate, ± %RU].

The results are presented in Table VII and summarized as follows:

Spiking Specie (S)	Spiking Rate, Lb S/Hr	Specie Spiking Rate Uncertainty:	
		Absolute Uncertainty, ±Lb S/Hr	Relative Uncertainty, ±% RU
Ash	14.12	± 0.0064 Lb Ash/Hr	± 0.045% RU
Naphthalene	26.52	± 0.0119 Lb Nap/Hr	± 0.045% RU
Toluene	67.54	± 0.0323 Lb Toluene/Hr	± 0.045% RU

Table VII Effect of Composition Uncertainty Associated with the *Laboratory Standard Method* on Specie Spiking Rate Uncertainty

Spiking Specie, (S)	Effect of Composition Uncertainty on:						
	Mass/Run:				Apparent Spiking Rate, Lb S/Hr	Absolute Specie Spiking Rate Uncertainty, ± Lb S/Hr	Relative Specie Spiking Rate Uncertainty, ± % RU
	Material		Specie				
	± Lb	± %	± Lb	±%			
Ash[1]	0.2	0.18	0.05	0.18	14.12	± 0.0064 Lb/Hr	± 0.045 %RU
Nap[2]	0.2	0.06	0.06	0.06	26.52	± 0.0119 Lb/Hr	± 0.045 %RU
Toluene[3]	0.2	0.08	0.15	0.08	67.54	± 0.0323 Lb/Hr	± 0.045 %RU

1. Total Ash has an indicated composition of 25.23 wt % ± 0.045 wt % in 110.49 Lb TiO_2 Dispersion/Run (Table III)
2. Naphthalene has an indicated composition of 26.96 wt % ± 0.045 wt % in 317.96 Lb Nap Sol/Run (Table V)
3. Toluene has an indicated composition of 72.95 wt % ± 0.045 w % in 246.88 Lb Nap Solution/Run (Table VI)

Inspection of these results indicates that the compositional uncertainty associated with the *laboratory standard method* of demonstrating spiking material composition resulted in very modest spiking rate uncertainties whether on an absolute and relative uncertainty basis.

COMPARISON OF *Laboratory Standard Method* AND *Sample and Analyze Method* UNCERTAINTIES

In order to complete this analysis by comparing the uncertainties associated with the *laboratory standard method* to the corresponding uncertainties associated with the *sample and analyze method*, it is first necessary to estimate the measurement uncertainties associated with the analytical methods (SW846 or similar methods) which are most likely to be used to determine the composition of spiking materials in a HWC Test context.

Three approaches were utilized to estimate measurement uncertainties of the applicable SW846 (& ASTM) Methods:

1. Reviewing a recent, Agency approved QAPP for guidance using the acceptable analyte recovery range for a given method in duplicate spiked samples,

2. Reviewing Agency Guidance, specifically QA Objectives for method accuracy (defined for a given method as the acceptable analyte recovery range in duplicate spiked samples), and

3. Polling Analytical/Trial Burn Experts for opinions based on experience.

Table VIII summarizes the results of that effort.

Table VIII Estimated Measurement Uncertainties for Selected Analytical Methods

Spiking:		Analytical Method[1]	Source of Method Uncertainty Estimates:		
Specie	Material		Recent QAPP(2)	Guidance(3)[2]	Expert Opinion(4)[3]
Ash	TiO_2 Dispersion	ASTM D-482	± 10 %	± 25 %	NA
Metals	Dispersion or Solution	6010 & 7470	± 30 %	± 30 %	6 - 41 %
Naphthalene	Nap & Toluene Solution	8270	-90 to -54,+50 %	± 50 %	6 - 40 %
Toluene	Nap & Toluene Solution	8260	-50, +30 %	± 50 %	10 - 30 %

Footnotes:
1. SW846 unless otherwise noted.
2. Reference (3), QA Objectives for TB, Table III-1, Process Samples.
3. Reference (4), based on low [analyte concentration] level sample analysis.

Inspection of Table VIII prompts three significant observations:

1. There is a relatively wide range within the measurement uncertainty estimates;

2. The expert opinion estimates of measurement uncertainty are based on low [analyte concentration] level analyses and, as such, probably under state the measurement uncertainty which would be present with analysis of high level spiking material samples. Conversely, the QAPP and Guidance estimates are based on a wide range of analytical laboratories and, as a result, probably over state uncertainties associated with analytical results from a laboratory with a strong QA/QC Program; and

3. The level of measurement uncertainty associated with each of these analytical methods (*sample and analyze method*) is at least two (2) orders of magnitude larger than the measurement uncertainty associated with the *laboratory standard method* (e.g., ± 5% vs. ± 0.045%).

As a result of the last observation, no further effort was invested to refine the measurement uncertainty for the analytical methods. As a result of the first two observations, the following method specific measurement uncertainties were, somewhat arbitrarily, selected:

Method	Analyte	Measurement Uncertainty, ±%
ASTM D-482	Ash	± 10%
SW846 8270	Naphthalene	± 30%
SW846 8260	Toluene	± 30%

These measurement uncertainty estimates were used to calculate the absolute and relative spiking rate uncertainties on the same case study basis and with the results were summarized in Table IX, below.

Table IX Effect of Compositional Uncertainty Associated with the *Sample & Analyze Method* on Specie Spiking Rate

Spiking Specie, (S)	Apparent Spiking Rate, Lb S/Hr	Est'ed Measurement Uncertainty, ± %	Absolute Spiking Rate Uncertainty, ± Lb S/Hr	Relative Spiking Rate Uncertainty, ± %RU
Ash	14.12 Lb/Hr	± 10%	± 1.41 Lb Ash/Hr	± 10%RU
Naphthalene	26.52 Lb/Hr	± 30%	± 7.96 Lb Nap/Hr	± 30%RU
Toluene	67.54 Lb/Hr	± 30%	± 20.3 Lb Toluene/Hr	± 30%RU

The absolute and relative specie spiking rate uncertainties based on the *laboratory standard method* and the *sample and analyze method* were then taken from Tables VII & IX, respectively, and compiled as a comparison in Table X. Inspection of Table X reveals significantly larger spiking rate uncertainties with the *sample and analyze method* than the *laboratory standard method* for all species and on both absolute and relative uncertainty bases.

Table X Comparison of Spiking Rate Uncertainties Associated with the *Laboratory Standard and Sample & Analyze Methods*

Spiking Specie, (S)	Specie Spiking Rate Uncertainty			
	Absolute Uncertainty, ± Lb S/Hr		Relative Uncertainty (RU), ±%RU	
	Laboratory Standard	*Sample & Analyze*	*Laboratory Standard*	*Sample & Analyze*
Ash	± 0.0064 Lb Ash /Hr	± 1.41 Lb Ash /Hr	± 0.045%RU	± 10%RU
Naphthalene	± 0.0119 Lb Nap/Hr	± 7.96 Lb Nap/Hr	± 0.045%RU	± 30%RU
Toluene	± 0.0323 Lb Toluene /Hr	± 20.3 Lb Toluene /Hr	± 0.045%RU	± 30%RU

Independent Assurance of Spiking Material Composition while Using the *Laboratory Standard Method*:

If there are regulatorily sensitive circumstances or other reasons that spiking material composition must be independently verified, the authors propose the following approach which would incur little or no additional cost compared to typical commercial analytical costs for GC/MS &/or ICP/CVAA analyses. The proposed approach would provide for the agency hiring a qualified, independent Professional Engineer (PE, or similar independent technically qualified individual) based near the material preparer's facility to observe the materials being prepared including all materials packages being opened, all measurement equipment being calibrated and all measurements being made and recorded, the Certificates of Analyses (CoAs) for all of the raw materials used, and the placement of a seal on all openings of the finished materials shipping containers, if required, and to obtain copies of all records related to the composition of the spiking materials including but not limited to: (1) calibration procedures for all measurement instrument/equipment, traceability of all standards used, and all applicable calibration records, (2) CoAs for all raw materials used, (3) all applicable material preparation procedures and measurement results, (4) all calculations based on the calibrations, standards, measurements, and procedures used to determine the spiking material composition, and (5) the PE's notes related to his/her observation of the materials being made, containerized, and sealed prior to shipment.

CONCLUSIONS

As a result of the information provided herein the authors have derived the following conclusions:

1. The compositional uncertainty of the two spiking materials prepared for the Case Study Trial Burn using the *laboratory standard method* as well as the impact of this compositional uncertainty on spiking rate are very modest (e.g., ± 0.045 wt% for each of the three spiking species: Ash, Naphthalene, and Toluene).

2. The *laboratory standard method* of demonstrating spiking material composition provides a much smaller uncertainty (by at least two orders of magnitude) in terms of both spiking material composition and spiking rate than is currently possible with the *sample and analyze method* due to inherent limitations/uncertainties of the current complex analytical methods. This uncertainty advantage is expected to remain even if analytical methods designed for high level samples were used, due to the very large magnitude of the uncertainty advantage compared to low level methods, and since all of the analytical method uncertainties remain with high level methods except those associated with sample dilutions.

3. Should there be sensitive regulatorily or other circumstances which make independent verification of spiking material composition mandatory, the use of an independent, technically qualified observer to confirm the details of the spiking material preparation using the *laboratory standard method* would be a logistically and economically viable alternative to the far less accurate *sample and analyze method.*

REFERENCES

1. Trial Burn Spiking Report, 2003, Confidential Client & Location.

2. Quality Assurance Project Plan, Confidential Client & Location.

3. USEPA, Region VI, Center for Combustion Science and Engineering, HWC Unit Permitting Manual, Component 2, "How to Review a QAPP", prepared by Tetra Tech, Inc., 1996

4. Private phone and email communications by one of the authors (WRS) with Julius Fulop, et. al., Philip Analytical Services, February, 2004.

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[i] As used herein **Spiking Material (M)** refers to the material which is actually spiked, i.e., a metal solution, a TiO_2 and/or metal dispersion, and/or an individual or a mixture of POHCs. **Spiking Species (S)** refers to the portion of the **Spiking Material** which is of specific interest in meeting the test objectives, i.e., individual metals, ash, individual POHCs, Cl^-, etc.

[ii] The weight of TiO_2 per drum assuming that the gross weight measurements for each of the three TiO_2 sub-batches were higher than indicated weight by an amount equal to the full uncertainty, and all net weight measurements for TiO_2 were less by the full uncertainty, which yields a quantity of TiO_2 = 91.65 + 0.06 = 91.71 Lb TiO_2/Drum.

[iii] The Lb TiO_2 /drum based on the opposite assumptions to footnote ii above, which yields TiO_2 = 91.65 - 0.06 = 91.59 Lb. TiO_2/Drum.

[iv] The term "indicated" as used within herein refers to the apparent weight or weight percent of a substance as "indicated" on the digital readout devices (digital indicators) employed in this work.

[v] The following assumptions were made in estimating the maximum Naphthalene concentration (e.g., the cumulative positive uncertainty) for a given drum:

All four Naphthalene tare weights were assumed to be smaller than the indicated weight by the full measurement uncertainty,

All four Naphthalene gross weights were assumed larger,

The Toluene tare weight (drum + Naphthalene) was assumed larger, and

The Toluene gross weight was assumed smaller.

In toto, this series of worst case assumptions results in the Naphthalene weight being 0.08 Lb larger than indicated and the Toluene weight being 0.2 Lb smaller than the indicated weights. These assumptions resulted in the maximum Naphthalene concentration. The opposite assumptions would produce the minimum Naphthalene concentration. See Table V for further explanation

Attachment V. Effect of Measurement Uncertainty on Spiking Rate Uncertainty

A. Schofield, PhD, PE, Bill, Anthony R Eicher, and Sean O'Brien, The Effect of Measurement Uncertainty on Material Composition and Spiking Rate Uncertainties, 2004 IT3 Conference Proceedings, Paper #103, Phoenix, AZ, May, 2004

THE EFFECT OF MEASUREMENT UNCERTAINTY ON SPIKING MATERIAL COMPOSITION AND SPIKING RATE UNCERTAINTIES

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ABSTRACT

It is not unusual for an agency, a client, or even a supplier of spiking materials to assert without justification that the only way to "know" the composition of a spiking material is through sampling and analysis of that spiking material. While this approach [which will be identified herein as the *sample and analyze method*] offers the advantage of determining composition independently of the spiking material supplier, it suffers the disadvantage of large measurement uncertainties resulting from inherent limitations in the analytical methods employed. However, there is another, fundamentally different, approach which is based on long standing principles of analytical chemistry and provides spiking material compositions with significantly smaller uncertainties. Conceptually, this approach is analogous to an analytical chemist preparing a laboratory standard for calibrating a sensitive analytical instrument. This approach, which will be identified herein as the *laboratory standard method* for preparing spiking materials, provides very accurate spiking specie concentrations.

Estimates of compositional uncertainty with the *laboratory standard method* developed herein are based on: (1) the test-specific details of a Case Study (e.g., a 2003 TB conducted at a private, US based HWC Unit), and (2) a series of worst case assumptions related to both the magnitude and direction of individual measurement uncertainties to produce the largest cumulative compositional uncertainty. Conversely, the assumption was made that no blatant operator mistakes were made since: (1) all measurements affecting composition were straight forward weight measurements using non-interpretive digital indicators, (2) the material preparation procedures were simple, and clear, (3) the documentation and record keeping procedures used were thorough, comprehensive, and consistently followed; and (4) the preparation procedure had built-in cross checks which included the utilization of two independent measurement observers and data recorders for most measurements. The premises on which the uncertainty analysis is based are explicitly identified, and rationales provided for their validity in the HWC Spiking context. Also, due to the small magnitude of all first-order uncertainties, second order uncertainties were ignored. Using first-order uncertainties developed for the case study example, the validity of this assumption was demonstrated.

The compositional uncertainty with the *laboratory standard method* is smaller (e.g., on the order of ± 0.1%) than is possible with the *sample and analyze method* using commercially available analytical methods. Depending on the method, matrix, and specie, the measurement uncertainty with commercially available analytical methods could vary from ± 5% up to ± 50%, assuming no uncertainty associated with sample collection and preparation.

This paper: (1) describes the spiking material preparation procedures used, (2) develops the apparent concentration of each spiking specie using the *laboratory standard method*, (3) describes the calculation procedures used to estimate uncertainty and presents the resulting estimates of uncertainty of spiking specie concentrations, (4) presents the impact of compositional uncertainty on spiking rate uncertainty, (5) estimates measurement uncertainty for the analytical methods most likely used to analyze spiking material composition in a HWC testing context, (6) presents the resulting impact on spiking rate, (7) presents a comparison of the composition and spiking rate uncertainties based on the *laboratory standard method* to those based on the *sample and analyze method*, and (8) proposes an approach for verifying spiking material composition independently of the material preparation firm should that be required in a regulatorily sensitive circumstance.

INTRODUCTION AND BACKGROUND

It is not unusual for an agency, a client, or even a supplier of spiking materials to assert that the only way to "know" the composition of a spiking material (e.g., a metal solution, an organic solution, a dispersion, and/or a "neat" POHC) is through sampling and analysis of that spiking material. While this approach [which will be identified herein as the *sample and analyze method*] offers the advantage of determining composition independently of the spiking material supplier, it suffers the disadvantage of large measurement uncertainties resulting from inherent limitations in the analytical methods employed. However, there is another, fundamentally different, approach which is based on long standing principles of analytical chemistry and provides spiking material compositions with significantly smaller uncertainties.

Conceptually, this approach is analogous to an analytical chemist preparing a laboratory standard for calibrating a sensitive analytical instrument and requires that one:

1. Know, with as much accuracy as possible, the purity of each reagent used in the preparation of a laboratory standard; especially as it relates to the chemical specie to be analyzed;

2. Use highly accurate, and carefully maintained measuring devices which are calibrated prior to use with NIST traceable standards; and

3. Maintain careful records for each step in the preparation of the laboratory standard.

This approach, which will be identified herein as the *laboratory standard method* for preparing spiking materials, provides very accurate spiking specie concentrations. The concentration uncertainty with this approach is smaller (e.g., on the order of ± 0.1%) than is possible with commercially available analytical methods (i.e., which, depending on the method, matrix, and specie could vary from ± 5% up to ± 50% without consideration of possible sample collection and preparation uncertainties).

Large concentration uncertainties are especially likely in spiking applications in which the use of SW846 and similar "low [analyte concentration] level" methods is required. As a result of large dilutions, these methods are generally not suitable for obtaining highly accurate analyses of the high analyte concentrations frequently encountered with spiking materials. Further, the *laboratory standard method* is expected to have smaller uncertainties than commercially available analytical methods which have been designed for analysis of samples with high analyte concentrations, due to the very large magnitude of the uncertainty advantage compared to low level methods, and since all of the analytical method uncertainties remain with high level methods except those associated with sample dilutions.

Estimates of compositional uncertainty with the *laboratory standard method* developed herein are based on: (1) the test-specific details of a Case Study (e.g., a 2003 TB conducted at a private, US based HWC

Unit), and (2) a series of worst case assumptions related to both the magnitude and direction of individual measurement uncertainties to produce the largest possible cumulative compositional uncertainty. Conversely, due to fact that: (1) all measurements are made with an absolute measurement method [based on the most fundamental parameter, e.g., gravity = mass, paraphrased from Reference (4)], (2) the use of non-interpretive digital indicators for all measurements, (3) the simplicity and clarity of the material preparation procedures used, (4) the use of thorough record keeping for each procedural step and measurement, (5) the experience and training of the personnel weighing the ingredients and preparing the finished spiking materials, and (6) built-in procedural cross checks including the utilization of two independent measurement observers and data recorders for most measurements; blatant operator mistakes are assumed to not be present. Also, due to the small magnitude of all first-order uncertainties, second order uncertainties were ignored.

Please note that a significant number of calculations are made in this uncertainty analysis. To avoid rounding errors and to retain the integrity of the uncertainty estimates developed herein, a relatively large number of significant figures are carried through the calculations and presented in the tables. The authors are not claiming the accuracy &/or precision in these figures that would normally be implied by the standard significant figures rules.

Engineered Spiking Solutions, Inc. (*ESS*) was retained to provide spiking materials as well as all necessary spiking equipment, and services for a Trial Burn (TB) which was conducted on a confidential, non-commercial, HWC Unit during 2003. The spiking materials used were: (1) a TiO_2 Dispersion (@ a nominal 25wt% Total Ash), and (2) a Naphthalene in Toluene Solution (@ a nominal 27wt% Naphthalene). The *laboratory standard method* was used in the case study with excellent results.

This paper: (1) describes the spiking material preparation procedures used, (2) develops the apparent concentration of each spiking specie using the *laboratory standard method*, (3) describes the calculation procedures used to estimate uncertainty and presents the resulting estimates of uncertainty of spiking specie concentrations, (4) presents the impact of compositional uncertainty on spiking rate uncertainty, (5) estimates measurement uncertainty for the analytical methods most likely used to analyze spiking material composition in a HWC testing context, (6) presents the resulting impact on spiking rate, (7) presents a comparison of the composition and spiking rate uncertainties based on the *laboratory standard method* to those based on the *sample and analyze method*, and (8) proposes an approach for verifying spiking material composition independently of the material preparation firm should that be required in a regulatorily sensitive circumstance.

Description of the "Case Study" Trial Burn

The Case Study TB consisted of two Test Conditions (TC) which were defined as follows: (1) TC #1: Maximum Waste Feed, and (2) TC #2: Minimum Temperature (DRE). The spiking materials[i] consisted of a 27% Naphthalene in Toluene Solution [Nap Sol] and a 25% TiO_2 Dispersion. The testing/spiking schedule is summarized as follows:

Test Condition	Date Conducted	Spiking With:	
		Nap Sol	Dispersion
TC #1	2003	✓	✓
TC #2	2003	✓	

The spiking function for this TB involved three spiking species[i] (e.g., Total Ash, Naphthalene, and Toluene) which were contained in two spiking materials[i] (e.g., TiO_2 Dispersion and Naphthalene in

Toluene Solution). The dispersion was used as an ash surrogate with ash contributions from both the TiO_2 (primary) and the proprietary dispersing agent (secondary). The Naphthalene in Toluene Solution spiking material contained both POHCs, e.g., Naphthalene and Toluene.

Conceptual Basis for the *Laboratory Standard Method* to Demonstrating Spiking Material Composition

The *laboratory standard method* for preparing and demonstrating the composition of spiking materials is analogous in concept to the approach employed by analytical chemists to prepare a laboratory standard for use in calibrating sensitive analytical instruments:

1. Make every effort to know, with as much accuracy as possible, the purity of each ingredient used in the preparation of a spiking material; especially as it relates to the specie being spiked, e.g. Naphthalene, Toluene, or Total Ash.

2. Have and carefully maintain a range (e.g., 1 Lb, 10 Lb, 50 Lb, 300 Lb, and 1,000 Lb) of highly accurate (Measurement Uncertainty = ± 0.01% of Full Scale Capacity, or better) weigh scales for accurately determining the quantity of each ingredient used. Use the most accurate (smallest) scale practical for a given application. Calibrate each scale with NIST traceable weight standards prior to each use.

3. Carefully record every spiking materials preparation step to facilitate documentation of the resulting spiking material composition, and QA audits.

Premises [with Supporting Rationales] on which this Uncertainty Analysis Was Based

The following premises were used as a basis for the Uncertainty Analysis provided herein. A rationale which demonstrates the validity of each premise in the HWC Testing/Spiking context is also provided:

1. **Premise:** No Chemical reactions will occur between the raw materials used to prepare a spiking material.

   Rationale: The two spiking materials used in the Case Study Trial Burn (e.g., Naphthalene in a Toluene solution, & TiO_2 in a mineral oil based dispersion) are typical of spiking materials in general, in that they well known in terms of chemistry and have been successfully used many times over a period of more than a decade. Naphthalene does not react chemically with toluene. Similarly TiO_2 and the proprietary dispersing agent are both chemically inert and furthermore would revert back to the same ash producing Ti & Si oxides in the combustion chamber if chemical reactions were to occur. Other systems, such as metal nitrates in an aqueous solution, will produce a weak nitric acid which could very well react with an unlined steel drum, will require special containers (lined or plastic drums) to ensure that such reactions do not occur.

2. **Premise:** Precipitation of spiking species out of solutions will not occur.

   Rationale: The solubility of the vast majority of spiking species (Naphthalene in Toluene, and metal salts in aqueous solutions) is well known (Merck Index, Perry's, etc.), and have been successfully used many times over a period of more than a decade. Solutions are never prepared at ≥90% of saturation (where cost has an impact, such as a Naphthalene in Toluene solution) and usually <50% of saturation (where there

is essentially no cost impact, such as aqueous solutions). Whenever there is any uncertainty, metal salts are not combined into the same solution as a means of ensuring that common ion and similar solubility effects do not bring composition in doubt.

3. **Premise:** Vapor Losses will have negligible impact on composition, or can be easily corrected for.

Rationale: All solutions are prepared in closed top drums which are kept sealed except when solute is added and mixed. Almost all spiking material solutions are aqueous solutions prepared at concentrations which are far from saturation. Thus, mixing times and associated vapor losses from the closed top drums are modest. For solutions prepared with higher vapor pressure solvents (e.g., Toluene), the quantity of vapor losses can be determined (by weight loss) and, if necessary, corrections made.

Measurement Uncertainty with Weight Measurements

All spiking material quantity measurements [which could affect the composition of the spiking materials discussed in this paper] were made using two weigh scales: (1) a 50 Lb bench scale, Model #: CQ25R33 manufactured by Ohaus Corporation, and (2) a 1,000 Lb floor scale, Model: Survivor FB2424-1000 manufactured by Rice Lake Weighing Systems. Selected (accuracy related) specifications for both of these scales are provided in Table I.

Table I Weighing Equipment Specifications

Specification	Units	Weigh Scale Manufacturer	
		Ohaus	Rice Lake
Capacity @ Full Scale (FS)	Lb (Kg)	50 (25)	1,000 (500)
Divisions[1](d)/FS			
NTEP[2]	d/FS	5,000	5,000
Non-NTEP[2]	d/FS	10,000	10,000
Lb/Division (%FS/d)			
NTEP[2]	Lb/d (%FS/d)	0.01 (0.01%)	0.02 (0.02%)
Non-NTEP[2]	Lb/d (%FS/d)	0.005 (0.005%)	0.01 (0.01%)
Non-Linearity	0.03% FS	NA	0.03% FS
Hysterises	0.02% FS	NA	0.02% FS

Footnotes:
1. The number of divisions/FS is an indication of scale sensitivity. For example, a division is the smallest weight increment discernable by the weighing system according to a given set of accuracy, calibration frequency, and environmental condition requirements.
2. NTEP is a quasi governmental organization established to regulate weights and measures used for commercial purposes. NTEP certified equipment has a conservative classification to properly reflect how measuring equipment may be used in commerce [i.e., infrequently calibrated, handled roughly, operated in a wide range of environmental conditions] while still providing acceptable accuracy. For the purposes of weighing ingredients for spiking materials with very frequent equipment maintenance & calibrations, and in controlled conditions of temperatures and humidity, the Non-NTEP division count is generally considered to be representative of scale accuracy. This observation has been confirmed by extensive pre-use and post-used calibration verifications with NIST traceable standards which consistently demonstrated deviations from the standards of $\leq$ 0.01% or equivalently d/FS $\geq$ 10,000.

PREPARATION OF SPIKING MATERIALS: TiO_2 DISPERSION

This section provides: (1) a description of the TiO_2 Dispersion preparation procedure, (2) the calculation procedure for determining Total Ash concentration and the calculated results, (3) the calculation procedure for estimating uncertainty in the apparent Total Ash concentration and the calculated results, and (4) the Certification of Composition for the TiO_2 Dispersion.

TiO_2 Dispersion Preparation Procedure (Summarized)

1. Setup and calibrate the 50 Lb ± 0.01 Lb, and 1,000 Lb ± 0.1 Lb weigh scales using NIST Traceable Weight Standards;

2. Add Mineral Seal Oil (MSO) to the Dispersion Matrix (DM) blend tank. Weigh each drum before and after the MSO transfer with the 1,000 Lb weigh scale. Record the drum gross and tare weights;

3. Weigh out the dispersion agent (DA) in four (4) batches on the 50 Lb ± 0.01 Lb weigh scale. Record the tare and gross weights;

4. Slowly add the dispersion agent to the blend tank and mix with maximum shear;

5. Weigh out the activator in five (5) batches on the 50 Lb ± 0.01 Lb weigh scale. Record the tare and gross weights;

6. Slowly add the activator to the blend tank and mix with maximum shear until the dispersing system is fully developed;

7. Drum off the DM per the Drum Weight Schedule provided. Weigh each numbered DM drum (Drum # 1-6 for the TiO_2 Dispersion) before and after adding DM and record the tare and gross weights;

8. Prepare six (6) batches (numbered 1-6) of TiO_2 for addition to the corresponding six (6) numbered drums of the TiO_2 Dispersion being prepared. Determine the quantity of TiO_2 in each batch on the basis of 0.3228 Lb TiO_2/Lb DM in the corresponding DM drum. Prepare each batch of TiO_2 in three sub-batches, (numbered as 1A, 1B, 1C; 2A, 2B,.....6B, 6C), record each tare and gross weight;

9. Slowly add each TiO_2 sub-batch to the corresponding drum of DM and shear thoroughly; and

10. Tightly seal, label and prepare each drum for shipment to the test site.

Calculation of Dispersion Matrix (DM) Composition

The DM composition data (e.g., the weights developed in procedure steps 2, 3, & 5 above) were compiled and summarized in Table II. Table II provides the DM composition information on weight (Lb), and weight percent (wt%) bases with corresponding uncertainty estimates. The term "indicated" as used herein refers to the apparent weight or weight percent of a substance as "indicated" on the digital readout devices [indicators] employed in this work.

Table II Composition of Dispersion Matrix

Constituent[1]	DM[1] #1 Composition By:					
	Weight, Lb			Weight Per Cent, Wt%		
	Target	Indicated	Uncertainty[2]	Target	Indicated	Uncertainty
MSO[1]	4,862.00	4,832.70	± 2.60[2]	94.16	94.12	± 0.0506
DA[1]	146.64	146.94	± 0.08[2]	2.84	2.8618	± 0.0016
Activator	154.91	154.98	± 0.10[2]	3.00	3.0183	± 0.0019
Total	5,163.55	5,134.62	± 2.78	100.00	100.00	± 0.0541

Footnotes:
1. **DM** = Dispersion Matrix, **MSO** = Mineral Seal Oil, & **DA** = Dispersing Agent.
2. Basis: A. Obtaining the total MSO weight involved a total of 26 individual weight measurements (e.g., gross and tare weights for 13 drums), each with an estimated measurement uncertainty of ± 0.1 Lb/weight measurement.
   B. Obtaining the total DA weight involved a total of 8 individual weight measurements (i.e., tare and gross weights for four batches of DA), each with an estimated measurement uncertainty of ± 0.01 Lb/weight measurement.
   C. Obtaining the total Activator weight involved a total of 10 individual weight measurements (i.e., tare and gross weights for five batches of activator), each with an uncertainty of ± 0.01 Lb/weight measurement.
   D. Each weight measurement is assumed to have measurement uncertainties in the direction which would produce the largest cumulative positive or negative uncertainty.

Calculation of the Total Ash Drum Concentrations

The TiO_2 Dispersion composition data from procedure steps 7 & 8 above, and Table II are summarized in Table III. Additionally, measured values for ash concentration (mass fraction ash) in the TiO_2 and the dispersion agent were provided by their respective manufacturers. These values were used to calculate the total ash content (expressed as Lb ash/Drum, and wt% ash) for each drum of finished TiO_2 Dispersion.

Estimated Uncertainty in Total Ash Concentration

The uncertainty in the total ash concentration (wt%) in a given drum is comprised of four primary components of uncertainty which were estimated as follows:

1. **The measurement uncertainty in determining the net weight of Dispersion Matrix (DM) per drum:** This measurement uncertainty is estimated as the sum of the uncertainties in the two [tare and gross] weight measurements obtained in preparation procedure (step 7 above) and is calculated as follows:

   - **DM Uncertainty** = 2 [weigh measurements] x ± 0.1 Lb DM [the uncertainty associated with each measurement].

   Thus, the net weight of the DM present in Drum #1, for example, [see Table III, column (4)], is estimated to be:

   - **DM/Drum** = 283.97 Lb ± 0.2 Lb DM/Drum.

Table III TiO_2 Dispersion, Total Ash Concentration

Drum #	Indicated Weight TiO_2 Disp Lb/Dr	DM Wt, Lb/Dr		Net Weight, Lb/Drum						Total Ash, Wt %
		Target	Indicated	TiO_2			DA		Total Ash	
				Target	Indicated	Ash[2]	Indicated	Ash[3]		
(1)[1]	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)
1	375.53	283.9	283.9	91.63	91.63	90.44	8.12	4.30	94.74	25.23[4]
2	375.53	283.9	283.9	91.63	91.63	90.44	8.12	4.30	94.74	25.23[4]
3	375.53	283.9	283.9	91.63	91.63	90.44	8.12	4.30	94.74	25.23[4]
4	375.53	283.9	283.9	91.63	91.63	90.44	8.12	4.30	94.74	25.23[4]
5	376.72	283.9	284.8	91.92	91.92	90.73	8.15	4.32	95.05	25.23[4]
6	374.87	283.9	283.4	91.47	91.47	90.28	8.11	4.30	94.58	25.23[4]
Ave	375.62	283.9	283.97	91.65	91.65	90.46	8.12	4.303	94.77	25.23

Footnotes:
1. The bracketed numbers, i.e. (1), (2) ...(11), in this row signify the Column numbers which are used in the calculation explanations to the right.
2. The TiO_2 is 98.7 wt% ash based on manufacturer's CoA.
3. The DA is 52.96 wt% ash based on manufacturer's CoA.
4. These values range from 25.2283% for Drums #1, 2, 3, & 4; to 25.2309% for Drum #5; and 25.2301% for Drum #6, for an average of 25.2290 wt % and a range of -0.0007 wt % to + 0.0019 wt %.
5. Mass fraction of DA in DM from Table II.

Information Sources:
1. Values provided in Columns (4) and (6) are based on measured weights.
2. Values provided in Columns (3) and (5) are targets provided in the detailed dispersion preparation SOP.

Calculations:
1. Weight TiO_2 Disp (Column 2) = Columns (4) + (6)
2. "Ash" content of TiO_2 (Column 7) = Column (6) x 0.987[2].
3. DA content (Column 8) = Column (4) x 0.0286[5]
4. Ash content of DA (Column 9) = Column (8) x 0.5296[3].
5. Total Ash (Column 10) = Columns (7) + (9).
6. Total Ash, wt % (Column 11) = Column (10) ÷ Column (2) x 100%.

2. **The measurement uncertainty in the ash contribution from TiO_2:** This uncertainty is estimated as the sum of:

(a) the weight measurement uncertainty in the quantity of TiO_2 added to each drum is estimated as follows:

± Ash (Lb ash/Drum) = [two weight measurements per TiO_2 sub-batch] x 3 [three sub-batches/drum] x ± 0.01 Lb [the uncertainty per weight measurement] x 0.987 [the mass fraction of TiO_2 which is ash]
= ± 0.0592 Lb ash/Drum), and

(b) the uncertainty in the Lb ash/drum due to uncertainty in the % ash in TiO_2 measurement. This uncertainty is estimated to be 98.7% ± 0.1% ash (or 0.987 ± 0.001 expressed as a mass fraction) in the ash content measurement times 91.65 Lb TiO_2/Drum [from Table III, column (6)], or:

± Ash (Lb ash/Drum) = (91.65 Lb TiO_2/Drum) x (± 0.001 Lb ash/Lb TiO_2)
= ± 0.09165 Lb ash/Drum.

The total estimated uncertainty in the mass of ash per drum from the TiO_2 is then ± 0.1509 Lb ash/Drum (e.g., ± 0.0592 Lb ash/Drum ± 0.09165 Lb ash/Drum = ± 0.1509 Lb ash/Drum).

Note: Consistent with the assumption that second-order uncertainties can be ignored, these two first-order uncertainties are simply added. The validity of this assumption is demonstrated below.

3. **The measurement uncertainty in the weight of TiO_2 per drum:** Following the logic [and the TiO_2 related math] of step 2(a) above, the weight measurement uncertainty in the quantity of TiO_2 added to each drum is estimated as follows:

   ± TiO_2 (Lb ash/Drum) = 2 [two weight measurements per TiO_2 sub-batch]
   x 3 [three sub-batches/drum]
   x ± 0.01 Lb [the uncertainty per weight measurement]

   Then, following the format of step 1 above:

   TiO_2/Drum = 91.65 Lb ± 0.06 Lb TiO_2/Drum.

4. **The uncertainty in the ash contribution from the dispersing agent:** Following the logic of step 2 above, this uncertainty is estimated as the sum of:

   (a) the uncertainty in the DA content per drum (± 0.08 Lb DA [from the fourth column from the left in Table II] divided by the number of DM drums produced in this DM lot [19 drums]) x (0.5296 Lb ash/Lb DA), or:

   ± Ash (Lb ash/Drum) = (± 0.08 Lb DA/19 Drums) x (0.5296 Lb ash/Lb DA)
   = ± 0.0022 Lb ash/Drum, and

   (b) the uncertainty in the weight loss on ignition measurement which was estimated at 52.96 % ± 0.1 % (or 0.5296 ± 0.001 expressed as a mass fraction) times 8.12 Lb DA/Drum [From Table III, column (8)]), or:

   ± Ash (Lb ash/Drum) = (± 0.001 Lb ash/Lb DA) x (8.12 Lb DA/Drum)
   = ± 0.00812 Lb ash/Drum.

   The total ash contribution from DA is ± 0.0103 Lb ash/Drum (± 0.0022 Lb ± 0.00812 Lb ash/Drum).

Uncertainty in the Total Ash Content per Drum:

The uncertainty in the total ash content per drum is then:

Ash Content Uncertainty from TiO_2	= ± 0.1509	Lb ash/Drum
Ash Content Uncertainty from DA	= ± 0.0103	Lb ash/Drum
Total Ash Content Uncertainty	= ± 0.1612	Lb ash/Drum

The uncertainty in ash concentration (expressed on a wt% basis) is estimated as follows (Drum #1 is used as an example):

$$\text{Uncertainty in wt\% Ash} = \frac{\pm\ 0.1612 \text{ Lb ash/Drum} \times 100\%}{(283.97 \pm 0.2 \text{ Lb DM/Drum}) + (91.65 \pm 0.06 \text{ Lb } TiO_2\text{/Drum})}$$

Note that wt% uncertainty is maximized when the DM weight is assumed to be the indicated weight minus the measurement uncertainty. [The smaller DM weight will minimize the denominator which in turn maximizes the wt% uncertainty.] Therefore, the maximum:

Positive Uncertainty in wt% Ash $= \frac{+0.1612 \text{ Lb ash X } 100\%}{(283.77\text{Lb} + 91.71^{ii} \text{ Lb})} = +0.0429$ wt % ash.

Negative Uncertainty in wt% Ash $= \frac{-0.1612 \text{ Lb ash X } 100\%}{(283.77\text{Lb} + 91.59^{iii} \text{ Lb})} = -0.0429$ wt % ash.

Thus, the TiO_2 Dispersion is 25.23% ± 0.0429% which was revised upward to 25.23% ± 0.045% to compensate for the minor drum to drum ash concentration difference described in Table III, footnote 4.

Certification of Composition for the TiO_2 Dispersion:

Based on this information, a Certification of Composition (CoC) for the TiO_2 Dispersion was prepared (See Fig. 1 for a highly abbreviated version of the TiO_2 Dispersion CoC).

Fig. 1 CERTIFICATE OF COMPOSITION: TiO_2 DISPERSION (Highly Abbreviated Format)

Product:	**TiO_2 DISPERSION**
Composition:	**Total Ash: 25.23 wt %[1]**

CERTIFICATION OF COMPOSITION:
I hereby certify that the composition information provided above and in the footnote is true and accurate to the best of my knowledge and belief.

Signed: _____ _____
W.R. (Bill) Schofield, PhD, PE **Date**
***ESS* Project Manager**

Footnotes:
[1] Based on an analysis of:
(a) the measurement uncertainty of weigh scales used to produce this material,
(b) the raw material composition information provided by the manufacturers, and
(c) the procedures which *ESS* used to produce this material;
I have concluded that the composition of this TiO_2 dispersion is 25.23% ± 0.045 wt% ash.

Demonstrating the Validity of the Assumption that Second-Order Uncertainties Can Be Ignored

This analysis of measurement uncertainty is partially based on the assumption that second-order uncertainties can be ignored. Using the first-order uncertainties calculated above, we can demonstrate the validity of this assumption. For example, we demonstrated above that the uncertainty in the quantity of ash from TiO_2: (1) due to weight measurement uncertainties was ± **0.0592 Lb ash/Drum**, and (2) due to ash concentration measurement uncertainty was ± **0.09165 Lb ash/Drum.** We will now calculate the second-order ash content uncertainty due to both TiO_2 weight measurement uncertainty and ash concentration uncertainty as follows:

± Ash (Lb ash/Drum) = [(± 0.0592 Lb ash/Drum)/(0.987 Lb ash/Lb TiO_2)] x (± 0.001 Lb Ash/Lb TiO_2)

= [± 0.0600 Lb TiO_2/Drum] x (± 0.001 Lb Ash/Lb TiO_2)

= ± 0.00006 Lb Ash/Drum

Obviously, an uncertainty of 6 parts in 100,000 parts is not significant even in the HWC Testing context. Similarly insignificant results would occur with other second-order uncertainties, simply due to the very small first-order uncertainties present.

PREPARATION OF SPIKING MATERIALS: NAPHTHALENE IN TOLUENE SOLUTION

This section provides: (1) a description of the Naphthalene in Toluene Solution preparation procedure, (2) the calculation procedure for determining Naphthalene and Toluene Concentrations and the calculated results, (3) the calculation procedure for estimating uncertainties in the apparent Naphthalene and Toluene Concentrations and the calculated results, and (4) the Certification of Composition for the Naphthalene in Toluene Solution.

Naphthalene in Toluene Solution Preparation Procedure (Summarized):

1. Setup and Calibrate the 50.00 Lb ± 0.01 Lb, and 1,000.0 Lb ± 0.1 Lb weigh scales using NIST Traceable Weight Standards;

2. Number fourteen (14) closed top "DOT" drums as Drum #1 through Drum #14;

3. Prepare fourteen (14) numbered batches (numbered I through 14) of 100.71 Lb of Naphthalene Flake. Weigh each batch on the 50.00 Lb bench scale as four sub-batches in sealed containers which are numbered as 1A, 1B, 1C, and 1D; through 14A, 14B, 14C, and 14D. Weigh each container before (tare weight) and after (gross weight) adding the Naphthalene and record the weights;

4. Weigh each drum and record the tare weight;

5. Add each Naphthalene sub-batch to the corresponding numbered closed top drum;

6. Weigh each drum after adding the Naphthalene and record the weight;

7. Add 272.3 Lb of Toluene to each drum and record the weight;

8. Mix the Naphthalene and Toluene contents of each drum thoroughly; and

9. Tightly seal, label and prepare each drum for shipment to the test site.

Calculation of Naphthalene Concentrations:

Table IV below provides the measured or indicated[iv] weights of each batch of Naphthalene, and the Toluene added to each drum; the estimated measurement uncertainty associated with each weigh scale reading (indication of weight); the Naphthalene purity (per the Manufacturer's Certificate of Analysis for the lot of Naphthalene used); and the calculated apparent or indicated Naphthalene concentration (wt%, assuming all weight measurements are accurate), as well as the cumulative Naphthalene concentrations uncertainty (based on the cumulative uncertainties assuming that each measurement was made with the maximum [error] measurement uncertainty and with the direction of each measurement uncertainty [error] which would result in largest increased[v] or decreased[v] concentrations, respectively, e.g., which would result in the maximum cumulative uncertainty).

Table IV Composition of Naphthalene in Toluene Solution by Drum

Nap Batch # & Drum #	Indicated Nap Weight, Lb/Batch	Scale Uncertainty[1], ± Lb	Indicated Toluene Weight, Lb/Drum	Scale Uncertainty[1], ± Lb	Nap Purity Correction, Mass Fraction	Wt% Naphthalene		
						Indicated	Min[2]	Max[2]
1	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
2	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
3	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
4	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
5	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
6	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
7	100.71	± 0.01	272.5	± 0.1	0.9985	26.944	26.914	26.974
8	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
9	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
10	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
11	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
12	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
13	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
14	100.71	± 0.01	272.3	± 0.1	0.9985	26.96	26.929	26.989
Average	100.71	± 0.01	272.31	± 0.1	0.9985	26.958	26.928	26.988

Footnotes:

1. Estimated measurement uncertainty for a single weight measurement on the weigh scale used.
2. The following assumptions were made in estimating the maximum Naphthalene concentration for a given drum:
   All four Naphthalene tare Weights were assumed to be smaller by the scale measurement uncertainty,
   All four Naphthalene gross weights were assumed larger,
   The Toluene tare weight (drum + Naphthalene) was assumed larger, and
   The Toluene gross weight was assumed smaller.

In Toto, these worst case assumptions result in the Naphthalene weight being 0.08 Lb larger than indicated weight and the Toluene weight being 0.2 Lb smaller than the indicated weights. These assumptions resulted in the maximum Naphthalene concentration. The opposite assumptions would produce the minimum Naphthalene concentration. See Table V for further explanation.

Estimated Uncertainty in the Average Naphthalene Concentration:

Table V below describes the computational method and information used to estimate the concentration uncertainty for Naphthalene in the Naphthalene in Toluene Solution.

TABLE V Calculation of the Minimum and Maximum Naphthalene Drum Concentrations Based on Worst Case Cumulative Measurement Uncertainty Assumptions

Drum #	Weight, Lb						Naphthalene Concentration, Wt %		
	Naphthalene (Purity Corrected[1])			Toluene					
	Indicated[1]	Min[3]	Max[3]	Indicated[2]	Min[4]	Max[4]	Indicated[5]	Min[6]	Max[7]
1-6, & 8-14	100.559	100.479	100.639	272.3	272.1	272.5	26.959	26.929	26.989
7	100.559	100.479	100.639	272.5	272.3	272.7	26.944	26.914	26.974
Ave	100.559	100.479	100.639	272.31	272.11	272.51	26.958	26.928	26.988
Range	100.559	100.479	100.639	272.3 -272.5	272.1 -272.3	272.5 -272.7	26.944-26.959	26.914-26.929	26.974-26.989

Summary Results: Maximum Naphthalene Concentration Range = 26.914 to 26.989 wt %
Deviation from Indicated Average Concentration = - 0.044 to + 0.031 wt % Naphthalene
[Known] Naphthalene Concentration = 26.958 ± 0.044 wt % = 26.96 wt % ± 0.045 wt % Nap.

Footnotes:

1. Indicated Naphthalene Weight [from second column from left in Table IV] x 0.9985 [Nap Purity, Mass Fraction].
2. Indicated Toluene Weight from fourth column from left in Table IV.
3. Max Nap Weights (wt} = Indicated Nap wt + (4 [sub-batches]) x {(+0.01 [positive deviation[8] of gross wt]) – (-0.01 Lb [negative deviation[8] of tare wt])} = Indicated Nap wt + 0.08 Lb.
4. Max Toluene Weight = Indicated Toluene weight + (+0.1 Lb [positive[8] deviation of gross weight]) – (-0.01 Lb [negative[8] deviation of tare weight]) = Indicated Toluene wt + 0.2 Lb.
5. Indicated Naphthalene wt % = [(Indicated Nap[9] weight)/(Indicated Nap[10] weight + Indicated Toluene[10] weight)] x 100%.
6. Min Naphthalene wt % = [Min Nap[9] wt/(Min Nap[10] wt + Max Toluene[10] wt)] x 100%.
7. Max Naphthalene wt % = [Max Nap[9] wt/(Max Nap[10] wt + Min Toluene[10] wt}] x 100%.
8. Reverse wt deviations for minimum Nap and Toluene wts.
9. Purity Corrected.
10. Not Purity Corrected.

Estimated Toluene Concentration Uncertainty

Table VI below describes the computational method and information used to estimate the concentration uncertainty for Toluene in the Naphthalene in Toluene Solution.

TABLE VI Calculation of the Minimum and Maximum Toluene Drum Concentrations Based on Worst Case Cumulative Measurement Uncertainty Assumptions

Drum #	Weight, Lb						Toluene Concentration, Wt %		
	Naphthalene (w/o Purity Corrected[1])			Toluene (with Purity Corrected[2])					
	Indicated[1]	Min[3]	Max[3]	Indicated[2]	Min[4]	Max[4]	Indicated[5]	Min[6]	Max[7]
1-6, & 8-14	100.71	100.63	100.79	272.11	271.91	272.31	72.95	72.92	72.98
7	100.71	100.63	100.79	272.31	272.11	272.51	72.964	72.934	72.994
Ave	100.71	100.63	100.79	272.1243	271.9243	272.3243	72.951	72.921	72.981
Range	100.71	100.63	100.79	272.11 -272.31	271.91 -272.11	272.31 -272.51	72.95 –72.964	72.92- 72.934	72.98– 72.994

Summary Results: Maximum Toluene Concentration Range = 72.92 to 72.994 wt %.
Deviation from Indicated Average Concentration = - 0.031 to + 0.043 wt %.
[Known] Toluene Concentration = 72.951 wt % ± 0.043 wt % = 72.95 wt % ± 0.045 wt %.

Footnotes:

1. Indicated Naphthalene Weight [from second column from left in Table III, without purity correction].
2. Indicated Toluene Weight from fourth column from left in Table III x 0.9993 [Toluene purity, mass fraction, see attached manufacturer's CoA].
3. Max Nap Weights (wt) = Indicated Nap wt + (4 [sub-batches]) x {(+0.01 [positive deviation[8] of gross wt]) – (-0.01 Lb [negative deviation[8] of tare wt])} = Indicated Nap wt + 0.08 Lb.
4. Max Toluene Weight = Indicated Toluene weight + (+0.1 Lb [positive[8] deviation of gross weight]) – (-0.01 Lb [negative[8] deviation of tare weight]) = Indicated Toluene wt + 0.2 Lb.
5. Indicated Toluene wt % = [(Indicated Toluene[9] weight)/(Indicated Toluene[10] weight + Indicated Nap[10] weight)] x 100%.
6. Min Toluene wt % = [Min Toluene[9] wt/(Max Nap[10] wt + Min Toluene[10] wt)] x 100%.
7. Max Toluene wt % = [Max Toluene[9] wt/(Min Nap[10] wt + Max Toluene[10] wt)] x 100%.
8. Reverse wt deviations for minimum Nap and Toluene wts.
9. Purity Corrected.
10. Not Purity Corrected

Certificate of Composition Naphthalene in Toluene Solution

Based on the information provided herein, the Certificate of Composition (CoC) for the Naphthalene in Toluene Solution was prepared (See Fig. 2 for a highly abbreviated version of the Naphthalene in Toluene Solution CoC).

Fig. 2 CERTIFICATE OF COMPOSITION: NAPHTHALENE IN TOLUENE SOLUTION (Highly Abbreviated Format)

Product:	**Naphthalene in Toluene Solution**
Composition:	**Naphthalene[1]: 26.96 wt %** **Toluene[1]: 72.95 wt %**

CERTIFICATION OF COMPOSITION:
I hereby certify that the composition information provided above and in the footnote is true and accurate to the best of my knowledge and belief.

Signed: _____ _____
W.R. (Bill) Schofield, PhD, PE **Date**
***ESS* Project Manager**

Footnotes:
[1] Based on an analysis of:
(a) the measurement uncertainty of the weigh scales used to produce this material,
(b) the Naphthalene and Toluene manufacturers' Certifications of Analysis, and
(c) the procedures which were used to produce this material,
I have concluded that the composition of the Naphthalene in Toluene Solution is:
(a) Naphthalene = 26.96 wt % ± 0.045 wt %, and
(b) Toluene = 72.95 wt % ± 0.045 wt %.

IMPACT OF COMPOSITION UNCERTAINTY ON ABSOLUTE AND RELATIVE SPIKING RATE UNCERTAINTY

The impact of compositional uncertainty discussed above on the Species (S) spiking rate uncertainty was calculated on two bases:

1. Absolute Species (S) Spiking Rate Uncertainty, ± Lb S/Hr, and

2. Relative Species Spiking Rate Uncertainty [uncertainty expressed as a % of the indicated spiking rate, ± %RU].

The results are presented in Table VII and summarized as follows:

Spiking Specie (S)	Spiking Rate, Lb S/Hr	Specie Spiking Rate Uncertainty:	
		Absolute Uncertainty, ±Lb S/Hr	Relative Uncertainty, ±% RU
Ash	14.12	± 0.0064 Lb Ash/Hr	± 0.045% RU
Naphthalene	26.52	± 0.0119 Lb Nap/Hr	± 0.045% RU
Toluene	67.54	± 0.0323 Lb Toluene/Hr	± 0.045% RU

Table VII Effect of Composition Uncertainty Associated with the *Laboratory Standard Method* on Specie Spiking Rate Uncertainty

Spiking Specie, (S)	Effect of Composition Uncertainty on:						
	Mass/Run:				Apparent Spiking Rate, Lb S/Hr	Absolute Specie Spiking Rate Uncertainty, ± Lb S/Hr	Relative Specie Spiking Rate Uncertainty, ± % RU
	Material		Specie				
	± Lb	± %	± Lb	±%			
Ash[1]	0.2	0.18	0.05	0.18	14.12	± 0.0064 Lb/Hr	± 0.045 %RU
Nap[2]	0.2	0.06	0.06	0.06	26.52	± 0.0119 Lb/Hr	± 0.045 %RU
Toluene[3]	0.2	0.08	0.15	0.08	67.54	± 0.0323 Lb/Hr	± 0.045 %RU

1. Total Ash has an indicated composition of 25.23 wt % ± 0.045 wt % in 110.49 Lb TiO_2 Dispersion/Run (Table III)
2. Naphthalene has an indicated composition of 26.96 wt % ± 0.045 wt % in 317.96 Lb Nap Sol/Run (Table V)
3. Toluene has an indicated composition of 72.95 wt % ± 0.045 w % in 246.88 Lb Nap Solution/Run (Table VI)

Inspection of these results indicates that the compositional uncertainty associated with the *laboratory standard method* of demonstrating spiking material composition resulted in very modest spiking rate uncertainties whether on an absolute and relative uncertainty basis.

COMPARISON OF *Laboratory Standard Method* AND *Sample and Analyze Method* UNCERTAINTIES

In order to complete this analysis by comparing the uncertainties associated with the *laboratory standard method* to the corresponding uncertainties associated with the *sample and analyze method*, it is first necessary to estimate the measurement uncertainties associated with the analytical methods (SW846 or similar methods) which are most likely to be used to determine the composition of spiking materials in a HWC Test context.

Three approaches were utilized to estimate measurement uncertainties of the applicable SW846 (& ASTM) Methods:

1. Reviewing a recent, Agency approved QAPP for guidance using the acceptable analyte recovery range for a given method in duplicate spiked samples,

2. Reviewing Agency Guidance, specifically QA Objectives for method accuracy (defined for a given method as the acceptable analyte recovery range in duplicate spiked samples), and

3. Polling Analytical/Trial Burn Experts for opinions based on experience.

Table VIII summarizes the results of that effort.

Table VIII Estimated Measurement Uncertainties for Selected Analytical Methods

Spiking:		Analytical Method[1]	Source of Method Uncertainty Estimates:		
Specie	Material		Recent QAPP(2)	Guidance(3)[2]	Expert Opinion(4)[3]
Ash	TiO_2 Dispersion	ASTM D-482	± 10 %	± 25 %	NA
Metals	Dispersion or Solution	6010 & 7470	± 30 %	± 30 %	6 - 41 %
Naphthalene	Nap & Toluene Solution	8270	-90 to -54,+50 %	± 50 %	6 - 40 %
Toluene	Nap & Toluene Solution	8260	-50, +30 %	± 50 %	10 - 30 %

Footnotes:
1. SW846 unless otherwise noted.
2. Reference (3), QA Objectives for TB, Table III-1, Process Samples.
3. Reference (4), based on low [analyte concentration] level sample analysis.

Inspection of Table VIII prompts three significant observations:

1. There is a relatively wide range within the measurement uncertainty estimates;

2. The expert opinion estimates of measurement uncertainty are based on low [analyte concentration] level analyses and, as such, probably under state the measurement uncertainty which would be present with analysis of high level spiking material samples. Conversely, the QAPP and Guidance estimates are based on a wide range of analytical laboratories and, as a result, probably over state uncertainties associated with analytical results from a laboratory with a strong QA/QC Program; and

3. The level of measurement uncertainty associated with each of these analytical methods (*sample and analyze method*) is at least two (2) orders of magnitude larger than the measurement uncertainty associated with the *laboratory standard method* (e.g., ± 5% vs. ± 0.045%).

As a result of the last observation, no further effort was invested to refine the measurement uncertainty for the analytical methods. As a result of the first two observations, the following method specific measurement uncertainties were, somewhat arbitrarily, selected:

Method	Analyte	Measurement Uncertainty, ±%
ASTM D-482	Ash	± 10%
SW846 8270	Naphthalene	± 30%
SW846 8260	Toluene	± 30%

These measurement uncertainty estimates were used to calculate the absolute and relative spiking rate uncertainties on the same case study basis and with the results were summarized in Table IX, below.

Table IX Effect of Compositional Uncertainty Associated with the *Sample & Analyze Method* on Specie Spiking Rate

Spiking Specie, (S)	Apparent Spiking Rate, Lb S/Hr	Est'ed Measurement Uncertainty, ± %	Absolute Spiking Rate Uncertainty, ± Lb S/Hr	Relative Spiking Rate Uncertainty, ± %RU
Ash	14.12 Lb/Hr	± 10%	± 1.41 Lb Ash/Hr	± 10%RU
Naphthalene	26.52 Lb/Hr	± 30%	± 7.96 Lb Nap/Hr	± 30%RU
Toluene	67.54 Lb/Hr	± 30%	± 20.3 Lb Toluene/Hr	± 30%RU

The absolute and relative specie spiking rate uncertainties based on the *laboratory standard method* and the *sample and analyze method* were then taken from Tables VII & IX, respectively, and compiled as a comparison in Table X. Inspection of Table X reveals significantly larger spiking rate uncertainties with the *sample and analyze method* than the *laboratory standard method* for all species and on both absolute and relative uncertainty bases.

Table X Comparison of Spiking Rate Uncertainties Associated with the *Laboratory Standard and Sample & Analyze Methods*

Spiking Specie, (S)	Specie Spiking Rate Uncertainty			
	Absolute Uncertainty, ± Lb S/Hr		Relative Uncertainty (RU), ±%RU	
	Laboratory Standard	*Sample & Analyze*	*Laboratory Standard*	*Sample & Analyze*
Ash	± 0.0064 Lb Ash /Hr	± 1.41 Lb Ash /Hr	± 0.045%RU	± 10%RU
Naphthalene	± 0.0119 Lb Nap/Hr	± 7.96 Lb Nap/Hr	± 0.045%RU	± 30%RU
Toluene	± 0.0323 Lb Toluene /Hr	± 20.3 Lb Toluene /Hr	± 0.045%RU	± 30%RU

Independent Assurance of Spiking Material Composition while Using the *Laboratory Standard Method*:

If there are regulatorily sensitive circumstances or other reasons that spiking material composition must be independently verified, the authors propose the following approach which would incur little or no additional cost compared to typical commercial analytical costs for GC/MS &/or ICP/CVAA analyses. The proposed approach would provide for the agency hiring a qualified, independent Professional Engineer (PE, or similar independent technically qualified individual) based near the material preparer's facility to observe the materials being prepared including all materials packages being opened, all measurement equipment being calibrated and all measurements being made and recorded, the Certificates of Analyses (CoAs) for all of the raw materials used, and the placement of a seal on all openings of the finished materials shipping containers, if required, and to obtain copies of all records related to the composition of the spiking materials including but not limited to: (1) calibration procedures for all measurement instrument/equipment, traceability of all standards used, and all applicable calibration records, (2) CoAs for all raw materials used, (3) all applicable material preparation procedures and measurement results, (4) all calculations based on the calibrations, standards, measurements, and procedures used to determine the spiking material composition, and (5) the PE's notes related to his/her observation of the materials being made, containerized, and sealed prior to shipment.

CONCLUSIONS

As a result of the information provided herein the authors have derived the following conclusions:

1. The compositional uncertainty of the two spiking materials prepared for the Case Study Trial Burn using the *laboratory standard method* as well as the impact of this compositional uncertainty on spiking rate are very modest (e.g., ± 0.045 wt% for each of the three spiking species: Ash, Naphthalene, and Toluene).

2. The *laboratory standard method* of demonstrating spiking material composition provides a much smaller uncertainty (by at least two orders of magnitude) in terms of both spiking material composition and spiking rate than is currently possible with the *sample and analyze method* due to inherent limitations/uncertainties of the current complex analytical methods. This uncertainty advantage is expected to remain even if analytical methods designed for high level samples were used, due to the very large magnitude of the uncertainty advantage compared to low level methods, and since all of the analytical method uncertainties remain with high level methods except those associated with sample dilutions.

3. Should there be sensitive regulatorily or other circumstances which make independent verification of spiking material composition mandatory, the use of an independent, technically qualified observer to confirm the details of the spiking material preparation using the *laboratory standard method* would be a logistically and economically viable alternative to the far less accurate *sample and analyze method.*

REFERENCES

1. Trial Burn Spiking Report, 2003, Confidential Client & Location.

2. Quality Assurance Project Plan, Confidential Client & Location.

3. USEPA, Region VI, Center for Combustion Science and Engineering, HWC Unit Permitting Manual, Component 2, "How to Review a QAPP", prepared by Tetra Tech, Inc., 1996

4. Private phone and email communications by one of the authors (WRS) with Julius Fulop, et. al., Philip Analytical Services, February, 2004.

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[i] As used herein **Spiking Material (M)** refers to the material which is actually spiked, i.e., a metal solution, a TiO_2 and/or metal dispersion, and/or an individual or a mixture of POHCs. **Spiking Species (S)** refers to the portion of the **Spiking Material** which is of specific interest in meeting the test objectives, i.e., individual metals, ash, individual POHCs, Cl^-, etc.

[ii] The weight of TiO_2 per drum assuming that the gross weight measurements for each of the three TiO_2 sub-batches were higher than indicated weight by an amount equal to the full uncertainty, and all net weight measurements for TiO_2 were less by the full uncertainty, which yields a quantity of TiO_2 = 91.65 + 0.06 = 91.71 Lb TiO_2/Drum.

[iii] The Lb TiO_2 /drum based on the opposite assumptions to footnote ii above, which yields TiO_2 = 91.65 - 0.06 = 91.59 Lb. TiO_2/Drum.

[iv] The term "indicated" as used within herein refers to the apparent weight or weight percent of a substance as "indicated" on the digital readout devices (digital indicators) employed in this work.

[v] The following assumptions were made in estimating the maximum Naphthalene concentration (e.g., the cumulative positive uncertainty) for a given drum:

All four Naphthalene tare weights were assumed to be smaller than the indicated weight by the full measurement uncertainty,

All four Naphthalene gross weights were assumed larger,

The Toluene tare weight (drum + Naphthalene) was assumed larger, and

The Toluene gross weight was assumed smaller.

In toto, this series of worst case assumptions results in the Naphthalene weight being 0.08 Lb larger than indicated and the Toluene weight being 0.2 Lb smaller than the indicated weights. These assumptions resulted in the maximum Naphthalene concentration. The opposite assumptions would produce the minimum Naphthalene concentration. See Table V for further explanation

Attachment V. Effect of Measurement Uncertainty on Spiking Rate Uncertainty

B. Schofield, PhD, PE, Bill, Anthony R Eicher, and Sean O'Brien, The Effect of Measurement Uncertainty on Field Spiking Rate and Overall Specie Spiking Rate Uncertainties, 2004 IT3 Conference Proceedings, Paper #102, Phoenix, AZ, May, 2004

THE EFFECT OF MEASUREMENT UNCERTAINTY ON FIELD SPIKING RATE AND OVERALL SPECIE SPIKING RATE UNCERTAINTIES

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ABSTRACT

Objectives

The objectives of this paper are:

1. To develop estimates for Measurement Uncertainty on a common basis for the two primary field methods of measuring spiking rate, e.g., weigh cell and mass flow meter technologies;
2. To determine the impact of these measurement uncertainties on field spiking rate and overall spiking rate uncertainties; and
3. To compare the combined spiking rate uncertainties due to measurement uncertainties associated with: (a) spiking material composition, (b) field spiking rate measurement, and (c) spiking material composition plus field spiking rate measurements.

Methodology

Estimates of field spiking rate uncertainties are developed on the basis of:

- Case Study Basis for Calculations: The test-specific details of a 2003 TB [conducted at a private, US based HWC Unit] are used as a Case Study basis for preparing quantitative comparisons on a consistent basis.

- The two field spiking methods and associated modes of operation considered in this uncertainty analysis were:
  - Weight loss versus time method with manual operation, and
  - Mass flow meter method with computer control.

- The Following Assumptions Were Made in Estimating and Propagating Uncertainties:
  - For both spiking methods:
    - No undetected operator mistakes, equipment/software mal-functions, and/or data reduction/reporting errors have occurred,
    - Second-order uncertainties are not significant, and
    - All spiking materials are uniform in composition throughout the test.

- For the weight loss versus time method:
  - Measurement uncertainty can be conservatively estimated based on a series of worst case conditions concerning both the magnitude and direction of individual measurement uncertainties, and
  - Indeterminative errors are adequately addressed with the conservative approach used to estimate determinative weight measurement uncertainty.
- For the mass flow meter method:
  - The mass flow meter manufacturer's published specification for equipment accuracy is an appropriate estimate of field measurement uncertainty, and
  - The function (accuracy) of the mass flow meter sensor is not adversely affected by corrosion, erosion, and/or uneven spiking material deposition onto the interior surfaces of the sensor tube.

- The compositional uncertainty estimates from a companion paper are combined with the field spiking rate uncertainties developed herein to produce the overall spiking system uncertainties, as follows:

Total System Uncertainty	=	Compositional Uncertainties with the:	+	Spiking Rate Uncertainties with the:
System #1 Uncertainty	=	Laboratory Standard Method	+	Weight Loss versus Time Method
System #2 Uncertainty	=	Sample and Analyze Method	+	Mass Flow Meter Method

Results

Uncertainty comparisons are made for the two systems on three bases:

- Overall spiking system uncertainties associated with spiking material composition uncertainty alone,
- Overall spiking system uncertainties associated with field spiking rate uncertainty alone, and
- Overall spiking system uncertainties due to the combined impact of both composition and field spiking rate uncertainties.

INTRODUCTION AND BACKGROUND

A great deal of effort has been expended over the last decade to investigate, understand, and improve the Quality Assurance (QA) aspects of the sampling and analytical methods used in Hazardous Waste Combustion (HWC) Risk Burns, Trial Burns, and HWC MACT Comprehensive Performance Tests. To date a comparable effort has not been made concerning the spiking function in these same tests. Additionally, conflicting information is being provided by proponents of the two most widely used methods of demonstrating spiking material composition and of measuring field spiking rates.

Objectives

The objectives of this paper are threefold:

1. To develop estimates for Measurement Uncertainty on a common basis for the two primary field methods of measuring spiking rate, e.g., weigh cell and mass flow meter technologies;
2. To determine the impact of these measurement uncertainties on field spiking rate and overall spiking rate uncertainties; and
3. To compare the spiking rate uncertainties due to measurement uncertainties associated with: (a) spiking material composition, (b) field spiking rate measurement and (c) spiking material composition plus field spiking rate measurements.

This paper also examines the effect of measurement uncertainty, imperfect knowledge, and/or error at each step of the spiking function from the point of setting a target spiking rate in a test plan, through spiking material design and preparation, the on-site spiking rate measurement and data collection functions, and the ultimate reporting of spiking rate results. Each major type of deviation, error, and/or uncertainty in the spiking function is identified and discussed in the context of the facility owners test objectives and applicable regulatory requirements.

Methodology: Estimating the Effect of Measurement Uncertainty

The effect of measurement uncertainty on field spiking rate and overall spiking system uncertainties are estimated on the following basis:

- **Case Study** basis for comparisons: The test-specific details of a 2003 TB [conducted at a private, US based HWC Unit] are used herein as a Case Study for preparing quantitative comparisons on a consistent basis;

- The **Two Field Spiking Methods** and associated **Modes of Operation** are used as the primary subject of this uncertainty analysis:
  - Weight loss versus time method with manual operation, and
  - Mass flow meter method with computer control.

  [While **ESS** currently deploys computer based technology for spiking system monitoring, feedback control, data acquisition, archiving, and output; and both mass flow meter and weigh cell technologies for measuring field spiking rates; the data presented herein were obtained prior to the mass flow meter and computer control technology becoming operationally available.]

- The following **Assumptions** were made in estimating and propagating uncertainties:
  - **For both spiking methods:**
    - No undetected operator mistakes, equipment/software mal-functions and/or data reduction errors have occurred,
    - Second-order uncertainties are not significant:
      [Because of the very small magnitude of all first-order uncertainties, no second order uncertainties are considered. The validity of this assumption is demonstrated within the companion paper (Ref.2), using first-order uncertainties estimates developed for the case study example], and
    - All spiking materials were uniform in composition throughout the test:
      [The two spiking materials used in the Case Study (e.g., Naphthalene in a Toluene solution, & TiO_2 in a mineral oil based dispersion) are typical of spiking materials, in that they well known in terms of chemistry and have been successfully used many times over a period of more than a decade. The solubility of Naphthalene is known and the Naphthalene in Toluene spiking material is prepared as an unsaturated solution.

      While there are reports that some plating or deposition of TiO_2 onto the inner surfaces of tubing can occur, this phenomena would largely occur during the initial equipment conditioning (pre-test) phase, and the mass of TiO_2 which could plate out in this case prior to blocking the relatively short, small diameter (1/2" ID) tubing is very small (<< 0.1 Lb) in comparison to the total quantity of TiO_2 spiked during a given run (> 100 Lb).]
  - **For the weight loss versus time method:**
    - The impact of weight measurement uncertainty on field spiking rate uncertainty can be estimated based on a series of worst case assumptions related to both the magnitude and direction of individual measurement uncertainties.

[The maximum measurement uncertainty together with the direction of each measurement uncertainty which would produce the largest cumulative field spiking rate uncertainty are used in all uncertainty propagation calculations. Specifically, weight measurement uncertainty is estimated on the basis of a large determinative uncertainty (**± U Lb**) based on the equipment vendor's specification of measurement uncertainty, typically, U = 0.01% of the full scale capacity of the equipment used). This uncertainty is carried throughout the uncertainty propagation calculations as if it were part of the weight (**W**) value (i.e., **W** was replaced with **W ± U Lb**). Once the calculation was completed, the + or - uncertainty directions for U which would result in the largest cumulative spiking rate uncertainty are selected.], and

- Indeterminative errors are adequately considered with the conservative approach used to estimate determinative weight measurement uncertainty.
  [The magnitude of determinative uncertainty (**± U**) is chosen to be sufficiently large and the weigh scale indicator setting is set such that random variation in the weight measurements (indeterminative uncertainty) is hidden in the decimal places which are not displayed. As a result, indeterminative uncertainty is not expected have a material impact on the results of this analysis.]

- For the **mass flow meter method:**
  - The equipment manufacturer's published specification for sensor accuracy (Refs: 3, 4, 5) can be used without modification as the mass flow meter field measurement uncertainty, and
  - The function (accuracy) of the mass flow meter sensor is not adversely affected by corrosion, erosion and/or uneven spiking material deposition onto the interior surfaces of the sensor tube (Refs: 3, 4, 5).

- Significant figures:
  - A large number of calculations are made in this uncertainty analysis, many with extremely small numbers. To avoid rounding errors and to retain the integrity of the uncertainty estimates developed herein, a relatively large number of significant figures are carried through the calculations and presented in the tables.
  - The authors do not claim the accuracy and/or precision in these figures that would normally be implied by the standard significant figures rules.

- The compositional uncertainty estimates from a companion paper are combined with the field spiking rate uncertainties developed herein to produce the overall spiking system uncertainties, as follows:

Total System Uncertain	=	Compositional Uncertainties with the:	+	Spiking Rate Uncertainties with the:
System #1 Uncertainty	=	*Laboratory* Standard Method	+	Weight Loss versus Time Method
System #2 Uncertainty	=	Sample and Analyze Method	+	Mass Flow Meter Method

[The uncertainties associated with composition and field spiking rate are combined in this manner to reflect the standard practices of representative spiking firms within the spiking industry.]

Results

Uncertainty comparisons are made for these two, frequently used spiking systems on three bases:

- Overall spiking system uncertainties associated with spiking material composition uncertainty alone,
- Overall spiking system uncertainties associated with field spiking rate uncertainty alone, and

- Overall spiking system uncertainties due to the combined impact of both composition and field spiking rate uncertainties.

Description of the "Case Study" Trial Burn

The Case Study TB was conducted on a confidential, non-commercial, HWC Unit during 2003, and consisted of two Test Conditions (TC) which were defined as follows: (1) TC #1: Maximum Waste Feed, and (2) TC #2: Minimum Temperature (DRE). The spiking materials[1] consisted of a Naphthalene in Toluene Solution (@ a nominal 27 wt % Naphthalene) and a TiO_2 Dispersion (@ a nominal 25 wt % Total Ash). The testing/spiking schedule is summarized as follows:

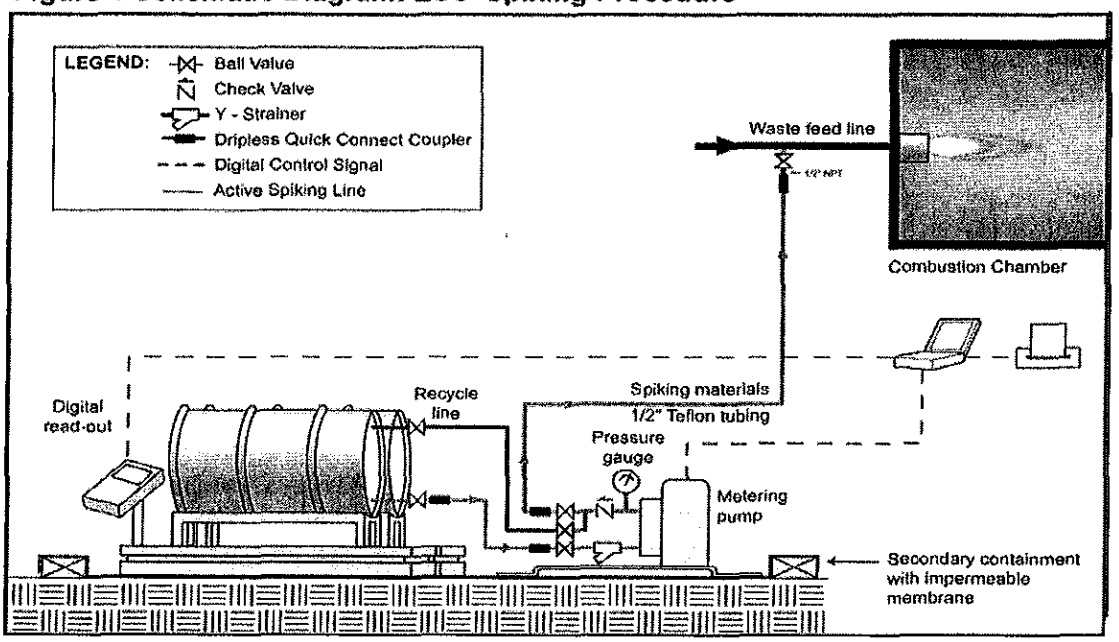
Test Condition	Date Conducted	Spiking With:	
		Nap Sol	Dispersion
TC #1	2003	✓	✓
TC #2	2003	✓	

The spiking function for this TB involved three spiking species[i] (e.g., Total Ash, Naphthalene, and Toluene) which were contained in two spiking materials[1] (e.g., TiO_2 Dispersion, and Naphthalene in Toluene Solution). The dispersion was used as an ash surrogate with ash contributions from both the TiO_2 (primary) and the proprietary dispersing agent (secondary). The Naphthalene in Toluene Solution spiking material contained both POHCs, e.g., Naphthalene and Toluene.

Equipment Setup and Operation for the Weight Loss Versus Time Spiking Approach

Typically a drum (or tote tank or gas cylinder) of spiking material is placed on an appropriately sized weigh scale (the smallest [most accurate] scale which can weigh the full container of spiking material) and connected with SS, dripless, quick-connect fittings to the metering pump, which is similarly connected to the waste feed line. As material is pumped out of the drum (and into the waste feed line) the mass on the weigh scale drops (see Figure 1). The weight of spiking material remaining on the weigh scale is recorded and the spiking rate calculated frequently based on the rate of change of mass on the weigh scale.

Figure 1 Schematic Diagram: ESS' Spiking Procedure



The weigh scales[ii] (See Table 1) are calibrated before the test and the calibration is verified on-site immediately before and after the test with NIST traceable standards[iii] The pre- and post-test calibration verifications generally indicate no deviations (e.g., $\pm$ 0.0 Lb deviation) for most if not all points over the full calibration range (typically, 0.0 – 650.0 Lb).

Table I Weighing Equipment Accuracy Related Specifications

Specification	Units	Weigh Scale Manufacturer
		Rice Lake
Capacity @ Full Scale(FS)	Lb (Kg)	1,000 (500)
Divisions[1] for Full Scale, (d)/FS:		
NTEP[2]	d/FS	5,000
Non-NTEP[2]	d/FS	10,000
Lb/Division (%FS/d):		
NTEP[2]	Lb/d (%FS/d)	0.02 (0.02%)
Non-NTEP[2]	Lb/d (%FS/d)	0.01 (0.01%)
Performance Specifications:		
Non-Linearity	0.03% FS	0.03% FS
Hysterises	0.02% FS	0.02% FS

Footnotes: 1. The number of divisions/FS is an indication of scale sensitivity. For example, a division is the smallest weight increment discernable by the weighing system according to a given set of accuracy, calibration frequency, and environmental condition requirements.

2. NTEP is a quasi governmental organization established to regulate weights and measures used for commercial purposes. NTEP certified equipment has a conservative classification to properly reflect how measuring equipment may be used in commerce [i.e., infrequently calibrated, handled roughly, operated in a wide range of environmental conditions] while still providing acceptable accuracy. For the purposes of weighing ingredients for spiking materials with very frequent equipment maintenance & calibrations, and in controlled conditions of temperatures and humidity, the Non-NTEP division count is generally considered to be representative of scale accuracy. This observation has been confirmed by extensive pre-use and post-used calibration verifications with NIST traceable standards which consistently demonstrated deviations from the standards of $\leq$ 0.01% or equivalently d/FS $\geq$ 10,000.

Procedures for Calculating Spiking Rate

Standard Spiking Rate Calculation Procedure

The standard procedure for calculating the spiking rate for a given run with the Weight Loss Versus Time Method is to include spiking rate data for the time period beginning when the stack sampling probe is first introduced into the stack [or from the beginning of sampling with the first VOST tube pair], through port changes [or VOST tube replacement] until the probe is removed from the stack at the end of that run [or until sampling with the last VOST tube pair is completed] unless some abnormal event occurs such as an extended combustor operational problem, or the rare sampling train leak check failure. Because metering pumps (which maintain essentially constant feed rates throughout the run[iv]) are used (instead of simple transfer pumps which are susceptible to throughput swings in response to waste feed line pressure changes), this approach has a number of advantages (e.g., simplified data reduction, and reduced

measurement uncertainty [see discussion below]), and no disadvantages. If problems were to occur which might bring operating, sampling, or spiking performance data into question, then the spiking data from that period would be excluded from the spiking rate calculations.

With this procedure, calculation of the spiking rate for a given run typically requires the recording and use of two weight measurements, i.e., the beginning mass and the final mass. Thus, weighing systems measurement uncertainty could occur twice.

A conservative estimate of measurement uncertainty in the mass of spiked material per run would assume that: (1) the weight measurement for the beginning mass measurement and for the ending mass reading are each in "error", (2) the "error" is equal to the full measurement uncertainty, and (3) the two "errors" are in opposite directions (so that the measurement uncertainties would be additive and would not cancel each other).

If one were to assume a measurement uncertainty of ± 0.1 Lb associated with each weight measurement reading, then the maximum measurement uncertainty for the total mass of spiking material (M) fed during a run would be ± 0.2 Lb M/Run.

For a run with 300 Lbs M/Run, the relative uncertainty (RU, expressed as a per cent) would be:

RU = (± 0.2 Lb M/Run)/(300 Lb M/Run) X 100 % RU
= ± 0.0667 % RU, a very small relative uncertainty.

For a run with 100 Lbs M/Run, the maximum measurement uncertainty for the total mass of spiking material fed during a run would remain ± 0.2 Lb M/Run, but the relative uncertainty would be:

RU = (± 0.2 Lb M/Run)/(100 Lb M/Run) X 100 % RU
= ± 0.2 % RU, still a very small relative uncertainty.

Similarly, if the spiking material were to contain the spiking specie (S) at a 20 wt% concentration, then the corresponding absolute measurement uncertainty would be ± 0.04 Lb S/Run, and the corresponding RU values for specie uncertainties would not change.

A More Conservative Spiking Rate Calculation Procedure

If, however, one were to decide to use only spiking data during test periods when spiking and stack sampling for that specie were both occurring, the spiking rate calculations could involved two or more separate spiking periods during each run. As before, each sampling period (Sx Period) required two weight measurements (at the beginning and the end of each period), each with its own measurement uncertainty.

For a run with four sampling periods and 300 Lbs M/Run, the maximum measurement uncertainty for the total mass of spiking material fed during a run would be ± 0.8 Lb M/Run and the relative uncertainty would be:

RU = (± 0.2 Lb M/Sx Period) X (4 Sx Periods/Run)/(300 Lb M/Run)X100 % RU
= (± 0.8 Lb M/Run)/(300 Lb M/Run) X 100 % RU
= ± 0.2667 % RU, a very modest relative uncertainty.

And for a run with four sampling periods and 100 Lbs M/Run, the maximum measurement uncertainty for the total mass of spiking material fed during a run would remain ± 0.8 Lb M/Run, but the relative uncertainty would be:

RE = (± 0.2 Lb M/Sx Period) X (4 Sx Periods/Run)/(100 Lb M/Run)X100 % RU
= (± 0.8 Lb M/Run)/(100 Lb M/Run) X 100 % RU
= ± 0.8 % RU, still a modest relative uncertainty.

Similarly, if the spiking material were to contain the spiking specie at 20 wt %, then the maximum measurement uncertainty for the total mass of spiking specie fed during a run would be ± 0.16 Lb S/Run and the corresponding RU values for specie uncertainties would not change.

Additional quantitative analyses of the effect of measurement uncertainty on field spiking rate based on the Case Study TB are provided below.

FIELD SPIKING RATE RESULTS

The Spiking Log Sheets completed during the Case Study TB were used together with the Certificates of Composition to calculate the specie spiking rates using both the standard and the more conservative procedures described above.

Note: During the Case Study 2003 TB, each spiking specie (Ash/PM, Naphthalene, and Toluene) was sampled using a different sampling method and over different sampling periods.

The resulting field spiking rate results for Ash, Naphthalene, and Toluene are presented in Tables II, III, and IV, respectively.

Total Ash Spiking Rate Results

Table II provides the average TiO_2 Dispersion, and concentration corrected Total Ash spiking rates for each of the three TC #1 runs, as well as for TC #1 in toto.

Table II Average TiO_2 Dispersion (Spiking Material[1]) and Total Ash (Spiking Specie[1]) Spiking Rates by Run for TC #1

TC#1/ Run#	Mass Fraction				Disp Spiking Rate[3], lb/min	Ash Spiking Rate[4]:	
	$[TiO_2]^2$	TiO_2 Purity[2]	Stoich. Content[2]	[Ash][2]		lb/min	lb/hour
Run #1	0.244	98.7	1.0477	**0.2523**	**0.8752**	**0.221**	**13.25**
Run #2	0.244	98.7	1.0477	**0.2523**	**0.9726**	**0.245**	**14.72**
Run #3	0.244	98.7	1.0477	**0.2523**	**0.9508**	**0.240**	**14.39**
TC #1 Ave	**0.244**	**98.7**	**1.0477**	**0.2523**	**0.9329**	**0.235**	**14.12**

Footnotes:

1. As used throughout this paper, **Spiking Material (M)** refers to the material as it is actually spiked, i.e., a Naphthalene in Toluene Solution, and/or a TiO_2 dispersion. **Spiking Species (S)** refers to the portion of the **Spiking Material** which is of specific interest in meeting the test objectives, i.e., Naphthalene, toluene, ash, POHC, Cl^-, etc.
2. **Concentration** refers to the concentration of the compound of interest in the **Spiking Material**, for example Nap in the Naphthalene in Toluene Solution assuming 100% purity. **Purity** refers to the assay, or purity of the Naphthalene, for example, used to make up the solution to the desired concentration. **Stoich. Content** refers to the stoichiometric content of the specie of interest in the compound, for example the Cl^- content in Perc or metal content in the metal compound. **[Specie]** indicates the specie concentration (usually expressed as Lb Specie/Lb Material, or mass fraction) and is defined as:
   [Specie] = Concentration x Purity x Stoich. Content. **[Specie]** is used to convert the **Spiking Material** spiking rate to the corresponding **Spiking Specie** spiking rate. Usually, all four of the "correction" terms are expressed as mass fractions.
3. Without Correction for **[Specie]**. Calculated from field spiking data.
4. With Correction for **[Specie]**

Naphthalene Spiking Rate Results

Table III provides the average Naphthalene in Toluene Solution spiking rates as well as the concentration and purity corrected Naphthalene spiking results for: (1) each of the six TC #1 and TC #2 runs, (2) each of the two TCs, and (3) the overall trial burn.

Table III Average Naphthalene Solution (Spiking Material[1]), and Naphthalene (Spiking Specie[1]) Spiking Rate Results by Run and TC.

TC#/ Run#	Correction Factors, Mass Fraction				Nap Sol Spiking Rate[1], lb/min	Nap Spiking Rate[1]	
	Nap Concentration[1]	Nap Purity[1]	Stoich. Content[1]	[Nap][1]		lb/min	lb/hr
TC#1/Run#1	0.2700	0.9985	1.000	**0.2696**	**1.7546**	**0.473**	**28.38**
TC#1/Run#2	0.2700	0.9985	1.000	**0.2696**	**1.8538**	**0.500**	**29.98**
TC#1/Run#3	0.2700	0.9985	1.000	**0.2696**	**1.6068**	**0.433[2]**	**25.99[2]**
TC#1 Ave	**0.2700**	**0.9985**	**1.000**	**0.2696**	**1.7384**	**0.469**	**28.12**
TC#2/Run#1	0.2700	0.9985	1.000	**0.2696**	**1.5416**	**0.4156[2]**	**24.94[2]**
TC#2/Run#2	0.2700	0.9985	1.000	**0.2696**	**1.5479**	**0.4173[2]**	**25.04[2]**
TC#2/Run#3	0.2700	0.9985	1.000	**0.2696**	**1.5321**	**0.4130[2]**	**24.78[2]**
TC#2 Ave	**0.2700**	**0.9985**	**1.000**	**0.2696**	**1.5405**	**0.415**	**24.92**
TB Ave	**0.2700**	**0.9985**	**1.000**	**0.2696**	**1.6395**	**0.442**	**26.52**

1. See footnotes in Table II for definitions for these terms.
2. **ESS** was directed to reduce the target spiking rate for these runs as a means of conserving limited stocks of spiking materials.

Table IV provides the average Naphthalene in Toluene Solution spiking rates as well as the concentration and purity corrected Toluene spiking results for: (1) each of the six TC #1 and TC #2 runs, (2) each of the two TCs, and (3) the overall trial burn.

Table IV Average Naphthalene in Toluene Solution (Spiking Material), and Toluene (Spiking Specie) Spiking Rate Results by Run and TC.

TC # / Run No.	Correction Factors, Mass Fraction				Nap Sol Spiking Rate[1], lb/min	Tolu Spiking Rate[1]	
	Toluene Concentration[1]	Toluene Purity[1]	Stoich. Content[1]	[Toluene][1]		lb/min	lb/hr
TC#1/Run# 1	0.7300	0.9993	1.000	0.7295	1.6261	1.186	71.17
TC#1/Run# 2	0.7300	0.9993	1.000	0.7295	1.7826	1.300	78.02
TC#1/Run# 3	0.7300	0.9993	1.000	0.7295	1.2100	0.8827[2]	52.96[2]
TC #1 Ave	0.7300	0.9993	1.000	0.7295	1.5396	1.123	67.39
TC#2/Run# 1	0.7300	0.9993	1.000	0.7295	1.5421	1.125[2]	67.50[2]
TC#2/Run# 2	0.7300	0.9993	1.000	0.7295	1.5550	1.134[2]	68.06[2]
TC#2/Run# 3	0.7300	0.9993	1.000	0.7295	1.5423	1.125[2]	67.50[2]
TC #2 Ave	0.7300	0.9993	1.000	0.7295	1.5464	1.128	67.69
TB Ave	0.7300	0.9993	1.000	0.7295	1.5430	1.126	67.54

1. See footnotes in Table II for definitions for these terms.
2. *ESS* was directed to reduce the target spiking rate for these runs as a means of conserving limited stocks of spiking materials.

THE EFFECT OF MEASUREMENT UNCERTAINTY ON SPIKING RATE RESULTS: METHODOLOGY & RESULTS

Measurement Uncertainty Associated with Field Weight Measurements with the weight loss versus time method

This section provides a summary of the measurement uncertainty aspects of: (1) the compositions of the two spiking materials which **ESS** prepared and supplied for this Trial Burn, (2) the weigh scale calibrations, pre- and post-test calibration verifications, and sensitivities, and (3) field spiking rate results. Additionally, an extensive uncertainty analysis was completed on spiking materials compositions and spiking rate results. The methodology used with respect to spiking rates is outlined below together with the results of both the composition and spiking rate analysis.

All weigh scales used during this trial burn were calibrated prior to the test and the calibrations were verified on-site (with ± 0.0 lb deviations at each point in the calibration range) immediately before and after the tests with **ESS'** NIST traceable weight standards. Thus, the field spiking rate data for this Trial Burn are deemed to meet all appropriate QC and QA standards and are demonstratably accurate within ± 0.1 Lb M/weight measurement.

Spiking		Sampling Method	Ave Sampling Period, Hours	Average Mass of Material Spiked per Run, Lb M/Run
Material:	Specie:			
TiO_2 Dispersion	Total Ash	Method 5	1.972	110.49 Lb TiO_2 Dispersion
Nap Solution	Naphthalene	Method 0010 (SVOC)	3.000	317.96 Lb Nap Solution
Nap Solution	Toluene	VOST (VOC)	2.667	246.88 Lb Nap Solution

The spiking data from the 2003 Case Study TB were used to calculate the quantity of each spiking material spiked per run and while the corresponding sampling method for that specie was being used. The results are summarized as follows:

With a weigh scale measurement uncertainty of ± 0.1 Lb M/weight measurement, and the assumption that all measurement uncertainty [error] occurs in the direction which would result in the maximum cumulative uncertainty, the maximum uncertainty in measuring the quantity of spiking material per run would be calculated as follows:

Field Measurement Uncertainty = (4 Sx Periods/Run) x (2 Weight Measurements/Sx Period) X (± 0.1 Lb M/Measurement)
= ± 0.8 Lb M/Run

The Effect of Measurement Uncertainty in Spiking Rate Results

Table V presents spiking rate uncertainty expressed on the following bases:

1 Absolute Uncertainty (Lb/Run AU) Basis:
   a. Spiking Material (Dispersion or Nap Sol) — Column 2
   b. Spiking Specie (Ash, Naphthalene, & Toluene) — Column 4
2. Relative Uncertainty (%RU) Basis:
   a. Spiking Material — Column 3
   b. Spiking Specie — Column 5
3. Absolute Specie Spiking Rate Uncertainty[1] (Lb Specie/Hr AU) Basis: — Columns 7, 8, & 9
4. Relative Specie Spiking Rate Uncertainty[1] (%RU) Basis: — Columns 10, 11, & 12

[1]The spiking rate uncertainties are presented on three measurement uncertainty bases: (1) field measurement uncertainty (Columns 7 & 10), (2) composition measurement uncertainty (Columns 8 & 11), and (3) the combined field spiking rate measurement plus composition uncertainty (Columns 9 & 12).

Table V **Effect of Field Measurement Uncertainty (By *Weight Loss Versus Time Method*), Compositional Uncertainty (By *Laboratory Standard Method*), and Combined Field Plus Compositional Uncertainties on Overall Spiking Rate Uncertainty**

Spiking Specie (1)[4]	Field Measurement Uncertainties expressed as Absolute Uncertainty (±Mass/Run AU), and Relative Uncertainty (±%RU)				Indicated Specie Spiking Rate, Lb S/Hr (6)	Cumulative Specie Spiking Rate Uncertainties expressed as:					
						Absolute Specie Spiking Rate Uncertainty Due to:			Relative Specie Spiking Rate Uncertainties Due to:		
	Spiking Material (M)		Spiking Specie (S)			Field Measurement ± Lb S/Hr AU (7)	Composition ± Lb S/Hr AU (8)	Combined ± Lb S/Hr AU (9)	Field Measurement ± % RU (10)	Composition ± % RU (11)	Combined ± % RU (12)
	± Lb M/Run, (2)	± %RU, (3)	± Lb S/Run, (4)	± %RU, (5)							
Four Spiking Periods Per Run (Measurement Uncertainty = ± 0.8 Lb M/Run)											
Ash[1]	0.8	0.72	0.20	0.72	14.12	0.102	0.0064	0.108	0.72	0.045	0.76
Nap[2]	0.8	0.25	0.22	0.25	26.52	0.066	0.0119	0.078	0.25	0.045	0.29
Toluene[3]	0.8	0.32	0.58	0.32	67.54	0.216r	0.0323	0.248	0.32	0.045	0.36
One Spiking Period Per Run (Measurement Uncertainty = ± 0.2 Lb M/Run)											
Ash[1]	0.2	0.18	0.05	0.18	14.12	0.025	0.0064	0.0314	0.18	0.045	0.22
Nap[2]	0.2	0.06	0.06	0.06	26.52	0.016	0.0119	0.0279	0.06	0.045	0.11
Toluene[3]	0.2	0.08	0.15	0.08	67.54	0.054	0.0323	0.0863	0.08	0.045	0.13

1. Total Ash has an indicated composition of 25.23 wt % ± 0.045 wt % in 110.49 Lb TiO_2 Dispersion/Run
2. Naphthalene has an indicated composition of 26.96 wt % ± 0.045 wt % in 317.96 Lb Nap Sol/Run
3. Toluene has an indicated composition of 72.95 wt % ± 0.045 w % in 246.88 Lb Nap Solution/Run
4. Column #'s which are used in text to identify uncertainty analysis results.

Additionally, for comparison purposes the entire analysis was repeated on the basis of **ESS'** standard spiking rate calculation procedures described above, i.e., spiking rate based on one spiking period with maximum field error of ± 0.2 Lb/Run. These results are presented in the bottom half of Table V.

Measurement Uncertainty Associated with Field Weight Measurements with the mass flow meter method

After reviewing available Micro Motion® Sales Literature and Product Specifications for the most sensitive (ELETE®) sensor and having numerous discussions with the Micro Motion ® technical sales and engineering staff, it appeared that a comprehensive analysis of measurement uncertainty in a "field" as opposed to a test bench setting was not available. As a result of the discussion with Mr. Tim Patten, Director of Measurement for Micro Motion®, one of the authors (WRS) concluded that the best approach to estimating field measurement uncertainty would be to assume that the published specification for sensor accuracy (Refs: 3, 4, 5) can be used without modification as the mass flow meter field measurement uncertainty. This approach allowed the uncertainty analysis to be completed without arbitrary revisions of the manufactures product specification. However, it should not necessarily be inferred that the manufactures product specification of accuracy is a complete measure of this equipments measurement uncertainty under field conditions.

Never the less, using the published accuracy specification of ± 0.1% as an estimate of field measurement uncertainty and the Case Study comparison basis described above, the absolute and relative spiking rates results were calculated and summarized in Table VI.

Table VI Effect of Field Measurement Uncertainty (By *Mass Flow Meter Method*), Compositional Uncertainty (By *Sample & Analyze Method*), and Combined Field Plus Compositional Uncertainties on Overall Spiking Rate Uncertainty

Spiking Specie (1)[4]	Field Measurement Uncertainties expressed as Absolute Uncertainty (±Mass/Run AU), and Relative Uncertainty (±%RU)				Indicated Specie Spiking Rate, Lb S/Hr (6)	Cumulative Specie Spiking Rate Uncertainties expressed as:					
						Absolute Specie Spiking Rate Uncertainty Due to:			Relative Specie Spiking Rate Uncertainties Due to:		
	Spiking Material (M)		Spiking Specie (S)			Field Measurement ± Lb S/Hr AU (7)	Composition ± Lb S/Hr AU (8)	Combined ± Lb S/Hr AU (9)	Field Measurement ± % RU (10)	Composition ± % RU (11)	Combined ± % RU (12)
	± Lb M/Run, (2)	± %RU, (3)	± Lb S/Run, (4)	± %RU, (5)							
Four Spiking Periods Per Run (Measurement Uncertainty = ± 0.8 Lb M/Run)											
Ash[1]	0.8	0.72	0.20	0.72	14.12	0.102	0.0064	0.108	0.72	0.045	0.76
Nap[2]	0.8	0.25	0.22	0.25	26.52	0.066	0.0119	0.078	0.25	0.045	0.29
Toluene[3]	0.8	0.32	0.58	0.32	67.54	0.216r	0.0323	0.248	0.32	0.045	0.36
One Spiking Period Per Run (Measurement Uncertainty = ± 0.2 Lb M/Run)											
Ash[1]	0.2	0.18	0.05	0.18	14.12	0.025	0.0064	0.0314	0.18	0.045	0.22
Nap[2]	0.2	0.06	0.06	0.06	26.52	0.016	0.0119	0.0279	0.06	0.045	0.11
Toluene[3]	0.2	0.08	0.15	0.08	67.54	0.054	0.0323	0.0863	0.08	0.045	0.13

1. Total Ash has an indicated composition of 25.23 wt % ± 0.045 wt % in 110.49 Lb TiO_2 Dispersion/Run
2. Naphthalene has an indicated composition of 26.96 wt % ± 0.045 wt % in 317.96 Lb Nap Sol/Run
3. Toluene has an indicated composition of 72.95 wt % ± 0.045 w % in 246.88 Lb Nap Solution/Run
4. Column #'s which are used in text to identify uncertainty analysis results.

QUANTITATIVE COMPARISON OF WEIGH CELL AND MASS FLOW METER MEASUREMENT UNCERTAINTIES

The absolute and relative specie spiking rate uncertainties based on the **weight loss versus time** and **mass flow meter methods** were then taken from Tables V & VI, respectively, and compiled as a comparison in Table VII. Inspection of Table VI reveals that **weight loss versus time** and the **mass flow meter methods** for measuring field spiking rate are essentially identical for all spiking species and on both absolute and relative uncertainty bases. However, the much greater measurement uncertainty associated with **Sample and Analyze Method** of demonstrating spiking material composition compared with the **Laboratory Standard Method** resulted in much higher total system spiking rate uncertainty for the combine **Mass Flow Meter & Sample & Analyze Approach** in comparison to the **Laboratory Standard & Weight Loss Versus Time Approach.**

Table VII Comparison of System Spiking Rate Uncertainties Due to Measurement Uncertainty in: (1) Composition by Two Methods, (2) Field Spiking Rates by Two Methods, and (3) Combined Composition + Field Spiking Rate Uncertainties

Spiking Specie	Specie Spiking Rate Uncertainty Due to:											
	Compositional Uncertainty:				Field Spiking Rate Uncertainty:				Composition + Spiking Rate Uncertainty			
	Lab Standard		Sample & Analyze		Weigh Cell		Mass Flow Meter		Lab Stand + Weigh		Sx & Anal. + MFM	
	± Lb S/Hr	± % RU	± Lb S/Hr	± % RU	± Lb S/Hr	± % RU	± Lb S/Hr	± % RU	± Lb S/Hr	± % RU	± Lb S/Hr	± % RU
Four Sampling Periods Per Run												
Ash	0.0064	0.045	1.41	10	0.102	0.72	0.014	0.1	0.108	0.76	1.42	10.1
Nap	0.0119	0.045	7.96	30	0.066	0.25	0.027	0.1	0.078	0.29	7.99	30.1
Toluene	0.0323	0.045	20.3	30	0.216	0.32	0.068	0.1	0.248	0.36	20.4	30.1
One Sampling Period Per Run												
Ash	0.0064	0.045	1.41	10	0.025	0.18	0.014	0.1	0.0314	0.22	1.42	10.1
Nap	0.0119	0.045	7.96	30	0.016	0.06	0.027	0.1	0.0279	0.11	7.99	30.1
Toluene	0.0323	0.045	20.3	30	0.054	0.08	0.068	0.1	0.0863	0.13	20.4	30.1

THE HWC CONTEXT FOR EVALUATING THE SPIKING FUNCTION

It is difficult if not impossible to evaluate the performance of one or more methods or technologies in meeting the requirements of their assigned function without some consideration of the application &/or context in which the methods/technologies are expected to perform. For example, there are several situations in which spiking occurs in HWC tests:

1. POHC spiking for DRE demonstration,
2. Acid gas precursor spiking for demonstrating the performance of and setting precursor feed rate limits for a wet scrubber, for example.
3. Ash spiking for similar purposes, and
4. Heavy metal spiking for demonstrating APC performance and feed rate APCS operating limit setting.

As a context for evaluating the performance of these competing technologies in the spiking function, we have somewhat arbitrarily assumed a case in which one metal is spiked into a HWC unit for the purpose of setting a feed rate limit for that metal. Within this general circumstance, each step in the process of designing, conducting and reporting the results a HWC test is identified and an order of magnitude estimate of the uncertainty associated with each step is provided in Table VIII.

Inspection of Table VIII prompts the following observations:

1. Uncertainties associated with the spiking function represent a relatively minor portion of the total uncertainty involved.
2. Within the spiking function, utilization of computer control and demonstrating spiking material composition with *the* Laboratory Standard Method clearly offer advantages in reducing spiking rate uncertainty.
3. Uncertainties associated with: (a) waste stream composition, (b) target spiking rate selection, (c) stack sampling, and (d) sample analysis all represent larger uncertainties than does the spiking function.

Table VIII The HWC Context for Evaluating the Spiking Function

Order of Magnitude Estimates of Uncertainties/Errors in the HWC Process of Setting Constituent Feed Rate Limits				
Areas of Uncertainty	**Mass Flow Meters With Computer Control and Composition by Sx & Analysis Method**	**Weighing Systems with:**		
		Manual Control	**Computer Control**	**[Specie] by Lab Standard**
Imperfect Knowledge of Waste Composition	±10%	±10%	±10%	±10%
Non-Optimum Target Spiking Rate	±30%	±30%	±30%	±30%
Off-set of Average Rate from Target Spiking Rate Due to:				
Imperfect Control	±1%	±3%	±1%	±1%
Imperfect Measurement	±1%	±1%	±1%	±1%
Imperfect Knowledge of [Specie]	±30%	±1%	±1%	±1%
Variations Around Average Spiking Rate Due to:				
Non-Homogeneous Materials	±1%	±1%	±1%	±1%
Imperfect Control	±1%	±1-5%	±1%	±1%
Measurement Uncertainty in Stack Sampling	±10%	±10%	±10%	±10%
Measurement Uncertainty in Sample Analysis	±30%	±30%	±30%	±30%

QUALITATIVE COMPARISON OF WEIGH CELL AND MASS FLOW METER TECHNOLOGIES FOR MEASURING FIELD SPIKING RATE

Up to this point, all discussion has been concerned with quantitative calculations and comparisons of measurement uncertainty between the two most widely used methods of measuring field spiking rate. There are however, other more qualitative attributes of both technologies which recommend their use. These attributes as well as the attributes of computer control and data acquisition are summarized within this section.

Both spiking rate measurement methods benefit similarly from the use of computer based process control and data acquisition technology. These benefits are summarized as follows:

1. The ability to control the spiking rate more uniformly and more closely to the target spiking rate than is possible with manual control.
2. Acquisition, archiving, analysis, and reporting of data in real time.
3. The ability to more rapidly effect spiking rate changes, as needed during miniburns for example.

The relative advantages and disadvantages of the two methods of measuring field spiking rate are summarized in the following table:

Major Features of the Two Field Spiking Rate Measurement Technologies	
Mass Flow Meters	**Weighing Systems**
Advantages:	**Advantages:**
Continuous, Direct Measurement of Flow	Rapid, Tangible Field Demonstration of Accuracy
More Rapid Detection of Rate Changes	Direct Measurement of Mass/Run
Very High Accuracy	Very High Accuracy
Disadvantages:	**Disadvantages:**
Very Difficult to Demonstrate Accuracy in the Field	Indirect Measurement of Rate

CONCLUSIONS:

As a result of the information provided herein, the authors have drawn the following conclusions:

1. Uncertainties associated with the spiking function in a HWC testing program are likely to be a modest part of the total uncertainty associated with the total regulatory/testing process for setting a metal feed rate limit.
2. Both the Mass Flow Meter Method and the Weigh Loss Versus Time Method of measuring field spiking rate provide highly accurate results.
3. The overall lowest level of spiking rate uncertainty is achieved with the Laboratory Standard Method of demonstrating spiking material composition combined with either of the Mass Flow Meter Method or the Weight Loss Versus Time Method of measuring field spiking rate.

REFERENCES

1. Trial Burn Spiking Report, 2003, Confidential Client & Location.

2. Schofield, Bill, Anthony R. Eicher, Sean O'Brien, THE EFFECT OF MEASUREMENT UNCERTAINTY ON SPIKING MATERIAL COMPOSITION AND SPIKING RATE UNCERTAINTIES, 2004 IT3 Conference Proceedings, Phoenix, AZ, May, 2004.

3. Micro Motion ® Product Data Sheet, PS-00230, Rev. A, October, 2003, Pages 9, 10, & 11, (Re: the CMF010 ELITE Sensor).

4. Traceability and Uncertainty Analysis for a Calibration Process for Flowmeters, Using Coriolis Flowmeters as Reference, Robert DeBoom, et. al., Micro Motion ®, June, 2000.

5. Private phone and email communications by one of the authors (WRS) with Mr. Tim Patten, Director of Flow Measurement, Micro Motion ®, March, 2004.

6. Neptune Chemical Pump Company, Inc, Bulletin DP-1100-2001, Series 500 dia-Pump, 2001, Page 10, Note 2.

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Tim Patten, and David Hollek of Micro Motion ® for sharing a significant quantity of written information, as well as providing numerous detailed explanations of various technical, sensitivity, reliability, and economic aspects of the ELITE Sensor Mass Flow Meter which their firm offers.

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[i] As used herein **Spiking Material (M)** refers to the material which is actually spiked, i.e., a metal solution, a TiO_2 and/or metal dispersion, and/or an individual or a mixture of POHCs. **Spiking Species (S)** refers to the portion of the **Spiking Material** which is of specific interest in meeting the test objectives, i.e., individual metals, ash, individual POHCs, Cl^-, etc.

[ii] Typically, the maximum error ± 0.005 to ±0.01% of the scale's capacity, or in terms of weight, ± 0.05 to ± 0.1 lb for our most frequently used 1,000 lb scales.

[iii] **ESS'** 50 lb field standards are certified annually by the State of Texas to be within ± 0.008 lb (approx. ± 0.02% RE) of NIST Primary Standards.

[iv] The pump through-put to line pressure sensitivity is: -1.5 %/100 psig (Ref 6), i.e., with a constant pump through-put setting, a waste feed line pressure increase of 100 psig would result in a pumping rate decrease of only 1.5%.

Attachment V. Effect of Measurement Uncertainty on Spiking Rate Uncertainty

B. Schofield, PhD, PE, Bill, Anthony R Eicher, and Sean O'Brien, The Effect of Measurement Uncertainty on Field Spiking Rate and Overall Specie Spiking Rate Uncertainties, 2004 IT3 Conference Proceedings, Paper #102, Phoenix, AZ, May, 2004

THE EFFECT OF MEASUREMENT UNCERTAINTY ON FIELD SPIKING RATE AND OVERALL SPECIE SPIKING RATE UNCERTAINTIES

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ABSTRACT

Objectives

The objectives of this paper are:

1. To develop estimates for Measurement Uncertainty on a common basis for the two primary field methods of measuring spiking rate, e.g., weigh cell and mass flow meter technologies;
2. To determine the impact of these measurement uncertainties on field spiking rate and overall spiking rate uncertainties; and
3. To compare the combined spiking rate uncertainties due to measurement uncertainties associated with: (a) spiking material composition, (b) field spiking rate measurement, and (c) spiking material composition plus field spiking rate measurements.

Methodology

Estimates of field spiking rate uncertainties are developed on the basis of:

- Case Study Basis for Calculations: The test-specific details of a 2003 TB [conducted at a private, US based HWC Unit] are used as a Case Study basis for preparing quantitative comparisons on a consistent basis.

- The two field spiking methods and associated modes of operation considered in this uncertainty analysis were:
  - Weight loss versus time method with manual operation, and
  - Mass flow meter method with computer control.

- The Following Assumptions Were Made in Estimating and Propagating Uncertainties:
  - For both spiking methods:
    - No undetected operator mistakes, equipment/software mal-functions, and/or data reduction/reporting errors have occurred,
    - Second-order uncertainties are not significant, and
    - All spiking materials are uniform in composition throughout the test.

- For the weight loss versus time method:
  - Measurement uncertainty can be conservatively estimated based on a series of worst case conditions concerning both the magnitude and direction of individual measurement uncertainties, and
  - Indeterminative errors are adequately addressed with the conservative approach used to estimate determinative weight measurement uncertainty.
- For the mass flow meter method:
  - The mass flow meter manufacturer's published specification for equipment accuracy is an appropriate estimate of field measurement uncertainty, and
  - The function (accuracy) of the mass flow meter sensor is not adversely affected by corrosion, erosion, and/or uneven spiking material deposition onto the interior surfaces of the sensor tube.

- The compositional uncertainty estimates from a companion paper are combined with the field spiking rate uncertainties developed herein to produce the overall spiking system uncertainties, as follows:

Total System Uncertainty	=	Compositional Uncertainties with the:	+	Spiking Rate Uncertainties with the:
System #1 Uncertainty	=	Laboratory Standard Method	+	Weight Loss versus Time Method
System #2 Uncertainty	=	Sample and Analyze Method	+	Mass Flow Meter Method

Results

Uncertainty comparisons are made for the two systems on three bases:

- Overall spiking system uncertainties associated with spiking material composition uncertainty alone,
- Overall spiking system uncertainties associated with field spiking rate uncertainty alone, and
- Overall spiking system uncertainties due to the combined impact of both composition and field spiking rate uncertainties.

INTRODUCTION AND BACKGROUND

A great deal of effort has been expended over the last decade to investigate, understand, and improve the Quality Assurance (QA) aspects of the sampling and analytical methods used in Hazardous Waste Combustion (HWC) Risk Burns, Trial Burns, and HWC MACT Comprehensive Performance Tests. To date a comparable effort has not been made concerning the spiking function in these same tests. Additionally, conflicting information is being provided by proponents of the two most widely used methods of demonstrating spiking material composition and of measuring field spiking rates.

Objectives

The objectives of this paper are threefold:

1. To develop estimates for Measurement Uncertainty on a common basis for the two primary field methods of measuring spiking rate, e.g., weigh cell and mass flow meter technologies;
2. To determine the impact of these measurement uncertainties on field spiking rate and overall spiking rate uncertainties; and
3. To compare the spiking rate uncertainties due to measurement uncertainties associated with: (a) spiking material composition, (b) field spiking rate measurement and (c) spiking material composition plus field spiking rate measurements.

This paper also examines the effect of measurement uncertainty, imperfect knowledge, and/or error at each step of the spiking function from the point of setting a target spiking rate in a test plan, through spiking material design and preparation, the on-site spiking rate measurement and data collection functions, and the ultimate reporting of spiking rate results. Each major type of deviation, error, and/or uncertainty in the spiking function is identified and discussed in the context of the facility owners test objectives and applicable regulatory requirements.

Methodology: Estimating the Effect of Measurement Uncertainty

The effect of measurement uncertainty on field spiking rate and overall spiking system uncertainties are estimated on the following basis:

- **Case Study** basis for comparisons: The test-specific details of a 2003 TB [conducted at a private, US based HWC Unit] are used herein as a Case Study for preparing quantitative comparisons on a consistent basis;

- The **Two Field Spiking Methods** and associated **Modes of Operation** are used as the primary subject of this uncertainty analysis:
  - Weight loss versus time method with manual operation, and
  - Mass flow meter method with computer control.

  [While **ESS** currently deploys computer based technology for spiking system monitoring, feedback control, data acquisition, archiving, and output; and both mass flow meter and weigh cell technologies for measuring field spiking rates; the data presented herein were obtained prior to the mass flow meter and computer control technology becoming operationally available.]

- The following **Assumptions** were made in estimating and propagating uncertainties:
  - **For both spiking methods:**
    - No undetected operator mistakes, equipment/software mal-functions and/or data reduction errors have occurred,
    - Second-order uncertainties are not significant:
      [Because of the very small magnitude of all first-order uncertainties, no second order uncertainties are considered. The validity of this assumption is demonstrated within the companion paper (Ref.2), using first-order uncertainties estimates developed for the case study example], and
    - All spiking materials were uniform in composition throughout the test:
      [The two spiking materials used in the Case Study (e.g., Naphthalene in a Toluene solution, & TiO_2 in a mineral oil based dispersion) are typical of spiking materials, in that they well known in terms of chemistry and have been successfully used many times over a period of more than a decade. The solubility of Naphthalene is known and the Naphthalene in Toluene spiking material is prepared as an unsaturated solution.

      While there are reports that some plating or deposition of TiO_2 onto the inner surfaces of tubing can occur, this phenomena would largely occur during the initial equipment conditioning (pre-test) phase, and the mass of TiO_2 which could plate out in this case prior to blocking the relatively short, small diameter (1/2" ID) tubing is very small (<< 0.1 Lb) in comparison to the total quantity of TiO_2 spiked during a given run (> 100 Lb).]
  - **For the weight loss versus time method:**
    - The impact of weight measurement uncertainty on field spiking rate uncertainty can be estimated based on a series of worst case assumptions related to both the magnitude and direction of individual measurement uncertainties.

[The maximum measurement uncertainty together with the direction of each measurement uncertainty which would produce the largest cumulative field spiking rate uncertainty are used in all uncertainty propagation calculations. Specifically, weight measurement uncertainty is estimated on the basis of a large determinative uncertainty (**± U Lb**) based on the equipment vendor's specification of measurement uncertainty, typically, U = 0.01% of the full scale capacity of the equipment used). This uncertainty is carried throughout the uncertainty propagation calculations as if it were part of the weight (**W**) value (i.e., **W** was replaced with **W ± U Lb**). Once the calculation was completed, the + or - uncertainty directions for U which would result in the largest cumulative spiking rate uncertainty are selected.], and

- Indeterminative errors are adequately considered with the conservative approach used to estimate determinative weight measurement uncertainty.
  [The magnitude of determinative uncertainty (**± U**) is chosen to be sufficiently large and the weigh scale indicator setting is set such that random variation in the weight measurements (indeterminative uncertainty) is hidden in the decimal places which are not displayed. As a result, indeterminative uncertainty is not expected have a material impact on the results of this analysis.]

- For the **mass flow meter method:**
  - The equipment manufacturer's published specification for sensor accuracy (Refs: 3, 4, 5) can be used without modification as the mass flow meter field measurement uncertainty, and
  - The function (accuracy) of the mass flow meter sensor is not adversely affected by corrosion, erosion and/or uneven spiking material deposition onto the interior surfaces of the sensor tube (Refs: 3, 4, 5).

- Significant figures:
  - A large number of calculations are made in this uncertainty analysis, many with extremely small numbers. To avoid rounding errors and to retain the integrity of the uncertainty estimates developed herein, a relatively large number of significant figures are carried through the calculations and presented in the tables.
  - The authors do not claim the accuracy and/or precision in these figures that would normally be implied by the standard significant figures rules.

- The compositional uncertainty estimates from a companion paper are combined with the field spiking rate uncertainties developed herein to produce the overall spiking system uncertainties, as follows:

Total System Uncertain	=	Compositional Uncertainties with the:	+	Spiking Rate Uncertainties with the:
System #1 Uncertainty	=	*Laboratory* Standard Method	+	Weight Loss versus Time Method
System #2 Uncertainty	=	Sample and Analyze Method	+	Mass Flow Meter Method

[The uncertainties associated with composition and field spiking rate are combined in this manner to reflect the standard practices of representative spiking firms within the spiking industry.]

Results

Uncertainty comparisons are made for these two, frequently used spiking systems on three bases:

- Overall spiking system uncertainties associated with spiking material composition uncertainty alone,
- Overall spiking system uncertainties associated with field spiking rate uncertainty alone, and

- Overall spiking system uncertainties due to the combined impact of both composition and field spiking rate uncertainties.

Description of the "Case Study" Trial Burn

The Case Study TB was conducted on a confidential, non-commercial, HWC Unit during 2003, and consisted of two Test Conditions (TC) which were defined as follows: (1) TC #1: Maximum Waste Feed, and (2) TC #2: Minimum Temperature (DRE). The spiking materials[1] consisted of a Naphthalene in Toluene Solution (@ a nominal 27 wt % Naphthalene) and a TiO_2 Dispersion (@ a nominal 25 wt % Total Ash). The testing/spiking schedule is summarized as follows:

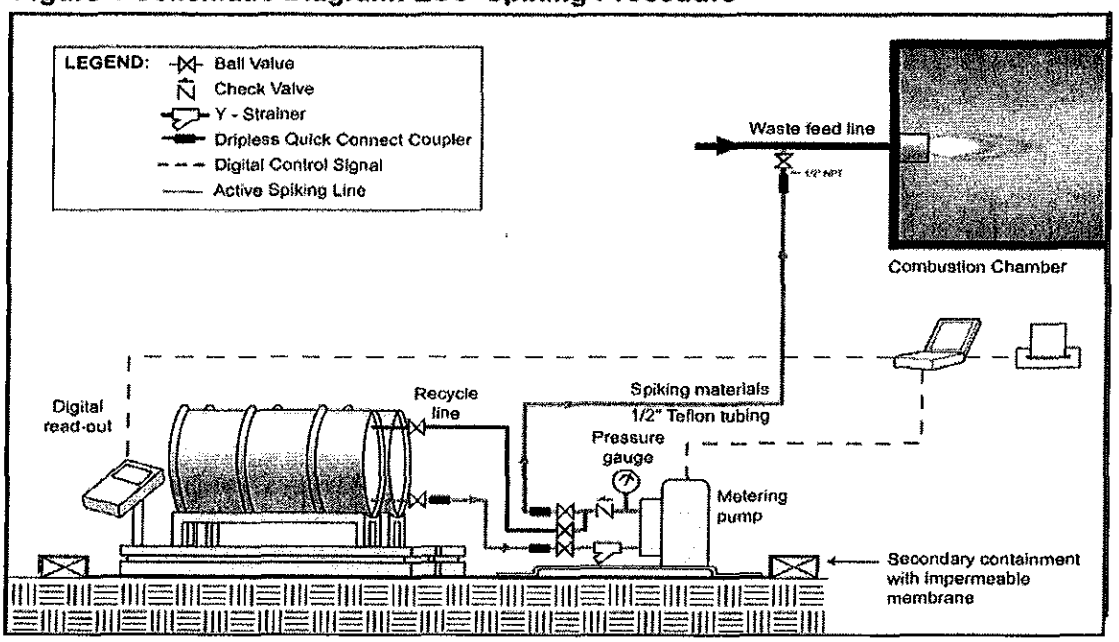
Test Condition	Date Conducted	Spiking With:	
		Nap Sol	Dispersion
TC #1	2003	✓	✓
TC #2	2003	✓	

The spiking function for this TB involved three spiking species[i] (e.g., Total Ash, Naphthalene, and Toluene) which were contained in two spiking materials[1] (e.g., TiO_2 Dispersion, and Naphthalene in Toluene Solution). The dispersion was used as an ash surrogate with ash contributions from both the TiO_2 (primary) and the proprietary dispersing agent (secondary). The Naphthalene in Toluene Solution spiking material contained both POHCs, e.g., Naphthalene and Toluene.

Equipment Setup and Operation for the Weight Loss Versus Time Spiking Approach

Typically a drum (or tote tank or gas cylinder) of spiking material is placed on an appropriately sized weigh scale (the smallest [most accurate] scale which can weigh the full container of spiking material) and connected with SS, dripless, quick-connect fittings to the metering pump, which is similarly connected to the waste feed line. As material is pumped out of the drum (and into the waste feed line) the mass on the weigh scale drops (see Figure 1). The weight of spiking material remaining on the weigh scale is recorded and the spiking rate calculated frequently based on the rate of change of mass on the weigh scale.

Figure 1 Schematic Diagram: ESS' Spiking Procedure



The weigh scales[ii] (See Table 1) are calibrated before the test and the calibration is verified on-site immediately before and after the test with NIST traceable standards[iii] The pre- and post-test calibration verifications generally indicate no deviations (e.g., $\pm$ 0.0 Lb deviation) for most if not all points over the full calibration range (typically, 0.0 – 650.0 Lb).

Table I Weighing Equipment Accuracy Related Specifications

Specification	Units	Weigh Scale Manufacturer
		Rice Lake
Capacity @ Full Scale(FS)	Lb (Kg)	1,000 (500)
Divisions[1] for Full Scale, (d)/FS:		
NTEP[2]	d/FS	5,000
Non-NTEP[2]	d/FS	10,000
Lb/Division (%FS/d):		
NTEP[2]	Lb/d (%FS/d)	0.02 (0.02%)
Non-NTEP[2]	Lb/d (%FS/d)	0.01 (0.01%)
Performance Specifications:		
Non-Linearity	0.03% FS	0.03% FS
Hysterises	0.02% FS	0.02% FS

Footnotes: 1. The number of divisions/FS is an indication of scale sensitivity. For example, a division is the smallest weight increment discernable by the weighing system according to a given set of accuracy, calibration frequency, and environmental condition requirements.

2. NTEP is a quasi governmental organization established to regulate weights and measures used for commercial purposes. NTEP certified equipment has a conservative classification to properly reflect how measuring equipment may be used in commerce [i.e., infrequently calibrated, handled roughly, operated in a wide range of environmental conditions] while still providing acceptable accuracy. For the purposes of weighing ingredients for spiking materials with very frequent equipment maintenance & calibrations, and in controlled conditions of temperatures and humidity, the Non-NTEP division count is generally considered to be representative of scale accuracy. This observation has been confirmed by extensive pre-use and post-used calibration verifications with NIST traceable standards which consistently demonstrated deviations from the standards of $\leq$ 0.01% or equivalently d/FS $\geq$ 10,000.

Procedures for Calculating Spiking Rate

Standard Spiking Rate Calculation Procedure

The standard procedure for calculating the spiking rate for a given run with the Weight Loss Versus Time Method is to include spiking rate data for the time period beginning when the stack sampling probe is first introduced into the stack [or from the beginning of sampling with the first VOST tube pair], through port changes [or VOST tube replacement] until the probe is removed from the stack at the end of that run [or until sampling with the last VOST tube pair is completed] unless some abnormal event occurs such as an extended combustor operational problem, or the rare sampling train leak check failure. Because metering pumps (which maintain essentially constant feed rates throughout the run[iv]) are used (instead of simple transfer pumps which are susceptible to throughput swings in response to waste feed line pressure changes), this approach has a number of advantages (e.g., simplified data reduction, and reduced

measurement uncertainty [see discussion below]), and no disadvantages. If problems were to occur which might bring operating, sampling, or spiking performance data into question, then the spiking data from that period would be excluded from the spiking rate calculations.

With this procedure, calculation of the spiking rate for a given run typically requires the recording and use of two weight measurements, i.e., the beginning mass and the final mass. Thus, weighing systems measurement uncertainty could occur twice.

A conservative estimate of measurement uncertainty in the mass of spiked material per run would assume that: (1) the weight measurement for the beginning mass measurement and for the ending mass reading are each in "error", (2) the "error" is equal to the full measurement uncertainty, and (3) the two "errors" are in opposite directions (so that the measurement uncertainties would be additive and would not cancel each other).

If one were to assume a measurement uncertainty of ± 0.1 Lb associated with each weight measurement reading, then the maximum measurement uncertainty for the total mass of spiking material (M) fed during a run would be ± 0.2 Lb M/Run.

For a run with 300 Lbs M/Run, the relative uncertainty (RU, expressed as a per cent) would be:

RU = (± 0.2 Lb M/Run)/(300 Lb M/Run) X 100 % RU
= ± 0.0667 % RU, a very small relative uncertainty.

For a run with 100 Lbs M/Run, the maximum measurement uncertainty for the total mass of spiking material fed during a run would remain ± 0.2 Lb M/Run, but the relative uncertainty would be:

RU = (± 0.2 Lb M/Run)/(100 Lb M/Run) X 100 % RU
= ± 0.2 % RU, still a very small relative uncertainty.

Similarly, if the spiking material were to contain the spiking specie (S) at a 20 wt% concentration, then the corresponding absolute measurement uncertainty would be ± 0.04 Lb S/Run, and the corresponding RU values for specie uncertainties would not change.

A More Conservative Spiking Rate Calculation Procedure

If, however, one were to decide to use only spiking data during test periods when spiking and stack sampling for that specie were both occurring, the spiking rate calculations could involved two or more separate spiking periods during each run. As before, each sampling period (Sx Period) required two weight measurements (at the beginning and the end of each period), each with its own measurement uncertainty.

For a run with four sampling periods and 300 Lbs M/Run, the maximum measurement uncertainty for the total mass of spiking material fed during a run would be ± 0.8 Lb M/Run and the relative uncertainty would be:

RU = (± 0.2 Lb M/Sx Period) X (4 Sx Periods/Run)/(300 Lb M/Run)X100 % RU
= (± 0.8 Lb M/Run)/(300 Lb M/Run) X 100 % RU
= ± 0.2667 % RU, a very modest relative uncertainty.

And for a run with four sampling periods and 100 Lbs M/Run, the maximum measurement uncertainty for the total mass of spiking material fed during a run would remain ± 0.8 Lb M/Run, but the relative uncertainty would be:

RE = (± 0.2 Lb M/Sx Period) X (4 Sx Periods/Run)/(100 Lb M/Run)X100 % RU
= (± 0.8 Lb M/Run)/(100 Lb M/Run) X 100 % RU
= ± 0.8 % RU, still a modest relative uncertainty.

Similarly, if the spiking material were to contain the spiking specie at 20 wt %, then the maximum measurement uncertainty for the total mass of spiking specie fed during a run would be ± 0.16 Lb S/Run and the corresponding RU values for specie uncertainties would not change.

Additional quantitative analyses of the effect of measurement uncertainty on field spiking rate based on the Case Study TB are provided below.

FIELD SPIKING RATE RESULTS

The Spiking Log Sheets completed during the Case Study TB were used together with the Certificates of Composition to calculate the specie spiking rates using both the standard and the more conservative procedures described above.

Note: During the Case Study 2003 TB, each spiking specie (Ash/PM, Naphthalene, and Toluene) was sampled using a different sampling method and over different sampling periods.

The resulting field spiking rate results for Ash, Naphthalene, and Toluene are presented in Tables II, III, and IV, respectively.

Total Ash Spiking Rate Results

Table II provides the average TiO_2 Dispersion, and concentration corrected Total Ash spiking rates for each of the three TC #1 runs, as well as for TC #1 in toto.

Table II Average TiO_2 Dispersion (Spiking Material[1]) and Total Ash (Spiking Specie[1]) Spiking Rates by Run for TC #1

TC#1/ Run#	Mass Fraction				Disp Spiking Rate[3], lb/min	Ash Spiking Rate[4]:	
	$[TiO_2]^2$	TiO_2 Purity[2]	Stoich. Content[2]	[Ash][2]		lb/min	lb/hour
Run #1	0.244	98.7	1.0477	**0.2523**	**0.8752**	**0.221**	**13.25**
Run #2	0.244	98.7	1.0477	**0.2523**	**0.9726**	**0.245**	**14.72**
Run #3	0.244	98.7	1.0477	**0.2523**	**0.9508**	**0.240**	**14.39**
TC #1 Ave	**0.244**	**98.7**	**1.0477**	**0.2523**	**0.9329**	**0.235**	**14.12**

Footnotes:

1. As used throughout this paper, **Spiking Material (M)** refers to the material as it is actually spiked, i.e., a Naphthalene in Toluene Solution, and/or a TiO_2 dispersion. **Spiking Species (S)** refers to the portion of the **Spiking Material** which is of specific interest in meeting the test objectives, i.e., Naphthalene, toluene, ash, POHC, Cl^-, etc.
2. **Concentration** refers to the concentration of the compound of interest in the **Spiking Material**, for example Nap in the Naphthalene in Toluene Solution assuming 100% purity. **Purity** refers to the assay, or purity of the Naphthalene, for example, used to make up the solution to the desired concentration. **Stoich. Content** refers to the stoichiometric content of the specie of interest in the compound, for example the Cl^- content in Perc or metal content in the metal compound. **[Specie]** indicates the specie concentration (usually expressed as Lb Specie/Lb Material, or mass fraction) and is defined as:
   [Specie] = Concentration x Purity x Stoich. Content. **[Specie]** is used to convert the **Spiking Material** spiking rate to the corresponding **Spiking Specie** spiking rate. Usually, all four of the "correction" terms are expressed as mass fractions.
3. Without Correction for **[Specie]**. Calculated from field spiking data.
4. With Correction for **[Specie]**

Naphthalene Spiking Rate Results

Table III provides the average Naphthalene in Toluene Solution spiking rates as well as the concentration and purity corrected Naphthalene spiking results for: (1) each of the six TC #1 and TC #2 runs, (2) each of the two TCs, and (3) the overall trial burn.

Table III Average Naphthalene Solution (Spiking Material[1]), and Naphthalene (Spiking Specie[1]) Spiking Rate Results by Run and TC.

TC#/ Run#	Correction Factors, Mass Fraction				Nap Sol Spiking Rate[1], lb/min	Nap Spiking Rate[1]	
	Nap Concentration[1]	Nap Purity[1]	Stoich. Content[1]	[Nap][1]		lb/min	lb/hr
TC#1/Run#1	0.2700	0.9985	1.000	**0.2696**	**1.7546**	**0.473**	**28.38**
TC#1/Run#2	0.2700	0.9985	1.000	**0.2696**	**1.8538**	**0.500**	**29.98**
TC#1/Run#3	0.2700	0.9985	1.000	**0.2696**	**1.6068**	**0.433[2]**	**25.99[2]**
TC#1 Ave	**0.2700**	**0.9985**	**1.000**	**0.2696**	**1.7384**	**0.469**	**28.12**
TC#2/Run#1	0.2700	0.9985	1.000	**0.2696**	**1.5416**	**0.4156[2]**	**24.94[2]**
TC#2/Run#2	0.2700	0.9985	1.000	**0.2696**	**1.5479**	**0.4173[2]**	**25.04[2]**
TC#2/Run#3	0.2700	0.9985	1.000	**0.2696**	**1.5321**	**0.4130[2]**	**24.78[2]**
TC#2 Ave	**0.2700**	**0.9985**	**1.000**	**0.2696**	**1.5405**	**0.415**	**24.92**
TB Ave	**0.2700**	**0.9985**	**1.000**	**0.2696**	**1.6395**	**0.442**	**26.52**

1. See footnotes in Table II for definitions for these terms.
2. **ESS** was directed to reduce the target spiking rate for these runs as a means of conserving limited stocks of spiking materials.

Table IV provides the average Naphthalene in Toluene Solution spiking rates as well as the concentration and purity corrected Toluene spiking results for: (1) each of the six TC #1 and TC #2 runs, (2) each of the two TCs, and (3) the overall trial burn.

Table IV Average Naphthalene in Toluene Solution (Spiking Material), and Toluene (Spiking Specie) Spiking Rate Results by Run and TC.

TC # / Run No.	Correction Factors, Mass Fraction				Nap Sol Spiking Rate[1], lb/min	Tolu Spiking Rate[1]	
	Toluene Concentration[1]	Toluene Purity[1]	Stoich. Content[1]	[Toluene][1]		lb/min	lb/hr
TC#1/Run# 1	0.7300	0.9993	1.000	0.7295	1.6261	1.186	71.17
TC#1/Run# 2	0.7300	0.9993	1.000	0.7295	1.7826	1.300	78.02
TC#1/Run# 3	0.7300	0.9993	1.000	0.7295	1.2100	0.8827[2]	52.96[2]
TC #1 Ave	0.7300	0.9993	1.000	0.7295	1.5396	1.123	67.39
TC#2/Run# 1	0.7300	0.9993	1.000	0.7295	1.5421	1.125[2]	67.50[2]
TC#2/Run# 2	0.7300	0.9993	1.000	0.7295	1.5550	1.134[2]	68.06[2]
TC#2/Run# 3	0.7300	0.9993	1.000	0.7295	1.5423	1.125[2]	67.50[2]
TC #2 Ave	0.7300	0.9993	1.000	0.7295	1.5464	1.128	67.69
TB Ave	0.7300	0.9993	1.000	0.7295	1.5430	1.126	67.54

1. See footnotes in Table II for definitions for these terms.
2. *ESS* was directed to reduce the target spiking rate for these runs as a means of conserving limited stocks of spiking materials.

THE EFFECT OF MEASUREMENT UNCERTAINTY ON SPIKING RATE RESULTS: METHODOLOGY & RESULTS

Measurement Uncertainty Associated with Field Weight Measurements with the weight loss versus time method

This section provides a summary of the measurement uncertainty aspects of: (1) the compositions of the two spiking materials which **ESS** prepared and supplied for this Trial Burn, (2) the weigh scale calibrations, pre- and post-test calibration verifications, and sensitivities, and (3) field spiking rate results. Additionally, an extensive uncertainty analysis was completed on spiking materials compositions and spiking rate results. The methodology used with respect to spiking rates is outlined below together with the results of both the composition and spiking rate analysis.

All weigh scales used during this trial burn were calibrated prior to the test and the calibrations were verified on-site (with ± 0.0 lb deviations at each point in the calibration range) immediately before and after the tests with **ESS'** NIST traceable weight standards. Thus, the field spiking rate data for this Trial Burn are deemed to meet all appropriate QC and QA standards and are demonstratably accurate within ± 0.1 Lb M/weight measurement.

Spiking		Sampling Method	Ave Sampling Period, Hours	Average Mass of Material Spiked per Run, Lb M/Run
Material:	Specie:			
TiO_2 Dispersion	Total Ash	Method 5	1.972	110.49 Lb TiO_2 Dispersion
Nap Solution	Naphthalene	Method 0010 (SVOC)	3.000	317.96 Lb Nap Solution
Nap Solution	Toluene	VOST (VOC)	2.667	246.88 Lb Nap Solution

The spiking data from the 2003 Case Study TB were used to calculate the quantity of each spiking material spiked per run and while the corresponding sampling method for that specie was being used. The results are summarized as follows:

With a weigh scale measurement uncertainty of ± 0.1 Lb M/weight measurement, and the assumption that all measurement uncertainty [error] occurs in the direction which would result in the maximum cumulative uncertainty, the maximum uncertainty in measuring the quantity of spiking material per run would be calculated as follows:

Field Measurement Uncertainty = (4 Sx Periods/Run) x (2 Weight Measurements/Sx Period) X (± 0.1 Lb M/Measurement)
= ± 0.8 Lb M/Run

The Effect of Measurement Uncertainty in Spiking Rate Results

Table V presents spiking rate uncertainty expressed on the following bases:

1 Absolute Uncertainty (Lb/Run AU) Basis:
   a. Spiking Material (Dispersion or Nap Sol) — Column 2
   b. Spiking Specie (Ash, Naphthalene, & Toluene) — Column 4
2. Relative Uncertainty (%RU) Basis:
   a. Spiking Material — Column 3
   b. Spiking Specie — Column 5
3. Absolute Specie Spiking Rate Uncertainty[1] (Lb Specie/Hr AU) Basis: — Columns 7, 8, & 9
4. Relative Specie Spiking Rate Uncertainty[1] (%RU) Basis: — Columns 10, 11, & 12

[1]The spiking rate uncertainties are presented on three measurement uncertainty bases: (1) field measurement uncertainty (Columns 7 & 10), (2) composition measurement uncertainty (Columns 8 & 11), and (3) the combined field spiking rate measurement plus composition uncertainty (Columns 9 & 12).

Table V **Effect of Field Measurement Uncertainty (By *Weight Loss Versus Time Method*), Compositional Uncertainty (By *Laboratory Standard Method*), and Combined Field Plus Compositional Uncertainties on Overall Spiking Rate Uncertainty**

Spiking Specie (1)[4]	Field Measurement Uncertainties expressed as Absolute Uncertainty (±Mass/Run AU), and Relative Uncertainty (±%RU)				Indicated Specie Spiking Rate, Lb S/Hr (6)	Cumulative Specie Spiking Rate Uncertainties expressed as:					
						Absolute Specie Spiking Rate Uncertainty Due to:			Relative Specie Spiking Rate Uncertainties Due to:		
	Spiking Material (M)		Spiking Specie (S)			Field Measurement ± Lb S/Hr AU (7)	Composition ± Lb S/Hr AU (8)	Combined ± Lb S/Hr AU (9)	Field Measurement ± % RU (10)	Composition ± % RU (11)	Combined ± % RU (12)
	± Lb M/Run, (2)	± %RU, (3)	± Lb S/Run, (4)	± %RU, (5)							
Four Spiking Periods Per Run (Measurement Uncertainty = ± 0.8 Lb M/Run)											
Ash[1]	0.8	0.72	0.20	0.72	14.12	0.102	0.0064	0.108	0.72	0.045	0.76
Nap[2]	0.8	0.25	0.22	0.25	26.52	0.066	0.0119	0.078	0.25	0.045	0.29
Toluene[3]	0.8	0.32	0.58	0.32	67.54	0.216r	0.0323	0.248	0.32	0.045	0.36
One Spiking Period Per Run (Measurement Uncertainty = ± 0.2 Lb M/Run)											
Ash[1]	0.2	0.18	0.05	0.18	14.12	0.025	0.0064	0.0314	0.18	0.045	0.22
Nap[2]	0.2	0.06	0.06	0.06	26.52	0.016	0.0119	0.0279	0.06	0.045	0.11
Toluene[3]	0.2	0.08	0.15	0.08	67.54	0.054	0.0323	0.0863	0.08	0.045	0.13

1. Total Ash has an indicated composition of 25.23 wt % ± 0.045 wt % in 110.49 Lb TiO_2 Dispersion/Run
2. Naphthalene has an indicated composition of 26.96 wt % ± 0.045 wt % in 317.96 Lb Nap Sol/Run
3. Toluene has an indicated composition of 72.95 wt % ± 0.045 w % in 246.88 Lb Nap Solution/Run
4. Column #'s which are used in text to identify uncertainty analysis results.

Additionally, for comparison purposes the entire analysis was repeated on the basis of **ESS'** standard spiking rate calculation procedures described above, i.e., spiking rate based on one spiking period with maximum field error of ± 0.2 Lb/Run. These results are presented in the bottom half of Table V.

Measurement Uncertainty Associated with Field Weight Measurements with the mass flow meter method

After reviewing available Micro Motion® Sales Literature and Product Specifications for the most sensitive (ELETE®) sensor and having numerous discussions with the Micro Motion ® technical sales and engineering staff, it appeared that a comprehensive analysis of measurement uncertainty in a "field" as opposed to a test bench setting was not available. As a result of the discussion with Mr. Tim Patten, Director of Measurement for Micro Motion®, one of the authors (WRS) concluded that the best approach to estimating field measurement uncertainty would be to assume that the published specification for sensor accuracy (Refs: 3, 4, 5) can be used without modification as the mass flow meter field measurement uncertainty. This approach allowed the uncertainty analysis to be completed without arbitrary revisions of the manufactures product specification. However, it should not necessarily be inferred that the manufactures product specification of accuracy is a complete measure of this equipments measurement uncertainty under field conditions.

Never the less, using the published accuracy specification of ± 0.1% as an estimate of field measurement uncertainty and the Case Study comparison basis described above, the absolute and relative spiking rates results were calculated and summarized in Table VI.

Table VI Effect of Field Measurement Uncertainty (By *Mass Flow Meter Method*), Compositional Uncertainty (By *Sample & Analyze Method*), and Combined Field Plus Compositional Uncertainties on Overall Spiking Rate Uncertainty

Spiking Specie (1)[4]	Field Measurement Uncertainties expressed as Absolute Uncertainty (±Mass/Run AU), and Relative Uncertainty (±%RU)				Indicated Specie Spiking Rate, Lb S/Hr (6)	Cumulative Specie Spiking Rate Uncertainties expressed as:					
						Absolute Specie Spiking Rate Uncertainty Due to:			Relative Specie Spiking Rate Uncertainties Due to:		
	Spiking Material (M)		Spiking Specie (S)			Field Measurement ± Lb S/Hr AU (7)	Composition ± Lb S/Hr AU (8)	Combined ± Lb S/Hr AU (9)	Field Measurement ± % RU (10)	Composition ± % RU (11)	Combined ± % RU (12)
	± Lb M/Run, (2)	± %RU, (3)	± Lb S/Run, (4)	± %RU, (5)							
Four Spiking Periods Per Run (Measurement Uncertainty = ± 0.8 Lb M/Run)											
Ash[1]	0.8	0.72	0.20	0.72	14.12	0.102	0.0064	0.108	0.72	0.045	0.76
Nap[2]	0.8	0.25	0.22	0.25	26.52	0.066	0.0119	0.078	0.25	0.045	0.29
Toluene[3]	0.8	0.32	0.58	0.32	67.54	0.216r	0.0323	0.248	0.32	0.045	0.36
One Spiking Period Per Run (Measurement Uncertainty = ± 0.2 Lb M/Run)											
Ash[1]	0.2	0.18	0.05	0.18	14.12	0.025	0.0064	0.0314	0.18	0.045	0.22
Nap[2]	0.2	0.06	0.06	0.06	26.52	0.016	0.0119	0.0279	0.06	0.045	0.11
Toluene[3]	0.2	0.08	0.15	0.08	67.54	0.054	0.0323	0.0863	0.08	0.045	0.13

1. Total Ash has an indicated composition of 25.23 wt % ± 0.045 wt % in 110.49 Lb TiO_2 Dispersion/Run
2. Naphthalene has an indicated composition of 26.96 wt % ± 0.045 wt % in 317.96 Lb Nap Sol/Run
3. Toluene has an indicated composition of 72.95 wt % ± 0.045 w % in 246.88 Lb Nap Solution/Run
4. Column #'s which are used in text to identify uncertainty analysis results.

QUANTITATIVE COMPARISON OF WEIGH CELL AND MASS FLOW METER MEASUREMENT UNCERTAINTIES

The absolute and relative specie spiking rate uncertainties based on the **weight loss versus time** and **mass flow meter methods** were then taken from Tables V & VI, respectively, and compiled as a comparison in Table VII. Inspection of Table VI reveals that **weight loss versus time** and the **mass flow meter methods** for measuring field spiking rate are essentially identical for all spiking species and on both absolute and relative uncertainty bases. However, the much greater measurement uncertainty associated with **Sample and Analyze Method** of demonstrating spiking material composition compared with the **Laboratory Standard Method** resulted in much higher total system spiking rate uncertainty for the combine **Mass Flow Meter & Sample & Analyze Approach** in comparison to the **Laboratory Standard & Weight Loss Versus Time Approach.**

Table VII Comparison of System Spiking Rate Uncertainties Due to Measurement Uncertainty in: (1) Composition by Two Methods, (2) Field Spiking Rates by Two Methods, and (3) Combined Composition + Field Spiking Rate Uncertainties

Spiking Specie	Specie Spiking Rate Uncertainty Due to:											
	Compositional Uncertainty:				Field Spiking Rate Uncertainty:				Composition + Spiking Rate Uncertainty			
	Lab Standard		Sample & Analyze		Weigh Cell		Mass Flow Meter		Lab Stand + Weigh		Sx & Anal. + MFM	
	± Lb S/Hr	± % RU	± Lb S/Hr	± % RU	± Lb S/Hr	± % RU	± Lb S/Hr	± % RU	± Lb S/Hr	± % RU	± Lb S/Hr	± % RU
Four Sampling Periods Per Run												
Ash	0.0064	0.045	1.41	10	0.102	0.72	0.014	0.1	0.108	0.76	1.42	10.1
Nap	0.0119	0.045	7.96	30	0.066	0.25	0.027	0.1	0.078	0.29	7.99	30.1
Toluene	0.0323	0.045	20.3	30	0.216	0.32	0.068	0.1	0.248	0.36	20.4	30.1
One Sampling Period Per Run												
Ash	0.0064	0.045	1.41	10	0.025	0.18	0.014	0.1	0.0314	0.22	1.42	10.1
Nap	0.0119	0.045	7.96	30	0.016	0.06	0.027	0.1	0.0279	0.11	7.99	30.1
Toluene	0.0323	0.045	20.3	30	0.054	0.08	0.068	0.1	0.0863	0.13	20.4	30.1

THE HWC CONTEXT FOR EVALUATING THE SPIKING FUNCTION

It is difficult if not impossible to evaluate the performance of one or more methods or technologies in meeting the requirements of their assigned function without some consideration of the application &/or context in which the methods/technologies are expected to perform. For example, there are several situations in which spiking occurs in HWC tests:

1. POHC spiking for DRE demonstration,
2. Acid gas precursor spiking for demonstrating the performance of and setting precursor feed rate limits for a wet scrubber, for example.
3. Ash spiking for similar purposes, and
4. Heavy metal spiking for demonstrating APC performance and feed rate APCS operating limit setting.

As a context for evaluating the performance of these competing technologies in the spiking function, we have somewhat arbitrarily assumed a case in which one metal is spiked into a HWC unit for the purpose of setting a feed rate limit for that metal. Within this general circumstance, each step in the process of designing, conducting and reporting the results a HWC test is identified and an order of magnitude estimate of the uncertainty associated with each step is provided in Table VIII.

Inspection of Table VIII prompts the following observations:

1. Uncertainties associated with the spiking function represent a relatively minor portion of the total uncertainty involved.
2. Within the spiking function, utilization of computer control and demonstrating spiking material composition with *the* Laboratory Standard Method clearly offer advantages in reducing spiking rate uncertainty.
3. Uncertainties associated with: (a) waste stream composition, (b) target spiking rate selection, (c) stack sampling, and (d) sample analysis all represent larger uncertainties than does the spiking function.

Table VIII The HWC Context for Evaluating the Spiking Function

Order of Magnitude Estimates of Uncertainties/Errors in the HWC Process of Setting Constituent Feed Rate Limits				
Areas of Uncertainty	**Mass Flow Meters With Computer Control and Composition by Sx & Analysis Method**	**Weighing Systems with:**		
		Manual Control	**Computer Control**	**[Specie] by Lab Standard**
Imperfect Knowledge of Waste Composition	±10%	±10%	±10%	±10%
Non-Optimum Target Spiking Rate	±30%	±30%	±30%	±30%
Off-set of Average Rate from Target Spiking Rate Due to:				
Imperfect Control	±1%	±3%	±1%	±1%
Imperfect Measurement	±1%	±1%	±1%	±1%
Imperfect Knowledge of [Specie]	±30%	±1%	±1%	±1%
Variations Around Average Spiking Rate Due to:				
Non-Homogeneous Materials	±1%	±1%	±1%	±1%
Imperfect Control	±1%	±1-5%	±1%	±1%
Measurement Uncertainty in Stack Sampling	±10%	±10%	±10%	±10%
Measurement Uncertainty in Sample Analysis	±30%	±30%	±30%	±30%

QUALITATIVE COMPARISON OF WEIGH CELL AND MASS FLOW METER TECHNOLOGIES FOR MEASURING FIELD SPIKING RATE

Up to this point, all discussion has been concerned with quantitative calculations and comparisons of measurement uncertainty between the two most widely used methods of measuring field spiking rate. There are however, other more qualitative attributes of both technologies which recommend their use. These attributes as well as the attributes of computer control and data acquisition are summarized within this section.

Both spiking rate measurement methods benefit similarly from the use of computer based process control and data acquisition technology. These benefits are summarized as follows:

1. The ability to control the spiking rate more uniformly and more closely to the target spiking rate than is possible with manual control.
2. Acquisition, archiving, analysis, and reporting of data in real time.
3. The ability to more rapidly effect spiking rate changes, as needed during miniburns for example.

The relative advantages and disadvantages of the two methods of measuring field spiking rate are summarized in the following table:

Major Features of the Two Field Spiking Rate Measurement Technologies	
Mass Flow Meters	**Weighing Systems**
Advantages:	**Advantages:**
Continuous, Direct Measurement of Flow	Rapid, Tangible Field Demonstration of Accuracy
More Rapid Detection of Rate Changes	Direct Measurement of Mass/Run
Very High Accuracy	Very High Accuracy
Disadvantages:	**Disadvantages:**
Very Difficult to Demonstrate Accuracy in the Field	Indirect Measurement of Rate

CONCLUSIONS:

As a result of the information provided herein, the authors have drawn the following conclusions:

1. Uncertainties associated with the spiking function in a HWC testing program are likely to be a modest part of the total uncertainty associated with the total regulatory/testing process for setting a metal feed rate limit.
2. Both the Mass Flow Meter Method and the Weigh Loss Versus Time Method of measuring field spiking rate provide highly accurate results.
3. The overall lowest level of spiking rate uncertainty is achieved with the Laboratory Standard Method of demonstrating spiking material composition combined with either of the Mass Flow Meter Method or the Weight Loss Versus Time Method of measuring field spiking rate.

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[i] As used herein **Spiking Material (M)** refers to the material which is actually spiked, i.e., a metal solution, a TiO_2 and/or metal dispersion, and/or an individual or a mixture of POHCs. **Spiking Species (S)** refers to the portion of the **Spiking Material** which is of specific interest in meeting the test objectives, i.e., individual metals, ash, individual POHCs, Cl^-, etc.

[ii] Typically, the maximum error ± 0.005 to ±0.01% of the scale's capacity, or in terms of weight, ± 0.05 to ± 0.1 lb for our most frequently used 1,000 lb scales.

[iii] **ESS'** 50 lb field standards are certified annually by the State of Texas to be within ± 0.008 lb (approx. ± 0.02% RE) of NIST Primary Standards.

[iv] The pump through-put to line pressure sensitivity is: -1.5 %/100 psig (Ref 6), i.e., with a constant pump through-put setting, a waste feed line pressure increase of 100 psig would result in a pumping rate decrease of only 1.5%.