John Ruggero
4 56172

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION III
CENTRAL REGIONAL LABORATORY
839 BESTGATE ROAD
ANNAPOLIS, MARYLAND 21401

301-224-2740
FTS-922-3752

DATE July 16, 1985

SUBJECT: QA Review Dioxin; Armour Creek
Case No. 4603-1, DC015301-4

ORIGINAL
(red)

FROM John Austin (3ES21) JA
Chemist

TO John Ruggero (3ES11)

THRU: Patricia J. Krantz (3ES20)
DPOQA

The samples DC015301-DC015304 were successfully analyzed for 2,3,7,8-TCDD. Each sample result is annotated with a "V" to indicate that the results have been verified according to standard Region III Dioxin Quality Assurance Review (see attachment 1). The verified results are attached.

cc: Jack Wolf, West Virginia DNR

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JUL 22 1985
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SERVICES DIVISION

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ATTACHMENT #1

The quality assurance review performed on each data package consists of a review of all calibrations, ion ratios, column performance checks, duplicate precision, surrogate recoveries and a recalculation of all findings.

Isotope dilution selected ion mass spectrometry was used to test for the presence of 2,3,7,8-TCDD. In order for a result to be reported as dioxin, each of the following criteria had to be met:

1. Retention time (at maximum peak height) of the sample component must be within 3 seconds of the retention time of the $13C_{12}$-2,3,7,8-TCDD.

2. The integrated ion currents detected for m/z 257, 320 and 322 must maximize simultaneously.

3. The integrated ion current for each analyte and surrogate compound ion (m/z 257, 320, 322, and 328) must be at least 2.5 times background noise and must not have saturated the detector; internal standard ions (m/z 332 and 334) must be at least 10 times background and must not have saturated the detector.

4. Abundance of integrated ion counts detected for m/z 320 must be $\geq$67% and $\leq$87% of integrated ion counts detected for m/z 322.

If any of the above criteria were not met, the result was not reported as dioxin but flagged with the qualifier "EM", representing the estimated maximum possible concentration, and a foot note to the failed criteria.

CASE For samples in which no unlabeled 2,3,7,8-TCDD was detected, the estimated minimum detectable concentration, which is the concentration required to produce a signal with area (or peak height) of 2.5 times the background signal area (or peak height), was reported.

ORIGINAL
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Dioxin Calibration check

CASE ________ Date ________ Instr. ID________ LAB ________

	Trial 1	Trial 2	Trial 3	NATIVE RF AVERAGE	SD	%RSD	TRIAL 1	TRIAL 2	TRIAL 3	SURROGATE AVERAGE	SD	%RSD
CC1	0.910	0.880	0.830	0.873	0.04	4.58	0.980	0.960	0.910	0.95	0.036	3.789
CC2	0.950	0.990	0.970	0.97	0.02	2.06	0.970	1.020	0.920	0.97	0.05	5.155
CC3	0.970	0.990	0.990	0.98	0.012	1.22	0.960	0.970	0.990	0.973	0.015	1.542
CC4	0.970	1.020	1.020	1	0.029	2.9						
CC5	1.020	0.980	0.970	0.99	0.026	2.63						
			5PT. MEAN	0.9626	0.051	5.3			3PT. MEAN	0.964	0.013	1.349
			LCL	0.86634					LCL	0.8676		
			UCL	1.05886					UCL	1.0604		

+++

SAMPLE #	WEIGHT (gms)	320	322	257	AREA 328c	332	334	257/322	RATIOS 320/322	332/334	SURR%ACC	TCDD(PPB)	D.L. (320)	D.L. (320+3[illegible]
RB	10.00				395272	770448	976832	ERR	ERR	0.789	117.33	0.000	0.000	0.0
DC015301N	10.00	121280	156784	37552	321572	607380	775679	0.240	0.774	0.783	120.60	1.044	2.593	2.6
DC015302D	10.00				108296	209820	276920	ERR	ERR	0.758	115.40	0.000	0.000	0.0
DC015304	10.00	3241870	3994390	972655	1173080	2200910	2821430	0.244	0.812	0.780	121.15	7.484	19.127	18.7
DC015302	10.00	[illegible]			227968	457007	576232	ERR	ERR	0.793	119.46	0.025	0.140	0.0
DC015303	10.00				98448	197096	249960	ERR	ERR	0.799	114.22	0.000	0.000	0.0

ORIGINAL (red)

CASE: 4603-1

FORM B-3 CONTINUING CALIBRATION SUMMARY

INST ID	DATE	TIME	SOL. ID	PEAK AREA (OR HEIGHT) 320	322	323	328	332	334	MEAS. RF NATIVE	MEAN RF NATIVE	MEAS. RF SURR.	MEAN RF SURR.	TCD ISOM RESOL
5	7/9/85	15:05	PC	338432	428912	57088	522527	1118010	1429450	--	--	1.02	0.96	2
5	7/9/85	16:46	CC1	429176	537471	--	311156	2056810	2580820	1.04	0.96	1.10	0.96	--
5	7/9/85	23:00	PC	349768	431952	61584	517776	1132290	1415420	--	--	1.01	0.96	1

Solution ID Codes:

PC = Performance check solution
CC1 = Concentration calibration solution #1 = 0.2 ng/ml
CC2 = Concentration calibration solution #2 = 1.0 ng/ml
CC3 = Concentration calibration solution #3 = 5.0 ng/ml
CC4 = Concentration calibration solution #4 = 20.0 ng/ml
CC5 = Concentration calibration solution #5 = 40.0 ng/ml

ORIGINAL
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FORM B-2 INITIAL CALIBRATION SUMMARY

INST ID	DATE	TIME	SOL. ID	PEAK AREA (OR HEIGHT) 320	322	323	328	332	334	MEAS. RF NATIVE	MEAN RF NATIVE	MEAS. RF SURR.	MEAN RF SURR.	TCDD ISOMERS RESOLUT'NS
5	10/17/84	03:32	PC	27848	38294	4148	31192	66548	83996	--	--	1.02	0.91	1
5	10/17/84	22:23	CC1	55200	74448	--	42576	318808	391376	0.91	0.88	0.98	0.95	--
5	10/18/84	10:49	CC1	40000	54240	--	31288	241680	294424	0.88	0.88	0.96	0.95	--
5	10/18/84	16:20	CC1	20344	23936	--	14720	115912	149312	0.83	0.88	0.91	0.95	--
5	10/18/84	06:15	CC2	303552	396304	--	89184	333112	401704	0.95	0.97	0.97	0.97	--
5	10/18/84	11:25	CC2	315328	404464	--	92352	328496	399520	0.99	0.97	1.02	0.97	--
5	10/18/84	17:28	CC2	141839	175136	--	37648	148928	179048	0.97	0.97	0.92	0.97	--
	10/17/84	23:47	CC3	1406390	1771270	--	145472	291192	360768	0.97	0.98	0.99	0.97	--
5	10/18/84	13:34	CC3	1160000	1459330	--	115920	239680	292016	0.99	0.98	0.97	0.97	--
5	10/18/84	18:07	CC3	1178050	1476660	--	116592	237551	298048	0.99	0.98	0.96	0.97	--
5	10/19/84	10:01	CC4	4151310	5064410	--	40976	208640	267375	0.97	1.00	--	--	--
5	10/19/84	10:45	CC4	2660570	3338030	--	29344	128608	163976	1.02	1.00	--	--	--
5	10/19/84	15:28	CC4	3178470	3956090	--	30920	155760	193856	1.02	1.00	--	--	--
5	10/19/84	13:42	CC5	5299900	6425510	--	59528	132408	169568	0.97	0.99	--	--	--
5	10/19/84	14:18	CC5	6573930	7970910	--	70312	164656	206176	0.98	0.99	--	--	--
5	10/19/84	14:53	CC5	4794460	5809850	--	49296	114440	144240	1.02	0.99	--	--	--
									OVERALL AVERAGE		0.96		0.96	
									OVERALL RSD		9.2		1.4	

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CC4 = Concentration calibration solution #4 = 20.0 ng/ml
CC5 = Concentration calibration solution #5 = 40.0 ng/ml

LAB: COMPUCHEM LABORATORIES, INC. FORM B-1. TCDD DATA REPORT FORM REPORT DATE: 7/11/85

CASE NO. 4603-1 COLUMN: CP SIL 88

EPA SAMPLE NO.	CC SAMPLE NO.	EXTR'N DATE	EXTRA CLEAN UP	ALIQUOT WET WT. (GRAMS)	PPB TCDD MEAS.	PPB TCDD D.L.	GC/MS ANALYSIS INSTR. ID	DATE	TIME	REL. ION ABUND 320/322	REL. ION ABUND 332/334	PPB SURROGATE MEAS.	% RECOV	INTEGRATED PEAK AREA OR HEIGHT 320	322	257	328**	332	334	COMMENTS
RB	23366	7-9-85	Y	10.0	ND	0.04	5	7/9/85	17:54	--	0.79	1.18	118	--	--	--	395272	770448	976832	✓
DC015301N	23325	7-9-85	Y	10.0	1.00	--	5	7/9/85	18:29	0.77	0.78	1.21	121	121280	156784	37552	321572	607380	775679	✓
DC015302D	23333	7-9-85	Y	10.0	ND	0.25	5	7/9/85	19:00	--	0.76	1.16	116	--	--	--	108296	209820	276920	✓
DC015304 *	23309	7-9-85	Y	10.0	7.50	--	5	7/9/85	19:29	0.81	0.78	1.22	122	3241870	3994390	972655	1173080	2200910	2821430	✓
DC015302	23341	7-9-85	Y	10.0	ND	0.10	5	7/9/85	19:59	--	0.79	1.20	120	4940	--	--	237968	457007	576232	✓
DC015303	23358	7-9-85	Y	10.0	ND	0.18	5	7/9/85	20:28	--	0.79	1.15	115	--	--	--	98448	197096	249960	✓

* PE Sample T.V. 8.67
Limits not Available

RB = REAGENT BLANK
P = PARTIAL SCAN/ CONFIRMATORY ANALYSIS
N = NATIVE TCDD SPIKE/FORTIFIED FIELD BLANK
D = DUPLICATE

FB =FIELD BLANK
ND = NOT DETECTED
DL = DETECTION LIMIT

**CORRECTED FOR CONTRIBUTION BY NATIVE TCDD; 0.9% OF m/z 322 SUBTRACTED.
@HEIGHT USED

ORIGINAL
(red)