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

301 224-2740
FTS-922-3752

DATE : November 14, 1984

SUBJECT: Dioxin QA Review Case 3276-5
Monsanto

Monsanto landfill

FROM : John Austin (3ES21) JA
Chemist

Diana Pickens (3ES21) DP
Chemist

TO : Walter Lee (3HW12)
Environmental Scientist

ORIGINAL
(red)

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 meet:

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. Relative abundance of m/z 257 to m/z 322 should be $\geq$20% and $\leq$45%.

5. 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.

For samples in which no unlabled 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.

Attached are the analytical results for each batch of samples and any associated data qualifiers.

JA/jlm

Attachments

Case: 3276-5

Lab: Compuchem

Samples: DC008901-8922

ORIGINAL
(red)

Data qualifiers:

° The following results should be reported as maximum estimated concentrations because all identification criteria were not met:

	TCDD ppb Measured	TCDD ppd Detection Limit	Comment
DC008903	EM	.34	A
DC008912	EM	.53	A
DC008915	EM	.83	A
DC008905	EM	.21	A

A m/z 257/322 out of control.

° Recalculation of the following values found reporting differences:

	Lab Reported TCDD ppb	QA Review TCDD ppb
DC008913	11.7	11.4

LAB: COMPUCHEM LABORATORIES, INC. FORM 8-1. TCDD DATA REPORT FORM REPORT DATE: 10/25/84

CASE NO. 3276-5 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 SURRAGATE MEAS.	PPB SURRAGATE % ACCY	INTEGRATED PEAK AREA OR HEIGHT 320	322	257	328**	332	334	COMMENTS
RB	12591	10/02/84	Y	10.0	ND	0.03	5	10/21/84	13:03	--	0.80	1.09	109	--	--	--	213136	454528	565296	
DC008902D	12369	10/02/84	Y	10.5	ND	0.68	5	10/21/84	14:27	--	0.75	0.83	82.8	--	--	--	7696	20800	27624	
DC008901 *	12344	10/02/84	Y	10.1	3.10	--	5	10/21/84	15:08	0.74	0.78	1.08	108	56208	75608	28568	44831	94560	121984	
DC008903	12385	10/02/84	Y	10.6	~~ND~~ EM	~~0.64~~ .36	5	10/22/84	19:54	--	0.86	0.83	83.3	6744	7976	--	33504	97040	112384	A
DC008906	12419	10/02/84	Y	10.5	ND	0.04	5	10/22/84	21:32	--	0.82	0.99	99.0	--	4432	--	392991	932272	1136210	
DC008909	12443	10/02/84	N	10.0	ND	0.07	5	10/22/84	22:11	--	0.84	0.97	97.1	--	--	--	156607	385000	455167	
DC008911	12468	10/02/84	Y	10.6	ND	0.69	5	10/22/84	22:54	--	0.73	0.78	78.1	--	--	--	9224	26000	35496	
DC008912	12476	10/02/84	Y	10.1	~~ND~~ EM	~~0.91~~ .53	5	10/22/84	23:42	--	0.78	0.78	78.0	4936	7200	--	17584	51360	66016	A
DC008916	12518	10/02/84	Y	10.1	0.12	--	5	10/23/84	00:20	0.86	0.81	0.99	99.1	16544	19240	5344	301178	707727	874832	
DC008917	12526	10/02/84	Y	10.4	ND	0.14	5	10/23/84	01:19	--	0.81	0.99	98.5	--	--	--	164384	390207	479103	
RB	12575	10/02/84	Y	10.0	ND	0.01	3	10/24/84	05:07	--	0.77	1.01	101	3552	--	--	1529650	3276750	4241090	
DC008913	12484	10/02/84	Y	10.3	~~11.7~~ 11.4	--	3	10/24/84	05:57	0.70	0.80	0.99	98.8	1137700	1634130	365160	258228	573752	719848	
DC008915	12500	10/02/84	Y	10.0	~~ND~~ EM	~~0.72~~ .83	3	10/24/84	06:42	--	0.76	1.10	110	25680	33088	--	86744	167904	221648	A
DC008908	12435	10/04/84	Y	10.2	2.80	--	3	10/25/84	00:47	0.72	0.79	0.68	67.8	120672	168064	42624	75767	245072	308530	
DC008922N	12351	10/02/84	Y	10.0	0.80	--	3	10/25/84	02:13	0.68	0.85	0.83	82.8	200666	293323	85522	568896	1568250	1834810	
DC008918	12534	10/02/84	Y	10.4	ND	0.04	3	10/25/84	03:41	--	0.82	0.99	98.7	--	--	7808	577728	1302390	1594690	
DC008919	12542	10/02/84	Y	10.8	ND	0.03	3	10/25/84	04:29	--	0.78	1.04	104	--	--	--	485856	1006870	1297790	
DC008903	12393	10/02/84	Y	10.6	ND	0.23	3	10/25/84	07:29	--	0.77	1.06	106	--	9141	--	102256	206336	268768	
DC008907	12427	10/02/84	Y	10.8	10.4	--	3	10/25/84	08:42	0.75	0.77	1.04	104	7478780	9939980	3024010	1791730	3721950	4813140	
DC008902	12377	10/02/84	Y	10.4	ND	0.16	3	10/25/84	09:27	--	0.83	1.00	99.7	--	9376	--	161480	362754	439071	
DC008914	12492	10/02/84	Y	10.6	ND	0.16	3	10/25/84	10:14	--	0.80	0.92	92.2	--	10688	--	163680	374736	504240	
RB	13235	10/04/84	Y	10.0	ND	0.28	3	10/25/84	11:51	--	0.78	0.99	98.6	--	--	--	70976	156400	199824	

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

A m/z 257/322 out of control

**CORRECTED FOR CONTRIBUTION BY NATIVE TCDD; 0.92 OF m/z 322 SUBTRACTED.
#HEIGHT USED

* PE sample TV = 4.20 ppb

ORIGINAL (red)

LAB: COMPUCHEM LABORATORIES, INC.

FORM B-1. TCDD DATA REPORT FORM

REPORT DATE: 11/05/84

CASE NO. 3276-5

COLUMN: CP SIL 88

EPA SAMPLE NO.	CC SAMPLE NO.	EXTR'N DATE	EXTRA CLEAN UP	ALIQUOT WET WT. (GRAMS)	F -- ME	SIS ------ TIME	REL. ION ABUND 320/322	REL. ION ABUND 332/334	PPB SURRAGATE MEAS.	% ACCY	INTEGRATED PEAK AREA OR HEIGHT 320	322	257	328**	332	334	COMMENTS
RB	13573	10/31/84	Y	10.0	ND	4 10:31	--	0.77	1.01	101	--	417968	--	68630	145696	190112	
DC008910	12450	10/31/84	Y	10.1	ND	4 11:14	--	0.78	0.84	83.7	--	--	--	92236	239040	306208	
DC008921	12567	10/31/84	Y	10.0	ND	4 11:58	--	0.76	1.02	102	--	70800	--	144053	302528	395656	
DC008905	12401	10/31/84	Y	10.1	~~ND~~	4 12:43	--	0.74	1.13	113	5672	8800	--	102896	193168	259328	A
DC008920	12559	10/31/84	Y	10.0	ND	4 13:27	--	0.82	1.17	117	--	154752	--	79104	150960	183192	

A m/z 257/322 out of control

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

ORIGINAL (red)

LAB: COMPUCHEM LABORATORIES, INC. FORM B-1. TCDD DATA REPORT FORM REPORT DATE: 10/25/84

CASE NO. 3276-5 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 SURRAGATE MEAS.	PPB SURRAGATE % ACCY	INTEGRATED PEAK AREA OR HEIGHT 320	322	257	328**	332	334	COMMENTS
RB	12591	10/02/84	Y	10.0	ND	0.03	5	10/21/84	13:03	--	0.80	1.09	109	--	--	--	213136	454528	565296	
DC008902D	12369	10/02/84	Y	10.5	ND	0.68	5	10/21/84	14:27	--	0.75	0.83	82.8	--	--	--	7696	20800	27624	
DC008901	12344	10/02/84	Y	10.1	3.10	--	5	10/21/84	15:08	0.74	0.78	1.08	108	56208	75608	28568	44831	94560	121984	
DC008903	12385	10/02/84	Y	10.6	ND	0.64	5	10/22/84	19:54	--	0.86	0.83	83.3	6744	7976	--	33504	97040	112384	
DC008906	12419	10/02/84	Y	10.5	ND	0.04	5	10/22/84	21:32	--	0.82	0.99	99.0	--	4432	--	392991	932272	1136210	
DC008909	12443	10/02/84	N	10.0	ND	0.07	5	10/22/84	22:11	--	0.84	0.97	97.1	--	--	--	156607	385000	455167	
DC008911	12468	10/02/84	Y	10.6	ND	0.69	5	10/22/84	22:54	--	0.73	0.78	78.1	--	--	--	9224	26000	35496	
DC008912	12476	10/02/84	Y	10.1	ND	0.91	5	10/22/84	23:42	--	0.78	0.78	78.0	4936	7200	--	17584	51360	66016	
DC008916	12518	10/02/84	Y	10.1	0.12	--	5	10/23/84	00:20	0.86	0.81	0.99	99.1	16544	19240	5344	301178	707727	874832	
DC008917	12526	10/02/84	Y	10.4	ND	0.14	5	10/23/84	01:19	--	0.81	0.99	98.5	--	--	--	164384	390207	479103	
RB	125[illegible]5	10/02/84	Y	10.0	ND	0.01	3	10/24/84	05:07	--	0.77	1.01	101	3552	--	--	1529650	3276750	4241090	
DC008913	12484	10/02/84	Y	10.3	11.7	--	3	10/24/84	05:57	0.70	0.80	0.99	98.8	1137700	1634130	365160	258228	573752	719848	
DC008915	12500	10/02/84	Y	10.0	ND	0.72	3	10/24/84	06:42	--	0.76	1.10	110	25680	33088	--	86744	167904	221648	
DC008908	12435	10/04/84	Y	10.2	2.80	--	3	10/25/84	00:47	0.72	0.79	0.68	67.8	120672	168064	42624	75767	245072	308530	
DC008922N	12351	10/02/84	Y	10.0	0.80	--	3	10/25/84	02:13	0.68	0.85	0.83	82.8	200666	293323	85522	568896	1568250	1834810	
DC008918	12534	10/02/84	Y	10.4	ND	0.04	3	10/25/84	03:41	--	0.82	0.99	98.7	--	--	7808	577728	1302390	1594690	
DC008919	12542	10/02/84	Y	10.8	ND	0.03	3	10/25/84	04:29	--	0.78	1.04	104	--	--	--	485856	1006870	1297790	
DC008703	12393	10/02/84	Y	10.6	ND	0.23	3	10/25/84	07:29	--	0.77	1.06	106	--	9141	--	102256	206336	268768	
DC008907	12427	10/02/84	Y	10.8	10.4	--	3	10/25/84	08:42	0.75	0.77	1.04	104	7478780	9939980	3024010	1791730	3721[illegible]50	4813140	
DC008902	12377	10/02/84	Y	10.4	ND	0.16	3	10/25/84	09:27	--	0.83	1.00	99.7	--	9376	--	161480	362754	439071	
DC008914	12492	10/02/84	Y	10.6	ND	0.16	3	10/25/84	10:14	--	80.80	0.92	92.2	--	10688	--	163680	374736	504240	
RB	13235	10/04/84	Y	10.0	ND	0.28	3	10/25/84	11:51	--	0.78	0.99	98.6	--	--	--	70976	156400	199824	

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; 0.92 OF m/z 322 SUBTRACTED.
@HEIGHT USED

ORIGINAL (red)

LAB: COMPUCHEM LABORATORIES, INC. FORM 8-1. TCDD DATA REPORT FORM REPORT DATE: 11/05/84

CASE NO. 3276-5 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 SURRAGATE MEAS.	% ACCY	INTEGRATED PEAK AREA OR HEIGHT 320	322	257	328**	332	334	COMMENTS
RB	13573	10/31/84	Y	10.0	ND	0.27	3	11/01/84	10:31	--	0.77	1.01	101	--	417968	--	68630	145696	190112	
DC008910	12450	10/31/84	Y	10.1	ND	0.24	3	11/01/84	11:14	--	0.78	0.84	83.7	--	--	--	92236	239040	306208	
DC008921	12567	10/31/84	Y	10.0	ND	0.34	3	11/01/84	11:58	--	0.76	1.02	102	--	70800	--	144053	302528	395656	
DC008905	12401	10/31/84	Y	10.1	ND	0.21	3	11/01/84	12:43	--	0.74	1.13	113	5672	8800	--	102896	193168	259328	
DC008920	12559	10/31/84	Y	10.0	ND	0.62	3	11/01/84	13:27	--	0.82	1.17	117	--	154752	--	79104	150960	183192	

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.

ORIGINAL
(red)