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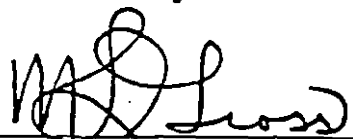
Monsanto Industrial Chemicals

Trace Analysis of TCDD

University of Nebraska
Department of Chemistry
Lincoln, Nebraska 68588

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Submitted by:



M. L. Gross
Director
Midwest Center for
Mass Spectrometry



P. A. Lyon
Assistant Director
Midwest Center for
Mass Spectrometry

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GC/HRMS Analysis

A Kratos MS-5076 ultra high resolution mass spectrometer was used for this analysis. The mass spectrometer was interfaced to a Perkin Elmer Sigma II gas liquid chromatograph. Data acquisition was accomplished with a Nicolet Model 1170 signal averaging computer.

A. Gas Chromatography

The column was a 6' x 1/4" O.D. glass containing a Dow mixed phase packing. Typical operating conditions were: Helium flow rate of 17 cc/min; injector 270° C; column temperature program: 1.5 min at 250° and then ramped at 10° c/min to 300° C and held there until the dioxin has eluted. The GC/MS interface was a simple glass lined stainless steel capillary (no separator) and was held at an average temperature of 250° C. Typical retention time for 2,3,7,8 TCDD was 4.0 minutes (peak width at 10% height approximately 60 seconds).

B. Mass Spectrometer Conditions

The electron impact source was used at 70eV ionizing energy and an accelerating voltage of 8KV. The source was set at 260° C. The instrument was tuned to a resolving power of 10,000 (10% valley definition).

Data were acquired by the standard ion switching feature. The initial run was made monitoring the 320,322 peaks for the 2,3,7,8-TCDD. Then the instrument was adjusted to look at the 286/288 ions for the trichlorodioxins during a second run (retention time 2.0 min). The time required to sample two ions of interest was .8 sec. (.4 sec. per peak). The sweep width was 300 parts-per-trillion ($\pm$ 96 millimass units with the various TCDD signals at the center of the scan). The data were integrated for about 75 sweeps over the retention time of TCDD. The amplifier bandwidth was set at 3K Hertz.

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C. Calculation of Results

For these analyses, a calibration curve was prepared using standard solutions of trichloro and tetrachlorodioxins supplied by Monsanto for this study. From the sample signal intensity, the concentration of dioxin could be determined from the calibration plot. The detection limit was calculated by taking 2.5 times the noise level at 322(288) as the minimum detectable signal. The data in the attached tables summarizes our results.

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Table I

Analysis of Cl₄-dibenzodioxin

by

GLC High Resolution Mass Spectrometry

ID	Conc.(ng/ml)*	Detection Limit (ng/ml)	i _{320/322}
6AM	nd	.07	-
5AM	5	.3	.61
1BM	.064	.02	.82
Method Blank	.35	.09	.96
#2	2600	40	.79
#3	2700	140	.86
	2200	80	.77
#4	6100	450	.72
	1300	20	.77
#5	350	28	.77

*

Many samples showed previously eluting peaks which may be isomers other than 2,3,7,8-TCDD.

- #2, 3, 4, 5 normalized to 1 ml; but is correctly normalized to 5 ml of sample; hence data should be divided by 5.

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Table II

Analysis of Cl₃-dibenzodioxin
by
GLC High Resolution Mass Spectroscopy

ID	Conc. (ng/ml)	Detection Limit (ng/ml)	i 320/322
#2	440	8	.98
#3	2100	35	.99
#4	900	14	.96
#5	130	2	.95
A later eluting peak (possibly another isomer)			
#3	12000	200	.94
#4	30000	400	.96

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