REPORT

DETERMINATION OF THE MASS SPECTRUM OF

TETRABROMOBISPHENOL A

NOTOX Project 292837
NOTOX Substance 100422

1 STATEMENT OF GLP COMPLIANCE

NOTOX B.V., 's-Hertogenbosch, The Netherlands

The study described in this report has been correctly reported and was conducted in compliance with the most recent edition of:

The OECD Principles of Good Laboratory Practice

Which are essentially in conformity with:

The Unites States Food and Drug Administration. Title 21 Code of Federal Regulations Part 58.

The United States Environmental Protection Agency (FIFRA). Title 40 Code of Federal Regulations Part 160.

The United States Environmental Protection Agency (TSCA). Title 40 Code of Federal Regulations Part 792.

Study Director
Drs. H. Stieltjes

Date: 6 July 2000

Head of Chemistry
Dr. Ir. H. Willems

Date: July 07, 2000

2 QUALITY ASSURANCE STATEMENT

NOTOX B.V., 's-Hertogenbosch, The Netherlands

This report was audited by the NOTOX Quality Assurance Unit to ensure that the methods and results accurately reflect the raw data.

The dates of Quality Assurance inspections and audits are given below. During the on-site inspections procedures applicable to this type of study were inspected.

DATES OF QAU INSPECTIONS/AUDITS	REPORTING DATES
On-site inspections	
12 May, 2000	12 May, 2000
Protocol inspection	
19 April, 2000	19 April, 2000
Report audits	
18 May,2000	18 May,2000

Head of Quality Assurance
C.J. Mitchell B.Sc.

Date: 7-7-2000

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4 SUMMARY

A mass spectrum of Tetrabromobisphenol A has been recorded in the negative mode from m/z 30 to 3000.

The molecular ion (Br_4-cluster around m/z 542.7) was clearly present. Fragmentation of this mass confirmed the identity of the substance.

A number of masses below m/z 200 were observed and may have originated from impurities. Also a Br_5-cluster around m/z 605.2 was observed. This corresponds to the replacement of a proton with a Br in the parent molecule.

Deviations from unit masses are caused by the mass defect.

5 PREFACE

Sponsor	Dr. M. Spiegelstein Bromine Science & Environmental Forum (BSEF) BSEF Secretariat co/Burson-Marsteller 118, Avenue de Cortenbergh 1000 Brussels
Study Monitor	Dead Sea Bromine Group Health, Safety and Environment P.O. Box 180 Beer-Sheva 84101
Testing Facility	NOTOX B.V. Hambakenwetering 3 5231 DD 's-Hertogenbosch The Netherlands
Study Director	Drs. H. Stieltjes
Project Number	NOTOX Project: 292837
Study plan	Start : April 19, 2000 End : April 19, 2000 Report : July 6, 2000

6 GUIDELINES

The study procedure described in this protocol is based on the following guideline:

Working Document of the 18th Meeting of competent Authorities.
Conception to Harmonize Requirements for Spectral Data, to be submitted according to Directive 79/831/EEC.

7 ARCHIVING

NOTOX B.V. will archive the following data for at least 10 years:
protocol, report, test substance reference sample and raw data. Thereafter, no data will be withdrawn without the sponsor's written consent.

8 PURPOSE AND PRINCIPLE

The purpose of the study is to record a mass spectrum of the test substance.

9 TEST SUBSTANCE

Identification	Tetrabromobisphenol A
Description	White crystalline powder
Batch	Not indicated
Purity	Described in NOTOX project 292815
Test substance storage	At room temperature in the dark
Stability under storage Conditions	Stable
Expiry date	10 April 2001 (allocated by NOTOX)

The sponsor is responsible for all test substance data unless determined by NOTOX .

Note: the test substance consisted of a composite of Tetrabromobisphenol A samples received from three manufactures. The materials identities and dates received from each of the manufactures is given below:

Manufacturer	Batch	Date received	NOTOX substance
Albemarle Corp	8721-33	10 April 2000	100737
Bromine Compounds	20001096	18 April 2000	82512/B
Great leaks Chemical Corp.	20001096	18 April 2000	82512/B

An equal quantity of each of the manufacturer's Tetrabromobisphenol A material was placed in a 500-ml glass jar with screw cap. The jar was placed on a reciprocating shaker for two hours.

10 PERFORMANCE OF THE TEST

10.1 Experimental procedures

Calibration and performance of the MS-system were carried out using standard procedures described in the corresponding manual and SOP.

10.2 Equipment

The sample was measured with MS/MS.

Mass spectrometery was performed with a Perkin Elmer SCIEX API 300 mass spectrometer, equipped with a Turbo-ion-spray source. Mass spectra were recorded with Analyst™ 1.0.

10.3 Test procedure

The test substance was dissolved in methanol (HPLC-grade, Labscan Limited Co., Dublin, Ireland) at a concentration of 1.026 g/l. This solution was diluted to 10.00 mg/l with methanol/water 90/10 with an overall ammoniumacetate concentration of 2.5 mM (pH=6.8)(fractopur, Merck, Darmstadt, Germany). The resulting solution was transferred to the MS/MS using a syringe pump with a flow of 10 µl/min.

A mass spectrum in the mass of m/z 30 to 3000 (ionspray voltage (IS): -4000, entrance potential (EP): 7.0, focusing potential (FP): -340, declustering potential (DP): -111) was recorded in the negative ion mode. Also, at the same conditions a mass spectrum of the blank solvent was recorded.

Product-ion spectra of the main m/z signals were recorded.

11 RESULTS

11.1 Calibration

The mass accuracy was between –0.035 and 0.006 dalton in the range from m/z 59 to 2242.6. The resolution (peak width at half height) was 0.747 or lower in the same range. This is within specifications (accuracy must be < 0.1 dalton, resolution must be < 0.8 dalton).

11.2 Mass spectra

The structural formula of Tetrabromobisphenol A is shown in Figure 1.

In the positive mode (spectrum not shown) no significant signals were observed. The negative mode mass spectrum of Tetrabromobisphenol A recorded from m/z 30 to 3000, including a detailed mass range is shown in Figure 2a. The spectrum of the blank solvent (Figure 2b) did not reveal any significant masses. Mass 59.0 represents the acetate ion.

A Product-Ion spectrum of the most significant mass is shown in Figure 3.

11.3 Identification

Deviations of the observed m/z values from unit masses are caused by the mass defect.

Masses	possible assignment
<200	No assignments could be given. The blank spectrum does not show those signals. They might originate from impurities that are easy to ionize. Their high abundance does not reflect the amount of imputities.
539.0 to 546.9	$(M-H)^-$, showing a typical Br_4-isotope pattern with 542.9 as most abundant mass: Two ^{79}Br and two ^{81}Br isotopes.
599 to 609	Both mass and isotope pattern indicate 5 Br-atoms in the molecule. Probably a bromo atom replaces a proton.

From mass 542.9 (referred to as mass 542.7) a product-ion spectrum was recorded (Figure 3).

- Mass 78.7 and 80.8 refer to the Bromine ion (both isotopes).
- The triplet at mass 290.8 refers to the molecule that has lost a dibromophenol group.
- No assignment could be given for masses 416.6, 417.6 and 419.7. The ratio does not reflect any pattern for bromo-containing substances.
- Mass 445.6 and 447.9 refer to the molecule with the loss of one bromo atom.

FIGURE 1: STRUCTURAL FORMULA OF TETRABROMOBISPHENOL A

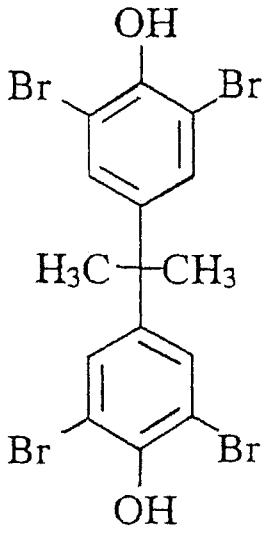

FIGURE 2a: NEGATIVE ION MASS SPECTRUM OF TETRABROMOBISPHENOL A

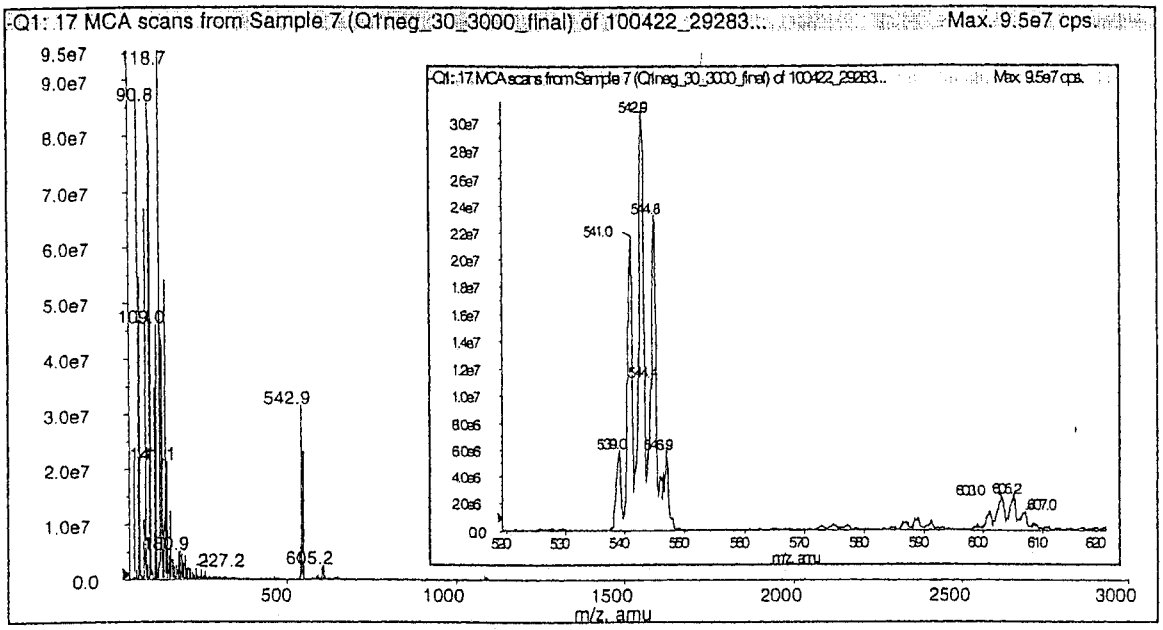

FIGURE 2b: NEGATIVE ION MASS SPECTRUM OF BLANK SOLVENT

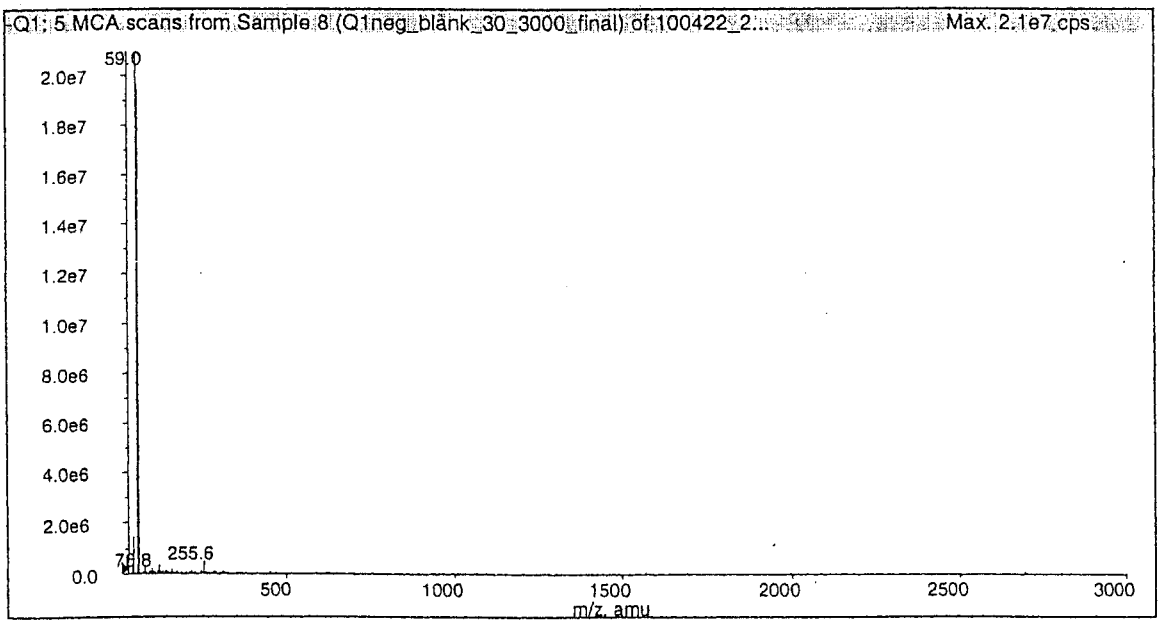

FIGURE 3: PI SPECTRUM OF MASS 542.7