

Chemical Name: Tetra bromobisphenol A  
Trade Name(s): BA-59P  
CAS No: 79-94-7  
Lab Study ID No: WILDLIFE # 439C-128 (Vapor Pressure Determination)

### FINAL REPORT ACTION ITEM CHECK-OFF LIST

☒ Reviewed for possible:

- ☐ FIFRA 6 (a) (2) and/or
- ☒ TSCA Section 8 (e) reporting

☐ Copy of FIFRA 6 (a) (2) and/or TSCA Section 8 (e) letter to the following Agency(ies), if applicable:

- ☐ EPA-FIFRA
- ☐ EPA-TSCA
- ☐ California [FIFRA 6 (a) (2)s]
- ☐ Other States [FIFRA 6 (a) (2)s]: \_\_\_\_\_

☐ Confidentiality Statement page addressed, signed, and dated in FIFRA reports

☐ GLP Compliance page signed and dated in FIFRA reports

☐ Flagging Statement page addressed, signed and dated in FIFRA reports

☒ Copy of report submitted to the Agency(ies) in conjunction and/or support of one or more of the following:

- ☐ TSCA Consent Order/Agreement
- ☐ FIFRA Registration or Re-registration
- ☐ California Registration
- ☐ EU Notification
- ☐ Japanese MITI Notification
- ☐ Japanese MAFF Notification
- ☐ Canadian (DSL) Notification
- ☐ FIFRA 6 (a) (2) Submission
- ☐ TSCA 8 (e) Submission
- ☐ TSCA 8 (d) Data-Call-In
- ☐ PMN Submission

☒ Other: EU Risk Assessment (Steve Dungey of UK)

☒ All information regarding the chemical and the study report entered into the IUCLID Toxicity Data Base

☒ Study Report reviewed for MSDS information *Copied to product file for addition to MSDS @ next update JSD 12/2/01*

☒ Copy of Cover & Summary report pages to Business Unit MSDS information center (domestic and international)

*Director Schmunn*

☐ Active Study file merged with final report in Regulatory Affairs file room

DETERMINATION OF THE VAPOR PRESSURE OF TETRABROMOBISPHENOL A  
USING THE SPINNING ROTOR GAUGE METHOD

WILDLIFE INTERNATIONAL, LTD. PROJECT NUMBER: 439C-128

OECD Guideline for Testing of Chemicals, 104, *Vapor Pressure Curve*  
U.S. EPA OPPTS 830 7950 *Vapor Pressure*

AUTHORS:

Frank J. Lezotte, B.S.  
Willard B. Nixon, Ph.D.

STUDY INITIATION DATE: August 28, 2001

STUDY COMPLETION DATE: October 18, 2001

SUBMITTED TO:

American Chemistry Council's  
Brominated Flame Retardant Industry Panel  
1300 Wilson Boulevard  
Arlington, VA 22209

*Wildlife International, Ltd.*

8598 Commerce Drive  
Easton, Maryland 21601  
USA  
1 (410) 822-8600

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**GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT**

SPONSOR: American Chemistry Council's  
Brominated Flame Retardant Industry Panel

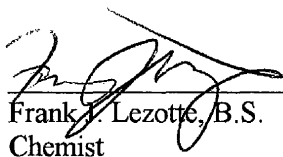
TITLE Determination of the Vapor Pressure of Tetrabromobisphenol A Using the Spinning  
Rotor Gauge Method

WILDLIFE INTERNATIONAL, LTD. PROJECT NUMBER: 439C-128

STUDY COMPLETION: October 18, 2001

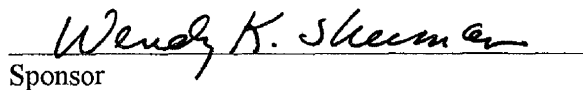
The study was conducted in compliance with Good Laboratory Practice Standards as published by the U.S. Environmental Protection Agency, 40 CFR Part 160, 17 August 1989 and OECD Principles of Good Laboratory Practice (ENV/MC/CHEM (98) 17).

STUDY DIRECTOR:

  
Frank J. Lezotte, B.S.  
Chemist

10-18-01  
DATE

SPONSOR APPROVAL:

  
Sponsor

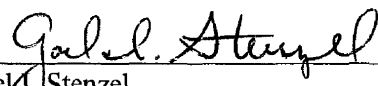
10/19/01  
DATE

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**QUALITY ASSURANCE STATEMENT**

This study was examined for compliance with Good Laboratory Practice Standards as published in the OECD Principles of Good Laboratory Practice, (ENV/MC/CHEM (98)17). The dates of all inspections and audits and the dates that any findings were reported to the Study Director and Laboratory Management were as follows:

ACTIVITY:	DATE CONDUCTED:	DATE REPORTED TO:	
		STUDY DIRECTOR:	MANAGEMENT.
Offset Measurement	August 28, 2001	August 28, 2001	August 31, 2001
Vapor Pressure Measurement	September 11, 2001	September 11, 2001	September 25, 2001
Data and Draft Report	September 26, 2001	September 26, 2001	September 27, 2001
Final Report	October 18, 2001	October 18, 2001	October 18, 2001

  
\_\_\_\_\_  
Joel I. Stenzel  
Systems Analyst

10/18/01  
DATE

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REPORT APPROVAL

SPONSOR. American Chemistry Council's  
Brominated Flame Retardant Industry Panel

TITLE: Determination of the Vapor Pressure of Tetrabromobisphenol A Using the Spinning Rotor  
Gauge Method

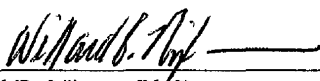
WILDLIFE INTERNATIONAL, LTD. PROJECT NUMBER. 439C-128

STUDY DIRECTOR:

  
\_\_\_\_\_  
Frank J. Lezotte, B.S.  
Chemist

10-18-01  
\_\_\_\_\_  
DATE

MANAGEMENT.

  
\_\_\_\_\_  
Willard B. Nixon, Ph.D.  
Director of Analytical Chemistry

10/18/01  
\_\_\_\_\_  
DATE

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**SUMMARY**

SPONSOR:	American Chemistry Council's Brominated Flame Retardant Industry Panel
SPONSOR'S REPRESENTATIVE	Ms. Wendy Sherman
LOCATION OF STUDY, RAW DATA AND A COPY OF THE FINAL REPORT	Wildlife International, Ltd. Easton, Maryland 21601

WILDLIFE INTERNATIONAL, LTD. PROJECT NUMBER:	439C-128
TEST SUBSTANCE.	Tetrabromobisphenol A
STUDY:	Determination of the Vapor Pressure of Tetrabromobisphenol A Using the Spinning Rotor Gauge Method
TEST DATES	Experimental Start – August 28, 2001 Experimental Termination – September 12, 2001

SUMMARY	The vapor pressure of Tetrabromobisphenol A was determined to be $< 1.9 \times 10^{-5}$ Pa at 20°C using the spinning rotor gauge method.
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## INTRODUCTION

The vapor pressure of Tetrabromobisphenol A (TBBPA) was determined using the spinning rotor gauge method. The study was conducted by Wildlife International, Ltd. for American Chemistry Council's Brominated Flame Retardant Industry Panel at the Wildlife International, Ltd. analytical chemistry facility in Easton, Maryland. Tests were performed following procedures in the OECD Guideline for Testing of Chemicals, 104, *Vapor Pressure Curve* (1) and Product Properties Test Guidelines, OPPTS 830.7950, *Vapor Pressure* (2). Vapor pressure analyses were conducted from August 28 to September 12, 2001. Raw data generated by Wildlife International, Ltd. and a copy of the final report are filed under Project Number 439C-128 in archives located on the Wildlife International, Ltd. site.

## OBJECTIVE

The objective of this study was to determine the vapor pressure of TBBPA using the spinning rotor gauge method at approximately 20 °C.

## EXPERIMENTAL DESIGN

The spinning rotor gauge (SRG) system was used to determine vapor pressure. The SRG measured the rotational frequency of a stainless steel ball that was magnetically suspended within a vacuum chamber. In the presence of a sample, the deceleration rate of ball rotation was proportional to the vapor pressure of the sample. Initially, the background pressure and out-gassing rate of an empty sample chamber were determined a minimum of two times in the absence of sample. The vapor pressures of a reference material and the test material were then sequentially determined at  $20 \pm 1$  °C by measuring the pressure increase over background with the respective material loaded in the test chamber.

## MATERIALS AND METHODS

This study was conducted following procedures outlined in the protocol "Determination of the Vapor Pressure of Tetrabromobisphenol A Using the Spinning Rotor Gauge Method". The protocol was based on procedures found in OECD Guidelines for Testing of Chemicals, Method 104 (1), and U.S. EPA Product Properties Test Guidelines, OPPTS 830.7950 (2).

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**Test Substance**

The test substance consisted of a composite of Tetrabromobisphenol A samples received from three manufacturers. The material's identity and date received from each of the manufacturers is given below

<u>Manufacturer</u>	<u>Lot/Batch</u>	<u>Date Received</u>	Wildlife International, Ltd <u>Identification Number</u>
Albemarle Corporation	25938C-1	July, 27 2000	5318
Great Lakes Chemical Corporation	008JG21C	July 25, 2000	5315
Bromine Compounds Ltd.	000135	August 17, 2000	5354

The composite test substance was assigned Wildlife International, Ltd. identification number 5381 and was stored under ambient conditions. The composite test substance was shipped to Albemarle Corporation for characterization and purity analyses (Appendix 1). The conclusion of the characterization was that the test article was Tetrabromobisphenol A with a purity of 98.91%.

**Reference Substance**

The hexachlorobenzene reference substance was received from Aldrich Chemical Company on February 10, 1997, and was assigned Wildlife International, Ltd identification number 3948 upon receipt. The reference substance, a powder, was identified as: Hexachlorobenzene, catalog no 17105-0; Lot Number 04416KY; CAS Number 118-74-1. The reference substance had a reported purity of 99% and was stored under ambient conditions.

**System Configuration**

The high vacuum system consisted of a vacuum chamber configured with a mechanical vacuum pump (Leybold TRIVAC S 1,6 B), a turbomolecular pump (Leybold TURBOVAC 50), a spinning rotor gauge (Leybold VISCovac VM 212), all-metal isolation valves, and miscellaneous vacuum fittings required to assemble the components. The system also incorporated a pair of capacitance manometers, with an associated controller, for higher-level vapor pressure work. The capacitance manometers were not required in the present study. The valves and metal tubing of the high vacuum system were insulated with foam. The spinning rotor gauge controller was connected to a computer

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via the serial interface (RS-232 port). A diagram of the apparatus configuration is presented in Figure 1.

The sample chamber consisted of a Pyrex tube (~2.5 cm O.D. X 12 cm) joined to a quick-fit type metal flange (NW-25). Empty sample chambers were used for background measurement. The temperature of the sample chamber was maintained at  $20 \pm 1^\circ\text{C}$  during pressure measurements. A refrigerated recirculator, attached to a copper water jacket, regulated the temperature of the sample chamber. The temperature of the sample chamber was periodically monitored using a digital thermometer.

### **Test Procedures**

The entire system was initially evacuated to achieve the ultimate pressure at the target operating temperature for the study ( $20^\circ\text{C}$ ). Under these conditions, the background deceleration rate (DCR) of the ball was monitored for at least thirty minutes to establish an offset value (background correction) for subsequent measurements with reference or test substance present. The parameters of the spinning rotor gauge were set to record a deceleration measurement every 30 seconds. Once obtained, the offset value (expressed as mean deceleration rate) was entered into the spinning rotor gauge controller. Typical experimental parameters for SRG measurements are summarized in Table 1.

For each sample type (reference and test substance), a background vapor pressure, due to a slow linear increase in pressure due to out-gassing and permeation, was determined a minimum of three times at  $20 \pm 1^\circ\text{C}$  using the appropriate molecular weight parameter (see Table 1). The sample chamber was evacuated to achieve a steady-state pressure. The mean and standard deviation of the steady state pressure were used as an indication of equilibration. After the system was pumped to a relatively stable pressure, the background pressure was recorded. Then the isolation valve between the vacuum pumps and the sensor head was closed and the pressure increase was monitored until a slow linear pressure increase was recorded.

A one-gram sample of hexachlorobenzene was used as a vapor pressure reference to verify the accuracy of the system configuration. The vapor pressure of hexachlorobenzene measured by the

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SRG method was compared to published values of  $2.6 \times 10^{-3}$  Pa @ 20°C by the gas saturation method and  $1.1 \times 10^3$  Pa @ 20°C by the vapor pressure balance method (2). A molecular weight of 284.78 was entered into the SRG program for the vapor pressure measurement of hexachlorobenzene.

A one-gram sample of the test substance, TBBPA, was used for vapor pressure determination. A molecular weight of 543.9 was entered into the SRG program for the vapor pressure measurement of TBBPA.

### Calculations

The pressure increase data were analyzed using least-squares-fit linear regression analysis. A line was fit to the portion of the data that was determined to have achieved equilibrium pressure with the sample. The slope and intercept were calculated for each measurement. The out-gassing rate was determined from the slope of the line and the vapor pressure was determined from the intercept value.

## RESULTS AND DISCUSSION

The offset value was  $9.11 \times 10^{-8}$  s<sup>-1</sup> based on the average deceleration rate measured with an empty sample chamber.

Individual and mean measured vapor pressures (intercept of linear regression function) for the reference and test substance are presented in Table 2. The corresponding outgas rates (slope of linear regression function) are presented in Table 3.

A Limit of Detection (LOD) and Limit of Quantitation (LOQ) was determined for the reference and test substance. The LOD was defined as three times the standard deviation for the vapor pressure measurements of each substance's corresponding blank ( $1.31 \times 10^{-5}$  Pa for hexachlorobenzene;  $3.57 \times 10^{-6}$  Pa for TBBPA). The LOQ was defined as ten times the standard deviation for the vapor pressure measurements of each substance's corresponding blank ( $4.36 \times 10^{-5}$  for hexachlorobenzene;  $1.19 \times 10^{-5}$  for TBBPA).

The mean background vapor pressure of a blank tube using hexachlorobenzene parameters was  $< 4.36 \times 10^{-5}$  Pa @ 20°C. The mean measured vapor pressure of hexachlorobenzene by the SRG

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method was  $5.12 \times 10^{-4}$  Pa @ 20°C. This vapor pressure was consistent with published values (2). The measured values for the vapor pressure measurement of hexachlorobenzene and the associated blank are presented in Table 2. Representative vapor pressure plots for hexachlorobenzene and the associated blank are presented in Figure 2.

The mean background vapor pressure of a blank tube using TBBPA parameters was  $< 1.19 \times 10^{-5}$  Pa @ 20°C. The mean measured vapor pressure of TBBPA by the SRG method was  $< 1.19 \times 10^{-5}$  Pa @ 20°C. The measured values for the vapor pressure measurement of TBBPA and the associated blank are presented in Table 2. Representative vapor pressure plots for TBBPA and the associated blank are presented in Figure 3.

Estimations of the magnitude of vapor pressure increase with temperature using the appropriate equations found in the OECD Guideline 104 (1) indicated that additional determinations at elevated temperatures were not warranted. The temperature range suggested by the guideline (0 to 50°C) would not significantly raise the vapor pressure.

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**REFERENCES**

1. **Organisation for Economic Cooperation and Development.** 1995. Guideline for Testing of Chemicals, Method 104, "Vapour Pressure Curve"
2. **U.S. Environmental Protection Agency.** 1996 Product Properties Test Guidelines, OPPTS 830.7950, Vapor Pressure. Washington, D.C
3. **Leybold AG.** Operating Instructions, VISCovac VM 212 Research Facility GmbH, Jülich, F.R.G.

Table 1

Typical Operating Parameters for Pressure Measurements by Spinning Rotor Gauge (3)

PRESSURE GAUGE: Leybold VISCOVAC VM 212 Gauge Controller Leybold VISCOVAC VK201 Gauge Head		
<u>Program parameter</u>	<u>Setting</u>	<u>Description (1)</u>
DISPLAY.	DCR or PA	Deceleration Rate or Pascal.
MTIME:	30	Measurement time in seconds
OFFSET.	$8.23 \times 10^{-8} \text{ s}^{-1}$	Mean DCR for empty sample tube
TEMP:	293	Temperature in °K (273 + °C).
MOLWT.	See footnote 1	Molecular weight of measured gas, if known.
VISC.	See footnote 2	Viscosity of measured gas in Pa·s, if known
SIGMA.	1.000	Describes properties of ball surface.
BDENS	$7.72 \times 10^3$	Density of stainless steel in kg/m <sup>3</sup>
BDIAM:	4.500	Ball diameter in mm.
LIMITS:	OFF	Built-in triggering relays remain in rest position.
OUTPUT	OFF	Analog output is passive.
PRINT	CONT	Output to printer port is active.
INTVL:	0	Printout interval in minutes Zero switches this mode off
SAMPLE:	10	Number of values to print out in one interval of SAMP or STAT modes. Not used (CONT mode)

<sup>1</sup> Effective molecular weight of air is 28.96  
Molecular weight of hexachlorobenzene is 284.78  
Molecular weight of TBBPA is 543.9

<sup>2</sup>  $1.82 \times 10^{-5}$ , the viscosity of air at 20°C, was entered. Value entered for viscosity does not significantly influence measured values < 1 Pa. A value of zero turns linearization off.

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Table 2

Vapor Pressure Measurements of  
Hexachlorobenzene and TBBPA by the Spinning Rotor Gauge Method

Sample	Measured Vapor Pressure <sup>1</sup> , Pa	Mean Vapor Pressure <sup>1</sup> , Pa	Standard Deviation
Blank	$1.87 \times 10^{-5}$	$<4.36 \times 10^{-5}$	$4.36 \times 10^{-6}$
(Hexachlorobenzene parameters)	$1.06 \times 10^{-5}$		
	$1.08 \times 10^{-5}$		
	$8.99 \times 10^{-6}$		
Hexachlorobenzene	$5.30 \times 10^{-4}$	$5.12 \times 10^{-4}$	$1.54 \times 10^{-5}$
	$5.05 \times 10^{-4}$		
	$5.02 \times 10^{-4}$		
Blank	$7.05 \times 10^{-6}$	$<1.19 \times 10^{-5}$	$1.19 \times 10^{-6}$
(TBBPA)	$4.93 \times 10^{-6}$		
	$5.06 \times 10^{-6}$		
TBBPA	$<1.19 \times 10^{-5}$	$<1.19 \times 10^{-5}$	N/A
	$<1.19 \times 10^{-5}$		
	$<1.19 \times 10^{-5}$		

<sup>1</sup> The Limit of Quantitation for vapor pressure measurements with the SRG was defined as ten times the standard deviation for the vapor pressure measurements of each substance's corresponding blank =  $4.36 \times 10^{-5}$  Pa for Hexachlorobenzene;  $1.19 \times 10^{-5}$  for TBBPA.



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Table 3

Out-gassing Rates of  
Hexachlorobenzene and TBBPA by the Spinning Rotor Gauge Method

Sample	Measured Out-gassing Rate, Pa/s	Mean Out-gassing Rate, Pa/s	Standard Deviation
Blank (Hexachlorobenzene parameters)	$3.30 \times 10^{-8}$	$2.46 \times 10^{-8}$	$5.67 \times 10^{-9}$
	$2.27 \times 10^{-8}$		
	$2.13 \times 10^{-8}$		
	$2.12 \times 10^{-8}$		
Hexachlorobenzene	$7.43 \times 10^{-8}$	$5.04 \times 10^{-8}$	$2.07 \times 10^{-8}$
	$3.76 \times 10^{-8}$		
	$3.94 \times 10^{-8}$		
Blank (TBBPA)	$1.73 \times 10^{-8}$	$1.57 \times 10^{-8}$	$1.40 \times 10^{-9}$
	$1.51 \times 10^{-8}$		
	$1.47 \times 10^{-8}$		
TBBPA	$7.69 \times 10^{-8}$	$7.62 \times 10^{-8}$	$2.68 \times 10^{-9}$
	$7.32 \times 10^{-8}$		
	$7.84 \times 10^{-8}$		

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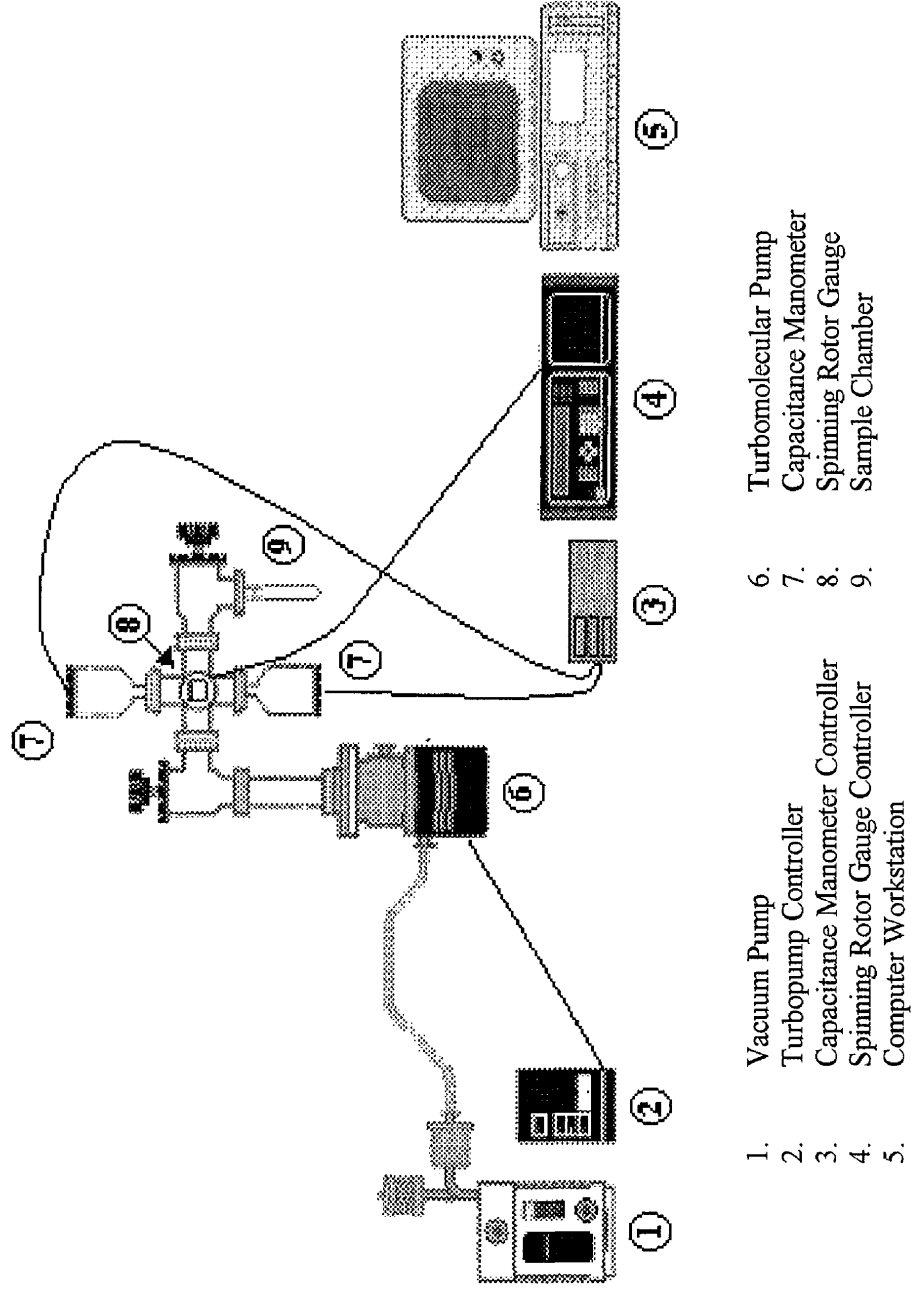
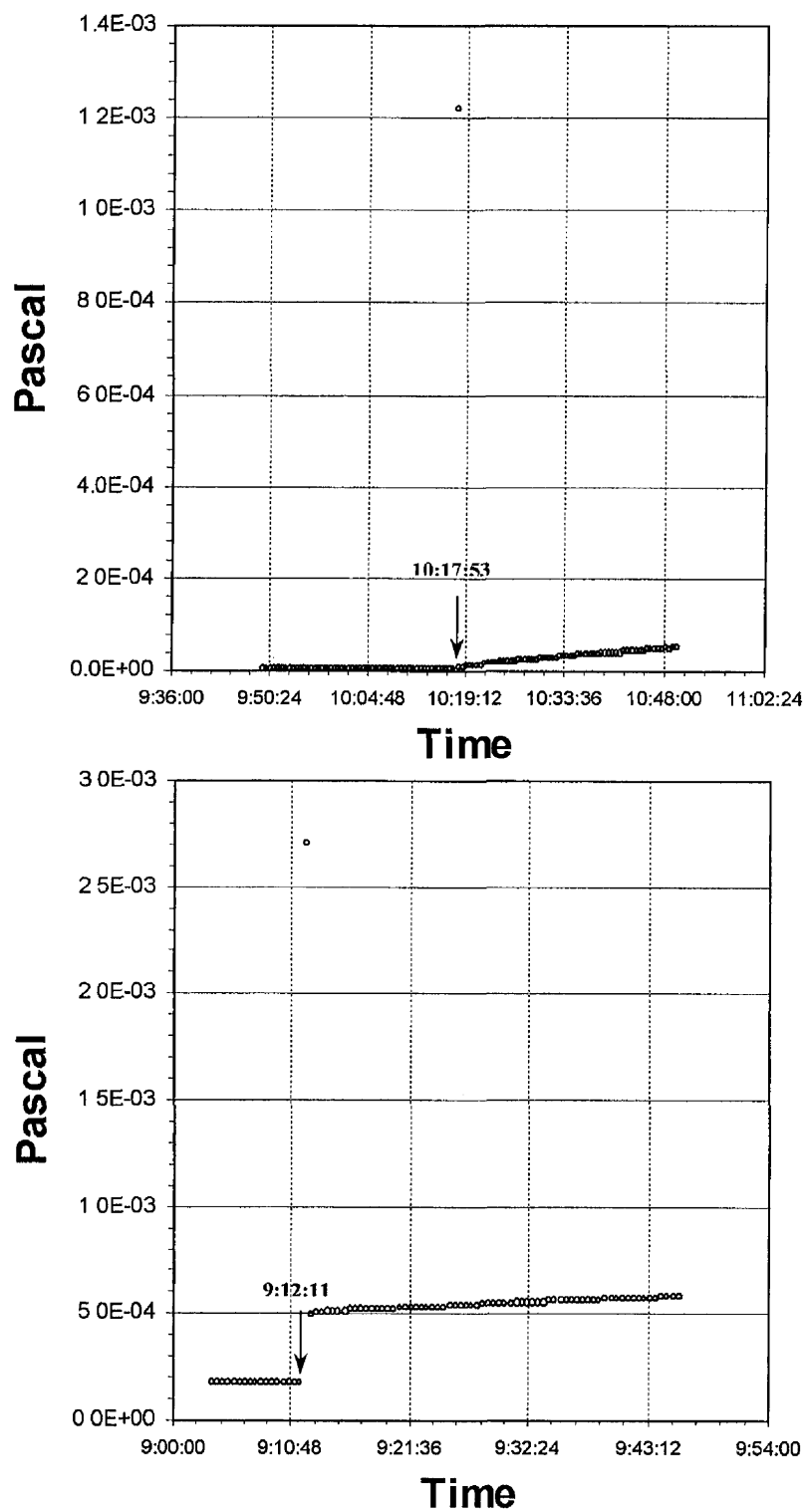


Figure 1. Diagram of high vacuum system configuration.

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**Figure 2.** Representative vapor pressure plots for hexachlorobenzene (BOTTOM) and the associated blank (TOP). Arrows indicate time of valve closing (vacuum isolation).

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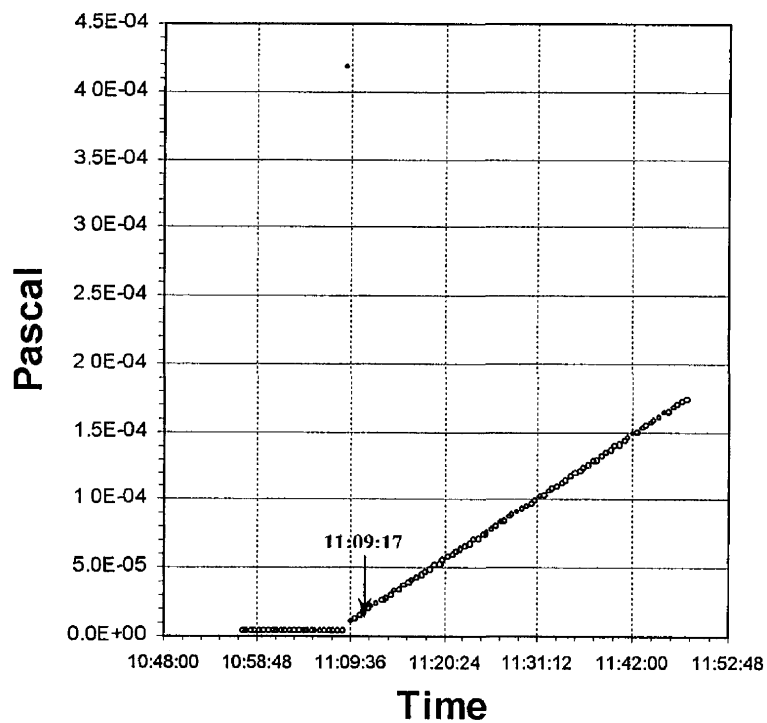
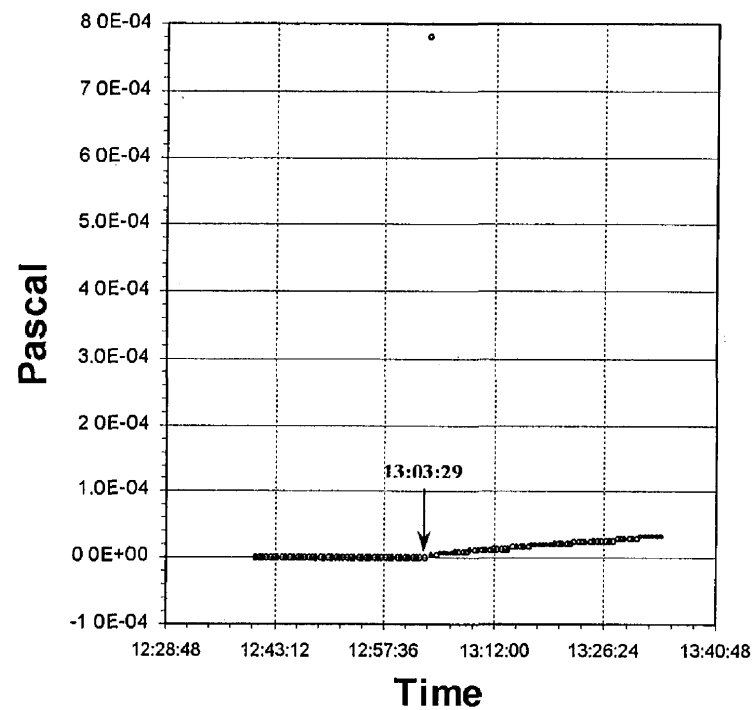


Figure 3. Representative vapor pressure plots for TBBPA (BOTTOM) and the associated blank (TOP). Arrows indicate time of valve closing (vacuum isolation).

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**Appendix 1**

Protocol and Protocol Amendment

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PROTOCOL

DETERMINATION OF THE VAPOR PRESSURE OF TETRABROMOBISPHENOL A  
USING THE SPINNING ROTOR GAUGE METHOD

OECD Guideline for Testing of Chemicals, 104  
Vapor Pressure Curve

Submitted to

American Chemistry Council's  
Brominated Flame Retardant Industry Panel  
1300 Wilson Boulevard  
Arlington, Virginia 22209

*Wildlife International, Ltd.*

8598 Commerce Drive  
Easton, Maryland 21601  
(410) 822-8600

August 27, 2001

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Wildlife International, Ltd.

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DETERMINATION OF THE VAPOR PRESSURE OF TETRABROMOBISPHENOL A  
USING THE SPINNING ROTOR GAUGE METHOD

SPONSOR: American Chemistry Council's  
Brominated Flame Retardant Industry Panel  
1300 Wilson Boulevard  
Arlington, Virginia 22209

SPONSOR'S REPRESENTATIVE: Ms. Wendy Sherman

TESTING FACILITY: Wildlife International, Ltd.  
8598 Commerce Drive  
Easton, Maryland 21601

STUDY DIRECTOR: Frank Lezotte  
Chemist

LABORATORY MANAGEMENT: Willard B. Nixon, Ph.D.  
Manager of Analytical Chemistry

FOR LABORATORY USE ONLY

Proposed Dates:	
Experimental Start Date: <u>8-28-01</u>	Experimental Termination Date: <u>9-30-01</u>
Project No.: <u>439C-128</u>	
Test Substance No.: <u>5381</u>	

PROTOCOL APPROVAL

  
STUDY DIRECTOR

8-28-01  
DATE

  
LABORATORY MANAGEMENT

8/28/01  
DATE

  
SPONSOR'S REPRESENTATIVE

8/28/01  
DATE

PROTOCOL NO.: 439/082701/104/SUB439

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**Wildlife International, Ltd.**

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**INTRODUCTION**

Wildlife International, Ltd. will determine the vapor pressure of the test substance, Tetrabromobisphenol A (TBBPA), using the spinning rotor gauge method. The study will be conducted at the Wildlife International, Ltd. analytical chemistry facility in Easton, Maryland. The study will be performed following procedures in the OECD Guideline for Testing of Chemicals, 104, *Vapor Pressure Curve* (1) and Product Properties Test Guidelines, OPPTS 830.7950, *Vapor Pressure* (2). Raw data for all work performed at Wildlife International, Ltd. and a copy of the final report will be filed by project number in archives located at Wildlife International, Ltd. or at an alternative location specified in the final report.

**OBJECTIVE**

The objective of this study is to determine the vapor pressure of TBBPA using the spinning rotor gauge method at approximately 20° C.

**EXPERIMENTAL DESIGN**

The spinning rotor gauge (SRG) method described in this protocol is applicable to both liquids and solids with vapor pressures in the approximate range of 0.5 to 10<sup>-4</sup> Pascal (Pa). Measurement is initiated with a determination of the system background pressure and out-gassing rate in the absence of sample. The background pressure and out-gassing rate are determined a minimum of two times. The vacuum system and SRG are then exposed to the test substance, maintained at a temperature of 20° ± 1.0°C, until a steady state pressure is obtained. The sample and SRG are then isolated from the vacuum pumps and the pressure increase is monitored by the SRG at 30-second intervals. The vapor pressure is determined from the intercept of the line fit to the pressure increase data upon isolating the sample and SRG from the vacuum pumps.

**MATERIALS AND METHODS****Test Substance**

The test substance consisted of a composite of HBCD samples received from three manufacturers. The material's identity and date received from each of the manufacturers is given below:

<u>Manufacturer</u>	<u>Lot/Batch</u>	<u>Date Received</u>	<u>Wildlife International Ltd. Identification Number</u>
Great Lakes Chemical Corporation	008JG21C	July 25, 2000	5381
Albemarle Corporation	25938C-1	July 27, 2000	5318
Bromine Compounds, Ltd.	000135	August 17, 2000	5354



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## *Wildlife International, Ltd.*

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The composite test substance was assigned Wildlife International Ltd. identification number 5381 and was stored under ambient conditions.

The Sponsor is responsible for all information related to the test substance and agrees to accept any unused test substance and/or test substance containers remaining at the end of the study.

### **Apparatus**

The high vacuum system consists of a vacuum chamber configured with a mechanical pump, a turbomolecular pump, a spinning rotor gauge, isolation valves, a sample chamber and miscellaneous vacuum fittings. The system configuration and components may be adapted as necessary to achieve high vacuum and sufficiently stable pressure readings to conduct the study. The system configuration will be described in the final report.

### **Test Procedure**

Prior to analysis of the test substance, the vacuum system, including the sample chamber, will be evacuated to achieve ultimate vacuum with no reference material or test substance present in the sample chamber. The system may be heated, if necessary, to achieve adequate vacuum.

An offset value (background deceleration rate) for the spinning rotor gauge will be determined at the system's ultimate pressure at the operating temperature(s) used for the study. The sample chamber will be controlled at  $20^{\circ} \pm 1^{\circ}\text{C}$  during vapor pressure analysis. The rest of the apparatus may be at or above the target temperature.

The background pressure and the rate of pressure increase due to out-gassing and permeation, using the parameters for air, will be determined at least three times at  $20^{\circ} \pm 1^{\circ}\text{C}$ . The steady state pressure of the vacuum system and the rate of pressure increase due to out-gassing and permeation in the presence of the test material will be determined at least three times at  $20^{\circ} \pm 1^{\circ}\text{C}$ . Background pressure and pressure changes will each be measured for at least five minutes prior to and after cycling the isolation valve to the pump.

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*Wildlife International, Ltd.*

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A vapor pressure reference compound may be used to confirm the system configuration and the measurement procedures. The measured vapor pressure of the reference material is not used for calculating the vapor pressure of the test substance, but may be reported and used for comparison with other measurement methods.

**Calculations**

A linear regression (least squares fit) will be performed on the vapor pressure measurements collected after the valves to the vacuum pumps are closed to determine the out-gassing rate. The out-gassing rate of the system will be equal to the slope of the regression equation in the absence of sample. The uncorrected vapor pressure of the sample will be equal to the intercept of the regression equation, i.e. the vapor pressure at the moment the valves are closed. If significant, the vapor pressure determined for the blank sample chamber will be considered the baseline or background pressure and will be subtracted from each vapor pressure measurement for the test substance. The means and standard deviations of the vapor pressures and out-gassing rates will be calculated.

**Sample Handling and Safety**

The Sponsor will identify any special handling or safety precautions to be used with the above referenced test substance. All normal precautions with respect to handling and storage will be taken.

**Sample and Test Substance Retention**

Upon completion of testing, portions of the test substance used as part of this study will be disposed of in accordance with federal, state and local regulations. Any unused portion of the test substance will be returned to the Sponsor.

**RECORDS TO BE MAINTAINED**

Records to be maintained for data generated by Wildlife International, Ltd. will include, but not be limited to:

1. A copy of the signed protocol
2. Identification and characterization of the test substance, if provided by the Sponsor.
3. Dates of initiation and completion of the study.
4. Dates of experimental start and termination.

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5. Storage conditions of the test substance.
6. Test substance use log.
7. Spinning rotor gauge pressure readings.
8. Statistical calculations.
9. Test conditions.
10. A copy of the final report.

**FINAL REPORT**

Wildlife International, Ltd will prepare a final report of the results of the study. The report will include, but not be limited to the following, when applicable:

1. Name and address of the facility performing the study.
2. Dates upon which the study was initiated and completed.
3. A statement of compliance signed by the Study Director addressing any exceptions to Good Laboratory Practice Standards
4. Purpose and procedure, as stated in the approved protocol, including all amendments and deviations to the protocol.
5. A copy of the protocol and protocol amendments.
6. The test substance identification, including name, chemical abstract number or code number, purity, composition, empirical formula, molecular formula, manufacturer's lot/batch number, dissociation in water, method of analysis, or other information provided by the Sponsor.
7. Description of the test method or reference to the method used along with any modifications made. Individual and mean values obtained for out-gassing rates and vapor pressure.
8. Description of any problems experienced and how they were resolved.
9. A statement prepared by the Quality Assurance Unit listing the dates that study inspections and audits were made and any findings that were recorded.

**CHANGING OF PROTOCOL**

Planned changes to the protocol will be in the form of written amendments signed by the Study Director and the Sponsor. Amendments will be considered as part of the protocol and will be attached to the final protocol. Any other changes will be in the form of written deviations filed with the raw data. All changes to the protocol will be indicated in the final report

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**GOOD LABORATORY PRACTICES**

This study will be conducted according to the Good Laboratory Practices described in OECD (ISBN 92-84-12367-9). Each study conducted by Wildlife International, Ltd. is routinely examined by the Wildlife International, Ltd. Quality Assurance Unit for compliance with Good Laboratory Practices, Standard Operating Procedures and the specified protocol. A statement of compliance with Good Laboratory Practices will be prepared for all portions of the study conducted by Wildlife International, Ltd. The Sponsor will be responsible for compliance with Good Laboratory Practices for procedures performed by other laboratories. Raw data for all work performed at Wildlife International, Ltd. and a copy of the final report will be filed by project number in archives located on the Wildlife International, Ltd. site or at an alternative location to be specified in the final report

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**REFERENCES**

- 1    **Organisation for Economic Cooperation and Development.** 1995. Guideline for Testing of Chemicals, 104: *Vapor Pressure Curve*.
- 2    **Product Properties Test Guidelines** 1996. OPPTS 830.7950 *Vapor Pressure*.

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## APPENDIX I

## IDENTIFICATION OF TEST SUBSTANCE BY SPONSOR

To be Completed by Sponsor

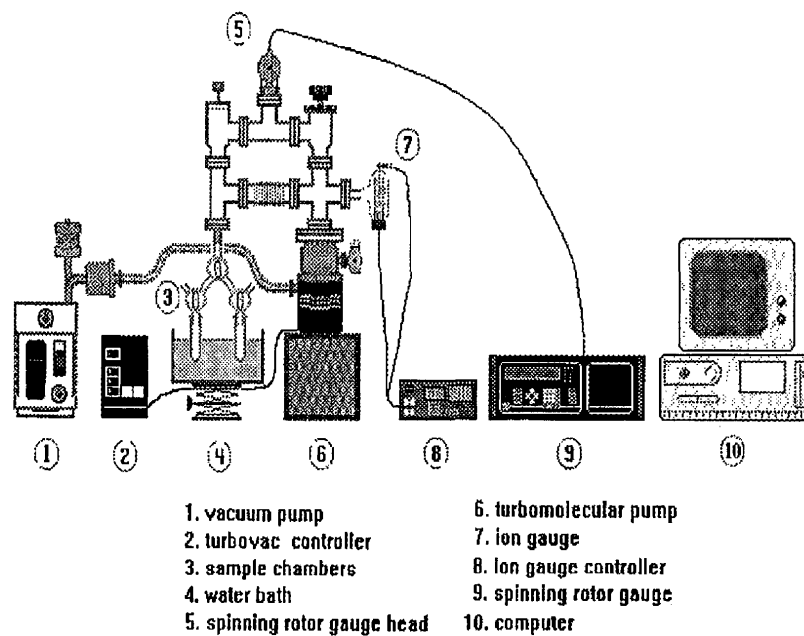
- I. Test Substance Identity (name to be used in the report): Tetrabromobisphenol-A  
Reference Standard (if applicable): N/A  
Test Substance Sample Code or Batch Number: Wildlife International, Ltd. No. 5381  
Test Substance Purity (% Active Ingredient): 98.91% Expiration Date: August 1, 2002
- II. Test Substance Characterization  
Have the identity, strength, purity and composition or other characteristics which appropriately define the test substance and reference standard been determined prior to its use in this study in accordance with GLP Standards? X Yes    No
- III. Test Substance Storage Conditions  
Please indicate the recommended storage conditions at Wildlife International, Ltd.,  
Ambient temperature; protect from light and moisture  
Has the stability of the test substance under these storage conditions been determined in accordance with GLP Standards? X Yes    No  
Other pertinent stability information:  
N/A
- IV. Toxicity Information:  
Mammalian: Rat LD50 > 5 g/kg Mouse LD50: > 10 g/kg  
Aquatic: Invertebrate Toxicity (EC/LC50) N/A Fish Toxicity (LC50) N/A  
Other Toxicity Information (including findings of chronic and subchronic tests):
- V. Classification of the Compound:  
   Insecticide    Herbicide    Fungicide  
   Microbial Agent    Economic Poison  
Other: Halogenated flame retardant

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Figure 1 Schematic of Spinning Rotor Gauge Apparatus.



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Project Number 439C-128

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AMENDMENT TO STUDY PROTOCOL

**STUDY TITLE:** Determination of the Vapor Pressure of Tetrabromobisphenol A Using the Spinning Rotor Gauge Method

**PROTOCOL NO.:** 439/082701/104/SUB439

**AMENDMENT NO.:** 1

**SPONSOR:** American Chemistry Council's  
Brominated Flame Retardant Industry Panel

**PROJECT NUMBER:** 439C-128

**SPONSOR STUDY NO.:** NA

**EFFECTIVE DATE:** August 27, 2001

**AMENDMENT:** Test Substance, Page 3  
Change: HBCD


To: Tetrabromobisphenol A

**REASON:** Typographical error

**AMENDMENT:** Test Substance, Page 3  
Change: 5381

To: 5315

**REASON:** Typographical error

  
STUDY DIRECTOR

9-24-01  
DATE

  
LABORATORY MANAGEMENT

9/24/01  
DATE

  
SPONSOR'S REPRESENTATIVE

9/25/01  
DATE

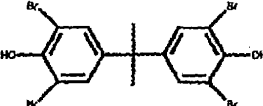
Reviewed by QA (SLC) 9-24-01



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Appendix 2  
Certificate of Analysis

## CONCLUSIONS AND TEST ARTICLE ANALYTICAL DATA. 1.

CHEMICAL NAME: Tetrabromobisphenol-A C.A.S. No.: 79-94-7 MOLECULAR FORMULA: $C_{15}H_7Br_4O_2$ PHYSICAL FORM: White Powder CHEMICAL STRUCTURE:					
					
ANALYSIS	RESULTS			ANALYSIS DATES	ANALYST
FT-IR	The sample FT-IR spectrum matched that of the reference spectrum. All spectra are on file with the original data.			01/04/01	W. T. Cobb
HPLC					
Sample	Purity (area% TBBPA)	Average	Difference (%) from average		
top center	98.92	98.91	<5%	01/05/01	J. S. Arroyave
middle center	98.89	98.91	<5%	01/05/01	J. S. Arroyave
bottom center	98.91	98.91	0	01/05/01	J. S. Arroyave
			Difference		
pre-study	98.91 (average)			01/05/01	J. S. Arroyave
day zero	98.95		< 5%	05/09/01	J. S. Arroyave
end-of-study	98.91		< 5%	05/09/01	J. S. Arroyave
CONCLUSION: Based on these analytical data, the test article identity was confirmed as tetrabromobisphenol-A. The composite sample was shown to be homogeneous with a purity of 98.91%. The test article was stable during the study.					

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**Appendix 3**

**Personnel Involved in the Study**

The following key Wildlife International, Ltd. personnel were involved in the conduct or management of this study:

1. Willard B. Nixon, Ph.D., Director of Analytical Chemistry
2. Raymond L. VanHoven, Ph.D., Scientist
3. Frank J. Lezotte, B.S., Chemist