

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
WASHINGTON, D.C. 20460

May 25, 2004



Office of Pesticide Programs

**MEMORANDUM**

**SUBJECT:** Data Evaluation Report for the Hydrolysis of PXTS (as a function of pH)

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**Thru:** Norm Cook, Chief *Norm Cook 6/2/04*  
Risk Assessment and Science Support Branch (RASSB)  
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**DP Barcode (s):** ~~D204272~~ *D298968*

**Pesticide Chemical No.:** *006925*

**Review Time:** 30 Hours

**MRID #:** 460626-23

Attached please find the Data Evaluation Report (DER) for the hydrolysis of PXTS, Polyxlenoltetrasulfide ( as a function of pH) submitted by the Akzo Nobel Functional Chemical LLC.

**EXECUTIVE SUMMARY:**

Hydrolysis of PXTS as a function of pH and at a nominal concentration of 0.125 mg/L was studied. The study was conducted following the procedures outlined in the laboratory protocol, which was based on OPPTS Guideline 835.2110 and OECD Guideline 111.

A test was conducted in sterile buffer solutions of pH 4 (acetate buffer), pH 7 (phosphate buffer), and pH 9 (borate buffer) at a temperature of 50°C for a period of 5 days (120 hrs). Samples of the test substance were analyzed at the start (0 hours) and end of the test (120 hours). Samples were analyzed using reverse phase, gradient elution using HPLC with UV detection.

In the test, the concentration of the test substance declined more than 50 percent, at all pH levels (4, 7, and 9). The test substance was considered to be hydrolytically unstable because none of the test substance was detectable after 5 days (120 hours) at 50 ± 1°C. The half-life/DT50 (50% decline time) of PXTS was estimated as significantly less than 1 year at pH 4, pH 7 and pH 9.

No additional tests were conducted. Therefore, a reaction constant and half-life of the test substance could not be determined. No supplementary experiments were discussed in the study report.

The procedure followed the requirements for a preliminary test in 835.2110(d)(5)(i). According to the test results, the test substance was unstable. However, the laboratory did not perform the required additional test for hydrolytically unstable substances called out in 835.2110(d)(5)(ii).

Therefore, while this study satisfies the guideline requirement for a preliminary hydrolysis (as a function of pH) study, is classified as unacceptable as a complete hydrolysis (as a function of pH) study. Another hydrolysis study, performed according to OPP guidelines, is required to support registration.

**RESULTS SYNOPSIS:**

	Half-life (unit)	Major transformation products
pH 5	<<1 year	none reported
pH 7	<<1 year	none reported
pH 9	<<1 year	none reported

**I. MATERIALS AND METHODS**

**GUIDELINE FOLLOWED:**

The study was conducted following the procedures outlined in the laboratory protocol. According to the study report, the laboratory protocol was based on OPPTS Guideline 835.2110 and OECD Guideline 111.

## GUIDELINE DEVIATIONS:

The procedure followed the requirements for a preliminary test in 835.2110(d)(5)(i). According to the test results, the test substance was unstable. However, the laboratory did not perform the required additional test for hydrolytically unstable substances called out in 835.2110(d)(5)(ii). The laboratory protocol was originally written as a hydrolysis (as a function of pH) study. The protocol was then amended to include only the preliminary study.

## COMPLIANCE:

Signed and dated copies of the "Statement of No Data Confidentiality Claims" and the "Good Laboratory Practice Compliance Statement" were provided for the study. The GLP statement indicates that the laboratory followed both the OECD principles of good laboratory practice and the national GLP regulations published at 40 CFR 160.

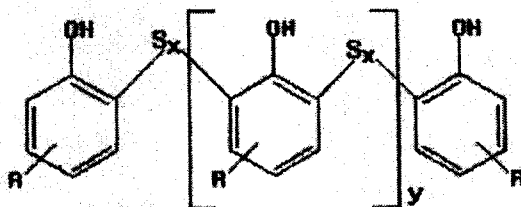
## A. MATERIALS:

### 1. Test Material

### Polyxylenotetrasulfide (PXTS)

#### Chemical Structure:

R = 95% Methyl  
x(avg) = 2.8  
y(avg) = 0.97



(Radio labeling was not mentioned in study report.)

**Description:** Dark solid

**Purity:** Analytical purity: 100%  
Batch No. 1685-23  
Lot No. 1685-25-2

Radiochemical purity: NA\*  
Specific activity: NA\*  
Locations of the label: NA\*

\*Not Applicable - Test material was not radio labeled

**Storage conditions of test chemicals:** Ambient

**Table 1: Physical-chemical properties of PXTS**

Parameter	Value	Comment
Water solubility	<12.5 µg/L	The study report does not indicate what the corresponding temperature is for this value.
Vapor pressure/volatility	not reported	
UV absorption	not reported	
pK <sub>s</sub>	not reported	
K <sub>ow</sub> /log K <sub>ow</sub>	not reported	
Stability of compound at room temperature, if provided	not reported	

**2. Buffer Solution:**

Buffer solutions were prepared with HPLC water as described in the following table. None of the compositions could be found in the Annex to OECD Guideline 111 or Table 13 of OPPTS 835.2110(d)(2)(I)(A)(2). However, the pH of each buffer solution was measured using a pH meter with an accuracy of 0.1 pH units.

**Table 2: Description of Buffer Solutions**

pH	Composition
4	720 mL of 0.2 N sodium acetate mixed with 3280 mL 0.2 M acetic acid
7	1200 mL of 0.1 N NaOH mixed with 2000 mL 0.1 M potassium dihydrogen phosphate and 800 mL of HPLC water
9	860 mL of 0.1 N NaOH mixed with 2000 mL 0.1 M boric acid in 0.1 M potassium chloride and 1140 mL of HPLC water

**B. EXPERIMENTAL CONDITIONS**

- 1) **Preliminary Study:** No preliminary studies were discussed in the study report.
- 2) **Experimental conditions:** Experimental parameters are presented in Table 3, below.

**Table 3: Experimental parameters**

Parameters		Details
Duration of the study		5 days
Test concentrations (mg a.i./L) nominal: measured:		concentrations in mg PXTS/L nominal: 0.125 measured: 0.0961 at pH 4 0.129 at pH 7 0.153 at pH 9
No. of replications		Single Samples
Preparation of test medium	Volume used/treatment	100 mL
	Method of sterilization	Sterile filtration
	Co-solvent (type/concentration)	Acetone / less than 1%
Test apparatus		Hewlett-Packard Model 1090 HPLC equipped with Hewlett-Packard Series 1100 Variable Wavelength Detector operated at 250 nm
Details of traps for volatile, if any		not reported
If no traps were used, is the test system closed/open		open
Is there any indication of the test material adsorbing to the walls of the test apparatus?		not reported
Experimental conditions Temperature (°C) pH		50±1°C 4, 7, 9
Other details, if any		NA

3). **Supplementary Experiments:** No supplementary experiments were discussed in the study report.

4). **Sampling:** Sampling details are presented in Table 4, below.

**Table 4: Sampling details**

Criteria	Details
Sampling intervals for the parent/transformation products	Start and end of test (0 and 120 hours)
Sampling method	Reverse phase, gradient elution using HPLC with UV detection.
Sampling methods for the volatile compounds, if any	not reported
Sampling intervals/times for: pH measurement temperature	Start and end of test (0 and 120 hours) not reported
Sample storage before analysis	ambient conditions
Other observation, if any (e.g.: precipitation, color change etc.)	not reported

### C. ANALYTICAL METHODS:

Identification and quantification of the parent compound was performed using a Hewlett-Packard Model 1090 HPLC equipped with a Hewlett-Packard Series 1100 Variable Wavelength Detector operated at 250 nm. HPLC conditions were as follows:

Analytical Column: Vydac 214TP Column (50 mm x 4.6 mm, 5- $\mu$ m particle size)  
 Guard Column: Supelco Supelguard LC-304 Column (20mm)  
 Stop Time: 20.00 minutes  
 Flow Rate: 1.000 mL/minute  
 Oven Temperature: 40.0 °C  
 Mobile Phase: Solvent A: 40:60 Acetonitrile: NANOpure® Water  
 Solvent B: 95:5 Acetonitrile: NANOpure® Water  
 Injection Volume: 150  $\mu$ L  
 PXTS Peak Retention Time: ~11.0 minutes  
 Primary Analytical Wavelength: 250 nm

The method limit of quantitation was set at 0.0625 mg/L, calculated as the product of the lowest calibrated standard (1.00 mg/L) and the dilution factor of the samples (0.0625).

Total <sup>14</sup>C was not measured.

Identification and quantification of transformation products and detection limits for the transformation products were not provided.

## **II. RESULTS AND DISCUSSION:**

### **A. TEST CONDITIONS:**

The study report did not indicate how pH, sterility, temperature and other experimental conditions were maintained throughout the study. Values for pH were presented only as integers; temperature was presented only as  $50 \pm 1^\circ\text{C}$ .

### **B. MASS BALANCE:**

Concentrations and Rates of Decline of PXTS are presented in Table 5, below.

**Table 5: Concentrations and Rates of Decline of PXTS**

pH		4	7	9
		Concentration of Test Material (mg/L)		
Hydrolysis Time (hours)	0	0.0961	0.129	0.153
	120	<0.0625	<0.0625	<0.0625
Rate of Decline		>50%	>50%	>50%

### **C. DEGRADATION OF TEST MATERIAL:**

Determination of potential degradants was not feasible due to the adverse influence of extracted buffer components on the gas chromatographic conditions.

#### **HALF-LIFE:**

The half-life of PXTS at different pH values, using reverse phase, gradient elution using HPLC with UV detection, was estimated as significantly less than one year.

### **D. SUPPLEMENTARY EXPERIMENT-RESULTS:**

No supplementary experiments were discussed in the study report.

## **III. STUDY DEFICIENCIES:**

According to the preliminary study, the test substance was unstable. However, the laboratory did not perform the required additional test for hydrolytically unstable substances. Therefore, the study satisfies the requirement for a preliminary hydrolysis (as a function of pH) study, but is

classified as unacceptable as a complete hydrolysis (as a function of pH) study. Another study, performed according to OPP guidelines, is required to support registration.

The water solubility of the test substance was provided. However, the study report did not indicate the associated temperature, so it is not known if this solubility corresponds to the test temperature of 50°C. In addition, the molar concentration of the test solution depends on whether the water solubility is greater or less than  $2 \times 10^{-2}$  M, however, the units given for solubility were in mass per unit volume. The molar concentration could not be determined because the molecular weight was not provided. For these reasons, the appropriate molar concentration of the test solution could not be verified.

The laboratory did not perform a hydrolysis test at pH 1.2 called out in 835.2110(d)(5)(iii), stating that the test substance was not physiologically important.

The readings (time and value) for temperature and pH were not found in the study report.

The laboratory protocol calls for triplicate analysis of the test substance concentrations. Only the results for single samples were found in the study report. In the opinion of the Laboratory Study Director, the deviation had no impact on the conclusion of the study.

#### **IV. REFERENCES:**

1. **Product Properties Test Guidelines.** 1998. OPPTS 835.2110, *Hydrolysis as a Function of pH.*
2. **Organization for Economic Co-operation and Development.** 1981. *Guideline for Testing of Chemicals, 111: Hydrolysis as a Function of pH.*

File: C:/Myfiles/2004 Reports/PXTS/ Data Evaluation Report for the Hydrolysis of PXTS  
(as a function of pH). wpd

CC: Siroos Mostaghimi/RASSB  
Chemical Files