

# CYANEX<sup>®</sup> 921 Extractant

## Solvent Extraction Reagent

- Selective for cobalt over nickel from sulfate and chloride media.
- Selective for zinc in the presence of calcium and cobalt.
- Extracts other metal cations.

**CYTEC**

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### ANALYTICAL METHODS

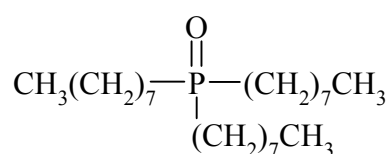
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# INTRODUCTION

CYANEX® 921 extractant, better known as trioctylphosphine oxide (TOPO), was the first member of a family of solvent extraction reagents developed by Cytec. This reagent has been used commercially for many years to recover uranium from wet process phosphoric acid. A more recent industrial application is in the extraction of acetic acid from effluent streams.

## Chemical Structure

The chemical structure of CYANEX 921 extractant is as follows:



Molecular Weight = 386

## Typical Properties

Trioctylphosphine oxides	: 93%
Appearance	: Off-white, waxy solid
Specific Gravity at 25°C	: 0.88
Specific Gravity at 61°C	: 0.84
Melting Point °C	: 47-52
Viscosity at 55°C	: 15.0 centipoise (15.0 mPa.s)
Brookfield Viscometer At 100°C	: 5.5 centipoise (5.5 mPa.s) (Viscosity is shown as a function of temperature in Figure 1)

## Compatible Materials

Stainless steel (304 and 316), mild steel and aluminum were found to corrode at <1 mil per year when in contact with a TOPO melt at 60-80°C.

## Stability

Trialkylphosphine oxides, of which CYANEX 921 is one example, are known to be the most stable members of the group of organophosphorus solvating reagents. <sup>(1,2,3,4)</sup>

Losses due to degradation are, therefore, likely to be lower than those associated with extractants such as tributylphosphate and dibutyl butylphosphonate.

## Solubility Losses

Losses of CYANEX 921 extractant by distribution to aqueous solutions of various composition were determined in some batch shake-out tests. The results are given below. <sup>(3)</sup>

Composition of Aqueous Phase	Extractant Solubility mg/L
Water	1.1±0.7
1 M HCl	0.2±0.07
0.5 M SO <sub>4</sub> + 0.1 M HNO <sub>3</sub>	0.2±0.07
2% Na <sub>2</sub> CO <sub>3</sub>	3.6±0.06
5% Na <sub>2</sub> CO <sub>3</sub>	3.6±1.3

The experimental conditions were as follows:

Solvent	:	0.1 M extractant (38.6 g/L) in decane
Temperature, °C	:	25
Equilibration Time	:	10 minutes
A/O	:	1

The soluble losses are generally lower than those of other extractants in commercial use, e.g. losses of amines to acidic sulphate solutions are reported to be in excess of 10 mg/L. <sup>(4,5)</sup>

## Organic Solubility

The solubility of CYANEX 921 extractant in four typical commercial diluents is shown as a function of temperature in Figure 4. Solubility is higher in the aromatic diluents, - Aromatic 100\* and Aromatic 150\* diluents than in the two essentially aliphatic diluents Escaid 110\* diluent and Kermac 470B\*\* diluent.

## Toxicity

The acute oral (rat) and acute dermal (rabbit) LD50 values for CYANEX 921 extractant are >10. 0 g/kg and 2.83 g/kg, respectively. Marked skin and eye irritation were produced during primary irritation studies with rabbits. Inhalation of airborne material may be irritating to the respiratory tract. CYANEX 921 was determined to be not mutagenic in the Ames Salmonella Assay.

\*Product of Exxon Co., U.S.A. \*\*Product of Kerr-McGee Refining Corp.

# COMMERCIAL APPLICATIONS

## Uranium Recovery From Wet Process Phosphoric Acid

Original work at the Oak Ridge National Laboratories led to the development of a solvent extraction process to recover the small quantities of uranium (0.1 to 0.2 g/L) present in wet process phosphoric acid. <sup>(6,7,8)</sup> The process, which is based on the extraction of hexavalent uranium using a synergic mixture of CYANEX 921 extractant and D2EHPA, is operating successfully in a number of commercial plants in the U.S., Canada and Belgium.

## Acetic Acid Recovery From Effluent Streams

Processes in petrochemical plants, wood pulp mills and other chemical facilities often generate aqueous effluent streams containing small concentrations of carboxylic acids; particularly acetic acid. Typically, the acids are present in concentrations of 1 to 50 gpl depending upon the source. <sup>(9)</sup>

The use of CYANEX 921 extractant and other reagents to recover acetic acid from these streams has formed the subject of several informative papers in recent years <sup>(10,11,12,13)</sup> and a number of patents have been issued in this area. <sup>(14,15)</sup> Briefly, the described processes consist of extracting the acid into a water-immiscible solvent, recovering or stripping the acid from the loaded solvent by distillation and recycling the solvent. One such plant, which recovers acetic acid and furfural, has been operated successfully by Lenzing AG in Austria since 1983.

The advantages of CYANEX 921 in this application lie in low solubility losses, stability during distillation at elevated temperatures and high extraction coefficients for acetic acid in comparison to other solvating reagents. These properties have the effect of minimizing plant operating and capital costs.

As an illustration of efficient performance characteristics, an acetic acid extraction isotherm obtained with a solvent containing 400 g/L CYANEX 921 extractant is shown in Figure 1. The corresponding experimental results and conditions are given in Table 1.

McCabe-Thiele interpolations from the isotherm indicate essentially quantitative recovery in three theoretical stages at O/A = 1.5; assuming an initial feed containing 10 g/L acetic acid.

Raffinates obtained from the above shake-out tests were analysed and found to contain <2 mg/L of CYANEX 921 extractant (detection limit of the method).

Rapid extraction kinetics play an important role in any solvent extraction process as good kinetic properties allow for the design of compact equipment and, consequently, have a favorable effect on capital expenditures. This property is

particularly important when differential (column) contactors are used because of the comparatively short contact times and low interfacial areas prevalent in this type of equipment.

Data on the rate of extraction of acetic acid with CYANEX 921 are presented in Table 2 and Figure 2 and indicate that extraction is essentially complete in <2 minutes. This rate is sufficiently high so as not to impose any kinetically based limitations on equipment selection for this process.

**TABLE 1**  
**Acetic Acid Extraction Isotherm**

Solvent	: 400 g/L CYANEX 921 extractant in DPA diluent*		
Aqueous	: 10.0 g/L acetic acid		
Temperature, °C	: 50		
Contact Time	: 5 minutes		
	Equilibrium Acetic Acid Concentration (g/L)		
A/O	Organic	Aqueous	
2	8.40	5.80	
1	7.50	2.50	
0.5	4.48	1.05	
0.2	1.92	0.40	
0.1	0.92	0.20	

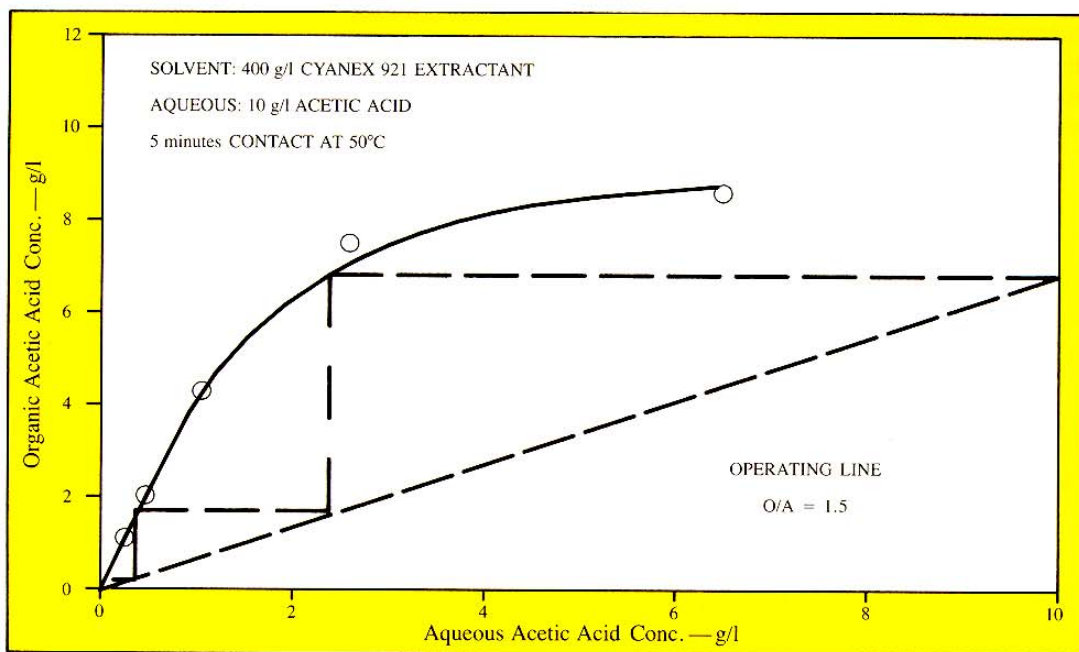
\*DPA, a high boiling (330-379°C) diphenylalkene diluent, is a product of Conoco Chemicals Co. (a division of Conoco)

**TABLE 2**  
**Acetic Acid Extraction Kinetics**

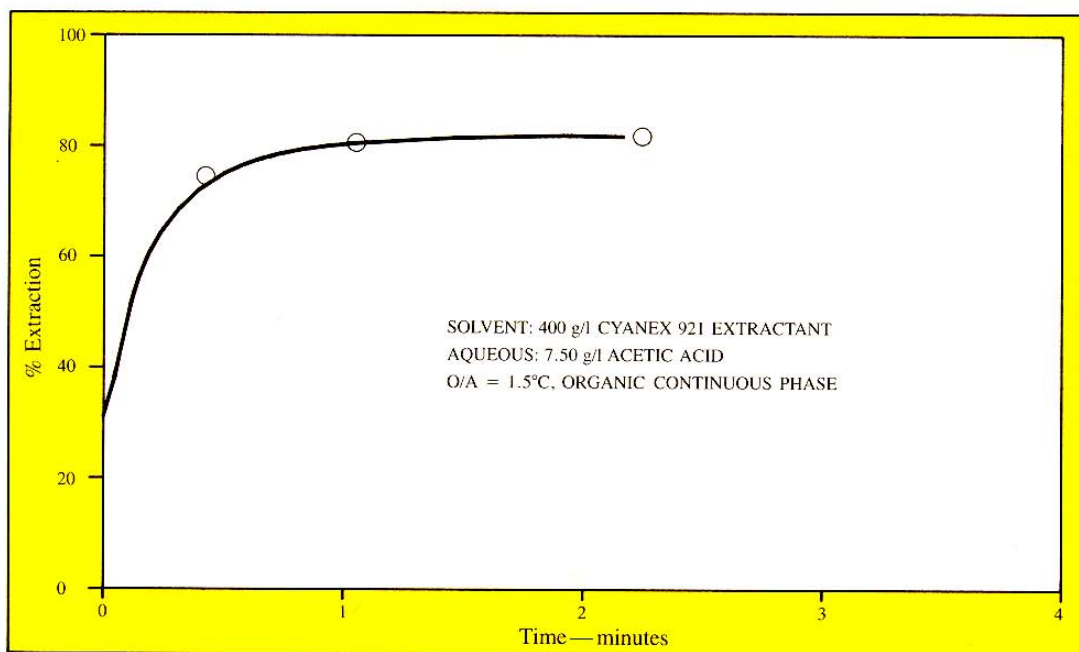
Solvent	: 400 g/L CYANEX 921 extractant In DPA diluent		
Aqueous	: Synthetic solution containing 7.50 g/L acetic acid		
Temperature, °C	: 50		
A/O	: 1 (Organic continuous phase)		
	Acetic Acid		Extraction Coefficient $E_A^o$
Time (mins)	Conc. In Raffinate (g/L)	% Extraction	
0	7.50	0	0
0.5	1.92	74.5	2.91
1	1.67	77.8	3.49
2	1.67	77.8	3.49

$E_A^o = \frac{\text{Concentration of Acetic Acid in Solvent at Equilibrium}}{\text{Concentration of Acetic Acid in Aqueous}}$

**FIG. 1—ACETIC ACID EXTRACTION ISOTHERM**



**FIG. 2—KINETICS OF ACETIC ACID EXTRACTION**



# POTENTIAL APPLICATIONS

## METAL EXTRACTION

### Niobium Tantalum Separation

A pilot-plant process using CYANEX 921 extractant to recover and separate niobium and tantalum has been demonstrated recently.<sup>(16,17)</sup> Both metals are extracted from a hydrofluoric-sulphuric acid leach liquor and then selectively stripped from the loaded organic. The advantages of CYANEX 921 extractant in comparison to the commercially used MIBK extractant were shown to be higher stability, lower aqueous solubility, rapid phase disengagement and, particularly, the production of high purity niobium oxide which meets the stringent specifications required for nuclear, optical and electronic applications.

Data comparing the impurities in niobium oxides derived by solvent extraction with MIBK and CYANEX 921 extractant are shown in Table 3 below.<sup>(16)</sup>

**TABLE 3**  
**NIOBIUM OXIDE PURITY**

COMPONENT	Concentration mg/L	
	CYANEX 921 EXTRACTANT	MIBK
Ta <sub>2</sub> O <sub>5</sub>	< 20	600
Fe <sub>2</sub> O <sub>3</sub>	< 5	< 500
CoO	1	10
CuO	1	5
MnO	1	10
MoO <sub>3</sub>	< 5	< 20
WO <sub>3</sub>	< 10	< 50
TiO <sub>2</sub>	< 5	< 200
SiO <sub>2</sub>	< 10	< 200
Cr <sub>2</sub> O <sub>3</sub>	< 3	< 50
P	< 10	< 200
Loss on Ignition	0.1%	0.1%

Table 3 reproduced by the kind permission of VCH Verlagsgesellschaft, Weinheim, Federal Republic of Germany.

### Rhenium Recovery

Rhenium is an essential element in the production of petroleum reforming catalysts. A recent patent granted to American Cyanamid Company<sup>(18)</sup> discloses the use of CYANEX 921 to recover rhenium from these source materials. The process involves leaching the spent catalyst with sulphuric acid, extracting rhenium and then stripping with ammonium sulphate. Excellent selectivity against typical impurity metals such as aluminum, calcium, magnesium and iron is achieved.

### Arsenic Extraction From Copper Electrolytes

The removal of arsenic impurities from copper electrolytes by solvent extraction with CYANEX 921 forms the subject of a recent European Patent issued to Vereinigte Metallwerke Ranshofen-Berndorf AG.<sup>(19)</sup> The process describes the selective extraction of arsenic followed by stripping with an acidic sodium sulphate solution and recovery of arsenic from the strip liquor by precipitation with H<sub>2</sub>S. CYANEX 921 is shown to be a stronger extractant for arsenic than tributylphosphate which is now used in two commercial arsenic solvent extraction plants. This property is claimed to result in lower staging requirements and solvent inventory.

### Lithium Recovery

The commercial significance of lithium has increased in recent years as a result of the development of new uses for this element, e.g. in batteries, and because of increased demand in established applications (ceramics, etc.).

A novel solvent extraction process using CYANEX 921 extractant to recover lithium from one of its sources (neutral brines) has been described<sup>(20)</sup>

### Additional Applications

CYANEX 921 is known to extract approximately 30 metals from aqueous solution as indicated in Table 4.<sup>(21)</sup>

Selective separations can be made depending upon a variety of factors, e.g. the valence state of the metal, the anionic nature of the solution (chloride, sulphate, etc.) and the concentration of the extractant in the solvent.

For example, one separation suggested by these data is the selective recovery of vanadium<sup>(5+)</sup> from chloride solutions containing aluminum, cobalt and nickel (these solutions may be derived from leaching spent catalysts used in the petroleum industry).

The reader is referred to two comprehensive reviews for more detailed information on the performance characteristics of TOPO.<sup>(22,23)</sup>

**TABLE 4 TRI *n*-OCTYLPHOSPHINE OXIDE (TOPO) EXTRACTION OF THE ELEMENTS**

Li	Be											B	C	N	O	P
Na	Mg											Al	Si	P	S	Cl
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br
			IV	V	VI	VII	III			II	II	III	IV	III		
			ac	a	ac	abc	a			a	a	a	a	a		
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I
			IV	V	VI	VII					II	III	IV	III V		
			ab	a	abc	acde					a	a	a	a		
Cs	Ba	La*	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At
			IV	V					IV	III	II			III		
			ab	a					a	a	a			b		
Fr	Ra	Ac†														
* Elements 58-71			Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
			IV													
			bc													
† Elements 90-103			Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	103
			IV		VI	IV	IV VI									
			ab		ab	bc	ab ab									
Key			Elements enclosed in color are extracted. Acid system from which favorable or selective extraction can be effected:													
Element			a-HCl; b-HNO <sub>3</sub> ; c-H <sub>2</sub> SO <sub>4</sub> ; d-HBr; e-H <sub>3</sub> PO <sub>4</sub>													
Valence																
System																

## ORGANIC EXTRACTION

### Phenol Recovery

Phenol occurs in aqueous effluents from many processes, e.g. in the petroleum, steel and coal gasification industries. CYANEX 921 is a strong extractant for phenol and offers a lower cost alternative to conventional Phenosolvan technology for phenol recovery.

This new process<sup>(24)</sup> involves dissolving CYANEX 921 extractant in a high boiling point diluent, extracting phenol and then stripping the solvent by distillation.

Extraction isotherms given in Table 5 and plotted in Figure 5 show the efficient extraction properties of CYANEX 921.

McCabe-Thiele interpretations from the isotherms indicated the following theoretical staging requirements and A/O ratios for essentially quantitative phenol recovery.

Extractant Conc. (g/L)	Theoretical Stages	A/O Ratio
100	2	2
200	2	3
325	2	5

**TABLE 5**  
**The Effect of Extractant Concentration on Phenol Recovery**

Solvents	:	100, 200 and 325 g/L CYANEX 921 extractant in Nalkylene 500 diluent*
Aqueous	:	10 g/L phenol (nominal) and 30 g/L Na <sub>2</sub> SO <sub>4</sub>
Temperature, °C	:	50
Contact Time	:	5 minutes

A/O	Equilibrium Phenol Concentration (g/L)					
	100 g/L		200 g/L		325 g/L	
	Org	Aq	Org	Aq	Org	Aq
0.25	2.54	0.02	2.46	0.01	2.63	0.02
0.5	5.07	0.05	4.93	0.02	5.25	0.03
1	10.1	0.10	9.84	0.04	10.5	0.04
2	19.4	0.47	19.6	0.10	20.9	0.08
5	34.3	3.34	45.0	0.87	-	-
7	40.0	4.48	56.6	1.79	68.2	0.79
10	-	-	-	-	88.8	1.65

\*Nalkylene 500, a linear alkyl benzene diluent, is a product of Conoco Chemicals Company (a division of Conoco).

FIG. 3—EFFECT OF TEMPERATURE ON VISCOSITY

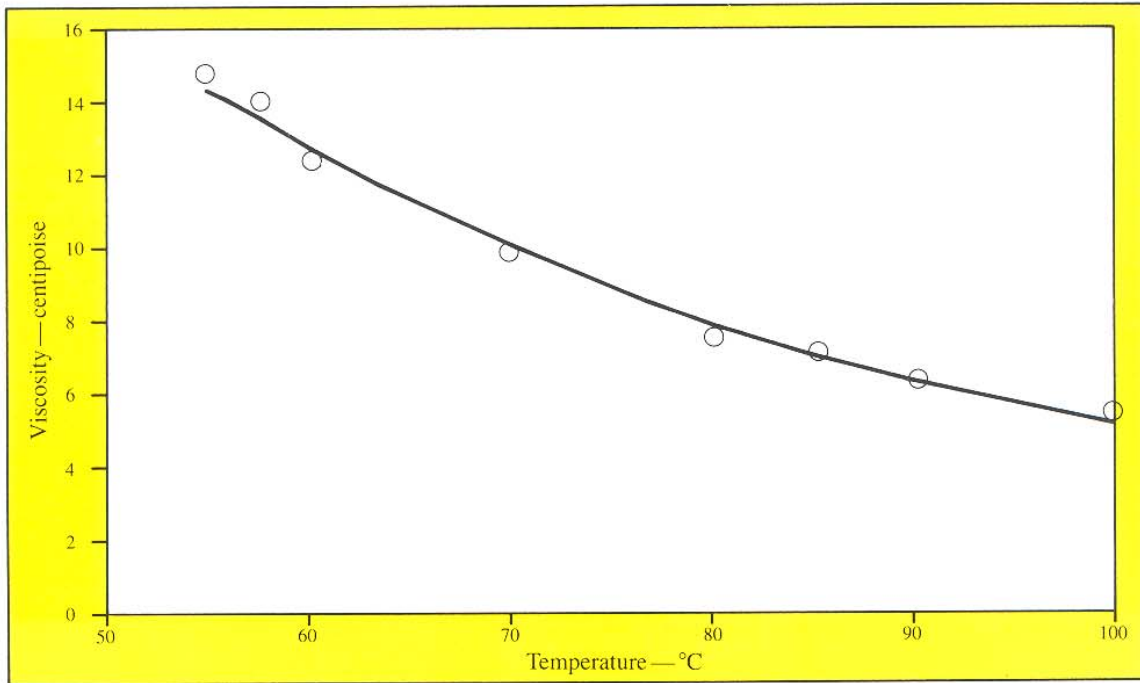


FIG. 4—EFFECT OF TEMPERATURE ON ORGANIC SOLUBILITY

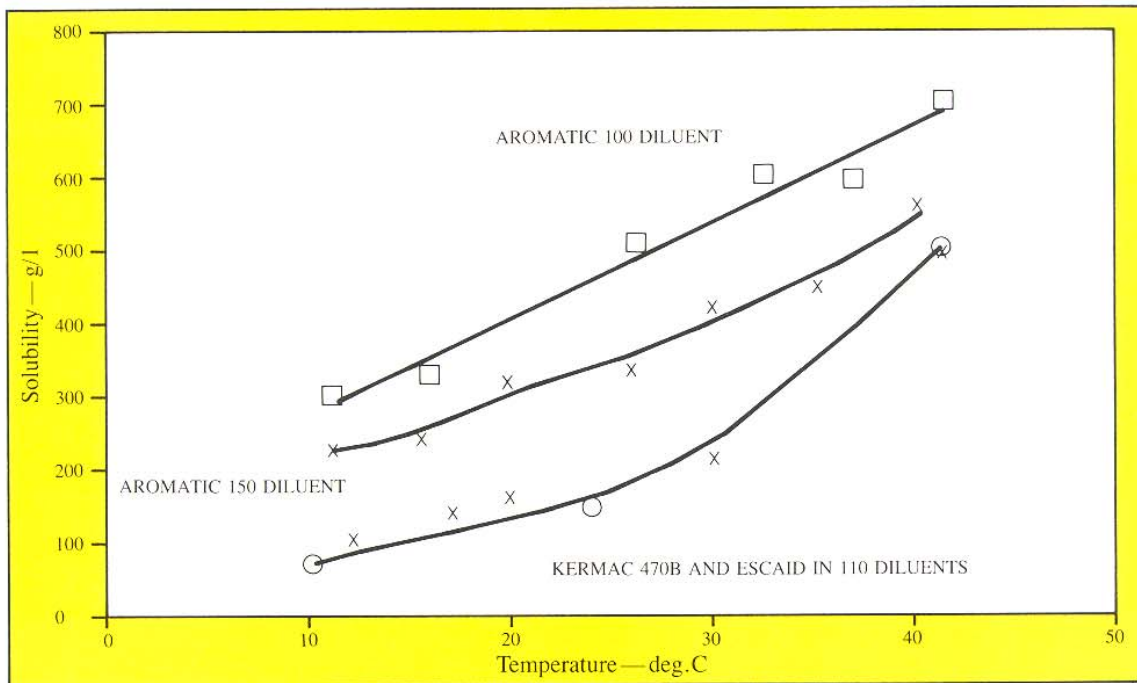
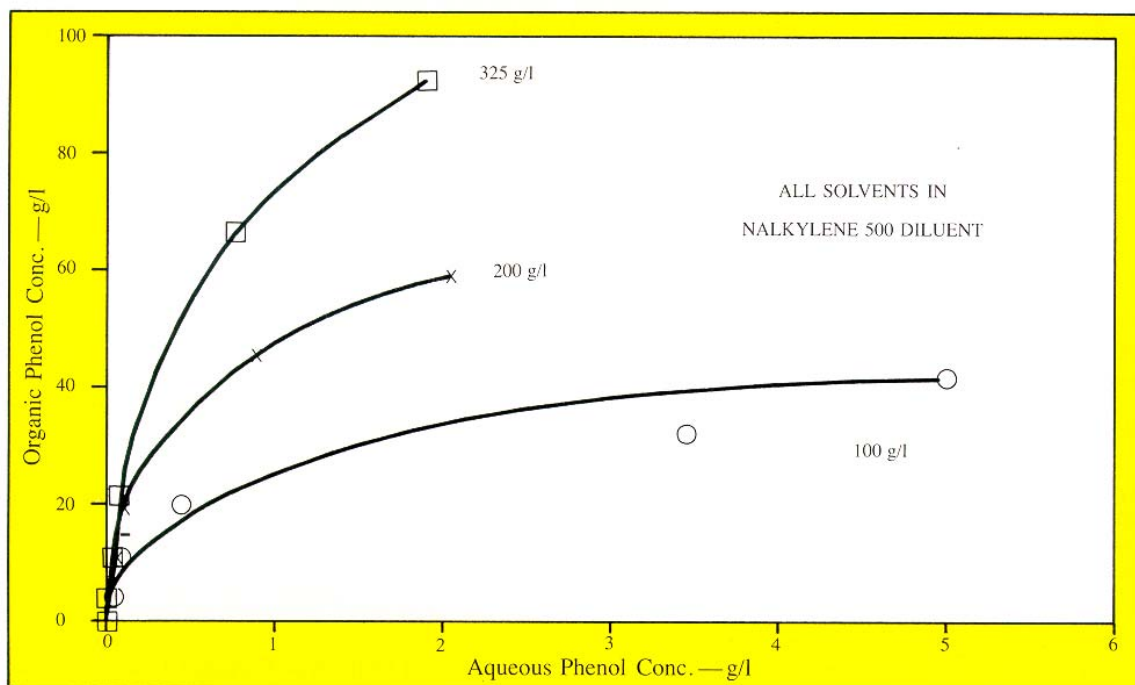


FIG. 5—EFFECT OF EXTRACTANT CONCENTRATION ON PHENOL RECOVERY



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# ANALYTICAL METHODS

## ANALYSIS IN ORGANIC SOLVENTS

As described, the gas chromatographic procedure is suitable for analyzing solvents containing  $120 \pm \text{g/L}$  TOPO. Other concentrations may be determined by making appropriate changes to the tenor of TOPO in the standard solution. (The method is based upon a single point standardization).

### Reagents

1. Didecylphthalate (AR grade-internal standard)
2. TOPO (purified-see note 1).
3. Process diluent (e.g. Escaid 110\* diluent, Aromatic 150\* diluent).

### Calibration

1. Accurately weigh ( $\pm 1 \text{ mg}$ )  $3 \pm 0.1 \text{ g}$  of pure TOPO and  $2 \pm 0.1 \text{ g}$  of didecylphthalate into a 50 mL volumetric flask. Dissolve and make up to volume using the process diluent.
2. Analyze the standard solution as described under "Gas Chromatographic Conditions" to determine the relative response factor of TOPO and didecylphthalate.

### Procedure

1. Centrifuge the solvent to be analyzed or filter through PS paper to remove entrained aqueous or suspended solids.
2. Accurately weigh ( $\pm 1 \text{ mg}$ )  $2 \pm 0.1 \text{ g}$  of didecylphthalate into a 50 mL volumetric flask and pipette 25 mL of the clarified solvent into the same flask. Make up to volume with the process diluent.  
Determine the relative response factor of TOPO and didecylphthalate as described under "Gas Chromatographic Conditions".

### Calculations

$$\text{Triocetylphosphine oxide (g/L)} = \frac{R_1}{R_2} \times 2 \times C_s$$

Where  $R_1$  = Relative response factor in standard solution  
 $R_2$  = Relative response factor in analyte solution  
 $C_s$  = TOPO concentration in standard solution (g/L)

### Gas Chromatographic Conditions

Instrument: Hewlett Packard 5880A or equivalent  
Column: 30 m X 0.32 mm fused silica capillary column coated with 0.25 $\mu\text{m}$  of SPB-1  
Detector: Thermal Conductivity  
Carrier Gas: Helium  
Temperature,  $^{\circ}\text{C}$   
Detector: 320  
Injector: 290  
Column: 270 (isothermal)

Column Flow: 1 mL/minute  
Vent Flow: 60 mL/minute  
Sample Injection  
Volume: 0.5  $\mu\text{L}$   
Peak Areas: Determined by electronic integration. Retention times for TOPO and didecylphthalate are approximately 6.5 and 10 minutes, respectively.

### Notes

1. Pure TOPO (>99.5%) can be obtained by recrystallizing commercial grade TOPO from hexane. (Three recrystallizations are necessary).
2. PS (Phase Separation) paper is available from Whatman, Inc.
3. Isomers of TOPO will also be detected using the subject method. These compounds may be assigned the same response factors as TOPO and determined accordingly. A typical chromatogram of commercial TOPO is shown in Figure 1A.
4. If the TOPO peak exhibits a leading tail this may effect the accuracy of the electronic integration. The effect can be minimized by increasing the column temperature by 10 to 20 $^{\circ}\text{C}$ .
5. Megabore columns can be used as an alternative to capillary columns.

## ANALYSIS IN AQUEOUS SOLUTION

Two methods have been developed which allow trace quantities of TOPO to be determined. The first method is suited to solutions which contain small concentrations of dissolved salts, while the second method is appropriate for high salt concentrations.

Both methods are based upon gas chromatography. In general, the solubility of TOPO in aqueous process solutions will be <5 mg/L.

### Low Dissolved Solids

The concentration of triocetylphosphine oxide (TOPO) is determined by evaporating the aqueous solution to dryness, leaching the residue with chloroform and analyzing the leachate by gas chromatography.

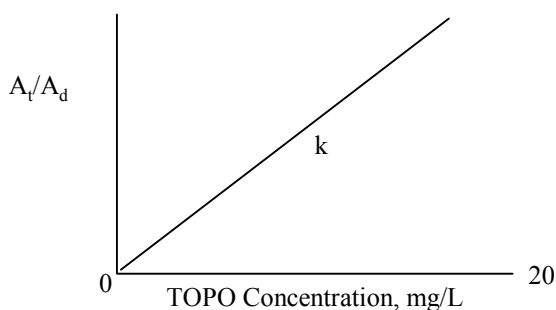
*\*Product of Exxon Co., U.S.A.*

## Reagents

1. Chloroform
2. Didecylphthalate
3. 25 mg/L solution of didecylphthalate (internal standard) in chloroform
4. TOPO (purified - see Note 1)
5. All reagents are AR grade

## Calibration

1. Prepare standard solutions containing 2, 5, 10 and 20 mg/L TOPO in chloroform. Each solution should also contain 25 mg/L didecylphthalate internal standard.
2. Analyze the solutions as described under "Gas Chromatographic Conditions".
3. Plot the calibration curve as follows and determine the slope "k".



Where  $A_t$  = TOPO peak area  
 $A_d$  = Didecylphthalate peak area

## Procedure:

1. Filter the aqueous solution to remove entrained organic.
2. Evaporate to dryness exactly 10 mL of the aqueous solution using a nitrogen-swept Pierce Reacti-Therm at 80°C.
3. At room temperature, leach the residue with exactly 1 mL of the chloroform solution containing 25 mg/L didecylphthalate.
4. Analyze the chloroform leachate as described under "Gas Chromatographic Conditions".

## Calculation

$$\text{Concentration of TOPO in the aqueous (mg/L)} = \frac{A_t}{A_d} \times \frac{1}{k} \times \frac{1}{10}$$

Where  $A_t$  = TOPO peak area  
 $A_d$  = Didecylphthalate peak area  
 $k$  = Slope of the calibration curve

## Gas Chromatographic Conditions

Instrument: Perking Elmer Sigma 115 or equivalent  
Column: 30 x 0.326 mm fused silica capillary column coated with 0.1  $\mu\text{m}$  Durabond 1 (15 or 60 m columns with a film thickness of 0.10 or 0.25  $\mu\text{m}$  may be substituted)

Split Ratio: 10 to 1  
Detector: FID  
Carrier Gas: Helium at 12 psig (30 m column)  
Vent Flow: 2.0 mL/minute (23 cm/sec)  
Temperatures, °C  
FID: 300  
Injection Port: 300  
Program, °C  
Isothermal: 270  
Sample Injection Volume: 5  $\mu\text{L}$

## Notes

1. Pure TOPO can be obtained by recrystallizing commercial grade TOPO from hexane. (Three recrystallizations are necessary).
2. TOPO and didecylphthalate will elute at approximately 5.4 and 7.8 minutes respectively.
3. Detection limit approximately 1 mg/L.
4. This method has not been validated.

## High Dissolved Solids

Aqueous solutions containing TOPO are analyzed by direct injection gas chromatography.

## Reagents

1. Tetrahydrofuran (THF). "Baker Analyzed Reagent, 100%"
2. TOPO (purified - see Note 1)

## Calibration

1. Prepare a stock solution by accurately weighing ( $1 \pm \text{mg}$ ) approximately 0.5 g of TOPO into a 50 mL volumetric flask. Dilute to 50 mL with THF and dissolve the TOPO.
2. Dilute aliquots of the stock solution with distilled water to prepare standard solutions containing 2, 5, 10 and 30 mg/L of TOPO.
3. Analyze the solutions as described under "Gas Chromatographic Conditions".

## Procedure

1. Filter the aqueous solution to remove entrained organic.
2. Inject 3  $\mu\text{L}$  of the analyte solution and analyze as described under "Gas Chromatographic Conditions".

## Calculation

\* Response Factor for TOPO (R)

$\frac{\text{Conc. of TOPO in Standard (mg/L)}}{\text{Area of TOPO, Standard Peak}}$

Concentration of TOPO in the aqueous (mg/L) = Area of TOPO peak (Analyte) X R

\* Response factor for the standard closest in concentration to the sample.

## Gas Chromatographic Conditions

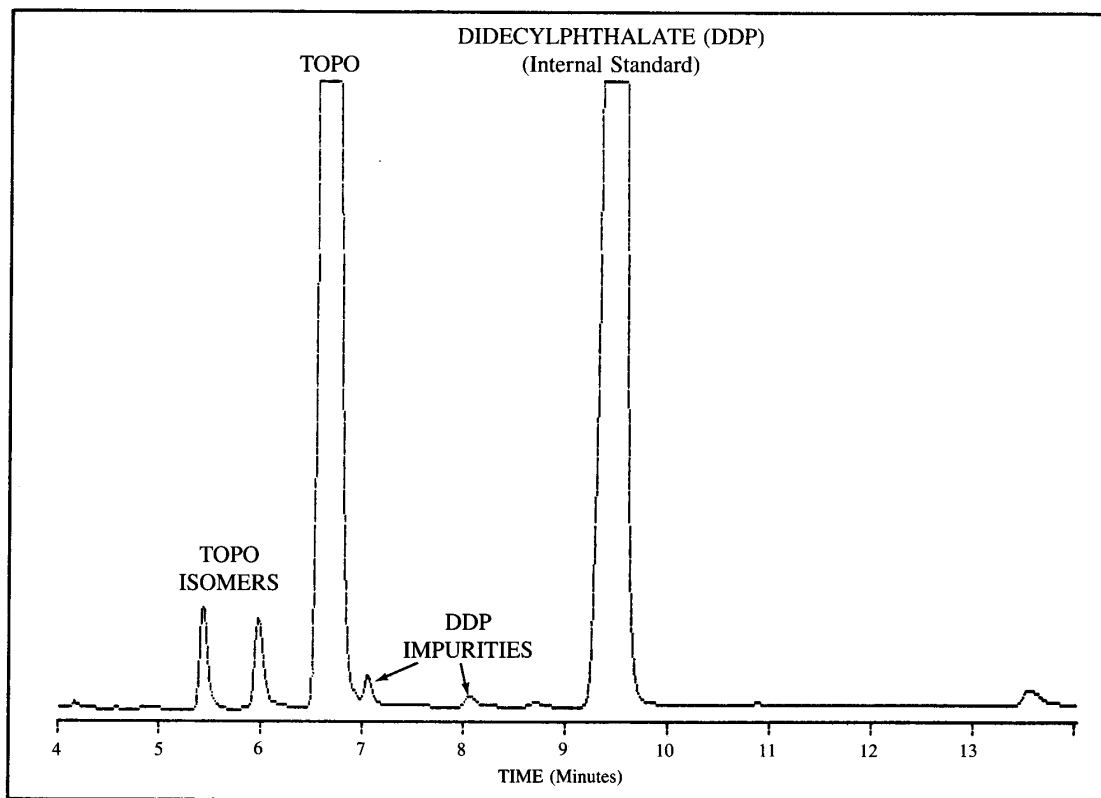
Instrument: Hewlett Packard 5730A or equivalent  
Column: 2 ft. x 1/4 inch OD x 2 mm ID glass  
Column packed with 9% OV-17 and 12% SP2401 on 80/100 mesh Supelcoport

Detector: FID  
Carrier Gas: Helium at 57 psi (flowrate 40 mL/minute)  
Temperatures, °C  
FID: 300  
Injection Port: 250  
Column: 230  
Program, °C  
Isothermal: 230  
Sample Injection Volume: 3 µl

## Notes

1. Pure TOPO can be obtained by recrystallizing commercial grade TOPO from hexane. (Three recrystallizations are necessary)
2. Retention time for TOPO is approximately 12 minutes
3. Detection limit approximately 2 mg/L.

FIG. 1A TYPICAL CHROMATOGRAM OF COMMERCIAL TOPO



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**IMPORTANT NOTICE**

The information and statements herein are believed to be reliable but are not to be construed as a warranty or representation for which we assume legal responsibility. Users should undertake sufficient verification and testing to determine the suitability for their own particular purpose of any information or products referred to herein. **NO WARRANTY OF FITNESS FOR A PARTICULAR PURPOSE IS MADE.**

Nothing herein is to be taken as permission, inducement or recommendation to practice any patented invention without a license.

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The logo for Cytec Industries Inc. features the word "CYTEC" in a bold, blue, sans-serif font. The letters are closely spaced and have a slightly irregular, industrial feel.

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