ORGANIC CHEMICALS DIVISION RESEARCH Report

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TENTATIVE PROCESS
FOR
NCS 978 (HYDRAUL 281)

DATE - April 15, 1970 WRITTEN BY: L. R. Stark

Monsanto

ST. LOUIS, MISSOURI

MONS 021018

MONSANTO COMPANY
Organic Chemicals Division
St. Louis Research Department

APPROVED

FINAL APPROVAL DATE 4-28-70

St. Louis Research Report No. 1560

TENTATIVE PROCESS

FOR

MCS 978 (PYDRAUL 281)

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TECHNICAL INFORMATION GROUP
1700 South Second

Date: April 15, 1970

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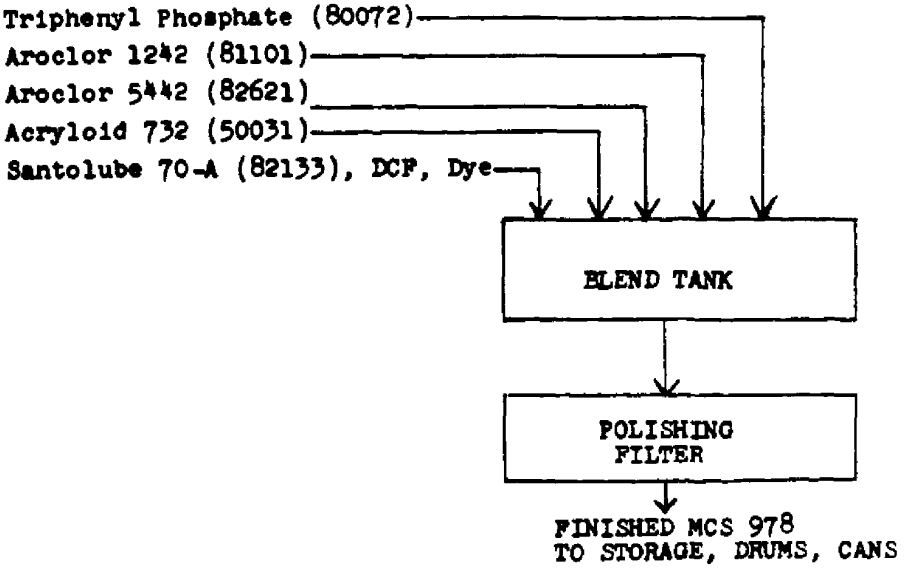
INTRODUCTION

MCS 978 (Pydraul 281) is a fire resistant hydraulic fluid. This formulation developed to eliminate Aroclor 1260 and 1248 from the Pydraul 280 blend and to use triphenyl phosphate to replace tricresyl phosphate.

SYNOPSIS OF PROCESS

MCS 978 is prepared by blending Aroclor 5442 and Aroclor 1242 with triphenyl phosphate and additives. The blended product is filtered to remove any stray debris from raw materials or blending and is transferred to containers or storage.

FLOW SHEET



BILL OF MATERIALS

Basis, 100 lb of product

Raw Material		Weight, lb
80072	Triphenyl Phosphate	22.8
81101	Aroclor 1242	54.6
82621	Aroclor 5442	19.3
50031	Acryloid 732	3.00
82133	Santolube 70-A	0.30
31100	Dow Corning 200 Fluid, 350 cs	0.002
20640	Dye, DuPont Oil Blue A	0.007

PROCESS IN DETAIL

The blending operation may be carried out in any suitable metal or glass-lined vessel. The materials are not corrosive and carbon steel equipment is suitable. An agitator should be provided.

To the clean dry vessel, charge the Aroclor 1242. Addition of Aroclor 5442 and triphenyl phosphate (TPP) will depend on local preference for handling. TPP can be added in the solid flaked form or heated to a molten liquid. Aroclor 5442 is best added in the molten state but can be added as a crushed solid.

Aroclor 5442 is a resinous, glassy solid at room temperature. In this state it is friable and can be crushed to small chunks for addition to the heel of CDP. If addition is made in the molten state, Aroclor 5442 should be heated to 180 to 200°F (82 to 93°C). At 180°F, viscosity is 300 to 500 centistokes and at 200°F, 80 to 120 cs.

TPP is normally supplied as a flaked solid which can be poured relatively easily from fiber-pak shipping containers. Future consideration may be given to handling in the molten state. TPP melts at approximately 50°C and becomes a liquid of relatively low viscosity.

Acryloid 732 and other additives are added last. Each of these should be carefully measured on a suitable scale or balance. Small variations of these components can cause large changes in viscosity, color, and other specification properties. Refer to Important Variables Section for further comments on these.

Acryloid 732 is a very viscous liquid at room temperature. Handling can be improved by heating to 50 to 70°C for no more than 6 to 8 hours. Prolonged exposure to high temperatures will cause product quality problems.

The dye should be carefully measured on a suitable scale or balance and added as a solid to the batch. Note: Relatively small changes in dye concentration or incomplete mixing can cause batch-to-batch color variations.

Santolube 70-A and Dow Corning Fluid are added in the quantities indicated. These, too, should be measured carefully since variations will affect acidity specs, anti-rust performance, and foam.

It is suggested the blend be warmed to 125 to 150°F (50 to 65°C) and agitation continued for sufficient time to insure complete component mixing. Thirty minutes to one hour should be sufficient. Lower blending temperatures may be used if experience indicates satisfactory mixing. Heat from molten Aroclor 5442 may be sufficient to raise batch temperature. The low viscosity of Aroclor 5442 at 200°F will aid dispersion in phosphate ester. The batch is then circulated through a polishing filter. Samples are taken as required for analytical purposes. The batch may be held for possible viscosity adjustment or be sent to storage. After blending is complete, it is recommended that the temperature be reduced to 80 to 129°F (26 to 49°C). Prolonged exposure at higher temperatures may cause some product quality deterioration.

DISCUSSION OF IMPORTANT VARIABLES

The most important specification property of MCS 978 is viscosity. Adjustments in the blend should be made to keep the 210 and 100°F viscosity within the required specification range.

The viscosity at 210°F is the most important followed by 100°F viscosity and finally density.

Table I will serve as guide to adjust the blend when viscosity is out of spec. Adjustments are made primarily on the basis of changes in the 210°F viscosity.

Another caution mentioned in the previous section concerned prolonged heating. It is suggested that maximum temperatures of 125 to 150°F be limited to 2 or 3 hours. Also, manner of heating the batch should control film temperatures so there are no significant hot spots. Prolonged heating may cause gradual darkening of the product as well as gradual increase in acidity due to decomposition of the TPP.

TABLE I

PYDRAUL 281 BLEND ADJUSTMENT

	100°F VISCOSITY LOW	100°F VISCOSITY HIGH
210°F VISCOSITY HIGH	Probably too much Acryloid 732. Per 100 lb of blend add 0.68 lb TPP and 1.62 lb 1242 and 0.58 lb 5442 to decrease 210°F visc. 0.85 cs.	Probably too much Aroclor 5442. Per 100 lb of blend add 1 lb TPP to decrease 210°F visc. 0.8 cs.
210°F VISCOSITY LOW	Probably not enough Acryloid 732. Per 100 lb of blend add 0.1 lb Acryloid to increase 210°F visc. 0.85 cs.	Probably too much Aroclor 5442 and not enough Acryloid 732. Per 100 lb blend add 0.3 lb TPP and 0.7 lb 1242 and 0.1 lb Acryloid to increase 210°F visc. 0.8 cs.

MATERIAL SPECIFICATIONS

Raw Materials

Refer to raw material specification sheets included in the Appendix.

Finished MCS 978

Typical analyses and suggested specification limits follow:

Property	Tentative Specification	Typical Value
Appearance	Cloudy blue liquid	
Viscosity, cs At 100°F At 210°F	 46 to 56 cs 5.60 to 6.40 cs	 51.0 5.95
Acidity, mgKOH/gm	0.08 to 0.25	0.20
Specific Gravity, 25/25°C	1.336 to 1.356	1.346
Moisture, KF	0.15 max.	0.02
Flash Point	350°F, min.	358°F

ANALYTICAL METHODS

No new or special analytical procedures are required for this product. A cross reference table appears in the Appendix listing standard analytical procedures used at J. F. Queeny Plant, W. G. Krummrich Plant, and at the Anniston Plant. These procedures are currently used on similar products.

TOXICITY AND HAZARDS

No unusual toxicity or hazards are expected with the raw materials or finished blend. The process is a simple blending procedure and no hazardous reactions should occur between the various components.

Aroclor 5442, 1242, and TPP have been known materials produced by Monsanto for many years. Aroclor 1242 has been used in Pydraul formulations.
(NOTE: Current specific toxicity information is being obtained from the Medical Department on these materials and it is suggested these be included in the Appendix of this report.)

General Precautions include the following:

1. Goggles should be worn where splashing or spillage may occur.

2. Any material in contact with the skin should be removed by washing with soap and water.

3. Prolonged or repeated exposure to the skin may cause irritation due to removal of skin oils with resulting dryness and chapping.

4. Material may be irritating to sensitive skin areas and eyes. In case of contact with eye tissue, flush copiously with water and then get medical attention.

5. In case of internal ingestion get immediate medical attention.

6. Avoid inhaling volation material or mists.

POLLUTION CONTROL

No waste by-products are generated by this process. Used filtering material, flushings from the system during cleaning, as well as any spoiled product should be disposed of as dictated by local regulations.

APPENDIX

L. R. Stark

ms

4/22/70

File Copy Approved by
Manager of Research:

_____ _____

W. R. Richard (Date)

IN 010

PHYSICAL CONSTANTS DATA

SUBSTANCE	STRUCTURAL FORMULA
MCS 978 (Pydraul 281)	Blend: 22.8% wt. Triphenyl phosphate 54.6% Aroclor 1242 19.3% Aroclor 5442 3.0% Acryloid 732 Additives
COMPILED BY / DATE	

NAME		SOURCE & DATE
Formula	Blend	
Molecular Weight		
Appearance	Slightly cloudy, blue oily liquid	
Boiling Point at 760 mm	670°F	*
Boiling Point at mm		
Boiling Point at mm		
Boiling Point at mm		
Crystallizing Point	None	
Melting Point	None	
Solution Point		
Specific Gravity @ 25/25°C	1.336 to 1.356	Spec Range
Specific Gravity @ Pounds per Gal. @ 25 °C	11.2	
Coefficient of Expansion cc/cc/°F	0.00040	*
Refractive Index @ °C		
Infra Red Spectrum (Reference)		
Ultra Violet Spectrum (Reference)		
Viscosity @ 210°F	5.60 to 6.40 cs	Spec Range
Viscosity @ 100°F	46 to 56 cs	Spec Range
Surface Tension		
Solubility in Water	Nil	
Solubility in		
Solubility in		
Pour Point		
Moisture (Karl Fisher)		
Thermal Conductivity (BTU/hr/ft./°F	0.066	*
Flash Point °F (Method) ASTM D-92	358°F typ. (350 min. Spec)	
Fire Point °C		
Heat of Combustion		
Heat of Formation		
Thermal Stability (cal./hr./min.)		
Latent Heat of Fusion		
Latent Heat of Vap.		
Specific Heat BTU/#/°F at 77°F	0.28	*
Critical Temperature		
Critical Pressure		
Dissociation Constant		
Acidity	0.08 to 0.25	Spec Range
Foam ASTM-D 972 at 75°F	40 ml	typical
Other Constants		
* Estimates based on Pydraul 280		
Toxicity (Give Reference)		

MONS 021028

Analytical Procedures

Property	WGK	JFO	Anniston	ASTM
Appearance	12809	16-B	C-1	-
Viscosity	12837	126-A	049	D-445
Flash & Fire Point	12803	50-A	114	D-92
Specific Gravity	12804	116-B	052	-
Pour Point	13005	100-A	053	D-97
Cloud Point	-	36-A	-	D-2500
Acidity	12794	80-B	050	D-664/D-974
Moisture	12810	78-B	047	D-1744
Foam	12805	54-A	-	D-892
Refractive Index	13123	104-A	048	D-1218
Distillation Range	10117	-	054	Modified D-20

→ Approximate equivalent ASTM method.

MONS 021029

Monsanto
ORGANIC CHEMICALS DIVISION

FINISHED PRODUCT SPECIFICATION

PRODUCT	DEPT.
Aroclor 5442	27-S

PLANT	PRODUCT CODE	USE
Anniston	826.21	Sales

DATE EFFECTIVE	SALES CODE
3-15-66	1040-470-04-009

APPROVALS

PRODUCTION	RESEARCH
R. C. Moody /s/ 12-29-65	L. R. Stark /s/ 3-11-66
SALES	QUALITY CONTROL (Chief Chemist)
W. E. Ault /s/ 3-2-66	O. E. Dolin /s/ 12-29-65

GENERAL DESCRIPTION

Mixture

CHEMICAL FORMULA

MOL WT.		
SUPERSEDES SPECS. OF	SAMPLE FOR ANALYSIS	ISSUED BY
4-25-61	1 x 16 oz. Can or W.M. Bottle	J. A. Liddle 11-30-65
CHARACTERISTIC	LIMITS	METHOD

UNRESTRICTED INFORMATION

(R) Appearance and Color	Clear, yellow sticky resin	G-1
(R) Color, HPA, Molten	2 Max.	G-4
(R) Softening Point, °C	46 - 52	115
Acid No., mg. KOH/g	0.05 Max.	116

MONS 021030

MONSANTO COMPANY
ORGANIC CHEMICALS DIVISION
J. F. QUEENY PLANT
RAW MATERIAL
SPECIFICATION

MATERIAL CODE: 8U01 or 82012

USING DEPTS.: Pydraul 150

SUPPLIER: Monsanto, WGK or Anniston Plants

MATERIAL: AROCLOR 1242 - NON-ELECTRICAL GRADE

CHEMICAL FORMULA: Mixture

MOL WT.: -----

SUPERSEDES SPEC OF: October 13, 1964

SAMPLE FOR ANALYSIS: 1 x 16-ounce NN Bottle

REQUIRED TESTS*	RESTRICTED INTERNAL LIMITS	JFQ CONTROL LIMITS	PROCEDURES WGK	PROCEDURES JFQ
Appearance	Clear, colorless to light yellow-green liquid	-----	10084	16-B
Color	APHA 100 max.	-----	10084	38-D
Refractive index @ 25°C	-----	1.6240 to 1.6260	10125	104-A
Specific gravity @ 25/15.5°C	1.381 to 1.392	-----	10114	116-A
TREND TESTS*				
Acidity (mg. KOH/g.)	0.010 max.	-----	10087	4-A
Moisture	50 ppm max.	-----	10620	78-C
Viscosity @ 100°F	82 to 92 SUS	-----	10086	126-A
General Tests (made only upon request at producing plant)				
Distilling range: (ASTM D-20 corrected): 10% distilled by wt. 325°C min. 90% distilled by wt. 360°C max.			10117	
Fire point	None	-----	10123	50-A
Flash point	338 to 392°F	-----	10123	50-A

*This information will be transmitted to JFQ Plant in accordance with Procedure 928 on all shipments from WGK Plant.

This specification is the property of Monsanto Company and is intended for internal use only.

MONS 021031

IM 63 (REV. 12/63)

MONSANTO COMPANY
ORGANIC CHEMICALS DIVISION
J. F. QUEENY PLANT
FINISHED PRODUCT SPECIFICATION

PRODUCT CODE: 80072
DEPT. OR JOB:
USE: Sales - W. F. Waychoff, Manager
SALES CODE: 8560-000-04-001

ISSUED JUNE 13, 1968
STANDARDS DEPARTMENT
John F. Queeny Plant
Paul A. Bowen

PRODUCT (Trade Name):
TYPE:

PRODUCT (Chemical Name): TRIPHENYL PHOSPHATE
TYPE: A GRADE

CHEMICAL FORMULA: $(C_6H_5)_3PO_4$

MOL. WT. 326.31

SUPERSEDES SPEC'S OF: October 8, 1964
SAMPLE FOR ANALYSIS: 1 x 32-ounce WM Bottle

TEST	UNRESTRICTED SALES LIMITS	INTERNAL CONTROL LIMITS	METHODS
On each lot			
Acidity	0.06 meq/100 grams max. (= 0.003% as H_3PO_4)	0.04 meq/100 grams max.	4-A
Appearance	White flakes	White flakes	16-A
Melt tests:			74-A
a. - Color of 100 ml.	----	APHA 20 maximum*	
b. - Turbidity	----	Clear*	
c. - Foreign particles	----	Passes*	
Melting point	48.5°C min. to start	49.2 to 49.8°C	76-A
Odor	Very faint aromatic max.	Very faint aromatic max.	84-A
Stability to $KMnO_4$	65% transmission min.	80 to 100% T	118-D

Sampling procedure

1. While a batch of this material is being flaked, catch 1 x 32-ounce WM bottle as the flakes fall from the flaker.
2. Label and submit to the laboratory for analysis.

*Disposition of material failing these limits will require approval of the Sales Dept.

MONS 021032

MS 84 (REV. 10/58)

J. F. QUILLEN PLANT

RAW MATERIAL SPECIFICATION

Pydraul E-9 & 150

L. R. Stark

RESIN, ACRYLOID 722

12/15/69 | 12/15/70 | Rohm and Haas | 1 x Resume

NOTE: All samples processed by this tentative specification will require disposition of the authorized release agent.

TEST	UNRESTRICTED PURCHASE LIMITS	INTERNAL CONTROL LIMITS	PROCEDURES
On each lot			
Appearance	Clear, viscous liquid, free of sediment, foreign particles, or polymer globules	—	16-B
Identification	—	Infrared Spectrum Normal	62-50030
Neutralization Number	Acid 0.50 max.	—	80-B
Viscosity @ 210°F	850-950 cs.	—	126-A
Separation Appearance Test	—	Clear with no cloudy or opaque appearance	See attached procedure
As requested			
Color, ASTM D-1500	4.5 max.		38-I
*Flash Point	375°F. min.		52-A

*After dilution with 3 parts of 375°F flash neutral oil to reduce viscosity.

MONS 021033

APPEARANCE TEST ON PYDRAUL 150 AND PYDRAUL F-9

PYDRAUL 150

Prepare the following blend in at least a 100 gram amount:

Santicizer 141	69.0%
Aroclor 1242	23.0%
Acryloid 710	8.0%

Stir the blend for 20-30 minutes at any convenient temperature up to 60°C Allow the fluid to cool to room temperature (65-75°F). The fluid should be very light yellow and water clear. If a cloudy or opaque appearance is observed, the blend is unacceptable and the lot of Acryloid 710 used should be rejected.

PYDRAUL F-9

Prepare the following blend in at least a 100 gram amount:

Aroclor 1248	52.5%
Tricresyl Phosphate	45.0%
Acryloid 710	2.5%

Stir the blend for 20-30 minutes at any convenient temperature up to 60°C. Allow the fluid to cool to room temperature (65-75°F). The fluid should be very light yellow and water clear. If a cloudy or opaque appearance is observed, the blend is unacceptable and the lot of Acryloid 710 used should be rejected.

IT IS IMPERATIVE FOR BOTH FLUIDS THAT THE TEST BE RUN BEFORE THE DOW CORNING 200 FLUID AND BLUE A DYE ARE ADDED AND THAT THE APPEARANCE OF THE FLUID IS OBSERVED WHEN THE FLUID IS AT ROOM TEMPERATURE. IF DOW CORNING 200 IS ADDED TO THE BLEND THE FLUID WILL BE CLOUDY IN ALL CASES. IF THE FLUID IS ABOVE ROOM TEMPERATURE, THE SOLUBILITY OF THE ACRYLOID 710 IS IMPROVED, THE CLOUDINESS DECREASES AND THE TEST IS NOT AS DEFINITIVE.

MONSANTO COMPANY
ORGANIC CHEMICALS DIVISION

J. F. QUEENY PLANT
RAW MATERIAL
SPECIFICATION

81797

USING DEPT.
Fluid Blends

SUPPLIER
Monsanto - Nitro Plant

MATERIAL
SANTOLUBE 70-A

CHEMICAL FORMULA
Blend of: Santolube 70 98.35%
Santolene C 1.65%

MOL WT

SUPERSEDES SPEC DT. 6-21-67 for Santolube 70, Nitro code 81787

SAMPLE FOR ANALYSIS
1 x 16-ounce WM bottle

TEST	RESTRICTED INTER-PLANT LIMITS	PROCEDURE NITRO	PROCEDURE
On each lot			
Appearance	Dark, oily liquid	6-C-01	10-D
Color ASTM Dilute	5.5 max.	6-C-21	35
Neutralization No.	51 to 61	6-A-24	80-A
Solubilities 5%/vol in PE	Clear	6-V-15	112-1
Specific gravity @ 25/25°C	0.89 to 0.97*	6-D-07	116
Upon request			
Emulsification test	40, 40, 0 (30 min.)	6-S-28	--
Santolene C presence	Positive phosphorous test	-----	-
Viscosity @ 210°F	70 to 300 SUS*	6-X-02	12

*Typical ranges submitted by Nitro

Special requirements

This material is shipped upon invoice certification of compliance with specifications. Prior permission will be obtained to ship non-specification grade material.

This specification is the property of Monsanto and is intended for internal use only.

MONS 021035

IN 35 REV 11 67

MONSANTO COMPANY
ORGANIC CHEMICALS DIVISION

J. F. QUEENY PLANT

RAW MATERIAL SPECIFICATION

MATERIAL NO. 81797

USING DEPT. Fluid Blends

SUPPLIER Monsanto - Nitro Plant

MATERIAL
SANTOLUBE 70-A

CHEMICAL FORMULA

Blend of Santolube 70 98.35%
Santolene C 1.65%

MOL WT

SUPERSEDES SPEC DT. 6-21-67 for Santolube 70, Nitro code 81787

SAMPLE FOR ANALYSIS 1 x 16 ounce WM Bottle

TEST	RESTRICTED INTER-PLANT LIMITS	PROCEDURE NITRO	PROCEDURE JEFF
On each lot			
Appearance	Dark, oily liquid	6-C-01	16-S
Color ASTM Dilute	5.5 max.	6-C-21	38
Neutralization No.	51 to 61	6-A-24	80-A
Solubilities 5%/vol. in PE	Clear	6-V 15	112-L
Specific gravity @ 25/25°C	0.89 to 0.97*	6-D-07	116 I
Upon request			
Emulsification test	40, 40, 0 (30 min.)	6-S 28	--
Santolene C presence	Positive phosphorous test	--	-
Viscosity @ 210°F	70 to 300 SUS*	6-X-02	120 V

*Typical ranges submitted by Nitro.

Special requirements

This material is shipped upon invoice certification of compliance with specifications. Prior permission will be obtained to ship non-specification grade material.

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IN 33 REV. 12-67

J. F. QUINCY PLANT
MONSANTO COMPANY
ORGANIC CHEMICALS DIVISION

RAW MATERIAL SPECIFICATION

MATERIAL CODE: 3100

USING DEPTS.: Fluid Blends

SUPPLIER: Dow Corning Corporation

MATERIAL: DOW CORNING 200 FLUID - 350 cs.

CHEMICAL FORMULA:

REVISED BY:

SUPERSEDES SPECS OF: October 28, 1964

SAMPLE FOR ANALYSIS: 1 x 8-ounce Bottle

TEST	UNRESTRICTED PURCHASE LIMITS	METHODS
On each lot		
Appearance	Syrupy liquid	16-B
Refractive index @ 25°C	1.4020 to 1.4040	104-A
Specific gravity @ 25/25°C	0.969 to 0.975	116-A
Viscosity @ 25°C	332.5 - 367.5	126-3100
Upon request		
Color - modified Barrett	1.5 max.	38-H
Flash point	600°F min.	50-A
Neutralization No.	0.01 max.	80-B
Volatility (24 hrs./150°C)	0.5% max.	128-B

MONS 021037

IN 86 (REV. 12/63)

Mon 2310

William H. Hunt - Medical

April 1, 1970

Pydraul 281, Lot QL-42

W. Richard
N. Johnson
D. Fowlkes

TO L.R. Stark

The undiluted sample was classed as practically non-toxic when administered orally to mixed sex rats. The acute oral LD_{50} was 10,400 mg/kg. Most deaths occurred in 3 days. Toxic Signs were reduced appetite and activity, lethargy, increasing weakness, collapse, and death. Macroscopic examination of those that succumbed revealed lung hyperemia, liver discoloration, and gastrointestinal inflammation. Macroscopic examination of those that were sacrificed revealed a normal viscera.

The undilted sample was classed as mildly toxic when applied to and retained for 24 hours on the clipped intact skin of mixed sex rabbits. The acute minimal lethal dose was greater than 1260 but less than 2000 mg/kg. Time to death was 5 to 13 days. Toxic signs were reduced appetite and activity, increasing weakness, tremors, collapse and death. Macroscopic examination of those that succumbed revealed hemorrhagic lungs, jaundiced liver, enlarged gall bladder, discolored kidneys and gastrointestinal inflammation. Macroscopic examination of those that were sacrificed revealed areas of lung congestion and slight liver discoloration.

The undiluted sample was classed as a moderate irritant when applied to and retained for. 24 hours on the clipped intact skin of mixed sex rabbits. The maximum irritation (2.6/8) occurred at 24 hours of contact. At this time the sample was removed. The maximum irritation observed was slight erythema and slight edema. Forty-eight hours after removal the skin was normal.

The undiluted sample was classed as a mild irritant when instilled and retained for 24 hours in the conjunctival sac of the rabbit eye. Maximum irritation occurred at 1 hour of contact (16/110). There was beefy-redness and slight edema. At 24 hours of contact the score was 4.6/110. At this time the eye was irrigated and 4 days later the eye was normal.

When male rats were exposed for 6 hours to the vapors generated at ambient temperature there were no toxic signs nor did any develop during the 10 days of observation. The sample was classed as practically non-toxic. Macroscopic examination of the sacrificed animals revealed normal visceras.

Handle as any other liquid Aroclor or Pydraul.

In case of skin contact wash off with soap and water.

In case of accidental eye contact flush out with water. If irritation persists consult a physician.

William H. Hunt

William H. Hunt, Ph.D.

MONS 021038