ORGANIC CHEMICALS DIVISION RESEARCH Report

REPORT NO. P-1563 JOB NO. 2-21-760.01-1113115

TENTATIVE PROCESS
FOR
MCS 1007 (PYDRAUL F-9A)

DATE - April 27, 1970 WRITTEN BY - P. D. Fowlkes

Monsanto
ST. LOUIS, MISSOURI

DSW 620208

STLCOPCB4094235

1) MCS 1007 (Pydraul F-9A) - a fire resistant hydraulic fluid

2) Aroclor 5442 - used for blending MCS 1007

3) Cresyl diphenyl phosphate - used for blending MCS 1007

4) Santicizer 711 - used for blending MCS 1007

SAFETY REVIEW COMMITTEE
APPROVED BY W.J. Knapp 5-5-70
[illegible] 5/5/70
M.A. Terpstra 5/5/70

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Organic Chemicals Division
St. Louis Research Department

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St. Louis Research Report No. P-1563

TENTATIVE PROCESS

FOR

MCS 1007 (PYDRAUL F-9A)

Job No. 2-21-760.01-1113115

Date: 4/27/70

Written by: P. D. Fowlkes

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L. F. Smith
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L. R. Stark

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MONSANTO INDUSTRIAL CHEMICALS COMPANY
Functional Products Group
Technology Department
October 20, 1971

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TENTATIVE AMENDMENT C
TO THE
TENTATIVE PROCESS
FOR
PYDRAUL F-9A

Report No. P-1563, dated April 27, 1970

- -

Job No. 2-21-760.01-1111015

Objective: Change of additives used in blend.

Present Practice

The present tentative process enables the blending of Pydraul F-9A, a fire resistant industrial hydraulic fluid.

Proposed Change

The revised formulation for Pydraul F-9A is presented. It is necessary to change the ratio of Aroclor 5432:Santicizer 140 to allow for the higher viscosities of the new Santicizer 140 and the "tighter specification" 5432. Also, the new rust inhibitor incorporated in Amendment B has formed a flocculant cloud in some batches of F-9A. Therefore, we will revert to the Santolube 70A as used originally. Material changes are described. This change supersedes Amendment B.

DSW 620211

STLCOPCB4094238

Bill of Materials

Basis: 100 lbs. of product

	Raw Material	Weight, lbs.
80151	Santicizer 140	48.5
86840	Santicizer 711	11.4
46310	Paraplex G-62	0.25
81797	Santolube 70A	0.30
60015	Tolyltriazole	0.05
31100	DC-200, 350 cs.	0.002
20640	DuPont Oil Blue A	0.0015
82623	Aroclor 5432	39.5

Process in Detail

All of the additives dissolve easily in Santicizer 140 or Santicizer 711. A suggested procedure is to charge the 140 and 711, commence heating, and add the Paraplex G-62, Santolube 70A, tolyltriazole, DC-200, and dye. The 5432 may be added at any convenient time. It will not affect the additive solubility and the heat of the 5432 would raise the temperature of the blend. The batch should be stirred for 1/2-1 hr. at 125-150°F. (50-65°C.).

The additives should be measured carefully on a suitable balance as they will affect the acidity and appearance specifications of the blend.

Aroclor 5432 is a resinous, sticky solid at room temperature. It may be handled easily if it is heated to 150-180°F. (65-82°C.). At 150°F. viscosity is 100-400 cs. and at 180, 40-100 cs.

Material Specifications

	Tentative Specification	Typical Value
Acidity, mg. KOH/g.	0.30 max.	0.25

Justification for Change

The change of 5432:Santicizer 140 ratio should bring the typical viscosity of the blend back down to the middle of the specification and end any problems with meeting viscosity specifications.

DSW 620212

STLCOPCB4094239

Precautions

No additional precautions are required due to this amendment to the Tentative Process.

P. D. Fowlkes

P. D. Fowlkes

L. R. Stark 10/25/71

L. R. Stark

File Copy approved by:

W R Richard — 10/26/71

Manager, R&D (date)

J R Savage — 10/27/71

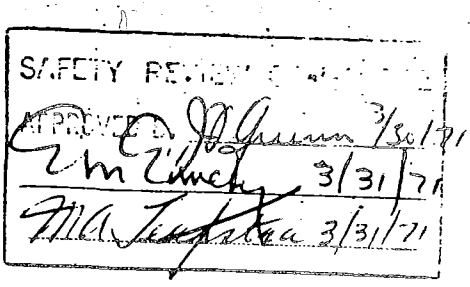
Manager of Manufacturing (date)

Oliver DeGarmo — 10/29/71

Manager of Process Technology (date)

/is
10/20/71

DSW 620213



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Organic Chemicals Division
St. Louis Technology Department
March 22, 1971

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TENTATIVE AMENDMENT B

TO THE

TENTATIVE PROCESS

FOR

PYDRAUL F-9A

Report No. P-1563, dated April 27, 1970

Job No. 2-21-760.01-1111015

Objective: Change of additives used in blend.

Present Practice

The present tentative process enables the blending of Pydraul F-9A, a fire resistant industrial hydraulic fluid.

Proposed Change

The revised formulation of Pydraul F-9A is presented. The changes are the addition of an acid acceptor to improve pump performance and the replacement of the rust inhibitor (Santolube 70-A) with a cheaper, more effective material. The material changes are described. Changes in blending procedure are delineated. This change supersedes Amendment A.

DSW 620214

STLCOPCB4094241

Bill of Materials

Basis: 100 lbs. of product

	Raw Material	Weight, lbs.
80151	Santicizer 140	45.5
86840	Santicizer 711	11.3
46310	Paraplex G-62	0.4
14010	Na-Sul BSB	0.2
60015	Tolyltriazole	0.1
31100	DC-200, 350 cs.	0.002
20640	DuPont Oil Blue A	0.0015
82623	Aroclor 5432	42.5

Process in Detail

All of the additives dissolve easily in Santicizer 140 or Santicizer 711. A suggested procedure is to charge the 140 and 711, commence heating, and add the Paraplex G-62, Na-Sul BSB, tolyltriazole, DC-200, and dye. The 5432 may be added at any convenient time. It will not affect the additive solubility and the heat of the 5432 would raise the temperature of the blend. The batch should be stirred for 1/2-1 hr. at 125-150°F. (50-65°C.)

The additives should be measured carefully on a suitable balance as they will affect the acidity and appearance specifications of the blend.

Aroclor 5432 is a resinous, sticky solid at room temperature. It may be handled easily if it is heated to 150-180°F. (65-82°C.). At 150°F. viscosity is 100-400 cs. and at 180, 40-100 cs.

Material Specifications

	Tentative Specification	Typical Value
Acidity, mg KOH/g.	0.30 max.	0.04

Justification for Change

The change of rust inhibitor results in a savings of $0.035/gal. and lowers the acidity of the fluid which will improve the service life of the product.

STLCOPCB4094242

Precautions

No additional precautions are required due to this amendment to the Tentative Process.

P. D. Fowlkes 3/26/71
P. D. Fowlkes

L. R. Stark 3/26/71
L. R. Stark

File copy approved by:

U R Richard 3/26/71
Manager, R&D (date)

Manager of Manufacturing (date)

3/30/71
Manager of Process Technology (date)

/ls
3/23/71

DSW 620216

STLCOPCB4094243

SAFETY REVIEW COMMITTEE
APPROVED BY [signature] 8/7/70
R. W. Redue 8/7/70
J. F. Queeny 8/10/70

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Organic Chemicals Division
St. Louis Research Department
August 3, 1970

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TENTATIVE AMENDMENT NO. A

TO THE

TENTATIVE PROCESS

FOR

MCS 1007 (PYDRAUL F-9A)

Report No. P-1563, dated April 27, 1970

Job No. 2-21-760.01-1113115

Objective: Incorporation of additives into blend

1. Brief Description of Present Practice

The present tentative process enables the blending of MCS 1007, (Pydraul F-9A), a fire resistant industrial hydraulic fluid.

DSW 620217

2. Description of Proposed Change

The revised formulation of MCS 1007 is presented. The changes were necessary in order to improve compatibility with Pydraul F-9 and also to enhance the functional properties of the fluid. The material specifications which are changed are described, and modifications to the blending procedure delineated.

BILL OF MATERIALS

Basis: 100 lbs of product

Raw Material		Weight, lbs.
82621	Aroclor 5432	42.5
80151	Santicizer 140	46.0
86840	Santicizer 711	11.1
51351	Santolube 70-A	0.3
60015	tolyltriazole	.1
31100	DC-200, 350 cs fluid	.002
20640	Dupont Oil Blue A	.0015

PROCESS IN DETAIL

All of the additives dissolve easily in Santicizer 140 or Santicizer 711. A suggested procedure is to blend the Santicizer 140 and 711, add the tolyltriazole, dye, Santolube 70-A, and DC 200, and mix for 1/2 to 1 hr at 125-150°F (50-65°C).

The additives should be measured carefully on a suitable balance as they will affect the acidity and appearance specifications of the blend.

Aroclor 5432 is a resinous, sticky solid at room temperature. It may be handled easily if it is heated to 150 to 180°F (65-82°C). At 150°F, viscosity is 100-400 cs and at 180, 40 to 100 cs.

DISCUSSION OF IMPORTANT VARIABLES

The most important specification property of MCS 1007 is viscosity. Adjustments in the blend should be made to keep the 100°F viscosity within the required specification range.

If the 100°F viscosity is low, add Aroclor 5432. One lb of Aroclor 5432 per 100 lb of blend will increase the 100°F viscosity about 1.5 centistokes.

If the 100°F viscosity is high, add Santicizer 140. One lb of Santicizer 140 per 100 lb of blend will decrease the 100°F viscosity about 1.0 cs.

STLCOPCB4094245

If it is necessary to add more than 1% of Santicizer 140 to bring the viscosity with specification, contact P. D. Fowlkes or L. R. Stark at the research center.

The quantity of dye added should be carefully measured on a suitable small balance or scale. This is important to insure uniform color from batch-to-batch.

MATERIAL SPECIFICATIONS

Property	Tentative Specification	Typical Value
Appearance	Clear to slightly hazy blue liquid	----
Viscosity, cs at 100°F at 210°F	48-57	53.36
Acidity, mgKOH/g	0.30 to 0.60	0.52
Sp. Gr. 25/25°C	1.20 - 1.23	1.215
Moisture, KF	0.15 max.	0.04
Pour Point (each 10th lot)	0°F	-5°F

3. Justification for Change

It was necessary to reduce the level of Santicizer 140 in the blend to increase compatibility with Pydraul F-9. The blend changes also lower the raw materials cost by $.14/gal.

4. Precautions

No additional precautions are required due to the changes to the tentative process.

P. D. Fowlkes

P. D. Fowlkes

ms
8/5/70

Recommended Review Date: Jan. 1, 1971

Approved by:

W. R. Richard 8/5/70

W. R. Richard (Date)
Manager of Research

DSW 620219

TABLE OF CONTENTS

	Page No.
INTRODUCTION	1
SYNOPSIS OF PROCESS	1
FLOW SHEET	1
BILL OF MATERIALS	1
PROCESS IN DETAIL	2
DISCUSSION OF IMPORTANT VARIABLES	2
MATERIAL SPECIFICATIONS	3
Raw Materials	3
Finished MCS 1007	3
ANALYTICAL METHODS	3
TOXICITY AND HAZARDS	4
POLLUTION CONTROL	4
APPENDIX	4

DSW 620220

STLCOPCB4094247

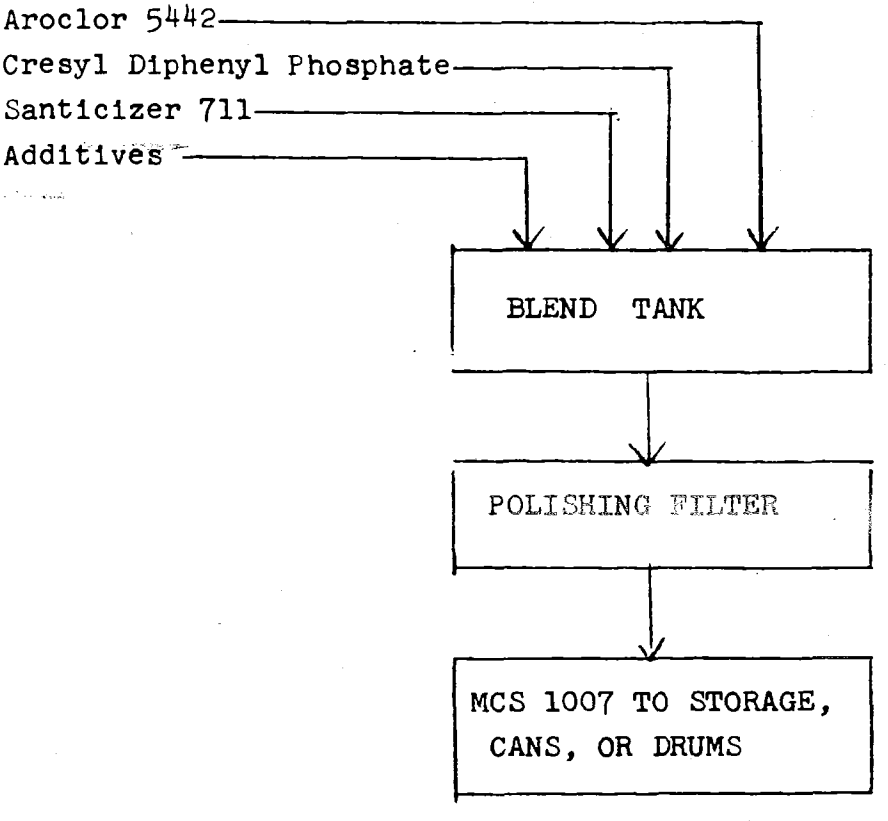
INTRODUCTION

MCS 1007 (Pydraul F-9A) is a fire resistant hydraulic fluid. This formulation was developed to eliminate Aroclor 1248 from the Pydraul F-9 blend and to use cresyl diphenyl phosphate to replace tricresyl phosphate.

SYNOPSIS OF PROCESS

MCS 1007 is prepared by blending Aroclor 5442, cresyl diphenyl phosphate, Santicizer 711, 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 lbs of Product

Raw Material		Wt., lb.
82621	Aroclor 5442	33.0
80077	Cresyl diphenyl phosphate	58.7
	Santicizer 711	8.0
81797	Santolube 70-A	0.3
31100	DC-200, 350 cs. fluid	.002
20640	Dupont Oil Blue A	.0015

DSW 620221

STLCOPCB4094248

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 cresyl diphenyl phosphate (CDP) and Santicizer 711. Addition of the Aroclor 5442 will depend on local preference for handling. The Aroclor can be added as a crushed solid or in the molten liquid state.

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.

The Santolube 70-A, dye, and Dow Corning fluid may be added to the CDP-711 mixture or after the 5442 is added. All of the additives dissolve readily and the time of addition is a matter of convenience. 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.

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 1007 is viscosity. Adjustments in the blend should be made to keep the 100°F viscosity within the required specification range.

If the 100°F viscosity is low, add Aroclor 5442. One lb. of Aroclor 5442 per 100 lb of blend will increase the 100°F viscosity about 2 centistokes.

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If the 100°F viscosity is high, add CDP. One lb of CDP per 100 lb of blend will decrease the 100°F viscosity about 1 cs.

The quantity of dye added should be carefully measured on a suitable small balance or scale. This is important to insure uniform color from batch-to-batch.

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 CDP.

MATERIAL SPECIFICATIONS

Raw Materials

Refer to raw material specification sheets included in the Appendix.

Finished MCS 1007

Typical analyses and suggested specification limits follow:

Property	Tentative Specification	Typical Value	JFQ Method
Appearance	Clear to hazy blue liquid	--	16-B
Viscosity, cs			
At 100°F	46-52	49.5	126-A
At 210°F	4.8-5.3	5.0	
Acidity, mgKOH/gm	0.30 max.	0.20	80-B
Sp. Gr., 25/25°C	1.25-1.28	1.259	116-A
Moisture, KF	0.15 max.	0.02	78-B
Pour Point (each 10th lot)	-5°F	-10°F	100-A

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.

STLCOPCB4094250

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 and CDP have been known materials produced by Monsanto for many years. CDP has been used in Pydraul fluid 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

P. D. Fowlkes

ms
4/28/70

File Copy Approved by
Manager of Research:

W. R. Richard

4/31/70
(Date)

DSW 620224

STLCOPCB4094251

APPENDIX

PHYSICAL CONSTANTS DATA

SUBSTANCE	STRUCTURAL FORMULA
MCS 1007	BLEND: 32.7% wt. Aroclor 54.2 59.0% Cresyl Diphenyl Phosphate 0.3% Santolube 70-A
COMPILED BY: P. D. Fowlkes	DATE: 4/27/70

NAME		SOURCE & DATE
Formula	Blend	
Molecular Weight		
Appearance	Hazy Blue Liquid	
Boiling Point at 760 mm		
Boiling Point at mm		
Boiling Point at mm		
Boiling Point at mm		
Crystallizing Point		
Melting Point		
Solution Point		
Specific Gravity @ 25/25	1.25-1.28	
Specific Gravity @		
Pounds per Gal. @ °C		
Coefficient of Expansion		
Refractive Index @ $n_D^{\bar{\ }}$		
Infra Red Spectrum *(Reference)*		
Ultra Violet Spectrum *(Reference)*		
Viscosity @ 100°F	49.2 cs	
Viscosity @ 210°F	5.01 cs	
Surface Tension		
Solubility in Water	Nil	
Solubility in		
Solubility in		
Pour Point	-10°F typical(-5°F max.)	
Moisture	0.02 (typical)	
Flash Point °F *(Method)*		
Fire Point °C		
Heat of Combustion		
Heat of Formation		
Thermal Stability (cal./kg/min.)		
Latent Heat of Fusion		
Latent Heat of Vap.		
Specific Heat		
Critical Temperature		
Critical Pressure		
Dissociation Constant		
Acidity, mgKOH/gm	0.30 max.	
Other Constants		
Toxicity *(Give Reference)*		

DSW 620225

NOTE: *These data should not be released outside of Monsanto Chemical Company. Use additional sheets if necessary.*

STLCOPCB4094252

Analytical Procedures

Property	WCK	JFC	Anniston	ASTM
Appearance	12809	16-B	G-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.

DSW 620226

STLCOPCB4094253

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. G. 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, NPA, Molten	2 Max.	G-4
(R) Softening Point, °C	46 - 52	115
Acid No., mg. KOH/g	0.05 Max.	116

DSW 620227

STLCOPCB4094254

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

PRODUCT CODE: 80077/80151
DEPT. OR JOB:
USE: Sales - W.F.Waychoff, Manager and Internal for blends
SALES CODE: 7680-940-04-001

ISSUED JAN. 23, 1967
STANDARDS DEPARTMENT
J. F. Queeny Plant
By Paul N. Brien

PRODUCT (Trade Name): SANTICIZER 140
TYPE: A GRADE

PRODUCT (Chemical Name): CRESYL DIPHENYL PHOSPHATE
TYPE: A GRADE

CHEMICAL FORMULA

MOL WT.

SUPERSEDES SPECS OF: November 26, 1963

SAMPLE FOR ANALYSIS: 1 x 16-ounce NN Flint Bottle

TEST	UNRESTRICTED SALES LIMITS	CONTROL LIMITS	METHODS
On each lot			
Acidity	0.20 meq/100 g. max. (=0.01% max. as H_3PO_4)	0.15 meq/100 g. max.	4-A
Appearance	Clear, mobile liquid	Clear, mobile liquid	16-B
Color	APHA 40 max.	APHA 15 to 35	38-D
Moisture (By KF in methanol)	0.15% max.	0.12% max.	78-B
Odor	Essentially odorless	Essentially odorless	84-A
Specific gravity @ 25/25°C	1.203 to 1.213	1.205 to 1.208	116-B
Stability to $KMnO_4$	65% transmission, min.	65 to 90% transmission	118-D
Upon request			
Refractive index @ 25°C	1.550 to 1.570	1.558 to 1.563	104-A

Sampling procedure

1. Circulate storage tank the night before filling shipping containers.
2. While filling drums or tank car, take one 16-ounce bottle from the filling line and submit to the laboratory for analysis.

DSW 620228

STLCOPCB4094255

CHEMICALS DIVISION

J. F. QUEENY PLANT
RAW MATERIAL
SPECIFICATION

USING DEPT.
Fluid Blends

SUPPLIER
Monsanto - Nitro Plant

MAY 6, 19

MATERIAL
SANTOLUBE 70-A-

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

MOL. WT.

SUPERSEDES SPECS OF 6-21-67 for Santolube 70; Nitro code 81787

SAMPLE FOR ANALYSIS
1 x 16-ounce WM Bottle

TEST	RESTRICTED INTER-PLANT LIMITS	PROCEDURES NITRO	PROCEDURES JFQ
On each lot			
Appearance	Dark, oily liquid	6-C-01	16-B
Color ASTM Dilute	5.5 max.	6-C-21	38-I
Neutralization No.	51 to 61	6-A-24	80-A
Solubilities: 5%/vol. in Pe	Clear	6-V-15	112-A
Specific gravity @ 25/25°C	0.89 to 0.97*	6-D-07	116-A
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	126-A

*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.

DSW 620229

IN-51 REV. 12-67

STLCOPCB4094256

J. F. QUEENY PLANT
MONSANTO COMPANY
ORGANIC CHEMICALS DIVISION

RAW MATERIAL SPECIFICATION

MATERIAL CODE: 31100

USING DEPTS.: Fluid Blends

SUPPLIER: Dow Corning Corporation

ISSUED FEB. 8, 1968

MATERIAL: DOW CORNING 200 FLUID - 350 cs.

CHEMICAL FORMULA:

MOL WT.

SUPERSEDES SPEC. 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	126-31100
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

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