2529

ORGANIC CHEMICALS DIVISION RESEARCH

St. Louis Research Report No. 2529

FINAL REPORT
ON
EVALUATION OF CHEMICAL MODIFIERS
FOR ASPHALTS
*****
Job No. 2-02-750-03-3082

Monsanto
ST. LOUIS, MO.

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Monsanto Chemical Company
Organic Chemicals Division
St. Louis Research Department

St. Louis Research Report No. 2529

FINAL REPORT ON EVALUATION OF CHEMICAL MODIFIERS FOR ASPHALTS

*****

Job No. 2-02-750.03-3082

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Reported by: D. W. Dinsmore

Work done by: E. P. Cunningham
W. M. Rinehart
D. W. Dinsmore

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INTRODUCTION

Many of the physical and chemical properties of asphalts can be improved by incorporation of chemical modifiers. This report summarized the screening evaluations completed in the Research Engine Laboratory during a one and one-half year period from November 1955 to April 1957.

SUMMARY

A literature and industry survey by Organic Development($_1$) indicated that the field of asphaltic materials would be an attractive market for Monsanto chemicals. A laboratory was constructed and evaluations begun with the scope of the project defined as follows:

I. Short Term Objectives

1. Development of techniques for evaluating and interpreting results.

2. Evaluation of alkylated polystyrene (APS) and other polymers as ductility improvers.

II. Longer Term Objectives

1. Improvement in temperature susceptibility.

2. Development of anti-staining agents for paper laminates.

3. Evaluation of antioxidants or anti-weathering agents.

4. Exploration in other areas where improvement in properties would be beneficial.

CONCLUSIONS

It was established that the functional groupings of benefit in other application areas also enhance the properties of asphalts.

Basic evaluation techniques were developed to a practical degree. Due to the complexity of asphalts, and the fact that one property cannot be modified without changing others, extended application experience is required for proficiency.

($_1$) Development Report OD-114, "Asphalt Manual", E. L. Will

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Alkylated polystyrene will improve the ductility of the five types of asphalts evaluated.

Several materials show promise of performance as anti-weathering or anti-staining agents.

RECOMMENDATIONS

1. Application work in this area should be reinstated at the earliest opportunity.

2. Prepare additional pilot plant quantities of APS for promotion to the industry.

3. Examine polymeric materials for effect on low temperature flexibility and tensile strength of laminating asphalts. Chlorinated materials, among others, should be evaluated as anti-staining agents in these asphalts.

4. Antioxidants of all types should be evaluated as anti-weathering agents. These should include materials with a cyclohexylamine group as well as phosphorodithioates, benzo-thiazoles and R.P.'s of P_2S_5 and polyolefins. Ultra-violet light modifiers should also be studied.

PATENT STATUS

Existing Monsanto patents on Santodex (APS) cover lubricating oils and petroleum waxes. It is unknown at this time whether they would also cover asphaltic materials. Century Refining Company filed an application regarding the use of Santodex in asphalt. To our knowledge no such patent has been issued.

A patent disclosure covering Aroclors as a viscosity modifier which does not significantly effect other properties was made by E. L. Will (Appendix A).

REFERENCES

1. Development Report OD-1114, "Asphalt Manual", E. L. Will.

2. St. Louis Research Report No. 1850, "Final Report on Asphalt Additives", Dr. R. R. Raube and Dr. E. J. Young.

DETAILED INFORMATION

A literature and industry survey by Mr. E. L. Will ($_1$) of the Development Department emphasised that improvement in asphalt properties by use of chemical modifiers is similar in many respects to work currently conducted within Monsanto. Areas of similarity are petroleum and rubber chemicals, functional fluids and plasticizer.

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A recommendation to establish facilities for evaluating modifiers was accepted and such a laboratory started in November 1955.

A. Training in Evaluation Techniques

Evaluating and interpreting test results differ greatly from that of petroleum products. Therefore, training in the basic techniques of evaluation was necessary before instigating an active program.

Lion Oil Division, El Dorado, Arkansas, produce a complete line of asphaltic materials. Their well equipped laboratory staffed with competent personnel was an ideal training ground. Consequently, ten days were spent at El Dorado with Mr. A. J. Hoiberg and Mr. C. Cowger learning basic techniques. This background was sufficient to allow initiation of a modest program.

As an aid in technique development two polymeric materials were evaluated at several concentrations in three asphalt samples. Results obtained with APS (100%) and Enjay's Parapol S-50 (copolymer of styrene and isobutylene) are recorded in detail in Appendix B and show that:

1. These polymeric materials are generally beneficial by increasing melting point and viscosity while decreasing penetration.

2. Ductility is erratic and probably due to insolubility of the 100% polymer.

3. Solubility can be improved by dissolving the polymer in oil. This is, however, detrimental to other physical properties.

B. Solubility of Chemical Modifiers

Solubility of chemical modifiers in asphalt is difficult to determine because of opaqueness. Solubility screening was therefore carried out in Skelly B (hexane) and found to be reasonably accurate. For application requiring good ductility and flexibility complete solubility is mandatory. The limitation of 0.5% (w) solubility in Skelly B was set as the criterion for evaluation.

Appendix C lists the hexane solubilities screened during this project as well as the appendix in which data for individual compounds are found.

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C. Alkylated Polystyrene

Century Refining Company, a small oil refinery in Western Kansas, reported that low concentrations of Santodex (APS) gave sufficient ductility improvement to their "off-grade" asphalt to make it meet paving grade specifications. This allowed marketing at $20 rather than $5 per ton. Other polymeric materials, such as the Acryloids, gave improvement at less economical dosages and with greater detrimental effect on other physical properties.

Until several years ago a 20% oil solution of APS had been marketed by Monsanto under the trade name of Santodex, a V.I. improver for lubricating oils. Since production was discontinued, techniques and raw materials required re-evaluation and other diluent materials needed to be examined. This work was carried out and recorded in St. Louis Research Report No. 1850 entitled, "Final Report on Asphalt Additives", by Dr. R. R Raube and Dr. E. J. Young. Application evaluations were carried out in the Research Engine Laboratory and are included in Appendix D.

The laboratory was successful in producing good quality APS. Either a special asphalt from Pure Oil Company's Smiths Bluff Refinery, recommended by Century, or Aroclor could successfully replace oil as the diluent.

Evaluation studies on the laboratory and pilot plant preparations of APS led to the following conclusions:

1. APS gives satisfactory ductility improvement in asphalts from each of the four major crude sources. Its use is not therefore restricted to a single type of asphalt.

2. Temperature susceptibility of the base asphalt remains essentially unchanged when low dosages of APS are incorporated.

3. Weatherability remains unchanged with blends containing even high dosages (2% wt.) of APS.

4. A loss in ductility upon ageing at moderately high temperatures (200-350°F) was reported by Lion Oil Division on blends containing low dosages of APS. Table I lists the significant data.

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Table I

LION DIVISION EVALUATION OF APS

Sample No.	Asphalt	Additive	Softening Point, °F	Penetration, mm. 32°	77°	115°	Ductility, cm. at 77°F
1	Lion - Roofing B	--	182	16	26	46	3.4
2	Lion - Paving	--	114	27	93	> 285	150+
3	43% No. 1, 57% No. 2	--	136	21	47	162	41
4	Sample No. 3	0.1% (w) APS*	137	21	49	168	81
5	Sample No. 3	0.5% (w) APS*	140	20	46	163	90
6	Sample No. 3 after 24 hrs. at 250°F		138	20	47	160	45
7	Sample No. 4 after 24 hrs. at 250°F		137	20	50	162	59
8	Sample No. 5 after 24 hrs. at 250°F		139	19	46	160	87

*50% Solution APS in Smiths Bluff asphalt.

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The loss in ductility is noted on the blend containing 0.1% APS and not on that containing 0.5% (w) APS. This indicates that greater than expected dosages may be necessary if the same pattern holds for other asphalts. Full details of this report are given in Appendix E.

Upon completion of these evaluations, APS was transferred to Development for promotion to industry (Appendix F).

D. Other Ductility Improvers

Table II shows the results of other polymeric materials evaluated as ductility improvers.

Table II

DUCTILITY IMPROVERS

	Ductility cm.	Penetration 1/10 mm.
Century Refining Straight Run B-109	79	102
0.25% (w) N-5262 Copolymer 80/20 decyl acrylate/ethyl acrylate	68	99
0.1% (w) Polyethylene No. 2-187 from Springfield	43	89
0.1% (w) Polyethylene No. 2-124 from Springfield	44	90
0.1% (w) Santicizer 300 butyl unsatisfactory fatty acid dibutyl fumarate adduct	39	91

All materials reduced both ductility and penetration of the base asphalt (Appendix D).

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E. Effect of Blending Time and Temperature

During the evaluation of APS formulations, blending techniques were found to be critical. Improvement in ductility could be overlooked if blending time was too short or too long. If the time was too short complete solubility, which could not be ascertained visually, was not accomplished. If blending time was too long, the base asphalt deteriorated and physical properties changed sufficiently to cloud the additive effect. Fortunately a normal blending time consistent with a reasonable temperature was established for each of the reference asphalts with APS, i.e., 30 to 60 minutes at 270-290°F. No great deviation from these conditions is anticipated with other modifiers. Results of this evaluation are included in Appendix F.

F. Anti-weathering Properties

Deterioration accompanies exposure of asphalts to climatic conditions whether it be a paving or a roofing grade material. Exposing thin films of these materials alternately to ultra-violet light and the washing and eroding action of water spray in a weatherometer is used to accelerate evaluations of this type. Testing was confined to roofing asphalts since uniform films could not be maintained on the vertical aluminum panels with more fluid materials. Even with roofing asphalts it was necessary to reduce maximum exposure temperatures from 180°F to 140°F to keep the film from flowing.

Failure of a test panel is ascertained by passing a high voltage spark producing unit over the surface. If a minimum of three sparks occur in separate sections of this panel it is reported as failing. Number of exposure days to failure is the final rating criteria.

Under the test conditions, three of the four types of roofing asphalts have similar weathering characteristics. The fourth, from Venezuelan Crude, appears to have slightly less resistance to oxidation.

Table III

WEATHEROMETER RESULTS
ASTM D-529-39T MODIFIED

Inventory No.	Type	Company	Days to Failure Avg.
B-102	Smackover	Lion Oil Company	124
B-103	Mid-Continent	Allied Materials Corp.	122
B-105	Venezuelan	American Bitumuls Co.	116
B-107	California	Trumbull Asphalt Co.	126

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Both panel thickness and spray water temperature affect the results in this study. A thinner panel coating and cold water (greater thermal shock) give more severe evaluations.

Table IV

EFFECT OF COATING THICKNESS AND WATER TEMPERATURE
ON
WEATHEROMETER RESULTS
ASTM D-529-39T MODIFIED

Inv. No.	Base Asphalt	Panel Thickness	Days to Failure Water Temp., °F 51.5	65	71
B-107	California-Trumbull Asphalt Co.	0.05 in.	126	186	--
		0.025 in.	--	--	154

The ASTM procedure calls for the use of deionized and cooled spray water. A limited budget precluded their use since rough relative ratings between modifiers could be ascertained without this equipment.

The roofing asphalt from Californian Crude was choosen as the base for evaluating anti-weathering agents. Three materials possessing oxidation inhibiting properties in lubricating oils were found also to enhance anti-weathering properties in asphalts.

Table V

WEATHEROMETER RESULTS
ASTM D-529-39T MODIFIED

Base	Additive, % (w)	Days to Failure Avg. of Duplicates Water Temp., °F 51.5	65
Californian B-107	None	126	186
	1.1 Sant. 393, Lot Z-2834*	150	
	1.0 Sant. 393, Lot V-20*		190
	2.0 Sant. 393, Lot V-20		190
	5.0 Sant. 393, Lot V-20	70	
	2.0 Sant. 393, Lot H-3**		219
	5.0 Sant. 393, Lot H-3	67	
	1.0 N-3720 Dicyclohexylamine	143	
	1.0 N-6787 N-cyclohexyl o-phenylenediamine	143	

* Dry process used in preparation - ZnO added to DTP acid.
** Wet process used in preparation - DTP acid added to slurry of ZnO and water.

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Treatment with excess Santolube 393 has a detrimental effect in asphalts just as it does in lubricating oils. Additionally, these data indicate Santolube 393 prepared by the wet process gives a greater inhibiting effect than the dry process material.

All materials evaluated in the Weatherometer are listed in Appendix G.

G. Anti-staining Properties

Techniques for evaluating this property were not fully established and confidence in the work is doubtful. However, several products from our initial screening are worthy of additional study.

Results using the modified Pressure Method (ASTM 1328-54T) for testing anti-staining properties are given as the number of layers of standard cigarette paper which are stained after contact with the asphalt blend at 210°F. Air pressure at 36 psi is used to force the liquid products through the asphalt and into the paper.

Promising candidates gave the following results:

Table VI

STAIN INDEX
ASTM-1328-54T

	No. of Layers Showing Stain	
	Avg.	Range
SM Laminate Type Asphalt R-1427-MO	15	12-21
SM + 1.0% CP-13020 R.P. propylene Polymer B (Sun Oil Co.) and chlorine 1:1 mole ratio	24.5	24-25
SM + 1.0% Santolube 393	18.5	18-19
SM + 0.5% CP-2457 Dodecyldisulfide	18	-

A complete list of materials tested appears in Appendix H.

H. Loss on Heating

This test is used to give a general indication of the volatile content of the asphalt under the conditions of the test and does not necessarily show correlation with evaporation in service due to differences in temperature and film thickness.

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Of more importance is a comparison of the penetration of the residue from this test with the original penetration which shows the amount of hardening resulting from this evaporation. Any hardening is detrimental and samples are considered undesirable if their penetration falls below approximately 80% of the original value.

Only a few evaluations of this kind were made and reported in Appendix I.

EVALUATION PROCEDURES

Standard ASTM test procedures were used in all work reported herein. Mention of any deviations or techniques not clearly defined is noted below.

A. Ductility - ASTM D113-44

In place of the amalgamated brass plate for molds, this laboratory used a masonite board, 4" x 6", coated with a mixture of talc and glycerine. The molds were easily removable.

All tests were run at a temperature of 77°F and at a rate of 5 cms. per minute.

B. Consistency

1. Softening Point - ASTM E28-51T

The masonite board covered with the talc-glycerine mixture is again used in preparing the molds. No mechanical stirrer was employed.

2. Penetration - ASTM D5-52

All tests were conducted at 77°F using the standard 100 gram load for 5 seconds duration.

3. Saybolt Furol Viscosity - ASTM E102-54T

The standard procedure was followed except that temperature was maintained at 275°F.

C. Loss on Heating - ASTM 6-39T

No deviation was made from the method outlined.

D. Accelerated Weathering Test - ASTM D529-39T
Modified Equipment

Equipment - Atlas Type SMC
Model No. WO-1277

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Cycle -	Time	- 21 hours
	Off	- 3 hours
	Ultra-violet Light	- 51 Min. per hour
	Water Spray*	- 9 Min. per hour
	Panel Temperature	- 140°

* Tap water was used at the delivered temperature.

E. Anti-staining - ASTM 1328-54T

No deviations from outlined procedure.

ACKNOWLEDGEMENTS

We are indebted to Lion Oil Division for training in evaluation techniques and to all who submitted samples for evaluation.

D. W. Dinsmore

bt
8-29-61

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APPENDIX A

PATENT DISCLOSURE

AROCLOR AS AN ASPHALT MODIFIER

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PATENT DISCLOSURE

AROCLOR AS AN ASPHALT MODIFIER

Following a discussion with a representative of Ashland Oil Refining Company in which it was mentioned that there was a need for additives to reduce the viscosity of certain asphalts at high temperatures, I suggested that certain of our Aroclors might have the desired effect and at the same time have other favorable effects. The suggestion was made to our Research Department and to Dr. Houlton of Ashland.

Laboratory work has been done by our Research Department to show that Aroclor 1232 has an appreciable effect on the viscosity of asphalt, without significant effect on its ductility and softening point. The work was done by Mr. D. W. Dinsmore and is recorded in detail in his notebooks. Specific data relative to the effect of Aroclor 1232 on asphalt are:

Asphalt	Penetration 77°F	Ductility 77°F	Softening Point °F	Furol Vis. 275°F
Venezuelan 80-100 Paving	93.5	150+	118	223.0
Venezuelan + 10% (w/w) Aroclor 1232	108.0	136	116	194.0

I claim that Aroclor 1232 and other chlorinated diphenyls and chlorinated terphenyls may be incorporated into asphalt for the following purposes:

1. Reducing the viscosity of asphalt for easier handling in the refinery.

2. Improving the fluidity of asphalt for impregnating paper and cloth and at the same time reducing the flammability of the finished product.

3. Providing a formulation more suitable for a process in which finely divided soil particles are uniformly coated with asphalt by spraying hot asphalt into a mixing device. The resulting product is useful for paving and soil stabilization.

E. L. Will

4-27-56

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APPENDIX B

SANTODEX EVALUATION IN ASPHALTS AT LION OIL DIVISION

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SANTODEX EVALUATION IN ASPHALTS AT LION OIL DIVISION

	Melt °F	Furol Vis. at 275°F	Penetration 32°	Penetration 77°	Ductility at 77°, cm	Fluidity Factor
Century Samples (Development)						
#1 - Typical production base	128			79	42	
#2 - #1 + 5% Prod. Santodex* polymer (100%)	140			66	104	
#3 - Base better than #1	124			48	98+**	
#4 - #3 + 5% Santodex* polymer (100%) lab material	131			45	150+	
Century Base (Supplied by Lion)***	130.5	247		80	33	128
" + 0.05% Santodex****	135		25	66	22.5	
" + 0.10% Santodex***	133	262		76	28	140
" + 0.50% Santodex	134	322	26	67.5	81.5	171
" + 1.00% Santodex	150	589	21.5	51	15	274
" + 5.00% Santodex	158		25.5	50.5	39	
" + 1.00% Parapol S-50	137	338	20.5	64.5	25	176
" + 2.00% Santodex solution, 25% in 200 neutral oil	133			71.5	142	
" + 4.00% Santodex solution, 25% in 200 neutral oil	128.5			81	150+	
Lion Base	128.5	256		70.5	85	131
" + 1% Santodex solution, 25% in 200 neutral oil	127.5	236		72.5	150+	119
" + 4% Santodex solution, 25% in 200 neutral oil	122.5			91.5	150+	
" + 4% Parapol S-50, 25% in 200 neutral oil	126			88.5	150+	

* Santodex from Springfield
** Thread touched bottom without breaking
*** Blends analyzed during October - base may have changed to some extent
**** Santodex furnished by Texas City (100% material)

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APPENDIX C

SOLUBILITY AND DATA INDEX

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RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: COMPOUND SOLUBILITY AND DATA INDEX

ALCOHOLS						IDENT. NO.		SKELLY B SOLUBILITY % WT.		DATA INDEX APPENDIX NO.
RP CHLORINATED PROPYLENE POLYMER "B" + 2 METHANOL						CP-1302		>0.5		
2-TRIISOBUTENYLAMINO ETHANOL						CP-1303		>0.5		
2,2'[N (1-TERT. BUTYL 2 METHYLALLYL)] IMINO DIETHANOL						CP-1304		>0.5		
N (1-TERT BUTYL 2 METHYLALLYL AMINO) 2 PROPANOL						CP-1305		>0.5		
2 (TETRA PROPENYLAMINO) ETHANOL						CP-1306		>0.5		
1 (TETRAPROPENYL) 2 PROPANOL						CP-1306		>0.5		
2,2' (TETRA PROPENYL IMINO) DIETHANOL						CP-1307		>0.5		
HYDROABIETYL ALCOHOL, TECHNICAL						CP-1308		>0.5		
RP TETRAPROPENYL SUCCINIC ANHYDRIDE + SALICYLO PROPANOL						CP-1301		>0.5		

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RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: COMPOUND SOLUBILITY AND DATA INDEX

AMINES AND IMINO COMPOUNDS.	IDENT. NO.		SKELLY B SOLUBILITY % WT.		DATA INDEX APPENDIX NO.
AEROLUBE "MB" - TETRAMETHYL DIAMINO DIPHENYL METHANE			>0.5		
ANTIOXIDANT 4010 - N-CYCLOHEXYL N-PHENYL p PHENYLENE					
DIAMINE	CP-13555		0.1 TO 0.5		
N-CYCLOHEXYL N'-PHENYL N, N' DINITROSO					
p PHENYLENE DIAMINE	CP-11377		<0.1		
RP. OLEIC ACID AND TRIETHYLENE TETRAMINE	CP-6109		0.1 TO 0.5		
N-PHENYL O-PHENYLENE DIAMINE	CP-10479		0.1 TO 0.5		
N-(1 TERT BUTYL 2-METHYL ALLYL) ETHYLENE DIAMINE	CP-11666		>0.5		
N-ISOPROPYL 1-TERT BUTYL 2 METHYL ALLYL AMINE	CP-12025		>0.5		
1,7, DIDODECENYL DIETHYLENE TRIAMINE	CP12685		>0.5		
1(TRIISOBUTENYL) DIETHYLENE TRIAMINE	CP-12977		>0.5		
RP. CHL. PROPYLENE POLYMER "B" AND DIETHYLENE TRIAMINE	CP-13021		>0.5		
N-CYCLOHEXYL TRIISOBUTENYL AMINE	CP-13024		>0.5		
N-(1 TERT. BUTYL 2 METHYL ALLYL) TERT BUTYL AMINE	CP-13030		>0.5		

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RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: COMPOUND SOLUBILITY AND DATA INDEX

AMINES & IMINO COMPOUNDS (CONT.)	IDENT. NO.		SKELLY β SOLUBILITY %		DATA INDEX APPENDIX NO.
N-TETRAPROPENYL ETHYLENEDIAMINE	CP-13045		>0.5		
N-TERT BUTYL TRIISOBUTENYLAMINE	CP-12082		>0.5		
RP. TETRAPROPENYL SUCCINIC ANHYDRIDE + DIOMEENT	CP-13300		>0.5		
RP. " " " " + DIETHYLENETRIAMINE	CP-13331		>0.5		
N-PHENYL 2 CYCLOHEXEN-1-YLAMINE	CP-17805		>0.5		
PHENYL 2-NAPHTHYLAMINE	N-653		0.1-0.5		
DIPHENYLAMINE - ACETONE CONDENSATION (30% FREE AMINE)	N-1634	18	<0.1		
DICYCLOHEXYLAMINE	N-3720		>0.5		
N,N' DISEC. BUTYL p PHENYLENEDIAMINE	N-4321		>0.5		
N-CYCLOHEXYL O-PHENYLENEDIAMINE	CP-10110		>0.5		
N(1 TERT BUTYL ETHYLALLIN) 1,1,3,3 TETRAMETHYL BUTYLAMINE	CP-13533		>0.5		
3 (TERT. BUTYLAMINO) PROPYLAMINE	—		>0.5		
3 (ISODECYLAMINO) PROPYLAMINE	-		>0.5		
RESIDUE FROM REDUCTIVE AMINATION OF ISODECYL ALCOHOL	P-988		>0.5		
AMINE DISTILLATION RESIDUE	P-1197		<0.1		

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RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: COMPOUND SOLUBILITY AND DATA INDEX

	IDENT. NO.	SKELLY B SOLUBILITY % wt.	DATA INDEX Appendix No
BENZOTHIAZOLES			
THIOFIDE - 2,2' DITHIO BIS BENZOTHIAZOLE	CP-1612	<0.1	
THIOTAX - 2 MERCAPTOBENZOTHIAZOLE	CP-1975	<0.1	
N-CYCLOHEXYL 2 BENZOTHIAZOLE SULFENAMIDE	CP-318	<0.1	
2 VINYL MERCAPTOBENZOTHIAZOLE	CP-4530	<0.1	
2-N-CYCLOHEXYLAMINO BENZOTHIAZOLE	CP-13[illegible]	>0.5	
6 DODECENYL 2-MERCAPTOBENZOTHIAZOLE	CP-13815	>0.5	
CARBAMATES			
N-2CYCLOHEXYLAMINO N-CYCLOHEXYL DITHIOCARBAMIC ACID	CP-550	<0.1	
R.P. N-CYCLOHEXYLAMINE + P_2S_5	CP-17[illegible]	<0.1	
BIS (DIMETHYL THIOCARBAMYL) SULFIDE	CP-2113	<0.1	
2 CHLOROALLYL DIETHYL DITHIOCARBAMATE	CP-4712	>0.5	
2 CHLOROALLYL N(2 CHLOROALLYL) CYCLOHEXYL DITHIOCARBAMATE	CP-7547	>0.5	
2 CHLOROALLYL N(2 CARBAMOYLETHYL) CYCLOHEXYL DITHIOCARBAMATE	CP-8307	<0.1	
BIS (DIMETHYL THIOCARBAMYL) DISULFIDE	N-8072	<0.1	

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RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: COMPOUND SOLUBILITY AND DATA INDEX.

	IDENT. NO.		SHELLY B SOLUBILITY % WT.		DATA INDEX APPENDIX NO
CHLORINATED MATERIALS					
AROCLOR 1232 - CHLORINATED BIPHENYL	—		>0.5		
MONTAR #4 - RESIDUE FROM AROCHLOR 1260	—		>0.5		
RP. PROPYLENE POLYMER "B" (SUN) AND CHLORINE	CP-13020		>0.5		
RP. TRIISOBUTYLENE AND CHLORINE	CP-13023		>0.5		
CHLORINATED KEROSENE (60%)	—		>0.5		
CRESOLS AND PHENOLS					
SANTOWHITE POWDER - 4,4' BUTYLIDENE BIS (6 TERT. BUTYL M-CRESOL)	CP-3888		0.1 TO 0.5		
ANTIOXIDANT 700 ETHYL CORP. 4,4' METHYLENE BIS (2,6 DITERT BUTYL PHENOL)	CP-13150		>0.5		
CYCLOHEXYLAMINO PENTACHLOROPHENATE	CP-167		<0.1		
DIISOBUTYL α PIPERIDINO P-CRESOL	CP-12713		>0.5		
α,α' (BUTYLIMINO) BIS (2,6 DITERT BUTYL P-CRESOL)	CP-13151		>0.5		
α,α',α'',α''' (HEXAMETHYLENE DINITRO) TETRA BIS (2,6 DI TERT BUTYL P-CRESOL)	CP-13229		>0.5		

DSW 621893

STLCOPCB4095924

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: COMPOUND SOLUBILITY AND DATA INDEX

						IDENT. NO.		SKELLY C SOLUBILITY %		DATA INDEX APPENDIX No.
CRESOLS + PHENOLS (CONT)										
2,6 DITERT BUTYL 4 NITROSO PHENOL						CP-13[illegible]		< 0.1		
$\alpha^2, \alpha^4, \alpha^6$ TRIS (DIETHYLAMINO) MESITOL						CP-14[illegible]		> 0.5		
α DIMETHYLAMINO O-CRESOL						CP-14290		< 0.1		
4,4' THIO BIS (6-TERT BUTYL M-CRESOL)						N-5[illegible]		0.1 TO 0.5		
ESTERS										
SANTOCIZER 300 - BUTYL UNSAT FATTY ACID DIBUTYL FUMARATE ADDUCT.								> 0.5		
PARAPLEX G-62 EPOXIDIZED SAFFLOWER OIL								0.1 TO 0.5		
PLASTOLEIN 9720 POLYESTER ADIPIC ACID, 1,2 PROPANEDIOL, FATTY ACID								< 0.1		
PX-800 PITTSBURG COKE - EPOXIDIZED VEGETABLE OIL								< 0.1		
DIHYDROABIETYL MALATE						CP-13[illegible]5		> 0.5		
ETHYL N-NITROSO N-PHENYL β ALAMINATE						CP-13779		> 0.5		
DIBUTYL 8,12 EICOSADIENEDIOATE - DIBUTYL FUMARATE						CP-14[illegible]2		> 0.5		
COPOLYMER 2ETHYL HEXYL (20%) + ETHYL (30%) ACRYLATES						N-52[illegible]		> 0.5		

DSW 621894

STLCOPCB4095925

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: COMPOUND SOLUBILITY AND DATA INDEX.

						IDENT. NO.		SOLUBILITY % wt.		DATA-INDEX SUMMARY NO
GUANIDINES										
GUANTAL -	DIPHENYLGUANIDINE PHTHALATE					CP-309		<0.1		
DIPHENYL GUANIDINE						N-3011		<0.1		
MORPHOLINES										
SULFOSAN R -	4,4' DITHIO MORPHOLINE					CP-307		ALMOST		
N-(1 TERT BUTYL 2 METHYL ALLYL)	MORPHOLINE					CP-1307		>0.5		
PHOSPHORAMIDATES										
O-ETHYL TETRAMETHYL	PHOSPHORODIAMIDOTHIOATE					CP-1015		>0.5		
2 ETHYL HEXYL TETRAMETHYL	PHOSPHORODIAMIDATE					CP-1330		>0.5		
N-NITROPHENYL	TETRAPROPYL PHOSPHORODIAMIDATE					CP-1378-A		>0.5		
DIISOPROPYL	DIPROPYL PHOSPHOROAMIDATE					CP-1378		>0.5		
HEXAMETHYL	PHOSPHOROAMIDE					N-1469		>0.5		

DSW 621895

STLCOPCB4095926

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: COMPOUND SOLUBILITY AND DATA INDEX

						IDENT. No.		SOLUBILITY % wt.		DATA INDEX APPENDIX No.
PHOSPHOROTHIONATES										
SANTOLUBE 393 - Di (1,3 Dimethyl Butyl) Phosphorodithioate								>0.5		
O,O DIETHYL PHOSPHORODITHIOATE						P-2490		>0.5		
CYCLOHEXYL AMINE SALT		DI 1,3 DIMETHYL BUTYL								
	PHOSPHORODITHIOIC ACID					P-2457		>0.5		
TRIETHYL THIOL PHOSPHATE						P-2457		>0.5		
POLYMERIC MATERIALS										
ALKYLATED POLYSTYRENE								>0.5		
COPOLYMER 2 ETHYL-HEXYL (20%) ETHYL (80%) Acrylate						P-5202		>0.5		
QUINOLINES										
SANTOFLEX A.W. 6-ETHOXY 1,2 DIHYDRO 2,2,4 TRIMETHYL						CP-4020		<0.1		
6-DODECYL 1,2 DIHYDRO 2,2,4 TRIMETHYL QUINOLINE					QUINOLINE POLYMER	CP-4057		0.1-0.5		
6 DODECYL 1,2 DIHYDRO 2,2,4 TRIMETHYL QUINOLINE						P-4020		>0.5		

DSW 621896

STLCOPCB4095927

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: COMPOUND SOLUBILITY AND DATA INDEX

						IDENT. NO.		SOLUBILITY %		DATA INDEX APPENDIX NO.
UREAS AND THIOUREAS										
EL-SIXTY - 1,3 BE (2 BENZOTHIAZOLYL THIOMETHYL) UREA						CP-[illegible]		<0.1		
1,3 DIBUTYL THIOUREA						CP-4101		<0.1		
ALLYL UREA						CP-4679		<0.1		
1,3 DIBENZYL 2 THIO UREA						CP-[illegible]		<0.1		
1 CYCLOHEXYL, 1-2 HYDROXY PROPYL, 3 PHENYL THIOUREA						CP-7115		<0.1		
1,3 DIALLYL THIOUREA						CP-7511		<0.1		
1-1 NAPHTHYL 3 OCTADECYL UREA						CP-7510		<0.1		
1 OCTADECYL 3 PHENYL UREA						CP-7601		<0.1		
1 CYCLOHEXYL 1-2 HYDROXY PROPYL 3 OCTADECYL UREA						CP-7604		>0.5		
MISCELANEOUS										
SANTOVAR - A - 2,5 DITECT AMYL HYDROQUINONE						CP-2181		0.1005		
SANTOLUBE 3940 - RP P2S5 AND α PINENE								>0.5		
ST-35 - SULFURIZED OLEFIN								>0.5		

DSW 621897

STLCOPCB4095928

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: COMPOUND SOLUBILITY AND DATA INDEX

	IDENT. No.		SKELLY P SOLUBILITY % WT.		DATA INDEX APPENDIX No
MISCELLANEOUS (CONT)					
M-372 MIXTURE SANT. 394 AND [illegible]			>0.5		
BLE - A DIPHENYLAMINE - ACETONE CONDENSATE	[illegible]		<0.1		
PHENOTHIAZINE	M-351		[illegible]		
DIPHENYL DISULFIDE	CP-[illegible]		<0.1		
DIBENZYL DITHIOFORMATE	CP-[illegible]		>0.5		
S-DICYCLOHEXYL THIURAM DISULFIDE	CP-[illegible]		<0.1		
DIAMETHYL BENZYL DISULFIDE	CP-[illegible]		>0.5		
DODECYL DISULFIDE	CP-[illegible]		>0.5		
1,4 DIHYDRO 4,4,6 TRIMETHYL 1 (p-PHENETYL)					
2 PYRIMIDINETHIOL	CP-[illegible]		<0.1		
1,4 DIHYDRO 4,4,6 TRIMETHYL 1 (p-NITROPHENYL)					
2 PYRIMIDINETHIOL	CP-[illegible]		<0.1		
ALLYL BENZYL SULFIDE	CP-[illegible]		>0.5		
X CYANO BIPHENYL	CP-[illegible]		>0.5		

DSW 621898

STLCOPCB4095929

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: COMPOUND SOLUBILITY AND DATA INDEX

MISCELLANEOUS (CONT.)						IDENT. No.		SKELLY B SOLUBILITY %		DATA INDEX APPENDIX No.
3,5' DI TERT BUTYL 4' HYDROXY ACETOPHENONE						CP-12621		[illegible]		
α FURYL N-PHENYL NITRONE						CP-12983		<0.1		
1(O-CHLORO PHENYL) 1,4 DIHYDRO 4,4,6 TRIMETHYL										
2 PYRIDIDINE THIOL						CP-[illegible]		<0.1		
N-(X DIDECYL PHENYL) 2,4,5 TRICHLORO PENZANE										
SULFENAMIDE						—		<0.1		
ALUMINA						CP-[illegible]		<0.1		
BARIUM FERRATE III						CP-[illegible]		<0.1		
TITANIUM IV OXIDE						CP-[illegible]		<0.1		
γ IRON III OXIDE						CP-[illegible]		<0.1		
α IRON III OXIDE						CP-[illegible]		<0.1		

DSW 621899

STLCOPCB4095930

APPENDIX D

EVALUATION OF DUCTILITY IMPROVERS

STLCOPCB4095931

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: EVALUATION OF DUCTILITY IMPROVERS.

	Century Refining Co			Base STOCK No.	Blending Time Hrs.	Blending Temp. °F	Ductility Avg Cm.	Penetration Avg 1/10 mm.	Softening Point Avg °F		
				B-100			0	6.5			
	Straight Run			B-109			79	102	125		
C-1	0.02% (w) A-70777			B-109			82	98.5			
C-2	0.5% (w) "			B-109			148	93.5			
C-3	1.5% (w) "			B-109				87			
C-4			0.25% 99.75%	B-100 B-109			77.5	98			
C-5	0.25% (w) N-5262			B-109			68	99			
C-6	0.5% (w) A-70778			B-109			71	112			
C-7	0.5% (w) N-1775 Sulfosan R			B-109			79	104		Insol.	
C-8	0.5% (w) N-603 Thiofide			B-109			23.5	89		Insol.	
C-9	0.5% (w) N-2415 Guantal			B-109			34	96.5		Insol.	
C-10	0.25% (w) CP-2425 El Sixty			B-109				96.5		Insol	
C-12	0.1% (w) A-70784			B-109			57.5	101.5			
C-13	0.1% (w) A-70777			B-109			103	100			

DSW 621901

STLCOPCB4095932

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: EVALUATION OF DUCTILITY IMPROVERS.

				Base Stock No.	Blending Time hr.	Blending Temp °F	Ductility Avg. Cm.	Penetration Avg 1/10 mm	Softening Point Avg. °F	Weather-Ometer Avg. Hours To Failure	
	Century Refining Co										
		Recd	4/12/57	B-110			40	90.5	126		
C-14	0.1% (2) A-70777			B-110			56	87	-		
C-14(2)	" "			B-710			56.5	86.5			
C-14(3)	" "			B-710	1 3/4	365°	36	81.5 86	130°		
C-14(4)	" " "			B-710	2	225°	49	88			
C-14(5)	" "			B-710	2 2 TR	200° 250°	37	83	127.5		
C-14(6)	" "			B-710	1/4	255°	38	88	128		
C-14(7)	" "			B-110	1/2	230-260	50	89	124		
C-14(8)	" "			B-110	3/4	250-270°	54	89	125		
C-14(9)	" "			B-110							
C-14(10)	" "			B-710	1	235	53				
C-14(11)	" "			B-710	3/4	235	46				
C-14(12)	" "			B-110	1 1/4	235	39				
C-14(13)	" "			B-110	1 1/4	260 270	35				

DSW 621902

STLCOPCB4095933

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: EVALUATION OF DUCTILITY IMPROVERS

	Century Refining Co			Base stock No.	Blending Hrs. Time	Blending Temp °F	Ductility Avg. cm.	Penetration Avg. Vacuum	Softening Point Avg. °F	Weather-Ometer Avg. Days to Failure	
C-15	0.1% Polyethylene No. 2-187			B-109			43	89	126		
C-16	0.1% " " No 2-124			B-109			44	90	126		
C-17											
C-18	0.1% + Santocel-C 300			B-110			39	91	127		
C-19	0.1% APS PPB-#1			B-110			43	89	125.5		
C-19a	" " "			B-110	3/4	260	39				
C-20	0.2% APS PPB-#1			B-110			53	85.5	127		
C-21	0.1% A-70800			B-110			46	87.5			
C-22	0.05% A-83003			B-110	3/4	260-270	44				
C-22a	" "			B-110	3/4	260	43				
C-23	0.05% A-83001			B-110			27				
C-24	0.12% A-83002A			B-110						Insoluble	
C-25	0.1% A-83008			B-110			33				
C-26	0.1% A-83007			B-110			27				

DSW 621903

STLCOPCB4095934

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: EVALUATION OF DUCTILITY IMPROVERS

	Century Refining Co			Base Stock No.	Blending Time (hr.)	Blending Temp °F	Ductility Avg. Cm.	Penetration Avg. 1/10 mm.	Softening Point Avg. °F	Weather-ometer Avg. Days to Failure	
C-27	—			B-110	3/4	260-270	28				
C-28	—			B-110	Melted	only	30.5				
C-29	—			B-110	"	"	17			Not Valid	
C-29(2)	—			B-110	"	"	30.5				
C-30	0.1% (w) A-70777			B-110	1/2	260	32				
C-31	" "			B-110	1/2	215-235	34.5				
C-32	" "			B-110	3/4	235	45				
C-33	" "			B-110	1/4	260	42.5				
C-33(2)	" "			B-110	"	"	41				
C-34	—			B-110	1/4	260	30.5				
C-34(2)	—			B-110	"	"	29				
C-34(3)	—			B-110	"	"	24				
C-35	0.1% (w) A-70777			B-110	1/4	260	34				
C-36	" "			B-110	3/4	260	40.5				600 gr. Blend.
C-37	0.1% (w) PPA # 1			B-110	1/4	260	30.5				

DSW 621904

STLCOPCB4095935

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: EVALUATION OF DUCTILITY IMPROVERS

	Century Refining Co.			Base Stock No.	Blending Time Hrs.	Blending Temp. °F	Ductility Avg. Cm.	Ductility Avg. 1/100 cm.	Softening Point Avg. °F	Weather Ometer Avg. Hrs. to Failure	
C-38	0.1% (a) A-83009			B-110	1/4	260°	18.5				
C-39	" "			B-110	3/4	260	27.5				Loop Blend
C-40	—			B-110	3/4	260	27.5	88			
C-41	0.1% (a) A-83034-A			B-110	1/2	260	30				Loop Blend
C-42	—			B-110	1/2	260	23.5				Loop Blend
C-43	0.1% (a) A-70777			B-110	1/2	260	33				Loop Blend

DSW 621905

STLCOPCB4095936

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: EVALUATION of DUCTILITY IMPROVERS

	Lion Oil Co.			Base Stock No.	Blending Time Hrs	Blending Temp °F	Ductility Avg. Cm.	Penetration Avg. 1/10 mm	Softening Point Avg. °F	Weather-Ometer Avg. Days to Failure	Water Temp Avg. °F
	Paving Grade			B-101			150+	90.5	113		
	Roofing Grade			B-102			3.1	24.5	159	124	51.5
L-1			40% 60%	B-101 B-102	1/2	300-320	14	29.5	153		
L-2			65% 35%	B-101 B-102	1/2	300-320	116	54	133		
L-3			55% 45%	B-101 B-102	1/2	300-320	47	45	137		
L-4			57% 43%	B-101 B-102	1/2	300-320	72	46	135		
L-5	0.1% (w) A-83042			L-4	1/2	270-290	150+	47	135		
L-5(2)	" "			L-4	3/4	270-290	150+	48	135		
L-5(3)	" "			L-4	1	270-290	150+	49	135.5		

DSW 621906

STLCOPCB4095937

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: EVALUATION OF DUCTILITY IMPROVERS

	Allied Materials Corp.			Base Stock No.	Blending Time Hrs	Blending Temp °F	Ductility Avg. Cm	Penetration Avg 1/10 mm.	Softening Point Avg. °F	Weather-Ometer Avg. Days to Failure	Weather-Ometer Avg. water Temp °F
	Mid-Continent Grade										
	Roofing Grade			B-103			27	23.5	203	122	51.5
	Paving Grade			B-104			131	92	118		
A-1			50% 50%	B-103 B-104	–	270	14				
A-2				A-1	1	325	13				
A-3			20% 80%	B-103 B-104	–	270	135				
A-4				A-3	1	270-284	132				
A-5			35% 65%	B-103 B-104	–	290	67	51	136		
A-6	0.25% (a) A-70777			A-5	3/4	270-290	150+	49			
A-7	0.1% (a) "			A-5	3/4	280-290	147				
A-8	0.1% (a) A-83009			A-5	3/4	280-290	65				
A-9	0.1% (a) A-83008			A-5	3/4	280-290	71				
A-10	0.1% (a) A-83003			A-5	3/4	280-290	149				

DSW 621907

STLCOPCB4095938

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: EVALUATION OF DUCTILITY IMPROVERS

				Base Stock No.	Blending Time Hrs	Blending Temp °F	Cooking Adj cm	Penetration Adj 1/10 mm	Softening Point Adj °F		
A-11	0.1% A-83009			A-5			51				
A-12	2.0% APS PPB-1			A-5			91				
A-13	0.1% A-83025			A-5			104	4/4			
A-14	0.1% A-83025			A-5			74				
A-15	0.1% A-83029 A			A-5			65				
A-16	0.1% A-83029 B			A-5			79				
A-17	0.1% A-83034 A			A-5			150+				
A-18	0.1% A-83034 B			A-5			148				
A-19	-		4/02 A03	B-103 B-104	1/2	270-290	38				
A-20	0.1% A-83034 A			A-79			56				
A-21	0.1% A-83036			A-5			150+				
A-22	0.1% APS BPB-1			A-79	3/4	270-290	62.5				
A-23	0.1% A-83003			A-79	3/4	270-290	82				
A-24	0.1% A-83034 A			A-79	3/4	270-290	56				

DSW 621908

STLCOPCB4095939

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: EVALUATION OF DUCTILITY IMPROVERS

				Base Stock No.	Blending Time Hrs.	Blending Temp. °F	Ductility Avg Cm.	Evaporation Avg 1/100 gm.	Softening Point Avg °F		
A-5(2)			35% / 65%	B-103 / A-104			88				
A-5(3)			37½ / 62½	B-103 / B-104			43	43	138		
A-27	0.1% A-83035 A			A-5(2)	¾	270-290	150+				
A-28	0.1% A-83035 B			A-5	¾	270-290	139				
A-28(2)	" "			A-5(2)	½	270-290	150+				
A-28(3)	" "			A-5(3)	½	270-290	82				
A-30	0.1% A-83042			A-5(3)	½	270-290	68				
A-30(2)	" "			A-5(3)	¾	270-290	150+	56	142.5		
A-30(3)	" "			A-5(3)	½	270-290	150	48	140		
A-30(4)	" "			A-5(3)	1	270-290	70	46	137		
A-31	0.1% A-83040			A-5(3)	½	270-290	82.5				
A-31(2)	" "			A-5(3)	½	270-290	150+	45			
A-32	0.1% A-93966			A-5(3)	½	270-290	137	53	137		

DSW 621909

STLCOPCB4095940

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: EVALUATION OF DUCTILITY IMPROVERS

				Base Stock No	Blending Time Hrs	Blending Temp. °F	Ductility Avg Cm.	Penetration Avg 1/10 mm.	Softening Point Avg. °F		
A-33			80% 20%	B-103 B-104	1/2	270-290		29			
A-34			70% 30%	B-103 B-104	1/2	270-290		33			
A-35			90% 10%	B-103 B-104	1/2	270-290		27			
A-36			75% 25%	B-103 B-104	1/2	270-290	45	31.5	176.5		
A-5[illegible]			37% 63%	B-103 B-104	3/4	270-290	31	42.5	134		
A-37	0.1% A-7-177			A-5(4)	3/4	270-290	23	47.5			
A-38	0.05% [illegible] A-7-177			A-5(4)	3/4	270-290	22	46			

DSW 621910

STLCOPCB4095941

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: EVALUATION OF DUCTILITY IMPROVERS

	American Bitumuls & Asphalt Co.			Base Stock No.	Blending Time Hrs	Blending Temp °F	Ductility Avg Cm.	Penetration Avg 1/mm	Softening Point Avg °F	Weather-Ag Days to Failure	Weather-Ag Water Temp °F
	Venezuelan Crude										
	Paving Grade			B-105			4.3	19	199	116	51.5
	Paving Grade			B-106			150+	93.5	118		
AB-1	0.1% Amoco 1232			B-106			136		116		
AB-2			35% 65%	B-105 B-106	1/2	270-290	108				
AB-3			45% 55%	B-105 B-106			62				
AB-4	0.1% A-93942			AB-5	1/2	270-290	98	43.7	139.5		
AB-4(a)	" "			AB-5	3/4	270-290	124	45	140		
AB-4(b)	" "			AB-5	1	270-290	147	45	142		
AB-4(c)	" "			AB-5	1/2	300-320	71	44	140.5		
AB-4(d)	" "			AB-5	3/4	300-320	102	40.3	140.5		
AB-4(e)	" "			AB-5	1	300-320	150+	41.5	140.5		
AB-5	—		40% 60%	B-105 B-106	1	270-290	51	49	141		
AB-6	0.1% A-93966			AB-5	3/4	270-290	96	49.7	141		

DSW 621911

STLCOPCB4095942

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: EVALUATION of DUCTILITY IMPROVERS

	Socony Mobil Oil Co.			Base Stock No.	Blending Time Hrs	Blending Temp. °F	Ductility Avg Cm.	Penetration Avg 1/10 mm.	Softening Point Avg. °F.		
?											
?											
SM-1				B-111	-	300-330	3.1	31	188.5		
SM-2	0.25% A-70777			B-111	1/4	300-330	3.4				

DSW 621912

STLCOPCB4095943

APPENDIX E

ALKYLATED POLYSTYRENE

EFFECT OF THERMAL AGEING ON DUCTILITY

STLCOPCB4095944

ALKYLATED POLYSTYRENE

EFFECT OF THERMAL AGEING ON DUCTILITY

Sample No.	Base Asphalt	Additive	Softening Point °F	Penetration at 32	Penetration at 77	Penetration at 115	Ductility at 77°F
1	Roofing A	---	143	29	60	133	10
2	Roofing A	5.0% Vistonex LM-MS	169	35	51	128	3.1
3	Roofing A	1.0% Vistonex LM-MS	150	24	50	106	7.0
4	Roofing A	1.0% Vistonex LM-MH	162	27	48	105	5.5
5	Roofing A	5.0% Thiokol LP-2	144	28	55	105	9.2
6	Roofing A	5.0% Thiokol LP-3	144	--	--	--	--
7	Roofing B	---	182	16	26	46	3.4
8	Paving	---	114	27	93	>285	150+
9	43% 7 + 57% 8	---	136	21	47	162	41
10	43% 7 + 57% 8	1.0% Vistonex LM-MS	144	17	46	143	12

DSW 621914

STLCOPCB4095945

Sample No.	Base Asphalt	Additive	Softening Point °F	Penetration at 32	Penetration at 77	Penetration at 115	Ductility at 77°F
11*	43% 7 + 57% 8	0.1% APS	137	21	49	168	81
12*	43% 7 + 57% 8	0.5% APS	140	20	46	163	90
13	Sample 9 after 24 hrs. at 250°F		138	20	47	160	45
14	Sample 11 after 24 hrs. at 250°F		137	20	50	162	59
15	Sample 12 after 24 hrs. at 250°F		139	19	46	160	87

* Alkylated Polystyrene (APS) was a blend of 50% APS-50% asphalt. Therefore, actual APS concentrations in Samples 11 and 12 were 0.05% and 0.25%, respectively.

DSW 621915

STLCOPCB4095946

APPENDIX F

EVALUATION OF APS AS AN ASPHALT MODIFIER

Project AP-134-57

DSW 621916

STLCOPCB4095947

MONSANTO CHEMICAL COMPANY

Research Engine Laboratory

PROJECT AP-134-57

EVALUATION OF ALKYLATED POLYSTYRENE AS AN ASPHALT MODIFIER

Work done by:

W. M. Rinehart
D. W. Dinsmore

Prepared by:

D. W. Dinsmore
5-22-57

DSW 621917

STLCOPCB4095948

Distribution:

E. P. Cunningham - TTT Building
L. E. Klein - Roberts Building
M. Kosmin - Dayton
J. H. Lum - Research
J. W. Newcombe - Roberts Building
W. W. Paris - Nitro
T. M. Patrick - Research
T. P. Sands - Roberts Building
M. C. Throdahl - Roberts Building
F. B. Zienty - Research

DSW 621918

STLCOPCB4095949

EVALUATION OF ALKYLATED POLYSTYRENE AS AN ASPHALT MODIFIER

Table of Contents

I. Summary

- A. Scope
- B. Conclusions
- C. Recommendations
- D. Application

II. Details Of Investigation

- A. Background
- B. Experimental Details and Procedures

III. Recommendations For Future Work

IV. Appendix

DSW 621919

STLCOPCB4095950

I. Summary

A. Scope

Upon receiving indications from the industry that alkylated polystyrene (APS) acted as a ductility improver in asphaltic materials, an evaluation program to determine the scope of this response was begun. The response was obtained in asphalts produced from each of the four major crude sources, i.e. Mid-Continent, Californian, Venezuelan and Smackover crudes. Although the ductility response was of primary importance, response toward penetration, softening point, blending conditions and weathering properties was also evaluated.

B. Conclusions

The following conclusions can be drawn from this work:

1. Satisfactory ductility improvement with low dosages of APS (0.1% wt.) can be obtained in each of the major types of asphalt.

2. The penetration and softening point of the asphalts are not significantly altered by incorporating small dosages of APS.

3. The weathering properties of the asphalt remain unchanged with the addition of 2% APS.

4. Asphalts from Venezuelan crude are more susceptible to blending conditions than those from other sources.

C. Recommendations

Based on the above conclusions, the following recommendations are made:

1. APS should be released to the Development Department for promotion to the asphalt industry as a ductility improver.

2. Pilot Plant quantities of APS in asphalt (50% solution) should be prepared for sampling the industry.

D. Application

Although APS has not been evaluated for all possible uses, it may have application in several types of asphaltic materials. These types may include paving grade, roofing grade, canal linings and laminate type asphalts. Additional uses may be discovered upon promotion to the industry.

STLCOPCB4095951

II. Details of Investigation

A. Background

APS has been produced and sold by Monsanto under the trade name of Santodex. This material functioned as a viscosity index improver for lubricating and gear oils.

The production of Santodex at the Springfield plant was discontinued several years ago. Since it was necessary to re-develop the manufacturing process and study available raw materials before embanking on an evaluation program, it was decided to replace the diluent oil at the same time. This was thought necessary since the presence of oil in asphaltic formulations is often detrimental to the oxidation resistance of the asphalt. The decision to use a brittle asphalt as diluent seemed logical since the mixture could be powdered, if desired, for easy handling and solution in the final product would be facilitated. Production of quality APS was necessary before the response of this material could be defined. This program was carried out by Dr. R. R. Raube under the direction of Dr. E. J. Young, with this laboratory assisting in evaluating the finished product.

An indication that APS acted as a ductility improver was obtained from a small producer in Western Kansas who found his production material failed to meet paving grade specification. Several materials gave satisfactory ductility improvement, with APS being the most economical and most advantageous from other standpoints. It was necessary for us to determine whether this material would function similarly in asphalts of other crude sources and refining processes before commercialization could be considered.

As expected, it was found that the ductility response of APS was dependent, in part, upon the ductility of the base asphalt. Thus, a paving grade asphalt which has a high ductility (above 100 cm) will need no improvement nor would it indicate maximum response. On the other hand, a roofing asphalt which is normally air blown to a high penetration and low ductility (2 to 6 cm) will not give pronounced improvement. By using a mixture of these two types of asphalts in ratios to give a base with intermediate ductility (70-80 cm), improvement can readily shown.

During this work it was found that the blending time and temperature play an important part in determining the response of APS in certain asphalts. It may also ensue that these same conditions will hold true for other chemical modifiers as well..

STLCOPCB4095952

The effect of APS upon properties other than ductility, which we were able to evaluate in this laboratory, are also indicated.

B. Experimental Details and Procedures

A summary of results in tabular and graphic form are included in the appendix. All procedures used in this evaluation are contained in "ASTM Standards on Bituminous Materials For Highway Construction, Waterproofing and Roofing".

III. Recommendations for Future Work

It is recommended that specific field problems encountered by the Development Department be studied in the laboratory to increase our knowledge of the application of APS.

STLCOPCB4095953

RESPONSE OF APS VS ASPHALT CRUDE SOURCE

Asphalt	Additive	Blending			Evaluations		
		Spl. Size Grams	Time Min.	Temp. °F	Ductility CM	Penetration 1/10 MM	Softening Point °F
Venezuelan Crude							
Roofing Grade B-105					4.3	19	199
Paving Grade B-106					150 +	93	118
40% Roofing, 60% Paving	None	400	60	270-290°	81	48	141
40% Roofing, 60% Paving	0.1% (w) APS(50%)*	400	30	270-290	98	44	139
40% Roofing, 60% Paving	0.1% (w) APS(50%)	400	45	270-290	124	45	140
40% Roofing, 60% Paving	0.1% (w) APS(50%)	400	60	270-290	147	45	142
40% Roofing, 60% Paving	0.1% (w) APS(50%)	400	30	300-320°	71	44	140
40% Roofing, 60% Paving	0.1% (w) APS(50%)	400	45	300-320	102	40	140
40% Roofing, 60% Paving	0.1% (w) APS(50%)	400	60	300-320	150 +	41.5	140
Mid-Continent Crude							
Roofing Grade B-103					2.7	23.5	203
Paving Grade B-104					131	92	118
37% Roofing, 63% Paving	None	400	30	270-290°	77	57	135
37% Roofing, 63% Paving	0.1% (w) APS(50%)	400	30	270-290	150	48	140
37% Roofing, 63% Paving	0.1% (w) APS(50%)	400	45	270-290	150 +	56	142
37% Roofing, 63% Paving	0.1% (w) APS(50%)	400	60	270-290	150 +	53	139
Californian Crude							
Roofing Grade B-107					3.9	22.5	193
Paving Grade B-108					150 +	93	113
50% Roofing, 50% Paving	None	400	30	270-290°	74	46	137
50% Roofing, 50% Paving	0.1% (w) APS(50%)	400	30	270-290	150 +	44	137
50% Roofing, 50% Paving	0.1% (w) APS(50%)	400	45	270-290	150 +	43.5	137
50% Roofing, 50% Paving	0.1% (w) APS(50%)	400	60	270-290	150 +	44	136

DSW 621923

STLCOPCB4095954

RESPONSE OF APS VS ASPHALT CRUDE SOURCE

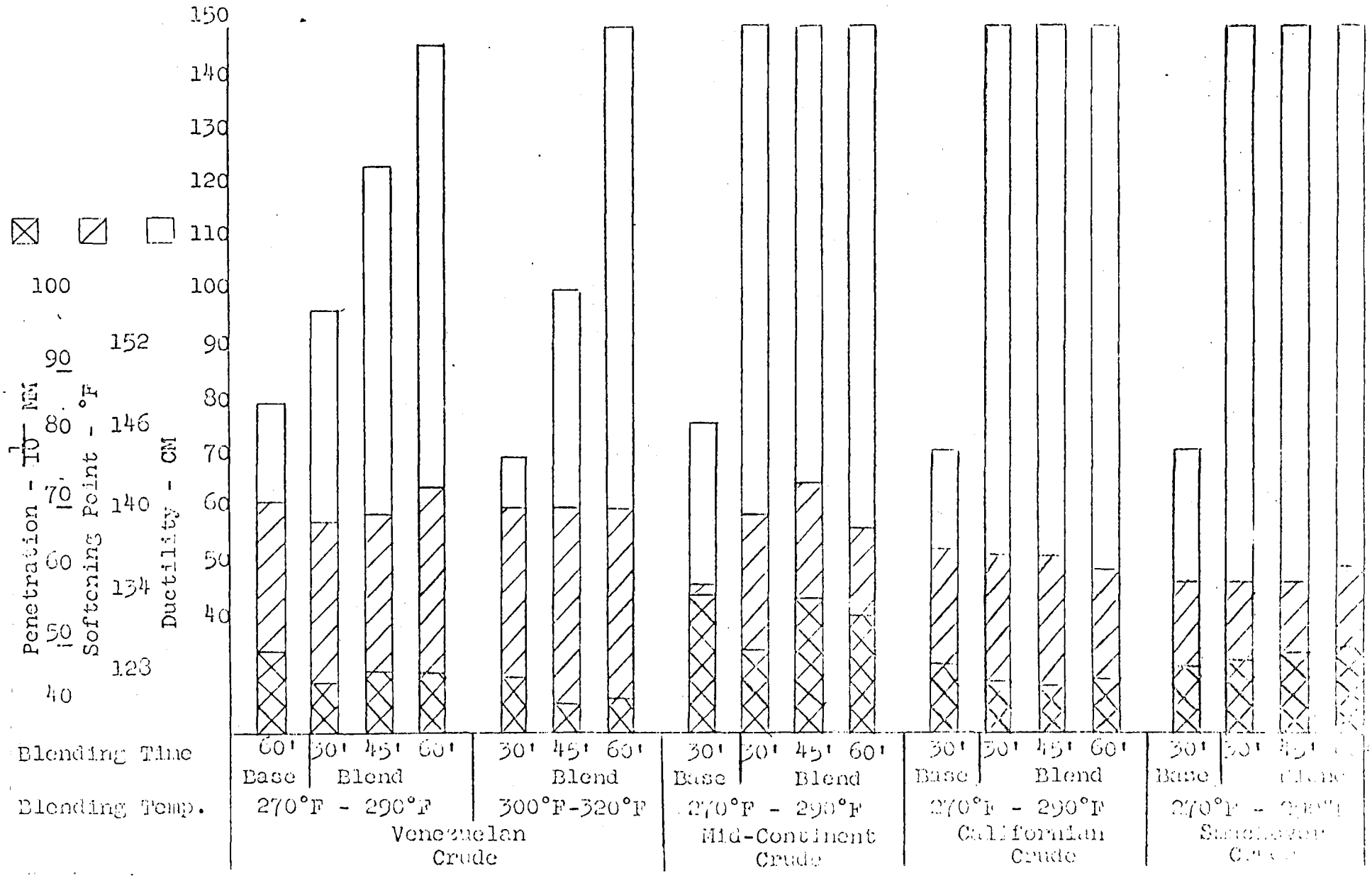
Asphalt	Additive	Blending			Evaluations		
		Spl. Size Grams	Time Min.	Temp. °F	Ductility CM	Penetration 1/10 MM	Softening Point °F
Smackover Crude							
Roofing Grade B-102					3.1	24.5	189
Paving Grade B-101					150 +	90.5	113
43% Roofing, 57% Paving	None	400	30	270-290°	72	46	135
43% Roofing, 57% Paving	0.1% (w) APS(50%)	400	30	270-290	150 +	47	135
43% Roofing, 57% Paving	0.1% (w) APS(50%)	400	45	270-290	150 +	48	135
43% Roofing, 57% Paving	0.1% (w) APS(50%)	400	60	270-290	150 +	49	136

* APS (50%) = A-83042

DSW 621924

STLCOPCB4095955

RESPONSE OF APS VS ASPHALT CRUDE SOURCE
Additive = 0.1% (w) APS (50%) in Asphalt (A-83042)



DSW 621925

STLCOPCB4095956

WEATHER-O-METER RESULTS

21 Hour Cycle Spray In Tap Water
At Delivered Temperatures For
9 Minutes Per Hour

	Days to Failure Average
Californian Crude Base	126
" + 2% (w) APS (50%)	125

DSW 621926

STLCOPCB4095957

APPENDIX G

EVALUATION OF ANTI-WEATHERING PROPERTIES

DSW 621927

STLCOPCB4095958

APPENDIX G

EVALUATION OF ANTI-WEATHERING PROPERTIES

STLCOPCB4095959

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: EVALUATION OF WEATHER ABILITY.

	Turnball Asphalt Co.			Base Stock No.	Blending Time Hrs	Blending Temp. °F	Ductility Avg. Cms.	Penetration Ave 1/10 mm.	Softening Point Ave. °F	Weather-Ometer Avg. Days To Failure	Water Temp Avg. °F	
	California Crude											
	Roofing Grade			B-107			3.9	22.5	193	126	51.5	
	Paving Grade			B-108			150+	93	113			
				B-107						186	65	
				B-107						154	71	0.025 in panels
T-1	0.5% (w) Aant 394-C			B-107						125	51.5	
T-2	1.13% (w) Aant 393 Z-2834	≡ 1.0% Pure Quill		B-107						150	51.5	
T-3	1.0% (w) Tenamene 2	N,N' di.sec. butyl p-phenylene diamine		B-707						115	51.5	
T-4	1.0% (w) N-4669	Hexamethyl phosphoramide		B-107						113	51.5	
T-5	1.0% (w) N-6060C	Xdodecyl 1,2 dihydro 2,2,4 Trimethyl quinoline		B-707						125	51.5	
T-6	1.0% (w) CP-13027	N(1 t-butyl/2me Allyl) morpholine		B-707						125	51.5	
T-7	1.0% (w) CP-4742	2Chl. Allyl diethyl dithiocarbamate		B-707						124	51.5	Insol.
T-8	1.0% (w) CP-7549 (2)	2Chl. Allyl N-2Chl. Allyl cyclohexyl dithiocarbamate		B-707						122	51.5	Insol.
T-9	1.0% (w) Monitor #4	Residue from Aroclor 1260		B-107						125	51.5	

DSW 621929

STLCOPCB4095960

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: EVALUATION OF WEATHERABILITY.

				Base Stock No.	Blending Time Hrs.	Blending Temp. °F	Ductility Avg. Cm.	Penetration Avg. 1/10 mm.	Softening Point Avg. °F	Weather-Ometer Avg. Days to Failure	Water Temp Avg °F	
T-10	1.0%(wt) CP-13815 6dodecenyl-2-mercaptobenzothiazole			B-107						125	51.5	
T-11	0.2%(wt) N-5215 Antioxidant 4010 (N-Cyclohexyl-N'-phenyl-p-phenylenediamine)			B-107						124	57.5	Insol.
T-12	2.0%(wt) A-70777 Alkylated polystyrene			B-107						125	51.5	
T-13	1.0%(wt) N-3720 Dicyclohexylamine			B-107						143	50	
T-14	1.0%(wt) N-6787 N-Cyclohexyl-p-phenylenediamine			B-107						143	50	
T-15	1.0%(wt) N-8057 1,7 didodecenyl diethylene Triamine			B-107						124	57.5	
T-16	1.0%(wt) P-988	Residue from reductive amination of Isodecyl aldehyde		B-107						123	57.5	
T-17	—		35% / 65%	B-107 / B-108	1/2	270-290°	150					
T-18	—		60% / 40%	B-107 / B-108	1/2	270-290°	17.5					
T-19	1.0%(wt) Santolube 393 Lot V-20			B-107						190	65	
T-20	2.0%(wt) " " "			B-107						190	68	
T-21	5.0%(wt) " " "			B-107						70	57.5	
T-22	2.0%(wt) " " Lot A-3			B-107						219	59	

DSW 621930

STLCOPCB4095961

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: EVALUATION OF WEATHERABILITY

				Base Stock No.	Blending Time Hrs.	Blending Temp. °F	Ductility Avg. Cm	Penetration Avg. 1/10 mm.	Softening Point Avg. °F	Weather-ometer Avg. Days to Failure	Weather-ometer Avg. water Temp °F
T-23	5.0%(w) Santolube 393 + LotN-3			B-107						67	57.5
T-24	2.0%(w) ST-35 Sulfurized	Olefin.		B-107						189	68
T-25	1.0%(w) 3(t-butylamino)	propyl Amine		B-107	1/2	300-320	3			199	67
T-26	1.0%(w) 3(isodecylamino)	propyl Amine		B-107	1/2	300 320	3.4			210	67
T-27	1.0%(w) N-8391			B-107						191	68
T-28	1.0%(w) N-8088 Dihydroabeityl Malate			B-107						200	67
T-29	1.0%(w) CP-7857 Allyl benzyl	Sulfide		B-107						185	67
T-30	1.4%(w) N-6672 Cd dihexylphosphorodithioate (70%)			B-107						195	67
T-31	1.4%(w) N-6673 Sn(ous) dihexylphosphorodithioate (70%)			B-107						172	68
T-32	1.4%(w) N-6675 Sb dihexylphosphorodithioate (70%)			B-107						190	67
T-33	2.0%(w) Santolube 394-C RP P2S5+α	pinene		B-107						186	68
T-34	2.0%(w) M-428 (75% Sant 393 + 25% Sant 394-C)			B-107						189	67
T-35	2.0%(w) M-428	"	"	B-107						191	67
T-36											

DSW 621931

STLCOPCB4095962

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: EVALUATION OF WEATHERABILITY

				Base Stock No.	Blending Time Hrs	Temp °F	Ductility Avg Cm.	Penetration Adj. 1/10 mm.	Softening Point Avg. °F	Weather-ometer Avg Days To Failure	Water Temp. °F	
T-37	1.0%(w) Calco MB			B-107						191	67	
T-38	1.0%(w) A-79357 Cyclohexylamine salt of dihexyl-phosphorodithioic Acid			B-107						186	67	
T-39	—		50%(w) 50%(w)	B-107 B-108	1/2	270-290	74	46.3	137.5			
T-40	0.1%(w) A-83042			T-39	1/2	270-290	150+	43.8	137			
T-40(a)	" "			T-39	3/4	270-290	150+	43.5	137			
T-40(a)	" "			T-39	1	270-290	150+	44	136			
T-41	1.0%(w) N-3720 dicyclohexylamine			B-107						155	71	0.025 in. Panel
T-42	1.0%(w) N-6786 N-Cyclohexyl-o-phenylenediamine			B-107						155	71	0.025 in. Panel
T-43	1.13%(w) Santoflube 393 Lot H-3			B-107						153	71	0.025 in. Panel

DSW 621932

STLCOPCB4095963

APPENDIX H

EVALUATION OF ANTI-STAINING PROPERTIES

STLCOPCB4095964

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: ANTI-STAINING EVALUATIONS

SOCONY LAMINATE (R-1427-MD)						BLENDING TIME MIN.	BLENDING TEMP °F		RATING NO. STAINED	RATING AVG.
Sm-1	ABOVE BASE D.V. B-111					0	290°		17, 21, 15, 12 13, 14	15
Sm-5	1%(w) CP-7604	1-CYCLOHEXYL	1-2 HYDROXYPROPYL	3-OCTADECYL UREA		30	290°		14, 14	14
Sm-6	2%(w) APS (50% Sol'n) ≡ 1%(w) PURE APS					30	290°		13, 15	14
Sm-7	1%(w) CP-1303	2-N CYCLOHEXYL AMINO	BENZOTHIAZOLE			30	290°		13, 16, 19	16
Sm-8	1%(w) WITCO	LITHium	HYDROXY STEARATE			30	290	Insol.	13, 18, 9, 15	14
Sm-8(2)	2% WITCO	"	"	"		30	290	Insol.	18, 19	18.5
Sm-9	1%(w) CP-1304G	1(TETRAPROPENYL)	2 PROPANOL			30	290°		17, 21	19
Sm-10	1% N-3720	DICYCLOHEXYL AMINE				30	290°		16, 15, 16	16
Sm-11	1% CP-10110	N-CYCLOHEXYL O-PHENYLENE DIAMINE				30	290°		13, 15, 16	15
Sm-12	1% CP-13020	RP PROPYLENE	POLYMER "B" (Sun)	& CHLORINE		30	290°		24, 25	24.5
Sm-13	1% N-526-2	COPOLYMER	2ETHYL HEXYL (70%)	& ETHYL (30%)	ACRYLATE	30	290°		14, 14	14
Sm-14	1% SANTOLUBE 393	(100% MATERIAL)				30	290°		18, 19	18.5

DSW 621934

STLCOPCB4095965

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose: ANTI-STAINING EVALUATIONS.

						BLEEDING Time MIN.	BLEEDING TEMP. °F.		RATING No. STAINED	RATING AVG.
SOCONY LAMINATE (R-1427-MO)										
SM-16	1% CP-12713	DIISOBUTYL	& PIPERIDINO	p-CRESOL		30	290		15, 18	17
SM-17	1% CP-2457	DODECYL	DISULFIDE			30	290		15, 18	18
SM-18	1% CP-13027	N(1-TERTBUTYL 2METHYL ALLYL)	MORPHOLINE			30	290		15, 18	17
ALLIED MATERIALS CORP - (MID-CONTINENT CRUDE)										
A-36	75% ROOFING GRADE									
	25% PAVING GRADE					30	290		10, 12	11

DSW 621935

STLCOPCB4095966

APPENDIX I

PHYSICAL PROPERTY LOSS ON HEATING

STLCOPCB4095967

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No: _____ Date _____

Purpose:

Loss on Heating

			Base Stock No		Weight Loss %		Penetration Loss % of Original				
	Century "off-grade" Paving		B-109		0.02		84.8				
C-1	0.02% A-70777		B-109		0		92.4				
C-2	0.5% A-70777		B-109		0		89.8				
C-3	1.5% "		B-109		—		94.3				
C-4		0.25% 99.75%	B-100 B-109		0		84.7				
C-5	0 0.25% N-5262		B-109		0		92.9				
C-6	0.5% A-70178		B-109		0		81.3				
C-12	0.1% A-70784		B-109		0		92				
C-13	0.1% A-70777		B-109		0.02		94.5				

DSW 621937

STLCOPCB4095968

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No: _____ Date _____

Purpose:

Loss on Heating

		Base Stock No.		Weight Loss %		Penetration Loss % of Original				
Paving Grade										
Lion - Smackover		B-101		0		88.4				
		B-101		0		86.7				
Allied Materials - Mid Continent		B-104		0		87.5				
		B-104		0		80.4				
American Bitumuls - Venezuelan		B-106		0.2		82.9				
		B-106		0.09		80.7				
Trumbull - California		B-108		0.2		81.2				
		B-108		0.08		78.5				
Miscellaneous										
Century - "off-grade" Paving		B-109		0.02		84.8				
Century - "off-grade" paving		B-100		0		92.3				

DSW 621938

STLCOPCB4095969

RESEARCH ENGINE LABORATORY

Project: Date Initiated: Summary No. _____ Date _____

Purpose:

Loss on Heating

		Base stock No.		Weight Loss %		Penetration Loss % of Original				
Roofing Grade										
Lion - Smackover		B-102		0.2		100%				
		B-102		0		93.4				
Allied Materials - Mid Continent		B-103		0		95.7				
		B-103		0		95.7				
American Bitumuls - Venezuelan		B-105		0		100				
		B-105		0.08		94.7				
Trumbull - Californian		B-107		0.4		100				
		B-107		0.5		91.1				

DSW 621939

STLCOPCB4095970

DSW 621940