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FINAL REPORT

ON

APPLICATION WORK ON PRESENT PLASTICIZERS, 1955

PART II

- -

Job No. 2-02-750.01-2896-II

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I. INTRODUCTION

When a Plasticizer Application Laboratory, such as ours, is responsible for the company-wide testing of exploratory and commercial organic compounds as potential plasticizers, a well defined screening system must be employed. The first phase of this operation is the preliminary evaluation, to which every compound is initially subjected. Upon occasion, promising compounds emerge from this preliminary screening and are then submitted to the second phase known as the intensive evaluation. In the intensive evaluation stage these compounds are given a more thorough examination for properties required in specific applications such as plastisols, vinyl foam, electricals, etc. Generally when the applications are large ones the work is separated into specific classifications with individual job numbers and appropriations. However, a large amount of the intensive evaluation is of a general nature and does not fit well into any of the specific application categories. This report comprises a compilation of the general application work completed during 1953 and is included as Part II on Final Report No. 1311 under Job No. 2-02-750.01-2896-I. (Ref. No. 1)

From time to time, the Sales Department has been given this application data to furnish them with valuable technical information in their customer contacts.

II. SUMMARY

Because of the diversified nature of the contents of this report, it is divided conveniently into four main classifications:

1. Application Work on Primary Plasticizers
2. Application Work on Secondary Plasticizers
3. Evaluation of New Process Plasticizers
4. Intensive Evaluation of Santicizer 2[illegible]3

The work on primary plasticizers includes an evaluation of the physical properties of a large number of the [illegible] plasticizers comprising the Santicizer 600 series. In some cases, a specific member of this series is compared directly to its closest competitor in the trade. Also included in this category is the work involved in finding a replacement for DNODP /di(n-octyl, n-decyl) phthalate/ which became scarce due to a restricted alcohol supply. Other studies on primary plasticizers reported herein are: the comparison of DIDA to its competitor Morflex P-20; an intensive evaluation of Flexol 77G (dipropylene glycol dibenzoate) - competitor for Santicizer 160; permanence properties of a number of plasticizers; and a fundamental study of the low temperature characteristics of plasticizers.

Although much of the work on secondary plasticizers was reported in an earlier report (Ref. 4), additional information was obtained in 1955. This includes a comparison of the Aroclors to competitive chlorinated paraffins, light stability work on HB-20, and an evaluation of Santicizer 212.

Application information pertinent to our plant processes are also included for the charcoal treatment of DOP at Everett and the laboratory distillation and topping of Santicizer 160.

Additional application information on Santicizer 213 and summary of its properties complete the final section of this report.

The original work on floor tile evaluation was included under this job number, however, the importance of the work warranted its separation under a separate job number (Ref. 10).

III. CONCLUSIONS

1. Santicizer 603 (DIDP/DBP 80:20) is equivalent to Flexol 426 (Carbide's butyl octyl phthalate) in all properties except light stability where Flexol 426 is superior. These two products are direct competitors in the trade.

2. Santicizer 615 (DIDP/DIDA 3:1) is the only mixed plasticizer found which compares in plasticization properties to DNODP. However, this mixture and all DIDP-DIDA combinations have lower compatibility limits than DNODP. This relative incompatibility poses a problem in its usage and indicates that caution must be used during formulation.

3. A mixture comprising 37.5% DIDP, 12.5% DIOP, 50% DIDA may be used as a replacement for Santicizer 601 (DNODP/DIOP 1:1) in applications where S-601 was recommended. When using this mixture most properties are equivalent, however a slight sacrifice in low temperature flexibility, water resistance and heat stability must be expected.

4. Ohio-Apex plasticizer RS-1313 (probably a mixture of tricresyl phosphate with a fatty acid ester like butyl oleate) is definitely less compatible than Santicizer 141. Moreover, the competitive mixture is decidedly less light stable than S-141. This discovery has definitely stopped the early competition encountered with RS-1313 in flame resistant formulations.

5. Morflex P-20 (Morton Withers' unidentified low temperature plasticizer) is completely equivalent in properties to DIDP. It is, in fact, slightly superior in low temperature flexibility.

6. Flexol 77-G fuses readily, its rate of fusion is somewhat lower than tricresyl phosphate and Santicizer 160. It does, however, fuse more rapidly than DOP or DIDP. The water extraction of Flexol 77-G is higher than Santicizer 160 but it is not as sensitive to soapy water extraction. It is inferior to DIDP in both aforementioned extraction properties.

7. From kerosene extraction results over a range of temperatures it was shown conclusively that DIDP is inferior to to DOP in this respect.

8. In the study of secondary plasticizers it was shown that, although the Aroclors are more compatible and flame retardant than the competitive chlorinated paraffins, they are nevertheless more water and soapy water sensitive. They (Aroclors) also suffer, to some extent, from poorer volatility and vapor toxicity.

9. The use of UV #9 (2-hydroxy-4-methoxy benzophenone) with Santowhite powder has produced the best light stability yet encountered in film containing appreciable amounts of HB-20. Also capable of imparting light resistance to HB-20 are mixtures of UV #9 and phenyl salicylate with Santowhite powder.

10. Santicizer 212 (Santicizer 300 distillate) exhibited interesting plasticization properties as a secondary plasticizer but showed poor compatibility-stability by the window exposure test. This product is contingent upon the success of Santicizer 300 which has not been encouraging so application effort has been terminated.

11. The new charcoal purified DOP from our Everett plant is completely equivalent to the original DOP production in plasticization properties.

12. The distillation of Santicizer 160 produces no apparent effect upon its plasticizing properties.

13. Topped (most volatile fraction removed) Santicizer 160 has an improved volatility from film as well as per se.

14. Santicizer 213, a blend of the exceptionally compatible ester Santicizer 160, and a less compatible material, alkyl benzene, is a primary plasticizer for polyvinyl chloride. Since its introduction to the trade in 1956, it has been received with interest. Its ability to impart surface "slip" to coated fabric, molded articles and transparent boats has been responsible for the enthusiasm shown for this product.

5. In order to distinguish more clearly the differences in volatility of monomeric and polymeric plasticizers, a more stringent volatility test was written. This new test involves a higher temperature and longer aging period than the standard A.S.T.M. activated carbon test.

IV. FUTURE WORK

1. To continue the intensive evaluation of potential exploratory plasticizers as well as competitive products.

2. To continue the study of secondary plasticizers.

3. To construct and compare modulus of elasticity vs. temperature curves of Monsanto and competitive plasticizers.

4. To study dodecyl benzene in admixture with other primary plasticizers such as DOP, DIDP and Santicizer 141.

V. PATENT STATUS

No disclosures have been submitted to the Patent Department as a result of this work.

VI. REFERENCES

1. L. R. Wangler, Application Work on Present Plasticizers, Special Application Report No. 1, A Study of Santicizer 213 in Polyvinyl Chloride. Report No. 1425 under Job No. 2-02-750.01-2896-1, February 22, 1956.

2. J. A. Cannon, Application Work on Present Plasticizers. Under Job No. 2-02-750.01-2616 (to be issued).

3. P. R. Graham, Plasticizer Test Method Development, Part I; A Study of the Clash-Berg Torsion Test. Report No. 1311 under Job No. 2-02-750.01-3044-I, September 27, 1955.

4. R. M. Parks, Screening Test on Monsanto and Competitive Plasticizers-1954; The Inteisive Evaluation of Secondary Plasticizers, Including the Aroclors. Final Report No. 1281 under Job No. 2-02-750.01-2321-I, May 16, 1955.

5. P. R. Graham, Special Plasticizer Application Report # 1, Evaluation of HB-20 as a substitute for HB-40. Report No. 779 under Job No. 117-2595, September 23, 1953.

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6. P. R. Graham, Special Plasticizer Application Report No. 2 on The Evaluation of HB-20 as a Substitute for HB-40. Report No. 1005 under Job No. 117-2616-II, July 26, 1954.

7. P. R. Graham, Final Report on the Evaluation of Stabilizers for Polyvinyl Chloride. Report No. 1504 under Job No. 2-02-750.01-2946, May 4, 1956.

8. L. R. Wangler, Screening Tests on Monsanto and Competitive Plasticizers-Special Application Report No. 1, Evaluation of Santicizer 300 Distillate as a Secondary Plasticizer for Polyvinyl Chloride. Report No. 1312 under Job No. 2-02-750.01-2894-I, October 17, 1955.

9. A. Y. Coran, Special Application Report No. 2 on Plasticizer Sales Development Research. Report No. 1340 under Job No. 2-02-750.01-2892, November 21, 1955.

10. Final Report under Job No. 2-02-750.01-3083, to be written.

VII. TEST METHODS

The test procedures employed in this study are contained in "Standard Test Methods of the Plasticizer Application Laboratory". They are in most cases adopted from standard A.S.T.M. procedure.

VIII. DETAILS

A. Application of Primary Plasticizers

1. Preliminary Evaluation of the Santicizer 600 Series (Ref. 9)

The members of the Santicizer 600 series are identified as follows:

Santicizer No.			
601	DnODP, DIO[illegible]	50:50	
602	DIOP, DIDP	50:50	
603	DIDP, DBP	80:20	
604	DnODP, 141	67:33	Discontinued
605	DOP, 141	67:33	Discontinued
606	DIDP, 160	67:33	
607	DOP, DOA	75:25	
608	S-601, 160	52.8:47.2	
609	DIDP, DOA, 1[illegible]	50:25:25	
610	DIDP, DBP, DIDA	60:15:25	
611	DIDP, 160	50:50	
61[illegible]	DOP, DOA	59.5:40.5	
61[illegible]	DIDP, 16[illegible]	33:67	
614	DIDA, DIDP	50:50	
61[illegible]	DIDP, DIDA	75:25	

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The results of those plasticizers which were evaluated will be found on Chart I in the Appendix.

It should be noted that Santicizers 607, 612 and 614 are quite flexibilizing. Santicizer 606 is inefficient and hence its formulations would require less resin to produce a composition of a certain hardness. Santicizer 614 has good permanence with respect to volatility. Santicizer 611 and especially Santicizer 613 exhibit superior resistance to kerosene extraction. In water sensitivity, most of these plasticizers are comparable. Santicizers 607, 612 and 614 exhibited preliminary indications of yielding formulations of good heat stability.

2. Comparison of Santicizer 603 to Flexol 426

Formulations containing Flexol 426 and those containing Santicizer 603 at concentrations of plasticizer of 20%, 30%, 40% and 50% were prepared.

At all concentrations, Flexol 426 showed a slightly better flexibilizing effect. There was little or no difference between the volatilities. From the Shore hardness results, it can be noted that Flexol 426 is slightly more efficient as a plasticizer. At lower concentrations of plasticizer, the kerosene extractions do not differ significantly. However, at 40% and 50% plasticizer, Flexol 426 is somewhat superior in this respect. There is very little difference between these plasticizers in respect to processing characteristics and water sensitivity.

In stability to heat, Flexol 426 is superior; however, the difference is not as well defined in the case of stability to light. In either case (stability to heat or stability to light), the differences were more apparent in the poorer stabilizing system than in the epoxy type system.

3. Comparative Evaluation of Santicizer 615 and DNODP

In an effort to achieve an efficient replacement for DNODP /di-(n-octyl, n-decyl) phthalate/ a series of combinations of DIDP (diisodecyl phthalate) and DIDA (diisodecyl adipate) were extensively tested in direct comparison with DNODP.

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Formulations

Series A:

	Opalon 410	Plasticizer A	Plasticizer B	Plasticizer C	Plasticizer D	G-62	Mark XI	Mark XX
1	100	64				3	2	1
2	"		22			"	"	"
3	"		37			"	"	"
4	"		64			"	"	"
5	"		97			"	"	"
6	"			22		"	"	"
7	"			37		"	"	"
8	"			64		"	"	"
9	"			97		"	"	"
10	"				22	"	"	"
11	"				37	"	"	"
12	"				64	"	"	"
13	"				97	"	"	"

Series B:

	Opalon 410	Plasticizer A	Plasticizer B	Plasticizer C	Plasticizer D	G-62	Mark XI	Mark XX	Bisphenol A
1	100	64				3	2	1	0.1
2	"	64				"	"	"	0.5
3	"		64			"	"	"	0.1
4	"		64			"	"	"	0.5
5	"			64		"	"	"	0.1
6	"			64		"	"	"	0.5
7	"				64	"	"	"	0.1
8	"				64	"	"	"	0.5
9	"		64 (+ 3 PHR POS-C; 0.75 PHR DS-207)						0.5

NOTE: Plasticizer A = DNODP
Plasticizer B = 25% DIDP; 75% DIDA
Plasticizer C = 50% DIDP; 50% DIDA
Plasticizer D = 75% DIDP; 25% DIDA (Santicizer 615)

a. Compatibility Test Results

As predicted, the most serious drawback to the use of DIDA/DIDP combinations in place of DNODP is the limited compatibility of the isodecyl esters. Loop compatibility tests, window exposures, compatibility in H_2O at 50°C., polished plate and Fadeometer compatibility lists collectively show the DIDA/DIDP combinations to be less compatible than DNODP. Various plasticizer concentrations (tabulated with a series of systems) displayed several degrees of incompatibility. It was found that compatibility is most seriously impaired when the lead-type stabilization system is used or if Bisphenol A is added. The combination of 75% DIDP-25% DIDA (Santicizer 615) was shown to be the most compatible of the series. Compatibility decreased proportionally with the increasing concentration of DIDA.

b. Fadeometer Light Stability

As in the previous study, DIDP/DIDA mixtures are less stable to light than DNODP. The best stability was obtained from the Santicizer 615 mixture. Poorer stability and more pronounced tack formation were evident in the mixtures containing the high concentrations of DIDA. Bisphenol A is detrimental to the light stability of Santicizer 615.

Fadeometer Exposure Results

No.	(800 Hours Exposure)	No.	(300 Hrs. Exposure)
1-A	No change	1-B	No change
2-A	Spotted	2-B	Sl. tackiness
3-A	Spotted, sl. exud.	3-B	Sl. spotting, exud.
4-A	Exuded	4-B	Noticeable exud.
5-A	Spotted, serious exud.	5-B	Sl. exudation
6-A	Very spotted	6-B	Noticeable exud.
7-A	Sl. spotting	7-B	Sl. tacky
8-A	Sl. exudation	8-B	Sl. tacky
9-A	Exuded	9-B	Spotted, serious exud.
10-A	Sl. exudation		
11-A	Very spotted		
12-A	Sl. spotted		
13-A	Very sl. exud.		

c.. Plasticizing Properties

The evaluation of the physical properties (volatility, T_f°C., etc.) of S-615 and DNODP will be found on Charts II, III and IV in the Appendix.

4. The Replacement of Santicizer 601 with a DIDP/DIDA/DIOP Mixture

Because of the dwindling supply of di(n-octyl,n-decyl) phthalate (DNODP), which is one of the components of Santicizer 601, it was necessary to find a replacement for Santicizer 601. In most respects, a mixture of DIDP, DIDA and DIOP, 37.5:12.5:50 is very similar to Santicizer 601. Though the new mixture exhibits less stability to heat in its formulation, the stability to light appears to be comparable.

Formulations containing Santicizer 601 (DNODP and DIOP 50:50) and those containing a mixture of DIDP, DIDA and DIOP, 37.5:12.5:50 (hereafter referred to as the "new mixture" in this report) at plasticizer concentrations of 20%, 30%, 40% and 50% were prepared.

At all concentrations, the new mixture was not quite as flexibilizing as S-601. Shore hardness showed no significant difference with respect to plasticizer efficiency. There was little or no difference noted in volatility at any of the concentrations tested. There was no difference in the kerosene extractions at most concentrations. The new mixture yielded formulations slightly more sensitive to water. However, it is doubtful that these differences are significant since the variations with respect to this property were slight at any given concentration of plasticizer.

Santicizer 601 was superior in stability to heat in both the lead and the "epoxy-cadmium" stabilizer systems. There has been little or no difference noted in respect to light stability in the lead system. After 600 hours in the fadeometer no difference in light stability was noted in the "epoxy-cadmium" system. Neither of these plasticizers showed satisfactory stability to light in the lead systems.

For more specific details see Charts V, VI and VII in the Appendix.

5. Volatility of Butyl Decyl Phthalate vs. Competitive Products

An extensive series of volatility tests were run on several laboratory prepared BDP (butyl decyl phthalate) samples in comparison with Flexol 426 and a number of other competitive materials. The results of this investigation follow:

Material	Volatile Loss*, % Run No. 1	Volatile Loss*, % Run No. 2
BDP (comp. A-65820 & 65825)	[illegible]	10.6
BDP (A-65831)	11.4	11.9
BDP (A-65836)	10.9	10.7
BDP (A-65837)	11.6	12.2
BDP (A-70253, distilled)	11.0	12.9
Flexol 426	9.9	11.4
PX 114	11.9	14.0
Elastex 4C-P	16.9	20.4
Butyl isodecyl phthalate (BIDP)	[illegible]	16.3
Santicizer 603	12.2	13.7
DOP	4.3	5.7
Santicizer 160	10.1	11.5

*Test specimens for runs 1 and 2 were taken from the same milled sheets.

6. Comparison of Santicizer 141 to Ohio-Apex RS-1313

The bulk of the work on this project is reported in the final report under Job No. 2-02-750.01-2616 (Ref. 2). However, additional work included in the scope of this report has shown RS-1313 to be decidedly inferior to S-141 from the standpoint of compatibility. After six weeks behind glass, RS-1313 developed serious incompatibility. It was also shown to be inferior to S-141 in light resistance.

7. Comparison of DIDA to Morton Withers' Morflex P-20

Morton Withers, in offering Morflex P-20 (unidentified) as a replacement for DIDA, have produced a product which is equivalent to Monsanto DIDA in all properties except low temperature flexibility, where it possesses a slight advantage over DIDA.

	40% P-20	40% DIDA	33-1/3% P-20	33-1/3% DIDA
T_f °C.	-69.0	-65.3	-53.1	-49.4
Volatility, % Plast. Lost	4.5	4.7		
Shore Hardness	73	77		
H_2O Extraction, % Plast. Lost	0.07	0.07		
H_2O Adsorption	0.64	0.55		
Kerosene Extraction, % Plast. Lost	76.7	75.5		

8. Intensive Evaluation of Flexol 77-G

Because of increased competition from Carbide and Carbon's plasticizer 77-G (dipropylene glycol dibenzoate) additional information has been obtained on its performance. From the standpoint of processing, Flexol 77-G fuses readily; its rate of fusion being somewhat lower than tricresyl phosphate and Santicizer 160. It does, however, fuse more readily than either DOP or DIDP.

The water extraction of Flexol 77-G is higher than Santicizer 160 but it is not as sensitive to soapy water extraction. It is inferior to DIDP in both environments.

The following are the water and soapy water extraction results of 40 mil samples of Flexol 77-G as compared to Santicizer 160, DIDP and Santicizer 606:

	Flexol 77-G	S-160	DIDP	S-606
Water Extraction, % Plasticizer Lost	0.39	0.24	0.02	0.09
Extraction by 1% Ivory Flakes, % Plasticizer Lost	2.46	4.37	0.19	3.03

9. A Study of the Kerosene Extraction of DOP and DIDP Formulations

A comparison of kerosene extraction properties of DOP and DIDA at various temperatures and concentrations was undertaken in order to clarify the kerosene sensitivity of these two plasticizers. See Chart No. IV.

Results:	5°C.	23°C.	40°C.	50°C.
33-1/3% DOP	11.2%	15.6%	19.3%	20.8%
33-1/3% DIDP	76.6	72.6	24.4	29.6
40% DOP	60.6	76.2	41.1	34.4
40% DIDP	73.7	75.7	63.5	44.4

NOTE: 96-hour immersion time

10. Extended Volatility Tests for both Monomeric and Polymeric Plasticizers

In order to distinguish more clearly the differences in volatility of monomeric and polymeric plasticizers, a more stringent activated carbon volatility test was designed; 33-1/3% formulations of di-isodecyl phthalate, DOP, tricresyl phosphate, Paraplex G-50, and Santicizer 300 were tested.

Most of the usual conditions for the carbon volatility test were fulfilled, except the term of heat aging was extended to 4 days and 6 days and a higher temperature was used.

	% Plasticizer Loss		
Plasticizer	1-Day Test, 87°C.	6-Day Test, 87°C.	4-Day Test, 105°C
DOP	4.5	[illegible]	46.4
TCP	1.0	[illegible]	24.[illegible]
DIDP	1.6	[illegible]	18.2
G-50	1.3	[illegible]	[illegible]
S-300	1.1	[illegible]	[illegible]

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11. Low Temperature Properties of Plasticizers

Much of the work involved in this study will be found in Final Report No. 1313 under Job No. 2-02-750.01-3044-1 (Ref. 3).

Since the appearance of the above report work was started on the new Tinius Olsen torsion tester. This instrument has been found to give accurate and reproducible results for the stiffness of plasticized PVC over a wide temperature range.

The following results were obtained for T_f and T_4 of DOP (50 phr) using the new Tinius Olsen tester.

T_4 (Temp. at which Modulus of Elasticity is 10^4 psi) - 6°C.

T_f (Temp. at which Modulus of Elasticity is 1.35×10^5 psi) -22°C.

These results are identical to those published by Carbide and Carbon Chemicals Corporation.

B. Application of Secondary Plasticizers

1. Intensive Comparison of Aroclors to Competitive Chlorinated Paraffins

This study is a continuation of the work previously reported in Final Report No. 1281 under Job No. 2-02-750.01-2821-I, May 16, 1955 (Ref. # 4).

Monsanto's participation in the secondary plasticizer market could be improved if a niche for the Aroclors (chlorinated biphenyls) could be found. An intensive study of the Aroclors vs. the chlorinated paraffins, their natural competitors, was therefore undertaken. Recent tests indicate that the Aroclors were more flame retardant and more extractable by soapy water than the chlorinated paraffins. Extraction studies in alkali solution indicate that there was a reaction between the NaOH and the chlorinated secondaries rather than an extraction.

Soapy water extraction results showed Aroclors 1254 and 1260 to be equivalent to Chlorowax 70 and Panaflex BN-1 (alkylated naphthalenes), however, all of the Aroclors were worse than the other chlorinated paraffins.

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TABLE I

Plasticizers		Soapy Water Extraction*	Flame-Out Time
Aroclor 1232	(1:5 DOP)	4.6%	31 sec.
Aroclor 1242	(1:5 DOP)	4.0	41 sec.
Aroclor 1248	(1:5 DOP)	3.7	34 sec.
Aroclor 1254	(1:5 DOP)	3.6	48 sec.
Aroclor 1260	(1:5 DOP)	3.5	48 sec.
Clorafin 40	(1:5 DOP)	1.7	60 sec.
Clorafin 40V	(1:5 DOP)	1.6	67 sec.
Clorafin 42	(1:5 DOP)	1.7	66 sec.
Clorafin 42S	(1:5 DOP)	2.0	40 sec.
Chlorowax 40	(1:5 DOP)	2.5	60 sec.
Chlorowax 70	(1:5 DOP)	3.5	51 sec.
Halowax 4004	(1:5 DOP)	1.6	97 sec.
Panaflex BN-1	(1:5 DOP)	3.6	75 sec.

* % plasticizer lost in 1% Ivory soap solution.

Flammability tests to determine flame-out times (General Cable Test) indicated that the Aroclors were more flame-retardant than the other secondaries tested except Clorafin 42S. Chlorowax 70 and Panaflex BN-1 burned until they were completely charred.

Extraction tests in 50% alkali solution gave results that varied from 0.21 g. lost to 0.21 g. gained, indicating that rather than extraction a chemical reaction of the secondary with NaOH occurred.

2. Light Stability of HB-20

Several stabilization systems comprising ultraviolet Absorber No. 9, Santowhite Powder mixtures, phenyl salicylate and UV No. 9 alone have produced the best light stability yet encountered in film containing appreciable amounts of HB-20. See Chart VIII in the Appendix.

For further information on the light stability of HB-20, see the following reports:

Final Report No. 779 under Job No. 117-2595 (Ref. # 5)
Final Report No. 1005 under Job No. 117-2616 (Ref. # 6)
Final Report No. 1504 under Job No. 2-02-750.01-2946-I (Ref. # 7)

3. Evaluation of Santicizer 212

A Special Report has been written concerning the performance of the Santicizer 300 distillate as a secondary-type plasticizer for PVC. This is Final Report No. 1312 under Job No. 2-02-750.01-2894-I (Ref. # 8).

The continued intensive evaluation of Santicizer 212 (Santicizer 300 topped distillate) indicates it is more sensitive to soapy water than is DOP. From tensile strength, elongation, and modulus determinations, it was shown that S-212 is very similar to DOP. Stabilization of S-212 has not been too successful to date. The usual stabilization systems have only prolonged compatibility from 50% to 100% and poor compatibility stability is evident with two of the four stabilizer systems used.

Santicizer 212 Soapy H_2O Extraction Results

(1% Ivory Soap Soln., 96 hrs. at 50°C.)

	% Loss
DOP	3.4%
S-212 (1:1 DOP)	5.8
S-212 (1:2 DOP)	5.4

Physical Properties of S-212 Formulations

	Tensile Strength (psi)	Modulus(100%)	Elongation(%)
DOP	2552	1376	391
S-212 (1:1 DOP)	2532	1296	435
S-212 (1:2 DOP)	2540	1324	415
S-212 (1:3 DOP)	2540	1308	394

Efforts to stabilize Santicizer 212 to light have not been too successful at the present time. In outdoor exposures the lead system started to degrade after 2 months. In the window exposure tests, the lead and Staycin I systems were compatible after six months; however, the "epoxy cadmium" and dibutyl tin dilaurate systems were incompatible after five months. The unstabilized formulations showed no signs of incompatibility for three to four months.

C. Evaluation of New Process Plasticizers

1. New Process DOP from Everett Plant

A sample of plant process DOP (di-2-ethylhexyl phthalate) from new Everett production was screened and found to be completely equivalent to present production DOP. Heat and light tests emphasized this same equivalence.

	T_f°C.	Volatility	Kerosene Extraction
DOP (Everett Composite)	-38.3	4.6%	54.3%
DOP (Standard)	-38.2	4.6	49.3

2. New Process Santicizer 160

a. Distilled S-160

In an effort to determine the effects of distillation upon the plasticizing properties of Santicizer 160, a comparative evaluation of S-160 was accomplished. Screening results were very closely paralleled, showing no deviations whatever, including volatility and soapy water extraction. See Chart IX in the Appendix.

b. Topped S-160

Topped (most volatile fraction removed) Santicizer 160 is improved in volatility both per se and in PVC at a 40% concentration. These results are found in Chart II in the Appendix.

D. Intensive Evaluation of Santicizer 213

The early work on Santicizer 213 (S-160/DDB, 3:1) is contained in Final Report No. 1425 under Job No. 2-02-750.01-2896-I (Ref. # 9). However, at the expense of some repitition a summary of the work presented up to the end of 1955 is included here.

Santicizer 213 was screened preliminarily at 20%, 30%, 40% and 50% concentrations in a direct comparison with Elastex 50-B (Barrett's butyl cyclohexyl phthalate), a product of apparently similar plasticizing characteristics. This similarity was evident in most of the screening tests. It is, however, more water sensitive than is 50-B. See Chart X.

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Both heat and light stability tests indicate S-213 to be slightly inferior in comparison to 50-B. This inferiority was more pronounced in the heat tests than in the light tests.

S-213 was shown to have a compatibility rating definitely superior to that of 50-B, which is an accepted primary plasticizer. Two weeks in the Loop Compatibility test gave very slight indications of incompatibility, with the signs of exudation being more pronounced in the case of 50-B. Unstabilized S-213 exposed to sunlight behind glass is still compatible after 3 months. Elastex 50-B developed slight tackiness after one week.

Plastisol evaluations of S-213 in an unstabilized formulation indicate this to be its most promising field. At 80 PHR, it shows excellent viscosity-stability after one month at room temperature. Its stability is similar to DOP at 40°C., in that it gels after 7 days. Fast gellation was evident at 50°C. -- it gelled within one day.

Santicizer 213 was tested in dry blending and extrusion as 50 PHR formulations. Faster fusion than S-160 or DOP was evidenced by shorter drying time and decreased extrusion rate. Substitution of S-213 for one fourth of the DOP in one formulation had the effect of increasing the drying and fusion rates. This formulation was also characterized by the "slip" quality of S-213. This increase in fusibility and surface "slip" were noted when S-213 was field tested at Sinclair and Rush, Inc.

On the basis of tests at Weymouth Art Leather Co., the performance of Santicizer 213 was essentially equivalent to that of DOP with the exception of volatility which is noticeably poorer than that of the control.

The phenomenon of surface "slip" previously noted in the use of Santicizer 213 in dip molding formulations is also evident in cloth coatings prepared from this plasticizer. This property was considered desirable by Weymouth because, at the present time, additional top coats are applied to surfaces having high "drag" to facilitate slipping.

Also worthy of comment was the decided superiority of S-213 from the standpoint of viscosity. Tests at Weymouth show S-213 modified plastisols to be about half as viscous as dispersions containing DOP. It is conceivable that the use of this material might diminish the amount of diluent required or even eliminate it entirely in some cases.

Coated Fabric Evaluation

	Formulations 1	2	3
Geon 121	100	100	100
DOP	60	---	---
S-213	---	60	30
DIDP	---	---	30
Ferro 1203X	2	2	2
Apco Thinner	5	5	5
Rex Orange (Imperial Pigment)	20	20	20

	Results 1	2	3
Flexibility* (Clark) mm	46	49	55
Abrasion Resistance	No difference between samples		
Thickness, mils	18	17	21
Spew Test, 1 hr.	N.T.	N.T.	N.T.**

* The lower the value, the higher the flexibility

** N.T. = No tack

APPENDIX - I

Notebook Pages

53744	64605	64647	68869	71289
58975	64606	64648	68870	72264
58976	64607	64649	68871	72265
59918	64613	64650	71251	72266
59922	64622	66753	71252	72267
59926	64627	66754	71270	72268
59927	64628	66755	71271	72269
59930	64629	66757	71272	72270
59945	64630	66758	71273	74908
59947	64641	66763	71274	74909
59950	64644	66792	71275	74911
64603	64645	66800	71276	74912
64604	64646	68867	71277	74913

P. R. Graham

R. Dirby

3-19-7[illegible]

A P P E N D I X - I I

CURVES AND CHARTS

CHART NO. I

PROPERTIES OF PLASTICIZED OPALON 300

Plasticizer 40% unless specified	Milling results: Fuming	Milling results: Odor	Milling results: Color	Molded sheet: Trans-parency	Molded sheet: Odor	Molded sheet: [illegible]	T_f °C.	Volatility % Plast. Lost	Hardness: Before Vol.	Hardness: After Vol.	Heat Stability: Color after 30 mins. at 150°C in air	Heat Stability: Rating	Water Absorption: % Soluble	[illegible]	[illegible]
Santicizer 606 [illegible]	Sl.	Sl.	V. lt. yel	Clea[illegible]	V. Sl.	Yel	-19.7	[illegible].2	85	83	Dk. Amb.	F. P	0.0[illegible]	[illegible]	[illegible]
Santicizer 606 40%	Sl.	Sl.	V. lt. yel	Clear	V. Sl.	Lt. Yel	-32.2	[illegible]	[illegible]	73	Amb.	F	0.08	0.45	[illegible]
Santicizer 607 [illegible]	Sl.	Sl.	V. lt. yel	Clear	V. Sl.	Lt. Yel	-46.9	8.5	[illegible]4	72	Lt. Amb.	G	0.0[illegible]	0.40	[illegible]
Santicizer 611 [illegible]	Sl.	Sl.	V. lt. yel	Clear	V. Sl.	Lt. Yel	-17.1	[illegible].1	87	83	Amb.	F	0.0[illegible]	[illegible]	[illegible]
Santicizer 611 [illegible]	Sl.	Sl.	V. lt. yel	Clear	V. Sl.	Lt. Yel	-31.[illegible]	[illegible].9	[illegible]	[illegible]	Amb.	F. P	0.10	0.45	[illegible]
Santicizer 612 [illegible]	Sl.	Sl.	V. lt. yel	Cle[illegible]	V. Sl.	Lt. Yel	-[illegible].3	3.[illegible]	83	80	Lt. Amb.	G	0.07	0.36	[illegible]
Santicizer 612 40%	Sl.	Sl.	V. lt. yel	Clear	V. Sl.	Lt. Yel	-49.1	8.8	74	72	Lt. Amb.	G	0.09	0.56	66.1
Santicizer 614 [illegible]	Sl.	Sl.	V. lt. yel.	Clear	V. Sl.	Lt. Yel.	-37.8	1.4	88	73	Lt. Amb.	F	0.0[illegible]	0.5[illegible]	[illegible]
Santicizer 614 40%	Sl.	Sl.	V. lt. yel.	Clea[illegible]	V. Sl.	Lt. Yel.	[illegible]	[illegible]	[illegible]	[illegible]	Amb.	G	0.07	0.43	78.[illegible]
Santicizer 613 [illegible]	[illegible]	Sl.	V. lt. yel.	Clear	[illegible] Sl.	Lt. Yel.	-14.8	[illegible].4	92				0.07	0.42	[illegible]2.9

C H A R T   I I

PROPERTIES OF PLASTICIZERS

	Tf°C.	Volatility	Hardness: Before Vol.	Hardness: After Vol.	Water Absorption: % Solubility	Water Absorption: % Absorption	Kerosene Extr'n.
20% S-615	+10.1	2.4	100	100	0.01	0.20	Sl. Inc.
20% DnODP	+11.2	2.8	100	90	0.00	0.23	Sl. Inc.
30% S-615	-20.8	2.7	94	91	0.01	0.23	30.9
30% DnODP	-25.1	2.9	88	87	0.02	0.37	22.2
40% S-615	-46.3	2.1	76	70	0.02	0.52	77.7
40% DnODP	-46.5	2.2	72	73	0.01	0.40	69.0
50% S-615	-65.0	2.5	58	53	0.04	0.61	85.0
50% DnODP	-63.9	2.9	61	56	0.01	0.53	77.6
S-160, Topped	-24.0	9.5	74	73	0.09	0.71	3.3
S-160 (Analytically Pure)	-23.7	9.7	[illegible]	[illegible]	0.09	0.74	3.5
S-160, Standard	-23.2	11.1	75	73	0.09	0.66	3.6

DSW 620995

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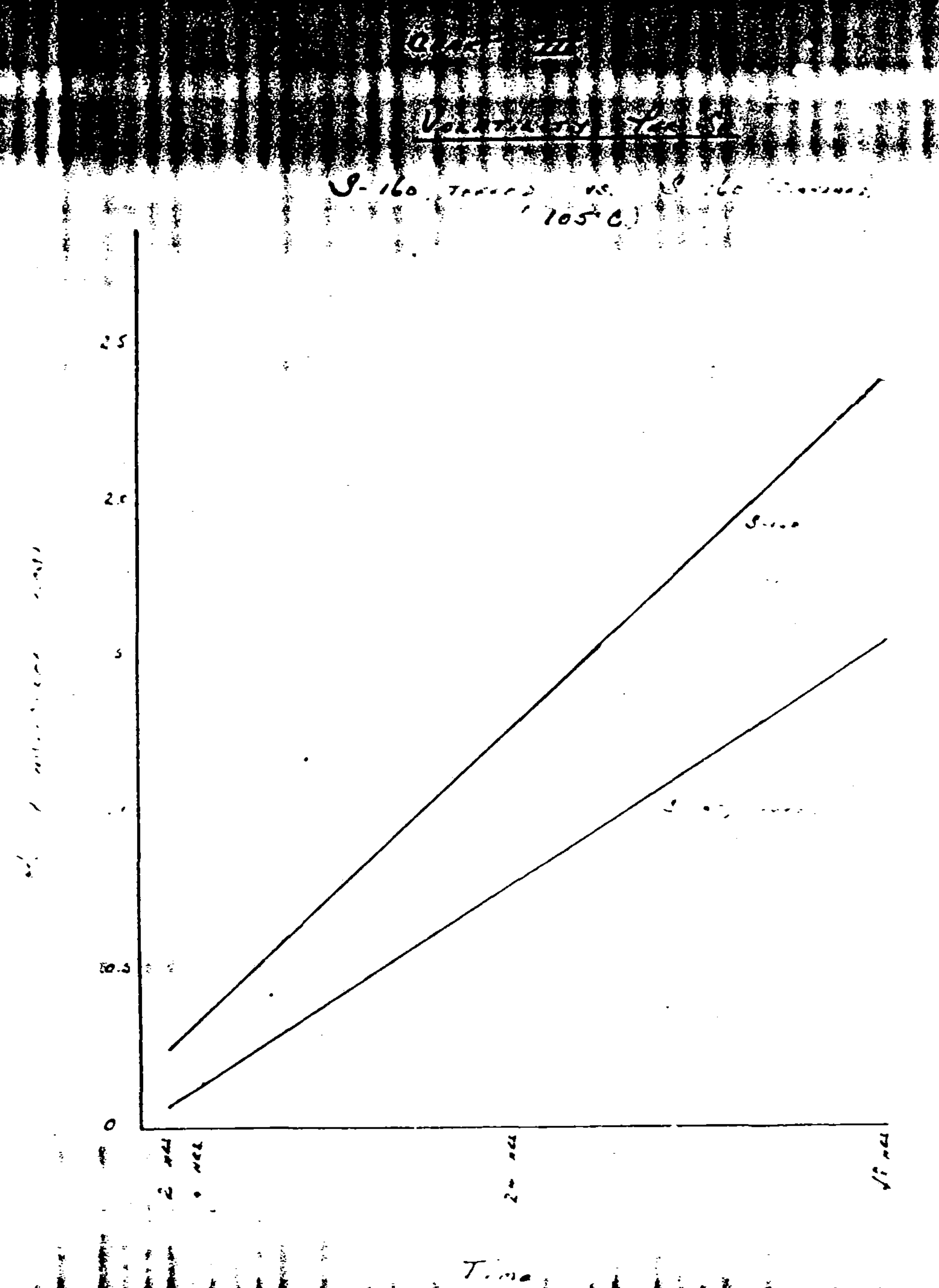
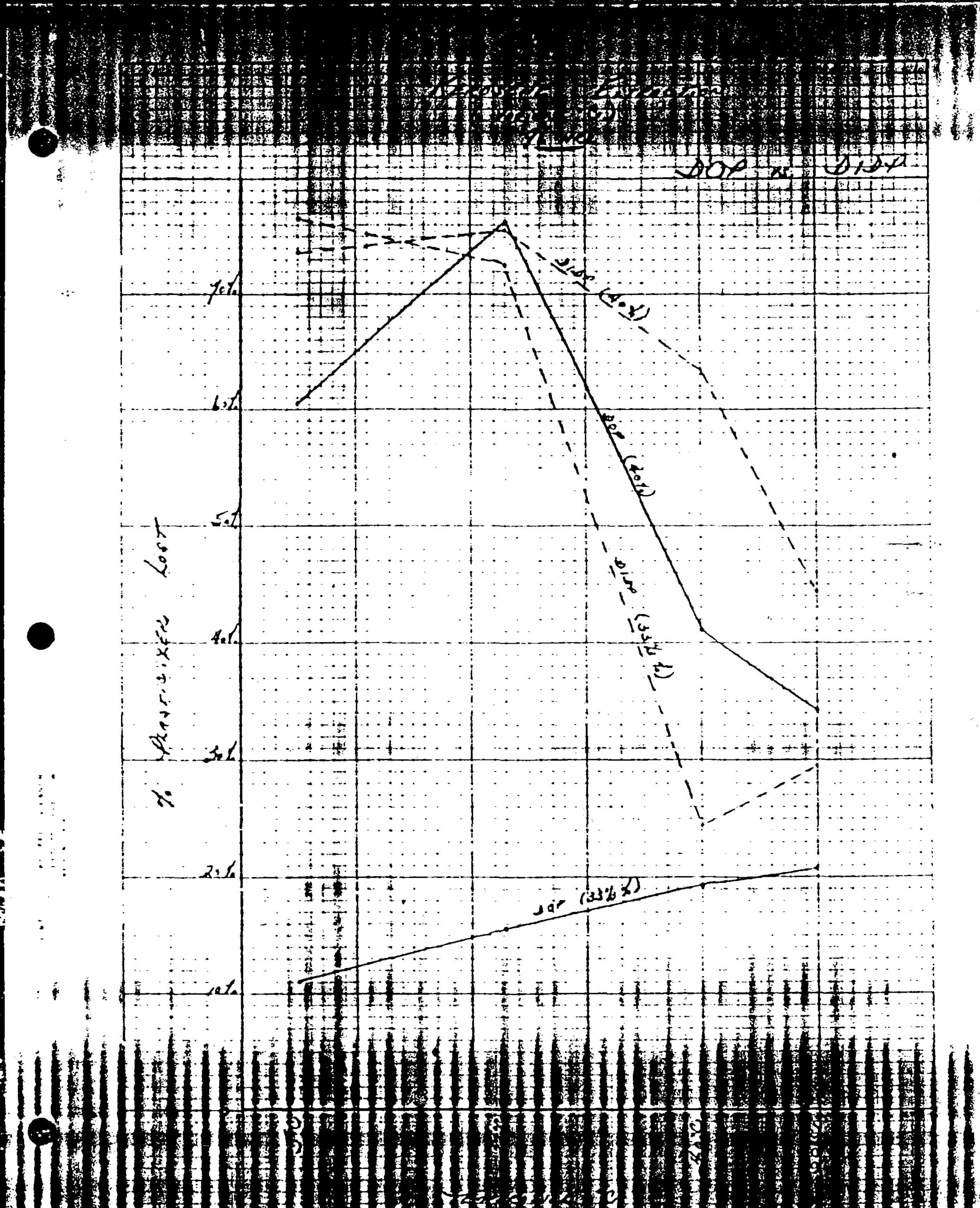


DSW 620996

STLCOPCB4095025


DOP vs DIDP
70%
60%
50%
40%
30%
20%
10%
% Plasticizer Lost
DIDP (40%)
DOP (40%)
DIDP (33⅓%)
DOP (33⅓%)

[illegible] PROPERTIES [illegible]

	Milling results			Molded sheet					Hardness		Heat stability		Water Absorption		
	Fusion	Odor	Color	Transparency	Odor	Color	[illegible]	Volatility	Before Vol	After Vol	Color after 30 mins 325°F.	Rating	% Soluble matter	% Absorption	
[illegible]	[illegible]	Sl.	Very Lt. yel.	V.Sl. Cloudy	Sl.	Lt. Yel	[illegible]	[illegible]					0.02	0.20	[illegible]
30%	"	"	"	"	"	V. Lt. Yel	[illegible]	[illegible]	[illegible]		INTENSIVE STUDY MADE		0.04	0.35	[illegible]
[illegible]	"	"	"	"	"	"	-40.0	2.40	[illegible]				0.02	0.49	
[illegible]	"	"	"	"	"	"	-52.2	2.51	62				0.11	0.71	
[illegible] 30%	"	"	"	"	"	Lt. Yel	[illegible]	[illegible]	[illegible]				0.03	0.28	[illegible]
30%	"	"	"	"	"	V. Lt. Yel	[illegible]	[illegible]	[illegible]				0.05	0.42	[illegible]
40%	"			"	"	"	[illegible]	[illegible]					0.07	0.61	
[illegible]	"						[illegible]	[illegible]					0.15	0.83	[illegible]

DSW 620998

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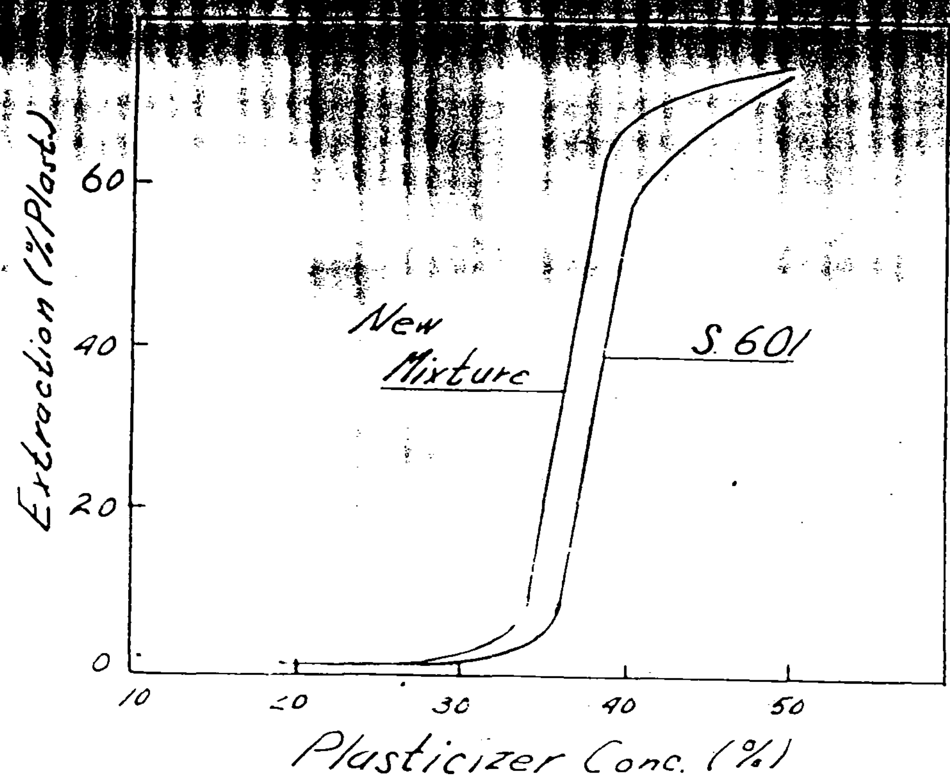
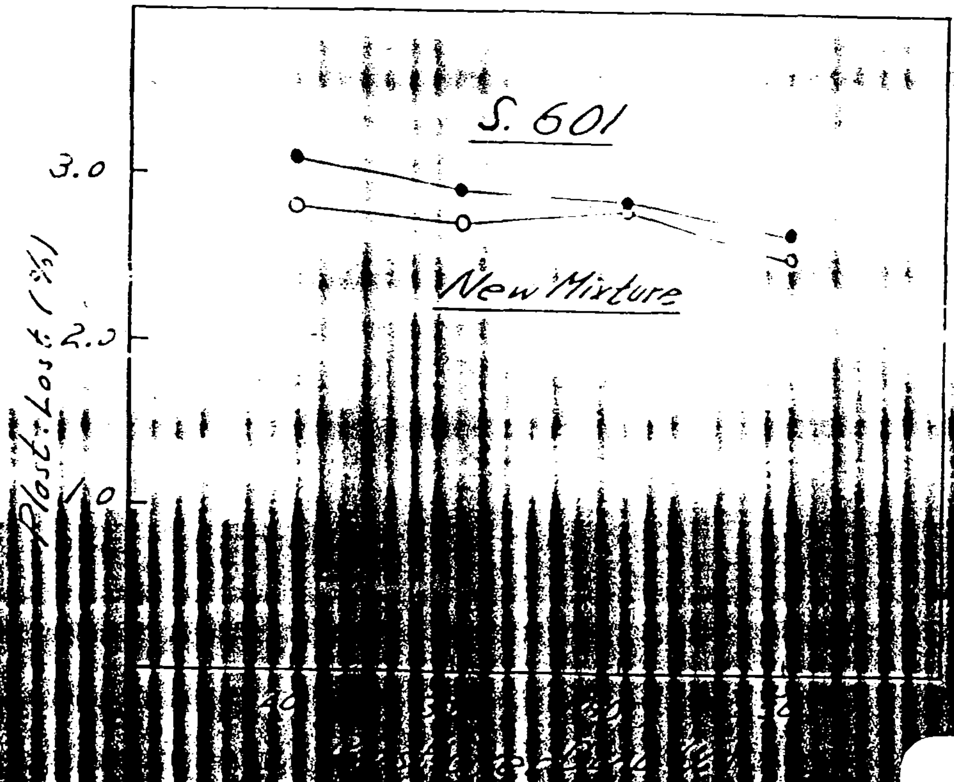
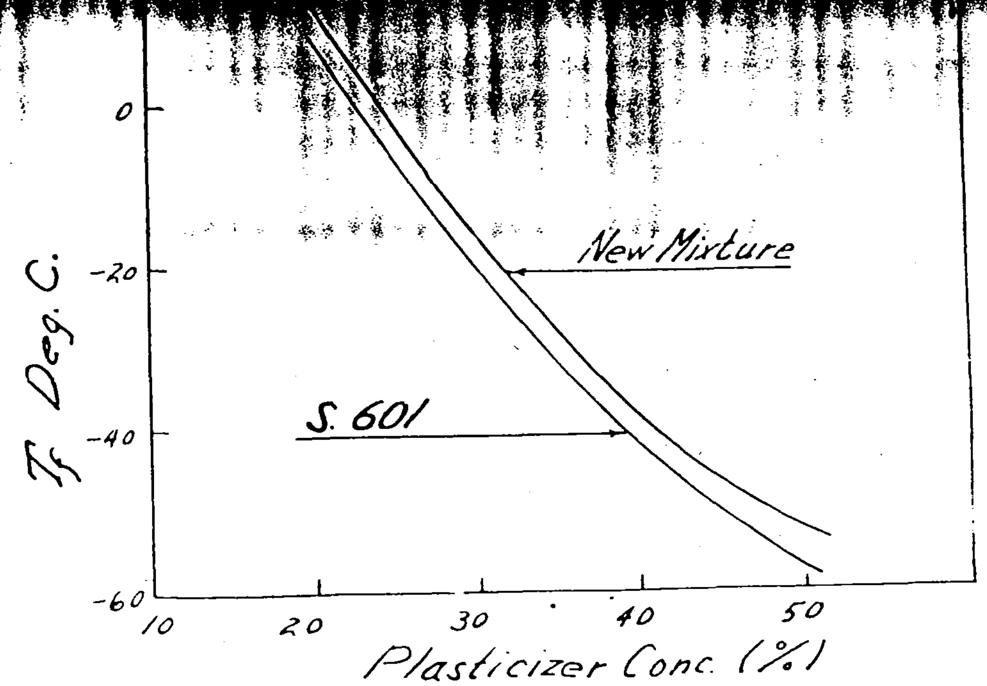


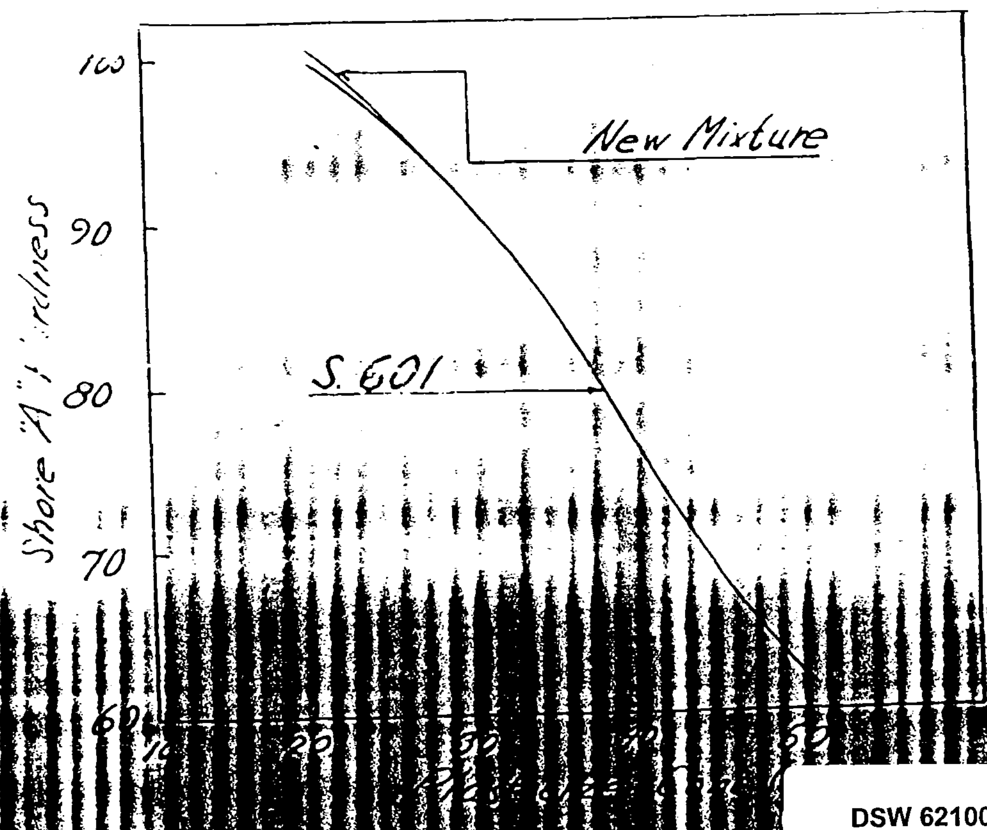
Kero-sene Extr.



Volatility



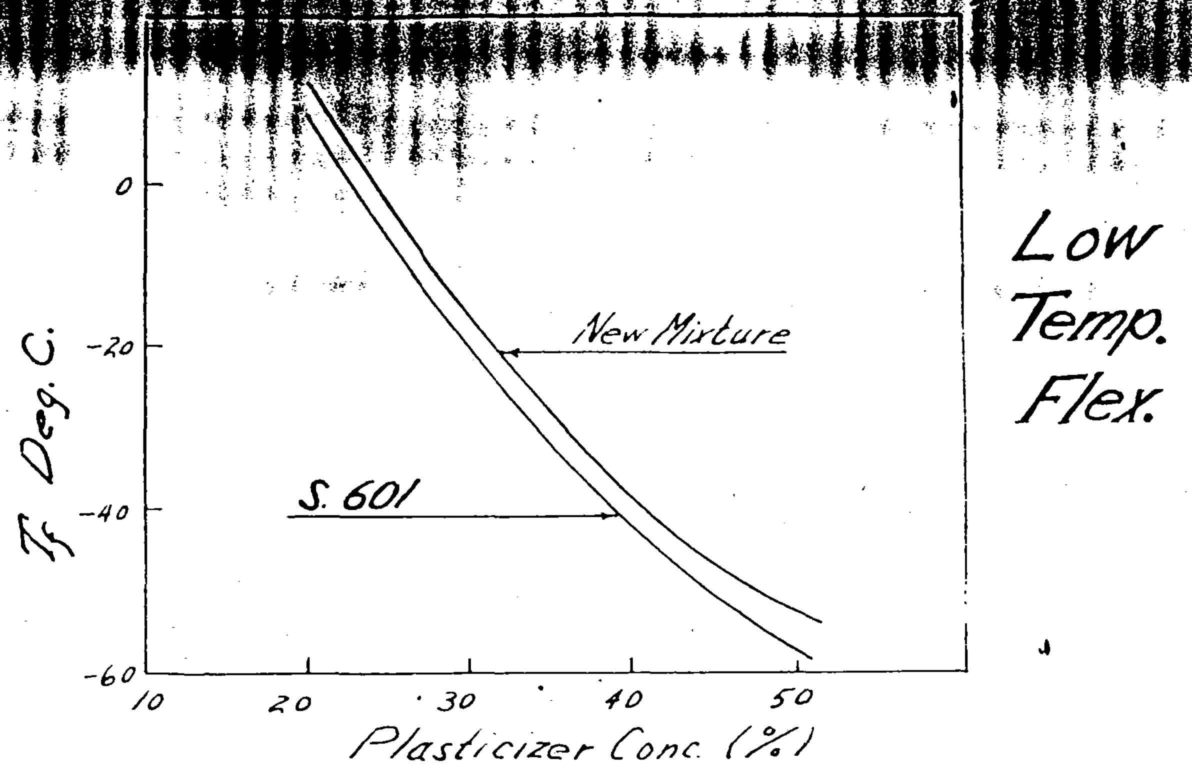
Low Temp. Flex.

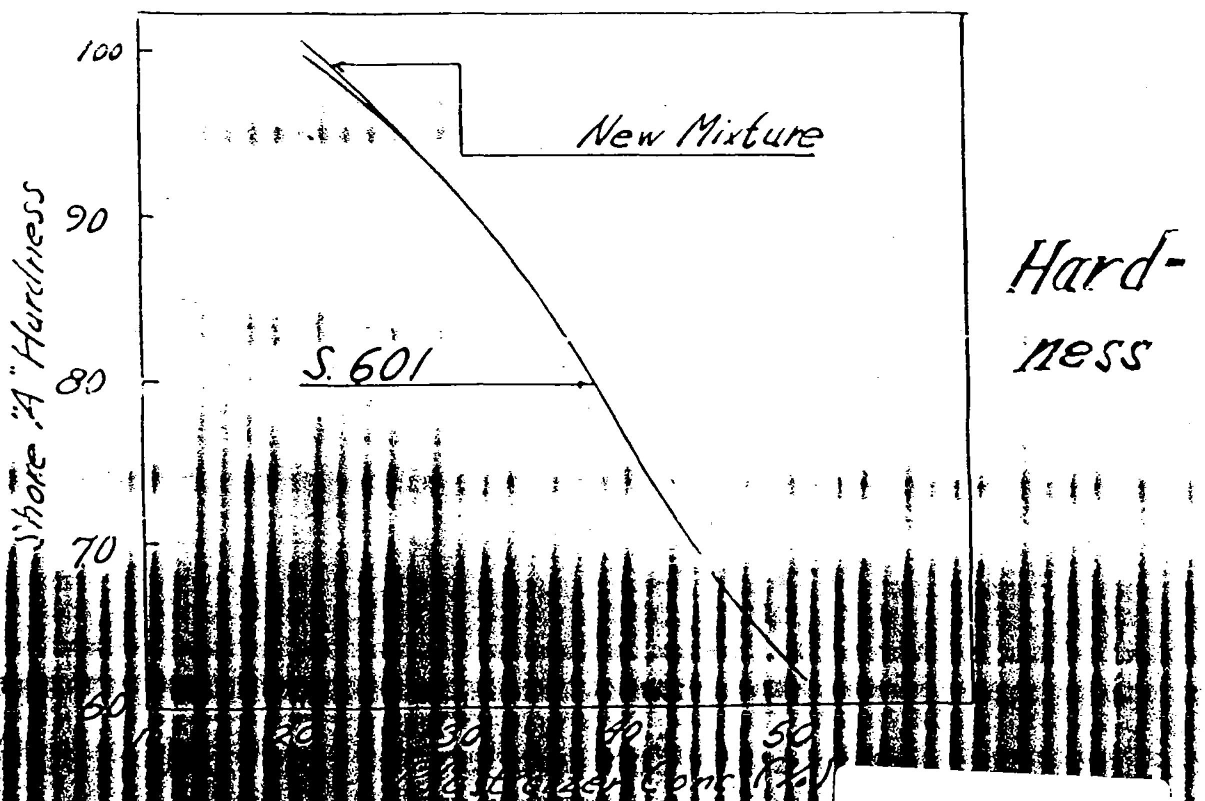


Hard-ness

[illegible]

EVALUATION OF SANTICIZER 301





DSW 621001

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CHART VIII

LIGHT STABILITY OF HB-20

Formulations

	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Opalon 300	100	100	100	100	100	100	100	100	100	100	100	100	100	100
DOP	25	33	37.5	25	25	20	33	33	33	23	37.5	37.5	37.5	37.5
HB-20	25	17	12.5	25	25	25	17	17	17	17	12.5	12.5	12.5	12.5
G-60	3	3	3	3	3	3	3	3	3	3	3	3	3	3
[illegible]	2	2	2	2	2	2	2	2	2	2	2	2	2	2
[illegible]	1	1	1	1	1	1	1	1	1	1	1	1	1	1
[illegible]				4			3		4		3		4	
UV No. [illegible]					2	2	1	2		2	1	2		2
Santowhite Powder					2			2				2		
Santicizer [illegible]						5				5				5

Hours Exposed in Weatherometer	1	2	3	4	5	6	7	8	9	10	11	12	13	14
0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
[illegible]	4	3	3	2	0	1	0	0	1	0	0	0	1	0
100	5	3	3	2	1	2	1	0	1	0	1	0	1	1
[illegible]	5	4	3	3	1	2	1	0	2	0	1	0	2	1
300	5	4	4	3	2	3	2	0	2	0	1	1	2	2
400	5	4	4	3	2	3	2	1	2	1	2	1	2	2
500	5	4	4	4	2	4	2	1	2	1	2	1	2	2
[illegible]	5	4	4	4	2	4	3	1	2	2	2	1	2	2
[illegible]	5	4	4	4	2	4	3	1	2	2	2	1	2	2
800	5	4	4	4	3	4	3	2	2	2	2	1	3	2
[illegible]	5	4	4	4	3	4	3	2	2	2	2	1	3	[illegible]

CODE

0	Colorless	3	Yellow	6	Dark amber
1	Sl. discoloration	4	Light amber	7	Brown
2	Light yellow	5	Amber	8	Black

DSW 621002

CHART IX

COMPARISON OF THE PROPERTIES OF STANDARD SANTICIZER 160 AND DISTILLED S-160

	Distilled S-160	Std. S-160
Milling Results		
Fuming	Mod. - heavy	Mod. - heavy
Odor	Mod.	Mod.
Color	V. lt. yellow	V. lt. yellow
Molded Sheet		
Transparency	Clear	Clear
Odor	Slight	Slight
Color	Lt. yellow	Lt. yellow
T_f°C.	-25°C.	-25.[illegible]°C.
Volatility, % Plast. Lost	10.[illegible]%	[illegible]%
Hardness		
Before Vol.	74	72
After Vol.	72	72
Heat Stability		
Color	Yellow	Yellow
Rating	Good	Good
H_2O Extraction, % Sol. Matl.	[illegible]	0.07%
H_2O Absorption	0.59%	0.59%
Kerosene Extraction	3.5%	5.3%
Soapy H_2O Extraction	17.[illegible]	1[illegible].1%

DSW 621003

STLCOPCB4095032

CHART [illegible]. X

PROPERTIES OF PLASTICIZED OPALON 300

[illegible] specified	Milling results: Fuming	Milling results: Odor	Milling results: Color	Molded sheet: Trans-parency	Molded sheet: Odor	Molded sheet: Color	T_f °C.	Volatility % Plast. lost	Hardness: Before Vol.	Hardness: After Vol.	Heat Stability: Color after 30 mins. at 325° F.	Heat Stability: Rating	Water Absorption: % Soluble matter lost	Water Absorption: % Absorbed [illegible]	[illegible]
[illegible]	Mod. Heavy	Sl.	Lt. Amb	Clear	Sl. Mod	Yel.	20	5.0	100	100	Amb.	F	0.00	0.45	[illegible]
[illegible]	Mod. Heavy	Sl.	V. Lt. Amb	Clear	Sl. Mod	Lt. Yel	20	4.7	100	100	Lt. Amb.	F	0.00	0.43	[illegible]
[illegible]	Mod. Heavy	Sl.	Lt. Amb	Clear	Sl. Mod	Lt. Yel.	- 1.7	10.2	98	97	Lt. Amb.	F	0.15	0.79	[illegible]
[illegible]	Mod. Heavy	Sl.	V. Disc	Clear	Sl.	Lt. Yel.	1.9	12.5	97	99	Dk. Yel.	G - F	0.09	0.89	[illegible]
Santicizer 213 [illegible]	Heavy	Mod Str	Cl.	Clear	Sl. Mod	V. Lt. Yel.	-21.0	13.1	32	35	Lt. Amb.	F	0.54	1.23	[illegible]
[illegible]	Heavy	Mod. Str.	Cl.	V. Sl. Cldy	Sl. Mod	V. Lt. Yel.	-1[illegible]	13.5	78	84	Yel.	G	0.09	0.95	[illegible]
[illegible]	Heavy	Mod. Str.	Cl.	Sl. Cldy.	Sl. Mod	V. Lt. Yel	-32.5	17.5	71	74	Yel.	G	0.60	1.65	[illegible]
[illegible]	Heavy	Mod. Str.	Cl.	Sl. Cldy.	Sl. Mod	V. Lt. Yel.	-31.3	22.1	66	66	Lt. Yel.	G	0.16	1.35	[illegible]

DSW 621004

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