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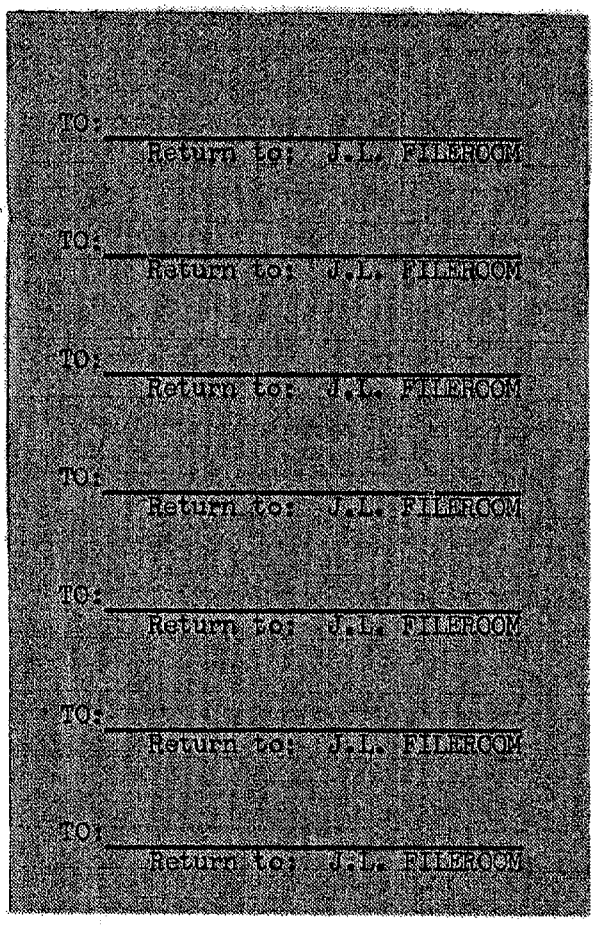
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JLR-35-63, No. 16

Serial Number 18708

Permanent Change in
Tent. Dye Works Process
18708-A

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CONFIDENTIAL

E. I. DuPont de Nemours & Company

Jackson Laboratory

November 13, 1943

"Lorol" Mercaptan from "Lorol" Chloride and "Lorol" Sulfate

Progress Report

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"Lorol" Mercaptan from "Lorol" Chloride and "Lorol" Sulfate
(Project Number 2267)

L. C. Holt

JLR-35-63, No. 16 Serial Number 18708

Object:

To complete the work on the manufacture of "Lorol" mercaptan from "Lorol" chloride, the beginning of which is described in JLR-35-63, No. 14; Serial Number 18590. The main purpose of the work was to determine what quality of "Lorol" chloride and sodium sulfhydrate are necessary to give optimum yields and highest quality of "Lorol" mercaptan.

To co-operate with the plant in efforts to improve and stabilize the present manufacturing process for "Lorol" mercaptan starting from "Lorol" sulfate or "Gardinol" WA Paste. ("Lorol" = fatty alcohol)

Period Covered by the Report:

August 12, 1943, to November 9, 1943.

Historical Background:

"Lorol" mercaptan is being manufactured at present from "Lorol" sulfate or "Gardinol" WA Paste by the action ofaqueous sodium sulfhydrate. This process will be replaced by one starting from "Lorol" chloride and high strength, solid sodium sulfhydrate as soon as the materials are available. D. P. Graham in JLR-35-63, No. 14; Serial Number 18590 describes the basic work on which this latter process is founded. In a plant demonstration of his process, Graham found that different lots of "Lorol" chloride gave widely different results. He also found that different samples of solid sodium sulfhydrate also gave varying results. The Grasselli Dept. of the DuPont Company has undertaken to manufacture these two necessary raw materials. Work has therefore been carried out in co-operation with the Grasselli Dept. on samples submitted by them to determine the necessary specifications of quality which will insure raw materials of uniform reactivity in the production of "Lorol" mercaptan with both high yield and quality.

The current plant process starting from "Gardinol" WA Paste has failed to give process yield. Much yield and operating time has been lost through the formation of emulsions which have rendered it impossible to completely separate the final product from the aqueous layer. Also losses have resulted from the failure of the emulsions to separate on long standing. Work has, therefore, been done to prevent the formation of emulsions in the process and also to break the emulsions already formed in order to recover the product thus tied up.

Conclusions:

It has been determined that "Lorol" chloride in order to give an optimum yield and high quality of "Lorol" mercaptan must contain close to the theoretical amount of chlorine, preferably as near 16% as possible and be comparatively free from distillation residues, preferably less than 5%.

Sodium sulfhydrate, to give optimum yield and high quality of "Lorol" mercaptan must have a low content of sodium thiosulfate, preferably 0.4% or less and contain about 75% or more of sodium sulfhydrate.

In order to avoid emulsion formation in the current process the "Gardinol" in the charge must be completely reacted. An extremely small amount of a sulfur-containing, high molecular weight by-product will produce emulsions in the final product. The plant has increased the excess of sodium sulfhydrate and time in the process and difficulties with emulsions have practically disappeared. Following a suggestion from the laboratory of the "Gardinol" plant, emulsions produced in the past operation have been broken by the use of a small amount of MP-189 Crude (a paraffin oil-sulfonate) solution. The product contained in these old emulsions has all been recovered and added to current production.

Summary:

1. Seventeen (17) samples of "Lorol" chloride from the Grasselli Department have been studied. Tests involving the conversion of the chloride to the mercaptan have been made using various samples of sulfhydrate. Because the change in molecular weight in passing from the chloride to the mercaptan is extremely small the purity of the mercaptan produced is equivalent to the yield, provided there is no mechanical loss. For this reason the results are stated entirely in terms of purity of the mercaptan, assuming a molecular weight of 216. The lowest purity

obtained in the tests was 80% and highest 96%. The chloride which gave a purity of 80% was shown to have a low chlorine content and to contain about 10% of distillation residue. The chloride which gave a purity of 96% was a laboratory prepared sample with a chlorine content of just over 16%.

2. The greater part of the high-boiling residue which is present in poor "Lorol" chloride samples is a product with the molecular weight of the ether. The attention of the Grasselli Department was called to the presence of this ether. On investigation they found that the hydroxyl of "Lorol" alcohol undergoing treatment with hydrogen chloride to produce the chloride decreases much faster than the chlorine content increases. The hydroxyl disappears completely long before the chlorine content of the reaction mass has reached the desired point. To obtain a satisfactory quality of chloride it is necessary to continue the treatment with hydrogen chloride until the ether is converted over to the chloride and the chlorine content has therefore reached a maximum.

3. Fourteen (14) samples of sodium sulfhydrate from the Grasselli Department were tested by converting selected "Lorol" chlorides to the mercaptans. Two samples of the same material from the Hooker Electrochemical Company were similarly tested. It was found that by far the best results were obtained from samples containing a minimum amount of sodium thiosulfate. Other impurities such as sodium sulfide and sodium carbonate did not seem to have any deleterious effect.

4. Emulsions similar to those occuring in the plant during operation of the process for making the mercaptan from "Gardinol" WA Paste were prepared by mixing as little as 0.08% of "Gardinol" with crude mercaptan containing an equal or even lesser amount of a high molecular weight, sulfur-containing body which was isolated from emulsions. On the basis of this showing the excess of sodium sulfhydrate used in the plant process was increased and the time of reaction was lengthened from four to five hours. These steps were taken with the object of completely reacting all "Gardinol" in the charge. Practically no emulsions have been produced since this step was taken.

5. Acting on a suggestion of the laboratory of the "Gardinol" plant samples of the many thousands of gallons of stored emulsion resulting from the "Gardinol" process were broken by the use of MP-189 crude solution. The mercaptan recovered from all these old emulsions by this method was added to current production.

Patent Situation:

Nothing of a patentable nature has resulted from work covered by this report. The patent situation is therefore unchanged from that described in JLR-35-63, No. 14; Serial Number 18590.

Plans for Future Work:

No further laboratory work is planned for the time being. When the Grasselli Department begins the regular manufacture of "Lorol" chloride in new apparatus now being installed, tests must be carried out on the conversion of their products to the mercaptan. Considerable of this type of work may be necessary while their plant is getting into operation. In the same manner tests must be applied to the sodium sulfhydrate which they will also produce in new equipment. As soon as satisfactory material is produced by them it is expected that regular production of the mercaptan from "Lorol" chloride and solid sodium sulfhydrate will begin. It will be necessary to co-operate with our plant during the early stages of the operation.

Experimental Results:

1. Tests on "Lorol" Chloride

When this work was started the only "Lorol" chloride available was a portion of plant Lot No. 4 and plant Lot No. 5. A carboy from Lot No. 4, designated as 61-30527, was obtained from stores. The only solid sodium sulfhydrate on hand was 100 pounds of 70% material supplied by the Hooker Electrochemical Company. This was a portion of the supply used in the original manufacture of the mercaptan from "Lorol" chloride as reported by D. P. Graham in report No. 14, referred to above. This sulfhydrate had been divided into gallon glass bottles and the tops sealed by paraffin to protect from moisture and oxidation.

Soon after the work was begun five carboys of "Lorol" chloride were received from the Grasselli Department. These were designated as numbers 34, 35, 36, 37, and 38. These were stated to represent experimental lots which were produced by significant changes in operation. Shortly after this, two samples of sodium sulfhydrate were received also from the Grasselli Department. These were high concentration samples produced by

the treatment of solid caustic soda with hydrogen sulfide followed by evaporation and centrifuging off the solid which separated as the mass cooled.

Tests were run on both the "Lorol" chloride and sulfhydrate samples in one or both of two autoclaves designated as Ex. 11 and Ex. 47. The former is a 1500-cc. and the later a 1000-cc. vessel. The charge in the former was 680 g. of "Lorol" chloride, 310 g. of 70% sodium sulfhydrate, or the equivalent amount of other strengths, 310 g. of 388 cc. of methanol and 3 g. of zinc dust. / The charge in the latter was 400 g. of "Lorol" chloride, 182 g. of 70% sodium sulfhydrate, 182 g. of methanol or 228 cc. and 1.1 g. of zinc dust.

The autoclaves were charged, the air displaced by raising the pressure to 25 lbs. by means of carbon dioxide and then releasing this pressure. The pressure was then raised by means of carbon dioxide to a somewhat variable point depending upon the amount of sodium sulfide in the sodium sulfhydrate used. The pressure was so set that during the six hours of heating at 135 to 140°C. the pressure in the autoclave would run from about 180 to 220 lbs. per square inch. The pressures generally used were 240 lbs. for Ex. 11 and 165 lbs. for Ex. 47. Variations in pressure during the reaction from about 180 lbs. to 220 lbs. appeared to make no difference in the results. Carbon dioxide was used to remove the air from the autoclave and also to convert the small amount of sodium sulfide in all of the sulfhydrate charged into sodium carbonate and sodium sulfhydrate.

Check runs were made with as many of the samples as possible. In such cases one charge was run in Ex. 11 and the other in Ex. 47. Runs were made at first at 5, 6, and 8 hours. There was no increase in yield after 6 hours so this was adopted as the standard time for all runs. As a rule results in the two autoclaves agreed within 2 to 2.5%.

The completed charges were worked up by removing from the autoclaves, washing the autoclaves with hot water and then filtering both the oil and water together through a filter mat of Filter-Cel. This removed a small amount of iron sulfide and made the separation of oil and aqueous layers easy. The oil layer was again filtered by gravity through a folded filter to remove traces of water and then warmed under reduced pressure to remove traces of hydrogen sulfide. The purity was determined by titrating about a 1-gram sample dissolved and suspended in

about 150 cc. of ethanol, with tenth normal iodine solution. The end point is a very faint yellow which persists after fifteen seconds of shaking. All titrations were made in duplicate and generally checked within about 0.3%. The purity was calculated by using the assumed molecular weight of 216. When the work covered by this report was about completed the Rubber Reserve Corporation decided that the mercaptan should be rated on the content of mercaptan sulfur. A purity of 96% based on the molecular weight of 216 is equivalent to a mercaptan sulfur content of 14.8%.

As already stated the purities reached in this work varied from a minimum of about 80% up to a maximum of 96%. The greater number of the results gathered in the region between 90 and 93%. It is now certain that a crude mercaptan with a purity better than 90% can be obtained provided only that the Grasselli Dept. furnishes "Lorol" chloride and sulfhydrate of consistently good quality.

Table I gives the results obtained with various "Lorol" chlorides when 70% sodium sulfhydrate supplied by the Hooker Electrochemical Company was used and Table II gives the results when a sample of sodium sulfhydrate supplied by the Grasselli Department and designated as No. 65 was used. It is to be noted that in all tests in which samples from the same batch of "Lorol" chloride were used with both Hooker 70% sodium sulfhydrate and Grasselli No. 65, superior results were obtained with the latter.

TABLE I

Tests with Hooker 70% Sodium Sulfhydrate*

Note Book Reference Book 4215 Pages	"Lorol" Chloride Used Sample No.	% Chlorine	% Purity		Average % Purity
31,68	61-30527	15.18 (Grasselli)	85.4	83.57	84.6
38,42	Dye Works Lot 5	15.26 (DuPont)	86.35	86.65	86.5
39,45	Dye Works Lot 6	15.22 (DuPont)	84.8	84.8	84.8
47,61	Grasselli #34	14.57 (Grasselli)	77.6	81.56	79.6
50,74	Grasselli #35	15.0 (Grasselli)	85.0	84.0	84.5
56,69	Grasselli #36	14.92 (DuPont)	87.3	86.9	87.1
57	Grasselli #37	15.23 (Grasselli)	79.6		79.6
58,70	Grasselli #38	15.05 (DuPont)	85.9	87.8	86.8

*Analysis by Jackson Laboratory - NaSH 68.44%, $Na_2S_2O_3$ 0.82%

TABLE II

Tests with Grasselli Cast Sodium Sulfhydrate #65*

Note Book Reference Book 4215 Pages	"Lorol" Chloride Used Sample No.	% Chlorine	% Purity
97	Dye Works Lot 5	15.26 (DuPont)	91.5
101	Dye Works Lot 6	15.22 (DuPont)	91.2
122,129	Dye Works Lot 7	14.55 (DuPont)	89.4
123	Dye Works Lot 8	15.10 (DuPont)	90.6
124	Dye Works Lot 9	15.00 (DuPont)	90.3
167	Grasselli No. 1	15.73 (DuPont)	93.9
165	Grasselli No. 2	15.38 (DuPont)	90.0
166	Grasselli No. 81	15.35 (DuPont)	91.1
164,176	Grasselli No. 82	15.85 (Grasselli)	93.3
161,168, 170	Grasselli No. 31011	15.59 (DuPont)	92.4
162,182	Grasselli No. 1 (First Sample)	15.50 (DuPont)	92.7
163	Grasselli No. 10 (Lab. Run)	16.03 (Grasselli)	96.3
108	Grasselli #34	14.57 (Grasselli)	86.22
104	Grasselli #35	15.00 (Grasselli)	88.3
105	Grasselli #36	14.92 (DuPont)	90.1
110	Grasselli #37	15.23 (Grasselli)	89.45
109	Grasselli #38	15.05 (DuPont)	89.5
114	Hooker #1	16.04 (DuPont)	93.9
111	Hooker #2	15.83 (DuPont)	94.5
115	Hooker #K-38-43	15.54 (DuPont)	92.1

*Analysis by Jackson Laboratory NaSH 80.4% $Na_2S_2O_3$ 0.47%

2. Tests on Sodium Sulfhydrate

These tests were carried out exactly like the tests on "Lorol" chloride except that portions of the same lot of "Lorol" chloride were used in each test while the sulfhydrate was varied.

Two types of samples were received from the Grasselli Dept. Both were made by treating solid caustic soda with hydrogen sulfide followed by evaporation. The lump samples were prepared by cooling the evaporated mass until a certain amount of solid had separated out and then whizzing on a heated centrifugal to remove the still liquid portion. The cast samples were made by evaporation to a certain point and then running the entire mass into drums and allowing it to harden. Some cast samples were treated with hydrogen sulfide gas during the evaporation stage and some were not treated. The latter would have a higher sodium sulfide content than the former. There is no evidence from our work that either sodium sulfide or sodium carbonate has any deleterious effect on the reaction, provided the former is converted to sodium carbonate and sulfhydrate by means of carbon dioxide at the start of the reaction.

Below are tabulated the results obtained with various samples of sodium sulfhydrate. The early tests were carried out using Batch 61-30527 of "Lorol" chloride. Later tests were all carried out using samples from Dye Works Lot 5. Sodium sulfhydrate No. 65 was tested with both "Lorol" chlorides so the results with this can be used to co-relate the two sets of tests.

TABLE III

Tests on Sodium Sulfhydrate Samples using "Lorol" Chloride No. 61-30527 (Cl Content 15.18%)

Note Book Reference Book 4215 Pages	Sodium Sulfhydrate Sample No.	Analysis Purity	Analysis $Na_2S_2O_3$	Purity of Mercaptan Produced (M.W. 216)
18,21	48 (Lump)	85.9	0.68 (Grasselli)	88.9
35	51 (Lump)	83.9	1.11 (DuPont)	83.4
75,80	60 (Lump)	88.4	1.25 (DuPont)	82.5
76,79	62 (Lump)	90.0	0.69 (Grasselli)	88.1
77,82	63 (Lump)	91.1	0.77 (Grasselli)	87.2
78,81	64 (Lump)	90.6	1.11 (Grasselli)	83.7
89	65 (Cast)	80.4	0.47 (DuPont)	90.1
88	68 (Lump)	90.7	0.47 (Grasselli)	89.7
31,68	Hooker 70% #1	68.4	0.82 (DuPont)	84.6

TABLE IV

Tests on Sodium Sulfhydrate Samples using "Lorol" Chloride - Dye Works Lot Number 5 (Cl - 15.26)

Note Book Reference Book 4215 Pages	Sodium Sulfhydrate Sample No.	Analysis Purity %	Analysis $Na_2S_2O_3$ %	Purity of Mercaptan (M.W.216) %
97	65 (Cast)	80.4	0.47 (DuPont)	91.5
130	69 (Lump)	90.1	0.51 (Grasselli)	90.0
128	73 (Cast)	79.4	0.66 (Grasselli)	88.3
131	71 (Lump)	90.1	0.57 (Grasselli)	89.9
132,138	76 (Cast)	78.8	0.44 (DuPont)	91.4
177	83 (Cast)	75.3	0.21 (Grasselli)	93.0
178	86 (Cast)	77.8	0.20 (Grasselli)	92.9
121,119	Hooker 70% #2 (XX940)	69.9	0.46 (DuPont)	91.9

The analyses of sodium sulfhydrates given in Tables III and IV are partly results supplied by the Grasselli Department and partly determinations carried out in Jackson Laboratory. The source of data is indicated in each case. The results for the critical impurity, sodium thiosulfate, differ widely in the two laboratories. The amount of this impurity found by Jackson Laboratory is always much higher than that reported by Grasselli. The suggestion that 0.4% be set as the specification upper limit is based on Grasselli figures. Before any specification figure can be set, a method of analysis to be used in both laboratories must be agreed upon.

3. Distillations of "Lorol" Chloride Samples

Several samples of "Lorol" chloride were distilled under reduced pressure with the object of determining the quantity and nature of the relatively non-volatile residue which might remain. The distillations were carried out in a short-necked flask with the neck well insulated. No sharp cut could be made and, therefore, the results obtained are not accurate quantitatively, but still they threw considerable light on the nature of impurities in inferior "Lorol" chlorides.

(a) Batch No. 34 - "Lorol" Chloride (Notebook 4215-48)

843 Grams was distilled as above under 7 mm. of pressure. 746 Grams or 88.4% of distillate was obtained up to 191°C. vapor temperature. The residue in the flask was 89 g. or 10.5%. The residue solidified on cooling. Some oil was sucked from this residue on a Buchner funnel and the remaining solid was well washed with ethanol. After drying the solid was sent for analytical examination.

The chlorine content was reported to be 1.16%, the hydroxyl number 13.8 and molecular weight 394. This composition and molecular weight suggested that the main component of the residue might be di-"Lorol" ether which would have a molecular weight of about 382.

(b) Batch No. 35 - "Lorol" Chloride (Notebook 4215-53)

This distillation was carried out in the same way as (a) above except that 3 mm. of pressure was used. The residue was 9.4%. This residue was examined without purification. The chlorine content was 3.3%, the hydroxyl number 4.7 and the molecular weight was 403.

(c) Batch No. 61-30527 (Notebook 4215-62)

This distillation was run using a 24-inch column surrounded by a jacket in which the temperature could be controlled. The pressure was 8 mm. 7.8% distilled up to 122°C., 54.4% 122° to 136°C. and 23.3% 136° to 165°C. The residue of 13.4% was transferred to a small flask with a short, well insulated neck and the distillation continued at 2 mm. pressure. The residue remaining at 200°C. was 4.6%. This residue solidified but was not examined further.

It is to be noted from Table I that Batch No. 34 gave a mercaptan of only 79.6% purity while 61-30527 gave a purity of 84.6%.

(d) Hooker No. K 38-43 (Notebook 4215-85)

This "Lorol" chloride had an unusually large amount of high boiling material but a low amount of final residue. Thus 86.7% distilled over up to 220°C. under 6 mm. of pressure whereas even poor batch #34 (see (a)) gave 88.4% up to only 191° under 7 mm. of pressure. An additional 10% distilled off from 220° to 255° under 3 mm. of pressure leaving a residue of 3%.

The last 10% distilling over had a molecular weight of 255 and a chlorine content of 13.22%.

The residue of 3% had a molecular weight of 462 and a chlorine content of 2.65%.

This "Lorol" chloride gave a mercaptan of 92.1% purity when converted with Grasselli Sulfhydrate No. 65.

(e) Attempt to Convert Distillation Residues to Mercaptan (Notebook 4215-139)

The attention of the Grasselli Department was called to the fact that a high boiling product was present in their "Lorol" chloride which appeared to be a di-"Lorol" ether. On investigation they determined that as "Lorol" alcohol is treated with hydrogen chloride the hydroxyl number decreases much faster than the chlorine content increases. They found that all hydroxyl disppeared when only about 80% of the desired chlorine had been introduced. By continued treatment with hydrogen chloride the chlorine content rises. It appears then that an ether is produced

along with the chloride during the early stage of treatment with hydrogen chloride and this ether is then gradually broken down to the chloride. It seemed desirable to determine if the ether present in "Lorol" chloride could be converted to the mercaptan directly.

300 Grams of "Lorol" chloride, solid distillation residues with a chlorine content of 0.98% was charged to autoclave Ex. 47 and heated during 6 hours with an excess of sodium sulfhydrate using methanol as a solvent. The temperature of treatment was 136-138°C. and the pressure in the autoclave was 300 lbs. The final product contained only 6.1% of mercaptan which is just what would be expected from the original chlorine content of the residue. The ether cannot, therefore, be converted directly to the mercaptan under the conditions of our process.

4. Distillation of Mercaptan Samples

All crude mercaptan produced in the plant from "Lorol" chloride and sodium sulfhydrate must be vacuum distilled as soon as distilling equipment is available. It was, therefore, advisable to make laboratory distillations in order to further check on what may be expected in the plant.

(a) Distillation of Mercaptan from Dye Works Lot 5 of "Lorol" Chloride (Notebook 4215-102)

500 Grams of this mercaptan with the purity of 89.9% was distilled under 2 mm. of pressure until the distillate began to crystallize in the condenser. The distillate was 467 g. with a purity of 95.4%. The distillate weighed 93.2% of the starting material and contained 99.2% of the mercaptan.

(b) Distillation of High Purity Crude Mercaptan (Notebook 4215-169)

1502 Grams of a composite of three laboratory prepared crude mercaptans with an average purity of 95% was distilled under 2 mm. of pressure. The highest vapor temperature reached was 165°C. at which point crystals appeared in the condenser. The distillate was 1390 g. with a residue of 100 g. The weight recovery was 93.6%. The distillate had a purity of 98.3% so the recovery of mercaptan was 96%.

5. Plant Operation of "Lorol" Chloride Process (Notebook 4215-145)

A change in process (Serial Number 18708-A) was written covering operation at 180 to 190 lbs. pressure (Notebook 4215-121).

Three plant charges were run using Lot 5 of "Lorol" chloride with Hooker 70% sodium sulfhydrate XX940 (No. 2). The purities of the mercaptans obtained in the three charges were 90.6, 91.35 and 90.5%. The time at reaction temperature was 6 hours and the pressure was kept below 200 lbs. at all times so there was no venting of the charges. Charges 1 and 2 were mixed to give Lot 1. This had a purity of 90.6%. Note that the laboratory result with Lot 5 of "Lorol" chloride and Hooker 70% sodium sulfhydrate as given in Table IV above is 91.9%.

Two charges were run using Lot 7 of "Lorol" chloride with the same sulfhydrate as above. The first charge was run very cautiously so the temperature did not come up to the required point promptly. The resulting product had a purity of only 81.5%. At our request another charge was run with rapid heating. This charge gave a purity of 89.7% which checks closely the laboratory result of 89.22 (Notebook 4215-122).

6. Studies on Emulsion Formation in the Plant Process for Manufacture of "Lorol" Mercaptan from "Lorol" Sulfate or "Gardinol" WA Paste

Samples of emulsions were obtained from the plant. Those from current operation were readily broken by acidification with sulfuric or hydrochloric acid. Then a sample of emulsion from old charges was found to be unaffected by acidification. This latter type proved to be an emulsion of water in oil and the former of oil in water.

A portion of the latter type was autoclaved with sodium sulfhydrate (Notebook 4215-148). This destroyed the small residue of "Gardinol" in the emulsion. The oil then separated from the water. This oil had a mercaptan content of only 54.3%. It was distilled under 3 mm. pressure. 70% distilled and had a mercaptan content of 76%. The residue solidified. A portion of this was purified by dissolving in ether and then throwing out by adding an equal volume of methanol slowly. This purified product had a molecular weight of 456 and a sulfur content of 12.16%.

A series of experiments was now carried out in which emulsions were produced by adding small amounts of "Gardinol" and or the above high molecular weight sulfur-containing compound to a mixture of plant mercaptan and the alkaline aqueous layer resulting from the plant operation. In brief the result of these experiments (Notebook 4215-156, 159) was that as little as 0.08% of "Gardinol" when accompanied with 0.06% of the above sulfur-containing body would form emulsions. This demonstrated the necessity for completely reacting all "Gardinol" charged into the reaction if emulsions are to be avoided. To reach this condition the plant immediately increased the molecular ratio of sulfhydrate used from 1.35 to 1 of "Gardinol" to 1.50 to 1 of "Gardinol" and lengthened the time of heating from 4 to 5 hours. With that change emulsion formation ceased.

Several thousand gallons of emulsions resulting from past operations were in storage. A sample from these had the consistency of sour milk. It was impossible to break this composite emulsion by means of acid. At the suggestion of the "Gardinol" laboratory the action of MP-189 Crude solution was tried. (Notebook 4215-171) It was shown that 5% of the volume of the emulsion of 12% crude MP-189 solution would break the emulsion completely and rapidly. All of the old emulsions on hand were broken by this method. A sample of the resulting oil which might contain as much as 10% of MP-189, provided all of that used went into the oil layer, was submitted to the Elastomer Division for test. They reported that the activity of the mercaptan for use in the polymerization step in the production of a Buna type of Elastomer was unaffected by the MP-189. All of the recovered oil has, therefore, been added to current production.

Submitted for typing - November 23, 1943
Typed - December 9, 1943
rvw

Literature and References:

1 - JLR-35-63, No. 14; Serial Number 18590. Progress Report by D. P. Graham
2 - Dye Works Process No. 18590-A
3 - Dye Works Change in Process No. 18708-A
4 - Notebook 4215 pages 18 to 192.