COMPANY CONFIDENTIAL

RESEARCH AND DEVELOPMENT REPORT
ANNISTON PLANT

Report No. 1
Job No. 446-513
Date 1-12-70

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		W. R. Haupt

Title: Aroclor 5460 Insolubles (Acetone)

Personnel: G. L. Arnett (J. T. Bell)

Problem: Aroclor 5460 acetone insolubles caused by over-chlorinated terphenyls.

Justification: Low soluble Aroclor 5460 has resulted in the production of approximately 200 M lbs. off-grade material in 1969.

Summary:

1. Information has been derived, establishing Santowax R quaterphenyl level - Aroclor 5060 soft point relationship. This data is necessary to prevent the formation of over-chlorinated terphenyls.

2. Distillation yields of Aroclor 5460 are effected by the quaterphenyl content of Santowax R. Santowax R with 1% quaterphenyls gives distillation yield of 93%, whereas, 10% quaterphenyl material reduces yield to 80%.

3. Total chlorine content of Aroclor 5460 can not be used as an indicator for over-chlorination of terphenyls.

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Background

Aroclor 5460 acetone insolubles is a specification test run by preparing a solution of 25 grams 5460 in 25 grams acetone. The solution must contain no precipitate for a period of 1-24 hours depending on customer demands. It is highly desirable to produce all Aroclor 5460 with 24 hour solubilities. For several years, it has been understood that all acetone insolubles in Aroclor 5460 were caused by biphenyl being in the Santowax. (Biphenyl chlorinated to 68% is insoluble in acetone.) Therminol leaks are the most common contaminate. In October 1969, it was discovered by Mass Spectrophotometry that some acetone insolubles were due to over-chlorinated terphenyls and, as a result, this investigation was directed toward determining the causes of the formation of over-chlorinated terphenyls.

Experimental

All Santowax R used in this study was current production material with the exception of the 1% and 15% quaterphenyl material. The 1% Santowax was prepared by blending essentially pure o-m-p terphenyls. The higher quaterphenyl (15%) material was blended using Santowax C (24% quaterphenyl) and 10% quaterphenyl production Santowax R. See Table I for ratios.

The chlorinations were performed to duplicate plant conditions as nearly as possible. All chlorinations were controlled by soft point analysis to determine degree of chlorination.

Distillations were carried out using 9 mm pressure to a maximum pot temperature of 380°C.

Experimental Details

Aroclor 5460 having a soft point of 103°C and above produced from 1% quaterphenyl Santowax R yielded insolubles that ranged from 8 hours or less solubility in acetone. Material that had a soft point of less than 103°C produced 24 hour solubilities. See Table II for more detailed results.

Aroclor 5460 made from 4% quaterphenyl Santowax R gave 16 hours solubility in acetone when chlorinated to a soft point of 105°C. (Finished specification soft point is from 98.5°C to 105.5°C.) Finished material having a soft point of 103°C had 24 hour solubilities in acetone. This indicates that lower quaterphenyls Santowax R requires a lower Aroclor 5460 soft point to obtain high soluble material. Santowax R containing quaterphenyls of 10% and above appear to create little problem when chlorinated to Aroclor 5460 specification soft point of 105.5°C. However, when a 10% quaterphenyl batch was chlorinated to a soft point of 117°C (Aroclor 5060) or 109°C (Aroclor 5460), less than 30 minutes solubility was obtained. A graph was prepared to set tentative specification limit on soft point of Aroclor 5060 versus Santowax R quaterphenyl content (Graph No. 1).

Total chlorine analyses were run (Anniston Method No. 093) on all distilled material (Table II). The range of chlorine is from 58.0-62.0% and has no apparent relation to insolubles.

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Experimental Details (continued)

Distillation yields were taken on all batches of Aroclor 5060. Increased quaterphenyl level in Santowax R decreases Aroclor 5460 yield. (Graph No. 3)

Santowax R quaterphenyl content effects the 5060-5460 soft point ratio. High quaterphenyl material requires a higher soft point Aroclor 5060 to obtain specification material. (Graph No. 2)

Conclusions

1. High soluble Aroclor 5460 can be made with Santowax R containing 1-15% quaterphenyls.

2. Lower quaterphenyl Santowax R requires a lower Aroclor 5060 soft point to produce high soluble Aroclor 5460.

3. The difference in Aroclors 5060 and 5460 soft point is directly proportional to the quaterphenyl content of the Santowax R.

4. Santowax R quaterphenyl content has a definite effect on Aroclor 5460 yields.

Recommendations

1. All Santowax R charged to chlorinators should be analyzed to determine quaterphenyl content.

2. Based on Santowax R analyses, all Aroclor 5060 soft points should be held to operating limits. This would eliminate low soluble Aroclor 5460 caused by over-chlorination of terphenyls. (Graph No. 1)

3. Control quaterphenyl level in Santowax R to 7% or below to obtain increased yields in Aroclor 5460.

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TABLE I

Santowax R Composition

% Quaterphenyl	% Ortho-Terphenyl	% Meta-Terphenyl	% Para-Terphenyl	% Biphenyl
1	13.0	57.0	29.0	NDA
4.2	11.1	55.6	28.7	0.2
7.3	11.4	53.1	28.0	0.2
10.5	11.6	50.6	27.3	0.1
15.0	10.6	48.6	25.9	NDA

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TABLE II

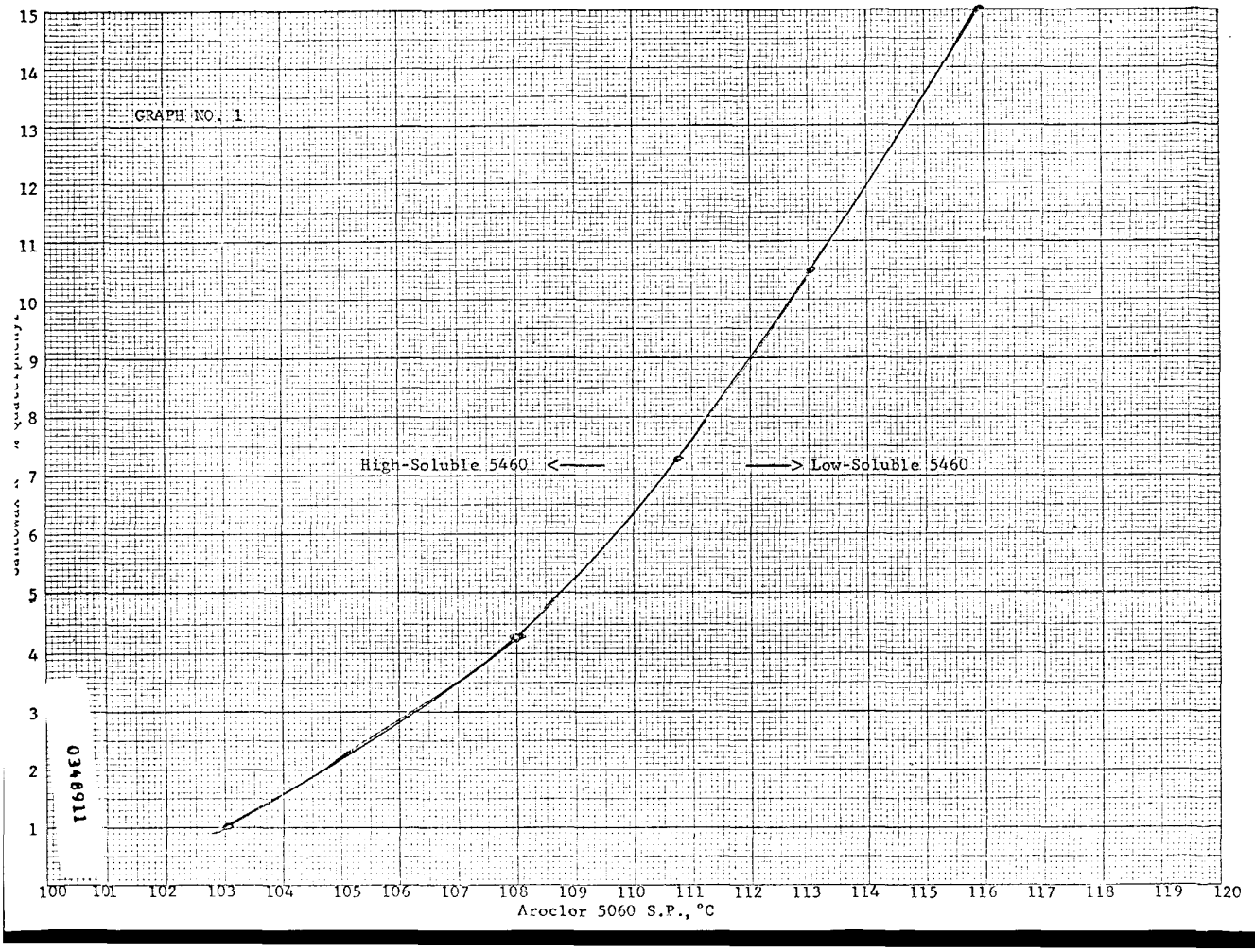
% Quaterphenyls	Soft Point 5060, °C	Soft Point 5460, °C	% Chlorine 5460	% Yield 5460	Solubility in Acetone
1.0	108.5	106.0	60.8	93.0	40 Min.
1.0	101.0	98.5	58.6	93.6	>24 Hrs.
1.0	105.5	103.0	58.9	93.5	8 Hrs.
4.2	109.0	105.0	60.2	91.1	16 Hrs.
4.2	107.5	103.0	60.1	91.1	>24 Hrs.
7.3	107.0	102.0	59.8	87.0	>24 Hrs.
7.3	102.0	97.0	58.7	88.0	>24 Hrs.
10.5	112.5	104.0	61.0	80.3	>24 Hrs.
10.5	117.0	109.0	61.6	80.2	<30 Min.
15.0	107.5	97.5	58.2	78.6	>24 Hrs.
15.0	116.0	105.5	59.4	76.9	>24 Hrs.

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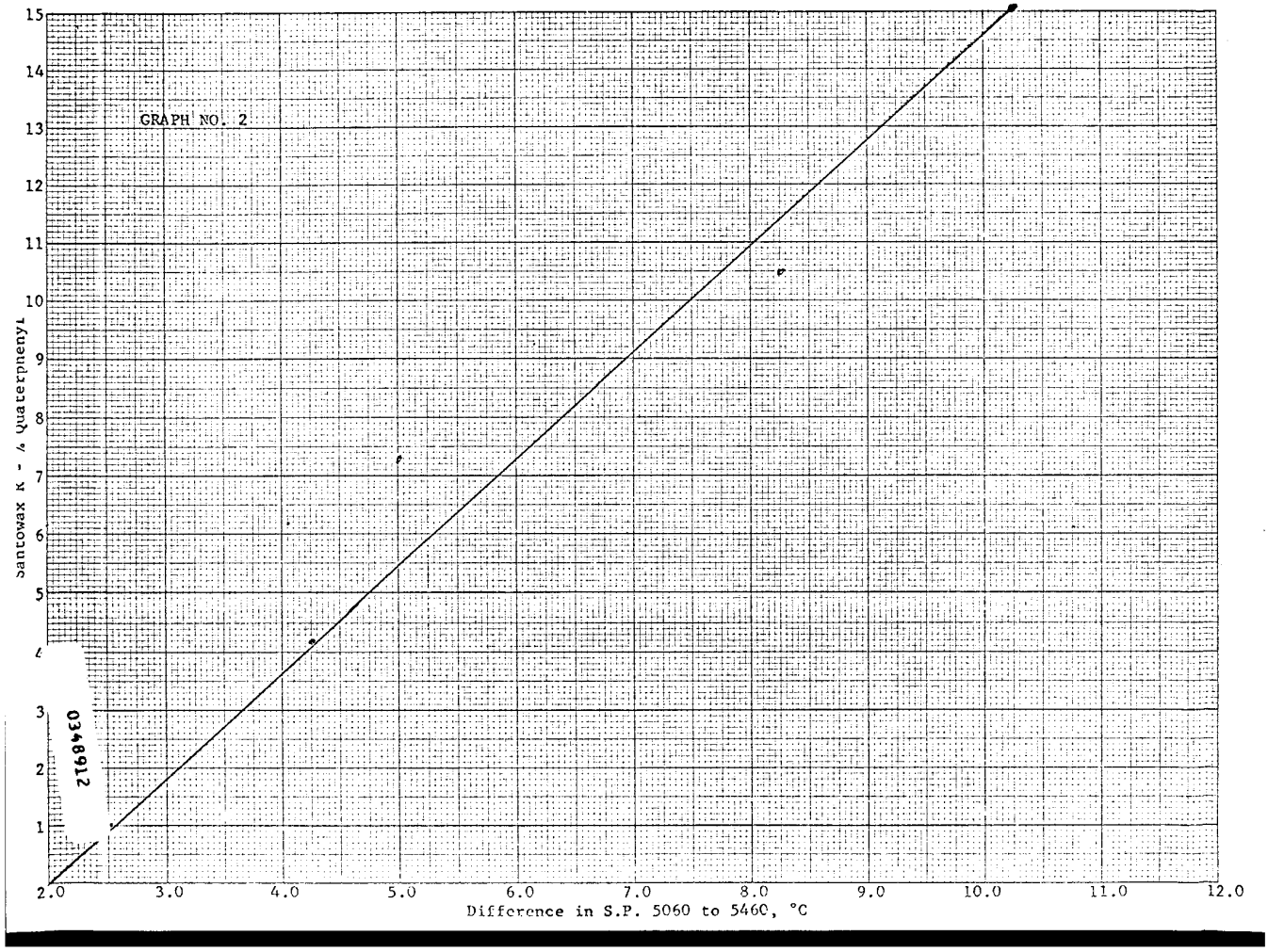


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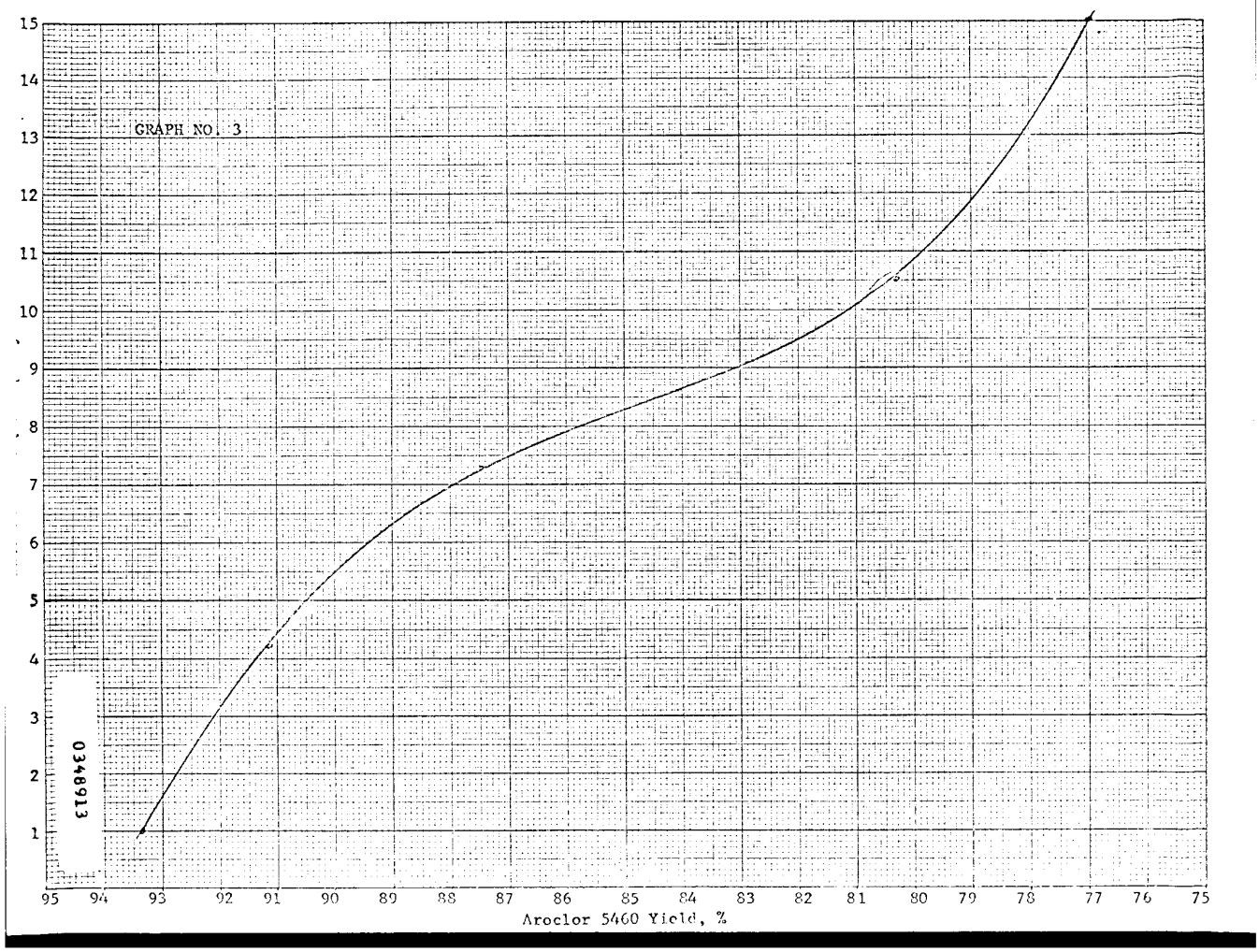


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