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NAVY DEPARTMENT

Report

on

The Solubility of Gums in Hydrocarbons at Temperatures Ranging from -22°F to 110°F.

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ANACOSTIA STATION
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ABSTRACT

The solubility of experimentally prepared gums from pure compounds has been studied. The solubilities were determined in three hydrocarbons and in a blend of these compounds at temperatures ranging from -22°F to 110°F. The compounds from which the gums were formed represent unstable structures which may be present in aviation gasoline or which may arise during oxidation in storage; the solvents wherein their solubility was investigated are hydrocarbons known to occur in aircraft fuels.

In general, the gums derived from cyclic hydrocarbons, tetralin, limonene and pinene and methyl tert-butyl ketone gum, exhibited the greatest solubilities, especially at high temperatures. Two ketone gums, acetonylacetone and cyclohexanone, were the least soluble. The comparative efficiency of the solvents was: xylene, a blend of xylene in methylcyclohexane and isooctane, methyl cyclohexane, and isooctane. The experimental values for the solubility of most of the gums in the blend did not agree with the ones calculated on the basis of the solubilities of the pure compounds. Decrease in temperature did not necessarily mean decreased solubility for although some of the gums exhibited great differences in solubility at different temperatures, the solubility of others was affected only slightly.

The findings of this study have been related to the problem of gum contamination in stored gasolines and engine use of gummed fuels.

INTRODUCTION

(a) Authorization

1. This problem was authorized by Bureau of Aeronautics Project Order No. 29/40 of 30 June 1939.

(b) Statement of Problem

2. This report will discuss the solubility of gums formed from pure compounds. The solubilities were determined in hydrocarbon solvents at temperatures ranging from -22°F to 110°F.

(c) Known Facts Bearing on the Problem

3. The detrimental effects of oxygen on certain types of compounds in a gasoline has led in this Laboratory to an investigation of the stability of pure compounds of diverse structures under various conditions. The structural characteristics of some pure compounds versus their stability are given by Naval Research Laboratory report No. P-1527, progress report "The Stability of Acetone, Hexone, and Cyclohexanone on Storage" included in Naval Research Laboratory letter of 24 April 1939 to the Bureau of Aeronautics, and also in a paper by Berger and Bost[1]. In these reports, as well as in the work reported in the literature on the stability of gasolines, the solubility of gums is considered chiefly from the standpoint of the solubility in the parent substances, that is, the substances from which the gums are derived. The solubility of gums in various common organic solvents is given in some cases but quantitative studies over any definite range of temperature, or even at any one temperature, have not been made.

4. This report deals with a quantitative investigation of the solubility of gums obtained during previous studies on the gumming tendencies of pure hydrocarbons and ketones. Three hydrocarbons, pure and blended with each other, were used as the solvents and the studies conducted over a 132°F range of temperature.

(d) Theoretical Considerations

5. The stability of a gasoline depends largely on the constituents of the fuel. Although the rate of deterioration of a gasoline on storage may, through a knowledge of the stability of certain types of compounds, be retarded by the use of inhibitors (antioxidants)*, and by storage under optimum conditions, yet long time storage sometimes practiced by the Navy, and long time storage contingent on the upbuilding of stocks of aviation gasoline for emergency, encourage the deterioration of gasoline; furthermore, conditions exist in the intake system of the motor itself (adequate supply of oxygen in intimate contact with the fuel at elevated temperatures) which favor oxidations that may be accompanied by some gum

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* When this work was instituted, inhibitors were not permitted in Naval specifications aviation grade gasoline. Since that time, revised specifications permit the use of certain inhibitors to the extent of 1 pound per 5000 gallons of fuel (A&N Specification AN-VV-F-776 and AN-VV-F-781).

formation in the fuel on its passage to the cylinders. Trouble from this source is probably less apt to occur than when gasoline already containing quantities of preformed gum is used. Even though the most scrupulous care be exercised in procuring gum free gasoline of low gumming tendency, it may be expected that some degree of deterioration will take place when the storage period is long. Whether this storage alteration is slight or great is covered mainly by four factors:

(1) The intrinsic gum forming tendencies of the gasoline.
(2) The type and degree of inhibition of the fuel.
(3) Storage conditions.
(4) Time of storage.

6. Gum deposition in engines using badly contaminated gasoline is more or less related to the total quantity of preformed gum per unit volume of fuel supplied the motor. In other words, a gasoline low in preformed gum will generally tend to foul engine mechanisms less than will a high gum fuel. Current A&N specifications limit the quantity of preformed gum to a maximum of 6 milligrams per 100 cc. Such a level of gum usually will not cause crippling deposits on engine parts between overhauls. In old deteriorated fuel this 6 milligram limit may be considerably exceeded and can rise beyond the limits for safe operation. The extreme level to which the gum content of a given gasoline can rise is limited only by the solubility of that particular gum in the fuel. If the source of gum be decomposition and oxidation of unstable components of gasoline, then the quantity of gum in the gasoline would increase as the deterioration process continues until the saturation point is reached; whereafter, gum separates from the fuel as a distinct phase. If, on the other hand, the original gasoline is stable with low gumming tendency, but is placed in dirty storage contaminated with deposited gums from earlier batch of fuel, these deposited gums will enter the gasoline either until the supply of predeposited gum is exhausted or the limiting solubility of that gum in the fuel is reached.

7. Gum, therefore, may be present in gasoline in two physical states: (1) dissolved in the fuel, or (2) existing as a separate phase which usually lies as an oily or sticky layer beneath the gasoline. Such gum as has separated from the gasoline is relatively harmless in so far as engine operation is concerned since it can have access to vital engine parts only if carried there by the fuel. Unless it is entrained in the gasoline by agitation or mixing, the only way the fuel can carry the gum is in a state of direct solution. Gasoline overlying separated or deposited gum is saturated with that particular gum and so, though undissolved material remains in the storage tank, much of that which is dissolved is ultimately deposited on engine parts. Undissolved gum left in the storage tanks remains as a potential hazard to contaminate fresh fuel when the tank is refilled.

8. When an engine having been run on gum laden gasoline is examined at overhaul, the carburetor mechanism and cooler parts of the intake system will generally contain soft, sticky, resinous deposits. On the hotter surfaces, the undersides of the valves and valve stems, the deposits are found as hard concretions; these latter are practically insoluble in gasoline. The softer deposits, on the other hand, are

somewhat soluble so that if the engine is shifted from a gum contaminated gasoline to a gum-free one, some removal of the soft deposits may be expected. With these facts in mind, this study was undertaken with the view of estimating the solubility of gums of known constitution in pure hydrocarbons present in aviation gasoline for the purpose of indicating what can be expected of various types of gasoline when contacted with old gum deposits either in an engine or in a storage system, and to show the relationship of gasoline types to the maximum amount of dissolved gum that they may contain.

9. This work also includes the solubility behavior of gums at different temperatures for the reason that gum saturated fuels, when cooled from temperatures prevailing at the site of storage during high altitude flight, deposit part of their gum content directly in the fuel tank of the plane. These gums which are very viscous liquids or solids at temperatures below 0°F may cause trouble through blocking of strainers, fuel lines and pumps. Thus it is seen that under the wide range of operating conditions of aircraft, a badly gummed gasoline may foul not only the engine itself but the entire fuel system.

10. As in the previous stability studies made in this Laboratory, this investigation is based, not on the behavior of gums formed from gasoline, but on gums from pure compounds of known structure. Whereas the composition of the former type of resins may vary due to the composition of the gasoline and also to the conditions under which the gums were formed, the composition of the latter may vary only with the conditions of gum formation. In this manner, by a study of gums of comparatively simple structures, an insight is gained on the general solubility of gums formed from difference types of compounds under like conditions.

11. Pure hydrocarbons representative of general types of hydrocarbons present in liquid petroleum fuels and themselves known to occur in aviation gasoline, were used as the solvents in this study. In order to obtain information as to the behavior of gums in a mixture of hydrocarbons, the solubility of the gums in a blend of definite composition was determined. The experimental values for the solubility of the gums in the mixture of hydrocarbons were compared to values calculated on the basis of percentage composition, by volume, of the blend.

(e) Narrative of Original Work

12. The solubility of nine different gums, four hydrocarbons, four ketones, and a hydrocarbon-ketone blend, in three pure hydrocarbons and a blend of these compounds, was determined at four different temperatures. Known volumes of saturated solutions of the gums at each of the four different temperatures were evaporated at 110° - 120°C in tared weighing bottles and the amount of gum per 100 cc of solvent calculated.

13. The data obtained, as given in tabular form and supplemented with graphs, are listed and discussed in a following section. Along with this discussion, an account of the preparation of the gums and also an explanation of the methods used in this study, are given. Additional

information such as a description of the purification and the physical constants of the solvents, are to be found in the appendix. The results of the analyses of some of the gums as reported in the literature, are also given in this section.

PREPARATION OF MATERIALS

14. The determination of gum in the stability studies of pure compounds as given in Naval Research Laboratory progress report entitled "The Stability of Acetone, Hexone, and Cyclohexanone on Storage" and in a paper by Berger and Bost[1] was accomplished by evaporating 10 cc of material in a tared glass dish at 110° - 120°C. These gums were dissolved in acetone, the solution poured in a beaker, and the acetone evaporated from a steam bath. Each weekly analysis of the previous study was followed by a continuation of this process so that over a period of time large enough quantities of gums were collected for this study.

15. Procedure. The solubility of these gums in the hydrocarbons and blends was determined by preparing saturated solutions of the gums at different selected temperatures and analyzing these solutions for gum content. A more detailed description of this procedure is given in Appendix A.

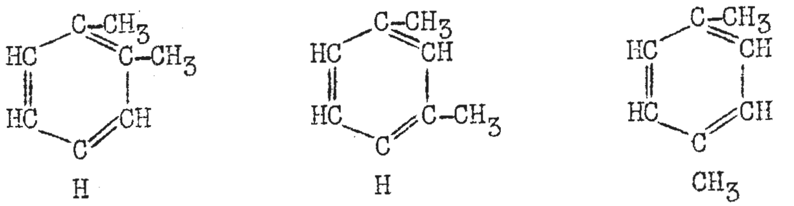
DATA OBTAINED

16. Data obtained, in tabular form, and a discussion of results are given below.

Table I

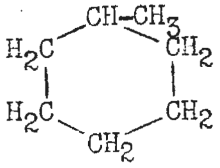
Solubility of Gums

Part I. Structure of Solvents

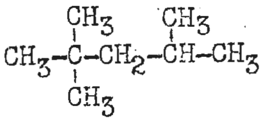


ortho meta para

Xylene



Methyl Cyclohexane



$CH_3-C(CH_3)_2-CH_2-CH(CH_3)-CH_3$

Isooctane

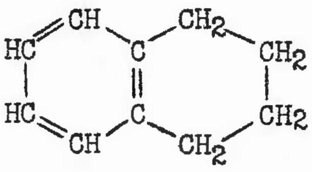
Xylene consisted of mixture of ortho, meta, and para isomers.

Blend contained 25% xylene, 20% methyl cyclohexane, and 55% isooctane by volume.

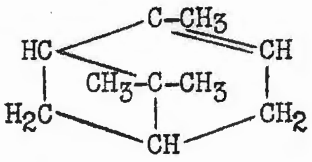
Table I (Continued)

Part II. Structure of Parent Compounds of Gum

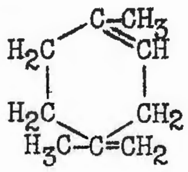
(a) Hydrocarbons



Tetralin



Pinene

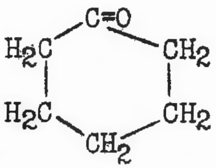


Limonene

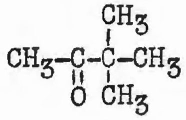
$CH_2=CH-CH_2-CH_2-CH=CH_2$

Diallyl

(B) Ketones

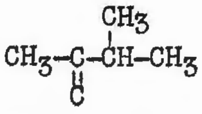


Cyclohexanone



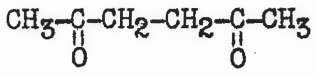
$CH_3-\underset{\underset{O}{\|}}{C}-\overset{CH_3}{\overset{|}{\underset{\underset{CH_3}{|}}{C}}}-CH_3$

Methyl tert-Butyl Ketone



$CH_3-\underset{\underset{C}{\|}}{C}-\overset{CH_3}{\overset{|}{C}}H-CH_3$

Methyl Isopropyl Ketone



$CH_3-\underset{\underset{O}{\|}}{C}-CH_2-CH_2-\underset{\underset{O}{\|}}{C}-CH_3$

Acetonylacetone

The methyl isopropyl ketone blend gum was formed from 10% methyl isopropyl ketone in isooctane by volume.

Table I (Continued)

Part III. (a) Solubility in Aromatic Hydrocarbon

Gum	Solvent	Temperature, °F.	Solubility Mg. per 100 cc
Tetralin	Xylene*	-22	426
		40	902
		70	1400
		110	1443
Pinene	Xylene	-22	524
		40	
		70	
		110	603
Limonene	Xylene	-22	750
		40	770
		70	776
		110	794
Diallyl	Xylene	-22	112
		40	
		70	116
		110	118
Methyl Isopropyl Ketone Blend	Xylene	-22	66
		40	
		70	
		110	188
Methyl tert-Butyl Ketone	Xylene	-22	144
		40	166
		70	222
		110	286

* See Part I.

Table I (Continued)

Part III. (a) Solubility in Aromatic Hydrocarbon

Gum	Solvent	Temperature, °F.	Solubility Mg. per 100 cc
Methyl Isopropyl Ketone	Xylene*	-22	131
		40	
		70	
		110	132
Acetonylacetone	Xylene	-22	70
		40	72
		70	72
		110	72
Cyclohexanone	Xylene	-22	63
		40	78
		70	88
		110	104

Part III. (b) Solubility in Hydroaromatic (Naphthenic) Hydrocarbon

Gum	Solvent	Temperature, °F.	Solubility Mg. per 100 cc
Tetralin	Methyl Cyclohexane	-22	25
		40	40
		70	44
		110	58
Pinene	Methyl Cyclohexane	-22	42
		40	
		70	
		110	120
Limonene	Methyl Cyclohexane	-22	63
		40	82
		70	112
		110	148

* See Part I.

Table I (Continued)

Part III. (b) Solubility in Hydroaromatic Hydrocarbon

Gum	Solvent	Temperature, °F.	Solubility Mg. per 100 cc
Diallyl	Methyl Cyclohexane	-22	30
		40	
		70	34
		110	36
Methyl Isopropyl Ketone Blend	Methyl Cyclohexane	-22	18
		40	
		70	24
		110	25
Methyl tert-Butyl Ketone	Methyl Cyclohexane	-22	76
		40	76
		70	76
		110	84
Methyl Isopropyl Ketone	Methyl Cyclohexane	-22	23
		40	
		70	24
		110	28
Acetonylacetone	Methyl Cyclohexane	-22	7
		40	9
		70	9
		110	12
Cyclohexanone	Methyl Cyclohexane	-22	7
		40	10
		70	10
		110	10

Table I (Continued)

Part III. (c) Solubility in Aliphatic Hydrocarbon

Gum	Solvent	Temperature, °F.	Solubility Mg. per 100 cc
Tetralin	Isooctane	-22	2
		40	12
		70	20
		110	36
Pinene	Isooctane	-22	81
		40	
		70	
		110	92
Limonene	Isooctane	-22	36
		40	38
		70	46
		110	54
Diallyl	Isooctane	-22	18
		40	
		70	28
		110	34
Methyl Isopropyl Ketone Blend	Isooctane	-22	8
		40	
		70	24
		110	32
Methyl tert-Butyl Ketone	Isooctane	-22	20
		40	22
		70	24
		110	27

Table I (Continued)

Part III. (c) Solubility in Aliphatic Hydrocarbon

Gum	Solvent	Temperature, °F.	Solubility Mg. per 100 cc
Methyl Isopropyl Ketone	Isooctane	-22	4
		40	
		70	5
		110	7
Acetonylacetone	Isooctane	-22	11
		40	12
		70	16
		110	18
Cyclohexanone	Isooctane	-22	0
		40	0
		70	0
		110	0

Part III. (c) Solubility in a Mixture of Hydrocarbons

			Experimental	Calculated
Tetralin	Blend*	-22	38	112.6
		40	48	239
		70	52	370
		110	58	392
Pinene	Blend	-22	198	184
		40		
		70		
		110	232	225
Limonene	Blend	-22	276	220
		40	302	229
		70	326	242
			338	257

* See Part I.

Table I (Continued)

Part III. (d) Solubility in a Mixture of Hydrocarbons

Gum	Solvent	Temperature, °F.	Solubility Mg. per 100 cc	
			Experimental	Calculated
Diallyl	Blend*	-22	40	43.9
		40		
		70	42	51.2
		110	44	55.4
Methyl Isopropyl Ketone Blend	Blend	-22	26	21.2
		40		
		70		
		110	46	69.6
Methyl tert-Butyl Ketone	Blend	-22	114	62.2
		40	115	68.8
		70	116	83.9
		110	118	103.0
Methyl Isopropyl Ketone	Blend	-22	20	39.6
		40		
		70	32	
		110	39	42.5
Acetonylacetone	Blend	-22	4	25
		40	10	26.4
		70	10	28.6
		110	14	30.3
Cyclohexanone	Blend	-22	2	17.2
		40	3	21.5
			6	24
			12	28

* See Part I.

(a) Discussion of Results

17. The nine gums whose solubilities were studied in this investigation were derivates of four hydrocarbons, four ketones, and one ketone-isooctane blend. The structures of the parent hydrocarbons of these resins and of the solvents used in this study are given along with the complete results of the solubility determinations in Table I. The discussion of the comparative solubilities and the relationships between solute and solvent is based, therefore, on the data as listed in this table.

18. The gums were most soluble in the aromatic hydrocarbon, xylene. The solubility in this compound was so much greater than in the others that in the blend, which contained 25% xylene by volume, most of the gums dissolved more readily than in either methyl cyclohexane or isooctane. The solids, with the exception of the one derived from acetonylacetone, exhibited a greater solubility in the hydroaromatic (methyl cyclohexane) than in the aliphatic hydrocarbon (isooctane) at 110°F. At the lowest temperature, -22°F, pinene as well as acetonylacetone gum was more soluble in the latter than in the former liquid.

19. Since a closer relationship exists between the structures of tetralin and xylene than between any of the other compounds and this solvent, the gum formed from this hydroaromatic hydrocarbon was much more soluble, especially at the higher temperatures, than the other gums. Limonene and pinene gums, the parent substances of which are cyclic but more saturated than the aromatic solvent, dissolved more readily than those gums formed from compounds unrelated in structure to xylene. The comparative solubilities of the gums in xylene are: tetralin > limonene > pinene > methyl tert-butyl ketone > methyl isopropyl ketone blend > diallyl > cyclohexanone > acetonylacetone.

20. In the hydroaromatic solvent, methyl cyclohexane, the gums derived from cyclic hydrocarbons, limonene and pinene, were most soluble. Although methyl tert-butyl ketone differs in structure from the solvent, the gum formed from it dissolved more readily than some of the gums with structures more related to methyl cyclohexane. The comparative solubilities of the gums in methyl cyclohexane are: limonene > pinene > methyl tert-butyl ketone > tetralin > diallyl > methyl isopropyl ketone > methyl isopropyl ketone blend > acetonylacetone > cyclohexanone.

21. The hydrocarbon gums, the ketone-isooctane blend included, were more soluble in isooctane than the ketone gums. The resins derived from the cyclic hydrocarbons dissolved more readily than diallyl gum, although diallyl itself is more similar in structure to the aliphatic solvent. The comparative solubilities of the gums in isooctane are: pinene > limonene > diallyl > methyl isopropyl ketone blend > tetralin > methyl tert-butyl ketone > acetonylacetone > methyl isopropyl ketone. Cyclohexanone gum was completely insoluble in this solvent.

22. The experimental values obtained for the solubilities of the gums in the blend were, in many cases, much different from the ones calculated on the basis of the solubilities of the solids in the pure

hydrocarbons. Due to the decreased solubility of tetralin gum in xylene in the presence of the other solvents, this gum was precipitated from a solution in xylene by the addition of methyl cyclohexane and isooctane. On the other hand, whereas limonene and methyl tert-butyl ketone gums enjoyed a greater solubility in the mixture than would be expected from their behavior in the pure compounds, the calculated values for acetonylacetone and cyclohexanone were greater than the experimental ones. In some cases, the solubilities of pinene, diallyl, methyl isopropyl ketone, and methyl isopropyl ketone blend gums agreed with their behavior in the pure compounds; i.e., their solubilities were additive on the basis of the values obtained in the hydrocarbons themselves. The comparative solubilities of the gums in the blend are: limonene> pinene> methyl tert-butyl ketone> tetralin> diallyl> methyl isopropyl ketone blend> methyl isopropyl ketone> acetonylacetone> cyclohexanone.

23. The changes of the solubility of the gums with temperature are shown by Plates 1 to 5. In xylene, tetralin, methyl isopropyl ketone blend, and methyl tert-butyl ketone gums exhibited the greatest decreases in solubilities with lowered temperatures. From Plate 1 it can be seen that a maximum solubility of tetralin gum in xylene was reached, for all practical purposes, at 70°F, for at the temperature higher than this only a comparatively slight increase in solubility occurred. The amounts of diallyl, acetonylacetone, and methyl isopropyl ketone gums which dissolved with an increase of temperature were very small. The behaviors of the other resins in this solvent were intermediate between the two types already mentioned.

24. The gums derived from the cyclic hydrocarbons, limonene, pinene, and tetralin showed the greatest decreases in solubilities in methyl cyclohexane with lowered temperatures, respectively. Although the amounts of the other gums in this hydrocarbon varied with temperature, the changes were slight as can be seen from Plate 2.

25. In isooctane most of the gums derived from the hydrocarbons, the blend included, showed the greatest decreases in solubilities with decreased temperatures. Plate 3 shows the changes of the solubility of the solutes in the aliphatic solvent with temperature.

26. The greatest differences in the solubilities in the blend at different temperatures were shown by tetralin, limonene, and methyl isopropyl ketone and its blend. The solubility of the other resins varied too, but the changes were gradual and less pronounced than the ones mentioned above. Plates 4 and 5 show the changes of the solubility of the gums in the blend with temperature on the basis of the experimental and calculated values.

CONCLUSIONS

27. The solubility of nine gums derived from pure compounds of known structures, have been determined for various temperatures in three pure hydrocarbons and in a blend of these compounds.

28. The comparative order for the efficiency of the solvents was: xylene, the blend, methyl cyclohexane, and isooctane.

29. In general, the gums derived from the cyclic hydrocarbons, tetralin, limonene, and pinene, and also methyl tert-butyl ketone gum, exhibited the greatest solubilities, especially at the higher temperatures. Two ketone gums, acetonylacetone and cyclohexanone, were the least soluble.

30. The experimental values for the solubility of most of the gums in the blend of the three solvents did not agree with the ones calculated on the basis of the solubilities in the pure compounds.

31. Decreases in temperature did not necessarily mean decreased solubility, for although some of the gums exhibited great differences in solubility at different temperatures, the solubility of many of the gums was affected only slightly.

32. Extending the findings of this work on gums of known structures and pure component hydrocarbons of aviation gasoline to the behavior of aviation gasoline itself, the following conclusions seem admissible:

(a) In general, the solubility of gum in gasoline increases as the temperature is raised and decreases as the temperature falls though, as has been indicated above, this is not always the case. Nonetheless, warm gasoline can carry more gum into the motor than cold gasoline and warm, gum-bearing gasoline, when cooled during high altitude flight, tends to deposit a portion of the gum load directly in the fuel tank and supply lines.

(b) The solubility of gasoline gum varies with the type of gasoline, aromatic fuels exhibiting a considerably greater solvent power for gums than fuels predominantly aliphatic in character. Therefore, aromatic fuels are subject to greater contamination when placed in dirty storage tanks or contacted with predeposited gum. When the source of gum is in the fuel itself, that is, gum formed from within the fuel during storage, aromatic gasolines are capable of retaining more of this gum solution than aliphatic or naphthenic gasolines and hence carry a heavier gum load to the engine.

RECOMMENDATIONS

33. On the basis of this investigation, it is recommended:

(a) That this study be extended to include gums formed from other pure compounds such as are given in Naval Research Laboratory Report No. P-1527.

(b) That this work be expanded to include studies of natural and artificial gums from aviation gasoline and that the solubility of such gums in the several current grades of A&N specification fuels be investigated.

(c) That a study be made of the solubility behavior of gums derived from hydrogen boiling above aviation gasoline in the range of the "safety" fuels.

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6. International Critical Tables, 1st ed.

APPENDIX A

PROCEDURE FOR DETERMINING SOLUBILITY OF GUMS STUDIED

1. Many of the gums collected, as described in the body of the report, were semi-solids and as such could not be used for solubility determinations. These semi-solids were converted to solids by dissolving the gums in acetone, evaporating the acetone on a steam bath in a current of air, and if necessary, placing the gums in an oven at 110° - 120°C for 2 to 3 hours. The gums were then scraped from the beakers and ground to fine powders. In this condition they were used in this study.

Description of Procedure[2]

2. The solubility of a gum in a hydrocarbon was based on the formation of a saturated solution and then the analysis of the liquid phase for solid material. The production of a saturated solution was carried out in an eight inch test tube equipped with a glass screw stirrer operated by an electric motor. A small quantity of the gum was added to the solvent in a tube immersed in a constant temperature bath until some of the solid remained undissolved. After the solution was stirred for 3 to 4 hours, the stirrer was removed from the tube and the latter closed with a cork, the solubility tube meanwhile kept in the thermostat.

3. After the solid had subsided, five cc of the solution were transferred to a tared weighing bottle by means of a pipette, the end of which was securely wrapped with filter paper to guard against the entrance of solid particles. The amount of solid in solution was determined by evaporation in an oven at 110° - 120°C and bringing the residue to constant weight.

4. To the solution a further quantity of the gum was added and stirring continued for another period of 1 to 2 hours. The composition of the solution was determined again. When two consecutive determinations showed that the concentration of the solution was constant, these results were averaged and expressed in milligrams of gum per 100 cc of solvent. The volume occupied by the gum was negligible in comparison to that of the hydrocarbon.

APPENDIX B

METHOD OF PURIFICATION OF SOLVENTS AND ANALYSES OF GUMS USED IN THE STUDY

1. Presented in this section is a description of the method of purification of the hydrocarbon solvents, the physical constants of these compounds as determined experimentally and compared to the values as reported in the literature, and the results of the analyses of some of the gums used in this study as also given in the literature.

Purification of Materials

2. Xylene, methyl cyclohexane, and isooctane were purified by the following treatment. Two gallon samples of the hydrocarbons were stirred vigorously over a period of 6 to 8 hours with fuming sulfuric acid. The hydrocarbon layers were washed successively with a 40% sodium hydroxide solution and then twice with water. The resulting materials, which in the cases of isooctane and methyl cyclohexane gave negative tests for unsaturation, were dried first over calcium chloride and then over sodium. The hydrocarbons were fractionally distilled in a three and one-half foot silvered vacuum-jacketed column. The boiling points and the refractive indices are listed in Table A.

3. Table B contains the results of the gum analyses as given in the literature.

Table A

Physical Constants of Hydrocarbons

	Boiling Point; 76 0mm/corr.		Refractive Index, d line	
Name	Determined	Value from Literature	Determined at 20°C	Value from Literature
Isooctane	99.5-100.2	99.3 (3) *	1.3911	1.3916 (3) * at 20°C
Methyl cyclohexane	100.3-100.4	100.8 (4)	1.4230	1.4235 (4) at 20°C
		101.0 (5)		1.4254 (5) at 20°C
Xylene **	138.7-141.0		1.4951	
ortho		143.66 (6)		1.50777 (6) at 15.5°C
meta		139.00 (6)		1.49962 (6) at 14.8°C
para		138.30 (6)		1.49734 (6) at 16.2°C

* Literature reference; see Bibliography.

** Xylene consisted of mixture of ortho, meta, and para isomers.

Table B

Gum Analyses[1]

Gum	M.P., °C	% C.	% H.	% O	Molecular Weight	Hydro-carbon Units	Oxygen Atoms
Pinene	85-90	71.92	9.31	18.77	504	3	6
Limonene	78-88	71.12	8.67	20.21	693	4	8
Cyclohexene		64.23	9.17	26.60	304	3	6
Tetralin		74.70	6.70	18.60	413	3	6
Diallyl	133-142				302		

(1) Literature reference; see Bibliography.

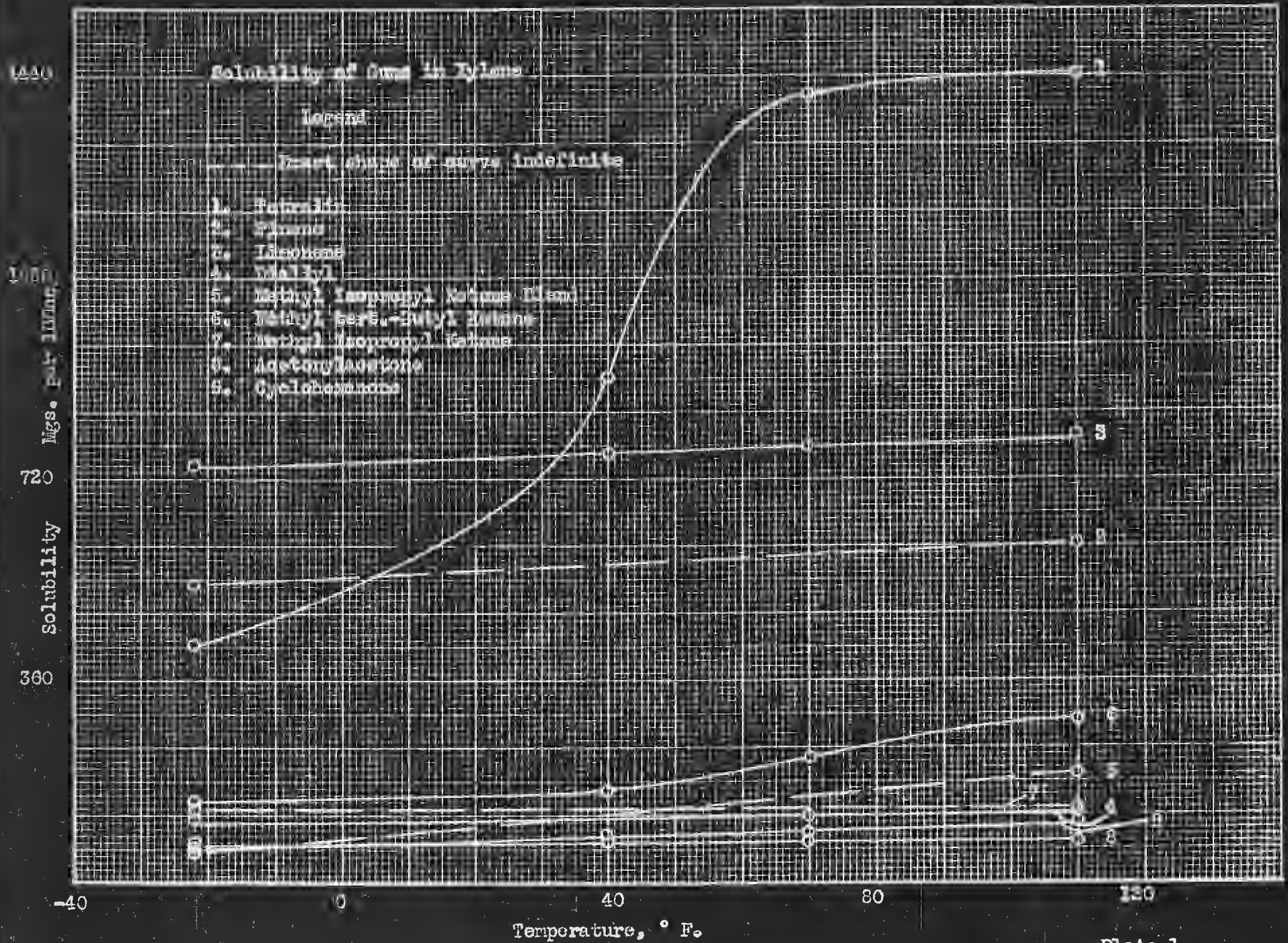
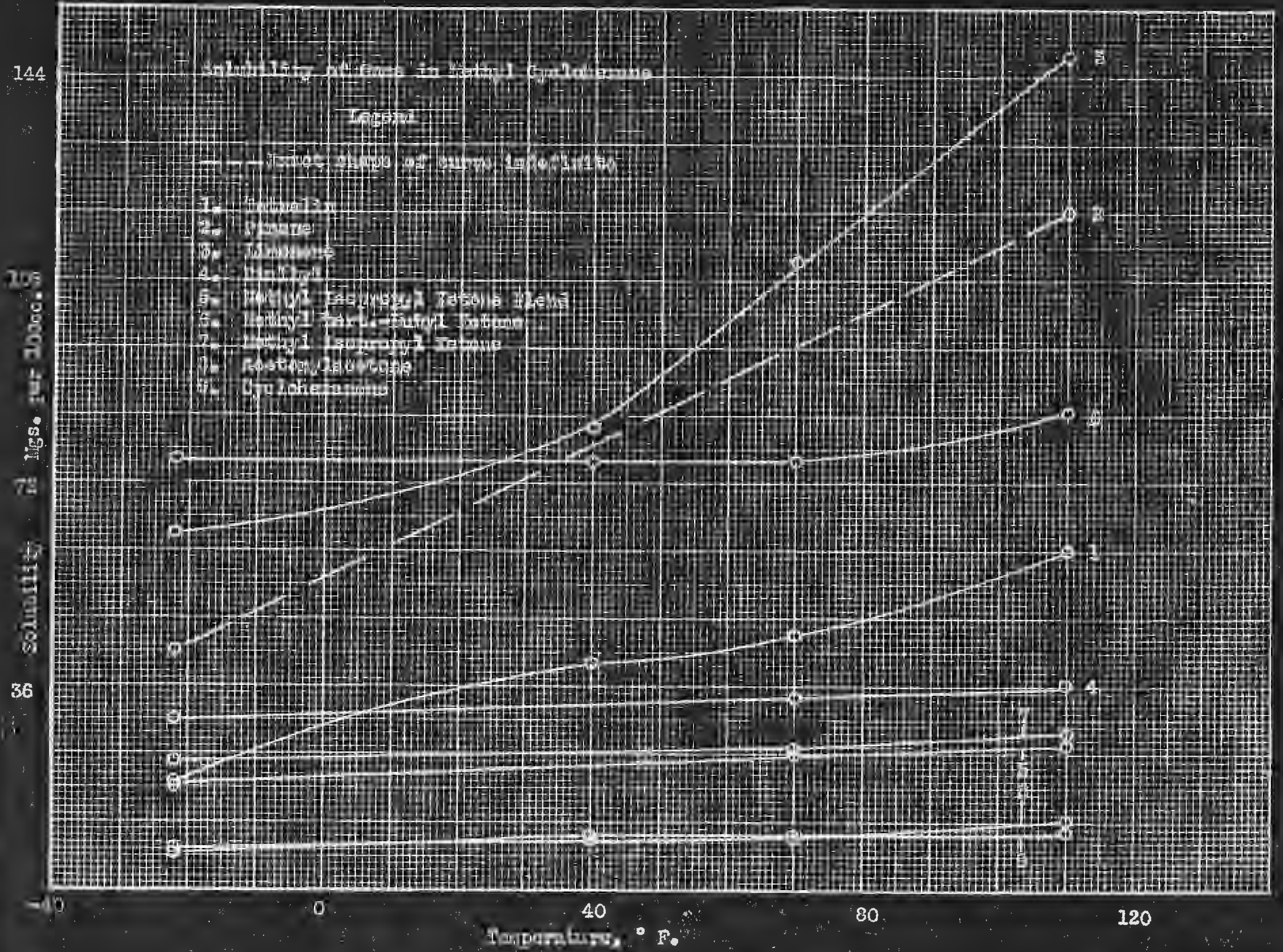

Solubility of Gums in Xylene
Legend
— — Exact shape of curve indefinite
1. [illegible]
2. [illegible]
3. Isophorone
4. [illegible]
5. Methyl Isobutyl Ketone (Hexone)
6. Methyl tert.-Butyl Ketone
7. Methyl Isopropyl Ketone
8. Acetonylacetone
9. Cyclohexanone
1440
1080
720
360
Solubility Mgs. per 100ml
-40
0
40
80
120
Temperature, ° F.
1
3
2
6
5
7
4
9
8

Plate 1

Solubility of Gases in Methyl Cyclohexane
Legend
— — — Exact shape of curve indefinite
1. Tetralin
2. [illegible]
3. [illegible]
4. [illegible]
5. Methyl Isopropyl Ketone Blend
6. Methyl tert.-Butyl Ketone
7. Methyl Isopropyl Ketone
8. Acetonylacetone
9. Cyclohexanone
144
108
72
36
Solubility Mgs. per 100cc.
-40
0
40
80
120
Temperature, ° F.
5
2
6
1
4
7
8
3
9

DATE

Plate 2

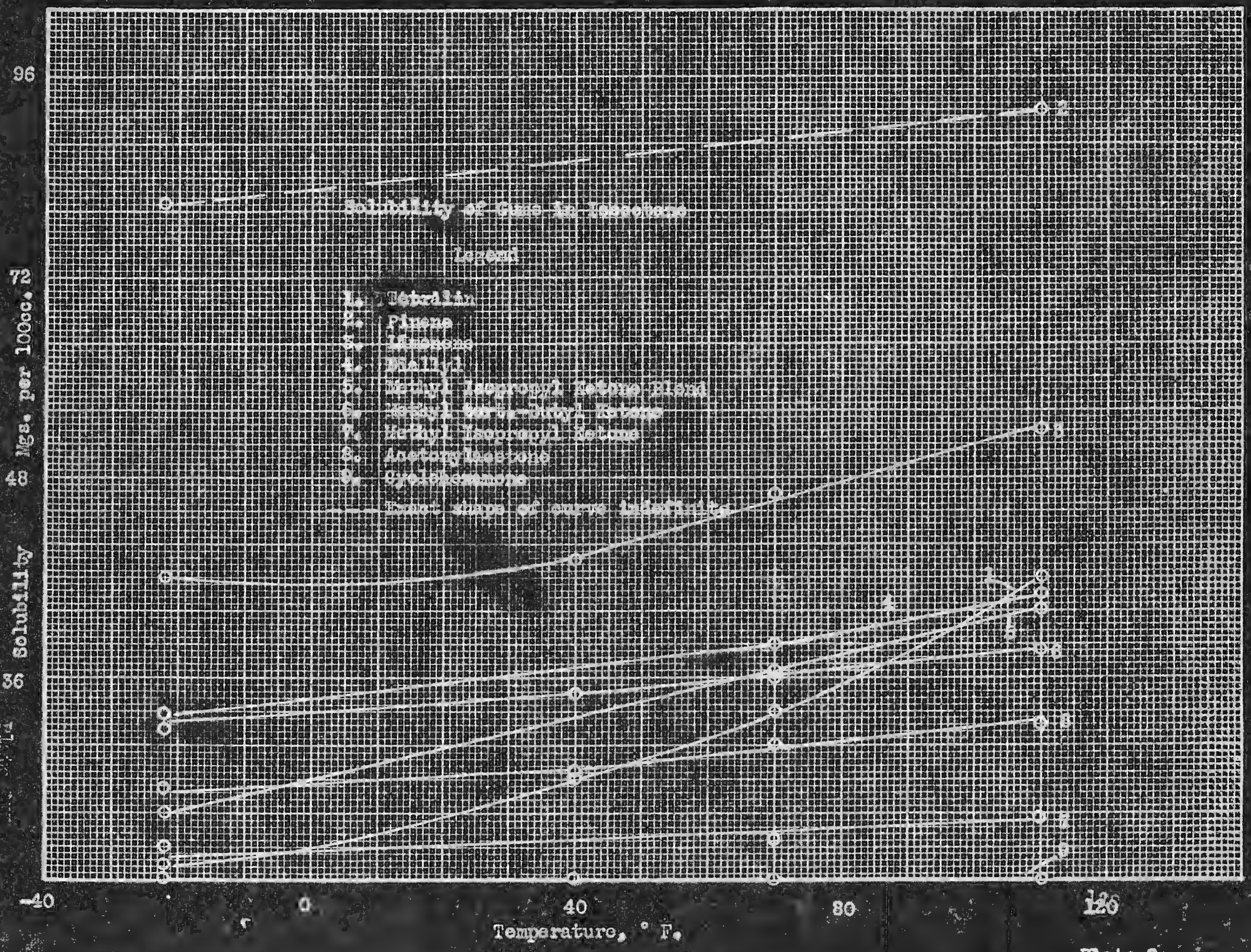

Solubility of Gums in Isooctane
Legend
1. Tetralin
2. Pinene
3. Limonene
4. [illegible]
5. Methyl Isopropyl Ketone Blend
6. Methyl [illegible]-Butyl Ketone
7. Methyl Isopropyl Ketone
8. Acetonylacetone
9. Cyclohexanone
Exact shape of curve indefinite
96
72
48
36
Solubility Mgs. per 100cc.
-40
0
40
80
120
Temperature, ° F.
2
1
4
6
8
7
9

Plate 3

U. S. NAVAL RESEARCH LABORATORY
BELLEVUE, D. C.

DATE

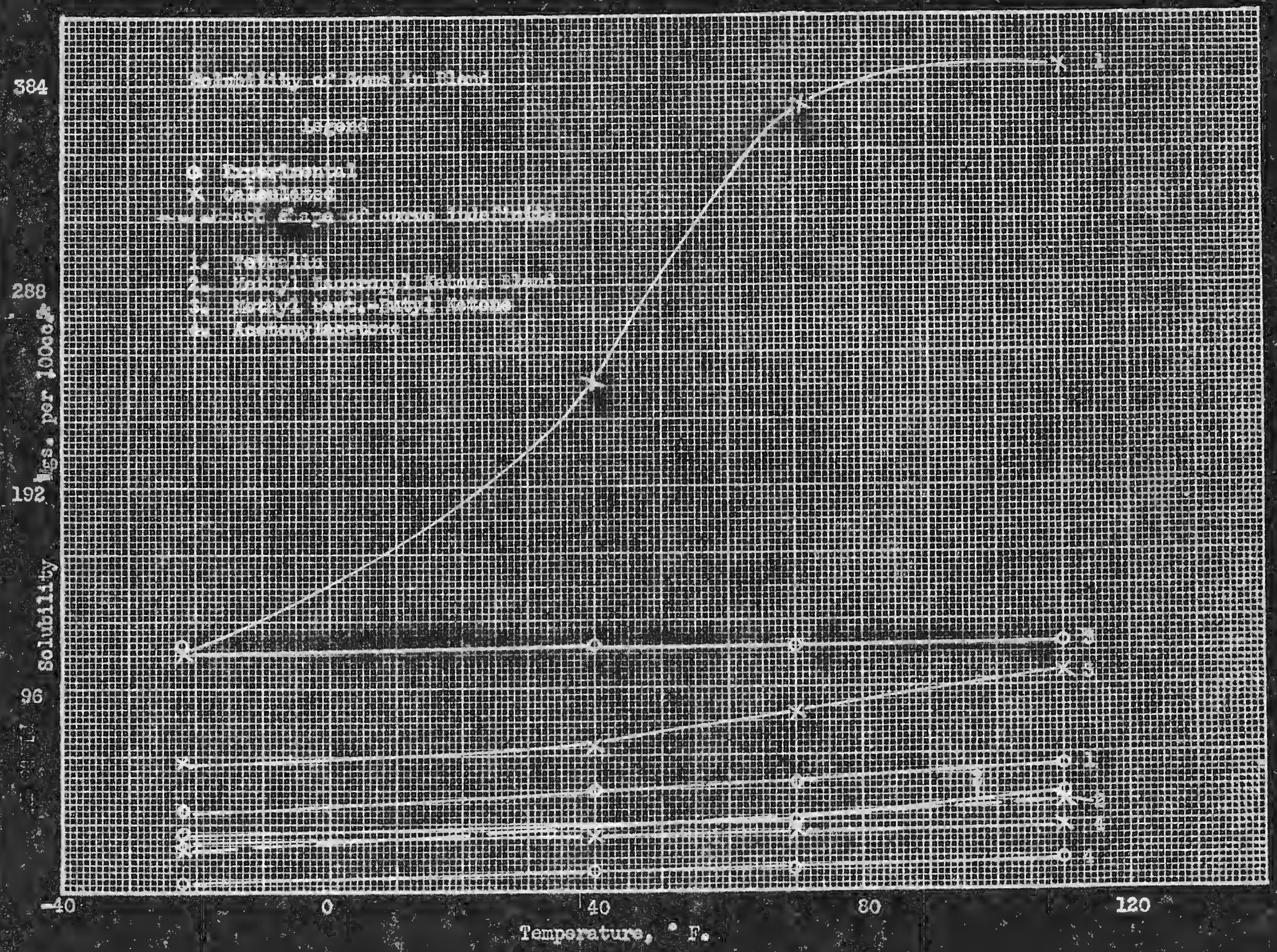


Plate 4

RESEARCH ATORY

DATE

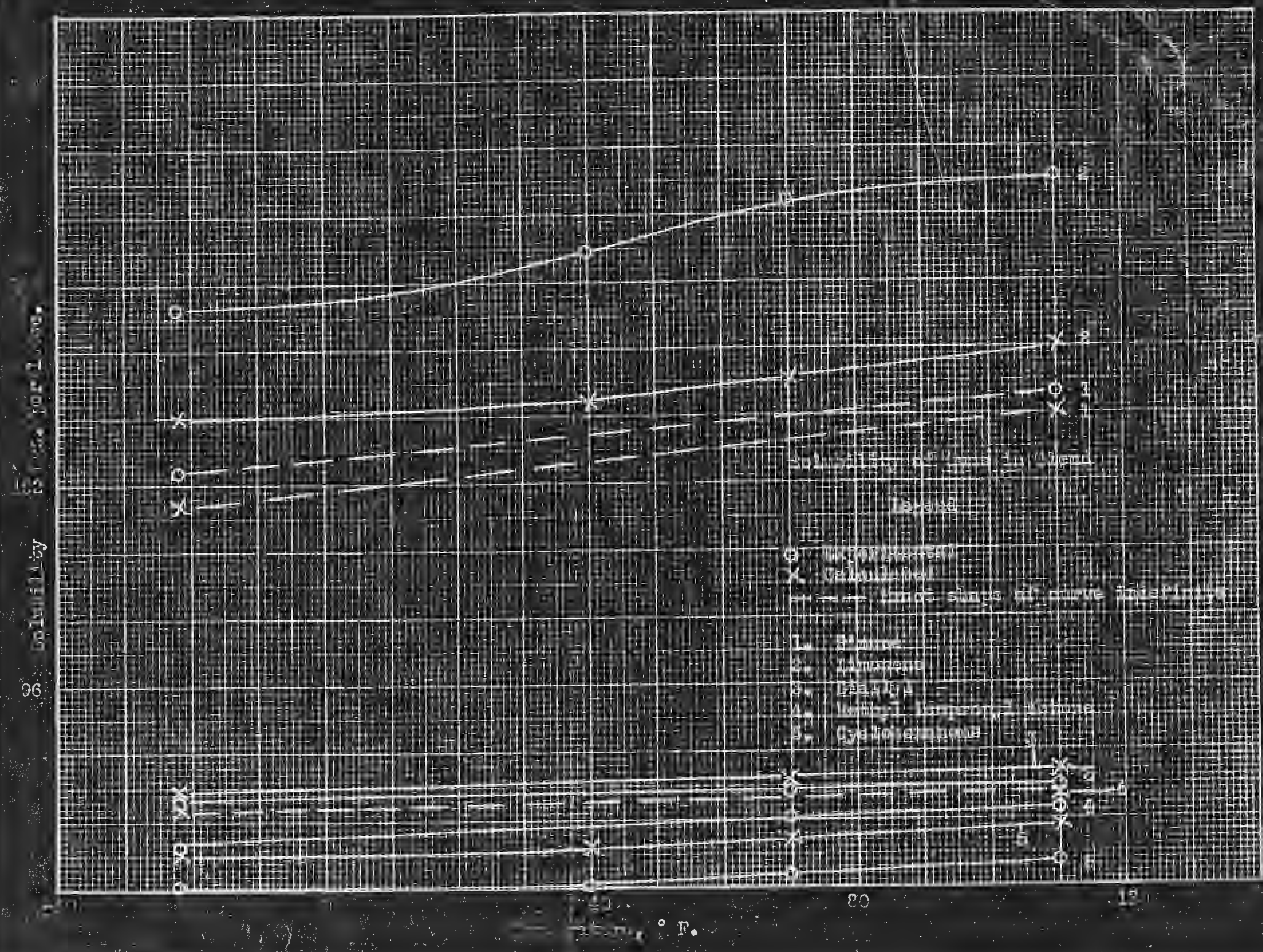


Plate 5