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SYNTHETIC HYDROCARBON LUBRICATING COMPOSITION

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U.S. Cl. 252—59 6 Claims

ABSTRACT OF THE DISCLOSURE

This disclosure concerns a synthetic hydrocarbon lubricating composition, having good viscosity properties and excellent pour point properties, which consists essentially of a mixture of dialkylbenzenes and alkyl-substituted tetrahydronaphthalenes, preferably trialkyl-substituted tetrahydronaphthalenes. An important aspect of the disclosure is the feature that the pour point of dialkylbenzenes can be lowered by incorporating therein alkyl-substituted tetrahydronaphthalenes.

BACKGROUND

Various petroleum fractions have been used as lubricants for many years. While the petroleum-derived lubricants have been satisfactory for most uses, there are fields of use, as for example, jet engine lubricants and arctic oils, wherein the requirements render the conventional petroleum-derived lubricants either unsatisfactory or of marginal utility. In an attempt to solve this problem, synthetic lubricants (for example, diesters) have been developed having improved viscosity and pour point properties. Unfortunately, however, the synthetic lubricants of the prior art have been relatively expensive.

In an effort to provide lower-cost, high-quality synthetic lubricants, work has been directed to synthetic hydrocarbons. For example, Pappas and Kant in U.S. 3,173,965 teach that dialkylbenzenes have properties which render them useful as lubricants. In addition, Becraft and Durr, in U.S. 3,288,176, teach that a bottoms fraction of the product resulting from the condensation of a substantially straight chain paraffinic hydrocarbon, containing in the range of about 8 to about 18 carbon atoms, with an aromatic hydrocarbon, has properties which render it particularly useful as a lubricant.

Applicant has discovered that the pour point of dialkylbenzenes can be lowered by incorporating therein alkyl-substituted tetrahydronaphthalenes, preferably trialkyl-substituted tetrahydronaphthalenes.

PRIOR ART

The most pertinent prior art is believed to be U.S. Pats. 3,173,965 and 3,288,176, referred to above.

With regard to 3,288,176 read in its entirety the patent teaches that tetrahydronaphthalenes are undesirable and contain no recognition that the presence of tetrahydronaphthalenes improves the pour point. Moreover it does not recognize the presence of trialkyl-substituted tetrahydronaphthalenes.

BRIEF SUMMARY OF THE INVENTION

Broadly stated, the present invention relates to a synthetic hydrocarbon lubricating composition consisting essentially of about 1 part dialkylbenzenes and from about 0.25 to about 1.6 parts alkyl-substituted tetrahydronaphthalenes, said composition containing less than 0.10 part of diphenylalkanes per part of dialkylbenzenes and having a pour point of at least —50° F.

In a preferred embodiment of this aspect of my invention, the composition contains an intentionally added

amount, sufficient to lower the pour point of the composition by at least 5° F., of alkyl-substituted tetrahydronaphthalenes. The intentionally added amount is from about 0.05 to about 1 part per part of said dialkylbenzenes.

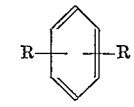
In another aspect the present invention relates to a process for lowering the pour point of asynthetic hydrocarbon lubricating composition containing at least 55 percent, preferably at least 70 percent, by volume, dialkylbenzenes, which comprises incorporating therein from about 0.25 to about 1.6 parts alkyl-substituted tetrahydronaphthalenes per part of dialkylbenzenes.

Preferably, the alkyl-substituted tetrahydronaphthalenes are trialkyl-substituted tetrahydronaphthalenes.

DETAILED DESCRIPTION

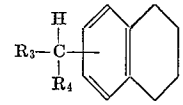
Components of the composition

The major components of the composition of my invention are dialkylbenzenes and alkyl-substituted tetrahydronaphthalenes. The dialkylbenzenes can be represented by the formula



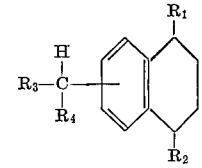
wherein R is a straight-chain alkyl group containing from about 10 to about 18 carbon atoms. The alkyl group is attached to the benzene ring through an internal carbon atom.

The alkyl-substituted tetrahydronaphthalenes can be represented by the formula

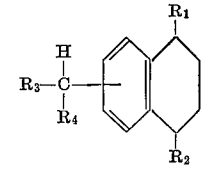


wherein R_3 and R_4 contain from 1 to about 16 carbon atoms each, with the sum of R_3 and R_4 being from about 9 to about 17. Both R_3 and R_4 are straight-chain alkyl groups.

The trialkyl-substituted tetrahydronaphthalenes, which are preferred, can be represented by the formula

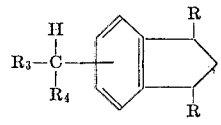


wherein R_1 and R_2 contain from 1 to about 13 carbon atoms each, with the sum of R_1 and R_2 being from about 6 to about 14, and R_3 and R_4 contain from 1 to about 16 carbon atoms, each wtih the sum of R_3 and R_4 being from about 9 to about 17. The alkyl groups, R_1, R_2, R_3 and R_4, are straight chain. For example, a C–11 trialkyl-substituted tetrahydronaphthalene would be represented by the formula



wherein R_1 and R_2 would have a total of 7 carbon atoms with R_1 or R_2 having at least a methyl group, and wherein the sum of R_3 and R_4 is 10. R_3 and R_4 can contain from 1 to 9 carbon atoms each.

The trialkyl-substituted tetrahydronaphthalenes can contain trace amounts of trialkyl-substituted indanes, which are represented by the formula



wherein the R_3—CH—R_4 grouping is the same as in the trialkyl-substituted tetrahydronaphthalenes.

For reason of simplicity, the above-described dialkylbenzenes, alkyl-substituted tetrahydronaphthalenes and trialkyl-substituted tetrahydronaphthalenes are often referred to as DAB, ATHN and TTHN, respectively, hereinafter.

Since mixtures of ATHN and TTHN are suitable in the composition of my invention, the term ATHN is generic and includes mixtures of ATHN and TTHN.

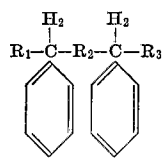
As indicated hereinbefore, addition of the ATHN to the DAB provides a reduction in the pour point of the resulting composition. Plotting the relative amounts of the ATHN and DAB against the pour point usually shows that a small amount of ATHN has a pronounced effect, producing a sharp upward "break" in the curve. Moreover, the curve produces a peak, which may be either sharp or relatively broad, after which a downward break results in the curve, thereby indicating that the ATHN actually increases the pour point. The effectiveness of the ATHN on lowering the pour point of the DAB varies, depending on the particular composition of the ATHN and the DAB.

Suitably, the composition of my invention contains from about 0.25 to about 1.6 parts by volume ATHN per part of DAB. Preferably, the compositions contain from about 0.30 to about 0.80 part by volume ATHN per part of DAB.

In one embodiment, the composition of my invention contains an intentionally added amount, sufficient to lower the pour point at least 5°, preferably at least 10° F., of ATHN. The intentionally added amount is from about 0.05 to about 1 part by volume, preferably from about 0.10 to about 0.80 part by volume ATHN per part DAB.

While DAB and ATHN are the essential materials of the composition of my invention, other materials can be present. Usually, the other materials are by-products of the processess of producing the essential materials.

One of the non-essential materials is diphenylalkanes (DPA). At one time these materials were believed to be beneficial in synthetic hydrocarbon lubricants in that they produced a lower pour point. It now appears that the DPA reduced the pour point by dilution rather than by depression, as is the case with the ATHN and TTHN. These diphenylalkanes are present in an amount of less than 0.10 part by volume, preferably less than 0.05 part by volume, per part of DAB. Typically, the DPA which are present are represented by the formula



where the sum of R_1, R_2 and R_3 is from about 8 to about 16 carbon atoms and R_1 and R_3 is at least a methyl group.

Other materials which can be present, even though they are not desirable, include monoalkylbenzenes and miscellaneous condensed alkyl aromatic compounds. These materials are present in less than about 0.25 part by volume, preferably less than about 0.10 part by volume, per part of DAB.

Properties of the compositions

The compositions of my invention have excellent (i.e. very low) pour points, excellent (i.e. very high) viscosity indexes, good flash points and acceptable —40° F. viscosities. For example, the compositions suitably have a pour point of less than —50° F., preferably less than —60° F., and more preferably less than —75° F. In addition, the composition suitably has a viscosity index of at least 90, preferably at least 100 and more preferably at least 110. Moreover, the compositions have a flash point of at least 400° F. and a —40° F. viscosity of less than 20,000 cs.

Preparation of the composition

The following description describes a particularly suitable process for preparing the composition.

The dialkylbenzenes are prepared by disproportionating monalkylbenzenes or by alkylating monoalkylbenzenes with an olefin or a chlorinated paraffin of suitable carbon content. Since disproportionation of monoalkylbenzenes provides a suitable dialkylbenzene product, this process will be described in detail.

A suitable monoalkylbenzene fraction for the disproportionation reaction is prepared by alkylating benzene with a mixture of substantially monohalogenated paraffins, containing 10 to 15 carbon atoms, using a Friedel-Crafts catalyst. One such method is described in detail in U.S. Pat. No. 3,316,294 which is made a part of this disclosure.

Briefly, U.S. 3,316,294 relates to a process of preparing a detergent alkylate, wherein the process comprises the following steps, broadly stated: (a) separating a fraction of substantially straight-chain C_8–C_{18} hydrocarbons from a petroleum distillate substantially free of olefins and containing said straight-chain hydrocarbons together with non-straight chain hydrocarbons, (b) chlorinating said fraction to the extent whereby between abonut 10 and about 35 mole percent of the straight-chain hydrocarbons present are substantially only monochlorinated, and (c) alkylating an aromatic compound, e.g. benzene, with the chlorination product of step (b) in the presence of an alkylation catalyst.

Disproportionation of the mono-n-alkylbenzenes to di-n-alkylbenzenes is conducted using a Friedel-Crafts catalyst. The term Friedel-Crafts catalyst is well understood in the art and refers, generally, to materials such as the aluminum halides, boron trifluoride, boron trichloride, antimony chlorides, stannic chloride, zinc chloride, and mercuric chloride. Preferably, the Friedel-Crafts catalyst is aluminum chloride or aluminum bromide. The more preferred catalyst is aluminum chloride, which also includes "in situ" prepared aluminum chloride, or, in other words, the reaction product of aluminum metal and hydrogen chloride.

In some cases it is desirable to use a proton-donor promoter with the Friedel-Crafts catalyst. Suitable promoters include any material which, when added to the catalyst, yields a proton. Preferred promoters are hydrogen chloride and water. The amount of promoter is typically about 4 weight percent based on the weight of the catalyst employed.

Preferably, the catalyst is added to the reaction mixture after the mono-n-alkylbenzenes are brought to within the correct temperature range, which will be described below. The amount of the catalyst which is used can vary from about 0.1 weight percent to about 10 weight percent based on the mono-n-alkylbenzene starting material. Preferably, the amount of catalyst is from about 0.5 weight percent to about 3 weight percent.

The disproportionation process, suitably, is conducted at a temperature of from about 20° C. to about 130° C. tained at temperatures between about 60° C. and 120° C., used, preferably the amount of catalyst is from about 1 to about 2 weight percent.

Following the reaction, the reaction mass is distilled in order to remove the benzene, paraffins and unreacted mono-n-alkylbenzenes. The desired disproportionation product is the bottoms fraction with a distillation cut point of 197° C. at 5 mm. Hg. In other words the desired product distills above 197° C. at 5 mm. Hg.

While we have described in detail the process conditions for the disproportionation reaction, the disproportionation process is not part of our invention. This process is disclosed and claimed in application Ser. No. 529,284, filed Feb. 23, 1966, having the same assignee as the present application.

The disproportionate product contains at least 55 percent by volume, more usually at least 70 percent by volume, dialkylbenzenes. It has a viscosity index (100–210° F. basis) of from about 105 to about 120 and a pour point of from about —55 to about —70° F. The disproportionate product usually contains a small amount of TTHN. The amount of TTHN varies with the disproportionation conditions and the amount of dialkyl-substituted tetrahydronaphthalenes in the monoalkylbenzene disproportionation charge. By careful selection of disproportionation conditions a disproportionated product can be prepared containing as much as 30% by volume (mass spectral analysis) TTHN. In instances where it is desired to have a product having a pour point of less than about —55° F. and a high flash point (over 450° F.) disproportionation conditions are selected which produce a product containing less than about 20% TTHN. Then additional TTHN is added in an amount sufficient to lower the pour point.

The benefits of my invention (i.e. the effect of ATHN on lowering the pour point of dialkylbenzene compositions) will be readily apparent to those skilled in this art. For example, as indicated above, the emphasis in the preparation of the dialkylbenzenes can be to produce a product which is strong in properties other than pour point. Then, the desired pour point can be attained by simply adding the ATHN or TTHN to the dialkylbenzenes.

The alkyl-substituted tetrahydronaphthalenes are prepared by alkylating tetrahydronaphthalene with an olefin or a chlorinated paraffin having the desired chain length. The trialkyl-substituted tetrahydronaphthalenes are prepared in a similar manner using dialkyl-substituted tetrahydronaphthalenes. The dialkyl-substituted tetrahydronaphthalenes can be obtained by careful fractionation of the product prepared by the process described by U.S. 3,316,294, described hereinbefore. Alternatively, the trialkyl-substituted tetrahydronaphthalenes can be obtained by careful fractionation of the disproportionated product. The first described procedure of preparing the trialkyl-substituted tetrahydronaphthalenes is preferred since in effect the value of the dialkyl-substituted tetrahydronaphthalenes is increased by conversion to the trialkyl-substituted tetrahydronaphthalenes.

The non-essential components of the composition (diphenylalkanes, monoalkylbenzenes, and miscellaneous condensed alkyl aromatic compounds) are by-products of the process of manufacturing the composition, which has been described herein.

In order to disclose the nature of the present invention still more clearly, the following illustrative examples will be given. It is to be understood that the invention is not to be limited to the specific conditions or details set forth in these examples except insofar as such limitations are specified in the appended claims.

EXAMPLE 1

(A) Preparation of the dialkylbenzenes

The dialkylbenzenes were prepared by disproportionation of a topped monoalkylbenzene fraction prepared by the process of U.S. Pat. No. 3,316,294. The monoalkylbenzenes had the following carbon content of the alkyl groups

<C–12—less than 5 percent by volume
C–12—10–30 percent
S–13—30 min. percent
C–14—20 min. percent
>C–14—less than 5 percent

An 80% overhead fraction was then obtained using a cut-point of 182° C. at 5 mm. Hg pressure. This 80% overhead fraction was the material disproportionated.

Prior to disproportionation the charge stock was pretreated with $AlCl_3$ as follows:

1100 grams of alkylate (80% overhead material) was contacted with 11.0 grams of anhydrous $AlCl_3$ at 60° C. for 30 minutes. The reaction mass was allowed to cool and settle for 1½ hours to separate the sludge. There was obtained 1086 grams of "pretreated" alkylate and 25 grams of sludge.

Disproportionation procedure

Charge:

1000 g. pretreated alkylate (described above)
10.0 g. anhydrous $AlCl_3$
0.4 ml. H_2O

Procedure:

The pretreated alkylate was added to a reaction flask. While stirring the admixture, heat was applied until the temperature of the admixture rose to 60° C. At this point the $AlCl_3$ and H_2O were added. The reaction mass was heated to 100–103° C. and held there for one hour. The admixture was then allowed to cool and settle. There was obtained 980.1 grams of crude alkylate and 30.8 grams of a sludge.

The crude alkylate was washed with deionized water,, followed by a wash with a 5% NaOH solution, and then again ashed with water. From this treatment, there was obtained 962 grams of crude alkylate.

The crude alkylate was then subjected to a distillation taking the benzene, paraffin, and monoalkylbenzene fractions overhead. The cut-point for the monoalkylbenzene-bottoms fraction was 197° C. at 5 mm. Hg.

The bottoms fraction (which was the dialkylbenzene fraction) was then treated with Filtrol clay. The yield of treated bottoms fraction was 235.9 grams.

The properties of the bottoms framtion (DAB) are shown in Table I.

(B) Preparation of trialkyl-substituted tetrahydronaphthalenes (TTHN)

The TTHN was prepared by alkylating a dialkyl-substituted tetrahydronaphthalene (DTHN) with dodecene-1. The DTHN was a fraction obtained from the product prepared by U.S. Pat. No. 3,316,294. Mass spectrometer analysis of the starting material indicated the composition to be as follows:

	Volume percent
C_{14} tetrahydronaphthalenes	89
C_{15} tetrahydronaphthalenes	2
$C_{14,15}$ alkylbenzene	5
Naphthalene	2
Other	2

The properties of the product (TTHN) are shown in Table I.

(C) Blends of trialkyl-substituted tetrahydronaphthalenes and dialkylbenzenes

In order to determine the effect on pour point, blends of TTHN and DAB were prepared, using the above-described fractions. The blends contained, 5, 10 and 20 weight percent TTHN fraction, with the remainder being DAB fractions.

The pour points of the 100 percent DAB fraction, 100 percent TTHN fraction and the various blends are shown below:

	Pour point, ° F.
100% DAB fraction	−60
100% TTHN fraction	−55
Blend A	−65
95% DAB fraction	
5% TTHN fraction	
Blend B	−65
90% DAB fraction	
10% TTHN fraction	
Blend C	−55
80% DAB fraction	
20% TTHN fraction	

The physical properties and complete data for the various blends are shown in Table I.

TABLE I

	100% DAB fraction	100% TTHN fraction	Blend A	Blend B	Blend C
TTHN fraction, wt. percent			5.0	10.0	20.0
DAB fraction, wt. percent			95.0	90.0	80.0
Viscosity, cps. at—					
−40° F.	9,526	95,759	10,794	11,500	13,308
100° F.	30.85	67.72	32.02	33.02	35.54
210° F.	5.35	8.00	5.38	5.48	5.69
V.I.	119	91	113	114	110
Pour point, ° F.	−60	−55	−65	−65	−55
Mass spectraanalysis, vol, percent:					
DAB	78.0	5.7	74.4	70.8	63.5
TTHN	16.0	68.3	19.4	21.9	27.0
DPA	0.5	10.0	1.0	1.5	2.4
Indenes	2.0	12.4	2.5	3.0	4.1
Misc. compounds	2.7	3.6	2.8	2.8	2.9
Ratio DAB/TTHN	1/0.21	1/12.0	1/0.26	1/0.31	1/0.43

DAB fraction, 100 percent TTHN fraction and the various blends are shown below:

	Pour point, ° F.
100% DAB fraction	−45
100% TTHN fraction	−55
Blend A	−45
95% DAB fraction	
5% TTHN fraction	
Blend B	−45
90% DAB fraction	
10% TTHN fraction	
Blend C	−45
85% DAB fraction	
15% TTHN fraction	
Blend D	−45
80% DAB fraction	
20% TTHN fraction	
Blend E	−60
75% DAB fraction	
25% TTHN fraction	
Blend F	−60
70% DAB fraction	
30%TTHN fraction	
Blend G	−55
65% DAB fraction	
35% TTHN fraction	

The physical properties and complete data for the various blends are shown in Table II.

TABLE II

	100% DAB fraction	Blend A	Blend B	Blend C	Blend D	Blend E	Blend F	Blend G
TTHN fraction, wt. percent		5.0	10.0	15.0	20.0	25.0	30.0	35.0
DAB fraction, wt. percent		95.0	90.0	85.0	80.0	75.0	70.0	65.0
Viscosity, cps. at—								
−40° F.	7,884							
100° F.	26.40							
210° F.	4.92							
V.I.	122							
Pour point, ° F.	−45	−45	−45	−45	−45	−60	−60	−55
Mass spectral analysis, vol. percent:	(2)		(1)		(1)	(1)	(1)	(1)
DAB	96.1		87.5		78.0	73.5	69.0	64.5
TTHN	3.7		10.1		16.7	19.8	23.0	26.3
DPA			1.0		2.0	2.5	3.0	3.5
Indenes			1.2		2.5	3.1	3.7	4.3
Misc. compounds	0.3		0.7		0.9	1.1	1.3	1.5
Ratio DBA/TTHN	1/0.04		1/0.11		1/0.21	1/0.21	1/0.33	1/0.41

[1] Calculated from 100 percent DAB fraction and 100 percent TTHN fraction.
[2] Contains 13.4 percent trialkylbenzene, no mono.

EXAMPLE 2

The dialkylbenzenes used in this example were prepared by disproportionation, using the same procedure as in Example 1. The material subjected to disproportionation differed slightly from that in Example 1. In this example the charge alkylate was the monalkylbenzene fraction without any overhead distillation (in other words, the monoalkylbenzene fraction per se was used).

The properties of the DAB fraction (bottoms) are shown in Table II following.

The TTHN fraction used in this example was the same as in Example 1.

In order to determine the effect on pour point, blends of TTHN fraction and the DAB fraction of this example were prepared. The blends contained 5, 10, 15, 20, 25, 30 and 35 weight percent TTHN fraction with the remainder being DAB fraction. The pour points of the 100 percent

EXAMPLE 3

This example shows the effect on pour point of blending a C_{13} TTHN with a C_{14} DAB. (By C_{13} TTHN is meant one wherein the grouping $R_3—CH—R_4$ has a total of 13 carbon atoms.)

The C_{13} TTHN was prepared by alkylating dialkyltetrahydronaphthalene with a C_{13} olefin.

The C_{14} DAB was prepared by disproportionation of pure tetradecylbenzene.

The analyses on the C_{13} TTHN and C_{14} DAB are shown in Table IV.

Various blends were prepared of the two materials. The effect on pour point and other pertinent information on these blends are shown in Table III.

EXAMPLE 4

This example shows the effect on pour point of blending a C_{13} TTHN or a C_{11} TTHN with a C_{12} DAB.

The C_{13} TTHN was the same as in Example 3.

The C_{11} TTHN was prepared by alkylating dialkyltetrahydronaphthalene with a C_{11} chlorinated paraffin.

The C_{12} DAB was prepared by alkylating a narrow cut C_{11}–C_{13} monoalkylbenzene with a C_{11}–C_{13} chlorinated n-paraffin. (Both materials had a predominance of C_{12}.)

The analyses on the C_{13} TTHN, C_{11} TTHN, and C_{12} DAB are shown in Table IV.

Various blends were prepared of the materials. The effect on pour point and other pertinent information on these blends are shown in Table III.

EXAMPLE 5

This examples shows the effect of blending a C_{11} TTHN with a C_{13} DAB.

The C_{11} TTHN was the same as in Example 4.

The C_{13} DAB was prepared by first alkylating benzene with a chlorinated C_{13} n-paraffin to produce a C_{13}-monoalkylbenzene. The latter material was then disproportionated to prepare the C_{13} DAB.

The analyses on the C_{11} TTHN and C_{13} DAB are shown in Table IV.

Various blends were prepared of the two materials. The effect on pour point and other pertinent information are shown in Table III.

of −75° F., which is 5° lower than that for 100% DAB Fraction and 25° lower than that for 100% TTHN Fraction. Also, in Example 4 compositions containing 0.69 and 1.32 parts C_{11} TTHN per part of C_{12} DAB had a pour point of −75° F., which is 5° lower than that for 100% DAB Fraction and 15° lower than that for 100% TTHN Fraction.

In Example 5 compositions containing 0.75 and 1.40 parts TTHN per part of DAB had a pour point of −70° F., which is 20° lower than that for 100% DAB Fraction and 10° lower than that for 100% TTHN Fraction.

Since addition of an optimum amount of TTHN to the DAB produces a composition having a lower pour point than either alone, and particularly lower than the TTHN alone, it appears that synergistic effect is present.

What is considered new and inventive in the present invention is defined in the hereunto appended claims, it being understood, of course, that equivalents known to those skilled in the art are to be construed as within the scope of the invention.

What is claimed is:

1. A synthetic hydrocarbon lubricating composition consisting essentially of about 1 part by volume dialkylbenzenes, the alkyl groups of which are straight-chain and contain from about 10 to about 16 carbon atoms each and an added amount, sufficient to lower the pour point of said dialkylbenzenes by at least 5° F., of alkyl-substituted tetrahydronaphthalenes, the total amount of said alkyl-substituted tetrahydronaphthalenes being from about 0.25 to about 1.6 parts by volume per part of said dialkylbenzenes, the added amount being from about 0.05 to about 1 part by volume per part of said dialkylbenzenes, said alkyl-substituted tetrahydronaphthalenes being represented by the formula

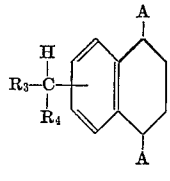


TABLE III

Example No.	DAB [1]	TTHN [1]	Wt. percent TTHN [1] in blend	Pour point, ° F.	Mass spectral analyses, vol. percent [2] DAB	TTHN	DPA	Misc.	Ratio DAB/TTHN	DPA/Misc.
3	C_{14}	C_{13}	10.0	−35						
			20.0	−40	80.2	16.3	0.7	2.8	1/0.20	0.01/0.04
			30.0	−45	72.3	23.4	0.6	3.7	1/0.32	0.01/0.05
			40.0	−55	64.3	30.6	0.6	4.5	1/0.48	0.01/0.07
			45.0	−50	60.3	34.1	0.6	5.0	1/0.57	0.01/0.08
4	C_{12}	C_{13}	10.0	−75	54.4	35.1	0.5	10.0	1/0.64	0.01/0.18
			25.0	−75	48.2	41.5	0.5	10.0	1/0.86	0.01/0.21
			35.0	−75	43.9	45.6	0.5	10.0	1/1.01	0.01/0.23
			40.0	−70	41.8	47.7	0.5	10.0	1/1.14	0.01/0.24
		C_{11}	10.0	−75	53.1	36.6	0.5	9.8	1/0.69	0.01/0.19
			25.0	−75						
			35.0	−75	38.8	51.3	0.5	9.4	1/1.32	0.01/0.24
			40.0	−65	35.9	54.3	0.5	9.3	1/1.51	0.01/0.26
5	C_{13}	C_{11}	10.0	−55	68.7	26.3	1.0	4.0	1/0.38	0.02/0.06
			20.0	−65	61.3	33.4	1.0	4.3	1/0.54	0.02/0.07
			30.0	−70	53.8	40.4	0.9	4.9	1/0.75	0.02/0.09
			50.0	−70	38.9	54.5	0.8	5.8	1/1.40	0.02/0.15

[1] Fraction.
[2] Calculated from analysis of DAB and TTHN materials used to prepare blends.

TABLE IV

Example Number	Type material	Alkyl chain	Method of preparation	Pour point, ° F.	Mass spectral analyses, vol. percent DAB	TTHN [a]	DPA	Misc.	Ratio DAB/TTHN	DPA/Misc.
3	DAB fraction	C_{14}	Disproportionation	−40	96.3	2.0	0.7	1.0	1/0.02	0.01/0.01
4	do	C_{12} [b]	Alkylation	−70	58.8	30.7	0.5	10.0	1/0.52	0.01/0.17
3 and 4	TTHN fraction	C_{13}	do	−50	16.2	73.4	0.5	9.9	1/4.5	0.03/0.61
5	DAB fraction	C_{13}	Disproportionation	−50	76.2	19.3	1.1	3.4	1/0.25	0.01/0.05
5	TTHN fraction	C_{11}	Alkylation	−60	1.6	89.6	0.5	8.3	1/56	0.3/5.2

[a] Includes trace amounts of indanes.
[b] Average.

A study of the data presented in the foregoing tables indicates the following:

In Example 1 compositions containing 0.26 and 0.31 part TTHN per part DAB had a pour point of −65° F., which is 5° lower than that for 100% DAB Fraction and 10° lower than that for 100% TTHN Fraction.

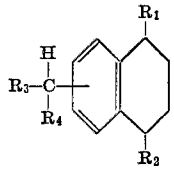
In Example 2 compositions containing 0.27 and 0.33 part TTHN per part DAB had a pour point of −60° F., which is 15° lower than that for 100% DAB Fraction and 5° lower than that for 100% TTHN Fraction.

In Example 3 a composition containing 0.48 part TTHN per part DAB had a pour point of −55° F., which is 15° lower than that for 100% DAB Fraction and 5° lower than that for TTHN Fraction.

In Example 4 compositions containing 0.64, 0.86, and 1.01 part C_{13} TTHN per part C_{12} DAB had a pour point

wherein A is hydrogen or a straight-chain alkyl group containing from 1 to about 13 carbon atoms, characterized in that when both A substituents are alkyl groups the total number of carbon atoms in both of the alkyl groups is from about 6 to about 14 and wherein R_3 and R_4 contain from 1 to about 16 carbon atoms each, with the sum of R_3 and R_4 being from about 9 to about 17, said composition containing less than 0.10 part by volume of diphenylalkanes per part of said dialkylbenzenes and having a pour point of below about —50° F.

2. The composition of claim 1 wherein the alkyl-substituted tetrahydronaphthalenes are trialkyl-substituted tetrahydronaphthalenes, which are represented by the formula



wherein R_1 and R_2 are straight chain alkyl groups containing from 1 to about 13 carbon atoms each, with the sum of R_1 and R_2 being from about 6 to about 14, R_3 and R_4 are straight chain alkyl groups containing from 1 to about 16 carbon atoms each, with the sum of R_3 and R_4 being from about 9 to about 17.

3. The composition of claim 2 wherein (1) it contains from about 0.30 to about 0.80 part by volume trialkyl-substituded tetrahydronaphthalenes per part of dialkylbenzenes and (2) has a pour point of at least —60° F.

4. The composition of claim 3 wherein the added amount of trialkyl-substituted tetrahydronaphthalenes is from about 0.10 to about 0.80 part per part of dialkylbenzenes.

5. The composition of claim 4 wherein the added amount of trialkyl-substituted tetrahydronaphthalenes is sufficient to lower the pour point of the dialkylbenzenes by at least 10° F.

6. The composition of claim 5 characterized further in that it has a pour point of at least —75° F.

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PO-1050
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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,598,739 **Dated** August 10, 1971

Inventor(s) Roy C. Sias

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 2, line 6, "asynthetic" should be --a synthetic--.
Column 2, line 56, "wtih" should be --with--.

Column 4, line 39, "abonut" should be --about--.
Column 4, line 72, after 130°C, insert --Since maximum yields of the di-n-alkylbenzenes are ob--.
Column 4, line 73, after 120°C, insert --these temperatures are preferred. The most preferred temperature is about 80°C. When this temperature is--.

Column 6, line 5, "S-13" should be --C-13--.
Column 6, line 38, "ashed" should be --washed--.
Column 6, line 47, "framtion" should be --fraction--.

Table II, under Blend E, Ratio DAB/TTHN, "1/0.21" should be --1/0.27--.

Column 9, line 14, "examples" should be --example--.

Signed and sealed this 31st day of October 1972.

(SEAL)
Attest:

EDWARD M.FLETCHER,JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents