

Multiply-Alkylated Cyclopentanes (MACs): A New Class of Synthesized Hydrocarbon Fluids[®]

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LUBRICATION ENGINEERING

A new class of synthesized hydrocarbon fluids has been prepared and evaluated. Examples within the following ranges of properties have been examined: viscosity (100°C), 2–20 mm²/sec; viscosity (40°C), 6–310 mm²/sec; viscosity index, 20–180; and pour points to less than –60°C. A typical example with molecular weight of 550 has viscosity (100°C) = 6 mm²/sec, viscosity index = 134, and pour point of <–55°C. A special low volatility example has viscosity (100°C) = 14.6 mm²/sec, viscosity index = 137, pour point –57°C, and evaporation loss (ASTM D972, 6.5 hr, 250°C) of less than 0.1 percent.

INTRODUCTION

Polyalphaolefins (PAOs) have become a standard material for the preparation of synthetic lubricant products. Little recent work in lubrication chemistry has dealt with the invention of new synthesized hydrocarbon fluids which are fundamentally different in chemical structure from PAOs.

The authors describe below a new material which can be prepared from dicyclopentadiene by reaction with aliphatic alcohols. Dicyclopentadiene is a commercial product used in the polymer industry which is produced as a by-product in ethylene production, and aliphatic alcohols are standard, relatively inexpensive, commodity chemicals. Properties of these multiply-alkylated cyclopentanes (MACs) can be tailored to specific applications by varying the nature of the alcohol used in the synthesis.

SYNTHETIC METHODOLOGY

The method of preparing multiply-alkylated cyclopentane fluids is simple, and involves novel extensions and improvements (1) on a process first reported by Bailey and Hirsch (2) and also Fritz (3). Yields are virtually quantitative.

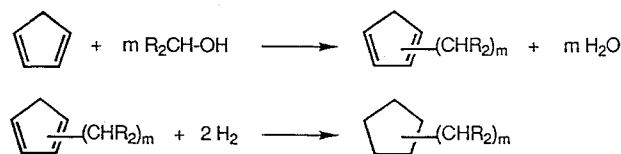
Dicyclopentadiene is cracked to cyclopentadiene and allowed to react with alcohols in the presence of strong base (Scheme 1). The products of this initial step are multiply-alkylated cyclopentadienes, which are then hydrogenated to the desired MACs under normal hydrogenation conditions. Alkylation of cyclopentadiene with alcohols proceeds sequentially and usually yields mixtures containing one or more of di-, tri-, tetra-, or penta-alkylated cyclopentadienes ($m = 2, 3, 4, 5$ in Scheme 1). The distribution is controlled by choice of reaction conditions, and one homolog usually predominates. Some of the examples given in Table 1, particularly those with $m = 2$, were prepared by an alternative procedure (4).

Relevant properties of products were determined by standard tests, which are noted in each section of the presentation of physical properties.

PHYSICAL PROPERTIES

Summaries of the physical properties of MACs are reported below. In this initial report, the authors will concentrate on those physical properties of most interest in the selection of base oils for the construction of a wide variety of lubricants and functional fluids, namely viscosity, the variation of viscosity with temperature, and the melting points (pour points) of the fluids.

In addition, discussions of the dependence of these properties on the specific molecular structures and comparisons of properties of alkylcyclopentanes to those of PAOs are also included. The data presented in Table 1 are representative of the variety of products available and illustrative of typical properties. Figures 1, 2, and 3 contain data from additional experiments.



Scheme 1—Method of preparation of multiply-alkylated cyclopentane synthesized hydrocarbon fluids

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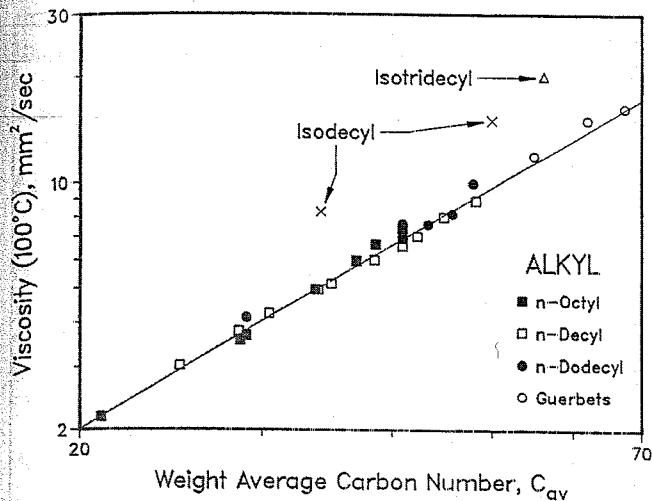


Fig. 1—Dependence of 100°C viscosity on average carbon number for multiply-alkylated cyclopentanes.

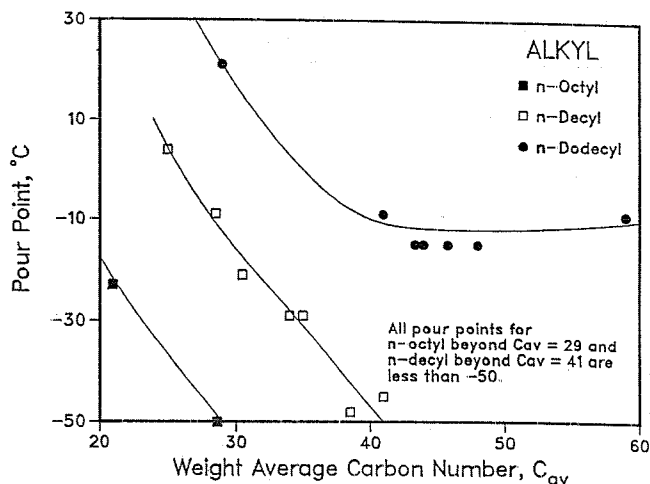


Fig. 3—Dependence of pour point on alkyl group and carbon number for multiply-alkylated cyclopentanes.

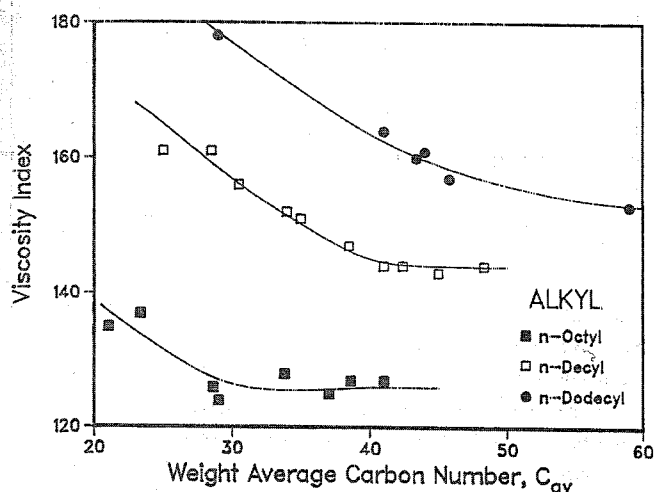


Fig. 2—Dependence of viscosity index on alkyl group and carbon number for multiply-alkylated cyclopentanes.

VISCOSITY (100°C)

The 100°C kinematic viscosities (ASTM D445) of representative examples of MACs are given in Table 1 and Fig. 1. The main structural parameter upon which viscosity depends is the average molecular weight of the material. The average molecular weight is approximated by C_{av} , the weight average number of carbons in a molecule, i.e.

$$C_{av} = \sum_i (\text{weight fraction of } i^{\text{th}} \text{ component}) \times (\text{carbon number of } i^{\text{th}} \text{ component})$$

Both the weight fraction and the carbon number can be obtained from gas chromatography (GC). For example, the GC of sample No. 9 in Table 1 shows only two groups of peaks at retention times corresponding approximately to 35 carbons (tri-*n*-decyl) and 45 carbons (tetra-*n*-decyl), respectively. The relative areas are 3 and 2, respectively. Hence, $C_{av} = 0.6 \times 35 + 0.4 \times 45 = 41$. A regression line relating $\log(\text{vis})$ to $\log(C_{av})$ for *n*-alkyl substituents is shown in Fig. 1. Thus, for *n*-alkyl MACs, the kinematic viscosity at 100°C can be readily predicted. The equation is $\log(\text{vis}) = -1.92 + 1.71 \log(C_{av})$. The analogous equation for *n*-alkanes with $C_{av} > 20$ is $\log(\text{vis}) = -2.36 + 2.03 \log(C_{av})$.

Highly branched alcohols, such as the Oxo alcohols, isodecanol (which is predominately isomeric trimethylheptanols) and isotridecanol (which is predominately isomeric tetramethylnonanols), give more viscous products than would be predicted from the regression line for the *n*-alkyl MACs. Note that the points for the products prepared from these alcohols are above the line in Fig. 1.

In contrast, multiply-alkylated cyclopentanes having substituents with a single branch, e.g., MACs derived from the so-called Guerbet alcohols, 2-octyldodecanol and 2-decyltetradecanol, seem to fit near the *n*-alkyl MAC regression line. The rightmost three points near the line in Fig. 1 are alkylcyclopentanes derived from Guerbet alcohols. That these molecules fit so closely to the line probably derives from the fact that the length of their *n*-alkyl branches are approximately the same as those of the *n*-alkyl groups used to derive the regression line, and the compact shape of both types of products are similar.

Thus, the 100°C kinematic viscosity can be readily selected by changing C_{av} in one of two ways: 1) by varying the average number of carbons in each R-group, or 2) by varying the number of R-groups attached to each cyclopentane.

For example, di(*n*-dodecyl)cyclopentane and tri(*n*-octyl)cyclopentane each must have 29 carbon atoms per molecule and the 100°C viscosities are 4.13 mm²/sec and 3.68 mm²/sec, respectively. The viscosities of mixtures also fit this relationship. Thus, tri(*n*-decyl)cyclopentane ($C_{av} = 35$) has a 100°C viscosity of 5.15 mm²/sec and tetra(*n*-decyl)cyclopentane ($C_{av} = 45$) has a 100°C viscosity of 7.99 mm²/sec. A 40:60 mixture of the two ($C_{av} = 41$) has a viscosity of 6.90 mm²/sec.

Although, for a given C_{av} , the choice of R-group does not have a large effect on the 100°C viscosity, it does have a marked effect on other properties.

VISCOSITY INDEX

The most common measure of the change of viscosity with temperature for lubricant products is the viscosity index (ASTM D2270). A high viscosity index signifies that the viscosity of the fluid changes less with temperature than that of a fluid with low viscosity index. In general, a high viscosity index is a desirable property. For example, com-

TABLE 1—LUBRICANT RELATED PROPERTIES OF MULTIPLY-ALKYLATED CYCLOPENTANE SYNTHESIZED HYDROCARBON FLUIDS

SAMPLE NUMBER	ALKYL, R*	m, NUMBER OF GROUPS	C_{av}^{**}	KINEMATIC VISCOSITY, ν , 100°C, mm ² /sec	VISCOSITY INDEX	POUR POINT, °C
1	n-octyl	2	21	2.18	135	-24
2	n-octyl	3	29	3.68	124	<-50
3	n-octyl	3,4	34	4.96	128	<-50
4	n-octyl	4	37	5.99	125	<-50
5	n-octyl	4,5	41	7.40	127	<-50
6	n-decyl	2	25	3.03	161	+6
7	n-decyl	2,3	29	3.78	161	-9
8	n-decyl	3	35	5.15	151	-27
9	n-decyl	3,4	41	6.90	144	-45
10	n-decyl	4	45	7.99	143	<-50
11	n-decyl	4,5	48	8.92	144	<-50
12	n-dodecyl	2	29	4.13	178	+21
13	n-dodecyl	3	41	6.99	164	-9
14	n-dodecyl	3,4	46	8.18	157	-15
15	n-dodecyl	4,5	59	11.91	153	-9
16	2-octyldodecyl	2,3	62	14.56	137	-57
17	2-ethylhexyl	3,4	31	5.35	40	<-50
18	isodecyl	3,4,5	46	11.68	83	-33
19	isotridecyl	3,4,5	56	20.09	71	-27
20	2-octyl	2,3	28	4.71	74	<-54
21	2-decyl	2,3	32	4.07	123	—

* R is the alkyl group in $\text{C}_5\text{H}_9\text{R}_m$

** $C_{av} = \sum_i (\text{weight fraction})_i \times (\text{carbon number})_i$

mercial "6 cSt" polyalphaolefins have a viscosity index of about 135 vs. about 95 for typical mineral oils. This property allows 5W-30 motor oils to be constructed more easily with PAO base oil.

For multiply-alkylated cyclopentanes, viscosity index changes in a regular and predictable way with molecular structure. Data are shown in Table 1 and Fig. 2.

The strongest dependence is on the nature of the R-group. The viscosity index depends most on the average chain length per substituent, CL_s . The average chain length per substituent is the weight average carbon number minus the five carbons in the ring divided by m , the number of alkyl groups per molecule.

$$CL_s = (C_{av} - 5)/m$$

For a MAC prepared with a single n-alkyl group, CL_s = the carbon chain length of the precursor alcohol. For mixtures of different starting n-alkanols, CL_s in the product MACs is closely approximated by the weight average CL_s of the starting alcohol mixture.

$$CL_s \approx \sum_i (\text{weight fraction of } i^{\text{th}} \text{ alcohol}) \times (\text{carbon number of } i^{\text{th}} \text{ alcohol}).$$

The differences between tri(n-octyl), $CL_s = 8$, $VI = 124$; tri(n-decyl), $CL_s = 10$, $VI = 151$; and tri(n-dodecyl), $CL_s = 12$, $VI = 164$ illustrate this dependence. In addition, the viscosity index is slightly affected by the number of substituents, VI decreasing with each additional substituent of the same average chain length per substituent. For example, di(n-decyl)cyclopentane has a VI of 161, tri(n-

decyl)cyclopentane has a VI of 151, and tetra(n-decyl)cyclopentane has a VI of 143.

For the same number of carbons in two different MACs, i.e. the same C_{av} , longer average chains give higher viscosity indexes. Examples with $C_{av} = 29$ give the following VI s: n-dodecyl, $CL_s = 12$, 178; n-decyl, $CL_s = 10$, 161; and n-octyl, $CL_s = 8$, 124.

MACs prepared from mixtures of alcohols exhibit the properties of the average chain length. For example, a 40:60 mixture of n-octanol and n-decanol can be used to make a multiply-alkylated cyclopentane product. The average chain length, CL_s , is 9.2 and the viscosity index of the resultant MAC in the $C_{av} = 40$ range is $VI = 134$, between the 144 of pure n-decyl products, $CL_s = 10$, and the 125 of the pure n-octyl products, $CL_s = 8$, of approximately the same molecular weight.

Physical mixtures of products also exhibit a viscosity index characteristic of average chain lengths. For example, a mixture of tri(n-octyl)cyclopentane ($VI = 124$) and tri(n-dodecyl)cyclopentane ($VI = 164$), 1:1, gives a calculated average chain length, CL_s , of 10. The mixture exhibits a viscosity index of 141, which is near the average of the two components and slightly below that of tri(n-decyl)cyclopentane ($VI = 151$), which also has an average chain length of 10 by synthesis.

Multiply-alkylated cyclopentane fluids with almost any 100°C viscosity and any viscosity index can be designed by a judicious choice of C_{av} , the carbon number of an average molecule, and m , the number of alkyl groups.

POUR POINT

These materials, like polyalphaolefins, tend to crystallize; that is, they tend to have sharp transitions between free-

flowing liquid and solid. Thus, the pour point measured in the typical ASTM D97 test gives an approximation of the melting point of the MACs. These materials may be quite fluid down to a temperature just above the pour point. Table 1 and Fig. 3 give summaries of pour point data for a variety of multiply-alkylated cyclopentanes.

Pour points depend on molecular structure in three ways: 1) for a given n-alkyl group, pour point decreases with increasing average carbon number, C_{av} , 2) for a given C_{av} , pour point decreases with increasing substitution, i.e., increasing m , and 3) branched alkyl groups give much lower pour points than do n-alkyl. These points will be taken in order.

Three entries in Table 1 (Nos. 6, 8 and 10) for n-decyl illustrate that, despite an increase in molecular weight of 140 units with each n-decyl group, the pour point decreases with increasing C_{av} . Despite the fact that the 100°C viscosity goes from 3 mm²/sec to 5 mm²/sec to 8 mm²/sec, pour point goes steadily down. The n-octyl derivatives show an even steeper curve: di(n-octyl)cyclopentane, -24°C, and tri(n-octyl)cyclopentane < -50°C.

Tri(n-octyl)cyclopentane (pour point = < -50°C) and di(n-dodecyl)cyclopentane (pour point = +21°C), both of which have $C_{av} = 29$, amply illustrate that pour point is extraordinarily sensitive to the exact structure. A mixture of di-

and tri(n-decyl)cyclopentanes with $C_{av} = 29$ shows a pour point of -9°C, between the two extremes.

As would be expected from the behavior of pure n-alkanes and isoparaffins, branching has a profound effect on pour point. A mixture of di- and tri(2-octyldodecyl)cyclopentanes rich in tri, with a $C_{av} = 62$ (molecular weight = 868, and viscosity at 100°C = 14.6 mm²/sec), has a pour point of -57°C, see Table 4).

DISCUSSION

A wide variety of physical properties are readily available in the alkylcyclopentane class of synthesized hydrocarbon fluids. Variation of the nature and number of substituents allows the construction of molecules with almost any physical properties desired. These variations are understandable in comparison to the properties of previously reported synthesized hydrocarbon fluids.

API Project 42 (5) reported viscosity and pour point data for substituted paraffins in which the regularity of the paraffin chain is interrupted by substituents. Selected data is given in Table 2. Much of the Project 42 data has been summarized recently by Denis (6). Normal paraffins have very high viscosity indexes, see Nos. 1 and 2 in Table 2, but also very high melting points (pour points). The branched

TABLE 2—SELECTED PROPERTIES OF SOME SYNTHESIZED HYDROCARBONS*

SAMPLE NUMBER	SYNTHESIZED HYDROCARBON	FORMULA	100°C VISCOSITY, mm ² /sec	VISCOSITY INDEX	MELTING POINT, °C
1	n-Octacosane	C ₂₈ H ₅₈	3.74	196	+61
2	n-Dotriacontane	C ₃₂ H ₆₆	4.92	206	+69
3	Tri(n-hexyl)methane	C ₁₉ H ₄₀	1.49	79	-28
4	Tri(n-octyl)methane	C ₂₅ H ₅₂	2.44	116	-14
5	Tri(n-decyl)methane R in R(n-C ₈ H ₁₇) ₂ CH	C ₃₁ H ₆₄	3.59	144	+9
6	n-hexyl	C ₂₃ H ₄₈	2.09	100	-19
7	n-octyl	C ₂₅ H ₄₈	2.44	116	-14
8	n-undecyl	C ₂₈ H ₅₈	3.04	132	0
9	n-tridecyl	C ₃₀ H ₆₂	3.49	144	+9
10	n-pentadecyl	C ₃₂ H ₆₆	4.02	151	—
11	n-heptadecyl	C ₃₄ H ₇₀	4.61	161	+23

* Data from Ref. (5).

TABLE 3—SELECTED PROPERTIES OF SOME SYNTHESIZED HYDROCARBONS*

HYDROCARBON	FORMULA	100°C VISCOSITY, mm ² /sec	VISCOSITY INDEX	POUR POINT, °C
1-Hexene trimer	C ₁₈ H ₃₈	1.4	88	< -55
1-Octene trimer	C ₂₄ H ₅₀	2.3	92	< -55
1-Decene trimer	C ₃₀ H ₆₂	3.7	122	< -55
1-Dodecene trimer	C ₃₆ H ₇₄	5.1	144	-45
1-Tetradecene trimer	C ₄₂ H ₈₆	6.7	157	-20
1-Hexene pentamer	C ₃₀ H ₆₂	3.8	96	< -40
1-Propene decamer	C ₃₀ H ₆₂	7.3	70	< -40
1-Decene tetramer	C ₄₀ H ₈₂	5.7	141	< -40
Squalane (isoprene hexamer)	C ₃₀ H ₆₂	4.1	117	

* Data of Refs. (5) and (7).

TABLE 4—EXTENDED PROPERTIES OF THREE MULTIPLY-ALKYLATED CYCLOPENTANES

R-GROUP	No. 1 Octyl/Decyl	No. 2 Octyl/Decyl	No. 3 2-Octyldodecyl
C_{av}	35*	41*	62
Vis, 100°C, mm ² /sec	5.23	6.91	14.56
Vis, 40°C, mm ² /sec	26.28	38.67	109.30
Viscosity Index	134	139	137
Pour Point, °C (°F)	< -50 (< -60)	-48 (-55)	-57 (-70)
Vis, -40°C, mPa·s	4,100	9,400	78 600
Flash Point, °C (°F)	246 (475)	257 (495)	307 (585)
Fire Point, °C (°F)	277 (530)	296 (565)	332 (630)
Evaporation Loss (D972) 6.5 hr. @ 250°C	10%	5%	<.1%

* Estimated from 100°C viscosity.

synthesized hydrocarbons in the Project 42 data exhibit much lower pour points than normal paraffins of the same molecular weight, but also lower viscosity indexes.

In this series, as in the MACs, VI increases regularly with increasing chain length. For example, sample Nos. 3, 4 and 5 in Table 2 are all symmetrical tri(n-alkyl)methanes, R₃CH. Their viscosity indexes increase markedly with chain length: tri(n-hexyl), VI = 79, mp = -28°C; tri(n-octyl), VI = 116, mp = -14°C, and; tri(n-decyl), VI = 144, mp = +9°C. The di(n-octyl)methane derivatives, RCH(n-octyl)₂, sample Nos. 6-11 in Table 2, show the same trend. VI increases from R = n-hexyl (C₆, VI = 100) to n-octyl (C₈, VI = 116) to n-undecyl (C₁₁, VI = 132) to n-tridecyl (C₁₃, VI = 144) to n-pentadecyl (C₁₅, VI = 151) to n-heptadecyl (C₁₇, VI = 161), and the pour point also increases in the same order.

Both polyalphaolefins and MACs have a decided advantage over these simple isoparaffins in that they are liquids to quite low temperatures.

There is much data on the properties of the commercially available poly(1-decene), but not nearly as much for other polyalphaolefins. In one study, Brennan (7) observed that the viscosity index and pour point of PAOs vary in much the same way with the size of the alkyl group as the authors observed for multiply-alkylated cyclopentanes. Table 3 shows data from his paper. The trimer of 1-hexene has a lower VI than the trimer of 1-octene which has a VI lower than that of 1-decene trimer, which has, in turn, a VI lower than that of 1-dodecene trimer. Pour points increase with increasing chain length, with the trimer of 1-tetradecene having the highest pour point.

Brennan (7) found that for poly(1-alkene)s of the same molecular weight, increased branching leads to increasing 100°C viscosity and decreasing viscosity index. The authors observed the same trends in MACs (see for example Nos. 2 and 12 in Table 1 and also Nos. 5 and 13).

The molecular weight distributions of n-decyl-MACs are narrow. Mixtures containing only tri- (C_{av} = 35) and tetra-substituted MACs (C_{av} = 45) are easy to prepare. Although the alkylation proceeds easily to the trialkylcyclopentadienes, the attachment of additional alkyl groups is significantly more difficult. No raw material is wasted. All of the alcohol and dicyclopentadiene are incorporated into the desired relatively narrow boiling range material without distillation of the product. Only stripping of residual alcohol is required. No undesirably light nor undesirably heavy ma-

terials which degrade the desirable properties of the fluid, are produced. Using the method described here, mixtures which are over 95 percent tri- and tetra(n-alkyl)cyclopentanes are readily prepared. Such undistilled mixtures contain no monoalkylcyclopentanes and only trace quantities of dialkyl- and pentaalkylcyclopentanes. The two example products below are prepared simply by hydrogenation of reaction mixtures from which solvent alcohol has been stripped. No fractionation was necessary to remove by-products.

EXAMPLES OF PARTICULAR FLUIDS

N-Octanol/N-Decanol Derived Multiply-Alkylated Cyclopentane

Because the pour point of tri(n-decyl)cyclopentane (pour point = -27°C) is too high for use in conventional automotive engine oils, N-alkylcyclopentanes were prepared using a mixture of n-octanol and n-decanol. Properties of two examples of these fluids are shown in Table 4. Sample No. 1 can be formulated into a fully synthetic 5W-30 motor oil.

The product is viscometrically equivalent to comparable engine oils formulated from commercially available PAOs, and in-house proprietary bench and engine tests suggest that the performance properties of these fluids are also equivalent to those of PAOs, see Table 5. In this same engine test a commercially available fully synthetic 5W-30 SG/CD engine oil has a viscosity increase of 27 percent.

2-Octyldodecanol Derived Multiply-Alkylated Cyclopentane

Probably the most interesting MACs prepared in this program are those derived from 2-octyldodecanol. One example is shown in Table 4. Note the remarkable combination of properties. Viscosity at 100°C of 14.6 mm²/sec, viscosity index of 137, pour point of -57°C, and evaporation loss at 250°C of less than 0.1 percent. This material will be useful in any application in which low volatility at high temperatures and good fluidity at low temperatures are requirements.

Carré has already reported wear data for this material (8). It is inadvertently mislabelled as PAO in his Table 2, Nos. 4 and 5. Because MACs are hydrocarbons and dissolve antiwear additives, Carré concludes that their performance is superior to perfluoropolyethers.

TABLE 5—COMPARISON OF ENGINE OILS MADE FROM
MULTIPLY-ALKYLATED CYCLOPENTANES AND PAOS

PROPERTIES	MAC BASE OIL* NO. 1 IN TABLE 4	PAO BASE OIL* 5.3 mm ² /sec
Vis, 100°C, mm ² /sec	10.99	10.88
Vis, 40°C, mm ² /sec	61.89	62.87
Viscosity Index	171	166
CCS -25°C, mPa·s	2250	2550
<u>In-house Engine Test Results</u>		
% Viscosity Increase at end of test	6	9
Rocker Arm Sludge**	9.50	9.80
Piston Skirt Varnish**	8.25	7.80
Oil Ring Land Face Deposits**	6.88	6.61
Oil Consumption, lbs	1.12	2.31

* Formulated as SAE 5W-30 engine oils using 57.3 wt. % of the indicated synthetic hydrocarbon, plus an identical package consisting of 24 wt. % di (isotridecyl) adipate, 11.5 wt. % of a commercially available DI package (all secondary zinc dithiophosphate, overbased calcium sulfonate, and bispolyisobutenylsuccinimide), 7.0 wt. % of a commercially available VI improver (non-dispersant OCP), 0.2 wt. % methylene-4,4'-bis(2,6-di(1,1-dimethylethyl)phenol).

** Rating, maximum merit score of 10.

CONCLUSION

Multiply-alkylated cyclopentanes, in which two to five alkyl groups are attached to a single five-membered carbon ring, are readily available from the reaction of dicyclopentadiene and commercially available alcohols. The physical properties of the materials are significantly dependent on the choice of alkyl group and the number of alkyl groups attached to the ring. Examples with viscosities (100°C) from 2 to 20 mm²/sec, viscosity indexes from 20 to 180, and with pour points to less than -60°C have been prepared.

Materials which are viscometrically equivalent to poly(1-decene)s have been prepared and all the tests performed to date suggest that they are also equivalent in chemical stability.

The versatility of the synthesis and the ready availability of a wide variety of commercial alcohols will allow the preparation of multiply-alkylated cyclopentanes with extraordinarily rich combinations of properties. The low volatility, low pour point example presented above is just one example of the unique properties achievable.

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