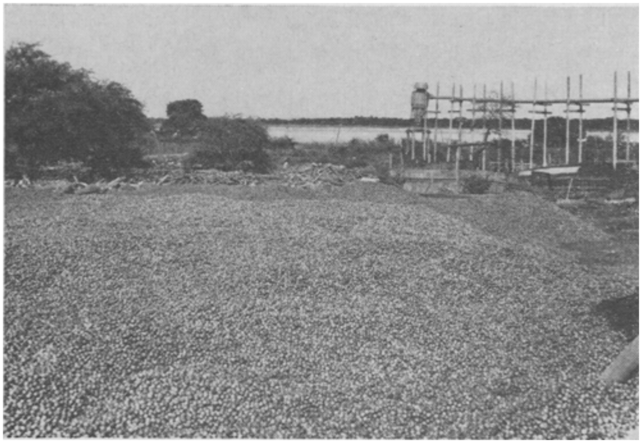

FIG. 8. Mbocayá fruit stored on ground outside of oil mill to dry prior to processing.

which is exploited for its pulp and kernel oils. Since the products of many of these palms are consumed locally or are exported to neighboring countries or Europe, they are little known in the United States.

The mbocayá is one of these palms which has been exploited commercially for its kernel oil for about 50 years and for a lesser time for its pulp oil. The kernel oil is similar in composition to that of other American oil palms but is unique in being more unsaturated and having a lower melting point which clearly reflects the more temperate environment in which this palm thrives.

Paraguay contains 6 to 7 million of these palms which produce annually an estimated 55,000 to 120,000 metric tons of fruit, only a part of which is processed for oil. In recent years production of kernel oil has varied between 2,000 and 2,700 metric tons and pulp oil between 500 and 1,100 metric tons, all of which has been consumed locally or exported to Argentina. Processing mbocayá fruit presents many difficulties not encountered with most oilseeds but which are similar to those encountered in processing most American oil palm fruits.

This is the first comprehensive report in English on the mbocayá palm and its economic importance.

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Isomerization of Polyunsaturated Fatty Acids and Their Esters by Sodium Amide in Liquid Ammonia[1]

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ALTHOUGH AMIDES of the alkali metals have been used extensively as reagents in numerous inorganic and organic reactions (1, 2, 3), their reaction with the fatty acids or derivatives has only been given limited attention. Khan *et al.* (4) used sodium amide in liquid ammonia in dehydrobromination of dibromostearic acid to stearolic acid. Attempts by the authors to apply the method for preparation of the diacetylenic derivative of tetrabromostearic acid were not successful. Consequently the study of the effect of sodium amide on a substance having a methylene-interrupted unsaturated system seemed to be necessary before tackling the problem of preparing polyacetylenic compounds. Because polyacetylenic fatty acids are unavailable, the polyethenoid fatty acids, linoleic and linolenic acids and their esters, were used in this study.

The alkali amides are the caustic alkalis of the ammonia system. One might expect some resemblance between the actions of the alkali amides and of the alkali hydroxides upon the polyunsaturated fatty acids. The alkali hydroxides at high temperatures are known to cause migration of the double bonds resulting in a conjugated system, the character of the products being dependent to a large degree on the concentration of the alkali reagent, the temperature of the reaction, and the ratio of the reagent to sample size. With the alkali amides in liquid ammonia one might expect greater effect on the polyunsaturated fatty acids under comparable conditions due to the basicity

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TABLE I

Isomerization of Methyl Linoleate, Methyl Linolenate, and Linolenic Acid by Sodamide in Liquid Ammonia

Time		Methyl linoleate	Methyl linolenate		Linolenic acid	
Hrs.	Min.	k_{2350}	k_{2350}	k_{2680}	k_{2350}	k_{2680}
	25	......	......	......	24.7	4.9
	50	......	......	......	27.9	11.4
1	30	......	......	......	38.5	10.5
					26.6	12.0
2	....	29.3	42.4	19.6	31.4	7.6
2	10	......	......	......	32.5	8.1
3	....	34.7	50.4	30.1	29.5	10.5
4	....	39.8	55.4	33.3	......	......
5	....	34.7	51.4	20.0	......	......
6	....	16.5	......	......	......	......

of the medium. It is of interest to note that reactions in liquid ammonia take place at —32°C., a temperature at which the alkali hydroxides have little or no effect on the isomerization of polyunsaturated fatty acids.

A review of the literature revealed that some cyclic dienes containing isolated double bonds could be converted to isomeric compounds containing conjugated double bonds when treated with alkali amides (5). Thus 2,5-dihydroanisole is isomerized to 2,3-dihydroanisole when treated with potassium amide in liquid ammonia. The authors discovered similar effects with the polyunsaturated fatty acids. The present paper describes the use of sodium amide in liquid ammonia in the isomerization of linoleic and linolenic acids and their esters.

Preliminary Experiments with Ethyl Linoleate

In a preliminary experiment ethyl linoleate (prepared by the bromination-debromination method) was used. The apparatus consisted of a 2-liter, three-necked flask. The center neck carried a mercury seal through which a mechanical stirrer operated. One of the other two necks was connected to a condenser cooled by dry ice—acetone while the third served for introducing the reactants. About 0.5 l. of liquid NH_3 was introduced, followed by 0.3 g. anhydrous $FeCl_3$ with stirring. Metallic sodium (8.0 g.) was added in small pieces over a period of 15 min. Stirring was continued for ½ hr., which is long enough for all the sodium to be converted to $NaNH_2$. Ten grams of ethyl linoleate were then added from a dropping funnel in a fine stream. Reaction was stopped after 5½ hrs. by adding 15 g. NH_4Cl to destroy the sodium amide. Most of the liquid NH_3 was then allowed to evaporate. Five hundred ml. of water were added, and the mixture was acidified with dilute HCl (1:1), followed by extraction with ether. The extract was then dried with Na_2SO_4, filtered, and evaporated under a stream of nitrogen. The yield was 7.9 g. (92.5%) of the crude product which had a melting point of 42–50°C. The ultraviolet absorption of the product in absolute ethanol showed a maximum at 2325 Å and had an extinction coefficient, k, of 51.6.

Recrystallization of the crude product from Skelly F yielded 6.2 g. of white product (amide), m.p. 56–60°C. About 1.3 g. of yellow oily liquid was recovered from the filtrate. The liquid, probably esters, had a k of 55.2 at 2325 Å.

Infrared spectra of both solid and liquid fractions showed that the solid fraction consisted mainly of amides while the liquid fraction contained very little amide. The infrared absorption between 5.6 and 6.0 μ of the liquid fraction indicated the presence of at least two different kinds of C═O groups. The band at 5.76 μ is characteristic of an ester group, and the smaller band at 5.84 μ may indicate a free carboxyl group or a ketonic group, possibly resulting from the self-condensation of ester molecules (6, 7, 8).

Conjugation in solid and liquid fractions was of both *cis, trans,* and *trans, trans* types. Both fractions had almost similar extinction coefficients, indicating that amide formation and conjugation of the double bonds are two independent reactions. Displacement chromatography of the solid amide, using ethyl stearate as displacer, indicated that several components were present.

Action of $NaNH_2$ on Methyl Linoleate, Methyl Linolenic Acid in Liquid Ammonia. A series of preparations starting with these substances was studied in which reaction time was the only variable. In a 500-ml. flask the following reagents were added in the order named: 25–30 ml. liquid ammonia, 0.1 g. $FeCl_3$, 1.5 g. sodium, and 1.0 g. acid or ester in 10 ml. dry ether. Ether was used because it reduced the amount of acid or ester which precipitated upon addition to the liquid ammonia. The data shown in Table I indicate that the reaction rate is not strictly reproducible. This is probably due to the heterogeneous character of the reaction medium. For both methyl linoleate and methyl linolenate the optimum reaction time seems to be about 4 hrs. The reaction with linolenic acid does not seem to proceed as far as it does with its ester.

Methyl Oleate. Methyl oleate was allowed to react with sodamide in liquid ammonia under the same conditions as described for linoleate and linolenate. From infrared spectra of the isolated product it was evident that the amide was formed, but the *cis* configuration of the double bond was unchanged.

Action of $NaNH_2$ on Linoleic Acid and Methyl Linoleate in Petroleum Ether. The $NaNH_2$ was prepared from 23 g. sodium, about 700 ml. liquid NH_3, and about 0.4 g. $FeCl_3$ as already described. To this 700 ml. of petroleum ether were added, and the ammonia was allowed to evaporate over night. Linoleic acid (0.8045 g.) was dissolved in petroleum ether and made up to 25 ml. One ml. of the solution was added to 1 ml. of the $NaNH_2$-petroleum ether suspension. Several such mixtures were shaken on a mechanical shaker for different time periods. At the end of each interval of time the mixture was made up to 25 ml. with absolute EtOH (which also served to destroy the $NaNH_2$), then filtered, and diluted for ultraviolet measurements in the Beckman spectrophotometer. The results are shown in Table II.

Four times the amount of catalyst used in the above experiments were added to 1 ml. of each of the

TABLE II

Isomerization of Linoleic Acid and Methyl Linoleate by Sodamide in Petroleum Ether

Shaking time	k_{2330}	
	Linolenic acid	Methyl linoleate
(min.)		
15	1.12	
30		0.42
50	5.28	
90		0.53
120	3.10	
150		0.72
190	2.25	
210		0.52
225	0.74	

acid and ester solutions. After shaking 1 hr. the specific extinction coefficients were 3.48 for linoleic acid and 1.11 for methyl linoleate.

These experiments showed that $NaNH_2$ in petroleum ether causes much less isomerization of linoleic acid than the same reagent in ammonia. The reaction is almost negligible with methyl linoleate, and a change in the amount of catalyst does not change the effect markedly. The reaction probably is ionic and requires a polar solvent.

Separation and Characterization of Reaction Products

Linoleyl Amides. A crude product having a specific absorption coefficient of k 34.7, at 2330 Å was recrystallized from petroleum ether and fractionated by the technique of displacement chromatography, using octadecyl iodide as displacer in absolute ethanol (Figure 1). The infrared spectra of the fractions are shown in Figure 2. Fraction A consists mainly of the amide of unchanged linoleate. It has some isolated *cis* and some isolated *trans* double bonds which probably existed in the bromination-debromination linoleate used. Fractions B and C are a continuation of A with some of the following fraction D appearing in them. Fraction D is an amide of *cis, trans* conjugated linoleate. It had a specific extinction coefficient k of 90.1 at 2320 Å.

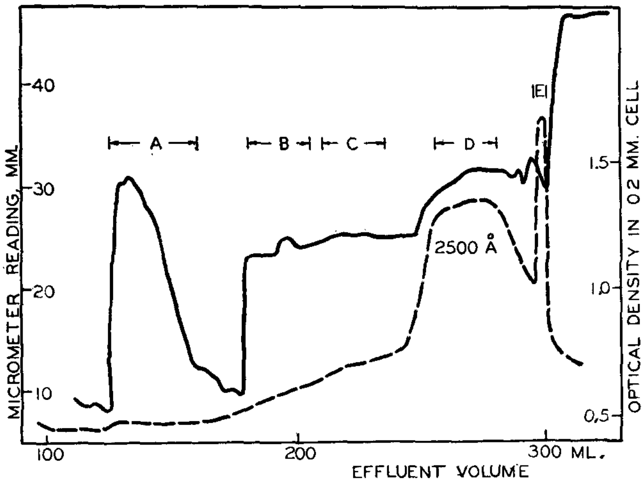

FIG. 1. Displacement separation of linoleyl amides.

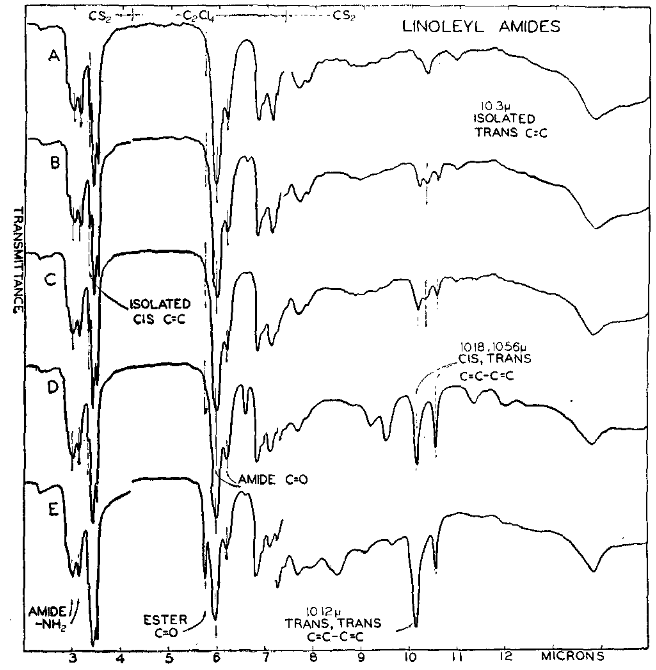

FIG. 2. Infrared spectra of linoleyl amide fractions. (See Figure 1.)

Fraction E is mainly *trans, trans* conjugated diene contaminated with some *cis, trans* conjugated diene from the preceding fraction. The infrared curve shows also a relatively strong band at 5.76 mμ which indicates an ester group. The *cis, trans* diene represents contamination of the preceding fraction, *cis, trans* diene amide, and the *trans, trans* substance is probably the ester. The dip in the interferometer curve separating fractions A and B represents an artifact caused by flooding the column with too much sample and by subsequent addition of another small column at the bottom of the chromatographic setup. A displacement separation was made on a similar product, using ethyl stearate as displacer in which the displacement showed no artifact. In this experiment only two major zones were indicated, a large zone of unconjugated material and a small zone of *cis, trans* conjugated diene. We chose to show the experiment with artifact because when octadecyl iodide was used as displacer, a third substance is displaced (*trans, trans* diene), and because infrared spectra in the latter experiment were measured on 10% solutions rather than films of unknown thickness.

Linolenyl Amides. A crude product having a k of 16.1 at 2680 Å and 43.5 at 2330 Å was used for this study. It was recrystallized twice from petroleum ether. The liquid fraction, which represented about 20% of the total, had a k of 16.2 at 2680 Å and 44.0 at 2330 Å. The solid fraction had an iodine value (Woburn, 3 hrs. at 0°C.) of 357 (theory 375). Hydrogenation of the latter, followed by hydrolysis with alcoholic KOH, acidifying, and recrystallizing the acid gave a product of m.p. 67.5–68°C. (uncorrected). This indicates that the 18 carbon chain of linolenate remained unaltered.

The solid fraction was subjected to displacement chromatography on a charcoal-Hyflo column (1:2), using ethyl stearate as displacer and 95% ethanol as solvent, using technique and an instrument described elsewhere (9). Density readings on a Beckman spectrophotometer at 2400 Å and 2900 Å were taken simultaneously with the interferometer readings on the effluent. The displacement diagram shown in Figure 3 indicates that the crystalline fraction contained several components. Appropriate cuts were made, and their infrared spectra were measured (Figure 4).

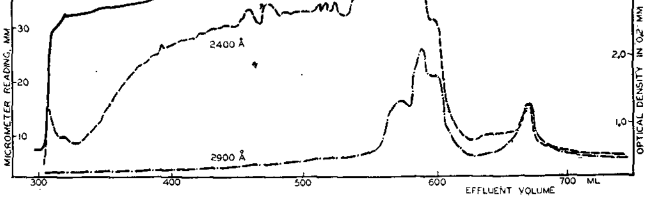

FIG. 3. Displacement separation of linolenyl amides.

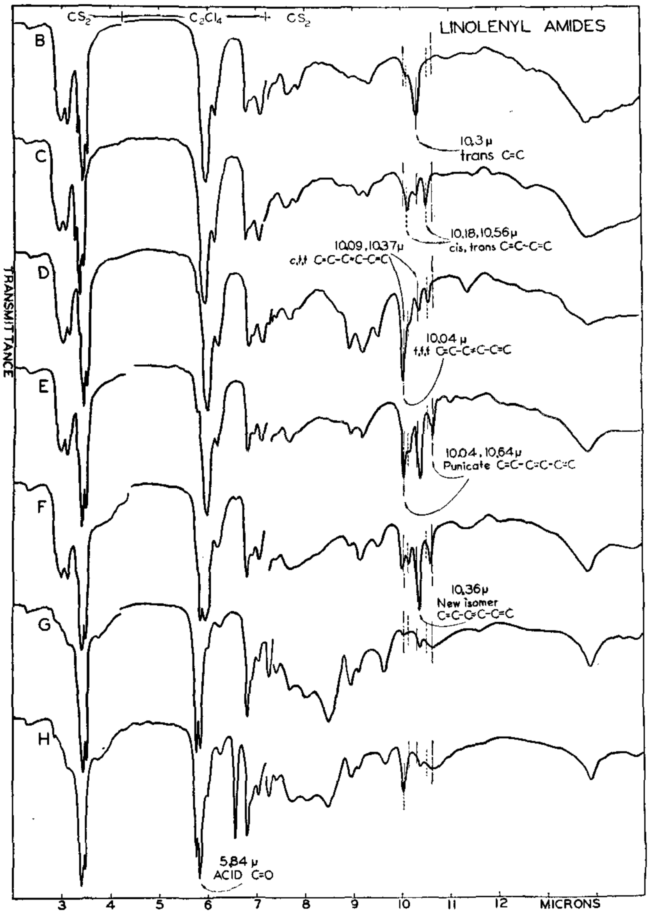

FIG. 4. Infrared spectra of linolenyl amides. (See Figure 3.)

Fraction A shows a density reading at 2400 Å higher than that of fraction B, which consists mainly of an amide with *cis* and *trans* isolated double bonds. Fraction C is mainly isolated *cis* and *cis, trans* diene-conjugated triene. The second fraction C is essentially the same as the first except for a small amount of triene conjugation (all *trans*). Fraction D represents an amide consisting mainly of all *trans* conjugated triene isomer (10). It also contains minor amounts of a *cis, trans* diene-conjugated triene, and the isolated *trans* isomer. Fraction E contains some amide isomer with all *trans* conjugation, and the two bands at 10.04 μ and 10.64 μ indicate some triene conjugated isomer of the punicic acid type (10). (Punicic acid is a naturally-occurring isomer of eleostearic acid. Its infrared absorption spectrum is quite different from that of either α or β eleostearic acid, but its exact geometric configuration is not yet known.) The shoulder at 10.18 μ may indicate some *cis, trans* diene conjugated triene. The strong band at 10.38 μ indicates some type of geometric isomerism which we cannot readily identify because of the lack of infrared spectra of reference compounds. However in the IR spectra of methyl eleostearates, reported by Paschke and associates, the absorption band at 10.37 μ distinguishes between α-eleostearate (*cis*-9, *trans*-11 *trans*-13) and β-eleostearate (*trans*-9, *trans*-11, *trans*-13), suggesting that this absorption is due to the *cis* conjugated double bond. It might be that the strong band at 10.38 μ in fraction E is due to an isomer having more than one *cis* conjugated double bond, such as *tri cis* or *cis, cis, trans* conjugated triene. The possibility also exists that the substance with the unusual infrared spectrum possesses a triene structure in which one double bond remains nonconjugated. However the only fractions having this strong band at 10.36 μ showed strong triene absorption in the ultraviolet region. Moreover our experience with displacement chromatography would lead us to predict that such an isomer with nonconjugated double bonds would be more weakly adsorbed than a conjugated triene.

Fraction F consists mainly of the new isomer with small amounts of the *cis, trans* diene-conjugated triene, and punicic acid amide. Fraction G is not an amide and has no appreciable conjugation. It has a strong band at 5.75 μ indicative of an ester, another at 5.84 μ indicative of free acid. It does not show any isolated *cis* band. This information suggests that fraction G consists mainly of a saturated ester mixed with a saturated acid. The only source could be the displacer.

Fraction H is not an amide, does not contain isolated *cis* double bond, has a strong band at 5.84 μ (free acid) and a weaker one at 5.75 μ (ester), has a relatively strong band at 10.04 μ (all *trans* conj. triene). This acid must come from the sample. Probably during the reaction some soap was formed which on acidification gave the free acid. The appearance of this acid within the displacer zone may represent a type of carrier displacement or elution. It is evident from this experiment that an amide is less strongly adsorbed than the corresponding free acid.

The study described here indicates that sodamide in ammonia affects the polyunsaturated fatty acids in a manner similar to potassium hydroxide at high temperature. It appears likely that all possible *cis, trans* isomers are produced, and some have been identified *via* infrared spectra. The combination of displacement chromatography with ultraviolet and infrared spectra has been demonstrated as a very useful tool for separation and identification of *cis-trans* isomers of dienes and trienes.

Summary

Sodium amide in liquid ammonia has been shown to cause isomerization of the polyunsaturated fatty acids. With methyl linoleate the products of reaction are a complicated mixture consisting of amides and esters, conjugated and nonconjugated. The conjugated products were mainly *cis, trans,* and a small part *trans, trans.* With linolenate the picture is much more complicated. *Cis, trans* diene-conjugated triene, all *trans,* and possibly *cis, cis, trans,* or all *cis* conjugated triene, and conjugated triene of the punicic acid type were demonstrated to be present in the reaction products.

Time of reaction has been shown to have a considerable effect on the extent of conjugation of the double bonds. With the system used, optimum time for maximum conjugation in linoleate and linolenate esters is about 4 hrs. Under these conditions the extinction coefficients of the isomerized esters were $k_{2350}=39.8$ for linoleate and $k_{2350}=55.4$ and $k_{2680}=33.3$ for linolenate.

Liquid ammonia is preferred to petroleum ether as a medium for the reaction.

The extent of isomerization is greater with the esters than with the free acids. This may be due to the

higher solubility of the esters. The amount of the catalyst did not show any appreciable effect on the extent of the isomerization in petroleum ether.

Displacement chromatography in conjunction with ultraviolet and infrared spectra is shown to be an effective means for separation and identification of *cis, trans* diene and triene isomers.

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Detergents from Synthetic Olefins[1]

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OLEFINS synthesized in the Fischer-Tropsch process, although commercially unavailable, are a potential source of raw material for detergents (11). They possess a higher degree of linearity and terminal unsaturation than the olefiin polymers from propylene (4) used in the commercial manufacture (6) of alkylaryl sulfonates. Because the hydrocarbon radical provides the hydrophobic part of surface-active agents, the nature of the olefin would be expected to affect detergent performance. In alkyl sulfates, straight-chain nonterminal olefins are known to produce good foaming and wetting properties whereas straight-chain terminal olefins give good detergency (9, 13).

Because of a shortage of natural fats, detergents from Fischer-Tropsch olefins were investigated in Germany during World War II. Although a plant had been constructed to make alkyl sulfates, the war ended and it was never placed in operation (9, 15).

In the course of research on possible uses of Fischer-Tropsch products we have experimentally studied the effectiveness of surface-active agents prepared from such olefins. Sodium alkylbenzene sulfonates and sodium alkyl sulfates were prepared from the olefins containing 11, 12, 13, 14, 15, and 16 carbon atoms. Individual sulfonates and the mixture of 11- through 15-carbon sulfonates corresponding to the distribution of olefins in the Fischer-Tropsch product were evaluated by comparison with a reference sulfonate prepared from propylene tetramer. The individual sulfates and the mixture of 12- through 17-carbon sulfates were evaluated by comparison with a reference sulfate prepared from lauryl alcohol. Conventional soil-removal, whiteness-retention, foaming, and wetting tests were used to estimate relative effectiveness. Observations of caking, hygroscopicity, and solubility were also made.

Preparation

The olefins were synthesized in a pilot plant by the fluidized-iron process (17, 18). Reaction of carbon monoxide and hydrogen at 300° to 340°C. under 15 to 40 atmospheres' pressure gave a liquid product containing a wide molecular-weight distribution of olefins, paraffins, and oxygenated compounds. The product was washed with caustic to remove organic acids and then fractionated. Analysis of the fractions gave the estimated composition shown in Table I. Individual fractions contain 13% to 29% normal terminal olefins whereas the propylene polymer contains very small amounts of such structures (4).

TABLE I

Properties of Olefin Fractions

Fraction	Boiling Range, °C.	Olefin %	Olefin Distribution, %			
			Normal		Branched	
			Terminal	Internal	Tertiary	Others
$C_{11}H_{22}$	174-196	55	29	24	19	28
$C_{12}H_{24}$	196-216	54	25	25	20	30
$C_{13}H_{26}$	216-237	54	21	25	20	34
$C_{14}H_{28}$	237-254	53	17	25	21	37
$C_{15}H_{30}$	254-273	53	16	23	21	40
$C_{16}H_{32}$	273-287	52	13	22	22	43

Alkylbenzenes were prepared from the fractions of 11- through 15-carbon atoms by alkylating benzene with anhydrous ethanesulfonic acid as the catalyst (1). Each alkylate was washed with caustic and fractionated to give a center cut for sulfonation. Physical properties of these center cuts are listed in Table II. Each was sulfonated with 100% sulfuric acid (10), neutralized with sodium hydroxide, deoiled, desalted, and dried.

TABLE II

Physical Properties of Alkylbenzenes

Alkylbenzene	Boiling Range, °C., 5 mm.	N_D^{20}	Specific Gravity 25°C.
$C_{11}H_{23}C_6H_5$	115-135	1.4863	0.873
$C_{12}H_{25}C_6H_5$	135-150	1.4871	0.878
$C_{13}H_{27}C_6H_5$	150-165	1.4926	0.890
$C_{14}H_{29}C_6H_5$	165-175	1.4875	0.894
$C_{15}H_{31}C_6H_5$	175-185	1.4848	0.883

Sodium alkyl sulfates were synthesized from alcohols obtained by the classical oxo reaction (8, 12, 14). The mixture of alcohols contains one more carbon atom than the starting olefins. Crude alcohols were fractionated to give center cuts with the properties listed in Table III. Each center cut was sulfated with chlorosulfonic acid (5) in absolute ether. Neutralization of the sulfates with sodium hydroxide was followed by deoiling, desalting, and drying.

For each detergent type a reference standard common to the trade was prepared in the same way as the experimental samples. A sodium dodecyl sulfonate was prepared from propylene tetramer. A sodium lauryl sulfate was prepared from lauryl alcohol melting at 23° to 24°C.

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