azelaic, and adipic acids but was greater than that of the phthalate ester.

A mechanistic scheme of plasticizer-polymer interaction has been presented, proposing that the rate of diffusion of plasticizer through the polymer mass is a controlling factor in both good low-temperature performance and the resulting high migration losses. Methyl esters, some aromatic esters, and esters containing three or more polar centers have improved permanence but show a more rapid change in torsional modulus as the temperature is lowered during the determination of the Clash-Berg stiffening temperature.

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New Reactions of Long-Chain Isocyanides[1]

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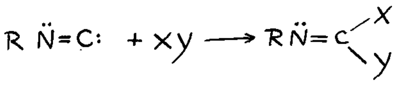
THE ISOCYANIDES were discovered about 100 years ago by Gautier, who prepared them by treating an alkyl iodide with silver cyanide (1). In the late nineteenth century Nef (2, 3) investigated reactions of short-chain and aromatic isocyanides with many reagents such as halogens, acid chlorides, sulfur, hydrogen, water, or phosgene. These reactions indicated a peculiar type of unsaturation of the isocyanide group located entirely at the terminal carbon atom; this is nowadays usually presented by the resonance structures:

$$R\ddot{N}=C: \longleftrightarrow R\overset{+}{N}\equiv\overset{-}{C}:$$

Addition reactions were reported to take place at the negative carbon atom, giving products with the following structures:



$$R\ddot{N}=C: + XY \longrightarrow R\ddot{N}=C\begin{smallmatrix}X\\Y\end{smallmatrix}$$

In 1920 Passerini (4, 5) re-investigated and confirmed many of the results of earlier workers and also discovered several new reactions. Some typical reactions are shown in Table I.

TABLE I
Reactions of Isocyanides

	Reference
1. $ArNC + RCHO \longrightarrow RCO.CH = NAr$	(4)
2. $ArNC + RCO.Cl \longrightarrow RCO\overset{Cl}{C} = NAr$	(2)
3. $RCN + COCl_2 \longrightarrow RN = \overset{Cl}{C} - CO - \overset{Cl}{C} = NR$	(5)
4. $RNC + R'MgX \longrightarrow RN = \overset{R'}{C} - MgX$	(6)
5. $RNC + Cl_2 \longrightarrow RN = CCl_2$	(7)
6. $RNC + HCl \longrightarrow RN = \overset{H}{C} - Cl$	(7)
7. $RNC + H_2 \longrightarrow RNHCH_3$	(7)
8. $ArNC + R'CHO + R''COOH \longrightarrow ArNHCO\underset{OCOR''}{C}HR'$	(8)

None of the earlier workers investigated the reactions of fatty isocyanides. In our present studies a series of fatty isocyanides were prepared by the "carbylamine reaction" by heating a primary amine with chloroform and potassium hydroxide in a solvent such as *n*-butanol:

$$C_{12}H_{25}NH_2 + CHCl_3 + 3KOH \longrightarrow C_{12}H_{25}N = C + 3KCl + 3H_2O$$

The reactions of the long-chain isocyanides were studied with three classes of compounds: halogens, alkylating agents (*e.g.*, benzyl chloride, methyl sulfate), and acid chlorides. During our investigation some startling differences were observed between the behavior of the fatty isocyanides and the reported behavior of the corresponding short-chain or aromatic isocyanides. These are the subject of this paper.

Experimental

Preparation of Isocyanides

All the isocyanides were prepared by the reaction of a primary amine with chloroform and potassium hydroxide. The preparation of dodecylisocyanide is used as a typical example.

Double-distilled dodecylamine (370 g., 2 moles) was dissolved in chloroform (400 g., 3.3 moles). This solution was added slowly from a dropping funnel to a well-stirred solution of potassium hydroxide (400 g. 85% pellets, 6 moles) in 1,400 ml. of *n*-butanol.

After all the amine-chloroform mixture had been added, the resultant solution was refluxed for five to six hours. Water was added to dissolve the salts, the butanol layer was separated and washed with water. The butanol was then stripped under vacuum, taking care to allow the pot temperature to reach no higher than 140°C. An infrared spectrum of the crude iso-

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[1] Presented at Fall Meeting, American Oil Chemists' Society, Chicago, Ill., October 20–22, 1958.

cyanide mixture showed a strong isocyanide band (4.72 microns), also a strong amide band (6.05 microns). The crude isocyanide was then distilled, and pure dodecylisocyanide was obtained in 60% yield, boiling point 104–105°C. at 0.5 mm. The residue consisted largely of N-dodecylformamide, distilling at 170–175°C. at .5 mm. and was identified by its boiling point and its infrared spectrum. The infrared spectrum of the isocyanide fraction showed strong absorption bands at 4.72 microns and at 6.28 microns; these have been reported as being characteristic of isocyanides (9).

Table II gives a list of the isocyanides prepared, yields, and boiling points.

TABLE II

Fatty Isocyanides

RNC R	Boiling point	Yield
	°C.	%
C_8H_{17}	34– 35/.4 mm.	76.4
$C_{10}H_{21}$	59– 60/.5 mm.	75.0
$C_{12}H_{25}$	104–105/.5 mm.	59.0
$C_{14}H_{29}$	129–131/.5 mm.	70.0
$C_{16}H_{33}$	140–142/.5 mm.	56.3
$C_{18}H_{37}$	160–162/.5 mm.	47.5
$C_{18}H_{35}$(oleyl)	152–154/.5 mm.	65.5

Reactions of Fatty Isocyanides

With Halogens: Chlorination of dodecylisocyanide is given as a typical example. Additions of bromide or iodine were carried out by similar procedures except that with iodine the solution was refluxed for several hours.

Dodecylisocyanide (9.75 g., 0.05 mole) was dissolved in 50 ml. of carbon tetrachloride, and dry chlorine gas was bubbled into the solution. The reaction is exothermic and was kept between 25–30°C. by external cooling. After a few minutes a white precipitate was formed. When the temperature no longer rose, the gas addition was stopped, the solution was cooled to 0°C., and the white solid was filtered off. Obtained 7 g. of crystalline material, m.p. 160–165°C. with decomposition.

Anal. Calcd. for $C_{12}H_{25}N.C.Cl$: neutral equivalent (potentiometric) 230.5; N, 6.07; Cl, 15.4; C, 67.6; H, 10.9; found: neutral equivalent, 230.5; N, 6.12; Cl, 15.6; C, 67.47; H, 10.83.

Results on the halogenated isocyanides are summarized in Table III.

TABLE III

Halogenated Isocyanides

Compound	% Halogen		% Nitrogen	
	Found	Calcd.	Found	Calcd.
$C_{12}H_{25}N.C.Cl$	15.6	15.4	6.12	6.07
$C_{12}H_{25}N.C.Br$	29.9	29.1	5.02	5.09
$C_{12}H_{25}N.Cl$	39.4	39.4	4.28	4.34
$C_{16}H_{33}NC.Cl$	12.3	12.6	5.07	5.00
$C_{18}H_{37}NC.Cl$	11.8	11.4	4.46	4.51

With Acid Chlorides: Preparation of Dodecylisocyanide-Acetyl Chloride Addition Product. To 48 g. (.24 mole) of dodecylisocyanide in 100 cc. of Skellysolve B were slowly added 16 g. (0.12 mole) of acetyl chloride in 100 cc. of Skellysolve B, using external cooling so that the temperature did not exceed 30°C. The product was allowed to stand several hours, was cooled to 15°C. and was filtered. Weight of product 32 g.; (1st crop); m.p. 148–149°C. A second crop of crystals formed; weight 14.5 g.; m.p. 145°C. Total weight yield: 46.5 g. or 73.4%. Recrystallizations of first and second crops yielded products melting at 151–152°C.

Anal. Calcd. for $C_{12}H_{25}N = C.CH_3COCl$:Cl, 12.9; N, 5.11; found, Cl, 12.6; N, 5.28.

Preparation of Dodecylisocyanide-Lauroyl Chloride Addition Product. To 24 g. (0.12 mole) of dodecylisocyanide in 100 ml. of Skellysolve B were added 23 g. (0.1 mole) of lauroyl chloride in 100 ml. of Skellysolve B, not allowing the temperature to rise above 30°C. The product was allowed to stand several hours, then was filtered, giving 38.0 g. of a crystalline product (yield 71.8%). M.p. 143–145°C. Recrystallization yielded a product, m.p. 148–149°C.

Anal. Calcd. as $C_{12}H_{25}NCC_{11}H_{23}COCl$: Cl, 8.58; N, 3.46; found Cl, 8.31; N, 3.66.

With Alkylating Agents: Several typical examples are listed below; analyses are summarized in Table IV.

Preparation of the Dodecylisocyanide-Lauroyl Bromide Addition Product. Twenty-four grams (0.12 mole) of dodecylisocyanide and 25 g. (0.12 mole) of lauryl bromide were dissolved separately in 100 ml. of Skellysolve B each. The two solutions were mixed together and refluxed 16 hrs. The mixture was then chilled to 15°C. until the crystals separated. These were filtered and dried. Weight, 15 g. The filtrate was refluxed another 8 hrs. and again chilled to 15°C., when a further crop of crystals was formed, weight, 10 g. Recrystallization from Skellysolve B yielded a crystalline material, m.p., 160–164°C.

Preparation of the Dodecylisocyanide-Benzyl Chloride Addition Product. Fifty grams of dodecylisocyanide (0.25 mole) in 100 cc. of Skellysolve B were added to 25 g. of benzyl chloride (0.2 mole) in 100 ml. of Skellysolve B, and the mixture refluxed 8 hrs. The solution was chilled to 15°C., and the crop of crystals was filtered off and washed with Skellysolve B. Weight, 10 g.; m.p., 153–156°C.

Preparation of Dodecylisocyanide-Ethyl Sulfate Addition Product. Diethyl sulfate (15.5 g., 0.1 mole) was slowly added with good agitation to 20 g. (0.1 mole) of dodecylisocyanide in 200 cc. of Skellysolve B, not allowing the temperature to rise above 25–30°C. The solution was allowed to stand for 8 hrs., then chilled to 15°C. overnight. The crystalline product was filtered off and washed with Skellysolve B. Weight of the product, 25 g. The product was gummy and was recrystallized from Skellysolve B. A crystalline product was obtained, m.p. 45–48°C.

Preparation of the Dodecylisocyanide-Methyl Chloride Addition Product. Methyl chloride was passed at atmospheric pressure into a solution of 50 g. of dodecylisocyanide in 200 cc. of Skellysolve B for 24 hrs., refluxing the solution at the same time. The solution on chilling to 15°C. yielded 45 g. of a crystalline material, m.p. 175–177°C.

A summary of the results of some of these addition reactions is given in Table IV.

Reactions of Isocyanide Addition Products with Base

Some typical decomposition experiments are given below.

Reactions of the Lauryl Bromide Addition Product with Sodium Hydroxide. Five grams of the crystalline dodecylisocyanide-lauryl bromide adduct were slurried into 200 cc. of warm water and the slurry

TABLE IV

Analyses of Alkylated Isocyanides

Product	Run No.	% Nitrogen		% Halogen		% Sulfur	
		Found	Theoretical[a]	Found	Theoretical[a]	Found	Theoretical[a]
$C_{12}H_{25}NC.C_{12}H_{25}Br$	1213A	3.10	3.16	17.70	17.83	......	......
$C_{12}H_{25}NC.C_6H_5CH_2Cl$	1213B	5.32	4.52	13.20	11.48	......	......
$C_{12}H_{25}NC(CH_3)_2SO_4$	1193	4.32	4.01	......	......	10.1	9.45
$C_{16}H_{33}NC.C_6H_5CH_2Cl$	....	......	......	11.80	9.42	......	......
$C_{12}H_{25}NC.CH_3Cl$	1212C	6.49	5.70	15.50	14.5	......	......
$C_{12}H_{25}NC.Et_2SO_4$	1228A	4.63	4.01	......	......	10.53	9.17

[a] All theoretical results are calculated on the basis of 1:1 adducts though it is realized that this is not the structure of our products in most cases. It should be noted though that in all cases the N:halogen or N:S ratio is essentially 1:1.

was made strongly alkaline with 50% base. An oily product separated immediately and was extracted with a 1:1 petroleum-ethyl ether solution. The ether extract was washed several times with water, dried over sodium sulfate, and evaporated to dryness. Four grams of material were obtained, which by titration for primary, secondary, and tertiary amines were found to consist of 89.8% secondary amine (calculated as didodecyl), 0% tertiary, and a trace of primary amine. An infrared spectrum on the product purified by vacuum distillation was identical with that from a known sample of didodecylamine.

Reaction of the Benzyl Chloride Addition Product with Base. Five grams of the benzyl chloride-dodecylisocyanide reaction product was treated with sodium hydroxide, and 4.1 g. of material were isolated which, by titration, were shown to be 45% primary amine (calculated as $C_{12}H_{25}NH_2$), 53% secondary amine (as $C_{12}H_{25}NHC_6H_5CH_2$), and 0% tertiary amine. The mixture was separated by distillation, and infrared spectra on the products were identical with those of known samples of dodecylbenzylamine and dodecylamine.

Reaction of the Methyl Chloride Addition Product with Sodium Hydroxide. Five grams of the methyl chloride-dodecylisocyanide addition product were reacted with base as above, and 3.8 g. of a product were obtained which, by titration and distillation, were shown to be 100% dodecylamine.

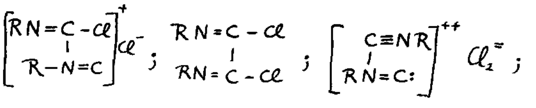
Reaction of Lauroyl Chloride Addition Product with Sodium Hydroxide. Five grams of the lauroyl chloride-dodecylisocyanide addition product were treated with base as above, obtaining 4.35 g. of a solid product which was neutral toward acid or base titrations and whose infrared spectrum was identical with a known sample of N-dodecyl-lauramide and had an m.p. of 45–46°C. Melting point of a known sample of N-dodecyl-lauramide was 46–47°C. A mixed melting-point showed no depression.

Discussion

Reactions with Halogens. The fatty isocyanides were found to react with chlorine, bromine, and iodine to yield products with the empirical formula $(RNCX)_n$. Reactions were run in an inert solvent and proceeded most readily with chlorine, least with iodine. The products are crystalline materials and soluble in water and appear to possess some surface activity. Nitrogen and halogen analyses and potentiometric titrations checked closely with the above empirical formula showing a 1:1 nitrogen:halogen ratio (Table III).

The behavior of the fatty isocyanides differs markedly from that reported for the corresponding aromatic or short-chain isocyanides, both of which gave alkyl iminocarbonyl halides, $RN = CCl_2$. Good evidence had been provided by the earlier workers for the 1:2 nitrogen:halogen ratio with the short-chain or aromatic adducts. This points to a basic difference in the chemical behavior of fatty isocyanides when compared with their low molecular weight homologues and analogues.

When considering the possible structures of the products formed from the fatty isocyanides, a definite complication arises since, by using the normal valencies for carbon, hydrogen, nitrogen, and halogens, it is impossible to write a simple monomeric formula. Rast camphor molecular-weight determinations were run and indicated that the compounds were probably dimeric or trimeric though exact figures could not be obtained because the nature of the halogen, *i.e.*, the relative amount of covalent or ionic character, could not be clearly established. Some of the possible structures are shown though at present we have insufficient evidence for distinguishing between them:

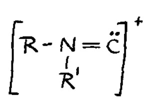


Reactions with Alkylating Agents. The reactions between isocyanides and alkyl halides or alkyl sulfates had not been investigated previously to any extent. It was found that fatty isocyanides reacted surprisingly readily with a large number of alkylating agents, such as benzyl chloride, methyl chloride, ethyl sulfate, or dodecyl bromide to form well-defined, salt-like solids resembling quaternary ammonium salts. Elemental analyses of some of these products indicated that a 1:1 addition took place:

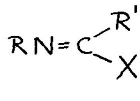
$$C_{12}H_{25}N = C + C_{12}H_{25}Br \longrightarrow C_{12}H_{25}N = C.C_{12}H_{25}Br.$$

With others the reaction was more complicated, and compounds were obtained which had the nitrogen and halogen or sulfur present in a 1:1 ratio but in excess for a simple 1:1 adduct, *i.e.*, a loss of carbon and/or hydrogen had occurred. Thus, with the dodecylisocyanide-dodecyl bromide adduct, 3.10 and 17.70% nitrogen and bromine, respectively, were found, which is close to theoretical for a 1:1 adduct. However, with the dodecylisocyanide-ethyl sulfate adduct, 4.63% nitrogen and 10.53% sulfur were found; theoretical are 4.01% and 9.17%, respectively, that is, the theoretical for both nitrogen and sulfur is lower by an equal amount (13%) than the found values.

We considered that several different types of structures could be formed during the reaction between alkylating agent and fatty isocyanides. The two simplest are based on attack on the nitrogen yielding a "quaternized isocyanide" (structure A) and attack on the divalent carbon yielding structure B.



(A)



(B)

Support for these types of structures was obtained by studying the reactions of the alkylated isocyanides with sodium hydroxide. Treatment of an aqueous solution of the alkylated isocyanides with sodium hydroxide yielded primary and/or secondary amines and sodium formate. The alkyl group of the primary amines formed corresponded to that of the starting isocyanide; the secondary amines contained one alkyl group from the isocyanide, the second from the alkylating agent, *e.g.*, the dodecyl isocyanide-benzyl chloride adduct yielded dodecylamine and dodecylbenzylamine. Some representative results are shown in Table V.

TABLE V

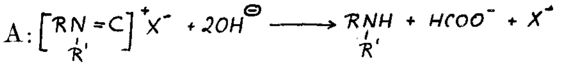
Decomposition of Alkylated Isocyanides

Number	Isocyanide $R'N=C$	Alkylating agent $R''X$	% Primary amine as $R'NH_2$	% Secondary amine as $R'R''NH$
1	$C_{12}H_{25}N=C$	$C_{12}H_{25}Br$	trace	90
2	$C_{12}H_{25}N=C$	CH_3Cl	100	nil
3	$C_{12}H_{25}N=C$	$(CH_3)_2SO_4$	55	33
4	$C_{12}H_{25}N=C$	$C_6H_5CH_2Cl$	45	53
5	$C_{16}H_{33}N=C$	$C_6H_5CH_2Cl$	62	37
6	$C_{18}H_{37}N=C$	$C_6H_5CH_2Cl$	64	34

From the results shown in Table V a considerable variation is noted in the relative amounts of the decomposition products formed. No tertiary amines were ever found but in Experiment 3 an unidentified neutral material was formed. Experiments 4 to 6 show a definite shift in the primary to secondary amine ratio as the molecular weight of the isocyanide radical increased.

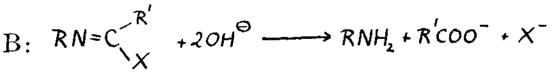
The possible source of the decomposition reaction is shown below, using the simple two forms proposed earlier:

From A:



$$\left[\begin{matrix}RN=C\\ |\\ R'\end{matrix}\right]^+X^- + 2OH^- \longrightarrow \begin{matrix}RNH\\ |\\ R'\end{matrix} + HCOO^- + X^-$$

From B:

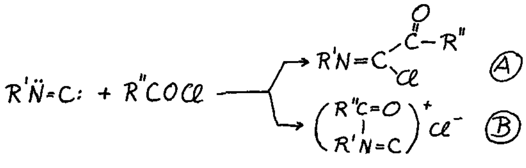


$$RN=C\begin{matrix}\diagup R'\\ \diagdown X\end{matrix} + 2OH^- \longrightarrow RNH_2 + R'COO^- + X^-$$

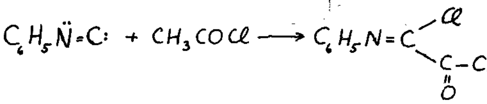
The analytical and the decomposition data from the dodecylisocyanide-dodecylbromide adduct check well with structure A. With other adducts, where decomposition gave mixtures of primary and secondary amines coupled with high but equivalent nitrogen and chlorine values, it is believed that attack by the alkylating agent is competitive at the nitrogen and the divalent carbon, giving structures of type A or B, respectively. Interaction of these structures to form higher molecular-weight products containing higher concentrations of nitrogen and halogen or sulfur also seem to occur and would fit our analytical data.

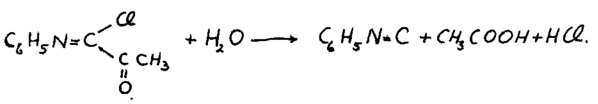
Although it is not yet possible to draw a precise chemical picture of the product formed by the reaction of fatty isocyanides and alkylating agents, this reaction represents a previously uninvestigated area of isocyanide chemistry with some interesting theoretical and practical implications. The isolation of secondary amines from the base decomposition reaction is a very definite indication that an attack on the nitrogen of the isocyanide group is possible by an electrophillic agent. It is believed that these are the first reported samples of such reactions by isocyanides.

Reactions with Acid Chlorides. When an isocyanide reacts with an acid chloride, the acyl group can become attached at either the divalent carbon or the nitrogen:



Nef had found that short-chain or aromatic isocyanides react with acid chlorides to form alkyl amido chlorides (structure A), which on treatment with base, decompose to the starting isocyanide (2, 3):



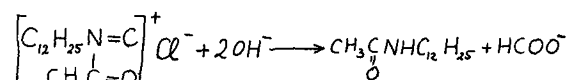


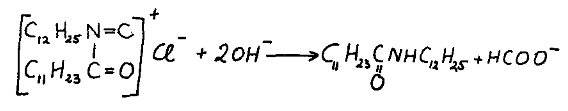
Fatty isocyanides were found to react readily with acid chlorides, such as acetyl chloride, to form products which on analysis indicated that a 1:1 addition had occurred. Decomposition experiments with aqueous base led to the formation of the corresponding N-alkylamides, which were identified by infrared, independent synthesis and mixed melting points.

The behavior of the fatty isocyanide acid chloride adducts on decomposition differs markedly from that of the corresponding short-chain or aromatic isocyanide derivatives. For the latter the alkylamido chloride structure (A) had been demonstrated by Nef.

$$\text{Structure B}-\left[\begin{matrix}R''-C=O\\ |\\ R'-N=C\end{matrix}\right]^+Cl^-$$

is proposed by us for the fatty isocyanide-acid chloride adducts. This view is supported by the salt-like properties of these compounds, their water solubility and surface activity, and the decomposition experiments with base, which are summarized below:





These reactions furnish further proof that, with fatty isocyanides, attack on the nitrogen plays an important role, which is in strong contrast to the behavior reported for the short-chain and aromatic isocyanides.

Properties. Some very interesting and practical properties are common to the three types of fatty isocyanide adducts discussed in this paper. They are all crystalline materials with varying degrees of water solubility. Their aqueous solutions have surface activity with definite foaming, wetting, and suspending propensity. All have a low oral toxicity and have good bactericidal activity against both gram positive and gram negative organisms.

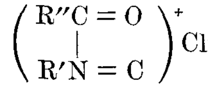
Summary and Conclusions

Three new reactions of fatty isocyanides were discovered and are summarized below.

a) With halogens, products of the type $(RNCX)_n$ were isolated; this differs from the aromatic or short-chain isocyanides which form $RNCX_2$ under similar conditions.

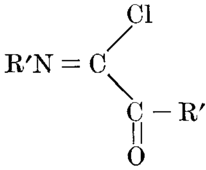
b) With alkylating agents, such as methyl chloride or ethyl sulfate, the fatty isocyanides formed quaternary-like surface-active derivatives. This type of reaction has not been reported previously.

c) With acid chlorides, the following type of structures were formed:



$$\left(\begin{array}{c} R''C = O \\ | \\ R'N = C \end{array}\right)^{+} Cl^{-}$$

this differs from the aromatic or short-chain isocyanides which are reported to form



under similar conditions.

In reactions of fatty isocyanides with alkylating agents and with acid chlorides evidence was presented to indicate that addition occurred at the nitrogen of the isocyanide group. These are the first examples in the literature of such reactions by isocyanides.

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Identification of Rutin and Isoquercitrin in Cottonseed[1]

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BOATNER (1) has stated that, although many of the pigments of the cotton flower have been isolated and identified as flavonoid compounds, there is little evidence that these or related pigments occur in the seed. Gurevich (2) reported microchemical tests which suggested the presence of flavones or closely related pigments in the epidermal cells of the cotyledons above the palisade cells. Goldvoskii (3), also Boatner (1), pointed out however that before one could draw any definite conclusions concerning the presence of flavonoid compounds in cottonseed, one would need to supplement the microchemical tests with the isolation and identification of these pigments. This present paper reports the separation and identification of two such pigments.

Paper chromatography of isopropyl alcohol extracts of crushed, delinted cottonseed (kernel and hull) in our laboratory has revealed the presence of at least six pigments which preliminary tests indicate to be flavonoid compounds. We have therefore undertaken studies to identify the individual flavonoid pigments present in the cottonseed. Two of these have been separated and identified as isoquercitrin (quercetin-3-glucoside) and rutin (quercetin-3-rhamnoglucoside). Quercetin is 3,3′,4′,5,7-pentahydroxyflavone.

Experimental

Extraction and Preliminary Separation of Pigments. Delinted cottonseed (1 kg.), obtained through the courtesy of the National Cottonseed Products Association Inc. from a cottonseed oil producer in Lubbock, Tex., was crushed with a food blendor and extracted in Soxhlet extractors with a total of 3 l. of 85% isopropyl alcohol-water until the solvent in contact with the seed no longer gave a bright yellow color. This extract was concentrated to 200 ml. *in vacuo*, then transferred to a beaker. To it were added 100 ml. of hot distilled water, with stirring, so that the oil collected together. The mixture was kept in the refrigerator (5–10°C.) for 3 hr. The lower layer was decanted and concentrated to 25 ml.; 50 ml. of 95% ethyl alcohol were added to the concentrate. The resulting solution was streaked onto 24 sheets of Whatman No. 3 MM chromatography paper, 18½ in. × 22¼ in., and the chromatograms were developed for 18–20 hr. in n-butyl alcohol-acetic acid-water (6:1:2 v./v.). Three major zones that fluoresced brown in long wavelength ultraviolet light (3660 Å) were observed. Rutin was one of the compounds present in the second brown zone (called zone 2) from the top, with an approximate R_f value of 0.39. Isoquercitrin was present in zone 3, with R_f value of approximately 0.59.

Separation of Rutin. Zone 2 from the n-butyl alcohol-acetic acid-water chromatography was cut from each paper, minced, and extracted in a Soxhlet extractor with 95% ethyl alcohol. The extract was concentrated *in vacuo* to 25 ml. and streaked onto sheets of Whatman 3 MM paper. The chromatograms were developed in 15% acetic acid-water for 5–6 hr. Three brown fluorescent zones resulted from the one original zone 2. The brown fluorescent zone with the lowest R_f value (about 0.61) of these three contained the rutin. Identification studies on the compounds of the other two zones are in progress.

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