1H,2H-2-oxo-imidazolo-[4,5-*b*]pyridine (XIX). To a stirred solution of 10.0 grams of the above product in 200 ml. of water was added, dropwise, a solution of 5.0 grams of sodium acetate in 25 ml. of water while cooling with an ice bath. The precipitate of crystalline XIX (7.32 grams) was collected, washed with water, and dried. Recrystallized material (needles from water) melted at 274–75° C. Anal. Calcd. for $C_6H_5N_3O$: C, 53.33; H, 3.73; N, 31.10, MW, 135. Found: C, 53.43; H, 3.84; N, 30.92; MW, 142.

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New *N*-Alkylimides

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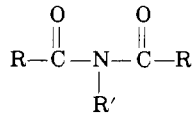
The preparation and physical properties of six new *N*-alkylimides are described. *N*-Alkylamides, which are formed as by-products in these preparations, were removed by column chromatography.

N-ALKYLIMIDES have been prepared by acylation of primary alkylamines (*4*) or *N*-alkylamides (*1*) with anhydrides in the presence of acid catalysts. A less convenient method involves the reaction of nitriles with carboxylic acids (*2, 3, 5*).

The authors chose to use the anhydride acylation of alkylamine hydrochlorides. In most cases *N*-alkylamides, which result as by-products, were removed by chromatography on Florisil. Purity of the *N*-alkylimides (listed in Table I) was determined by gas-liquid chromatography on a 6-mm. × 2-meter column packed with 20% Carbowax 20M on Chromosorb W. The absence of strong characteristic N-H absorption in the 3300-cm.$^{-1}$ region eliminated the likelihood of significant amounts of *N*-alkylamide con-

Table I. *N*-Alkylimides



Compound	R	R′	B.P., °C./Mm.	n_D^{20}	Empirical Formula	Analysis, %[a] C Calcd.	C Found	H Calcd.	H Found	N Calcd.	N Found
I[b,c]	CH_3	CH_3	105–06/39[d]	1.4550	$C_5H_9NO_2$						
II	CH_3	C_2H_5	96/26[e]	1.4499	$C_6H_{11}NO_2$	55.80	56.00	8.58	8.40	10.84	10.65
III[c]	CH_3	n-C_6H_{13}	91/1–3[c]	1.4580	$C_{10}H_{19}NO_2$	64.83	65.03	10.34	10.50	7.56	7.50
IV[c]	C_2H_5	C_2H_5	114/26[d]	1.4544	$C_8H_{15}NO_2$	61.12	61.00	9.62	9.45	8.91	8.76
V[b,c]	C_2H_5	CH_3	108–10/28[f]	1.4462	$C_7H_{13}NO_2$						
VI[b]	n-C_3H_7	CH_3	133/25.5[e]	1.4567	$C_9H_{17}NO_2$						
VII[c]	n-C_3H_7	n-C_3H_7	143/24[f]	1.4532	$C_{11}H_{21}NO_2$	66.29	66.17	10.62	10.47	7.03	7.07
VIII[c]	n-C_4H_9	CH_3	103–04/1[e]	1.4586	$C_{11}H_{21}NO_2$	66.29	66.28	10.62	10.46	7.03	6.93
IX[c]	CH_3	n-C_3H_7	106–07/26[e]	1.4510	$C_7H_{13}NO_2$	58.72	58.81	9.15	9.11	9.78	9.64

[a] Elemental analyses by Galbraith Laboratories, Knoxville, Tenn. [b] See (*1*). [c] Chromatographed on Florisil. [d] Distilled through 30-cm. Oldershaw column. [e] Distilled through 30.5-cm. Scanco semimicro concentric tube column. [f] Distilled through 13-cm. Vigreux column.

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taminants. Since no elemental analyses (see Table I) were obtained for known compounds I, V, and VI, a sample NMR spectrum was obtained for the lowest molecular weight compound (I). This spectrum (60 mc.) revealed two sharp singlets at 2.29 and 3.13 p.p.m., which upon integration gave the expected 2 to 1 ratio of acyl to *N*-alkyl protons.

EXPERIMENTAL

***N*-*n*-Propyldiacetimide (IX).** A mixture of 164 grams (1.61 moles) of acetic anhydride and 35.0 grams (0.37 mole) of *N*-*n*-propylamine hydrochloride was stirred for 6 hours with heating by means of an oil bath maintained at 160° C. A slow stream of dry nitrogen was passed through the mixture for 1 hour to expel hydrogen chloride. The excess anhydride was removed under reduced pressure and the residue was distilled to obtain 37.5 grams of a liquid (b.p. 116° C. at 40 mm. of Hg). A gas chromatogram indicated that this distillate contained a small amount of a less volatile component. Redistillation of the material through a 30.5-cm. Scanco semimicro concentric tube column gave 29.3 grams of a liquid (b.p. 96.5–100° C. at 19 mm. of Hg) which still contained some of the second component. This material was dissolved in 300 ml. of *n*-hexane and passed through a 2 × 50 cm. column of Florisil. An additional 300 ml. of *n*-hexane was used to elute the imide from the column. Concentration of the eluate and distillation of the residue gave 18.0 grams (34.4%) of a colorless liquid [b.p. 106–07° C. at 26 mm. of Hg, $\lambda_{max}^{liquid\ film}$ 1670 to 1710 cm.$^{-1}$ (imide C=O)].

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Infrared Spectra and Synthesis of Some Acetophenone Derivatives

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The acetophenone derivatives were synthesized by the Friedel-Crafts acylation of the symmetrical trialkylbenzenes. The nitroacetophenones were prepared by nitration of the ketone in acetic acid–acetic anhydride solution. Nine organic compounds heretofore unreported were prepared: 2,4,6-triisopropyl-α-methylbenzyl acetate (m.p., 62.5–63.5); 2,4,6-triisopropyl-α-methylbenzyl alcohol (m.p., 92–93); 2′,4′,6′-triisopropyl-3′-aminoacetophenone (m.p., 109–10); 2′,4′,6′-triisopropyl-2-bromoacetophenone (m.p., 53–54); 2′,4′,6′-triisopropyl-3′-nitroacetophenone (m.p., 110–12); 2′,4′,6′-triisopropylbutyrophenone (m.p., 41–42); 2′,4′,6′-triisopropyl-3′-nitrobutyrophenone (m.p., 101.5–2.5); 2′,4′,6′-triisopropylpropiophenone (m.p., 87–88); and 2′,4′,6′-triisopropyl-3′-nitropropiophenone (m.p., 113–14). The effect of ring substituent groups upon the infrared absorption frequency assigned to the carbonyl group of the acetophenones is discussed.

THE ACETOPHENONE derivatives were synthesized by the Friedel-Crafts acylation of the symmetrical trialkylbenzenes. The nitroacetophenones were prepared by nitration of the ketone in acetic acid–acetic anhydride solution. Nine organic compounds heretofore unreported were prepared: 2,4,6-triisopropyl-α-methylbenzyl acetate, 2,4,6-triisopropyl-α-methylbenzyl alcohol, 2′,4′,6′-triisopropyl-3′-aminoacetophenone, 2′,4′,6′-triisopropyl-2-bromoacetophenone, 2′,4′,6′-triisopropyl-3′-nitroacetophenone, 2′,4′,6′-triisopropylbutyrophenone, 2′,4′,6′-triisopropyl-3′-nitrobutyrophenone, 2′,4′,6′-triisopropylpropiophenone, and 2′,4′,6′-triisopropyl-3′-nitropropiophenone.

In addition, three known compounds were prepared. The effect of ring substituent groups upon the infrared absorption frequency assigned to the carbonyl group of the acetophenones was studied. The observed absorption frequencies assigned to the carbonyl group of the acetophenones indicated that this group was not in the plane of the aromatic ring.

SYNTHESIS

The best general method found for the synthesis of the acetophenones was a Friedel-Crafts acylation (*16*) of the symmetrical trialkylbenzenes. The melting point and analytical data of the compounds described in this article may be found in Table I. The differential thermal analysis curves for the three nitro compounds are shown in Figures 1, 2, and 3. All attempts to substitute 1,3,5-tri-*tert*-butylbenzene and 1,2,4,6-tetraisopropylbenzene by this method failed, only rearranged or dealkylated products being obtained.

The nitro group was introduced by nitration with concentrated nitric acid in acetic acid and acetic anhydride (*5*). Excellent yields of the desired products were obtained. Nitration with other nitration reagents, red fuming nitric acid (*9*), mixtures of concentrated nitric acid and concentrated sulfuric acid (*11*), and potassium nitrate and concentrated sulfuric acid (*17*), resulted in rearranged, oxidized, or dealkylated products. CAUTION! Nitration of organic