trated sulfuric acid and in ether, insoluble in dilute hydrochloric acid and sodium hydroxide, and formed a hydrazone (not analyzed) rapidly with 2,4-dinitrophenylhydrazine in perchloric acid.[6]

Anal. Calc'd for $C_{30}H_{59}NO_2$: C, 77.35; H, 12.77; N, 3.01. Found: C, 77.87; H, 12.91; N, 2.85.

N-(2-oxoheptadecyl)-N-methylpalmitamide (II). The reaction, as above, of sarcosine and palmitoyl chloride gave 37.1% of II, melting sharply at 82°, after recrystallizations from 85% ethanol, ethyl acetate, and from acetone-water mixtures. The properties of II were analogous to those described for I.

Anal. Calc'd for $C_{34}H_{67}NO_2$: C, 78.24; H, 12.94; N, 2.68. Found: C, 78.53; H, 12.99; N, 2.85.

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(6) Neuberg, Grauer, and Pisha, *Anal. Chim. Acta*, **7**, 238 (1952).

The Mono-2,4-dinitrophenylhydrazones of Diacetyl and Glyoxal

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Diacetyl monodinitrophenylhydrazone can easily be prepared in aqueous hydrochloric acid, in chloroform-acetic acid, or by refluxing an alcoholic solution of the components. In order to avoid osazone formation it is advisable to use an excess of diacetyl. The monodinitrophenylhydrazone shows an infrared band at 5.97 μ (keto group) and an absorption maximum at 350 mμ. This maximum is at a wave-length 13 mμ shorter than in acetone dinitrophenylhydrazone (λ_{max} 363 mμ). A similar effect of an adjacent keto group was previously observed in the spectra of methyl propyl ketone dinitrophenylhydrazone and hexane-2,3-dione monodinitrophenylhydrazone.[1] In distinction from the dinitrophenylhydrazones of other ketones that of diacetyl dissolves with a red color in alcoholic sodium hydroxide solution.

Neither method mentioned above could be used for the preparation of glyoxal monodinitrophenylhydrazone.[2] In every case only glyoxal dinitrophenylosazone was obtained, although an excess of glyoxal was used. It was found, however, that an alcoholic solution of glyoxal reacts very readily with dinitrophenylhydrazine at room temperature to give a mixture of the osazone and the monodinitrophenylhydrazone. Since the latter is soluble in ethanol, it can easily be separated from the completely insoluble osazone. The great reactivity of glyoxal is best illustrated by a comparison with acetone which, under similar conditions, gives only 12% dinitrophenylhydrazone. The glyoxal monodinitrophenylhydrazone shows an infrared band at 5.95 μ (aldehyde group). Since it exhibits maximum absorption at 343 mμ, the shift to shorter wavelengths in comparison with formaldehyde dinitrophenylhydrazone (λ_{max} 347 mμ) amounts only to 4 mμ.

EXPERIMENTAL[3]

Diacetyl monodinitrophenylhydrazone. (*a*). A solution of 0.1 ml. of diacetyl (98 mg.) in 5 ml. of ethanol was added to a suspension of 113 mg. of 2,4-dinitrophenylhydrazine in 68 ml. of chloroform and 11.3 ml. of glacial acetic acid. After standing overnight, the clear yellow solution was washed to neutrality, dried, and evaporated. The residue was chromatographed on 5 g. of neutral aluminum oxide Woelm and eluted with hexane-benzene 1:1. Two recrystallizations from chloroform-ethanol gave small yellow prisms, m.p. 176–177°; λ_{max} 350 mμ (ϵ 26,260); infrared band at 5.97 μ. The substance dissolved with red color in alcoholic NaOH. Large crystals sometimes were orange in color.

Anal. Calc'd for $C_{10}H_{10}N_4O_5$: N, 21.05. Found: N, 21.22.

(*b*). A mixture of 113 mg. of dinitrophenylhydrazine, 0.1 ml. of diacetyl, and 15 ml. of ethanol was refluxed for two hours. The orange crystals which separated on cooling were washed with cold ethanol, dried, and chromatographed as described above. After recrystallization from chloroform-ethanol, 79 mg. of yellow prisms were obtained which proved to be identical with the product from procedure (*a*).

(*c*). Diacetyl (58.8 mg.) in 6 ml. of 2 *N* HCl was added to 67.6 mg. of dinitrophenylhydrazine in 16.9 ml. of 2 *N* HCl. The yellow crystals (89.5 mg.) were filtered after 5 minutes, washed with water, dried, and recrystallized from chloroform-ethanol; m.p. 176–177°.

Glyoxal monodinitrophenylhydrazone. A suspension of 200 mg. of dinitrophenylhydrazine in 25 ml. of ethanol and 1 ml. of glyoxal was stirred for two hours. The dinitrophenylhydrazine dissolved and the orange glyoxal dinitrophenylosazone precipitated. The mixture was kept in the refrigerator overnight, filtered, and diluted with 25 ml. of water. Upon renewed cooling, light-orange crystals appeared which were washed with 40% ethanol and dried (yield 113 mg.). They gave a red-brown color with alcoholic NaOH and melted at 190°; λ_{max} 343 mμ (ϵ 24,210); infrared band at 5.95 μ.

Anal. Calc'd for $C_8H_6N_5O_4$: N, 23.53. Found: N, 23.63.

This dinitrophenylhydrazone could neither be chromatographed nor recrystallized satisfactorily. A sample was dissolved in hot ethanol and cooled. Upon addition of a few drops of concentrated hydrochloric acid, glyoxal dinitrophenylosazone precipitated almost immediately.

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(1) E. A. Braude and E. R. H. Jones, *J. Chem. Soc.*, 498 (1945).

(2) Glyoxal monophenylhydrazone was described by H. O. L. Fischer and C. Taube, *Ber.*, **59**, 856 (1926).

(3) The melting points were determined on a Fisher-Johns melting point block and are uncorrected. The ultraviolet spectra were taken in chloroform on a Cary recording spectrophotometer, the infrared spectra on a Baird double-beam spectrophotometer (KBr wafer technique). The microanalyses were carried out by Huffman Microanalytical Laboratories, Wheatridge, Colo.