Elmer model 12C infrared spectrometer with rock salt optics calibrated against water vapor and ammonia,[9] using the same fixed rock salt cell of thickness about 0.025 mm.

(NOTE: The complete spectrum will be available through the American Petroleum Institute.—Ed.)

[9] R. A. Oetjen, C. L. Kao and H. M. Randall, *Rev. Sci. Instruments*, **13**, 515 (1942).

JOINT CONTRIBUTION FROM THE DEPARTMENT OF PHYSICAL
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Trinitrobenzene Complexes of Various Indole Compounds[1]

BY LOWELL E. WELLER, THEODORE L. REBSTOCK AND HAROLD M. SELL

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We have prepared a number of new 1,3,5-trinitrobenzene complexes of various indole derivatives for use in characterization of these substances.

The properties of these derivatives, prepared as previously described,[2,3] are summarized in Table I.

TABLE I

PROPERTIES OF 1,3,5-TRINITROBENZENE ADDUCTS OF INDOLE COMPOUNDS

Trinitrobenzene derivative of	M.p., °C.	Color	Formula	Dumas nitrogen, % Calcd.	Dumas nitrogen, % Found
Isatin (2,3-indolinedione)	116	Light brown	$C_{14}H_8O_8N_4$	15.6	15.7
Oxindole (2,3-dihydro-2-oxoindole)	123	Yellow	$C_{14}H_{10}O_7N_4$	16.2	16.3
Indole-3-aldehyde	144.5	Yellow	$C_{15}H_{10}O_7N_4$	15.6	15.8
Skatol (3-methylindole)[a]	184	Red	$C_{15}H_{12}O_6N_4$	16.3	16.4
Tryptophol (indole-3-ethanol)	109.5	Orange	$C_{16}H_{14}O_7N_4$	15.0	15.1
β-(Indole-3)-propionic acid	135	Orange	$C_{17}H_{14}O_8N_4$	13.9	14.1
2-Methylindole-3-acetic acid	208	Red	$C_{17}H_{14}O_8N_4$	13.9	13.8
β-(Indole-3)-*n*-butyric acid	152	Red	$C_{18}H_{15}O_8N_4$	13.5	13.4
2,3-Diphenylindole	160	Red	$C_{26}H_{18}O_6N_4$	11.6	11.6

[a] van Romburgh[4] reported the m.p. as 183°, but gave no analysis.

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[1] Journal Article No. 1307 from the Michigan Agricultural Experiment Station, East Lansing. This work was supported in part by the Horace H. Rackham Research Endowment of Michigan State College.

[2] C. T. Redemann, S. H. Wittwer and H. M. Sell, THIS JOURNAL, **73**, 2957 (1951).

[3] J. J. Sudborough, *J. Chem. Soc.*, **109**, 1339 (1916).

[4] M. P. van Romburgh, *Rec. trav. chim.*, **14**, 67 (1895).

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Synthesis of β-Acetamido-β,β-dicarbethoxypropionaldehyde and DL-Aspartic Acid[1]

BY OWEN A. MOE AND DONALD T. WARNER

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Previous publications[2-4] from this Laboratory have disclosed the syntheses of γ-acetamido-γ,γ-dicarbethoxybutyraldehyde and other closely related aldehydo compounds, and the utilization of these aldehyde intermediates in the production of diverse amino acids. This report concerns the synthesis of the lower aldehydo homolog, namely, β-acetamido-β,β-dicarbethoxypropionaldehyde.

Ethyl allylacetamidomalonate[5] was prepared by the allylation of the corresponding malonic ester with allyl bromide. The action of ozone on the allyl derivative yielded the ozonide which in turn was converted by reductive cleavage to the desired aldehydo compound, a crystalline product melting at 80–81°. The β-acetamido-β,β-dicarbethoxypropionaldehyde was further characterized as the 2,4-dinitrophenylhydrazone melting at 117–119°. Oxidation of the aldehyde by alkaline permanganate yielded the corresponding acid diester melting at 147–148°. Hydrolysis by refluxing in concentrated hydrochloric acid converted the acid diester to DL-aspartic acid.

Experimental

β-Acetamido-β,β-dicarbethoxypropionaldehyde.—Ethyl allylacetamidomalonate[5] (30 g.) was dissolved in 50 cc. of absolute ethanol. The resulting alcoholic solution was treated with ozone at 0–5° for a period of 5 hours. The water-clear alcoholic solution of the ozonide was diluted with absolute ethanol to a total volume of 200 cc. Five per cent. palladium-on-charcoal (2 g.) was added and the reductive cleavage[6] of the ozonide was accomplished at an initial pressure of 30 p.s.i. After 90 minutes the reduction was complete. The reaction mixture was refrigerated overnight and then the catalyst was removed by filtration. The water-clear filtrate gave no test for ozonide and it was concentrated *in vacuo*. A solid crystalline cake resulted which possessed a pungent odor of formaldehyde. The yield of crude aldehydo compound melting at 65–67° was 29.3 g. Purification by crystallization from ethanol increased the melting point to 80–81°.

Anal. Calcd. for $C_{11}H_{17}O_6N$: C, 50.93; H, 6.61; N, 5.40. Found: C, 50.58; H, 6.39; N, 5.87.

The 2,4-dinitrophenylhydrazone of β-acetamido-β,β-dicarbethoxypropionaldehyde was prepared following the conventional procedure and after purification by crystallization from ethanol melted at 117–119°.

Anal. Calcd. for $C_{17}H_{21}O_9N_5$: C, 46.44; H, 4.82; N, 15.94. Found: C, 46.33; H, 4.41; N, 15.85.

The Diester Acid.—A water solution containing 3.3 g. of the aldehydo compound was mixed with 5–10 drops of 10% aqueous sodium hydroxide. The resulting clear solution was treated with portions of a saturated aqueous solution of potassium permanganate until a slight excess was indicated. The oxidation of the aldehydo compound was very rapid and it was necessary to cool the reaction mixture in a cold water-bath in order to avoid an increase in the reaction temperature. After the oxidation was complete, the manganese dioxide was removed by filtration and the mixture acidified to *p*H 3. The excess permanganate was

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[1] Paper 126, Journal Series, General Mills, Inc., Research Department.

[2] O. A. Moe and D. T. Warner, THIS JOURNAL, **70**, 2763 (1948).

[3] D. T. Warner and O. A. Moe, *ibid.*, **70**, 2765 (1948).

[4] D. T. Warner and O. A. Moe, *ibid.*, **70**, 3918 (1948).

[5] N. F. Albertson, *ibid.*, **68**, 450 (1946).

[6] H. S. Mason, *ibid.*, **67**, 420 (1945).