densation of water from the air space over the diluent have been applied. The corrected dilution heats are listed in Table I, and the derived values of the apparent and partial molal heat content of the solute and the partial molal heat content of the solvent are given in Table II. The average deviation of the experimental points from a smooth curve drawn through them is ±3 joules per mole, if the two runs at the lowest concentrations are omitted. One run recorded in the original paper has been discarded.

The chief effect of the application of these corrections is on the extrapolation to infinite dilution, which is an arbitrary procedure at best. Thus the relatively large changes in the values of $\Phi_H - \Phi_H^0$ correspond to considerably smaller changes in the actual heats of dilution.

STERLING CHEMISTRY LABORATORY
YALE UNIVERSITY
NEW HAVEN, CONN.
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Preparation of β-Alanine Methyl Ester[1]

BY HARRY H. WEINSTOCK, JR.,[2] AND EVERETTE L. MAY[2]

The use of β-alanine ester in the synthesis and "partial synthesis" of pantothenic acid[3] has made desirable a rapid and convenient method for the preparation of that substance. Because of the instability of the free ester,[4] quantities of the material cannot be kept on hand, and must be made up immediately before use. We have found the preparation of the ester from the ester hydrochloride to be inconvenient and the yields poor.

Kuhn and Brydowna[5] have prepared a number of α-amino acid esters by esterification with diazomethane. This method has been applied to the preparation of the methyl ester of β-alanine, the product being finally obtained in high purity and good yield. The comparative instability of the final product has necessitated working out certain conditions which must be followed closely for good results.

Experimental

To 4 g. of β-alanine was added one-half of an ether solution of diazomethane freshly prepared from 20 g. of nitrosomethylurea.[6,7] After 1 cc. of water had been added, the reaction mixture was stirred mechanically until the ether solution had become colorless (fifteen to thirty minutes). The remainder of the ether solution of diazomethane was added and stirring continued another two hours. At this time gas evolution was scarcely noticeable, and only a small amount of semi-solid material remained undissolved. Omission of mechanical stirring was found to reduce the yield considerably. The ether solution was decanted and dried over anhydrous sodium sulfate in the refrigerator for one hour. The ether solution was filtered and distilled from a 10 cc. "spitzkolben" under water pump pressure at a water-bath temperature not exceeding 35°. The flask containing the crude amino acid ester was removed from the bath, the bath was warmed quickly to 75–80°, the distillation flask was again immersed and the ester distilled under vacuum utilizing a good condensing system and well-cooled (0°) receivers. A fore-run boiling 40–49° (12 mm.) (0.55 g.) was collected. The main fraction of ester (2.9 g.) came over at 50–52° (12 mm.), 54–55° (13 mm.). Conversion of the ester in the fore run to the hydrochloride indicated an additional 0.2 g. of ester, making the total yield of β-alanine methyl ester 67%.

Identification was effected by heating a small portion of the ester for one hour with water, evaporation almost to dryness and addition of 95% ethanol. The product which crystallized out melted at 194–195°. A mixed melting point with an authentic specimen of β-alanine showed no depression. The ester was further characterized through its chloroplatinate which melted, without recrystallization, at 192°. After one recrystallization from 90% ethanol–ether the m. p. was 193°. The melting point of the crude chloroplatinate is indicative of the purity of the ester.

We are grateful for the suggestions of Dr. Roger J. Williams and Dr. Donald Price who, at different times, directed this research, and to Robert Eakin and Herschel K. Mitchell for preliminary work at Oregon State College.

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[2] Present address: Nopco Research Laboratories, National Oil Products Company, Harrison, New Jersey.

[3] Woolley, Waisman and Elvehjem, THIS JOURNAL, **61**, 977 (1939); Williams, *Science*, **89**, 486 (1939); Williams, Mitchell, Weinstock and Snell, THIS JOURNAL, **62**, 1784 (1940); Stiller, Harris, Finkelstein, Keresztesy and Folkers, *ibid.*, **62**, 1785 (1940).

[4] Abderhalden, *Z. physiol. Chem.*, **85**, 118 (1913).

[5] Kuhn and Brydowna, *Ber.*, **70**, 1333 (1937).

[6] Gatterman and Wieland, "Laboratory Methods of Organic Chemistry," Macmillan Co., New York, N. Y., p. 272.

[7] The ethereal solution was allowed to stand over a few potassium hydroxide pellets for fifteen minutes.

DEPARTMENT OF CHEMISTRY
OREGON STATE COLLEGE
CORVALLIS, OREGON
NOPCO RESEARCH LABORATORIES
NATIONAL OIL PRODUCTS COMPANY
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p-Nitrobenzoyl-*d*(−)- and *p*-Aminobenzoyl-*d*(−)-glutamic Acid

BY HARRY C. WINTER

Some contradictions exist in the literature as to the properties of the *p*-nitrobenzoyl derivatives of the optical isomers of glutamic acid. J. Van der Scheer and K. Landsteiner[1] reported the preparation of *p*-nitrobenzoyl-1(+)-glutamic acid and

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[1] Van der Scheer and Landsteiner, *J. Immunol.*, **29**, 371 (1935).