Preparation of Sodium [$^2H_1$]Acetate with Exceptional Isotopic Purity

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Summary Sodium [$^2H_1$]acetate of >99% isotopic purity was obtained from bromoacetic acid in a mild debromination procedure employing zinc dust.

IN connection with some n.m.r. correlation time studies, it was necessary to prepare sodium [$^2H_1$]acetate free of isotopic impurities. Previous syntheses[1] were either lengthy and complex, frequently without statement of isotopic composition or gave mixtures with varying extents of deuteriation of the acetate unit. We here report the synthesis from bromoacetic acid of sodium [$^2H_1$]acetate with an isotopic purity of $>99\%$ by a simple procedure analogous to that wherein Overberger and Cho[2] obtained optically active 3-methylpentanoic acid by debrominating the diastereoisomeric 2-bromo-derivatives.

Bromoacetic acid (0·08 mol), twice exchanged with D_2O, was stirred overnight with an excess of zinc dust in D_2O (room temperature). The insoluble zinc products were separated off, the solution was acidified to pH 1 with the relatively involatile conc. H_2SO_4 and then carefully distilled collecting the fraction boiling at 101·5—102·2° (1 atmos.), which was shown to be free of bromide and sulphate anions. The distillate after neutralisation, evaporation at reduced pressure, and prolonged drying of the resultant solid at 150° *in vacuo*, gave anhydrous sodium [$^2H_1$]acetate (60% yield), m.p. 334—336°. Mass spectrometric assay (20 and 70 eV) showed $<0{\cdot}5\%$ each of sodium [$^2H_2$]acetate and sodium acetate, whilst the ^1H n.m.r. spectra (D_2O solution) at 60 and 100 MHz comprised three lines of almost equal intensity (J 2·19 Hz) centred at δ 1·89 p.p.m. from internal DSS.

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