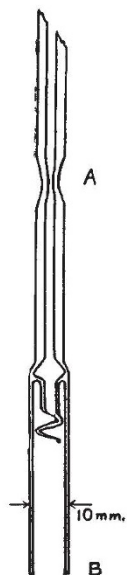


## Microsynthesis of C<sup>14</sup>-Labelled Ethyl Iodide

CARBON dioxide labelled with C<sup>14</sup> can be converted in good overall yield to ethyl iodide by way of acetylene and ethylene on the scale of about 10 micromoles. Acetylene can be produced from carbon dioxide<sup>1</sup> in 90–95 per cent yield, and it may be reduced almost quantitatively to ethylene by the method of Traube<sup>2,3</sup>, in which it is shaken for an hour with a large excess of freshly prepared chromous chloride solution at room temperature. The chromous chloride solution is prepared by the rapid reduction of a solution of 1 part green chromic chloride hexahydrate in 2 parts 25 per cent hydrochloric acid with zinc wool, until the solution turns almost a copper sulphate blue. About 0.4 ml. of this solution is pipetted into a glass reaction vessel (see diagram), frozen in liquid nitrogen and evacuated. The acetylene is condensed into the tube which is then sealed off at A and allowed to warm to room temperature. After agitation for an hour, carried out by intermittent rotation about its axis of the reaction vessel held



horizontally, the vessel is sealed on to the high vacuum apparatus at B and the break-seal opened. All the dissolved gas is boiled from the chromous chloride solution, and ethylene condensed at liquid nitrogen temperature into a trap connected with a McLeod gauge, water and any residual acetylene being held back in a U-tube at liquid oxygen temperature. The yield in terms of standard gas volume is 96–98 per cent in this stage, and the remaining few per cent of acetylene can be recovered from the oxygen-cooled trap.

The direct addition of hydrogen iodide to ethylene is not easy, but can be carried out with about 90–95 per cent yield on the 10-micromole scale by sealing up ethylene with a few drops of saturated hydriodic acid in a tube similar to that used for reduction of acetylene but of about half the internal volume. The closed tube is then heated for one hour at 144° C. (boiling *o*-xylene). After the break-seal has been opened, the whole contents of the tube are distilled into excess 50 per cent caustic soda, from which ethyl iodide vapour can be recovered, most of the water vapour being held back at –78° C., and the ethyl iodide vapour finally dried over about 100 mgm. of anhydrous magnesium perchlorate for five minutes.

The synthesis is intended for use with radiocarbon dioxide of the highest obtainable specific activity; but in order to obtain a preliminary check on the yield in terms of radioactivity, several samples of C<sup>14</sup>-labelled carbon dioxide of exceedingly low activity were converted to ethyl iodide by the method, this ethyl iodide being burnt and the carbon dioxide from it introduced into a Brown and Miller type  $\beta$ -counter<sup>4</sup>. It was found that whereas overall yields for CO<sub>2</sub> → C<sub>2</sub>H<sub>5</sub>I were about 80 per cent in terms of gas volume measurements, in terms of radioactivity they were generally some 10 per cent lower. About half the loss in radioactivity was found to occur in the stage CO<sub>2</sub> → C<sub>2</sub>H<sub>2</sub>, and the other half in the stage C<sub>2</sub>H<sub>4</sub> → C<sub>2</sub>H<sub>5</sub>I → CO<sub>2</sub>. No loss of activity at all occurred in the stage C<sub>2</sub>H<sub>2</sub> → C<sub>2</sub>H<sub>4</sub>. In two particular experi-

ments, the overall volume yields were 83.5 and 84 per cent, and the yields calculated from radioactivity measurements, 72 and 75 per cent. The activities of C<sup>14</sup> available in each experiment were only about three times the background of the counter and were insufficient to pursue further the small losses, the first of which was probably due to an unidentified exchange reaction. In fact, the total amount of C<sup>14</sup> available was 2 × 10<sup>-3</sup> microcuries, of which half had to be used for calibration of the counter.

Catalytic reduction of acetylene to ethylene has been found to be possible on this scale, and yields are about 75 per cent. The method of preparation of the palladized palladium catalyst is critical, however, and it is easily poisoned. Chromous chloride reduction is much to be preferred.

We wish to acknowledge grants from the Therapeutic Research Corporation, the Royal Society and the Central Research Fund of the University of London, and to thank the Medical Research Council for grants and for a specimen of radioactive barium carbonate. One of us (R. G.) is a Keddey Fletcher-Warr student of the University of London.

W. J. ARROL

RAYMOND GLASCOCK

Research Laboratories,  
Chemistry Department,  
College of the Pharmaceutical Society,  
University of London,  
17 Bloomsbury Square,  
London, W.C.1.  
March 2.

<sup>1</sup> Arrol and Glascock, *Nature*, **159**, 810 (1947).

<sup>2</sup> Traube and Passarge, *Ber.*, **49**, 1692 (1916).

<sup>3</sup> Patterson and du Vigneaud, *J. Biol. Chem.*, **123**, 327 (1938).

<sup>4</sup> Brown and Miller, *Rev. Sci. Instr.*, **18**, 496 (1947).

## Effect of Insulin on Intestinal Glucose Absorption in Alloxan Diabetic Rats

INTESTINAL absorption of glucose is increased in the diabetic rat<sup>1,2</sup>. According to Laszt and Vogel<sup>2</sup>, this increase is not observed when alloxan diabetic rats are submitted to insulin treatment, suggesting that insulin has an inhibiting action on intestinal glucose absorption.

In the diabetic animal, as a consequence of the lack of insulin, changes are produced in the enzymatic systems, some of which might themselves modify absorption. To determine whether insulin has a direct influence on the mechanism of absorption, we have investigated the intestinal glucose absorption in alloxan diabetic rats before and after intravenous injection of insulin in the same animal, by the Sols and Ponz method of successive absorptions<sup>3</sup>.

Diabetes was produced by intravenous injection of 5 mgm. of alloxan per 100 gm. body-weight. The animals were tested four to eight days after this injection. Previous to testing, a blood-sugar level greater than 300 mgm. per cent was recorded.

Within a period of 2½–3 hours, two or four absorption experiments (of 30 and 15 min. duration respectively) were done; then an intravenous injection of 2 units of insulin per 100 gm. body-weight was given, and immediately afterwards further absorption experiments, all other circumstances being kept constant. By way of control, the blood-sugar level was estimated periodically, usually once after each absorption experiment.

As a control of the absorption capacity in diabetic rats during the experimental period (about three