

NOTES

The Epimerization of Glucose-1-C¹⁴¹

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The mechanism (or mechanisms) by which the epimerization of glucose proceeds in alkaline solution is not clearly established, the commonly accepted intermediate formation of an enediol being inconsistent with the results of the investigations of Fredenhagen and Bonhoeffer² concerning the conversion of glucose to fructose in heavy water. A rearrangement of the carbon chain of glucose during the course of the reaction has not been excluded by any earlier work and could conceivably occur. Such a rearrangement would be detectable using glucose-1-C¹⁴ which has recently become available.³ We have performed this experiment and our results indicate that no rearrangement takes place.

Experimental

1.3 mg. of glucose-1-C¹⁴ having a specific activity of 120 m μ c/mg.⁴ was diluted with 30 mg. of inactive glucose and dissolved in 0.2 ml. of saturated calcium hydroxide solution. The solution was kept at 70° for 2 hours,⁵ then chilled and passed over 1 g. each of Amberlite IR-100-H and IRA-400 ion exchange resins. The volume with washings was 6 ml. The mixed sugars were diluted with 25 mg. of cold glucose and were degraded by the method of Wood, Lifson and Lorber.⁶ The specific activities obtained were: for the glucose carbon atoms 1 and 6, 4.4 m μ c/mg. of carbon; carbon atoms 2 and 5, 0.1 m μ c/mg.; carbon atoms 3 and 4, 0.09 m μ c/mg. A similar distribution of label in the lactic acid was obtained by degrading glucose-1-C¹⁴ which had not been epimerized.⁷

(1) Research carried out at Brookhaven National Laboratory under the auspices of the U. S. Atomic Energy Commission.

(2) Fredenhagen and Bonhoeffer, *Z. physik. Chem.*, **A181**, 392 (1938).

(3) Sowden, *Science*, **109**, 229 (1949).

(4) The glucose-1-C¹⁴ was most generously furnished us by Prof. J. C. Sowden.

(5) Lobry deBruyn and Van Ekenstein, *Rec. trav. chim.*, **14**, 203 (1895).

(6) Wood, Lifson and Lorber, *J. Biol. Chem.*, **159**, 475 (1945).

(7) Gibbs, Dumrose, Bennett and Bubeck, *ibid.*, **184**, 545 (1950).

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Separation of Seventh Group Anions by Ion-Exchange Chromatography

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An extension of the techniques of ion-exchange chromatography to the separation and concentration of anionic species has been made possible by virtue of the recent commercial availability of several stable mono-functional strong base anion exchange polymers.^{1,2} The principles governing anion fractionation are the same as those previ-

ously applied in the separation of the rare earths.^{3,4} This communication reports the efficient separation of the halide anions and of pertechnetate from perhenate ion. The separation of Cb(V) and Ta(V), and Zr(IV) and Hf(IV) through the formation of their complex halide anions has been described in a recent publication from this Laboratory.⁵

Halide Separations.—The fractionation was conducted by placing approximately 0.7 m. e. quantities of ammonium bifluoride, chloride, bromide and iodide on the top-most portion of a 0.72 sq. cm. X 91.5 cm. cylindrical bed of 100/120 mesh Dowex-2 initially in the nitrate form, and then eluting the mixture at a flow rate of 1.3 ml./min. at room temperature with a 1 M NaNO₃ solution adjusted to a pH of 10.4 with NaOH. The course of the separation was followed by continuously monitoring the radioactivity in the effluent from the bed. The 37 m. Cl³⁸, 34 h. Br⁸² and 8.0 d. I¹³¹ were used as indicators. Fluoride was determined chemically. Figure 1a shows the results taken from a typical experiment. The identities of the various activity peaks were established by measurements of decay and radiation characteristics on aliquots taken from the effluent. Interestingly, the first peak contained over 90% of the starting fluoride together with a

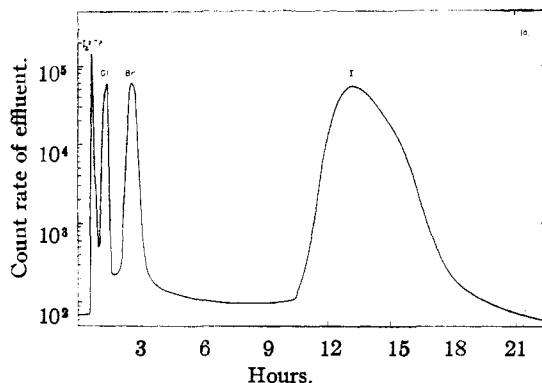


Fig. 1.—(a) Halide separation.

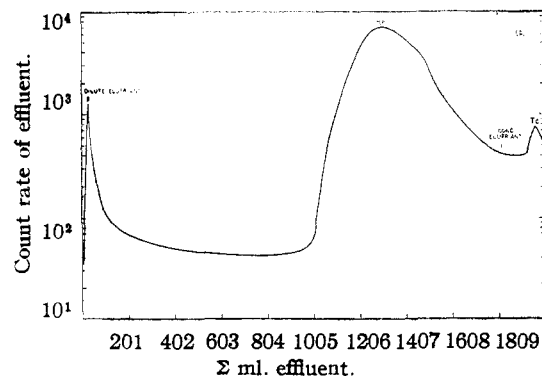


Fig. 1.—(b) Rhenium-technetium separation.

(1) Dow Chemical Company, Midland, Michigan.

(2) Resinous Products Division, Rohm and Haas Co., Philadelphia, Pa.

(3) B. H. Ketelle and G. E. Boyd, *THIS JOURNAL*, **69**, 2800 (1947).

(4) S. W. Mayer and E. R. Tompkins, *ibid.*, **69**, 2866 (1947).

(5) K. A. Kraus and G. E. Moore, *ibid.*, **71**, 3855 (1949).