TECHNICAL PAPERS

1-C14-D-Glucose and 1-C14-D-Mannose

John C. Sowden

Radiochemistry Laboratory, Department of Chemistry, Washington University, St. Louis

Radioactive C¹⁴ was first incorporated in the hexose molecule by allowing green plants to assimilate $C^{14}O_2$ (2). The resulting glucose contained C¹⁴ in all positions of the carbon chain, with the highest concentration of the radioactivity in carbons 3 and 4 (1). Starch labeled with isotopic C¹³ also has been prepared by growing plants in an atmosphere of C¹³O₂ (3).

Previously, glycogen containing isotopic C¹¹ had been produced by injecting NaHC¹¹O₈ into rats being fed nonradioactive sodium lactate (7). Evidence was subsequently obtained, using C¹³, that the biosynthetic glucose comprising the glycogen was labeled only in carbons 3 and 4 (9). This synthesis of labeled glucose has recently been employed with NaHC¹⁴O₈ (10).

For studies of the fate of hexose in experiments that involve fragmentation of the molecule, the need was apparent for a glucose containing C¹⁴ in only one known position of the carbon chain, preferably in the reactive reducing group of carbon 1.

D-Glucose and D-mannose labeled with C^{14} in the aldehyde carbon have now been prepared in the crystalline state by application of the nitromethane synthesis (8) to D-arabinose.

Sixteen grams of C¹⁴-methanol containing 4 me of radioactivity was converted to 60 gm of methyl iodide by the method of Norris (6). By application of the Victor Meyer reaction (4) with silver nitrite, the methyl iodide yielded 18 gm of nitromethane and 4 gm of the isomeric methyl nitrite. Condensation of the nitromethane with D-arabinose yielded, after separation and purification, 11.0 gm of 1-nitro-1-desoxy-D-mannitol and 6.0 gm of 1-nitro-1-desoxy-D-glucitol. Conversion of the respective nitroalcohols to the corresponding hexoses by the Nef reaction (5, 8) gave D-mannose phenylhydrazone in 80% yield and crystalline D-glucose in 60% yield. Crystalline D-mannose was obtained from the phenyl hydrazone in 90% yield by cleavage with benzaldehyde.

The sugars, whose calculated specific activity is .044 μ e/mg showed an observed activity of approximately 62,000 cpm/mg when counted from a thin layer in the R.C.L.-Nucleometer.¹

Experimental details will be published elsewhere.

References

- ARONOFF, S., BARKER, H. A., and CALVIN, M. J. biol. Chem., 1947, 169, 459.
- ARONOFF, S., BENSON, A., HASSID, W. Z., and CALVIN, M. Science, 1947, 105, 664; PUTMAN, E. W., HASSID, W. Z., KROTKOV, G., and BARKER, H. A. J. biol. Chem., 1948, 173, 785.
- ¹ Radiation Counter Laboratories, Chicago, Illinois.

- LIVINGSTON, L. G., and MEDES, G. J. gen. Physiol., 1947, 31, 75.
- 4. MEYER, V. Ann., 1874, 171, 23.
- 5. NEF, J. V. Ann., 1894, 280, 263.
- 6. NORRIS, J. F. Amer. chem. J., 1907, 38, 639.
- SOLOMON, A. K., VENNESLAND, B., KLEMPERER, F. W., BUCHANAN, J. M., and HASTINGS, A. B. J. biol. Chem., 1941, 140, 171.
- SOWDEN, J. C., and FISCHER, H. O. L. J. Amer. chem. Soc., 1944, 66, 1312; 1945, 67, 1713; 1946, 68, 1511; 1947, 69, 1048, 1963.
- WOOD, H. G., LIFSON, N., and LORBER, V. J. biol. Chem., 1945, 159, 475.
- ZILVERSMIT, D. B., CHAIKOFF, I. L., FELLER, D. D., and MASORO, E. J. J. biol. Chem., 1948, 176, 389.

Crushing Strengths of Minerals at Low Temperatures

James M. Weigle

Syracuse University

An attempt was made to measure and evaluate the effects of subnormal temperatures on the crushing strengths of several available common minerals (prochlorite, serpentine, graphite, selenite, and halite).

In all cases except that of halite, cylindrical samples 0.4'' in diameter were cut from mineral masses by means of **a** milled hollow tool. Ends of the samples were squared and polished on **a** lapping wheel to ensure equal distribution of the pressure applied. All halite samples were nearly perfect cubes cleaved from a single mass.

The apparatus used to produce and measure the force applied consisted essentially of a hydraulic jack with a registering pressure gauge attached. Containers fashioned of sheet aluminum, asbestos insulated, permitted cooling of the test samples in place. Dry ice and liquid oxygen were used as refrigerating agents, tests being made at room temperature, -78.7° C, and -183° C.

Pressure was applied at opposite ends of the samples, normal to the planes of cleavage when cleavage was present. Since halite cleaves in three planes at right angles, two planes of cleavage therefore lay parallel to the direction of force.

Graphite exhibited a decelerating increase in crushing strength with decrease in temperature, while prochlorite, serpentine, and selenite showed an accelerating increase. Halite, contrary to the others, showed an accelerating *decrease* in crushing strength with decrease in temperature. (The crushing strength of halite dropped from 11,850 PPSI to 3,250 PPSI with a decrease in temperature from 25° C to - 183° C.)

Since halite presented two planes of cleavage parallel to the direction of pressure applied, it is probable that in this case the results involved not only a change in crushing strength but an increased ease of cleaving with lowered temperature.

The changes in crushing strength with temperature