the hydrolysis fragments of the above dextran polyalcohol revealed also 2–2.5% glucose as determined by the phenol–sulfuric acid method.[5] This is believed to be derived from glucose residues which are immune to periodate oxidation as a consequence of being linked through positions 1 and 3, or 1, 2, and 4.

In an analogous manner it has been shown that glycogen and amylopectin contain about 1.0 and 0.5% glucose, respectively, which is immune to prolonged treatment with sodium periodate. This glucose, which still remains intact even when the derived polyalcohols themselves are treated with sodium periodate, could arise because of incomplete oxidation or because of fixed *trans* OH groups arising from stereochemical strain[6] but the present evidence suggests that it arises from glucose residues in the polysaccharides linked by 1, 3 bonds. While it is probable that these same glucose residues correspond to those which give rise to the 2,6-dimethyl-D-glucose fragment produced from the methylated polysaccharides by hydrolysis, the possibility exists that they correspond to glucose residues joined through positions 2 and 4.

In similar experiments on amylose and cellulose the indications are that these two polysaccharides contain approximately 0.2 to 0.5 and 0.1 to 0.2% glucose, respectively, which is immune to periodate oxidation. It is believed, therefore, that the possibility of a hitherto unrecognized linkage in these polysaccharides is worthy of some consideration. The details and constitutional significance of these and similar experiments on other polysaccharides such as fructosans, hemicelluloses, fungus glucosans, plant gums and degraded plant gums will be published later.

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RECEIVED AUGUST 4, 1952

X-RAY EXAMINATION OF IRON BISCYCLOPENTADIENYL

Sirs:

Crystals of iron biscyclopentadienyl, prepared by Dr. E. O. Brimm of Linde Air Products Co., and forwarded to us by Professor W. C. Fernelius, were examined by X-ray methods at the suggestion of the latter. Rotation and Weissenberg photographs, using Mo$K\alpha$ radiation, revealed a monoclinic cell, space group $P2_1/n$, with $a = 9.00$ Å; $b = 7.52$ Å., $c = 5.94$ Å., $\beta = 92.5°$. The measured density of 1.516 g./cc. showed 2 molecules per cell, and thus required the two iron atoms to be at the cell corner and body center. The cell symmetry requires that the molecule is centrosymmetric, with the iron atom at its center.

The molecular structure was determined automatically on X-RAC, the electronic computer for X-ray analysis,[1] by use of the non-negativity criterion, as previously utilized by us on the structure of fructose[2] and in several other analyses.[3] The iron atoms contribute positive phases to $(h,k,0)$ terms with $h + k = 2n$. All such structure factors were inserted into X-RAC with positive phases, and the strongest term with $h + k$ odd was also entered as positive. The effects of phase permutation of the remaining odd terms were examined consecutively and in order of decreasing amplitude, and phases were assigned so as to minimize negative excursions of the density function. A projection on the (x,y) plane concomitant with the "sandwich" structures proposed by Wilkinson, Rosenblum, Whiting and Woodward[4] and Woodward, Rosenblum and Whiting[5] immediately appeared. The density projection did not indicate that the cyclopentadiene groups were rotating, and the center of symmetry then demanded the anti-prismatic structure of Wilkinson, *et al.*[4]

A correct form factor for iron as it occurs here is not known, and thus a refinement of carbon positions has not yet been possible. Using an empirical Fe^{++} curve with an approximate temperature factor, an R-factor of 0.17 was found for a planar carbon ring with C–C distances of 1.41 Å. and Fe–C distance of 2.0 Å. A three-dimensional analysis is in progress, to establish the nature of the bonding and the electronic configuration of the iron atom.

We are grateful to Dr. Brimm and Prof. Fernelius for suggesting the problem and supplying the crystalline material.

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[2] P. F. Eiland and R. Pepinsky, *Acta Cryst.*, **3**, 160 (1950).

[3] X-RAC Computations supported by Office of Naval Research.

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[5] R. B. Woodward, M. Rosenblum and M. C. Whiting, *ibid.*, **74**, 3458 (1952).

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CHROMATOGRAPHIC SEPARATION OF THE ADRENOCORTICOTROPIC HORMONE ON PARTITION COLUMNS[1]

Sir:

Fractionation of pig and sheep pituitary extracts on oxycellulose columns[2] has yielded materials with ascorbic acid depleting activities up to 100 u./mg.[3] After *peptic* digestion of such fractions of porcine origin, Brink, *et al.*,[4] applied the countercurrent distribution technique in the isolation of an apparently homogeneous material with an activity of 300 u./mg.

[1] This work was aided in part by grants to Professor C. H. Li from the National Institutes of Health, the United States Public Health Service, the Armour Laboratories, Merck and Company, Inc., and the Eli Lilly Laboratories.

[2] (a) E. B. Astwood, M. S. Raben, R. W. Payne and A. B. Grady, THIS JOURNAL, **73**, 2969 (1951); (b) C. H. Li, *ibid.*, **74**, 2124 (1952).

[3] Assays reported here were performed by the adrenal ascorbic acid depletion method of M. A. Sayers, G. Sayers and L. A. Woodbury, *Endocrinology*, **42**, 379 (1948). Results are expressed in U. S. P. units per milligram.

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