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Preliminary communication

An X-ray crystallographic study of a metal–carbohydrate complex: α-D-xylose·$CaCl_2 \cdot 3H_2O$

GERALD F. RICHARDS

The Institute of Paper Chemistry, Appleton, Wisconsin 54911 (U.S.A.)

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Several monosaccharides and disaccharides react with calcium chloride to form hydrated, crystalline adducts[1]. The crystal structure of α-D-xylose·$CaCl_2 \cdot 3H_2O$ has been determined from low-temperature (approximately, −193°) three-dimensional X-ray data obtained by the multiple-film, equi-inclination Weissenberg technique (CuK_α radiation, $\lambda = 1.54182$ Å). Heavy-atom methods were used to solve the structure, and the block-diagonal, anisotropic refinement was terminated at a conventional R index of 0.079 for 1240 observations. One formula unit ($C_5H_{10}O_5 \cdot CaCl_2 \cdot 3H_2O$) comprises the asymmetric unit in a monoclinic unit-cell having low-temperature (approximately, −193°) cell parameters of $a = 8.375$ (10), $b = 12.599$ (25), $c = 7.194$ (10) Å, $\beta = 114.4$ (2) and space group $P2_1$. The atomic coordinates are given in Table I.

TABLE I

FRACTIONAL ATOMIC COORDINATES

Atom	x	y	z
Ca	0.11233(30)	0.0(0)	0.19213(29)
Cl-1	0.46615(33)	0.04954(24)	−0.12737(34)
Cl-2	−0.14906(38)	0.17723(23)	−0.43291(38)
O-1	0.25427(107)	0.14693(60)	0.44058(119)
O-2	0.23483(101)	0.14465(60)	0.06435(117)
O-3	0.06974(108)	0.34639(63)	−0.10081(108)
O-4	0.02186(105)	0.48005(61)	0.17313(113)
O-5	0.38676(107)	0.31444(68)	0.51923(121)
O-6	0.34374(114)	−0.09081(76)	0.16204(122)
O-7	0.19413(121)	−0.07981(68)	0.50903(116)
O-8	−0.13453(109)	0.10026(68)	0.16615(118)
C-1	0.36384(145)	0.21874(93)	0.40069(168)
C-2	0.28837(147)	0.24475(94)	0.17258(163)
C-3	0.12463(147)	0.31495(92)	0.10937(153)
C-4	0.17082(163)	0.41439(98)	0.24072(162)
C-5	0.22770(173)	0.37842(91)	0.45534(172)

Except for O-5 the ring oxygen atom, all of the oxygen atoms are coordinated to calcium ions. The oxygen atoms(O-6, O-7 and O-8) from the three molecules of water of hydration, the hydroxyl oxygen atoms, O-1 and O-2 from a xylose unit, and the oxygen atoms O-3′ and O-4′ from a symmetry-related xylose unit, are bonded to each calcium ion (Fig.1.). This sharing of xylose units by the calcium ions results in a chainlike structure parallel to the *b* axis similar to that reported[2] for the structure of β-D-mannofuranose·$CaCl_2 \cdot 4H_2O$. The sevenfold coordination of the oxygen atoms to each calcium ion is described by a distorted pentagonal bipyramid.

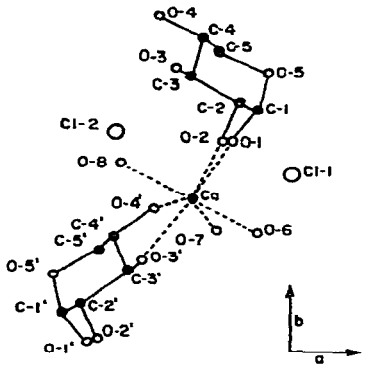

Fig.1. Structure of α-D-xylose·$CaCl_2 \cdot 3H_2O$, viewed along the *c* axis.

Calcium ion–oxygen bond distances (standard deviations, 0.008–0.010 Å) appear to vary according to the negative charge on the oxygen atoms. The distances from the calcium ion to the three oxygen atoms (O-6, O-7 and O-8) from the water of hydration are 2.336, 2.321, and 2.363 Å, whereas the distances from the calcium ion to the hydroxyl oxygen atoms O-1, O-2, O-3, and O-4 are 2.506, 2.449, 2.382, and 2.406 Å. The O-1 oxygen atom, which has the shortest carbon–oxygen distance (the C-1–O-1 distance is 1.400 Å) and the least negative charge, forms the longest calcium ion–oxygen bond.

Bond distances and bond angles for the α-xylose molecule are in agreement with those reported for the crystal structure of α-xylose[3].

REFERENCES

1 J. A. Rendleman, *Advan. Carbohyd. Chem.*, 21 (1966) 209.
2 D. C. Craig, N. C. Stephenson and J. D. Stevens, *Carbohyd. Res.*, 22 (1972) 494.
3 A. Hordvik, *Acta Chem. Scand.*, 25 (1971) 2175.