

Short Communications

A Study of the Ca—Na Salt of α -D-Galacturonic Acid by X-Ray DiffractionJOHAN HJORTÅS,^a BJØRN LARSEN^b
and SRINUAN THANOMKUL^{a*}^aInstitutt for røntgenteknikk and ^bInstitutt for marin biokjemi, Universitetet i Trondheim-NTH, N-7034 Trondheim-NTH, Norway

The studies of α -D-galacturonic acid derivatives, previously initiated with a structure determination of the methyl α -D-galacturonic acid methyl ester,¹ have been continued with an investigation of the Ca—Na salt, some preliminary results of which are given here.

Ca—Na- α -D-galacturonate was prepared by careful neutralization of an aqueous solution of α -D-galacturonic acid with Ca(OH)₂ to pH 6. The solution was then evaporated to crystallization and the product recrystallized from water at approximately 40 °C.**

Crystal data are: 2Ca²⁺+2Na⁺(C₆H₇O₇)₆·12H₂O, hexagonal space group P6₃, cell dimension (with estimated standard deviations in parentheses) $a = 13.493(2)$ Å, $c = 9.655(2)$ Å, measured density 1.665 g cm⁻³, calculated density 1.636 g cm⁻³.

1077 reflexions with intensity greater than twice the background were observed on an automatic Picker FACS-1 diffractometer, using MoK α radiation and the $\omega/2\theta$ scanning mode with max. $2\theta = 50^\circ$. The Ca and Na positions were obtained from a 3-dimensional Patterson map. The C and O atoms were found by successive Fourier calculations. Full matrix least squares refinement,² with isotropic temperature factors for the Ca, O, and C atoms, has yielded the conventional R index of 0.091. A projection of the molecule along the c axis is shown in Fig. 1.

The Ca atoms occupy positions on the 3-fold axes and are caged by O(5) and O(7) from the

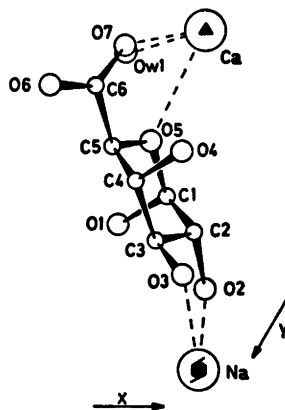


Fig. 1. Projection of the molecule along the c axis.

galacturonate molecule and O(w1) from a water molecule, with Ca—O distances of 2.83, 2.39, and 2.43 Å, respectively. Due to the multiplicity of the axis the coordination around Ca is 9-fold. The Na atom on the 6₃ axis has O(2) and O(3) from the ring as nearest neighbours at distances of 2.36 and 2.50 Å, respectively. The coordination of Na is six-fold. Further refinement is in progress.

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* Permanent address: Physics Department, Faculty of Science, Chulalongkorn University, Bangkok 5, Thailand.

** We originally planned to make the Ca salt but the product turned out to be the Ca—Na salt in low yield, the Na atoms probably coming from contamination. We later crystallized the Ca—Na salt from equal amounts of Ca(OH)₂ and NaOH and the crystals are identical to those previously obtained.