provided with a precision-ground stirrer, a reflux condenser with a $CaCl_2$-tube and a dropping funnel with a pressure-equalising tube. A solution of the acyloin (0.1 mole) in methylene chloride (100-400 ml, see below) was added through the dropping funnel in the course of 1 ½ h while a stream of dry nitrogen was passed through the apparatus from an inlet in the dropping funnel. Finally the solution was refluxed for 1 h. The hydrogen chloride evolved was collected in water and titrated with sodium hydroxide; usually a titer close to 95 % of the theoretical was found.

After the solution had been refluxed, the reflux condenser was replaced by a Claisen head with condenser and the solvent was distilled off. The triphenyl derivative crystallised; it was recrystallised from acetone. The other compounds were distilled *in vacuo*, moisture being carefully prevented from passing into the apparatus.

2,4,5-Triphenyl-1,3,2-dioxaborole. From 21.2 g of benzoin dissolved in 400 ml of methylene chloride. Recrystallised from dry acetone. Yield 16.7 g = 58 %. M.p. 110.5–111°C. (Found: C 79.80; H 5.05. Calc. for $C_{20}H_{15}BO_2$: C 80.50; H 5.07).

2-Phenyl-4,5-diethyl-1,3,2-dioxaborole. From 11.6 g of propioin in 100 ml of methylene chloride. Yield 10.0 g = 50 %. B.p./11 mm: 118–19°C. (Found: C 71.20; H 7.50. Calc. for $C_{12}H_{15}BO_2$: C 71.34; H 7.43).

2-Phenyl-4,5-dimethyl-1,3,2-dioxaborole. From 8.8 g of dimeric acetoin dissolved in 250 ml of methylene chloride. The reaction product, which boiled from 119 to 129°C at 11–12 mm Hg, crystallised on standing; it was recrystallised from pentane. Yield 9.3 g = 53 %. M.p. 43.5–44.5°C. The compound was very sensitive to humidity, and we did not succeed in obtaining quite correct analytical values. (Found: C 67.20; H 6.63. Calc. for $C_{10}H_{11}BO_2$: C 69.02; H 6.37).

2-Phenyl-4-methyl-5-oxo-1,3,2-dioxaborolane. From 11.8 g of ethyl lactate in 125 ml methylene chloride. Yield 8 g = 46 %, distilling from 127 to 128°C at 11 mm Hg. (Found: C 61.50; H 5.38. Calc. for $C_9H_9BO_3$: C 61.42; H 5.35).

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The Structure of β-Lyxose

ASBJÖRN HORDVIK

University College of Addis Ababa, Ethiopia

In 1947 Hassel and Ottar [1] advanced the assumption that the six membered pyranose ring is chair formed. The correctness of this assumption has been confirmed by the determination of several pyranose structures [2-6]. The chair formed pyranose ring, however, made it necessary to accept two possible structures for each pyranose isomer, one being formed from the other by a conversion of the ring. The existence of only one of the conversion forms of a pyranose in the crystalline state seems to be due to their different stability, and the hexa- and penta-pyranoses which so far have been determined [2-6] agree with the proposals of Reeves [7].

The purpose of the present work is to find out whether the same is valid for β-lyxose. This pentapyranose has the conversion forms 1*e*2*a*3*e*4*e* and 1*a*2*e*3*a*4*a*. According to Reeves' stability scheme the first form is the more stable, and is consequently expected to be found in β-lyxose crystals.

Crystallization of β-lyxose was tried from several different solvents, but always resulted in syrups. Eventually one crystal large enough for single crystal studies was obtained by placing some syrup on an objective glass and then making scratches in the glass. The crystals grown in this way are needle-shaped and elongated along the *c* axis.

Space group and axial lengths were determined from Weissenberg and oscillation photographs, using Cu$K\alpha$ radiation. The crystals are orthorhombic and the space group is $P2_12_12_1$. The unit cell dimensions are, a = 9.58 Å, b = 10.42 Å and c = 6.53 Å, to within 0.5 %. With four molecules in the unit cell the calculated density is 1.53 g/cm³, which agrees with the experimental value 1.54 g/cm³.

The intensities of the $hk0$ reflections were estimated visually from a series of Weissenberg photographs, using an intensity scale made from one of the reflections in this zone. 67 $hk0$ reflections were observed, representing about 52 % of the possible number observable under the experimental conditions. The intensities were corrected for the Lorenz and polarization factors in the usual way, and in order to derive the

unitary structure factors of the reflections, the observed structure factors were put on an approximately absolute scale by Wilson's method [8].

The carbon and oxygen contributions to calculated structure factors are based on the atomic scattering curves of Berghuis *et al.*[9]

The Patterson *c* projection revealed the general arrangement of the molecules (rather similar to the arrangement in the *a* projection of β-arabinose [6]), and this indicated that the strong reflection 400 should have a negative sign. The signs of 210 and 790 were chosen, and probable phases of 28 reflections were deduced by Zachariassen's method [10]. Eight sets of phases were found, but the corresponding Fourier maps were difficult to interpret, as none showed a maximum corresponding to an axial hydroxyl oxygen atom. An approximate map showing such a maximum, and also giving a rough outline of the molecule, was eventually obtained by selecting 15 phases by an application of a method proposed by Woolfson [11]. These 15 phases were common for two of the phase sets. Further phases for both sets were deduced for unitary structure factors larger than 0.1 by the equation $s(\mathbf{h}) = s(\Sigma_{\mathbf{h}'} U_{\mathbf{h}} U_{\mathbf{h}+\mathbf{h}'})$ (Cochran and Woolfson [12]). The two corresponding electron density maps were very nearly mirror images of each other around $(a/8, b/4)$. For one of these structures the refinement process stopped at an R_1 value of 0.26, while the other one, with coordinates as listed in Table 1, gave $R_1 = 0.15$ and so proved to be the correct one. In $R_1 = \Sigma|F_o - F_c|/\Sigma|F_o|$ also unobserved reflections are included. An overall temperature function $\exp(-3.0 \sin^2\Theta/\lambda^2)$ was used. Contributions from hydrogen atoms to the structure amplitudes have so far been ignored.

Table 1. Atomic coordinates, in fractions of corresponding cell edges. Origin on twofold screw axis.

	x	*y*
C_1	0.090	0.124
C_2	0.117	0.262
C_3	0.087	0.357
C_4	0.179	0.326
C_5	0.144	0.187
O_1	0.134	0.028
O_2	0.259	0.260
O_3	0.124	0.486
O_4	0.133	0.409
O_5	0.180	0.109

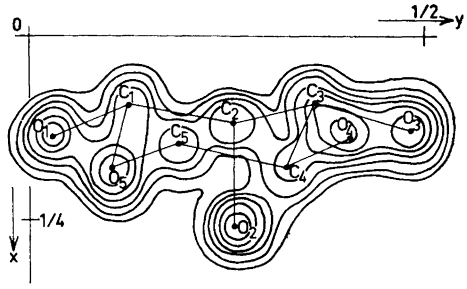

Fig. 1. Electron density projection of β-lyxose on the (001) plane, showing one asymmetric unit. Plane group *pgg*, origin at 2. Contours at arbitrary but equal intervals.

The final electron density map is reproduced in Fig. 1. It shows that β-lyxose has the configuration *1e2a3e4e* in agreement with Reeves' prediction.

An account of the crystal structure determination, when completed, and a discussion of the molecular structure will be published later.

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