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The crystal structure of d-ribose derived from powder diffraction data. Dubravka Sisak[a], Lynne McCusker[a], Christian Baerlocher[a], Jack D. Dunitz[b]
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The molecular structure of d-ribose is usually presented in textbooks in the β-furanose form, because that is the form it adopts in numerous biochemically important ribose derivatives. However, NMR experiments over forty years ago showed that in aqueous solutions it exists as a mixture of α- and β-furanoses and α- and β-pyranoses. Surprisingly, its structure in the solid state was not known until very recently [1]. Furberg et al. investigated crystalline d-ribose back in 1956 [2], but the crystals were small, twinned and of poor quality, so only the unit cell and space group could be derived ($P2_1$, a = 6.509 Å, b = 21.644 Å, c = 4.821 Å, β = 111.0°, Z=4). In view of the advances in structure determination methodology for polycrystalline materials that have been made in recent years and the importance of ribose in biochemical systems, an attempt to solve its structure from powder diffraction data was undertaken. Data were collected on the MS beamline at the SLS [3] using the Mythen II Si-microstrip detector [4]. Indexing confirmed the unit cell reported fifty years ago. Initially, the powder charge-flipping algorithm [5,6] in *Superflip* [7] was tried, but no satisfactory solution was found. Then an attempt was made to apply the direct-space global optimization program *FOX* [8] to the problem, even though it was not clear whether the d-ribose molecule would adopt a chain or one of the four ring structures in the solid state. With two molecules in the asymmetric unit, each with 5 possible configurations, there are 15 starting models for the optimization and up to 18 degrees of freedom. Several of these were tried using the parallel tempering algorithm in *FOX* with relaxed restraints, but the R values of the resulting structures did not differ significantly and a correct solution could not be identified. However, the flexibile chain models tended to yield a distorted U-shape geometry, and this suggested that the ribose probably adopts a ring structure. A long and tedious process of generating difference Fourier maps from various starting models was then undertaken. This eventually led to an approximate structure with one α- and one β-pyranose molecule in the asymmetric unit. Refinement converged with the R values R_F = 0.081 and R_{wp} = 0.097. Further refinement allowing an α/β disorder in both molecules showed that one molecule was in fact pure β and the other 73% α and 27% β, and resulted in a slight improvement in the R values (R_F = 0.077 and R_{wp} = 0.094). This model with a higher percentage of β-pyranose is consistent with solid state NMR results [8]. Because the structure solution was not straightforward, d-ribose is being used as a test case for combining *FOX* and *Superflip*. Preliminary results of this systematic study are promising.

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