In a previous report from this laboratory the successful determination of positions of H bonded to C or H using a spherical atom model was reported (Hope and Ottersen, Acta Cryst. (1978) B34, 3623). Attempts to position H in OH groups had been unsuccessful. For further-study of this problem we have used a pentaerythritol, C(CH₂OH)₄, model. The compound has only 4 molecules per asymmetric unit, and was expected to be crystallographically well behaved (Elferman and Rudman, Acta Cryst. (1979) B35, 2450).

All unique reflections accessible with MoKα to 2θ = 163° (7700) were measured on a Picker diffractometer (graphite monochromator, temp. 85 K, 0-2θ scan 2°/min in 2θ, spcralicrystal r = 0.60 mm). Only 35% of the intensities had I < 0.1, 85% had I > 0.6(1). The intensities were corrected for scan truncation errors (Denne, Acta Cryst. (1977) A33, 458).

Full-matrix least-squares refinements were performed with anisotropic temperature factors for C, O, and isotropic for H. In keeping with previous experience the H(C) atoms could be well determined by excluding data below S = sin θ/λ = 0.65 Å⁻¹, while the O-H distance was too short (0.90 Å). Calculations with varying S_min showed the largest deviations to occur for S_min chosen.

For derivation and symbols used see Helmholdt and Vos (Acta Cryst. (1977), A33, 38-63). Account of the actual frequency distribution $v_j(g)$ changes $1/v_j(g)$ in (1) to

$$I_j(e,G) = G^{-1} \int (e^2/v_j(g))dg$$

where G is the maximum value of $g$. The frequency distribution has to be measured by inelastic neutron scattering, which is not easy to accomplish for molecular crystals. It can also be found by lattice dynamical calculations. The program LATDYN developed by Kroon (see Kroon and Vos) has been extended in such a way that molecules with charged atoms can be treated. To this end the convergence acceleration technique described by Williams (Acta Cryst. (1971), A27, 452-455) has been added to the minimization procedure, and in the calculation of the dynamical matrix complete rigid body molecules are taken into account instead of all atoms within a certain distance from the atom considered. For different directions $\hat{g}$ in reciprocal space $I_j(e,G)$ is calculated as a series expansion in $G$. The summation technique of the program TDS2 developed by Helmholdt (see Helmholdt and Vos) is used for the computation of $a_j(\hat{g})$.