In a previous report from this laboratory the successful determination of positions of H bonded to C or N 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 pentamethylpentane model, C(CH₂OH)₅, C(CH₂OH)₅H₆. The compound has only one molecule per asymmetric unit, and was expected to be crystallographically well behaved (Ellerman and Rudman, Acta Cryst. (1975) B31, 2536).

All unique reflections accessible with MoKα to 2θ = 163° (~700) were measured on a Picker diffractometer (graphite monochromator, temp. 85 K, φ-2θ scan 2θ/min in 2θ = 0.20°/min. Only 35% of the intensities had I > 0.8σ(I). The intensities were corrected for scan truncation errors (Denne, Acta Cryst. (1977) A33, 438). 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(0) atom 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 Smin showed the largest deviations to occur for Smin = 0.75 Å⁻¹. In keeping with previous experience and with anisotropic temperature factors for C, 0.5 Smin was chosen.

The small member of observations per parameter required for very good data in the 0.7-0.8 reflection range) is expected to be crystallographically well behaved (Eilerman and Rudman, Acta Cryst. (1977) A33, 675-684). Attempts with Smin = 0.7 Å⁻¹, and with Smin = 0.6 Å⁻¹, the two peaks were of nearly equal height. Toward higher Smin values the bond peak diminished much more rapidly than the 'nuclear' peak. Apparently the refinement program places the H atom near the center of gravity of the two peaks, with undesirable consequences if an inappropriate Smin is chosen.

This approach described here (spherical atom model, very good data in the 0.7-0.9 Å⁻¹ range) is expected to yield reliable results unless high H thermal motion precludes significant H contribution to the structure factors.

More realistic models for the atomic form factors may lessen the requirements for good counting statistics (e.g. Hirshfeld and Hope, Acta Cryst. (1980) B36, 406).