

The Crystal Structure and Stereochemistry of Triol Q from *Erythroxylyon monogynum* Roxb.

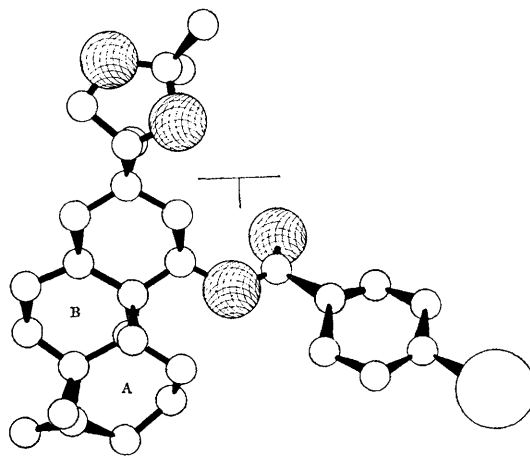
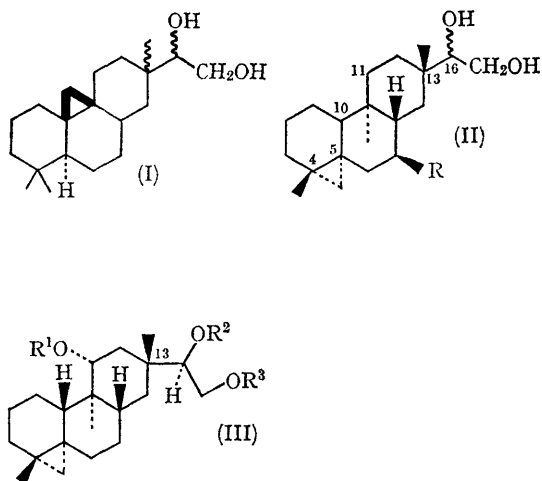
By GEORGE FERGUSON, J. W. B. FULKE, and R. MCCRINDLE

(Chemistry Department, The University, Glasgow, W.2)

THE elucidation of the constitution and stereochemistry of constituents of *Erythroxylyon monogynum* Roxb. has been the subject of a number of Papers.¹⁻⁴ The structure of erythroxydiol X was originally formulated¹ as (I) but later work² showed that a more likely structure was (II; R = H) with the cyclopropane ring at positions 4, 5. Triol Q had been tentatively formulated as (II; R = H, plus one secondary OH)² and as (II; R = OH)³; the stereochemistry at positions 4, 5, 10, and 16, the location of the cyclopropane ring and of the secondary hydroxyl group were still in doubt. The structure and stereochemistry of triol Q clearly merited further attention and we accordingly undertook an X-ray analysis of the *p*-iodobenzoate of triol Q acetone. While our analysis was in progress it was shown⁴ that the secondary OH group was attached α to C-11, that the cyclopropane ring was at positions 4, 5 and that triol Q could be converted into diol X by removal of the secondary OH group.

been determined by correlation with rosenonolactone,² the constitution and absolute stereochemistry of triol Q is thus defined by (III; R¹ = R² = R³ = H). In addition, our analysis establishes the stereochemistry and confirms the structure assigned² to the related diols isolated from this source.

The *p*-iodobenzoate of triol Q acetone crystallises in the orthorhombic system, space group $P2_12_12_1$ with four molecules of C₃₀H₄₁O₄I in a unit cell of dimensions $a = 10.34$, $b = 25.84$, $c = 10.44$ Å. Some 1300 equi-inclination Weissenberg data were estimated visually and used to determine the structure by the heavy-atom and three-dimensional Fourier methods. The analysis was complicated initially by the heavy-atom x -coordinate being close to 0.00, leading to the appearance of a false mirror plane in the first heavy-atom-phased electron-density distribution. The value of R is now 19% and refinement of the atomic parameters is continuing.



FIGURE

The atomic arrangement in a molecule of the *p*-iodobenzoate of triol Q acetone as viewed along the *c* crystal axis.

Our X-ray analysis clearly establishes the constitution and stereochemistry of the *p*-iodobenzoate of triol Q acetone as (III; R¹ = *p*-I.C₆H₄.CO⁺, R², R³ = Me₂C<). The absolute stereochemistry at position 13 having previously

The conformation and absolute stereochemistry of the derivative are shown in the Figure. The geometry of the A-B junction is evident as

is the distortion in ring-A induced by the cyclopropane ring fusion.

(Received, August 25th, 1966; Com. 629.)

¹ R. Soman and Sukh Dev, *Tetrahedron Letters*, 1964, 1181.

² J. D. Connolly, R. McCrindle, R. D. H. Murray, K. H. Overton, and A. Melera, *Tetrahedron Letters*, 1964, 1859; J. D. Connolly, R. McCrindle, R. D. H. Murray, A. J. Renfrew, K. H. Overton, and A. Melera, *J. Chem. Soc. (C)*, 1966, 268.

³ R. Soman, Sukh Dev, R. Misra, and R. C. Pandey, *Tetrahedron Letters*, 1964, 3767.

⁴ J. D. Connolly, D. M. Gunn, R. McCrindle, R. D. H. Murray, and K. H. Overton, *Tetrahedron Letters*, 1966, 2109.