Supporting Information to:

New Furanoditerpenoids from *Croton jatrophoides*

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Crystal Structure Determination of Isoteucvin (2)

A crystal with dimensions ca. 0.15 × 0.40 × 0.40 mm was used for data collection. A total of 1713 unique reflections were measured within the range 0 ≤ h ≤ 10, 0 ≤ k ≤ 14, 0 ≤ l ≤ 21. Of these, 1433 were above the significance level of 2.5 σ(Iobs) and were treated as observed. The range of (sin θ) / λ was 0.053–0.626 Å⁻¹ (4.7 ≤ θ ≤ 74.8°). Two reference reflections [(2 0 0), (1 2 2)] were measured hourly and showed no decrease during the 25-h collecting time. In addition, around 500 “Friedel” reflections were measured, which were used to determine the absolute configuration. Unit-cell parameters were refined by a least-squares fitting procedure using 23 reflections with 40.33 ≤ θ ≤ 43.92°. Corrections for Lorentz and polarization effects were applied. The structure was solved by the program package CRUNCH [1] and refined with XTAL [2] using scattering factors taken from Cromer and Mann and the International Tables for X-ray Crystallography [3], [4]. Structure validation, including the making of figures and tables, was carried out with PLATON [5]. The hydrogens were kept fixed at calculated positions, each with an isotropic atomic displacement parameter (U = 0.10 Å²). Anisotropic full-matrix least-squares refinement on F converged to R_f = 0.078, R_wF = 0.089, (Δσ)max = 0.01, S = 1.04. A weighting scheme w = [3. + 0.01*(σ(Fobs))² + 0.001/(σ(Fobs))]⁻¹ was used. The secondary isotropic extinction coefficient refined to g = 2291(291) [6], [7]. The absolute-structure Flack parameter refined to Xabs = 0.2, indicating that the correct enantiomer had been refined [8]. However, the large s.u. (= 12) of Xabs implies that the absolute structure is not established unambiguously. A final difference Fourier map revealed a residual electron density between −0.34 and 0.37 eÅ⁻³. The atom numbering of the refined crystal structure model is shown in Fig. 1S. The bond lengths and bond angles are close to values reported in the Cambridge Structural Database [9] for similar types of compounds.

Crystal Structure Determination of Jatrophoidin (3)

A crystal with dimensions ca. 0.20 × 0.30 × 0.75 mm was used for data collection. A total of 2022 unique reflections were measured within the range 0 ≤ h ≤ 8, 0 ≤ k ≤ 19, −10 ≤ l ≤ 10. Of these, 1884 were above the significance level of 2.5 σ(Iobs) and were treated as observed. The range of (sin θ) / λ was 0.058–0.626 Å⁻¹ (5.1 ≤ θ ≤ 74.8°). Two reference reflections [(2 0 2), (1 1 2)] were measured hourly and showed no decrease during the 70-h collecting time.
In addition, around 1500 “Friedel” reflections were measured, which were used to determine the absolute configuration. Unit-cell parameters were refined by a least-squares fitting procedure using 23 reflections with $40.00 \leq 0 \leq 50.61^\circ$. Corrections for Lorentz and polarization effects were applied. The structure was solved by the program package CRUNCH [1] and refined with XTAL [2] using scattering factors taken from Cromer and Mann and the International Tables for X-ray Crystallography [3], [4]. Structure validation, including the making of figures and tables, was carried out with PLATON [5]. The hydrogens were kept fixed at calculated positions, each with an isotropic atomic displacement parameter ($U = 0.10 \AA^2$). Anisotropic full-matrix least-squares refinement on $F$ converged to $R = 0.075$, $R_w = 0.070$, $(\Delta^f \sigma)_{\text{max}} = 0.10$, $S = 1.05$. A weighting scheme $w = [0.9 + 0.01*(\sigma(F_{\text{obs}}))^2 + 0.001/(\sigma(F_{\text{obs}}))^{-1}$ was used. The secondary isotropic extinction coefficient refined to $g = 1236(92)$ [6], [7]. The absolute structure Flack parameter refined to $X_{\text{abs}} = -0.2(6)$, indicating that the correct enantiomer was refined [8]. A final difference Fourier map revealed a residual electron density between $-0.23$ and $0.28$ eÅ$^{-3}$. The atom numbering of the refined crystal structure model is shown in Fig. 2S. The bond lengths and bond angles are close to values reported in the Cambridge Structural Database [9] for similar types of compounds.

References

2 Hall SR, Du Boulay DJ, Olthof-Hazekamp R. XTAL3.7 System. Lamb, Perth: University of Western Australia; 2000
3 Cromer DT, Mann JB. X-ray scattering factors computed from numerical Hartree-Fock wave functions. Acta Crystallogr 1968; A24: 321-4
5 Spek AL. Single-crystal structure validation with the program PLATON. J Appl Crystallogr 2003; 36: 7-13


Fig. 1S Isoteucvin (2).
Fig. 2S Jatrophoidin (3).