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REFERENCES

- 1. Nath, M. C. (1943) Ann. Biochem. Exptl. Med. 3, 55.
- Nath, M. C. (1948) Indian Pat. 36407 (1949) [Chem. Abstr. 43, 367a].
- Chow, S. Y., Chen, S. M., Yang, C. M. and Hsu, H. (1974) J. Formosan Med. Assoc. 73, 729.
- 4. Satyanarayana, K. (1969) J. Indian Chem. Soc. 46, 765.
- Qudrat-I-Khuda, M., Ali, M. E. and Rahman, M. A. (1964)
 Sci. Res. (Dacca) 1, 3, 57 [(1964) Chem. Abstr. 60, 11047e, g].

- Qudrat-I-Khuda, M., Ali, M. E. and Rahman, M. A. (1966) Sci. Res. (Dacca) 3, 84 [(1967) Chem. Abstr. 66, 52949y].
- 7. Chen, C. M. and Chen, M. T. (1976) Phytochemistry 15, 1997.
- 8. Stothers, J. B. (1972) Carbon-13 NMR Spectroscopy. Academic Press, New York.
- 9. Doddrell, D. M., Khong, P. W. and Lewis, K. G. (1974) Tetrahedron Letters 2381.
- 10. Seo, S., Tomita, Y. and Tori, K. (1975) Tetrahedron Letters 7.
- Majumder, P. L., Maiti, R. N., Panda, S. K., Mal, D., Raju, M. S. and Wenkert, E. (1979) J. Org. Chem. 44, 2811.
- Budzikiewicz, H., Wilson, J. M. and Djerassi, C. (1963) J. Am. Chem. Soc. 85, 3688.
- 13. Cheung, H. T. and Yan, T. C. (1972) Aust. J. Chem. 25, 2003.
- Biglino, G. (1961) Atti Accad. Sci. Torino. Cl. Sci. Fis., Mat. Nat. 94, 106 [(1964) Chem. Abstr. 61, 8351b].
- 15. Sim, K. Y. and Lee, H. T. (1972) Phytochemistry 11, 3341.
- Biglino, G., Cattel, L., Caputo, O. and Nobili, G. (1969) Gazz. Chim. Ital. 99, 830 [(1969) Chem. Abstr. 71, 124725a].
- Bowers, A., Halshall, T. G., Jones, E. R. H. and Lemin, A. J. (1953) J. Chem. Soc. 2555.

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PROCERANONE, A NEW TETRANORTRITERPENOID FROM CARAPA PROCERA

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Abstract—A new tetranortriterpenoid, named proceranone, has been isolated from the seeds of Carapa procera and its structure elucidated by IR, NMR and mass spectral studies.

We have recently reported [1] the isolation and structure elucidation of evodulone (1) from the seeds of Carapa procera. In continuation of our investigation on the same seeds, we have isolated α -obacunyl acetate [2], methyl angolensate [3] and a previously undescribed limonoid of novel structure which we name proceranone and formulate as structure 2.

Compound 2 had absorption bands in the IR (KBr) spectrum attributable to an acetate $(1725\,\mathrm{cm}^{-1})$, an α , β -

unsaturated carbonyl group ($1685\,\mathrm{cm}^{-1}$) and an α,β -substituted furan ($875\,\mathrm{cm}^{-1}$). Its 1H NMR spectrum was particularly informative and was similar to that of evodulone. Thus, evodulone and compound 2 both showed resonances of the same intensity and multiplicity for the protons at C-1, C-2, C-21, C-22 and C-23. The differences between the chemical shifts for corresponding hydrogens were not greater than 0.06 ppm. This similarity extended to the C-methyl region, except for the C-13

methyl group which appeared at δ 1.04 in the spectrum of 1 and δ 0.81 in that of 2. At the same time the expected difference between ring D of 1 and that of 2 was clearly discernible. Thus, the H-15 signal which appeared as a singlet (δ 3.41) in 1 was a triplet (δ 5.23, J=3 Hz) in 2. The 13 C NMR spectrum showed close parallels with that of evodulone, and a marked resemblance to that of tricoccin S₁₃ (3) [4] in ring D where the difference in the chemical shifts of C-14 and C-15 was 1.8 and 0.3 ppm, respectively.

In the mass spectrum of compound 2 additional support for the proposed structure was given by the peaks at m/e 81 and 197 corresponding to ions 4 and 5, which result from the fragmentation of rings D and B.

To conclude, we propose the structure assigned for compound 2 on the basis of the spectroscopic evidence outlined above. Further work is in progress to attempt conversion of proceranone to evodulone.

EXPERIMENTAL

Mps were recorded on a Kofler hot stage apparatus and are uncorr. Elemental microanalyses were performed by the Microanalytical Laboratory, Institut de Chimie, Strasbourg (France); IR spectra were recorded on KBr pellets and were calibrated against the 2850 and 1601 cm⁻¹ bands of polystyrene film. The 60 MHz 1 H NMR spectra were determined in CDCl₃ with TMS (δ 0) as internal standard.

Extraction and isolation. Plant material was collected and identified by the National Herbarium (1538 HNC). Dried powdered seeds (800 g) of Carapa procera, harvested near Yaoundé (Cameroon), were extracted with hexane in a Soxhlet. Evapn of the solvent under red. pres. gave a residue (200 g) which

was chromatographed over neutral alumina (1.2 kg). Elution with a mixture of hexane-EtOAc (4:1) gave an oil (150 g) and further elution with the same solvent mixture (1:1) gave a further amount of oil (40 g). TLC of the second crop after spraying with Ehrlich's reagent indicated the presence of furanoid compounds. This fraction was chromatographed over Si gel (0.063-0.200 mm mesh) and elution with hexane-EtOAc (17:3) afforded proceranone (255 mg), recrystallized from C₆H₆-EtOAc.(2:1) mp 178–179°; $[\alpha]_D^{20} + 28^{\circ}$ (CHCl₃); M⁺ 452.2549. Calc. M⁺ 452.57. (Found: C, 74.33; H, 7.96. Calc. for C₂₈H₃₆O₅: C, 74.30; H, 8.02 ° .). IR v_{max}^{KBr} cm⁻¹: 3130, 1725, 1685, 1620, 1580, 1490 and 875; ¹H NMR (CDCl₃): δ 6.53 (1H, d, J = 12 Hz), 5.87 (1H, d, J = 12 Hz), 5.40 (1H, t, J = 3 Hz), 5.23 (1H, t, J = 3 Hz), 7.25 and 6.28 (1H each), 0.81, 1.24, 1.32, 1.38, 1.47 (C-Me); ¹³C NMR (CDCl₃) (assignments were made by the aid of off-resonance and selective proton-decoupling techniques and by comparison): δ 155.7 (C-1, d), 119.8 (C-2, d), 167.6 (C-3, s), 84.8 (C-4, s), 49.2 (C-5, d), 27.4 (C-6, t), 74.3 (C-7, d), 47.0 (C-8, s), 41.2 (C-9, d), 42.2 (C-10, d), 18.2 (C-11, t), 34.4 (C-12, t), 44.1 (C-13, s), 158.3 (C-14, s), 119.6 (C-15, d), 33.4 (C-16, t), 51.7 (C-17, d), 124.5 (C-20, s), 139.7 (C-21, d), 111.0 (C-22, d), 142.6 (C-23, d), 170.1 and 21.2 (-OAc), 32.0, 27.0, 26.2, 21.1 and 16.0 (C-Me).

REFERENCES

- Sondengam, B. L., Kamga, C. S. and Connolly, J. D. (1979) Tetrahedron Letters 1357.
- 2. Dean, M. F. and Geissman, T. A. (1958) J. Org. Chem. 23, 596.
- 3. Bevan, C. W. L. (1962) J. Chem. Soc. 786.
- 4. Epe, B. and Mondon, A. (1978) Tetrahedron Letters 3901.