10 fractions par chromatographie sur colonne de polyamide (Macherey Nagel, SC6), avec élution par du C_6H_6 progressivement enrichi en MeCOEt puis en MeOH.[7] L'examen des diverses fractions par chromatographie monodimensionnelle sur CM de polyamide (Macherey Nagel, DC 11) (solvant C_6H_6, MeCOEt, MeOH en diverses proportions) et par spectrophotométrie UV permet de retenir la fraction 5 qui après une ultime purification (colonne de polyamide éluée par $CHCl_3$–MeOH–MeCOEt–Ac_2CH_2, 95:3:1,5:0,5) livre 50 mg de poudre cristalline jaune citron. Fluorescence: Brune: m.p. 300–301°: R_f sur Whatman No. 1. TBA 0,75 (lutéoline 0,77); BAW 0,77 (0,79); Forestal 0,69 (0,67); AcOH 60% 0,55 (0,52). UV. λ_{max} (EtOH): 282, 308, (328), (366) nm; composé instable dans les réactifs classiques. MS. Principaux pics situés en valeur *m/e* à: 286 (100%), 258, 257, 168 (100%), 140 (25%), 129 (12%), 121 (8%), 119 (12%), 118 (8%), 112 (17%), 84 (8%), 69 (11%). Métastables à: 233 (286 → 268), 116,6 (168 → 140), 98,6 (286 → 168), 89,6 (140 → 112), 63 (112 → 84). NMR. (CD_3OD, 60 MC); 2H, *d*, δ 7,80 × 10^{-6} (*J* 8,5 et 2,5 Hz); 2H, *d*, δ 6,80 × 10^{-6} (*J* 8,5 et 2,5 Hz); 1H, *s*, δ 6,43 × 10^{-6}; 1H, *s*, δ 6,19 × 10^{-6}. IR. *1650*, 1615, *1590*, 1565, *1510*, *1440*, 1415, *1340*, 1325, *1300*, 1275, *1240*, 1200, *1185*, 1160, 1090, *1005*, *960*, *830* cm^{-1}.

Réaction de transposition de Wessely-Moser. 20 mg d'isoscutellaréine traités par HCl 4 N au BM bouillant pendant 45 min donnent, après refroidissement et purification sur colonne de polyamide, 15 mg de poudre cristalline identifiée comme scutellaréine par comparaison spectrale et chromatographique avec un échantillon de référence fourni par le Dr. Harborne.

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ALKALOIDS, TRITERPENES AND OTHER CONSTITUENTS OF *DISCARIA CRENATA*

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(*Received* 31 *October* 1972. *Accepted* 28 *November* 1972)

Key Word Index—*Discaria crenata*; Rhamnaceae; triterpenes; benzylisoquinoline alkaloids; peptide alkaloid.

Plant. Discaria crenata (Clos.) Regal. *Source.* Concepcion. Material collected in September (winter).

Present work. The dried, powdered plant material (3·3 kg) was extracted with C_6H_6 and then with alcohol. The C_6H_6 extract (81 g) was treated with 1 N HCl (100 × 15 ml) until all the alkaloids were removed (negative Mayer test). The acid fraction was basified and extracted with $CHCl_3$ to afford an alkaloid fraction (M) (1·9 g). Evaporation of the remaining benzene extract gave a neutral fraction (N). The EtOH extract was similarly

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macerated with 1 N HCl until free from basic constituents. The acidic solution was basified and extracted with $CHCl_3$ to provide a second alkaloid fraction (O) (1·2 g).

Fraction N. This was chromatographed through alumina (5 g extract on 60 g Al_2O_3, grade III) to give; *Fatty alcohol* (eluted with light petrol.), m.p. 73–75°. Its MS fragmentation pattern indicated that this was a mixture of homologues. *Lupenone* (eluted with 2:1 light petrol.–C_6H_6), m.p. 160–166°, $[\alpha]_D^{21}$ +57° (*c* 1·0, $CHCl_3$) (lit.[1] +57°), identical to an authentic sample. *Sitosterol,* (2:1, light petrol.–C_6H_6), m.p. 135°, again identical to an authentic sample.[2] *Lupeol* (C_6H_6), m.p. 210–215°. Both it and its derived acetate were identical to an authentic sample. *Ursolic acid* (1:9, EtOH–C_6H_6), m.p. 285–290°, identified by direct comparison with an authentic sample and by preparation of its acetate, methyl ester, and its methyl ester acetate. *Unidentified acid,* m.p. 70°, possibly contaminated with homologues this material had ν_{max}^{NaCl} 2941, 1701, 1468, 1307, 940 cm^{-1}, *m/e* 396, 383, 368, 354, 340, 326, 312, 297, 185, 129, 115 and 111. Treatment with CH_2N_2 afforded a monomethyl ester, m.p. 43–45°. The acid is probably mainly docosanoic acid ($C_{22}H_{44}O_2$).

Fraction M. The material (1·9 g) was chromatographed through alumina (50 g, grade III) to give a crystalline *peptide alkaloid* (96 mg), m.p. 211–212° (EtOH), M^+ 568. This alkaloid did not correspond to any previously reported in the literature.[3] Work on its structure is continuing and will be reported on later.

Fraction O. After chromatography through alumina (50 g, grade III), this fraction afforded, *an alkaloid* (39 mg), (eluted with C_6H_6) m.p. 228–231° (EtOH), *m/e* 341, 325, 310, 221, 206, 190, 178, 157, 150, 135, 121 and 43. *Armepavine*[4] (eluted with 1:19, EtOH–C_6H_6), m.p. 136°, identical to an authentic sample. N-*methylcoclaurine* (1:1, EtOH–C_6H_6) (10 mg), m.p. 181–183° (lit.[5] m.p. 184–185°), identical with an authentic sample.

An extraction of the dried leaves of the plant (200 g) with EtOH, followed by concentration and dilution with H_2O afforded a sample of the flavanoid glycoside, *rutin* (0·5 g), m.p. 173–175°. This material was identical to an authentic sample[6] and, after hydrolysis, afforded quercetin, glucose, and rhamnose.

Acknowledgements—This work was supported by the Organization of American States (Grant PMC-8/1). We thank CONICYT, Chile for a Research Studentship (P.P.) and for financial support (S.M.A.). We thank Dr. Pfeifer for a sample of armepavaine, Professor C. Marticorena for the botanical identification and Dr. P. G. Sammes for the spectral information.

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