

*Palmitic Ester of a Triterpene*

IR (Nujol) 3050 (cyclopropane  $\text{CH}_2$ ) 1730 ( $\text{C}=\text{O}$ ) 1260  $\text{cm}^{-1}$  ( $\text{C}-\text{O}-\text{C}$ ). Mass spectrum  $m/e$  (%) 664-6131 (15)  $\text{M}^+$  ( $\text{C}_{46}\text{H}_{80}\text{O}_2$  requires 664-6150), 649 (8), 409 (30), 408 (17), 394 (8), 393 (25), 339 (5), 299 (4), 298 (4-5), 297 (5), 286 (4), 271 (6), 257 (7), 239 (10-5), 229 (13), 218 (25), 205 (35), 204 (40), 189 (60), 191 (25), 190 (25), 177 (20), 175 (25), 173 (15), 163 (17), 161 (25), 159 (15), 149 (33), 147 (35), 137 (40), 136 (30); other strong peaks at 135 (55), 123 (50), 121 (57), 109 (88), 107 (60) with base peaks at  $m/e$  41, 43, 55 and 57.

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## RANUNCULACEAE

IDENTIFICATION OF DELSOLINE FROM *DELPHINIUM AJACIS*\*<sup>1</sup>

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*Plant.* *Delphinium ajacis*—Commonly called Larkspur.

*Source.* Seeds purchased from G. J. Ball, Incorporated, West Chicago, Illinois, and grown both in the Horticulture Greenhouses and in the field plots of the Agricultural Experiment Station at Stillwater.

*Uses.* Possess insecticidal properties and plants were responsible for poisoning of cattle in western United States.<sup>1</sup>

*Previous work.* In earlier communications<sup>2,3</sup> on biosynthesis studies of diterpenoid alkaloids, the isolation of an unknown alkaloid designated as LBA-III was reported. This communication presents the conclusive identification of LBA-III as delsoline (I).<sup>4</sup>

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<sup>2</sup> G. M. FROST, R. L. HALE, G. R. WALLER, L. H. ZALKOW and N. N. GIROTRA, *Chem. & Ind.* **320** (1967).

<sup>3</sup> S. D. SASTRY, G. R. WALLER and H. BURSTROM, *Am. Chem. Soc. Abstr.*, 156th National Meeting, Atlantic City, New Jersey (1968), *Biol.* **20**.

<sup>4</sup> V. SKARIC and L. MARION, *J. Am. Chem. Soc.* **44** 34 (1958).

*Isolation of alkaloids.* The alkaloids were isolated from *Delphinium ajacis* seeds by chromatography on neutral alumina. The final purification was done using TLC in  $C_6H_6$ -EtOAc-HNEt<sub>2</sub> (7:2:1) using blank plates and by final recrystallization from MeOH-CHCl<sub>3</sub>.

*Delsoline.* Several crystallizations of the crude compound yielded an alkaloid with a constant m.p. of 217-218° which was not depressed when admixed with authentic delsoline. IR and mass spectra of the isolated sample were superimposable on those of an authentic sample. Significant fragment ions in the mass spectrum were  $M^+$  467,  $m/e$  452 [M-15-CH<sub>3</sub>],  $m/e$  450 [M-17 (-OH)],  $m/e$  436 [M-31 (-OCH<sub>3</sub>)],  $m/e$  434 [M(-18-15)(-H<sub>2</sub>O-CH<sub>3</sub>)] and  $m/e$  278 ( $C_{16}H_{24}NO_3$ )<sup>+</sup>. High resolution mass spectral analysis of the isolated delsoline supported these fragmentations.

*Mass Spectra.* A prototype<sup>5</sup> of the LKB-9000 gas chromatograph-mass spectrometer, (LKB Instruments Incorporated, Rockville, Maryland) and a CEC-110 B, DuPont Instruments, CEC Analytical Division, Monrovia, California, were used. The LKB mass spectrometer was operated at 70 eV, 3.5 kV accelerating voltage, 65  $\mu$ A trap current, 290° ion source temperature and 30-55° direct probe temperature; the high resolution spectra: accelerating voltage 8000 V, ionizing voltage 70 eV, probe temperature 160-170° with an indicated source pressure  $2 \times 10^{-6}$  mm.

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<sup>5</sup> G. R. WALLER, *Proc. Okla. Acad. Sci.* **47**, 295 (1968)