

"The Alkaloids of *Crotalaria spectabilis* Roth." By C. C. J. Culvenor
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The following paragraphs were omitted from the Experimental Section of the abovementioned paper:

(k) *Hydrogenolysis of Diacetylmonocrotaline*.—(i) Hydrogenation of diacetylmonocrotaline (1.0 g) in ethanol solution with Raney nickel catalyst ceased after 1.8 moles of hydrogen had been absorbed. After removal of catalyst and solvent, the residue was taken up in aqueous alkali and extracted with chloroform to give a basic gum (0.3 g) R_F 0.29, 0.37. Acidification of the aqueous residue and further ether extraction gave an acid (0.48 g) which was chromatographed on silica gel and crystallized from benzene when it formed plates, m.p. 115 °C, undepressed on admixture with anhydromonocrotalic acid. The basic product was chromatographed on alumina; light petroleum eluted the base of R_F 0.30 (0.12 g) which formed plates, m.p. 78 °C, undepressed on admixture with deoxyretronecine, and benzene eluted the base of R_F 0.37 (0.04 g) which appeared to be retronecanol but was not identified with certainty. The isolation of deoxyretronecine is a result of incomplete reduction.

(ii) The reduction of diacetylmonocrotaline was effected as in (i) but the acidic product was recovered before the basic product (i.e. it was not subjected to alkaline conditions). The acid obtained, a red oil (0.18 g) R_F 0.70, was chromatographed on silica gel. The major fraction formed a brucine salt, m.p. 186 °C, undepressed on admixture with the brucine salt of acetylmonocrotalic acid (Found: C, 63.3; H, 6.6; N, 4.5; O, 25.4%. Calc. for $C_{10}H_{14}O_6 \cdot C_{23}H_{26}O_4N_2$: C, 63.5; H, 6.4; N, 4.5; O, 25.6%).

(l) *Acetylmonocrotalic Acid*.—Monocrotalic acid (0.3 g) was refluxed for 7 hr with acetic anhydride (3 ml) containing a drop of pyridine, excess reagent evaporated under reduced pressure, and the product chromatographed on silica gel to give *acetylmonocrotalic acid* (0.21 g) as a syrup, R_F 0.72, which crystallized on keeping but which could not be recrystallized from a solvent. From ethanol it formed a *brucine salt*, m.p. 185 °C (Found: C, 62.9; H, 6.5; N, 4.6; O, 25.4%. Calc. for $C_{10}H_{14}O_6 \cdot C_{23}H_{26}O_4N_2$: C, 63.5; H, 6.4; N, 4.5; O, 25.6%).

Heating acetylmonocrotalic acid (0.8 g) in 10% aqueous HCl at 100 °C for 5 hr and extracting the solution with ether gave a crystalline acid (0.65 g) R_F 0.56, which formed from acetone, colourless needles, m.p. 183-184 °C, undepressed on admixture with monocrotalic acid.

Keeping acetylmonocrotalic acid in dilute alkaline solution at room temperature for 3 days, acidifying, and extracting with ether, gave an oily acid, R_F 0.63, which formed a brucine salt, m.p. 140.5-141.5 °C, from ethanol (Found: C, 63.0; H, 7.3; N, 5.0%. Calc. for $C_7H_{16}O_5 \cdot C_{23}H_{26}O_4N_2 \cdot 2H_2O$: C, 62.7; H, 7.3; N, 4.9%). Attempts to prepare pure monocrotic acid for comparison were unsuccessful.