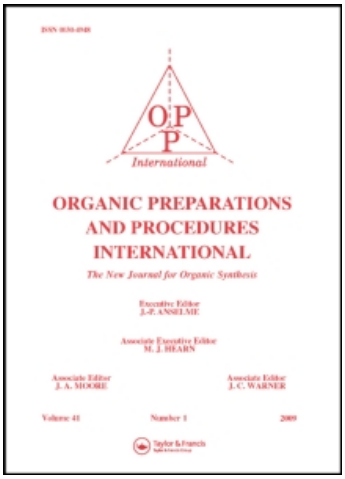

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α-ACETAMIDO-β-(1-AZULYL) PROPIONIC ACID

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α-ACETAMIDO-β-(1-AZULYL)PROPIONIC ACID

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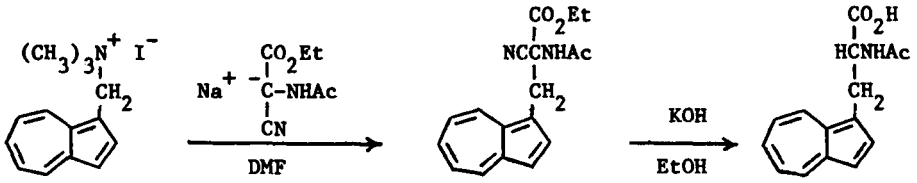

1 2 3

The title compound is a new azulene derivative and the azulene analog of N-acetylphenylalanine.

Experimental

α-Acetamido-β-(1-azulyl)propionic acid. To a stirred suspension of 0.6 g of NaH (mineral oil suspension) in 20 ml of anhydrous, redistilled DMF was added carefully, under an atmosphere of dry nitrogen, 1.3 g (7.73 mmol) of dry ethyl acetamidocyanoacetate.[1] After the evolution of hydrogen had ceased, 900 mg (2.75 mmol) of 1-azulylmethyltrimethylammonium iodide (1)[2] was added and the mixture was heated at

100-110° for 3.5 hr. After removal of the solvent in vacuo, the residue was chromatographed over neutral Al_2O_3. The blue fraction eluted with 3:1 CH_2Cl_2-petroleum ether afforded 836 mg of a blue oil formulated as ethyl α-acetamido-α-cyano-β-(1-azulyl)propionate (2): uv (CH_2Cl_2) (D_{max}) 235 (0.40), 279 (1.0), 284 (0.92), 289 (0.85), 343 (0.11) and 358 mμ (0.06); visible (CH_2Cl_2) (D_{max}) 582 (1.0), 630 (0.93), and 692 (0.31) mμ.

A solution of 340 mg of the blue oil in 20 ml of a 10% solution of KOH in 50% aqueous EtOH was heated under reflux for 2 hr. The solution was then added to 50 ml of H_2O in a separatory funnel and the mixture was extracted with 100 ml of ether. The separated aqueous layer was acidified with 6N hydrochloric acid, the organic phase extracted into ether, and the solvent removed from the dried (Na_2SO_4) ethereal solution. The residue was chromatographed over silica gel and the blue fraction eluted with ether yielded 179 mg (63%) of 3 as blue crystals, mp 122-126°. Recrystallization from water gave 171 mg (60.5%), mp 126-128°: uv (MeOH) (D_{max}) 238 (0.36), 277 (1.13), 283 (1.0), 331 (0.05), 343 (0.09) and 358 mμ (0.04); visible (MeOH) (D_{max}) 595 (1.04), 643 (0.87) and 710 mμ (0.33); ir (CCl_4 mull) 2.98, 3.45, 5.8 (shoulders), 6.22, 6.32, 6.5, 7.15, 7.23, 7.45 and 8.12 μ.

Anal. Calcd for $C_{15}H_{15}NO_3$: C, 69.99; H, 5.85; N, 5.44. Found: C, 69.66; H, 6.12; N, 5.20.

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