le charbon actif, le L-(+)-bornésitol est recristallisé dans MeOH. F. 206°, α_D^{25} + 32,1, IR, comparaison avec un échantillon authentique. Quantités isolées: 0,5 à 2% du matériel sec

La présence du L-(+)-bornésitol est uniforme à l'intérieur de la section Coelanthe et pourrait être caractéristique de cette dernière. De plus, elle est particulièrement intéressante pour la chimiotaxonomie au niveau des familles constituant l'ordre des Contortae ou Gentianales, le L-(+)-bornésitol n'ayant été décelé que dans deux familles proches des Gentianacées, les Apocynacées[4–9] et les Rubiacées.[10]

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OCCURRENCE OF A NEW AMINO ACID IN *CROTALARIA* SEEDS

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Key Word Index—*Crotalaria juncea*, Legaminosae, amino acids, 5-hydroxy-2-aminohexanoic acid

Seeds of *Crotalaria juncea* and several other species of this genus (*C. medicagenia, C. anagradis, C. striata* and *C. laburnifolia*) revealed, among other amino acids, the presence of an unidentifed compound in varying concentration that was strongly ninhydrin positive. Earlier investigators isolated from *C. juncea* seeds an optically inactive amino acid which they identified as β-hydroxy-*N*-methyl-(±)-norvaline. Pant and Kapur[2] also observed an unidentifiable peak in the seeds of *C medicagenia* while analysing them for their free amino acids in an automatic analyser. With a view to establish the identity of this compound with Adams and Gianturco's optically inactive amino acid, the unidentifiable ninhydrin positive compound was isolated from *C juncea* seeds in pure form The present communication describes the details of its isolation and assignment of structure.

A new amino acid has been isolated from *C. juncea* seeds. The MS of the compound reveals an (M + H) ion at *m/e* 148 indicating the MW of 147 ($C_6H_{13}NO_3$) The presence of ions at

[1] ADAMS, R. and GIANTUROC, M (1956) *J Am Chem Soc* **78,** 1919
[2] PANT, R and KAPUR, A S (1963) *Ann Biochem Exptl Med* **23,** 95

130 and 102 respectively and metastable ions at 114 1 and 71·1 further prove this. The important ion is *m/e* 84 which is the loss of $H_2O + COOH_2$. This proves that in addition to the COOH group, the compound has a hydroxyl group. This can also be seen by the small peak at *m/e* 112 for loss of $2H_2O$ from (M + H). These observations suggest that the compound could be 5-hydroxy-2-amino hexanoic acid

The NMR spectrum of the compound performed at 100 mc and at pH 10 showed five groups of peaks in the ratio of 3:2·2 1·1 at δ 1·16 (*d** J = 6 Hz), δ 1·52 (*m*), δ 1·80 (*m*), δ 3·61 (*m*), δ 3·79 (*q* J = Hz). These are assigned to the terminal methyl, the 4-methylene, the 3-methylene and the 2- and 5-hydrogens respectively. The splitting patterns appear straightforward; the 3 and 4 methylene groups exhibit complex coupling with each other and the adjacent hydrogens at C-2. Fortunately, the methylene group at C-4 is coupled to the hydrogen at C-5 in a manner that allows the coupling between the C-5 hydrogen and the C-6-methyl group (6 Hz) to be dominant. Proof that this assignment is correct is provided by the observation that the multiplet at δ 3·61 moves downfield at δ 3·72 at pH 2. Thus it is concluded that the compound is 5-hydroxy-2-amino-hexanoic acid. This compound (under the name of δ-hydroxy norleucine) was obtained by Takita and Naganawa[3] from various ilamycins. Hudlický and Kakáč[4] synthesized the compound by the hydrolysis of either ethyl-2-ethoxy-carbonyl-2-acetamido-5-fluorocaproate or 2-ethoxy-carbonyl-2-acetamido-δ-caprolactone. Although the elementary analysis and the empirical formula, MW etc. reported by these authors concur with our data, their reported IR bands do not appear to check with ours. However, this could be explained on the basis that the synthesized material probably contained four stereoisomeric components.

TABLE 1 R_f OF THE AMINO COMPOUND DEVELOPED WITH DIFFERENT SOLVENT SYSTEMS

Solvent	R_f
n-BuOH–HOAc–H_2O (12:3:5)	0 41
n-PrOH–H_2O (3 1)	0 56
i-BuOH–HCO_2H–H_2O (15.3 2)	0 87
EtOAc–HOAc–H_2O (3·1 1)	0·92
Pyridine–*i*-AmOH–H_2O (8 4.7)	0 53
Phenol–concn NH_4OH	0 76

EXPERIMENTAL

Seeds of *C juncea* obtained from Pratap Nursery, Dehradun, India, were powdered in a grinder to 100 mesh and defatted with light petrol (60–80°) 500 g defatted powder were extracted with EtOH (70% v/v) until the extract gave negative ninhydrin test The pooled extract was concentrated *in vacuo* to about 200 ml, filtered and passed through the anion exchanger column of De-Acidite FF in the Cl′ form The effluent was collected and the column washed with H_2O till the effluent was negative to ninhydrin test The effluent (2000 ml) was concentrated *in vacuo* to a small volume (*ca* 150 ml) and passed through the cation exchanger column of Zeo-Karb 225 in the H^+ form The column was washed with H_2O until the pH of the effluent rose to 5 and then eluted with aq 0 5 N ammonia Paper partition chromatography revealed the eluate to contain the unidentified amino compound along with traces of basic amino acids

The elutate was made ammonia-free by concentrating *in vacuo* and subsequently was evaporated to dryness The residue mainly consisting of the unknown base was dissolved in the smallest possible quantity of hot aq. EtOH (10 ml, 50% v/v) and filtered MeOH–acetone mixture (1.1, v/v) was then added to the filtrate in small

* *d* = doublet, *m* = multiplet, *q* = quadruplet

[3] TAKITA, T and NAGANAWA, H (1962) *Antibiotics (Tokyo), Ser. A* **16,** 246

[4] HUDLICKÝ, M and KAKÁČ, B (1966) *Collection Czechoslov Chem Commun* **31,** 1101

quantities (*ca* 50 ml) till the soln became turbid and then refrigerated overnight The white crystalline solid (*ca* 200 mg) was filtered, washed with Et_2O and dried over ($CaCl_2$) in vacuum The solid was re-crystallized from aq MeOH and finally from H_2O (Yield 150 mg) The compound melted at 235° with decomposition, ran as a single spot on the chromatogram in all the solvents tested (Table 1) and gave positive reaction with ninhydrin Unlike some other basic amino acids, this compound did not yield either a picrate, flavianate or copper complex (Found C, 47 69, H, 8 1, N 9 03 and O, 35 18)

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β-PHENETHYLAMINE AND TETRAHYDROISOQUINOLINE ALKALOIDS OF *DESMODIUM CEPHALOTES**

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Key Word Index—*Desmodium cephalotes* Leguminosae; β-phenethylamine; tyramine; hordenine; candicine; (±)-salsolidine

Plant. Desmodium cephalotes Wall (tribe: Lotoideae). The plant material was supplied by Messrs United Chemical and Allied Products, Calcutta. A voucher specimen has been preserved at the Pharmaceutical Chemistry Research Laboratory *Source*: The plant grows in India in the Northern Circars, Hills of the Deccan and Carnatic, and Western Ghats up to 3000 ft, in forest undergrowth, especially with teak in the South, with Sal in the North *Uses*: Different parts are used in the Indian system of medicine as a cure for dysentery, in bronchial spasms and coughs, as a central stimulant.

Previous work. On sister species, viz., *D pulchellum*,[1] *D gangeticum*,[2] *D triflorum*,[3] *D gyrans*,[4] *D tiliaefolium*,[5] *D. floribundum*[6]

Plant part examined. Stem-roots, leaves In a typical experiment, air-dried and powdered stem-roots (3 2 kg) were continuously extracted first with light petroleum, then with EtOH (16 hr, each). The extractives were separately processed according to a previously described procedure.[7] Separation of the mixture of alkaloids from the different fractions was accomplished by gradient-pH extraction, fractionation into phenolic and non-phenolic bases over Amberlite-IRA 400 (HO^-) resin column, and by column and layer chromatography. The identity of the individual entities was established by co-TLC with authentic markers, correspondence of m.p where possible, spectral evidence (UV, IR, PMR, MS), and derivatization.

* Part VI in the series "*Desmodium* Alkaloids" For Part V see Ref 5

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