EXPERIMENTAL

Chemicals Phellamurin has been isolated from the leaves of *Phellodendron amurense* by the method of Hasegawa and Shirato [1]

Culture Stock culture of *Aspergillus niger* was maintained on agar slants The growth medium was the modified Czapek-Dox medium with some microelements ($FeCl_3$ $6H_2O$, 20 mg $ZnSO_4$ $7H_2O$ 10 mg $MnSO_4$ $4H_2O$ 3 mg Na_2MoO_4 $2H_2O$ 1 5 mg $CuSO_4$ $5H_2O$ 1 mg) glucose 20 g and phellamurin 0 1 g and its pH was adjusted to 4 5 with HCl The soln of phellamurin and remaining ingredients were sterilized separately and combined aseptically in the flasks prior to inoculation 1 l of the liquid culture medium was inoculated with spores grown on 5 slants and incubated for 4 11 days at 25

Isolation 2 l of liquid medium were filtered and extracted with Et_2O After removal of Et_2O the remaining mass was dissolved in EtOH and applied to a column of polyamide The column was eluted successively with 100 ml each of 0 20 40, 60 80 100% aq EtOH The fractions eluted with 60 and 80% EtOH were concentrated and examined by TLC on silica gel plates with a solvent $CHCl_3$ EtOAc HCOOH (5 4 1) Neophellamuretin R_f 0 8) was isolated from silica gel plates with EtOH and recrystallized from EtOH H_2O

Neophellamuretin From EtOH m p 189 190 (Found C 67 30 H 5 73 $C_{20}H_{20}O_6$ requires C 67 41 H 5 61%) *m/e* 356 (25%) Acetate m p 125 126 (from dil EtOH) ν_{max}^{Nujol} 2860 2833 1760 1690 1610 1370 (δ) cm^{-1}

Hydrolysis of phellamurin Phellamurin (0 1 g) was hydrolyzed with 0 1 g β-glucosidase The aglycone neophellamuretin was extracted with Et_2O and recrystallized from EtOH m p 190 Sugar was determined as glucose with PC

Acid treatment Neophellamuretin (20 mg) was heated in 5% H_2SO_4 added with small vol of EtOH at 100 for 3 hr After evaporation of EtOH the solution was extracted with Et_2O phellamuretin obtained was recrystallized from EtOH m p 221 Acetate m p 199 NMR ppm 1 33 (6H s gem-dimethyl) 1 68 and 2 59 (2H each *t J* 7 Hz H-5′ and H-4″), 1 99 (3H s aliphatic acetyl group) 2 30 and 2 34 (3H each s aromatic acetyl group) 5 35 and 5 56 (1H each *d J* 12 Hz H-2 and H-3) 6 22 (1H, s H-6) 7 15 (2H *d J* 9 Hz H-3 5) 7 44 (2H *d J* 9 Hz H-2 6)

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ALKALOIDS OF *DATURA DISCOLOR*

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Key Word Index – *Datura discolor*, Solanaceae tropane alkaloids hyoscine tigloyl esters cuscohygrine

Plant *Datura discolor* Bernh A species native to the desert regions of south-eastern California and Mexico, sometimes confused with *D meteloides* D C which is found in similar locations but may be distinguished from the latter species by the presence of 5 purple flushes in the throat of the corolla and by its pentagonal calyx and characteristic seeds Taxonomically it accords with Safford's section Dutra[1] of the genus *Previous work* Pharmacognostical description assay of total alkaloids in morphological parts and characterization of hyoscine as principal alkaloid [2] Ontogenetic production of total alkaloids of

[1] SAFFORD, W E (1921) *J Wash Acad Sci* **11**, 173
[2] KALFMKIARIAN P H and MILLER O H (1957) *J Am Pharm Assoc* **46**, 393

Egyptian-grown plants [3] *Plant material* Raised from seeds collected 1959 in Mexico by Dr H S Gentry and kindly supplied by Dr A S Barclay, U S Dept of Agriculture Herbarium specimens of original collection (No 18 387) deposited in U S National Museum, Smithsonian Institute, Washington D C and in the Lundell Herbarium, Texas Research Foundation, Renner

Identification of alkaloids Alkaloids obtained by chromatographic fractionation of the Et_2O extract of the dried plant material are recorded below, characterization is indicated as follows P, m p and m m p of picrates, IRP, comparison of IR spectrum of picrate with that of authentic compound, C, co-chromatography (C1–C3, TLC), C1, alumina (Et_2O), C2, alumina (Et_2O–EtOH, 1 1), both visualized with I_2 in CCl_4, C3, silica gel ($CHCl_3$–Et_2NH, 9 1), visualized with iodoplatinate reagent, C4, paper (light petrol, b p 60–80°–amyl alcohol–HOAc–H_2O, 1 3 3 3) visualized with modified Dragendorff's reagent

Aerial parts Total alkaloid, 0 17% (dry wt) as hyoscine *Hyoscine* (principal alkaloid, 0 08%) C2, C3, P, IRP *Apohyoscine* C1 C2, P, IRP *Norhyoscine* C2, P, IRP *Hyoscyamine* (0 01%) C2, C3, P, IRP *Meteloidine* C2, C3, P, IRP *Tropine* C4 *ψ-Tropine* C4 In addition to the above, two weak bases giving alkaloid-positive reactions were detected, one had R_f 0 80 (C1) and the other had an R_f similar to that of 3-tigloyl-6-acetoxytropane (C1)

Roots Total alkaloid, 0 31% (dry wt) as hyoscine *Hyoscine* (0 02%) C2, C3, P, IRP *Norhyoscine* C2, C3, P, IRP *Atropine* (0 01%) C2, C3, P, IRP *Littorine* C2, C3, m p, m m p and IR spectrum of aurichloride [4] *Meteloidine* C2, C3, P, IRP 3α,6β-*Ditigloyloxytropane* C1, C2, P, IRP 3α,6β-*Ditigloyloxytropane-7β-ol* C1, C2, P, IRP *Cuscohygrine* (principal alkaloid, 0 06%) C1, C2, P, IRP *Tropine* C4 *ψ-Tropine* C4 Traces of bases having R_f (C3) similar to those of noratropine and acetoxytropane

The wide spectrum of alkaloids contained in the aerial parts and roots of *D discolor* is similar to that found in other species of the genus As with *D metel*, *D meteloides* and *D innoxia*, also of the section Dutra, hyoscine is the predominant alkaloid of the aerial parts (in the same section, *D leichhardtii* and *D prunosa* contain hyoscyamine as principal alkaloid) Cuscohygrine and littorine as constituents of the roots accord with findings[5] for other members of the genus but cuscohygrine (20% of total alkaloid of the roots, two samples examined) has not been recorded as the main base of other *Datura* roots 3α-Tigloyloxytropane and tigloidine, two minor components of a number of other species, were not detected

EXPERIMENTAL

The cultivation of plants and the extraction and fractionation of alkaloids was carried out as described previously[6] for *D prunosa*

[3] SABER, A H, BALBAA, S I, EL HOSSARY, G A and KARAWYA, M S (1970) *Lloydia* **33,** 401
[4] EVANS, W C and MAJOR, V A (1968) *J Chem Soc* 2775
[5] EVANS, W C, GHANI, A and WOOLLEY, V A (1972) *Phytochemistry* **11,** 2527
[6] EVANS, W C and TREAGUST, P G (1973) *Phytochemistry* **12,** 2077