RESEARCH PAPERS

THE ALKALOIDS OF THE GENUS *DATURA*, SECTION BRUGMANSIA

PART I. *D. CORNIGERA* HOOK

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Hyoscine has been isolated as the main alkaloid from the leaves, flowers, pericarp and seeds of *Datura cornigera* Hook. Noratropine occurs in the leaves, flowers and pericarp. The following alkaloids have been obtained from the roots, (−)-3α,6β-ditigloyloxytropane, 7-hydroxy-3,6-ditigloyloxytropane, hyoscine, hyoscyamine, atropine, norhyoscyamine, noratropine, 3α,6β-dihydroxytropane and the presence of others indicated. Hyoscyamine is the principal alkaloid of the whole roots but the root-wood, although giving a low yield of total alkaloids, contains a relatively high proportion of noratropine.

THE taxonomy of the *Brugmansia* group of species presents certain difficulties, the group being variously regarded as a section of the genus *Datura* (Bernhardi, 1833; Safford, 1921) or as having generic rank (Persoon, 1805; Lagerheim, 1895; van Zijp, 1920; van Steenis, 1930–31). Its members are widely distributed throughout Central and South America where they have been cultivated as ornamental plants and used by the Indians for their narcotic properties (Safford, 1920). Safford (1921) listed 14 species of this Section and Barclay (1959) has recently described a new species, *D. vulcanicola*. A number of the cultivated forms have been discussed by De Wolf (1956). Satina and Avery (1959) have indicated that more studies are necessary to clarify the taxonomy of the genus and in particular the relationship of the *Brugmansia* group of species to the other Sections. They suggest a fresh approach with new criteria and data because a classification based almost exclusively on morphological descriptions and on phenotypical resemblances and differences is apparently insufficient for the proper determination of these solanaceous species. But, for the present, it is necessary to identify species as adequately as possible by reference to the information available and, for this reason, we have given below, those features of the plant on which we have based our identification. *D. cornigera* was described by Hooker (1846) from a cultivated plant of unknown origin and was later recorded by Hemsley in 1882 in the valley of Mexico (Matuda, 1952) and by Lagerheim (1895) as cultivated around Quito and probably occurring wild somewhere in the forests of Ecuador. The species is closely related to *D. arborea* L.

No chemical investigation of *D. cornigera* appears to have been reported but it is pertinent to record the results of investigations carried out on the so-called *D. arborea*. Kircher (1905) investigated the distribution of alkaloids in *D. arborea*—"also known as *Brugmansia candida*". If the material was *B. candida* Pers., it can be regarded as *D. arborea* Ruiz and

Pavon., a plant having larger flowers than *D. arborea* L., with the margins of the limb between the corolla teeth being entire or rounded. Kircher's description of the plant does not make complete identification possible but the leaves with soft hairs on both sides are not consistent with De Wolf's (1956) characters of *D. arborea* L. The plants were grown in the botanical garden, Marburg and found to contain mainly hyoscine in the leaves and flowers and, hyoscine with some hyoscyamine in the young stems and roots. In contrast Schmidt and Kircher (1906) found the seeds of *D. arborea*, obtained from "foreign" plants, to contain hyoscine and hyoscyamine in the ratio 1 : 4. Montesinos (1939) reported "Datura Arbórea" of Peru to contain in the roots, leaves, flowers and seeds, 0·16, 0·15, 0·116 and 0·12 per cent alkaloids respectively. In the leaves hyoscine was the main alkaloid, together with small quantities of atropine and hyoscyamine. Three varieties of the plant are mentioned differing in the form of the flowers; from the photographs they could be *D. arborea* L., the double-flowered form of *D. cornigera*, often known as *D. knightii* and *D. candida* respectively. Montesinos used chiefly the third variety for his studies. Barriga Villalba, Medina and Albarracin (1945) give the alkaloidal content of the dry flowers, leaves and berries of *D. arborea* as 0·490, 0·287 and 0·063 per cent respectively, with hyoscine as the main alkaloid. In contrast Suárez (1952) found no significant amounts of alkaloid in a cultivated variety of *D. arborea* and, in a phytochemical investigation of a Peruvian sample, Aguero (1943) records only 0·02 per cent of alkaloids. Chlorogenic acid (Politas, 1948), scopoletin and aesculetin (Kala, 1958) have also been reported as constituents of *D. arborea*.

In view of this rather limited knowledge concerning the alkaloidal constituents of the white flowered tree daturas the following investigation on *D. cornigera* has been undertaken as part of a more extensive phytochemical examination of the whole genus.

Plant Material

The original seed samples were obtained in 1953 from Cochabamba, Bolivia and first year plants raised in 1954. Subsequent propagation was by cuttings and, more recently, by seed produced by the latter. Two leaf-varieties were evident among the plants, one with leaves having a sinuate margin and a covering of soft hairs (Type I) and the other with angular-toothed leaves and a rough texture (Type II). The flowers of both varieties were similar in form and in measurements of peduncle, calyx, corolla, corolla lobe, pistil, stamens and anthers. Type I produced more flowers than Type II; no fruits could be obtained from either variety by self-pollination but by cross-pollination of the two varieties, Type I bore fruits with viable seeds. This is consistent with the field studies of Barclay and Schultes who tell us that they find that in the wild state, the *Brugmansia* often require cross-pollination between different clones for the successful production of fruits.

We have identified these plants as *D. cornigera* Hook, a species very closely related to *D. arborea* L. and possibly a cultivar of it. The material

agrees well with Hooker's (1846) description of the species and is identical with his illustration. The plants can be distinguished from the other white-flowered forms of tree daturas by the following points. The deciduous calyx which is not toothed at the apex and the size of the flower are inconsistent with *D. candida* (Pers.) Safford (synonyms *D. arborea* Ruiz and Pavon; *Brugmansia candida* Pers.). The long calyx (up to 19 cm.) tapering to a horn-like recurved point, differentiates the plant from *D. arborea* L. which has a relatively short calyx and no horn-like point and from *D. affinis* Safford (synonym *Brugmansia arborea* Lagerh.) which also has a short calyx (8·5–10·0 cm.) but with five teeth. Hooker records no measurements for the flowers but Lagerheim (1895) in his study of the Ecuadorian species differentiates more fully between *D. cornigera* Hook. and a plant he regarded as *D. arborea* L., but which Safford (1921) subsequently renamed *D. affinis*. Our plants agree well with Lagerheim's description of *D. cornigera* with the exception of lengths of corolla and stamens which are intermediate between the values he records for the two species. Neither of the two types of leaves shown by Type I and Type II is inconsistent with the descriptions of Hooker and Lagerheim but leaf-shape as a distinguishing feature (De Wolf, 1956) would seem to be of doubtful value. Other white-flowered species, also differentiated in Safford's key by the toothed calyx but having other marked differences from *D. cornigera* are *D. suaveolens* Humb. and Bonpl., *D. dolichocarpa* (Lagerh.) Safford and *D. longifolia* (Lagerh.) Safford.

Experimental

Alkaloids of the Aerial Parts

The air-dry, coarsely-powdered leaves and small stems (500 g.) of *D. cornigera* were moistened with water (200 ml.) and stored overnight. Calcium hydroxide (30 g.) was stirred in and the mixture macerated with solvent ether (about 500 ml.) for 3 hr. The supernatant ether was drained off and the remaining alkaloids extracted by percolation with more ether (about 6 litres). Evaporation of the combined ether extracts to about 100 ml. caused deposition of solid material which was removed by filtration through paper. The final concentrate was passed through a column of purified kieselguhr (60 g.) supporting 5N sulphuric acid (30 ml.), ether (460 ml.) being used to elute most of the pigments and ammoniacal chloroform (2 litres) to collect the alkaloids. Removal of the chloroform afforded 1·9 g. of a greenish-brown gummy residue which was then fractionated by partition chromatography.

In a typical small-scale experiment the basic residue (0·12 g.) was treated with ether (2 ml.) and a few drops of chloroform to effect solution and poured on to a column of kieselguhr (20 g.) loaded with 0·5M phosphate buffer solution (10 ml.), pH 6·6. The chromatogram was developed successively with light petroleum (b.p. 60–80°), ether, and chloroform. The eluate was collected in 5 ml. fractions, each titrated with 0·005N sulphuric acid with bromocresol green as indicator and the separated alkaloids recovered as described previously (Evans and Wellendorf, 1959).

A small quantity of alkaloid having a high R_F value by paper chromatography* was recovered from the light petroleum eluate but no crystalline derivatives of it could be prepared. The titration curve obtained from the ether eluate showed a large peak followed by two smaller ones. The base corresponding to the first peak was identified as (−)-hyoscine by the preparation of hyoscine picrate m.p. 187–188°, undepressed on admixture with authentic (−)-hyoscine picrate and hyoscine aurichloride, m.p. and mixed m.p. with authentic material 204°. The oily base derived from the eluate corresponding to the second ether peak, when neutralised with dilute sulphuric and treated with sodium picrate, afforded prisms m.p. 229–230° undepressed on admixture with the alkaloid picrate m.p. 230°, of unknown constitution, previously derived from other species of *Datura* (Evans and Partridge, 1949; Evans and Wellendorf, 1959). No crystalline derivatives could be obtained from the eluate corresponding to the third ether peak. Titration of the fractions of chloroform eluate indicated at least four bases; no crystalline derivatives could be obtained for the first three of these but the last furnished a picrate, needles from aqueous ethanol, m.p. 226° undepressed on admixture with noratropine picrate, m.p. 227°.

TABLE I

DISTRIBUTION OF PRINCIPAL ALKALOIDS IN *Datura cornigera* HOOK.

	I*	II	III	IV	V	VI	VII
Leafy shoots	0·27			0·19		<0·01	0·06
Stems	0·23			0·13		<0·01	0·08
Flowers	0·96			0·57		0·12	0·24
Pericarp	0·50			0·24		0·13	0·11
Seeds—Bolivian sample, 8 years old	0·14			0·11			0·03
Seeds—Nottingham sample, viable	0·65			0·52			0·11
Roots—plants raised in the field from cuttings, 1 year old	0·95	0·03	0·05	0·12	0·48	0·09	0·17
Roots—plants 6 years old, raised under glass	0·24	0·01		0·06	0·06	0·07	0·04
Root-wood—plants 6 years old raised under glass	0·11	<0·01				0·05	0·05

* I, total alkaloids calculated as hyoscyamine; II, (−)-3α,6β-ditigloyloxytropane; III, 7-hydroxy-3,6 ditigloyloxytropane; IV, hyoscine; V, hyoscyamine and/or atropine; VI, norhyoscyamine and/or noratropine; VII, other alkaloids calculated as hyoscyamine. All per cent.

Samples (3 g.) of the ripe seeds produced in Nottingham and of the original seed-sample from Bolivia, 8 years old and no longer viable, were analysed by the method of Evans and Partridge (1952) with the modifications that light petroleum (b.p. 60–80°) replaced carbon tetrachloride and that the eluate was collected in 5 ml. fractions which were titrated individually. No bases were eluted with light petroleum. The ether eluate gave on the titration curve one very small peak which was followed by a main peak corresponding to hyoscine, m.p. and mixed m.p. of the picrate 186–187°. The chloroform contained very little alkaloid. A similar examination of the pericarp (5 g.) gave hyoscine as the main alkaloid, m.p. and mixed m.p. of the picrate 186–187° with two smaller peaks in the chloroform fraction, the second of which appeared to be noratropine,

* All paper chromatograms were prepared by the ascending method using light petroleum (b.p. 60–80°), amyl alcohol, glacial acetic acid and water (1 : 3 : 3 : 3) as the developing mixture.

m.p. of the picrate 224–225°, softening at 218° undepressed on admixture with noratropine picrate, m.p. 226–227°. The alkaloid mixture in the flowers (5 g.) was similar to that of the pericarp. The quantitative results for these determinations, together with similar ones for the leaves and stems are recorded in Table I.

Alkaloids of the Roots

Preliminary experiments involving partition chromatography of an ether extract of *D. cornigera* roots at pH 6·6 indicated the presence of at least 10 bases in the mixture. For a more detailed study of the individual alkaloids, powdered roots (600 g.) from plants one year old were moistened with water (300 ml.) and allowed to stand overnight. Calcium hydroxide (30 g.) was mixed in, the powder transferred to a percolator and the basic constituents extracted with ether (9 litres). Concentration of the percolate to about 200 ml. caused the deposition of some solid material; this was removed by filtration and the alkaloids collected in 0·1N sulphuric acid. The acidic solution was repeatedly shaken with chloroform and the separated chloroform extract (1·75 litres) basified with ammonia, washed with water and the solvent removed *in vacuo* leaving a light-brown residue (0·8 g.) designated "Fraction A". The yellow-brown, acid solution was made alkaline by the addition of a strong solution of ammonia and the liberated alkaloids collected in chloroform (2 litres). Removal of the chloroform under reduced pressure afforded a deep-brown syrupy residue (4·7 g.) designated "Fraction B". The marc remaining from the ether extraction was percolated with industrial methylated spirit (5 litres) and the solvent removed *in vacuo* from the extract leaving a brown, semi-solid residue (47·7 g.)—"Fraction C". Paper chromatography of these fractions showed A to contain mainly bases of high R_F values, B intermediate R_F values and C, low R_F values.

Fraction A was dissolved in chloroform (3 ml.) and submitted to partition chromatography on kieselguhr (30 g.) loaded with 0·5M phosphate buffer solution (15 ml.), pH 5·4; light petroleum (b.p. 60–80°), ether, and chloroform were used successively as developing solvents. Two bases were evident in the petroleum ether fraction. The first (initial 60 ml. eluate) was isolated as a colourless gummy mass (0·03 per cent) from the titration liquors and identified as (−)-3α,6β-ditigloyloxytropane by the preparation of the following derivatives: picrate, filamentous needles from aqueous ethanol, m.p. and mixed m.p. with authentic (−)-3α,6β-ditigloyloxytropane picrate, 151° (Found: C, 52·4; H, 5·3. Calc. for $C_{18}H_{27}NO_4,C_6H_3N_3O_7$: C, 52·4; H, 5·5 per cent); chloroplatinate, orange rosettes from dilute hydrochloric acid, m.p. 230° (decomp.). The addition of a saturated solution of ammonium reineckate to a neutral solution of the base afforded a *reineckate*, micro-rosettes from 20 per cent aqueous acetone, m.p. 172–173° after sintering at 167–168° (Found: C, 40·3; H, 5·1. $C_{18}H_{27}NO_4$, $H[Cr(SCN)_4(NH_3)_2]$, H_2O requires C, 40·1; H, 5·2 per cent). The second base, contained in the subsequent 335 ml. light petroleum, was shown to be 7-hydroxy-3,6-ditigloyloxytropane (0·02 per cent) by the following derivatives: picrate, plates from aqueous

ethanol, m.p. and mixed m.p. with authentic material 182–183° (Found: C, 51·2; H, 5·42. Calc. for $C_{18}H_{27}NO_5$, $C_6H_3N_3O_7$: C, 50·9; H, 5·3 per cent); chloroplatinate, orange prisms from 0·1N hydrochloric acid m.p. 251–252° (decomp.). A neutral solution of the base in water, afforded on the addition of a saturated solution of ammonium reineckate, a *reineckate*, micro-rosettes from aqueous acetone, m.p. 194–195° (decomp.) after sintering at 189–190° (Found: C, 40·2; H, 5·23. $C_{18}H_{27}NO_5$, $H[Cr(SCN)_4(NH_3)_2]$ requires C, 40·2; H, 5·06 per cent). The ether eluate showed two small peaks on the titration curve; no crystalline derivatives could be obtained from the eluate corresponding to the first peak but the second (0·004 per cent) afforded a picrate, plates from aqueous ethanol, m.p. 171–172° (Found: C, 55·3; H, 4·9 per cent). The chloroplatinate, m.p. 202–203° (decomp.), formed nodules from dilute hydrochloric acid and the base gave a positive Vitali-Morin reaction and had an R_F value intermediate between that of hyoscyamine and the ditigloyl esters. Insufficient material prevented further work on this alkaloid. The chloroform eluate contained only a small amount of basic material of which no crystalline derivatives could be prepared.

Fraction B was dissolved in ether (about 3 ml.) and submitted to partition chromatography using a column prepared from kieselguhr (60 g.) on which was distributed 45 ml. of 0·5M phosphate buffer solution, pH 6·6. The alkaloid mixtures derived from the light petroleum, ether and chloroform eluates were designated B1, B2 and B3 respectively. Ammoniacal chloroform was used to remove any residual alkaloids from the column—B4. B1 contained a small quantity of basic material with a high R_F value and probably represented residual Fraction A bases. B2 contained a mixture of the principal alkaloids, the separation of which was incomplete. Consequently it was divided into Subfraction B2a—the first 400 ml. of ether eluate and Subfraction B2b—the following 350 ml. eluate. The alkaloid mixture obtained from B2a was resubmitted to partition chromatography at pH 6·6. The first portion of ether eluate contained (−)-hyoscine which was characterised by the picrate, m.p. 184–185°, undepressed on admixture with authentic (−)-hyoscine picrate m.p. 188° and the reineckate m.p. 170–171° (decomp.) after sintering at 169–170°. The subsequent ether eluate (about 250 ml.), represented by a low hump on the titration curve, appeared to contain a single alkaloid having an R_F value similar to that of 3α-tigloyloxytropane (Evans and Wellendorf, 1959). However, no crystalline picrate could be isolated from the fraction and the reineckate, stout needles from aqueous acetone, m.p. 144–145° (decomp.), sintering at 130°, did not appear identical with the reineckate prepared from authentic 3α-tigloyloxytropane which had m.p. 159–160° (decomp.), sintering at 151–152°. From the chloroform eluate, atropine was characterised as the picrate, m.p. after several recrystallisations from aqueous ethanol 170–171°, mixed m.p. with authentic atropine picrate (m.p. 174°) 171–172°. The base derived from Subfraction B2b afforded in neutral solution with sodium picrate, hyoscyamine picrate m.p. and mixed m.p. 165° and, with ammonium reineckate solution, hyoscyamine reineckate m.p. 154–155° (decomp.)

after sintering at 148°. Fraction B3 was collected in three parts, B3a, represented by a small sharp peak on the elution titration curve, B3b the principal portion and B3c, the tail of the B3b peak. B3a on repeated chromatography at pH 6·6, gave three fractions, the largest of which afforded (—)-hyoscyamine which was characterised as the picrate. Paper chromatography indicated the presence of three bases in the Subfraction B3b but repeated chromatography at pH 6·8 and 6·4 did not resolve the mixture. The bases were therefore recovered from the eluates and submitted to counter-current extraction for which the immiscible phases were prepared by shaking together light petroleum (b.p. 60–80°), n-amyl alcohol, glacial acetic acid and water in the ratio 1:3:3:3 and using the upper and lower layers as moving and stationary phases respectively. Fifteen tubes were used and the degree of fractionation of the alkaloids was followed by paper chromatography. The alkaloid acetates from tubes 7 and 8 were combined, the free bases recovered and neutralised with 0·005N sulphuric acid (14 ml.). The addition of sodium picrate solution to the neutral solution gave norhyoscyamine picrate, needles from dilute ethanol, m.p. and mixed m.p. with authentic norhyoscyamine picrate, 220°. (Found: C, 52·4; H, 5·05. $C_{16}H_{21}NO_3,C_6H_3N_3O_7$ requires C, 52·4; H, 4·76 per cent.) From tubes 4 and 5, which paper chromatography indicated to contain two alkaloids, one having a R_F value the same as cuscohygrine, it was possible to isolate only norhyoscyamine. Tubes 1 and 2 also contained the cuscohygrine-like alkaloid (4 mg.) but no crystalline derivatives of it could be prepared. The same base appeared to be the principal constituent of B3c and a neutralised aqueous solution of it readily gave a picrate, prisms from dilute ethanol, m.p. 214–215° (decomp.) after collapsing into a viscous mass at 201–202° (Found: C, 51·8; H, 5·23; N, 11·3. Calc. for cuscohygrine dipicrate, $C_{13}H_{24}N_2O,2C_6H_3N_3O_7$: C, 43·9; H, 4·4; N, 16·4 per cent). Lack of material prevented a more thorough examination of this base.

Fraction C, a dark brown semi-solid was treated with commercial absolute ethanol (50 ml.) and acetone (100 ml.). Insoluble matter, consisting of inorganic material and other impurities, was removed by filtration, the filtrate concentrated to 25 ml. and an equal volume of acetone: ether mixture (50:50) added causing the deposition of considerable oily material. Removal of the solvent from the decanted, clear supernatant liquid left a residue (1·9 g.), which by paper chromatography showed the presence of at least two bases of low R_F value. For the attempted characterisation of these, a portion of the residue (0·8 g.) was esterified with tigloyl chloride and the mixture of isolated esters submitted to partition chromatography (Evans and Wellendorf, 1959). The light petroleum eluate from the column contained (—)-3α,6β-ditigloyloxytropane (0·03 g.) as shown by the following characters: picrate, filamentous needles from aqueous ethanol, m.p. and mixed m.p. 151° (Found: C, 52·5; H, 5·49 per cent), mixed m.p. with (+)-3α,6β-ditigloyloxytropane picrate (m.p. 152°) 173–174° after softening at 156° (Evans and Wellendorf, 1958); chloroplatinate, plates from dilute hydrochloric acid m.p. 230–231° (decomp.). Attempts to isolate other tigloyl esters (0·01 g.)

were unsuccessful although paper chromatography indicated their presence. The proportions of the principal alkaloids in the roots were determined by a slight modification of the method of Evans and Wellendorf (1959) and are given in Table I, together with similar figures for the roots of older plants.

Alkaloids of the Root-Wood

Paper chromatography indicated the distribution of alkaloids in the root-wood to differ from that of the whole root. The alkaloids from coarsely powdered root-wood (200 g.) of six-year old plants grown in a temperate greenhouse were extracted as previously described for the whole roots and collected in 0·05N sulphuric acid. Excess ammonia solution was added and the alkaloids extracted with chloroform; removal of the solvent gave a residue (0·75 g.) which was dissolved in ether (3 ml.) and submitted to partition chromatography at pH 6·6 using light petroleum, ether and chloroform in succession as eluants. A small petroleum ether fraction afforded (−)-3α,6β-ditigloyloxytropane (0·002 per cent), picrate filamentous needles, m.p. and mixed m.p. with authentic material 151–152°. No crystalline derivatives could be obtained from the ether eluate but the principal constituent of the chloroform was shown to be noratropine (0·05 per cent); picrate, stout needles from aqueous ethanol m.p. 226–227° (Found: C, 52·4; H, 4·8 per cent); aurichloride, m.p. 162–164°. The high m.p. of the aurichloride compared with that of authentic material (157°) could be ascribed to the presence of some unracemised norhyoscyamine, a possibility supported by the slight laevorotation of a solution of the base in ethanol. The quantitative analytical figures for the root-wood are given in Table I.

Discussion

The occurrence of hyoscine as the principal alkaloid of the leaves, stems and flowers of *D. cornigera* is consistent with other observations (*loc. cit.*) on closely related white-flowered tree daturas. Although we have been unable to confirm the presence of hyoscyamine or atropine in the leaves, their occurrence in small amounts cannot be excluded, as unresolvable mixtures, having R_F values on paper the same as hyoscyamine, have been obtained. An uncharacterised alkaloid, affording a picrate m.p. 230° and previously isolated from the aerial parts of *D. ferox*, Indian henbane and the roots of *D. innoxia* occurs in the leaves together with a small quantity of noratropine. The latter alkaloid is also reported, for the first time, in the flowers and pericarp of a *Datura* species. The isolation of hyoscine as the main alkaloid of *D. cornigera* seeds produced both in this country and in Bolivia supports the view that the alkaloid mixture of the seeds is a reflection of the alkaloid mixture in the aerial parts of the plant at the time of seed formation. Schmidt and Kircher (1906) suggested that their unexpected finding of a 4:1 ratio of hyoscyamine to hyoscine in the seeds from foreign plants of *D. arborea* might be explained by differences in climatic conditions. Subsequent investigations on *D.*

arborea grown in S. America (Montesinos, 1939; Villalba, Medina and Albarracin, 1945) have not yet indicated specimens having hyoscyamine as the principal alkaloid of the aerial parts.

Concerning the nature of the alkaloids of the roots, *D. cornigera* shows resemblances to those members of the Sections Dutra and Stramonium already studied in some detail (Evans and Partridge, 1957; Evans and Wellendorf, 1958, 1959). 7-Hydroxy-3,6-ditigloyloxytropane and (−)-3α,6β-ditigloyloxytropane appear to be confined to the roots. Irrespective of whether the aerial parts of the species contain either hyoscine or hyoscyamine as the major alkaloid, hyoscyamine or atropine usually appear to be the principal constituents of the roots with the exception of two species so far examined in which meteloidine predominates. Recently it has been shown that with a number of species, hyoscyamine produced in the roots, may be converted in the leaves to hyoscine via 6-hydroxyhyoscyamine (Romeike, 1959, 1960; Romeike and Fodor, 1960). A similar mechanism would explain in part the alkaloid pattern of *D. cornigera*. In our observations, the total alkaloidal content of the leafy aerial parts varied very little with age of plant but with the roots, those from 6 year old plants contained less alkaloid on a per cent dry weight basis than those from young plants. No 7-hydroxy-3,6-ditigloyloxytropane could be detected in the older roots but they contained a relatively high proportion of noratropine, which in the root-wood was the principal single alkaloid. The isolation of 3,6-dihydroxytropane gives another example of the presence of free alkamines in the roots of *Datura* species and the complexity of the root alkaloid mixture is further shown by the indication of other alkamines and uncharacterised bases.

A new derivative, suitable for the characterisation of the ditigloyl esters of the root is the reineckate.

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