

Cactaceae Alkaloids. I.

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The family *Cactaceae* comprises some 3000 species of cacti, frequently growing in the arid regions of North, Central and South America and the West Indies (4, 5). Open hybridization in cacti has induced large morphological variability which often has caused the same species to acquire different names. This in turn has created further taxonomical complications. The number of synonyms for a single species is often exasperating, e.g., *Lophocereus schottii* (Eng.) Br. & R., one of the large "organ pipe" cacti, has eight synonyms (22). The well known peyote cactus, *Lophophora williamsii* (Lem. ex SD.) Coult. (T.) is also recorded under at least three other names (27). The classification suggested by Backeberg (4, 5) will be followed in this paper.

In spite of the remarkable interest in peyote, whose hallucinogenic effects were described as early as in 1560 by the missionary Bernardino de Sahagun (cf., 27), the family *Cactaceae* has not been extensively investigated phytochemically.

One of the most accurate and critical reviews of the cactus alkaloids seems to be the survey by Reti in 1950 (27), listing only nine species containing alkaloids of known structure. Since then only a few new structurally clarified alkaloids and species containing previously known cactus alkaloids have been recorded. Some more important later contributions are summarized below.

Djerassi and co-workers have isolated pilocereine (12), first believed to be a "dimer" but then shown to be a "trimeric" alkaloid (11) from *Lophocereus schottii* (Eng.) Br. & R.,¹ *Lophocereus gatesii* M. E. Jon. and *Marginatocereus marginatus* (DC.) Backbg. A "monomeric" alkaloid lophocerine has also been isolated from *L. schottii* (13). Anhalonidine is known from *Pachycereus weberi* (Coult.) Backbg. (14) and mescaline from *Trichocereus pachanoi* (26). Recently Hodgkins *et al.* (16) isolated macromerine from *Lepidocoryphantha macromeris* (Eng.) Backbg. (T.) and the same alkaloid is also present in *Lepidocoryphantha runyonii* (Br. & R.) Backbg. (6) (synonyms: *Coryphantha macromeris* (Eng.) Lem. Br. & R. and *C. runyonii* Br. & R.). The structure proposed by Hodgkins *et al.*, (16) for gigantine isolated from *Carnegiea gigantea* (Eng.) Br. & R. (T.) was apparently not correct (10, 15). Dopamine, however, is known to occur in this species (32). Three new phenylethylamines, 3-methoxytyramine, 3,5-dimethoxy-4-hydroxyphenylethylamine and 3,4-dimethoxyphenylethylamine, were identified by us (1) together with mescaline in *Trichocereus pachanoi* Br. & R. and *Trichocereus werdermannianus* Backbg. In peyote, 3,4-dimethoxy-5-hydroxyphenylethylamine and 3,4-dimethoxyphenylethylamine are known to occur (2, 24). Kapadia and co-workers have recently isolated from peyote some quaternary alkaloids, *viz.* peyonine, anhalotine, lophotine and peyotine (19, 20), all closely related to mescaline and the tetrahydroisoquinolines. These workers have further isolated the alkaloid peyophorine from peyote (17) and, after the preparation of this manuscript, published (18) on the occurrence of no less than fourteen amides from peyote, all closely related to the known alkaloids and using a similar technique as suggested here.

Other surveys have been published, e.g., chapters on cactus alkaloids in Boitard's monograph (9) and in *The Alkaloids* (28) and an index of species containing alkaloids by Willaman (36). See also Reti's review for other references.

¹Pilocerine was also isolated from *L. australis* which now is considered as a variety of *L. schottii* (22).

The structures of the closely related to cactus alkaloids in several species obtained suggested that About 120 species of cacti in our laboratory and the

- (i). The presence of *Cereus, Echinops*
- (ii). The identity of t

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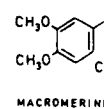
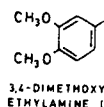
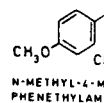


FIG. 1. Structures of known cacti alkaloids in subsequent sections. Quaternary or neutral is also known as 3-met

For our purpose it was known compounds were easily observed and specificity of mass spectra pointed out by Thomas choice.

The structures of the known cactus alkaloids are shown in fig. 1. Compounds closely related to cactus alkaloids and which may be expected to occur in cacti are presented in fig. 2. In connection with our biogenetic studies, a screening for alkaloids in several species of *Trichocereus* was carried out (1). The results obtained suggested that the occurrence of alkaloids in *Cactaceae* was wide spread. About 120 species of cacti have been tested for basic, non-quarternary alkaloids in our laboratory and the following data is presented in this paper:

- (i). The presence or absence of alkaloids in some cacti mainly belonging to *Cereus*, *Echinopsis*, *Helianthocereus* and *Trichocereus*.
- (ii). The identity of the alkaloids present in the species.

Our major object in this investigation was to identify new alkaloids particularly the ones of biosynthetic interest. Furthermore, this screening may present valuable chemotaxonomic criteria that may be of significance in the classification of such a taxonomically complex group.

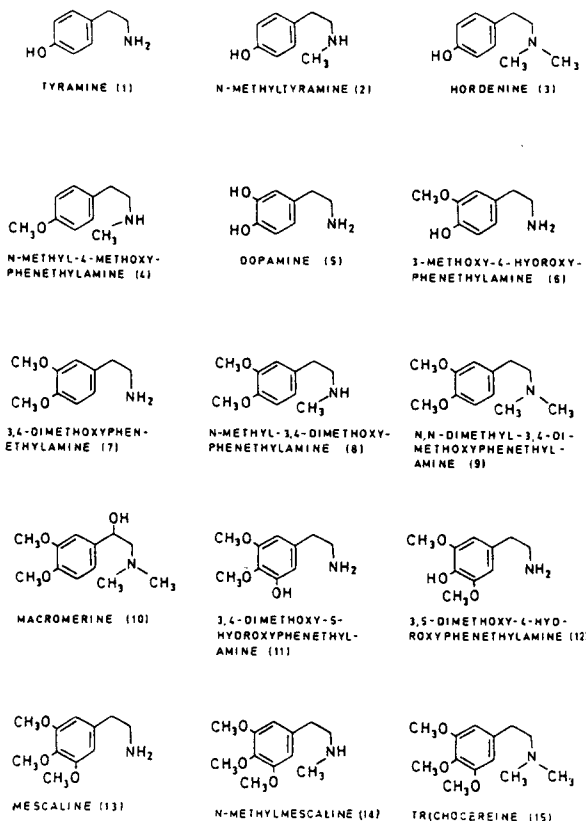


FIG. 1. Structures of known cactus alkaloids. Compounds 4 and 9 to be reported as isolated from cacti in subsequent papers in this series. Norcarnegine also known as salsolidine. Quarternary or neutral alkaloids e.g. N-acetylmescaline not included. Compound 6 is also known as 3-methoxytyramine and compound 16 as salsolidine.

For our purpose it was necessary to use a simple technique by which previously known compounds were readily recognized and identified and new interesting compounds were easily observed and characterized. Due to the great sensitivity and specificity of mass spectrometry combined with gas chromatography (GLC), as pointed out by Thomas and Biemann (34), it was adopted as the method of choice.

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During the preparation of this manuscript, Brown *et al.*, (10) reported on the occurrence of alkaloids in twelve of sixteen investigated cactus species. Their qualitative test was based on an alkaloid precipitating reagent and the number of alkaloids, which were not further identified, was estimated by gas chromatography.

This investigation has been carried out with commercially greenhouse grown cacti, which often differ morphologically from the plants growing under the conditions of the natural habitat. Furthermore it is not known whether these plants also differ in their ability to produce alkaloids. However, the green-house produced cacti so far tested (*Trichocereus pachanoi*, *T. terscheckii*, *T. lamprochlorus*, *T. candicans*, *Lophophora williamsii*, *Carnegie gigantea*, *Lophocereus schottii* and *Lepidocoryphantha runyonii*) agreed well in their alkaloid content with the data in the literature pertaining to wild species.

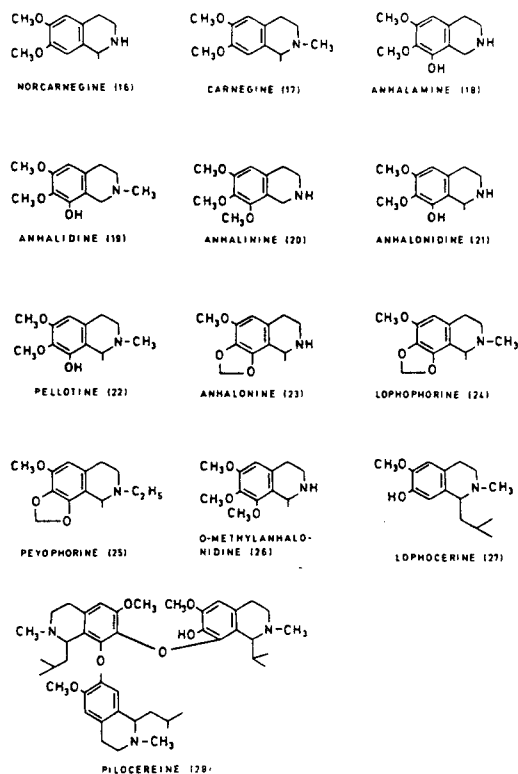


FIG. 1. *Continued.*

EXPERIMENTAL

Plant material.—Cacti used in this investigation are all commercially available and were purchased from K. Edelmann, Reeuwijk, The Netherlands; Walter Haage, Erfurt, DDR; or imported through the courtesy of B. Larsen, Kvarnby, Sweden.

The nomenclature of species as proposed by Backeberg (4) is used throughout. Cacti were checked to confirm with the macromorphological descriptions given by Backeberg (4, 5). However, only occasionally flowering species were available and cacti containing interesting compounds were then obtained from at least two different sources and their alkaloid content compared.

Isolation of alkaloids.—About 100 g plant material was homogenized in 250 ml methanol. After standing, protected from air, overnight at 4°, the methanolic extract was evaporated to dryness at low temperature. The residue was dissolved in a mixture of 25 ml 0.1 N HCl and 25 ml chloroform. Three percent acetic acid was later substituted for HCl since a number of alka-

loid hydrochlorides (*e.g.*, 1) in chloroform. The chloroform was extracted with 5 ml chloroform, the chloroform was extracted with 2×50 ml chloroform, and finally, after adjusting to pH 10, the chloroform extract dried to complete dryness.

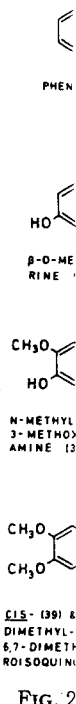


FIG. 2

The alkaloid extract was separated on a column of acid Celite (15 g) in chloroform. The column was saturated with conc. aq. ammonia and the extract was slowly passed through to form saturated with conc. aq. ammonia.

Separation of alkaloids was achieved by slowly passing (20 ml/h) a 10% aqueous methanol to yield 120 ml methanol, 60 ml water, and 60 ml benzene.

Column chromatography was carried out with benzene, benzene-chloroform, and chloroform-ethanol in different proportions.

Thin-layer chromatography.—Alkaloids were separated on silica gel G plates and developed with chloroform-ethanol-conc. ammonia. Phenols were located with 1% ferric chloride solution. Aqueous ammonia was used for the detection of basic compounds.

Gas chromatography.—The alkaloids were separated on a 1/8 in. (analytical) or 6 ft × 1/8 in. (AW-DMCS); 2% SE-52 column. The detector was a Varian model 20 (flame ionization detector); a

(10) reported on the cactus species. Their retention times and the number of peaks were determined by gas chromatography. The plants were greenhouse grown under the conditions whether these plants are the green-house product, *T. lamprochlorus*, *Phocereus schottii* and the content with the data

alkaloid hydrochlorides (e.g., pelletine, carnegine, dimethoxyphenylethylamines) are highly soluble in chloroform. The chloroform was discarded and the aqueous solution, after a further washing with 5 ml chloroform, was filtered and basified to pH 8 with ammonia. The aqueous phase was extracted with 2×50 ml chloroform followed by 50 ml of chloroform-ethanol (3:1) and then finally, after adjusting to pH 10-11 with another 2×50 ml of chloroform-ethanol (3:1). The chloroform extract dried over anhydrous sodium sulphate, was evaporated to dryness. Some simple amines are quite volatile and solutions of such compounds should not be evaporated to complete dryness.

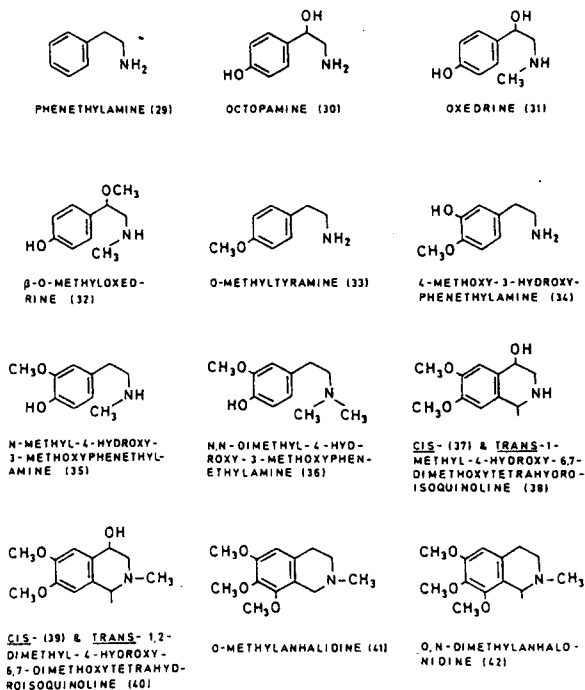


FIG. 2. Compounds related to known cactus alkaloids.

The alkaloid extract was dissolved in 100 ml chloroform and purified by passing through a column of acid Celite (15 g Celite 545 and 4 ml 0.5 M H_3PO_4). The column was washed with 100-200 ml chloroform to remove non-basic compounds. The alkaloids were eluted with chloroform saturated with conc. ammonia (23).

Separation of alkaloids into phenolic and non-phenolic compounds was accomplished by slowly passing (20 ml/h) a solution of the alkaloids in methanol over a column (1×20 cm) of Amberlite IRA-400 (OH^-) ion-exchange resin. The column was washed with 100 ml 30% aqueous methanol to yield non-phenolic compounds. Phenols were eluted with a solution of 120 ml methanol, 60 ml water and 20 ml glacial acetic acid.

Column chromatography was carried out on alumina (activity II-III) by elution successively with benzene, benzene-chloroform, chloroform, chloroform-methanol, methanol and methanol-water in different proportions.

Thin-layer chromatography.—Methods are described earlier (23). Silica gel G plates were chromatographed with chloroform-ethanol-diethylamine (85:5:10) followed by iodoplatinate to locate alkaloids. Phenols were visualized with *o*-dianisidine spray after chromatography with chloroform-ethanol-conc. ammonia (85:15:0.4) as solvent. Some simple phenylethylamines were also separated on silica gel G with *n*-butanol-acetic acid-water (4:1:1) as solvent.

Gas chromatography.—This technique has been described earlier (24). Glass columns, 6 ft × $\frac{1}{8}$ in. (analytical) or 6 ft × $\frac{1}{4}$ in. (preparative work) packed with 5% SE-30 on Gas Chrom P (AW-DMCS); 2% SE-52 on Aeropak 30; or 5% XE-60 on Chromosorb W (AW-DMCS) were used with Varian model 202 (thermal conductivity detector; preparative work) and model 204 (flame ionization detector; analytical work) Aerographs. The retention times of reference com-

mercially available and were from Haage, Erfurt, DDR; or

used throughout. Cacti were from Backeberg (4, 5). How- ever, the alkaloid content compared. The extract was evaporated to dryness in 250 ml methanol. The extract was evaporated to dryness in 25 ml 0.1 N HCl and 25 ml HCl since a number of alka-

pounds are shown in fig. 3. Preparative isolation of alkaloids was accomplished by collection of eluted alkaloids in glass wool packed Teflon tubings (3×60 mm) (Aerograph 202). Gas chromatography of enamines of primary amines was carried out by dissolving the amines in acetone (15). In certain cases, temperature programming was used to facilitate the gas chromatographic separation of compounds.

Spectrometric methods.—Gas chromatography–mass spectrometry was carried out with an LKB 9000 instrument (1). NMR spectra were recorded with a Varian A-60 instrument in deuteriochloroform or deuteropyridine solution with tetramethylsilane as internal standard. IR spectra were determined in KBr using a Perkin-Elmer 237 IR spectrophotometer and UV spectra in ethanol with a Beckman DB instrument. Compounds separated by preparative GLC were collected on 25 mg of KBr in a 3×60 mm Teflon tubing at the exit of the chromatograph (Aerograph 202) and used for the determination of the IR spectra. Melting points were taken on a Kofler micro hot-stage.

Reference compounds.—These are largely described in earlier publications (1, 23). The mp (base or hydrochloride) of synthesized compounds described below agree with literature values and further, mass spectra of final synthetic products were in agreement with the structural features. Purifications were carried out by column or sometimes by preparative paper or gas chromatography.

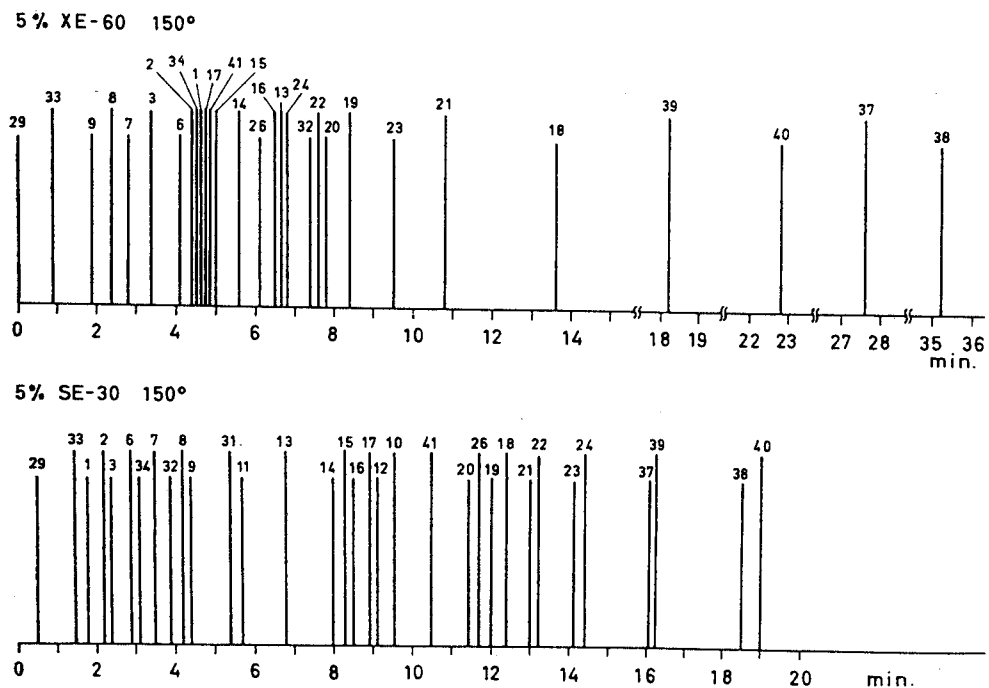


FIG. 3. Retention times of reference compounds. 5 ft× $\frac{1}{8}$ in. column of 5% SE-30 on Gas Chrom P, 150 or 5% XE-60 on Chromosorb W; 150. Compound no. as in fig. 1-2.

N-Methyl-3,4-dimethoxyphenylethylamine (hydrochloride, mp 136-137°) was prepared analogously to N-methyl-4-methoxyphenylethylamine (21).

Trichocereine and N,N-dimethyl-3,4-dimethoxyphenylethylamine (hydrochloride, mp 194-196°) were prepared as described by Reti for the synthesis of trichocereine (29).

Carnegine was synthesized according to Spath (31, see also 7) except for the reduction of the quarternary amine which was carried out with sodium borohydride in methanol. Norcarnegine was prepared by reduction of the intermediate dihydroisoquinoline with sodium borohydride (hydrochloride, mp 198-199°).

β -O-Methylodoxrine (β -O-methylsynephrine) was prepared as stated by Stewart and Wheaton (33).

Rac. cis- and trans-1-Methyl-4-hydroxy-6,7-dimethoxy-1,2,3,4-tetrahydroisoquinoline and their N-methylated forms were kindly donated by Dr. A. Brossi, Hoffman-La Roche Inc. (15). Macromerine was provided by Dr. J. McLaughlin, University of Washington.

Lophocerine was synthesized. **Screening and identification** of the presence of alkaloids together with GLC on SE-comparison with reference TLC data, an aliquot of the Teflon tubing. Comparison of the chromatograph. The mass spectrometry and/or The criteria for identification fig. 4.

Primary information Th ch (2)

Confirmatory information Ga ph an IR Pr rat un ma m) H

FIG. 4. Scheme for identification within parentheses.

Screening and Identification of the alkaloid fraction. This fraction was the platinate for non-phenolic alkaloids was indicated into phenolic and non-phenolic column at 150 and 2 m. wt less than about chromatogram the am by comparison with k.

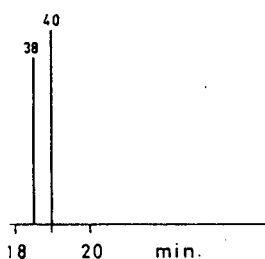
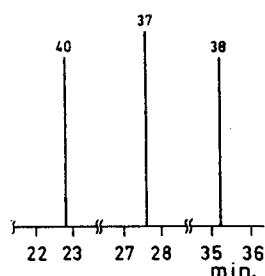
By comparison with constituents by GLC on SE-comparison with reference different separation procedure constituent was made chromatography–mass spectrometry.

Several major compounds by chromatography and additional means for structural identification indicated in fig. 4. The ring alkaloid was not identified from the mass spectrum.

accomplished by collection (Aerograph 202). Gas dissolving the amines in facilitate the gas chro-

was carried out with an A-60 instrument in an internal standard. IR spectrophotometer and UV separated by preparative the exit of the chroma- ra. Melting points were

publications (1, 23). The now agree with literature agreement with the structural preparative paper or gas



5% SE-30 on Gas Chrom no. as in fig. 1-2.

136-137°) was prepared

(hydrochloride, mp 194- reine (29).

cept for the reduction of n methanol. Norcarnegine with sodium borohydride

stated by Stewart and

etrahydroisoquinoline and ffman-La Roche Inc. (15). hington.

Lophocerine was synthesized essentially as described by Bobbitt and Chou (8).

Screening and identification techniques.—The methods used are outlined in fig. 4. Initially, the presence of alkaloids and phenolic alkaloids was ascertained by TLC. This information together with GLC on SE-30 at 150 and 240° usually allowed a preliminary identification by comparison with reference material. If there was an apparent discrepancy between GLC and TLC data, an aliquot of the extract was chromatographed at 260° on SE-30 and collected in a Teflon tubing. Comparison by TLC with the original extract showed if the alkaloids did pass the chromatograph. The preliminary identification was verified usually by gas chromatography-mass spectrometry and/or IR-spectrophotometry and/or by other means as shown in fig. 4. The criteria for identification is stated for each compound in table 1 using the abbreviations in fig. 4.

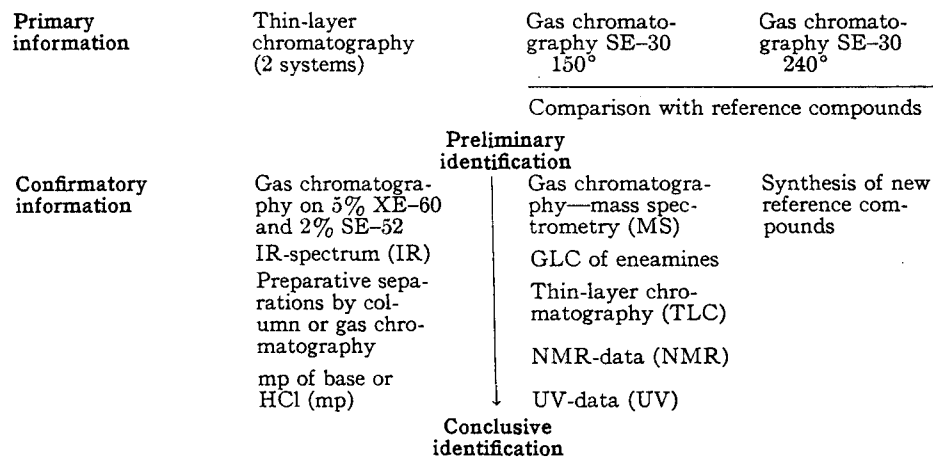


FIG. 4. Scheme for identification of alkaloids by comparison with reference material. Abbreviation within parenthesis are those used in table 1.

RESULTS AND DISCUSSION

Screening and Identification.—The basic technique consists of an initial extraction of the alkaloid fraction followed by purification on an acid Celite column. This fraction was then tested for alkaloids by TLC using spray reagents (iodoplatinate for non-phenolic; *o*-dianisidine for phenolic alkaloids). If the presence of alkaloids was indicated by TLC, the alkaloid extract, if necessary separated into phenolic and non-phenolic alkaloids, was subjected to GLC on an SE-30 column at 150 and 240°. This latter temperature will allow compounds with m. wt less than about 400 to pass through the chromatograph. From the gas chromatogram the amount of alkaloids, in mg/100 g fresh cactus, was determined by comparison with known amounts of mescaline.

By comparison with reference materials, a preliminary identification of constituents by GLC on SE-30 was obtained. This identification was complemented by comparison with reference materials also on an XE-60 column which has quite different separation properties (fig. 3). Finally, a positive identification of each constituent was made by comparison with reference materials using gas chromatography-mass spectrometry and/or IR spectrophotometry (fig. 4).

Several major components were readily isolated after preparative column chromatography and identified usually as hydrochlorides (mp). Other conventional means for structure elucidation used occasionally in the present studies are indicated in fig. 4. If a reference compound corresponding to the naturally occurring alkaloid was not available, an indication of the structure was usually obtained from the mass spectrum. Reference compounds were then synthesized accordingly.

Thus, the criteria in table 1 for identity of a compound is based on comparison with authentic reference material *e.g.*, identical mass or NMR spectra, together with the GLC data.

Fresh cacti were always used and during the work-up considerable precaution was taken to prevent the destruction of phenolic alkaloids. The extraction procedure also assured a satisfactory recovery of these chemically unstable but biosynthetically interesting entities. Cactus species were generally considered to contain no alkaloids if they contained less than 0.5 mg alkaloids/100 g plant. Plants containing more than 0.5 mg alkaloids/100 g, fresh weight, were considered to be alkaloid containing. Some 40% of the 120 screened species contained alkaloids. Of course, the percentage of cacti containing alkaloids would increase considerably by using a lower alkaloid content limit. Further results of the screening will be reported as the alkaloids are identified.

The occurrence of alkaloids in cacti appears not to be evenly distributed in the family; in some genera *e.g.*, *Coryphantha* (4 species tested), *Dolichothele* (5 species), *Echinocereus* (6 species), *Gymnocalycium* (4 species) and *Trichocereus* (12 species), the occurrence of several alkaloids in most of the tested species was common. Some of these genera will be further investigated in the future.

ALKALOID OCCURRENCE

The identified alkaloids, their approximate quantities, and relative abundance in the species investigated are reported in table 1. Pertinent references for the species in question and the criteria used to establish the identity of each alkaloid are also presented in the table.

Austrocylindropuntia.—*A. cylindrica* (Lam.) Backbg. (syn. *Opuntia cylindrica* (Lam.) SD.) was earlier apparently thought to be (26, 35) the botanical origin of a South American drug "San Pedro" which was used to prepare a hallucinogenic drink, "cimora" (30). The drug was found to contain mescaline (26, 35, 36) and consequently *A. cylindrica* is recorded as containing mescaline. But as pointed out by Poisson (26), "San Pedro" is not derived from *A. cylindrica* but most likely from *Trichocereus pachanoi* Br. & R. Also the cactus expert Backeberg (4) attributed "San Pedro" to *T. pachanoi* and on account of its effect suspected the presence of an alkaloid in the drug. *Opuntia cylindrica* tested now by us contained no alkaloids.

Cerus.—This genus is previously reported (36) to include species containing unknown alkaloids and caffeine (*C. jamacaru*). The species tested by us contained only tyramine and hordenine.

Helianthocereus.—A species (*H. huascha*) reported (36) to contain an unknown alkaloid yielded hordenine as did two other tested species.

Lepidocoryphantha.—This genus contains only two species (*L. macromesis*, *L. runyonii*) and both are known to contain macromerine (6, 16), which so far has not been encountered elsewhere in nature. Some of the trace alkaloids of *L. runyonii* were found to be: tyramine, hordenine and N-methyl-3,4-dimethoxyphenylethylamine. The latter compound was reported by us as a major component of *Echinocereus merkeri* Hildm. (3) and it is also present in *Ariocarpus trigonus* (Web.) K. Sch. (25).

Trichocereus.—Twelve species of this genus comprising in all 38 species have been investigated. Mescaline is earlier known to be present in *T. pachanoi* (26) and *T. terscheckii* (29). In a recent investigation, the biogenetic implications of which are reported elsewhere (1, 2), several other alkaloids including two new ones were reported to occur in *T. pachanoi* and *T. werdermannianus* (table 1). *T. macrogonus*, a species probably originating from Peru, and *T. bridgesii* from Bolivia contain the same alkaloids, including mescaline, as *T. werdermannianus*. It may be of interest to note that the mescaline producing cacti all have a stem and are branched or candelabra like. In contrast, the other investigated *Trichocereus*

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species, containing horizontal or columnar, creeping

Austrocylindropuntia	
<i>A. cylindrica</i> (Lam.)	
Backbg.	t
(syn <i>Opuntia cylindrica</i>	
Lam.)	t
Cerus	
<i>C. alacriportanus</i> Pfeiff.	t
<i>C. azureus</i> Parm.	t
<i>C. forbesii</i> O.	t
<i>C. glaucus</i> SD.	t
<i>C. peruvianus monstruosus</i>	
DC.	t
<i>C. peruvianus</i> (L.) Mill.	t
Cephalocereus	
<i>C. semilis</i> (Haw.) Pfeiff.	
(T.)	t
Echinopsis	
<i>E. eyriesii</i> G. (Turpin)	
Zucc.	t
<i>E. rhodotricha</i> K. Sch.	t
Helianthocereus	
<i>H. huascha</i> (Web.) Backbg.	t
<i>H. pasacana</i> (Web.)	
Backbg.	t
<i>H. poco</i> Backbg.	t
Lepidocoryphantha	
<i>L. runyonii</i> (Br. & R.)	
Backbg.	t
Trichocereus	
<i>T. bridgesii</i> (SD) Br. & R.	th
<i>T. camarguensis</i> Card.	th
<i>T. candicans</i> (Gill)	
Br. & R.	th
<i>T. chilensis</i> (Colla)	
Br. & R.	th
<i>T. lamprochlorus</i> (Lem.)	
Backbg.	th

species, containing hordenine or at most disubstituted phenylethylamines (table 1) are columnar, creeping or low.

TABLE 1. Occurrence of alkaloids.^a

	Lit. ref. ^b	Presence of alkaloids ^c	Percent of alk. fraction ^d	Alkaloids	Criteria ^e
Austrocylindropuntia					
<i>A. cylindrica</i> (Lam.)					
Backbg.....	35	—	—	(Mescaline) ^f	—
(syn <i>Opuntia cylindrica</i> Lam.).....	this paper	—	—	—	—
Cereus					
<i>C. alacriportanus</i> Pfeiff.....	this paper	+	4	hordenine	IR
<i>C. azureus</i> Parm.....	this paper	—	—	—	—
<i>C. forbesii</i> O.....	this paper	+++	4	tyramine	IR,NMR;MS
<i>C. glaucus</i> SD.....	this paper	+	3	tyramine	GLC
			1	hordenine	GLC
<i>C. peruvianus monstrosus</i> DC.....	this paper	++	3	tyramine	IR,MS
			1	unknown	—
<i>C. peruvianus</i> (L.) Mill.....	36	—	—	unknown	—
this paper		tr.	3	unknown tyramine	GLC
Cephalocereus					
<i>C. senilis</i> (Haw.) Pfeiff. (T.).....	this paper	—	—	—	—
Echinopsis					
<i>E. eyriesii</i> G. (Turpin) Zucc.....	36	—	—	unknown	—
this paper		+	3	hordenine	IR
<i>E. rhodotricha</i> K. Sch.....	this paper	—	—	—	—
Helianthocereus					
<i>H. huascha</i> (Web.) Backbg.....	36	—	—	unknown	—
this paper		++	4	hordenine	IR,mp
<i>H. pasacana</i> (Web.) Backbg.....	this paper	+	3	hordenine	MS
<i>H. poco</i> Backbg.....	this paper	+	3	hordenine	IR
Lepidocoryphantha					
<i>L. runyonii</i> (Br. & R.) Backbg.....	6	—	—	macromerine	—
this paper		+++	3	macromerine	MS,mp
			tr.	hordenine	MS
			tr.	tyramine	GLC
			tr.	N-methyl-3,4-dimethoxyphenylethylamine	MS
			tr.	unknowns	—
Trichocereus					
<i>T. bridgesii</i> (SD) Br. & R.....	this paper	+++	3	mescaline	MS,IR,mp
			1	3,4-dimethoxyphenylethylamine	MS
			1	3-methoxytyramine	MS,IR
			1	tyramine	MS
<i>T. camarguensis</i> Card.....	this paper	tr.	3	tyramine	MS
			1	3,4-dimethoxyphenylethylamine	MS
			tr.	3-methoxytyramine	MS
			tr.	N-methyltyramine	GLC
<i>T. candicans</i> (Gill) Br. & R.....	27	—	—	candicine	—
this paper		+++	3	hordenine	IR,mp
<i>T. chilensis</i> (Colla) Br. & R.....	this paper	—	—	—	—
<i>T. lamprochlorus</i> (Lem.) Backbg.....	27	—	—	hordenine	—
this paper		++	3	hordenine	IR

ased on comparison R spectra, together siderable precaution The extraction pro- y unstable but bio- rally considered to aloids/100 g plant. ight, were considered l species contained oids would increase ther results of the venly distributed in ed), *Dolichothele* (5 and *Trichocereus* (12 tested species was the future.

relative abundance t references for the tity of each alkaloid

Opuntia cylindrica e botanical origin of are a hallucinogenic scaline (26, 35, 36) mescaline. But as m *A. cylindrica* but us expert Backeberg i its effect suspected a tested now by us

e species containing sted by us contained

contain an unknown

s (*L. macromesis*, *L.* 6), which so far has race alkaloids of *L.* thyl-3,4-dimethoxy- us as a major com- resent in *Ariocarpus*

all 38 species have in *T. pachanoi* (26) netic implications of luding two new ones ianus (table 1). *T. bridgesii* from Bolivia mannianus. It may have a stem and are stigated *Trichocereus*

TABLE 1. —Continued.

	Lit. ref. ^b	Presence of alkaloids ^c	Percent of alk. fraction ^d	Alkaloids	Criteria ^e
<i>T. macrogonus</i> (SD.) Ricc...	this paper	++	3	mescaline	MS,IR
			1	3,4-dimethoxyphenylethylamine	MS
			1	3-methoxytyramine	MS
			1	tyramine	MS
<i>T. pachanoi</i> Br. & R.	26	—	—	mescaline	—
	1 and this paper	+++	3	mescaline	MS,IR,mp
			1	3,4-dimethoxyphenylethylamine	MS,IR
			tr.	hordenine	MS
			tr.	tyramine	MS
			1	3-methoxytyramine	MS,IR
			tr.	3,5-dimethoxy-4-hydroxyphenylethylamine	MS
			tr.	3,4-dimethoxy-5-hydroxyphenylethylamine	MS
			tr.	anhalonidine	MS
				several not identified alkaloids	—
<i>T. peruvianus</i> Br. & R.	10	—	—	tyramine	MS
	this paper	+	3	3-methoxytyramine	MS
			1	two unknown	—
<i>T. schickendantzii</i> (Web.) Br. & R.	this paper	+	3	hordenine	MS
			tr.	N-methyltyramine	GLC
<i>T. spachianus</i> (Lem.) Ricc.	27	—	—	candicine	—
	this paper	+	3	hordenine	IR
<i>T. terscheckii</i> (Parm.) Br. & R.	29	—	—	trichocereine	—
				mescaline	—
				mescaline	—
<i>T. werdermannianus</i> Backbg.	1	++	3	mescaline	MS,IR
			3	mescaline	MS,IR,mp
			1	3,4-dimethoxyphenylethylamine	MS,IR
			tr.	tyramine	MS
			1	3-methoxytyramine	MS,IR
			tr.	3,5-dimethoxy-4-hydroxyphenylethylamine	MS

^aQuarternary alkaloids or neutral compounds such as N-acetyl-mescaline not investigated in the present study.

^bTo limit the number of literature references, we refer largely to reviews for earlier work. For comparison earlier data is included on the occurrence of alkaloids.

^cPresence of alkaloids: +++=over 50 mg/100 g; ++=10-50 mg/100 g; +=1-10 mg/100 g; tr.=trace, less than 1 mg/100 g fresh plant.

^dPer cent of alkaloid fraction: 4=only alkaloid present; 3=over 50%; 2=10-50%; 1=1-10%; tr.=trace, less than 1% of alkaloid fraction. Estimated from gas chromatogram.

^eThe abbreviations used in table 1 are those in fig. 4. The remark "MS" in the column means that the mass spectrum of the isolated compound was identical with that of available reference material. "IR" means identical IR spectrum etc.

^f*Trichocereus pachanoi* was, as discussed, erroneously determined as *Austrocylindropuntia cylindrica* (26,35). Further Willaman's survey (36) contains another reference (*Gaz. Chim. Ital.* 86, 1305 (1956)) stating the presence of mescaline in *A. cylindrica*. This reference to the literature is not correct.

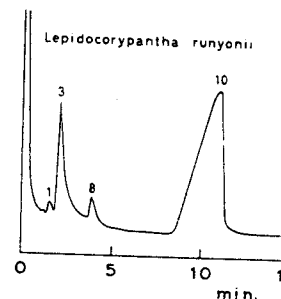


FIG. 5. Gas chromatogram SE-30; 150. Nur.

A rapid and sensitive method for the identification of cactus alkaloids by gas chromatography-mass spectrometry. Of 120 cactus species, 21 species of cactus alkaloids in 21 species of cactus, *Echinopsis* and *Trichocereus* and *T. macrogonus*. No alkaloids occur in nature for the first time. Introduction a short review from nature.

This investigation was supported by the Swedish Research Council, Drs. J. E. Lindberg and Dr. G. Gjerstad, Austin, Texas. The authors are indebted to Drs. G. Gjerstad, Austin, Texas, F. Sandberg, Stockholm, Sweden, and K. Olofsson and E. W. Bachmann, Uppsala, Sweden.

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	Criteria*
ethylamine	MS,IR MS MS MS
ethylamine	MS,IR.mp MS,IR MS MS MS,IR MS
ethylamine	MS
ethylamine	MS
alkaloids	MS MS
	MS GLC
	IR
	MS,IR
ethylamine	MS,IR.mp MS,IR MS MS,IR MS
ethylamine	MS

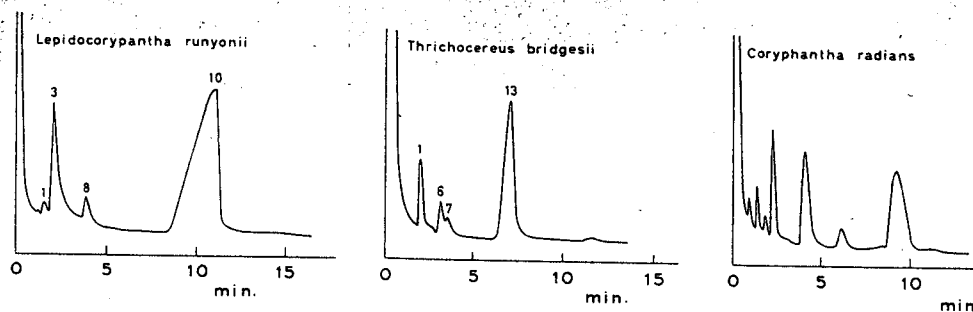


FIG. 5. Gas chromatogram of alkaloids extracts of *L. runyonii*, *T. bridgesii* and *C. radians* 5% SE-30; 150. Numbers refer to compound no. in fig. 1.

SUMMARY

A rapid and sensitive technique has been developed for the detection and identification of cactus alkaloid. The method is based on TLC, GLC and mass spectrometry. Of 120 cactus species tested about 40% contained alkaloids. The alkaloids in 21 species of cacti, mainly belonging to the genera *Cereus*, *Helianthocereus*, *Echinopsis* and *Trichocereus* are reported. Mescaline was isolated from *T. bridgesii* and *T. macrogonus*. N-methyl-3,4-dimethoxyphenylethylamine is reported to occur in nature for the first time (from *Lepidocoryphantha runyonii*). In the introduction a short review is given over the basic cactus alkaloids so far isolated from nature.

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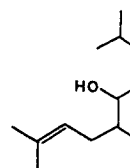
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