

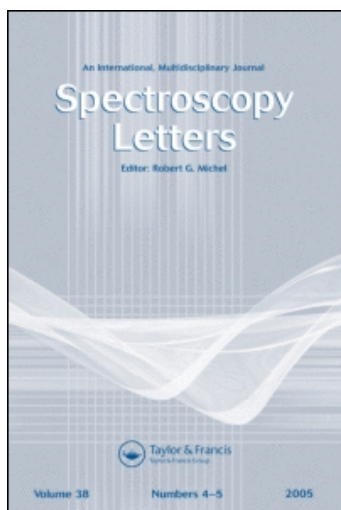
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GLC-Mass Spectroscopy of Distilled Alkaloids of Haloxylon Persicum¹

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GLC - MASS SPECTROSCOPY OF DISTILLED ALKALOIDS
OF HALOXYLON PERSICUM¹

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Key Word Index — Haloxylon Persicum; Chenopodiaceae; GLC -
Mass spectroscopy; Distilled alkaloids.

Abstract — GLC-Mass spectrometric study of the distilled alkaloids of Haloxylon persicum which grows wild in Saudi Arabia reveals the presence of cotinine as a minor alkaloid as well as anabasine which is a major alkaloid and nicotine which is also a minor alkaloid. The presence of both anabasine and nicotine has been previously established by other means. The occurrence of cotinine in the plant furnishes a supporting evidence for the presence of nicotine in Haloxylon persicum.

INTRODUCTION

Haloxylon Persicum Bunge. (Family Chenopodiaceae) grows wild in a specific saline area (near the Red sea coast)^a, and attains up

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1. An abstract of this research has been presented at the International Research Congress on Natural Products as Medicinal Agents, Strasbourg, France, July 6-11, 1980.

^a Voucher specimen is available in the herbarium of College of Pharmacy, Riyadh University.

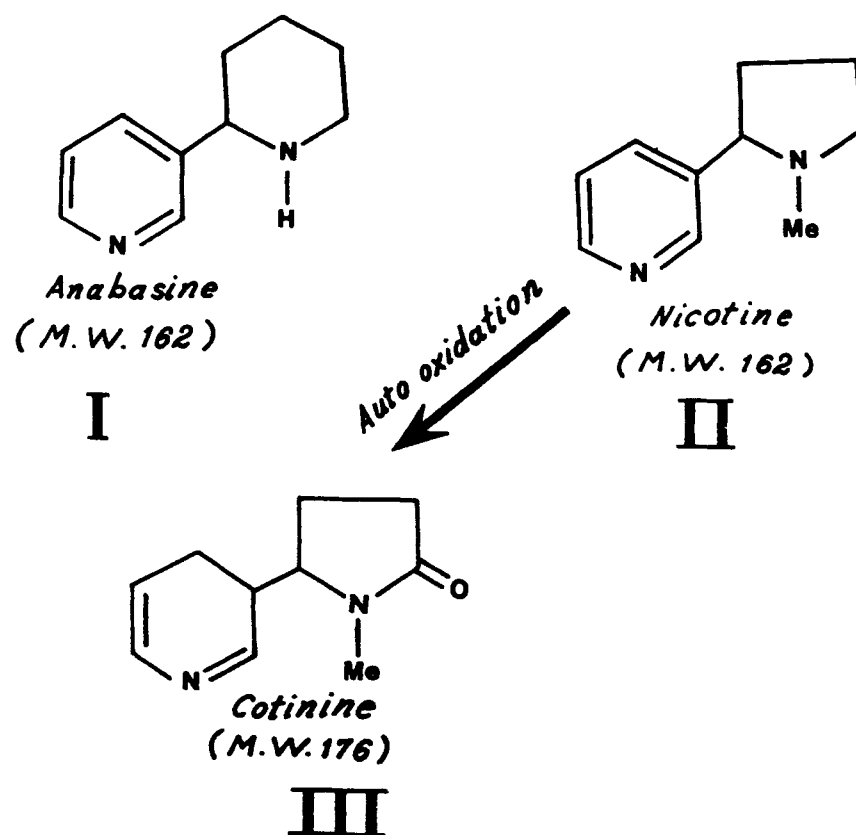
to 40 cm. long [1] caused death among grazing animals. For this reason we have investigated this plant for its chemical content. It was found to contain a high percentage of alkaloids (5.4%). Anabasine was isolated and found to be the major alkaloidal principle (constitutes almost 90% of the total alkaloids [2]).

In the present investigation we wish to confirm the presence of both anabasine and nicotine by GLC-Mass spectrometry. We also wish to report the identification of a third alkaloidal constituent by this technique.

RESULTS AND DISCUSSION

We have earlier established the presence of anabasine (by physical and chemical means) in *Haloxylon Persicum* in higher percentage than other commercial plant sources of anabasine. Nicotine was also identified by TLC analysis as a minor alkaloid. Its detection in *Haloxylon Persicum* furnished the first report of its occurrence within the family *Chenopodiaceae*. A third minor unidentified related alkaloid was also detected [2]. The present work was carried out to confirm the previously reported results as well as to identify the third alkaloid.

For this purpose the distilled total alkaloids have been subjected to GLC-Mass spectrometric analysis. Under the same conditions reference anabasine, nicotine and a mixture of both were also analysed. The GLC trace (Fig. 1) revealed a major component (retention time 3.59 min.) and a minor component (retention time 3.47 min.).



Scheme 1

It is to be noted that, although reference anabasine as well as reference nicotine gave slightly different retention times, yet a mixture of both (concentration dependant) gave the same retention time.

Under the area of the major component (retention time 3.59 min.), two mass fragmentation patterns were obtained both with M^+ 162 a.m.u. one of which was identical to that of reference anabasine I (Fig. 2) and the other was identical to reference nicotine II (Fig. 3) (3). The minor

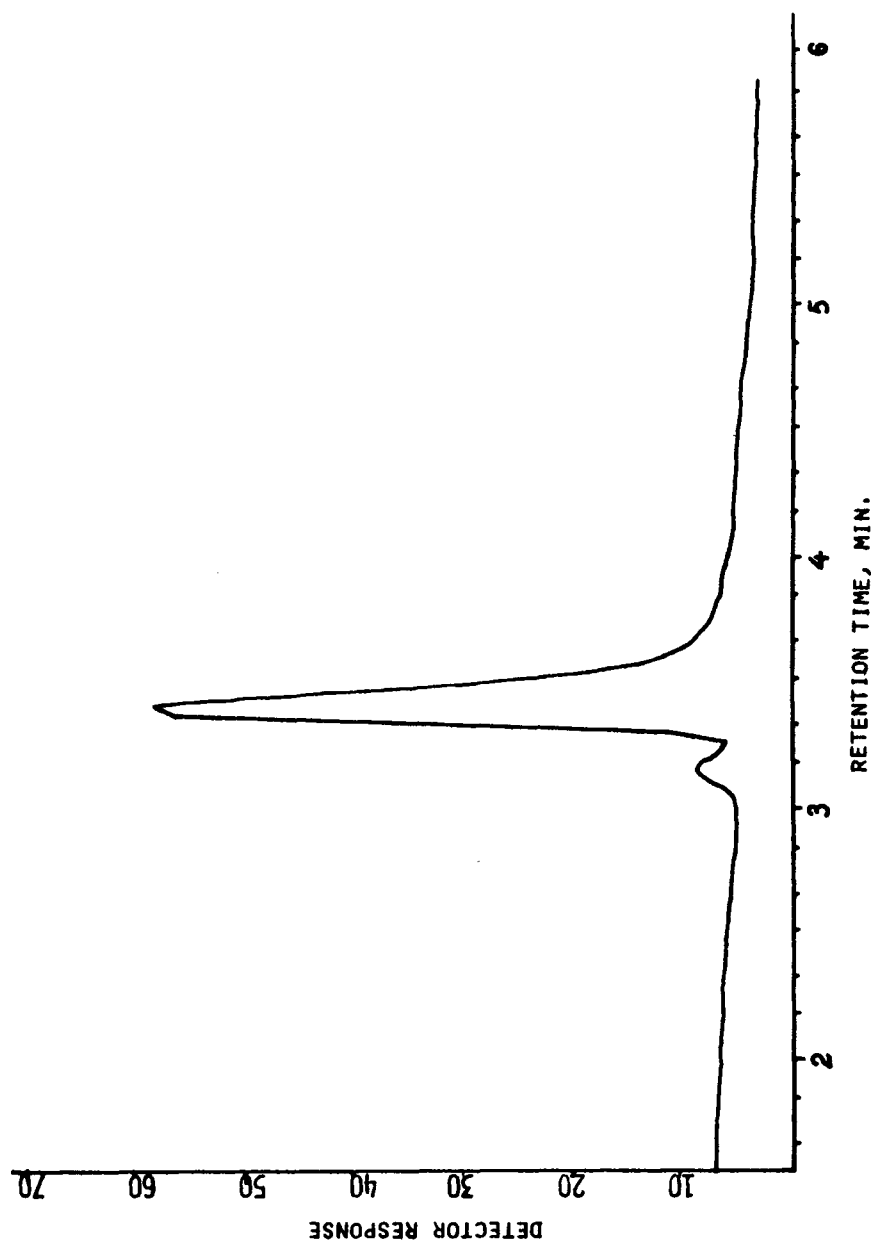


FIG. 1. GLC-MASS OF DISTILLED ALKALOIDS OF HALOXYLON PERSICUM.

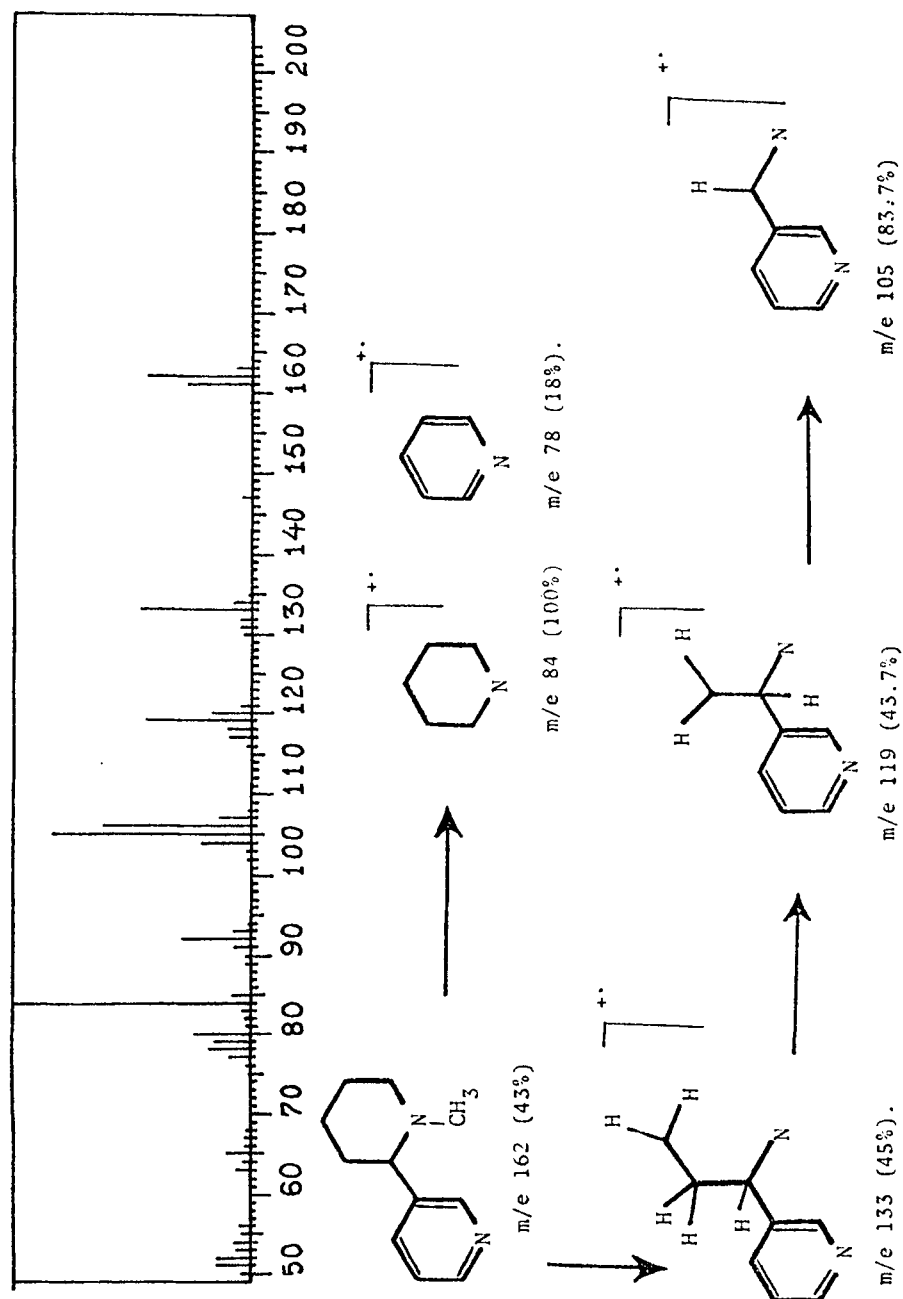


Fig. 2
Mass Spectrum of Anabasine

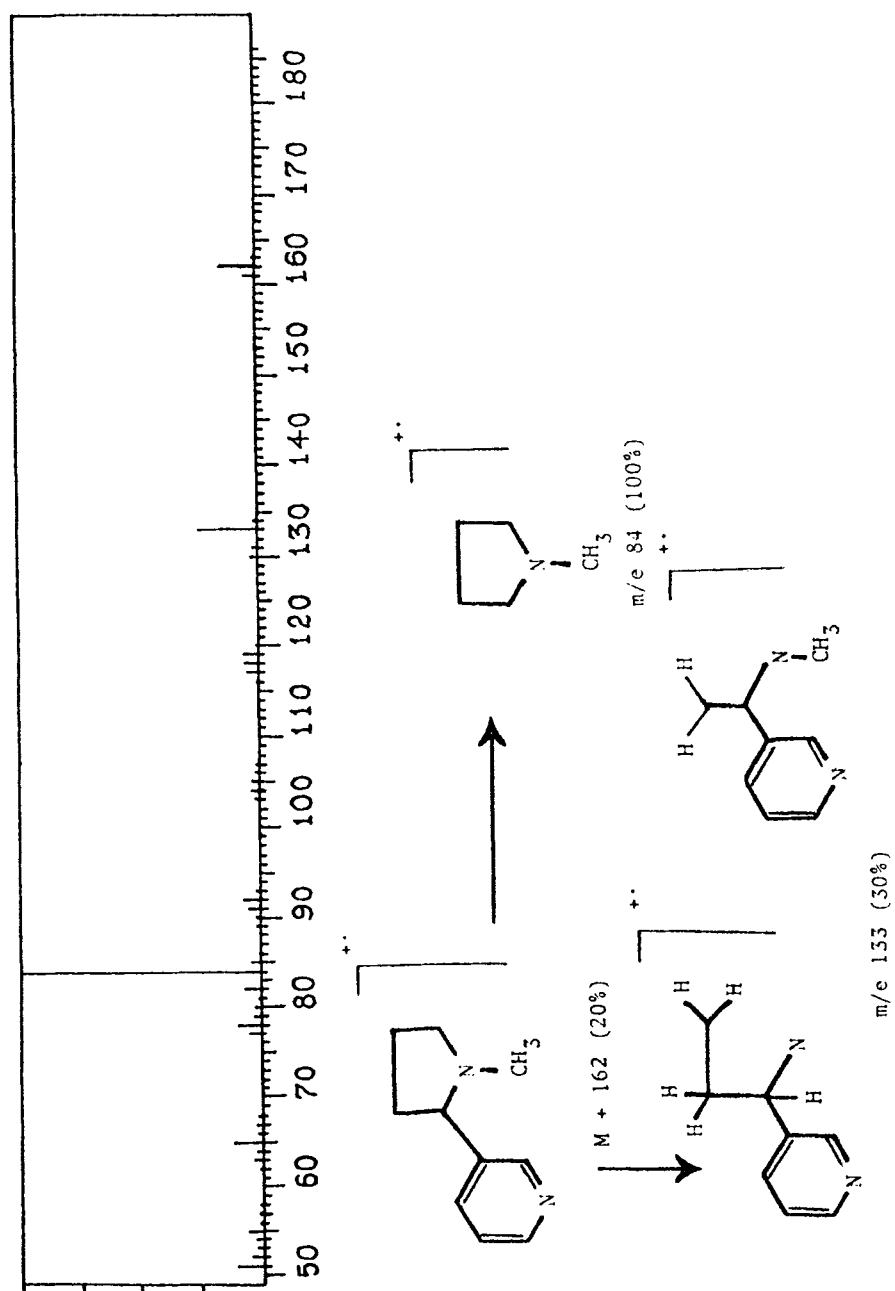


Fig. 3
Mass Spectrum of Nicotine

component (retention time 3.47 min.) showed a mass fragmentation pattern with M^+ 176 a.m.u. and this was attributed to continue III (Fig. 4). Cotinine which is an autooxidation product of nicotine (4), its presence is supporting evidence for the occurrence of nicotine.

EXPERIMENTAL

I) Extraction of the Alkaloids.

The dried powdered herb (1 Kg) was defatted with pet. ether (B.P. 60-80°). The defatted material was exhaustively extracted with alcohol (90%). The alcoholic extract was concentrated under vacuum, acidified and extracted with chloroform. The aqueous layer was basified with ammonia and the liberated bases were extracted with chloroform which was removed under vacuum. The resulting oily residue was fractionally distilled. The fraction boiling at 279-282° was collected and used for subsequent investigation.

II) GLC-Mass Spectrometry.

The distilled alkaloids were subjected to GLC-Mass spectrometric analysis using Ribermag GC/MS/DS Model R 10-10, Nermag, S.A. instrument. Reference Anabasine, Nicotine and a mixture of both were also run under the same conditions.

GLC Conditions:

3% methyl silicone polymer (SE-30) on chromosorb WHIP 100-180 mesh, packed on glass column of 2 m. long and 2 mm. diameter. The oven temperature which started from 150° was programmed at 10°/

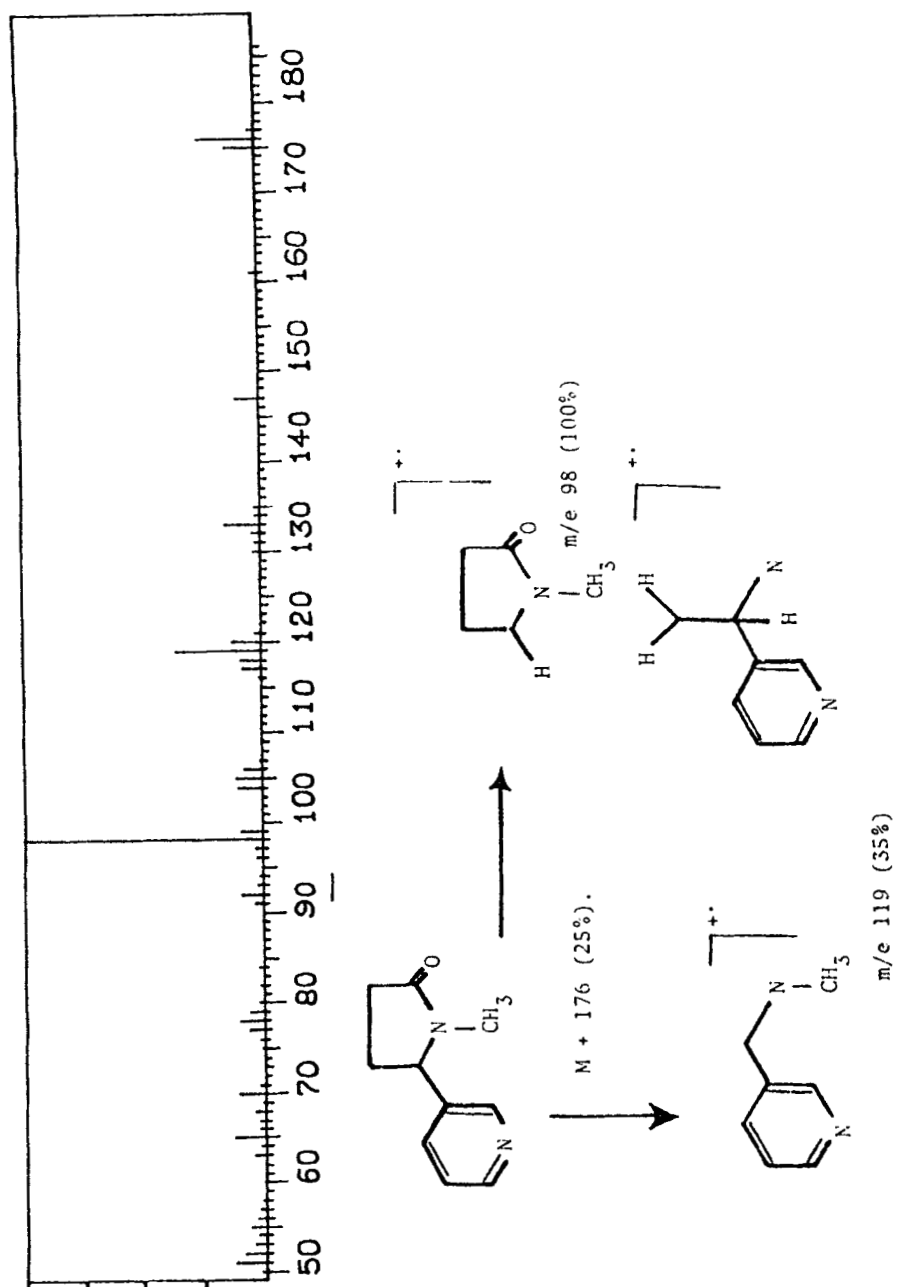


Fig. 4
Mass Spectrum of Cotinine

min. up to 240°. The injection port temperature was 240° and interface temperature 300°. Helium flow rate was adjusted to 20 c.c/min.

MS Conditions:

Mass spectra were obtained with electron impact mode (EI) with an electron energy 70 ev. The mass range was scanned from 50 to 450 a.m.u.

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