

SPARTEINE AND 13-HYDROXY SPARTEINE FROM
LEAVES OF SOPHORA SECUNDIFLORA (ORTEG.)LAG.

A.M. Abdel-Baky and M.A. Makboul
Department of Pharmacognosy, Faculty of Pharmacy, Assiut
University, Assiut, Egypt

ABSTRACT

The isolation of quinolizidine alkaloids, sparteine and 13-hydroxysparteine from leaves of Sophora secundiflora (Orteg.) Lag. Family Leguminosae cultivated in Egypt is reported.

INTRODUCTION

The first phytochemical investigation of Sophora secundiflora was published by Wood (1878) when the isolation of sophorine was reported¹. This compound was later shown to be identical to the quinolizidine alkaloid cytisine^{2,3}. Recently, Keller^{4,5} reported the isolation of rhombifoline, lupanine, N-methylcytisine and sparteine from the seeds of Sophora secundiflora. Izad-

doost⁶ detected two additional quinolizidine alkaloids, anagyrine and thermopsine, in the seeds of Sophora secundiflora.

Quinolizidine alkaloids are known to have widely different structural types. The cytisine and matrine-type alkaloids are characteristic components of the genus Sophora, while sparteine-type alkaloids are rare^{7,8,9}.

This paper describes the structure elucidation of two sparteine-type alkaloids isolated from leaves of Sophora secundiflora cultivated in Egypt.

EXPERIMENTAL

Material and Methods:

IR spectra were determined as KBr discs using a Perkin Elmer-267-grating infrared spectrophotometer. NMR spectra were run on a Hitachi-Perkin Elmer NMR spectrometer at 60 MHz using TMS as an internal standard and on a Varian EM-360 Brüker spectrometer at 200 MHz. Electron impact mass spectra were recorded on VG 12 F mass spectrometer at 70 ev. Melting points were determined on Thomas and Fisher Johns melting point apparatus. Plant material, was collected in May 1982 from Aswan Botanic Island.

Extraction and Fractionation:

The alkaloid fraction obtained from the air-dried leaves of Sophora secundiflora was subjected to alumina and silica gel column chromatography. Elution was started with chloroform and chloroform-methanol in increasing polarities. Preparative TLC was also used for separation of minor compounds (alkaloids I and II) using silica gel G plates and chloroform-methanol (90 : 10) as a solvent system. The method of extraction and isolation was described in a previous work by the authors¹⁰.

Perchloric acid salt of compound I:

Compound I obtained as a light yellow oily product (The base), was dissolved in methanol, the solution neutralized to congo red with 70% perchloric acid and diluted with ether to the point of incipient turbidity. On standing, the perchloric acid salt separated in colourless needles forming feathery aggregates, m.p. 171°C which was found consistent with that reported for the perchloric acid salt of d-sparteine¹¹.

Methiodide of Compound I:

The base was mixed with methyl iodide and left for half an hour, where crystalline methiodide salt was deposited as fine needles melting at 235-236°C (from alcohol-acetone), which was found in agreement with

that reported for sparteine methiodide¹¹.

Compound II:

Compound II crystallized as colourless prisms, m.p. 168-170°C (From pet. ether).

Acetylation of Compound II:

A solution of the base in chloroform was treated with acetic anhydride, the mixture was boiled under reflux for 4 hours and cooled. The resinous precipitate was passed through a column containing alumina and eluted with a mixture of chloroform-methanol (9:1). The crude product was purified by crystallisation from pet. ether (60-80°C) in the form of white needles melting at 137°C, which is in accordance with that reported for 13-hydroxysparteine acetate¹².

The picrate and methiodide salts of 13-hydroxysparteine acetate were also obtained and crystallised from acetone-ether and ethyl acetate, m.p. 224-226°C and 260-263°C respectively, which are consistent with published data of these derivatives¹².

RESULTS AND DISCUSSION

The IR spectrum (cm^{-1}) for compound I revealed the following bands: 2720, 2760, 2805 which are chara-

characteristic for the absorption of trans quinolizidines¹³⁻¹⁵.
 $C_{15}H_{26}N_2$: calculated, % C 76.9%; H 11.1% N 12.0% found C: 76.6%; H 11.2%; N 12.2%. This formula was further confirmed by determining the MS of compound I, which showed the M^+ ion at m/e value of 234. The other prominent fragments are shown in Fig. I A.

The PMR spectrum, run in $CDCl_3$, revealed the signals of the protons at the following chemical shift values: (δ) H_{17} at 2.50 ppm; H_{15} at 2.70 ppm, H_{10} at 2.42 and 1.90 ppm, H_9 at 1.65 ppm, H_8 at 0.90 and 2.02 ppm, H_7 at 1.30 ppm and H_2 at 2.45 ppm.

The aforementioned values are concordant with those reported for sparteine¹⁸⁻²¹.

The IR spectrum (cm^{-1}) for compound II revealed the following bands: 3620 (free hydroxyl group), 2720, 2760, 2805 (trans quinolizidine).

$C_{15}H_{26}N_2O$: calculated, C 72.0%, H 10.5% N 11.1%, found C: 72.2%, H 10.3, N 11.0%. This formula was further confirmed by determining the MS of compound II, which showed the M^+ ion at m/e value of 250. The other prominent fragments are shown in Fig. 1 B.

The 1H NMR (200 MHz, $CDCl_3$) spectrum of compound II is found similar to that of sparteine with the exception of the presence of a broad hydroxyl signal at 3.60 ppm. This signal is accordingly absent in the NMR

spectrum of the acetylated compound.

The above mentioned data demonstrate the identity of the isolated compounds I and II to the known sparteine and 13-hydroxysparteine alkaloids respectively.

This investigation describes the first report, on the isolation and identification of 13-hydroxysparteine from the leaves of Sophora secundiflora (Orteg.) Lag.

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Sparteine and 13-hydroxy sparteine from leaves of
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Fig. I A

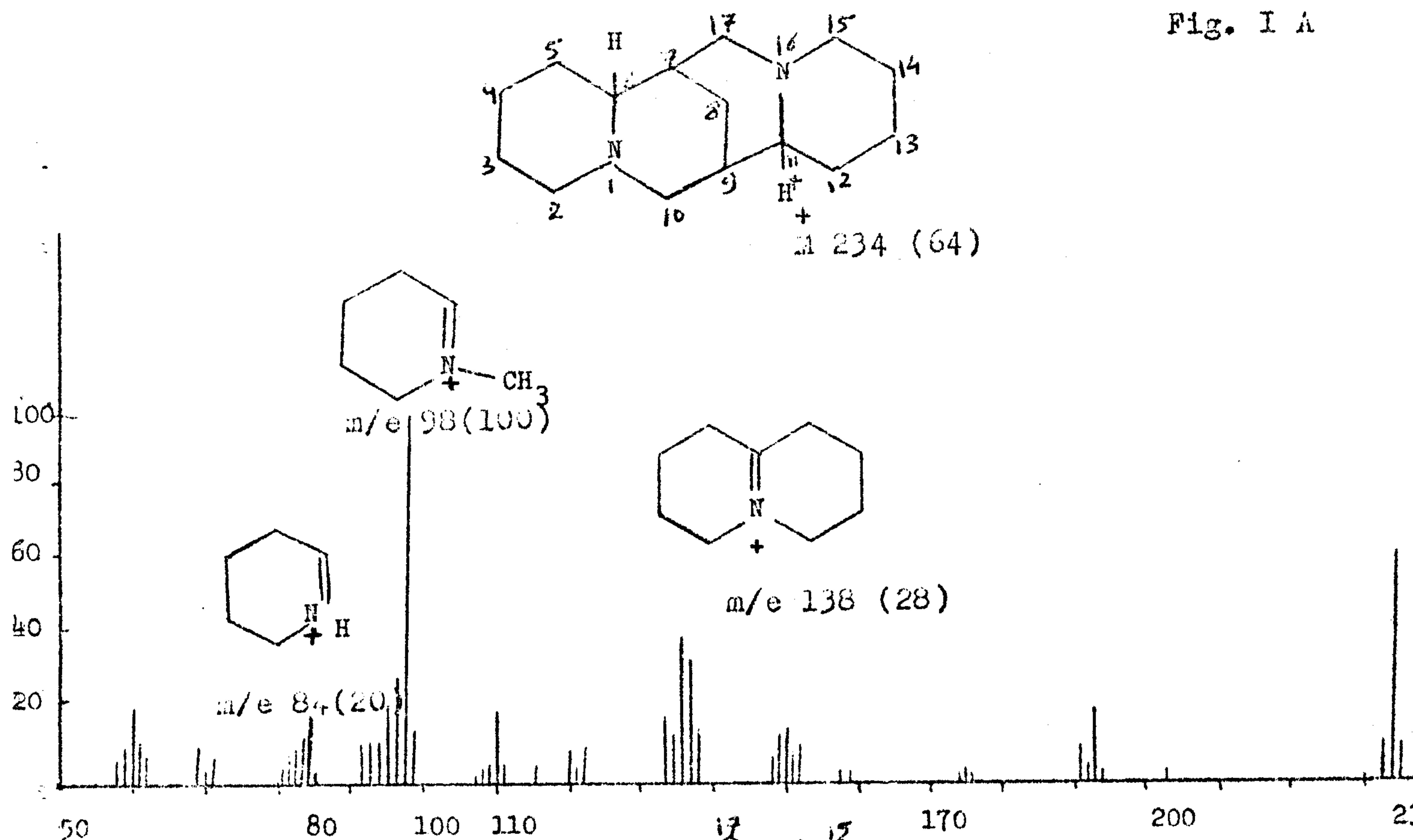


Fig. I B

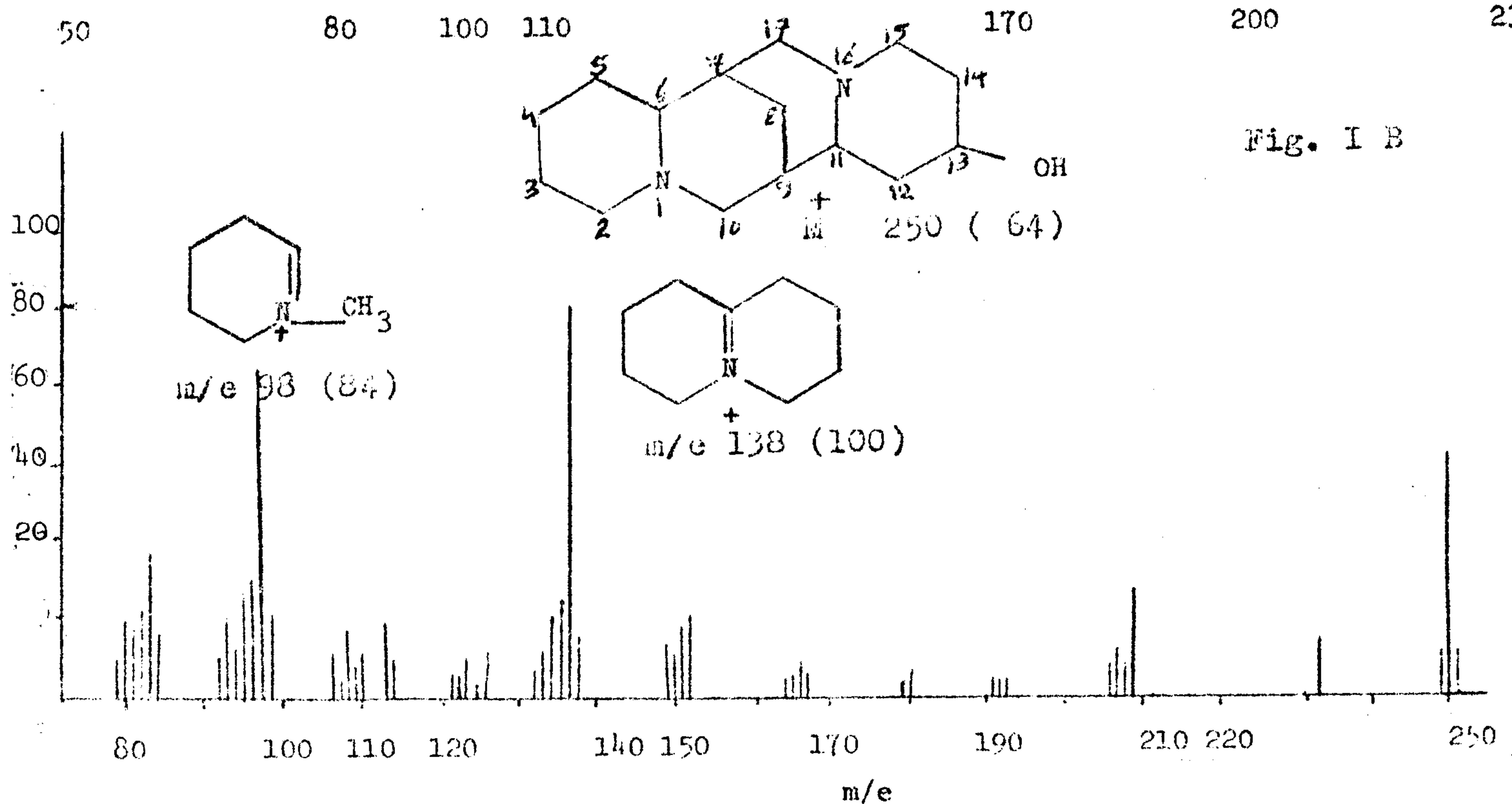


Fig. 1: Mass fragmentation pattern(70 ev) of:
A- Sparteine
B- 13-hydroxysparteine

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سبارتين و ١٣-هيدروكسي سبارتين
من أوراق سوفورا سكنديفلورا أورثو لاق

عفاف عبد الباقي - مقبول أحمد مقبول
قسم العقاقير - كلية الصيدلة - جامعة اسوط

في هذا البحث تم فصل قلويدى سبارتين و ١٣-هيدروكسي سبارتين
من نبات سوفورا سكنديفلورا المنزرع في مصر وذلك بطريقة كروماتوجرافيا
العمود مستخدما مادة أكسيد الألمنيوم والسليكا الهلامية، كما تم
استخدام كروماتوجرافيا الطبقة السميكة مستخدما كلوروفورم - ميثانول

وقد تمت دراسة هذين القلويدين باستخدام الرنين النووي المغناطيسي
ومطياف الكتلة وكذلك بتكوين املاح لهما مثل البكرات والبيركلورات وكذلك
مشتق الاستيل ومقارنة خواص هذه المشتقات بمثيلاتها المذكورة في المراجع
العلمية .

وأوضح من دراسة هذه الصفات أن قلويد سبارتين سبق فصله من هذا
النبات، أما ١٣-هيدروكسي سبارتين فقد تم فصله من هذا النبات
لأول مرة .

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