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CHENOPODIACEAE

ISOLATION OF TRIACETONAMINE FROM *SALSOLA TETRANDRA*

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Abstract—The alkaloids of the most common Egyptian *Salsola* species were studied. Four bases were isolated from *S. tetrandra*: a major constituent, triacetonamine and two other minor bases as well as betaine. TLC showed that the same compounds were present in *S. kali*, *S. longifolia* and *S. rigida*.

INTRODUCTION

THE ALKALOIDS salsoline and salsolidine were isolated from *Salsola richteri* Kar.[1,2] Salsamine isolated from this species[3] was established to be an artefact formed during the extraction of the alkaloids.[4] Borkowski *et al.*[5] also found salsoline in *S. kali* L., *S. soda* L. and *S. ruthenica* Iljin. Subaphylline was isolated from *S. subaphylla* C. A. Mey.[6]

Salsoline (6-hydroxy-7-methoxy-1-methyl-1,2,3,4-tetrahydroisoquinoline) and its hydrochloride are useful in the treatment of hypertension[7] and the hydrochloride salt was introduced in the U.S.S.R. Pharmacopoeia (Moskow 1968). The methyl ether derivative (saldolidine) is pharmacologically active against hypertension.[8]

Hsia T'ien Liang *et al.*[9] reported the presence of betaine in *S. ruthenica* Iljin but could detect neither salsoline nor salsolidine.

The present paper deals with the alkaloids of the most common Egyptian Salsola species, viz. *S. tetrandra* Forsk., *S. kali* L., *S. longifolia* Forsk. and *S. rigida* Pall.[10]

RESULTS

Triacetonamine, two other bases (II and III) as well as betaine are present in all species. Triacetonamine has been isolated from other plants, e.g. *Acalypha indica*.[11] However, its occurrence in *Salsola* is reported here for the first time. It is not an artefact arising from acetone and ammonia present during the extraction process since it can be separated from a dichloroethane extract.

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Base II seems to be a cyclic secondary amide either with or without an aromatic substitution. Base III contains an —NH— group and an ether linkage in the molecule. Due to the shortage of material, no further study could be carried out.

EXPERIMENTAL

Column chromatography of S. tetrandra *extracts.* Successive extracts of the defatted powdered plants (moistened with 25% NH_4OH and extracted by percolation with $(CH_2Cl)_2$, then with acetone and lastly with 75% EtOH) were fractionated separately on alumina column, after treating each extract after removal of the solvent with 2% HCl, till complete removal of the bases. The acidic layer was filtered and shaken with benzene to remove any extraneous matter. The acidic layer was neutralized with cold NH_4OH to pH 7, made alkaline with K_2CO_3 to pH 8·5–9 and extracted with $CHCl_3$. After washing and drying, the solvent was removed *in vacuo* at 45° (residue A, B and C). The aq. alkaline layer (M) of each extract still responded to alkaloidal tests.

The column was eluted with hexane, varying proportions of hexane–benzene, benzene, varying proportions of benzene–$CHCl_3$, $CHCl_3$, $CHCl_3$–MeOH (9:1, 4:1, 3:2 and 2:3) and MeOH. TLC of these fractions revealed the presence of three bases (I, II and III) in the organic phase. The aqueous phase revealed one spot (IV).

Base I. Base I, $C_9H_{17}NO$ (155), m.p. 48–50°; yellowish crystalline rectangular platelets, were obtained from hexane–benzene (3:2 and 1:1 fractions). It has a mouse-like odour, was soluble in most solvents and was homogeneous by TLC. The hydrochloride, m.p. 200°; (Found: C, 56·93; H, 9·63, N, 7·60; required for $C_7H_{17}NO$ HCl: C, 56·59; H, 9·50; N, 7·33%.$[\alpha]^{25}_{D}$ +14·3 (in EtOH)). Base I proved to be triacetonamine on the basis of mass, IR and NMR spectral and R_f comparison with an authentic sample.

Base II. Transparent crystals (m.p. 117–118°) obtained from the fractions eluted with benzene–$CHCl_3$ form 3:7 (residue A) and benzene–$CHCl_3$, 2.3 (residue C). Base II has R_f 0·11 in MeOH–H_2O (1:1) or in BuOH–HOAc–H_2O (4:1:2) and is not identical with salsoline or salsolidine. The IR spectrum shows two bands at 3400 cm^{-1} and 3180 cm^{-1} which are attributable to the NH absorption bands of a *cis*-bonded secondary amide. The two carbonyl absorptions of the amide are found at 1670 cm^{-1} and 1630 cm^{-1}. A phenyl group is probably absent since there is no absorption at 3030 cm^{-1}; however two weak bands are found at 1500 and 1580 cm^{-1} which may indicate the presence of an aromatic group.[12] It is also probable that this compound is a cyclic secondary amide, because of the absence of the NH absorption bands for non-cyclic amide at 1570–1515 cm^{-1}.

Base III. A dark brown oily residue obtained from $CHCl_3$–MeOH (9:1) fractions of residue A and C; giving positive alkaloidal tests and a single spot on TLC (BuOH–HOAc–H_2O) (4·1:2) with R_f 0·16 different from salsoline or salsolidine. The hydrochloride, also gave one spot (R_f 0·16) and melted at 283°. The IR spectrum (KBr) shows bands at 3045 cm^{-1} (=CH olefinic), at 2927 cm^{-1} and 2850 cm^{-1}, also at 1395 cm^{-1} and 1455 cm^{-1} (—CH_2— group), at 2650 cm^{-1} and 2450 cm^{-1} (ionized NH in the form of salt), at 1110 cm^{-1} (C—O—C ether linkage) and at 1510 cm^{-1} and 1570 cm^{-1} (deformation of NH).

Water soluble base IV. The extraction was conducted according to the reineckate method.[13] The TLC revealed two spots, a major one with R_f 0·58 (identical with betaine) and a minor one R_f 0·06 using MeOH–H_2O (1:1). After purification, base IV had m.p. 293° with decomposition, elementary analysis, (Found: C, 51·70, H, 10·29, N, 11·08, calc. for betaine ($C_5H_{11}NO_2$): C, 51·26; H, 9·46; N, 11·96; its hydrochloride, m.p. 240°, undepressed with authentic betaine hydrochloride and gave identical IR spectra). The UV spectrum shows absorption at 202 nm. The NMR spectrum in methanol shows the $(CH_3)_3N$-absorption at 7·05 τ (nine protons) and a methylene group (two protons) at 6·5 τ.

Investigation of the bases of the other Salsola *species.* TLC examination showed that betaine, triacetonamine, base II and base III are present in all the other three species. *S. longifolia* has an additional spot (R_f 0·36) in the BAW mixture. Salsoline and salsolidine are universally absent.

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