

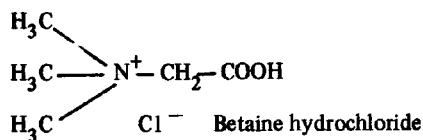
Isolation of Betaine from *Lycium Barbarum*

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Lycium barbarum Linn. (Solanaceae) (Syn. *L. europaeum*) grows wildy in Pakistan¹ and is known locally as Chirchitta. Its berries are reported to possess aphrodisiac properties². Its young leaves contain hydrocyanic acid². In 1968, M. Manzoor-i-Khuda and Miss Sitwat Sultana³ reported the isolation of β -sitosterol and a water soluble, hygroscopic alkaloid named by the authors as Lyceamin. Since lyceamin was reported to be a new base and no structure for it was suggested, we carried out a reinvestigation of the alkaloidal constituents of *Lycium barbarum*. With a modified procedure, as described in the experimental it was possible to isolate a hygroscopic colourless crystalline compound giving positive test with Dragendorff's reagent. The hydrochloride m.p. 227° (decomp.) and picrate m.p. 181° were however non-hygroscopic crystalline compounds and were used for further identification. The n.m.r. of the hydrochloride in D₂O using DSS as internal indicator showed only two singlets at δ 3.3 and δ 4.2 in the ratio of 9:2, in addition to the large peak due to solvent at δ 4.66. The compound had therefore a simple structure with no aromatic protons. Since the melting points of the salt and the n.m.r. spectral data resembled those of betaine, a direct comparison was carried out. The picrate and hydrochloride of the compound isolated by us showed no depression in melting point when admixed with an authentic sample of betaine picrate lit.⁴ m.p. 183°C and hydrochloride of the compound isolated by us showed no depression in melting point when admixed with an authentic sample of betaine picrate lit.⁴ m.p. 183°C and hydrochloride lit.⁴ m.p. 227-228° (decomp.) respectively. The i.r. spectra of the isolated and authentic samples of betaine hydrochloride were superimposable. The occurrence of betaine in *Lycium barbarum* is therefore proved.



Experimental

The fresh undried aerial part of the plant *Lycium barbarum* Linn. was extracted with methanol. The combined extract was evaporated under reduced pressure, the residue taken up in water and ethyl acetate. The aqueous layer which gave positive test with Dragendorff's reagent was separated off and passed through a column of cation exchanger (Merck Ion Exchanger I, H form). After washing the column thoroughly with distilled water in order to remove unabsorbed material, the column was eluted with 2N ammonium hydroxide solution. The eluent was evaporated on the water bath, the residue taken up in methanol and filtered. The residue obtained on evaporation of the solvent was purified through preparative layer chromatography on silica gel plates using methanol as developing solvent. A colourless hygroscopic crystalline residue was obtained which was converted into the hydrochloride by dissolving it in methanol containing a few drops of concentrated hydrochloric acid and evaporation. The hydrochloride as obtained above, was recrystallised from methanol m.p. 227° (decomp.), not depressed on admixture with authentic sample of betaine hydrochloride. It also showed identical R_f values as an authentic sample of betaine hydrochloride on silica gel plates (DC Microcards Riedel de Haen) using ethanol (96%)-glacial acetic acid (6:4 V/V) as developing solvent. The i.r. spectra of the two samples were superimposable.

NMR (D₂O): singlets at δ 3.3 (9H) 3 x *N*-methyl and δ 4.2 (2H) (CH₃)₃N⁺-CH₂-COOH.

The picrate was prepared by treatment of a solution of the hydrochloride in dry ethanol with an ethanolic solution of picric acid. The crystalline yellow precipitate was filtered and recrystallised from absolute ethanol yielding yellow needles, m.p. 181°C, undepressed on admixture with a sample of betaine picrate prepared similarly from authentic betaine hydrochloride.

References

1. R.R. Stewart, *An annotated Catalogue of the Vascular plants of West Pakistan and Kashmir*, in E. Nasir and S.I. Ali, (Editors) *Flora of West Pakistan* (1972) page: 641.
2. R.N. Chopra, S.L. Nayar and I.C. Chopra. *Glossary of Indian Medicinal Plants*, Council of Scientific and Industrial Research, New Delhi (1956) page: 158.
3. M. Manzoor-i-Khuda and Miss Sitwat Sultana, *Pak J. Sci. Ind. Res.*, 11, (3) 247 (1968).
4. J.R.A. Pollock and R. Stevens (Editors) *Dictionary of Organic Compounds*, 4th Edition (1965), Eyre and Spottiswoode Published Ltd. London, page: 389.