Nostocarboline: Isolation and Synthesis of a New Cholinesterase Inhibitor from *Nostoc* 78-12A

Supporting Information

Paul G. Becher, Julian Beuchat, Karl Gademann* and Friedrich Jüttner*

Limnological Station, Institute of Plant Biology, Seestr. 187,

8802 Kilchberg, Switzerland

and

Laboratorium für Organische Chemie der

Eidgenössischen Technischen Hochschule Zürich

ETH Hönggerberg, 8093 Zürich, Switzerland

Table of Contents

6-Cl-norharmane.

Modification of the procedure by Nakano, K.; Suyama, K.; Fukazawa, H.; Uchida, M.; Wakabayashi, K.; Shiozawa, T.; Terao, Y. *Mutation Research* **2000**, *470*, 141-146.

Norharmane (600 mg, 3.6 mmol) was dissolved in EtOH (22 mL) and acetic acid (12 mL). The solution was cooled to 0 °C and NaOCl solution (Javel water, commercial solution) was added dropwise over 20 min. The reaction mixture was stirred for 20 min. at 0°C and for 5 h at room temperature. The resulting suspension was diluted with EtOAc (200 mL) and the layers separated. The aqueous layer was twice extracted with EtOAc and the combined organic layers were twice washed with $NaHSO_3$ solution (7.5% v/v) and once with H_2O. These combined aqueous layers were made basic by the addition of $NaHCO_3$ and three times extracted with EtOAc. The combined organic layers were dried ($MgSO_4$) and the solvent evaporated. Flash chromatography (SiO_2, EtOAc) provided the title compound, 6-Cl-norharmane (420 mg, 2.1 mmol, 57 %) as off-white solid. The analytical data matched those reported.

Figure S-1: Nostocarboline (**1**), natural sample (CD_3OD, 300 MHz).

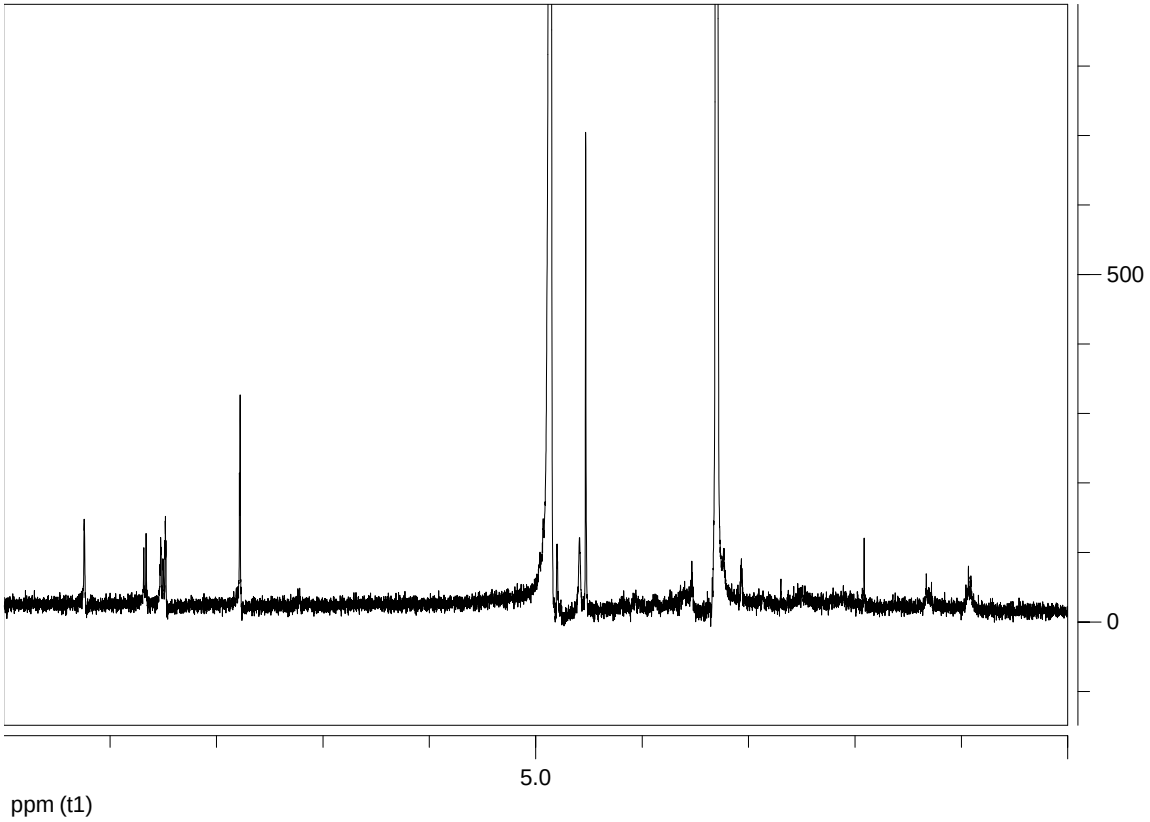

Figure S-2: Nostocarboline iodide (**1a**), synthetic sample (CD_3OD, 300 MHz).

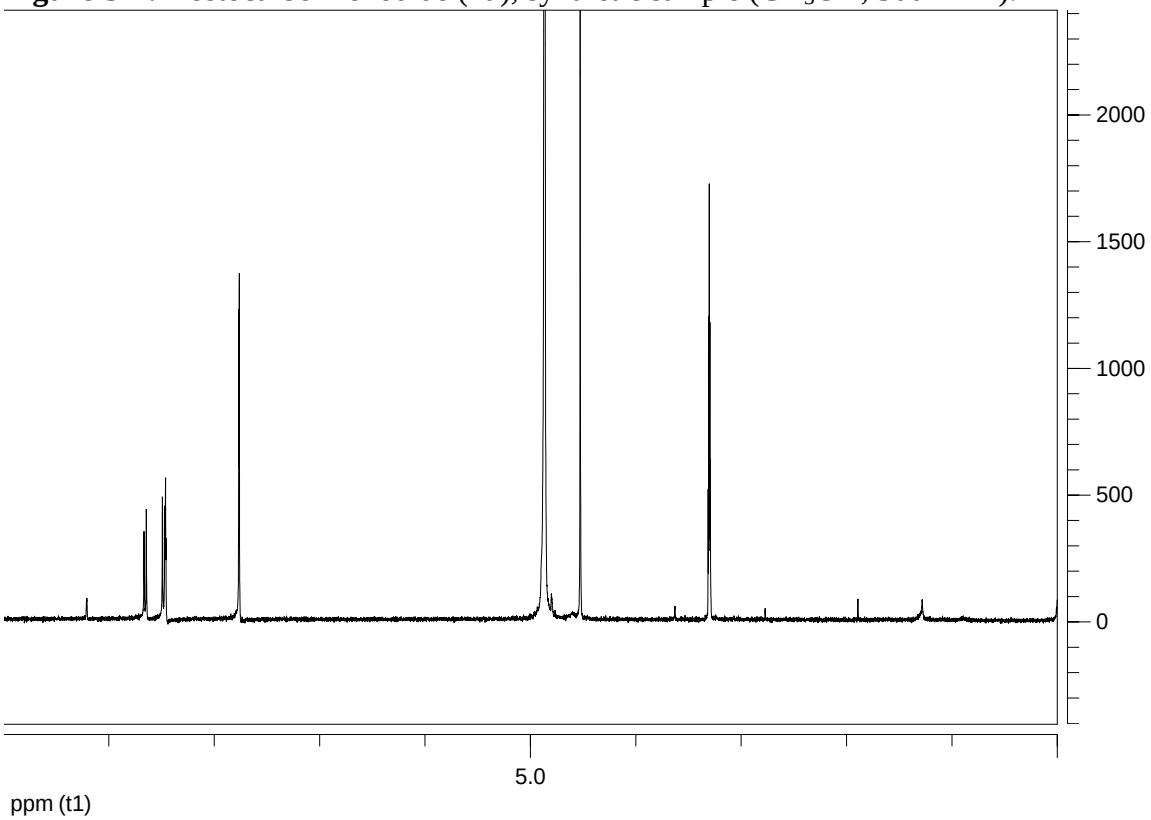

Figure S-3: Nostocarboline, mixture of natural (**1**) and synthetic (**1a**) samples (CD_3OD, 300 MHz).

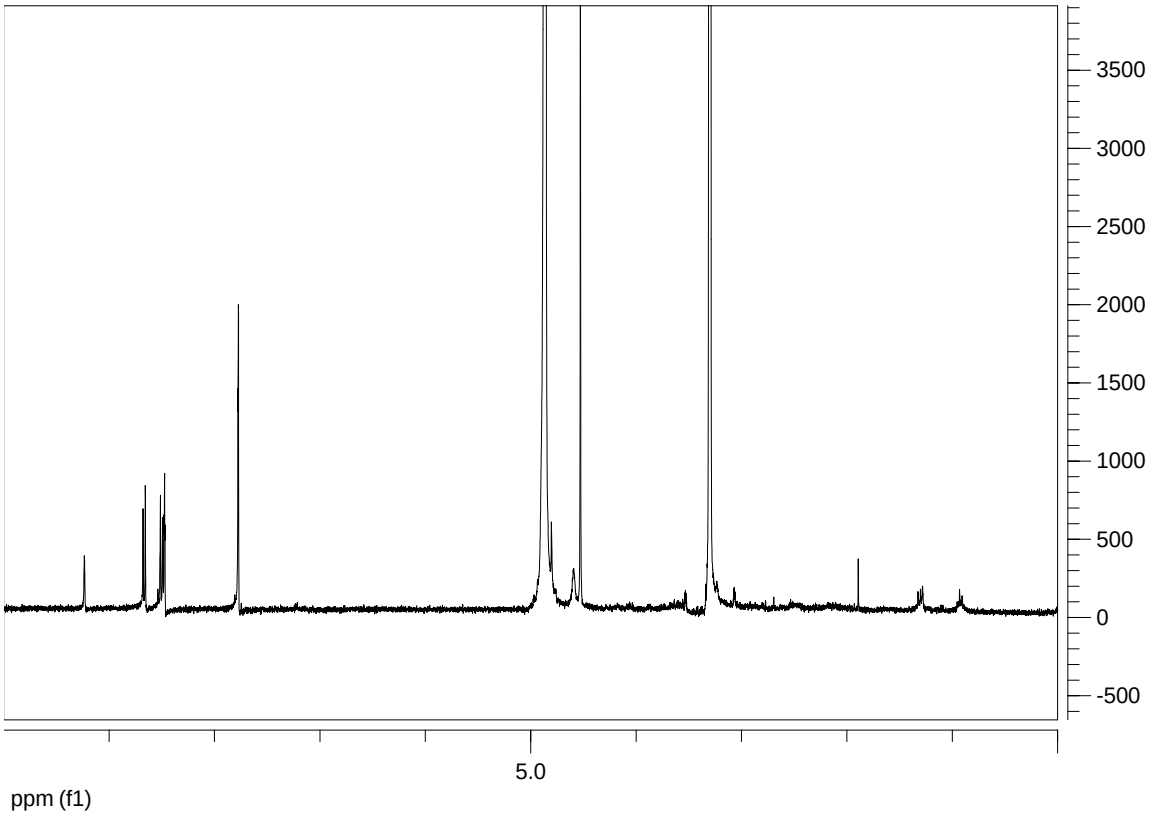

Figure S-4: Nostocarboline (**1**), natural sample (CD_3OD, 300 MHz).

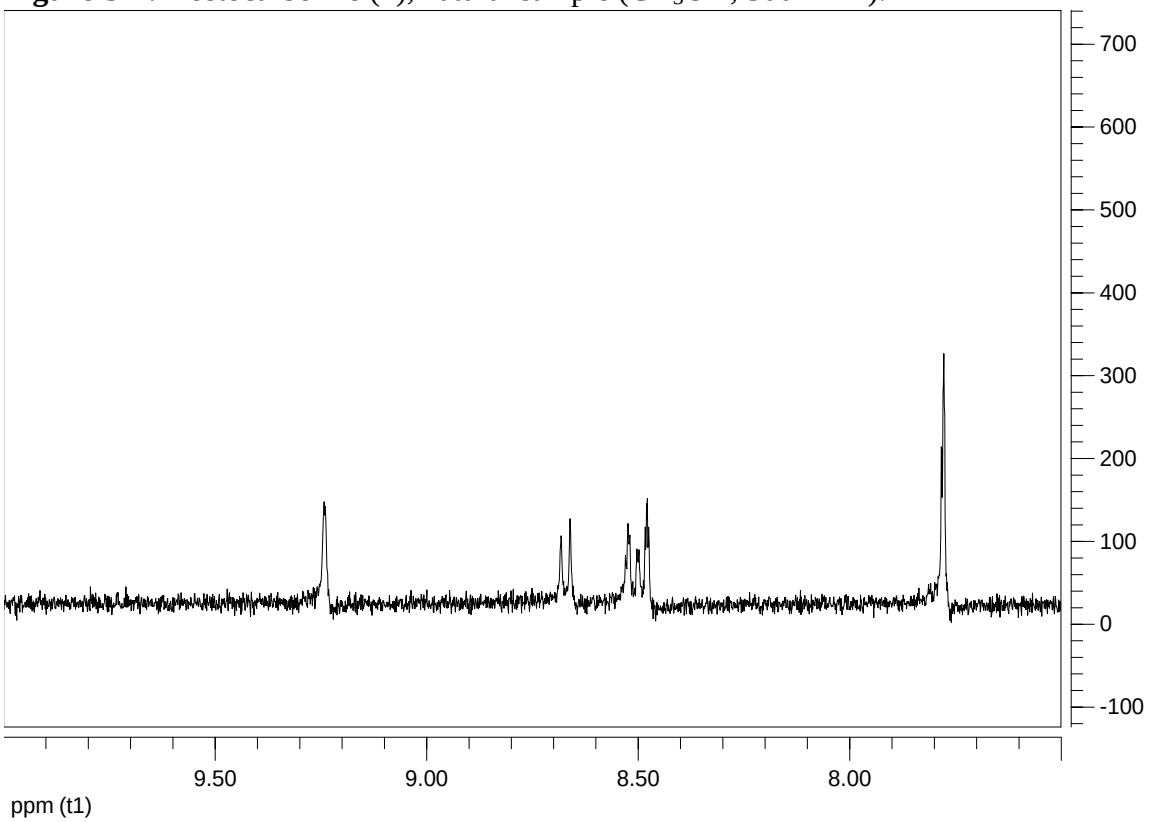

Figure S-5: Nostocarboline iodide (**1a**), synthetic sample (CD_3OD, 300 MHz).

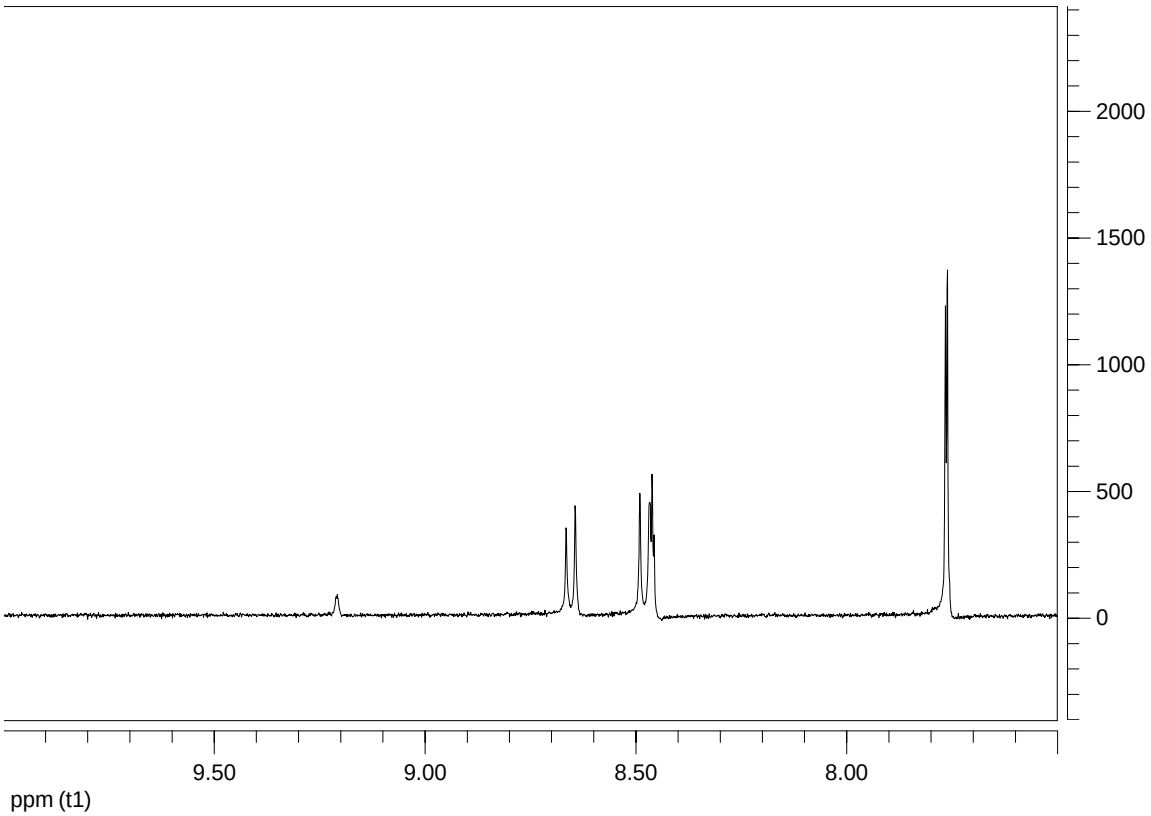

Figure S-6: Nostocarboline iodide, mixture of natural (**1**) and synthetic (**1a**) samples (CD_3OD, 300 MHz).

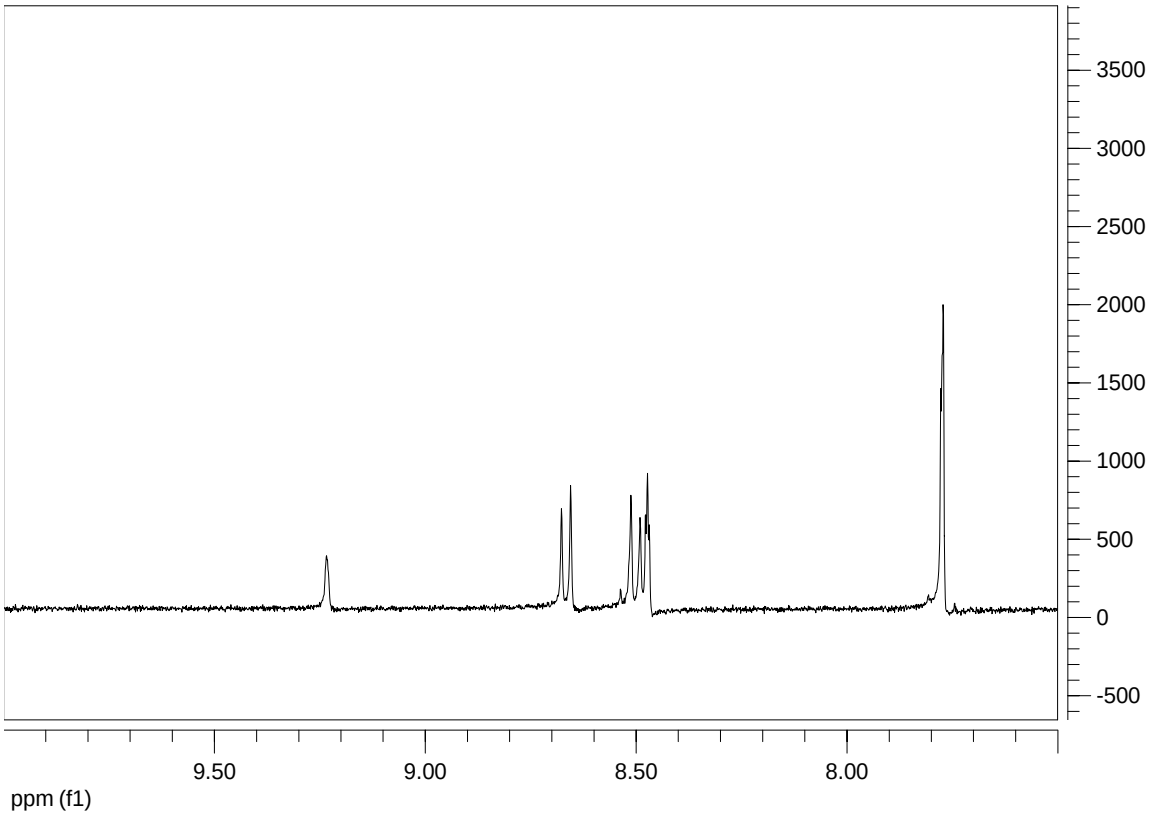

Figure S-7: Nostocarboline iodide (**1a**), synthetic sample (CD_3OD, 600 MHz). The inset is a part of the HSQC spectrum.