

Molecules **2000**, *5*, M186

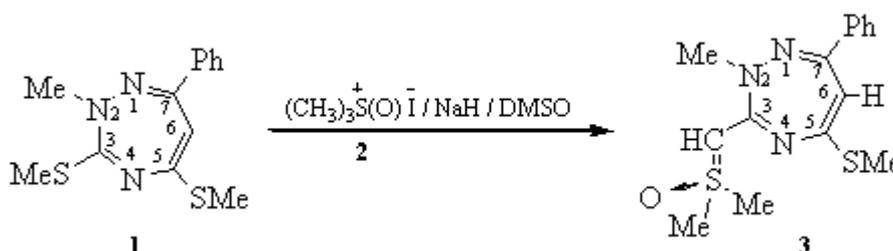
2-Methyl-5-methylmercapto-3-dimethylsulfoxymethine-7-phenyl-1,2,4-triazepine

My. Y. Ait Itto¹, A Hasnaoui¹, A. Riahi² and A. Huet³

¹Laboratoire de chimie des substances naturelles et des Hétérocycles, Département de Chimie, Faculté des Sciences Semlalia, B.P. 2390, 40001 Marrakech-Maroc.

²Université de Reims, UFR Sciences, UMR N° 6519, Réactions Sélectives et Applications, B.P. 1039, 51687 Reims Cedex 2 France. ³Laboratoire de Synthèse Organique, Université de Maine, Le Mans-France. *Phone: 2124434349, Fax: 2124437408, E-mail: aititto@ucam.ac.ma.

Received: 20 September 2000 / Accepted: 16 November 2000 / Published: 25 December 2000



All the following operations were performed under an inert atmosphere using standard vacuum line techniques. Trimethyloxosulfonium iodide (**2**, 0.73 g, 3.3 mmol) [1] was suspended in anhydrous DMSO (6 mL) and sodium hydride (80 % suspension in mineral oil, 0.1 g, 3.3 mmol) was added at room temperature. The mixture was vigorously stirred for 15 min and then cooled to 10°C before adding rapidly a solution of the 1,2,4-triazepine **1** (0.3 g, 1.1 mmol) [2] in anhydrous DMSO (3 mL). After stirring at room temperature during 48 hours, the reaction mixture was poured in cold water (40 mL) and extracted with chloroform (3 x 15 mL). The combined organic layers were washed with water (3 x 15 mL), dried over anhydrous MgSO₄. The solvent was then evaporated under vacuum. The pure product **3** was obtained as an orange solid after recrystallization from carbon tetrachloride (85 % yield).

Mp.: 169-170°C

¹H NMR (CDCl₃, 400 MHz): 2.35 (s, 3H, SCH₃); 3.11 (s, 3H, NCH₃); 3.47 (s, 6H, (CH₃)₂SO); 3.71 (s, 1H, H-C=S); 5.66 (s, 1H, H-C6); 7.31-7.70 (m, 5H, aromatic CH).

¹³C NMR (CDCl₃, 100 MHz, assignment based on NOESY, HMBC and COSY experiments): 14.82 (SCH₃); 41.42 (NCH₃); 42.43 ((CH₃)₂SO); 59.79 (H-C=S); 100.95 (C6); 127.98; 128.02; 129.39; 136.85 (aromatic C-H); 161.59 (C3); 163.18 (C7); 164.76 (C6).

MS-EI⁺(m/z, %): 321 (100, [M]⁺), 242 (30, [M-(CH₃)₂SO]⁺), 230 (5, [M-CH₃)₂SOCH]⁺).

Anal calcd. for C₁₅H₁₉N₃S₂O (321.46): C, 56.04; H, 5.96; N, 13.07; found C, 55.91; H, 6.04; N, 13.02 %.

References

- Corey, E. J.; Chakovsky, M. *J. Am. Chem. Soc.* **1965**, *87*, 1353.
- Ait Itto, My. Y.; Hasnaoui, A.; Riahi, A.; Lavergne, J.-P. *Tetrahedron Lett.* **1997**, *38*, 2087.

Sample availability: available from the authors and MDPI.

©2000 MDPI. All rights reserved. *Molecules* website www.mdpi.org/molecules/