Synthesis of 2,3-Disubstituted-4-oxothiazolidine 1-Oxides

M. T. Omar, A. E. El-Khamry and F. A. Sherif

Chemistry Department, Faculty of Science, A'in Shams University, Abbassia, Cairo, Egypt

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The reaction of chloramine-T with 2,3-disubstituted-4-oxothiazolidines **1a-c** give the corresponding sulfoxides **2a-c**, whose structures were based on analytical and spectral data.

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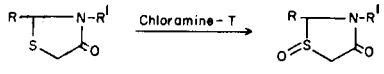
The synthesis of 3-acylthiazolidine 1-oxides has been reported by Ratner and Clarke (1). The recent finding that some thiazolidine 1-oxides are valuable as antidotes for herbicides (2,3) prompted us to synthesise the 4-oxothiazolidine 1-oxides, a new sulfoxide system.

Thus, chloramine-T reacts with 2,3-diaryl- and 2-aryl-3-(2-pyridyl)-4-oxothiazolidines **1a-c** in dioxane water mixture to give the corresponding 2,3-disubstituted-4-oxothiazolidin-1-oxides **2a-c**, respectively.



1 **2**

- **a** R = C_6H_5, R^1 = C_6H_5
- **b** R = 4-Cl-C_6H_5, R^1 = C_6H_5
- **c** R = C_6H_5, R^1 = 2-C_5H_4N

The structure of **2b** is based on analytical data and infrared spectrum which shows the absorptions for CH, C=O and S=O groups (4), and supported by the ^1H nmr spectrum which shows from low to high field the signals of aromatic (multiplet), heteroaromatic methine (apparent triplet) and heteroaromatic methylene (quartet with splitting at the two lower field signals) protons, with the integrated proton areas of 9:1:2, respectively, which are consistent with the proposed structure. The observed AB pattern is attributed to the magnetic nonequivalence of the methylene protons (5). The splitting observed for the methine as well as for the two lower field signals of the methylene protons is attributed to the long range coupling between the *cis* equatorial 2-H and 5-H; a similar coupling was previously reported (6) for 2,3-disubstituted thiazolidines.

The structure of the remaining sulfoxides **2a** and **2c** is based on analytical data, infrared spectra (*cf.* Experimental) and confirmed by analogy of their electronic spectra to that of **2b**.

EXPERIMENTAL

All melting points are not corrected. Infrared spectra were taken on a Unicam SP 1200 spectrophotometer as KBr discs. Electronic spectra were recorded on a Perkin-Elmer 4000A spectrophotometer in ethanol solutions. Nmr spectrum was taken on a Varian EM 360 instrument operating at 60 MHz in a solution of deuterated chloroform containing tetramethylsilane as an internal standard with chemical shifts (δ) expressed in ppm downfield from TMS.

2,3-Disubstituted-4-oxothiazolidine 1-Oxides (**2a-c**).

The mixed solutions of chloramine-T (1 g.) in water (10 ml.) and each of the 4-oxothiazolidines **1a-c** (1.3 g.) in pure dioxan (45 ml.) are heated in water bath for 5 hours. Evaporation in vacuum gives an oily residue which is triturated successively with small portions of aqueous cold sodium hydroxide solution (2%), light petroleum (b.p. 40-60°) and then with cold methanol untill solidification. The product is filtered off, washed throughly with water, dried and recrystallized from benzene methanol mixture to give the following compounds:

2,3-Diphenyl-4-oxothiazolidine 1-Oxide (**2a**).

This compound (0.6 g., 45%) had m.p. 178-180°; ir: 3080, 3000, 2940 cm^{-1} (CH), 1702 cm^{-1} (C=O), 1052 cm^{-1} (S=O); uv: 237 nm (4500).

Anal. Calcd. $C_{15}H_{13}NO_2S$: C, 66.40; H, 4.80; N, 5.15. Found: C, 66.60; H, 4.90; N, 5.0.

2-(4-Chlorophenyl)-3-phenyl-4-oxothiazolidine 1-Oxide (**2b**).

This compound (0.85 g., 53%) had m.p. 165-166°; ir: 3080, 2980, 2940 cm^{-1} (CH), 1700 cm^{-1} (C=O), 1052 cm^{-1} (S=O); uv: 236 nm (3850); nmr: δ 7.1-7.65 (m, 9, aromatic H), 5.95 (apparent t, 1, heteroaromatic methine H), 3.7 (q, 2, heteroaromatic methylene H).

Anal. Calcd. $C_{15}H_{12}ClNO_2S$: C, 58.90; H, 3.90; N, 4.60. Found: C, 58.85; H, 3.90; N, 4.60.

2-Phenyl-3-(2-pyridyl)-4-oxothiazolidine 1-Oxide (**2c**).

This compound (0.7 g., 51%) had m.p. 168-170°; ir: 3080, 3000, 2980, 2940 cm^{-1} (CH), 1708 cm^{-1} (C=O), 1055 cm^{-1} (S=O); uv: 240 nm (4250).

Anal. Calcd. $C_{14}H_{12}N_2O_2S$: C, 61.75; H, 4.40; N, 10.30. Found: C, 61.75; H, 4.50; N, 10.20.

REFERENCES AND NOTES

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