Chemistry of Heterocyclic Compounds, Vol. 36, No. 11, 2000

REACTIONS OF 1,2,5-THIADIAZOLE AND 1,2,5-SELENADIAZOLE N-OXIDES WITH HYDROGEN PEROXIDE AND SODIUM HYPOCHLORITE

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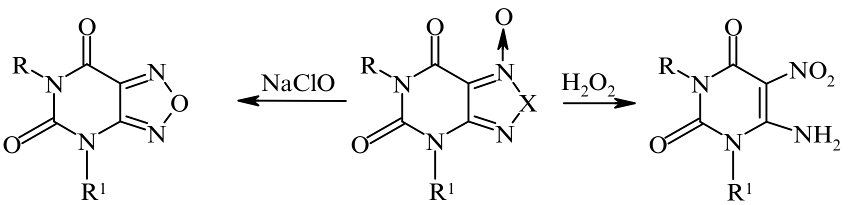
Keywords: 1,2,5-thiadiazoles, 1,2,5-selenadiazoles, oxidation.

The 1,2,5-thiadiazole ring is quite inert to most oxidizing agents [1]. Benzothiazole is not oxidized by 30% hydrogen peroxide in acetic acid at 100°C [2] and benzoselenadiazole is also stable under these conditions [3]. According to our results, 1,2,5-thia- and 1,2,5-selenadiazoles annelated with pyrimidine rings are no less stable.

A vigorous reaction occurred when 1,2,5-thia- and 1,2,5-selenadiazolo[3,4-*d*]pyrimidine-5,7-(4H,6H)dione N-oxides (**1c-f**) [4] were treated with 10-30% hydrogen peroxide to give 6-amino-5-nitrouracils (**2a,b**).



3a–c **1a–f** **2a–c**

1 a $R = R^1 = H, X = S$; **b** $R = R^1 = H, X = Se$; **c** $R = H, R^1 = CH_3, X = S$; **d** $R = H, R^1 = CH_3, X = Se$;
e $R = R^1 = CH_3, X = S$, **f** $R = R^1 = CH_3, X = Se$; **2 a** $R = R^1 = H$; **b** $R = H, R^1 = CH_3$;
c $R = R^1 = CH_3$; **3 a** $R = R^1 = H$; **b** $R = H, R^1 = CH_3$; **c** $R = R^1 = CH_3$

When 1,2,5-thia- and 1,2,5-selenadiazole N-oxides **1a-f** reacted with sodium hypochlorite they were readily converted into 1,2,5-oxadiazoles **3a-c**. Compounds **2** and **3** were identical to those prepared by a known method [5, 6].

Mass spectra were obtained with an MX 1321 apparatus with direct insertion of the sample into the ion source, with an ionizing current of 70 V and an ionizing chamber temperature of 220°C. IR spectra of nujol mulls were recorded with a Specord-80 instrument. Chromatographic monitoring was carried out on Silufol UV-254 strips with 2:1 acetone–hexane and 10:1 chloroform–methanol systems.

6-Amino-5-nitrouracils (2a-c). Compounds **1a-d** (1 mmol) were added to 10-30% hydrogen peroxide (5 ml). When the vigorous reaction ceased the mixture was carefully heated until it was decolorized and was then filtered. White crystals of **2a-c** were filtered off after cooling. Yields 65-70%. Compound **2a**: Found, %: C 27.87; H 2.36; N 32.58. $C_4H_4N_4O_4$. Calculated, %: C 27.92; H 2.34; N 32.55. Compound **2b** : Found, %: C 32.25; H 3.27; N 30.12. $C_5H_6N_4O_4$. Calculated, %: C 32.27; H 3.25; N 30.10. Compound **2c**: Found, %: C 36.00; H 4.05; N 28.03. $C_6H_8N_4O_4$. Calculated, %: C 36.01; H 4.03; N 27.99.

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1,2,5-Oxadiazolo[3,4-*d*]pyrimidine-5,7-(4H,6H)diones (3a-c). Compounds **1c-f** (1.0 mmol) were added at 5-10°C to aqueous sodium hypochlorite solution (7 ml) prepared from sodium hydroxide (1.6 g) and chlorine (1.4 g). After 1 h, the precipitate was filtered off and recrystallized from water or aqueous ethanol. Yields of **3a-c** 35-40%.

Compound **3a**. Found, %: C 31.20; H 1.30; N 36.37. $C_4H_2N_4O_3$. Calculated, %: C 31.18; H 1.31; N 36.36. Compound **3b**. Found, %: C 35.70; H 2.40; N 33.30. $C_5H_4N_4O_3$. Calculated, %: C 35.72; H 2.40; N 33.33. Compound **3c**. Found, %: C 39.50; H 3.30; N 30.82. $C_6H_6N_4O_3$. Calculated, %: C 39.57; H 3.32; N 30.76.

REFERENCES

1. V. G. Pesin, *Usp. Khim.*, **39**, 1950 (1970).
2. O. Hinsberg, *Ber.,* **48**, 1611 (1915).
3. E. Sawicki and A. Carr, *J. Org. Chem.,* **22**, 503 (1957).
4. A. A. Yavolovsky, E. A. Kuklenko, and E. I. Ivanov, *Khim. Geterotsikl. Soedin.,* 997 (1996).
5. E. C. Taylor and A. McKillop, *J. Org. Chem.,* **30**, 3153 (1965).
6. E. C. Taylor, Y. Maki, and A. McKillop, *J. Org. Chem.,* **37**, No. 10, 1601 (1972).