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Note

The Oxidation of 10-Acylphenoselenazines with Phenyl Iodosoacetate

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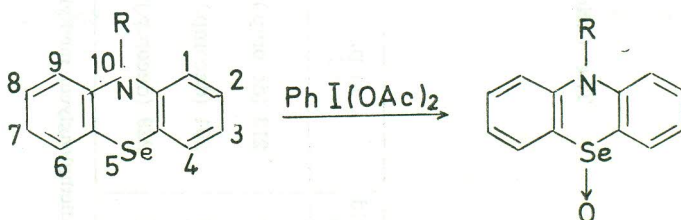
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Received December 24, 1971

Continuing our investigation¹ on the synthesis and chemical properties of phenoselenazine and its derivatives, in this paper we report on the oxidation of some 10-acylphenoselenazines.

For the oxidation of selenious atom in organic selenides, various oxidizing agents have been used: potassium dichromate in glacial acetic acid², potassium permanganate in diluted acetic acid³, 30% hydrogen peroxide⁴, sodium metaperiodate and iodobenzene dichloride⁵. To the best of our knowledge, the oxidation of phenoselenazine was previously accomplished only by Cornelius⁶, who had used nitric acid (sp. gr. 1.40) as oxidizing agent. He had found that by this reaction both oxidation of selenious atom to selenoxide and nitration in position 3 and 7 occurred.

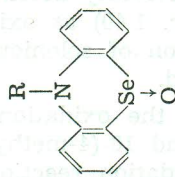
In the present work, we report on the oxidation of 10-benzoylphenoselenazine, 10-(2-furoyl)phenoselenazine and 10-(4-methylbenzoyl)phenoselenazine with phenyl iodosoacetate⁷. The oxidation reaction was carried out in 99% acetic acid at room temperature with constant shaking for 9 hours, using an excess of the oxidizing agent. Under such conditions, all mentioned 10-acylphenoselenazines were oxidized to the corresponding selenoxides. The formation of: 10-benzoylphenoselenazine-5-oxide, 10-(2-furoyl)phenoselenazine-5-oxide and 10-(4-methylbenzoyl)phenoselenazine-5-oxide was confirmed by elemental analysis.



R = Benzoyl-, 2-Furoyl-, 4-Methyl-benzoyl

To confirm the formation of the corresponding 10-acylphenoselenazine-5-oxides, their IR spectra were investigated. The presence of two strong bands in the regions 790—795 cm^{-1} and 810—838 cm^{-1} were observed. These

TABLE I
The Oxidation of 10-Acylphenoselenenazines with Phenyl Iodosuccinate



R	Yield %	m. p. °C	Brutto Formula	Calc'd. %			Found %			Se—O Strech. IR, cm ⁻¹
				C	H	N	C	H	N	
Benzoyl	75	213 (decomp.) ^a	C ₁₉ H ₁₃ NO ₃ Se	62.24	3.57	3.82	62.39	3.58	3.99	810 and 790
p-Toluy1	48	204 (decomp.) ^b	C ₂₀ H ₁₅ NO ₃ Se	63.10	3.97	3.68	63.10	4.01	3.90	835 and 790
2-Furoyl	36	229 (decomp.) ^c	C ₁₇ H ₁₁ NO ₃ Se	57.26	3.08	3.92	57.41	3.49	3.90	838 and 795

^a ethanol-water, ^b diethylenglycol methyleter, ^c ethanol.

two bands can be assigned to the Se-O group. The region 810—838 cm^{-1} is identical with the value for Se-O vibration in diphenyl selenoxyde⁸.

It was attempted also to hydrolyse the corresponding 10-acylphenoselenazine-5-oxides to the phenoselenazine-5-oxide, by means of 15% aqueous solution of sodium hydroxide in ethanol. All attempts to obtain analytically pure phenoselenazine-5-oxide were at that time unsuccessful.

EXPERIMENTAL

The IR spectra were taken in KBr pellets on a Perkin Elmer Model 521-Infrared spectrophotometer. Melting points are uncorrected.

General Procedure

To a solution of a 10-acylphenoselenazine (4 mmole) in glacial acetic acid (50 ml.), a solution of phenyl iodosoacetate (8 mmole) in glacial acetic acid (20 ml.) was added. The reaction mixture was shaken at room temperature for 9 hours. Acetic acid was evaporated *in vacuo* and the residue was crystallized several times till analytical purity was achieved. Solvents for crystallization, physical constants, yields, and analytical data are given in Table I.

REFERENCES

1. B. D. Podolešov and V. B. Jordanovska, *Croat. Chem. Acta* **42** (1970) 61.
2. F. Kraft and W. Vorster, *Ber.* **26** (1893) 2821.
3. D. G. Foster and S. F. Brown, *J. Am. Chem. Soc.* **50** (1928) 1182.
4. O. J. K. Edwards, W. R. Gaythwaite, J. Kenyon, and H. Phillips, *J. Chem. Soc.* (1928) 2293.
5. M. Cinquini, S. Colonna, and R. Giovini, *Chem. Ind. (London)* **48** (1969) 1737.
6. W. Cornelius, *J. Prakt. Chem.* **88** [2] (1912) 399.
7. K. H. Pausacker, *J. Chem. Soc.* (1953) 107.
8. D. Barnard, J. M. Fabian, and H. P. Koch, *J. Chem. Soc.* (1949) 2442.

IZVOD

Oksidacija 10-acilfenselenazinskih derivata pomoću fenil jodozoacetata

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Utvrđeno je da se oksidacijom 10-acilfenselenazinskih derivata pomoću fenil jodozoacetata u 99%-tnoj octenoj kiselini i pri sobnoj temperaturi dobivaju odgovarajući 10-acilfenselenazin-5-oksidi. Priređeni su: 10-benzoilfenselenazin-5-oxid, 10-(4-metilbenzoi)l)fenselenazin-5-oxid i 10-(2-furoil)fenselenazin-5-oxid.

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Prilmljeno 24. decembra 1971.