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Note

The Oxidation of 10-Acylphenoselenazines with Phenyl Iodosoacetate

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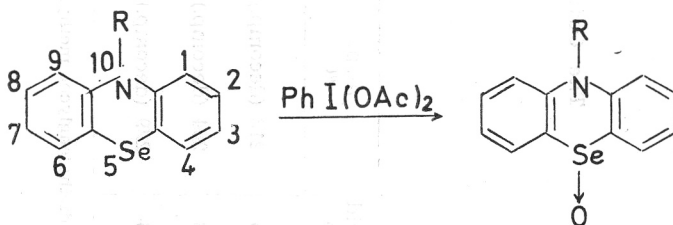
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Continuing our investigation¹ on the synthesis and chemical properties of phenoselenazine and its derivatives, in this paper we report on the oxidation of some 10-acylphenoselenazines.

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 Corneliuș⁶, 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.

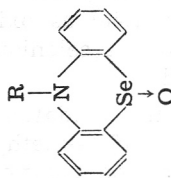
In the present work, we report on the oxidation of 10-benzoylphenoselenazine, 10-(2-furoyl)phenoselenazine and 10-(4-methylbenzoyl)phenoselenazine with phenyl iodosoacetate⁷. The oxidation reaction was carried out in 99% acetic acid at room temperature with constant shaking for 9 hours, using an excess 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
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R	Yield %	m. p. °C	Brutto Formula	Calcd. %			Found %			Se—O Strech. IR, cm ⁻¹
				C	H	N	C	H	N	
Benzoyl	75	213 (decomp.) ^a	C ₁₉ H ₁₃ NO ₂ 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 ₂ 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.

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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-metilbenzoil)fenselenazin-5-oxid i 10-(2-furoil)fenselenazin-5-oxid.

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