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Note

Preparation of Some Phenoselenazine Derivatives

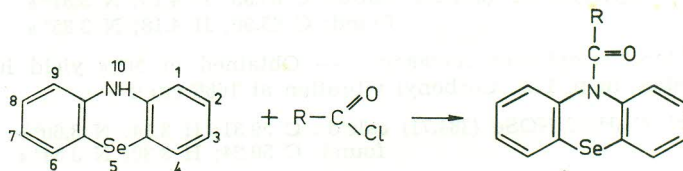
B. D. Podolešov and V. B. Jordanovska

Institute of Chemistry, Faculty of Science, University of Skopje,
Skopje, Macedonia, Yugoslavia

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In connection with our investigation of the chemical properties of phenoselenazine it was of interest to prepare some of its *N*-substituted derivatives.

The synthesis of phenoselenazine and its derivatives has been reported by several authors¹⁻⁶. For the preparation of phenoselenazine we attempted to employ the method suggested by Buu-Hoi *et al.*³, but despite our strict adherence to this method, we could not obtain phenoselenazine from the chloroform extract. Instead considerable quantities of a solid reaction product insoluble in chloroform were obtained. From this product phenoselenazine was isolated by extraction with glacial acetic acid. By action of excess of an aroyl



chloride on phenoselenazine the following *N*-aroyl derivatives were prepared: 10-(2-furoyl)phenoselenazine, 10-*p*-toluoylphenoselenazine, 10-benzoylphenoselenazine, 10-(*p*-chlorobenzoyl)phenoselenazine, 10-(1-naphthoyl)phenoselenazine and 10-(2-naphthoyl)phenoselenazine. The products were characterized by elemental analysis and by their infra-red spectra. A strong band around 1666—1650 cm^{-1} characteristic for the carbonyl vibration of tertiary amides⁷ was found in the IR spectra of all synthesized *N*-aroyl phenoselenazines.

EXPERIMENTAL

The IR spectra were run as KBr pellets on a Perkin Elmer Model 521 Infrared spectrophotometer. Melting points are uncorrected.

Phenoselenazine

To a solution of 40.8 g. (0.24 mole) diphenylamine in 200 ml. dry chloroform, 77 g. (0.33 mole) freshly prepared selenium chloride »mono« dissolved in 100 ml. dry chloroform was added in small portions with continual shaking. The reaction mixture was refluxed for 6 hrs. and after cooling 100 ml. chloroform was added. The product, insoluble in chloroform, was dissolved in acetic acid by refluxing for 4 hrs. After filtration the solution was diluted with water and the separated precipitate filtered off, washed with water and dried in dark. Thus, 35 g. (59%) of crude phenoselenazine m. p. about 150° was obtained. After sublimation at 155°/0.01 mm

yellowish crystals m.p. 180° resulted. From this product after two recrystallizations from ethanol shiny colorless crystals m.p. 194—5° (reported³ m.p. 195°) were obtained.

10-(2-Furoyl)phenoselenazine

To a solution of 10 g. (0.04 mole) phenoselenazine in 70 ml. dry benzene, 8 ml. of furoylchloride was added. The reaction mixture was refluxed for 4 hrs. The main part of benzene was then distilled off and petroleum ether was added to the residue. The separated precipitate was filtered off, washed with petroleum ether and dried. After recrystallization from ethanol-water, shiny colorless plates m.p. 172° were obtained. Carbonyl vibration at 1657 cm⁻¹.

Anal. C₁₇H₁₁NO₂Se (340.24) calc'd.: C 60.00; H. 3.26; N 4.11%
found: C 60.30; H 3.10; N 4.31%

The following compounds were prepared in the same manner by the action of excess of the corresponding aroyl chloride on phenoselenazine. The products were crystallized from ethanol-water unless stated otherwise.

10-Benzoylphenoselenazine. — A 70% yield of colorless needles, m. p. 168°. Carbonyl vibration at 1660 cm⁻¹

Anal. C₁₉H₁₃NOSe (350.26) calc'd.: C 65.14; H 3.74; N 3.99%
found: C 64.81; H 3.60; N 3.85%

10-p-Toluoylphenoselenazine. — Obtained in 57% yield as colorless plates, m. p. 212°. Carbonyl vibration at 1655 cm⁻¹.

Anal. C₂₀H₁₅NOSe (364.29) calc'd.: C 65.93; H 4.15; N 3.81%
found: C 65.90; H 4.18; N 3.85%

10-(p-Chlorobenzoyl)phenoselenazine. — Obtained in 50% yield in a form of colorless needles, m. p. 172°. Carbonyl vibration at 1666 cm⁻¹.

Anal. C₁₉H₁₂ClNOSe (384.71) calc'd.: C 59.31; H 3.14; N 3.60%
found: C 59.24; H 3.30; N 3.54%

10-(1-Naphthoyl)phenoselenazine. — It was obtained in 51% yield; colorless crystals, m. p. 194°. Carbonyl vibration at 1656 cm⁻¹.

Anal. C₂₃H₁₅NOSe (400.32) calc'd.: C 68.75; H 3.77; N 3.49%
found: C 68.80; H 3.85; N 3.50%

10-(2-Naphthoyl)phenoselenazine. — A 49% yield of colorless crystals, m. p. 191° was obtained by recrystallization from acetone-water. Carbonyl vibration at 1656 cm⁻¹.

Anal. C₂₃H₁₅NOSe (400.32) calc'd.: C 68.75; H 3.77; N 3.49%
found: C 68.95; H 3.90; N 3.54%

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ИЗВОД

Синтеза на некои феноселеназински деривати

Б. Д. Подолешов и В. Б. Јордановска

Опишана е синтезата на феноселеназин од дифениламин и диселен дихлорид. Исто така дадена е синтезата на: 10-(2-фууроил)феноселеназин, 10-бензоил-феноселеназин, 10-*n*-толуоилфеноселеназин, 10-(4-хлорбензоил)феноселеназин, 10-(1-нафтоил)феноселеназин и 10-(2-нафтоил)феноселеназин.

ХЕМИСКИ ИНСТИТУТ
ПРИРОДНОМАТЕМАТИЧКИ ФАКУЛТЕТ
СКОПЈЕ

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