

RECYCLIZATION REACTIONS.

26.* RECYCLIZATION OF HETEROARENIIUM SALTS BY ACTION OF ACID ANHYDRIDE IN THE PRESENCE OF BASE

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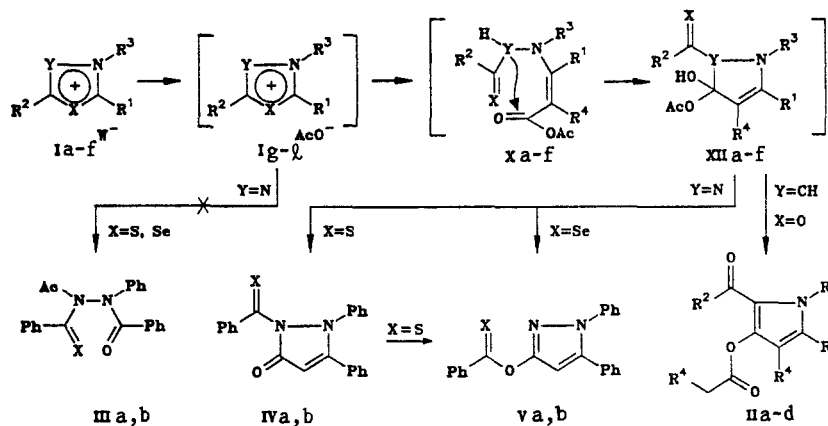
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When oxazolium, 1,3,4-thia- and -selenadiazolium, or pyrilium salts react with acid anhydride in the presence of base, they are recycled to pyrrol, pyrazole, and benzene derivatives, respectively. The typical features of cyclization involving anhydride in a number of five-membered and six-membered systems are discussed.

The reactions of X-azolium and 1,3,4-X-diazolium salts (where X = O, S, Se) with N-, O-, S-, and Se-nucleophiles have been studied in several publications [1-4]. The reactions of 1,3,4-oxadiazolium salts with C-nucleophiles formed from CH acids (malonic and cyanoacetic esters, cyclopentadiene, etc.) in the presence of base have been described [5].

The present work is a study of the reaction of oxazolium and 1,3,4-thia- and -selenadiazolium salts and their 6π-electron analogs, viz., pyrilium and pyridinium salts, with C-nucleophiles generated from acetic and propionic anhydrides (C-H₂COOCOCH₃; CH₃C-HCOOCOCH₂CH₃) in the presence of base.

Scheme 1



I, II, X, XII a, b l g, h' R¹=4-C₆H₄C₆H₅; I, X, XII c, e, f II c' l i, k, l' R¹=R²=R³=C₆H₅; I, II, X, XII d, l j R¹=R²=C₆H₅; I, II, X, XII a, b l g, h R²=C₆H₅; I, X, XII a, b II a, I g, h R³=CH₃; I, II, X, XII d, l j R³=3-C₆H₄CH₃; II, X, XII b R³=R⁴=CH₃; II a, c, d X, XII a, c-f R⁴=H; I, X, XII a-d, l g-j X=O; I, X, XII e, l k, III, IV a, V b X=S; I, X, XII f, l m, III, IV b, V a X=Se; I a, c-f; W=ClO₄; I b W=TsO; I, X, XII a-d, l g-j; Y=CH; I, X, XII e, f l k, l Y=N; I g, j-l, X, XII a, l c-f Ac=CH₃CO; I h, X, XII b' Ac=C₂H₅CO

We have previously shown that when 1,3,4-oxadiazolium salts are heated in acetic anhydride with an equimolar amount of potassium acetate they are converted to triacylhydrazines [6]. In the present work we have discovered that when oxazolium salts (Ia-d) (Scheme 1) are heated with acetic or propionic anhydride in the presence of the respective potassium alkanoate or triethylamine, the oxazoline ring is recycled to pyrrol, with the formation of the colorless crystalline compounds IIa-d. On the basis of elemental analysis and IR and PMR spectroscopy we identified these compounds as pyrrol derivatives. In the IR spectra of

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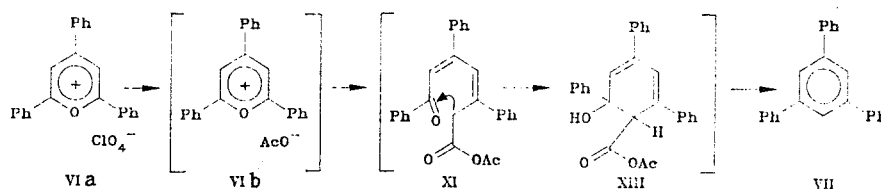
compounds IIa-d (see Experimental Part) two groups of C=O absorption bands can be distinguished: in the 1760-1730 cm^{-1} region ($\text{R}^4\text{CH}_2\text{CO}$) and in the low frequency 1630-1615 cm^{-1} region (R^2CO , conjugated with the hetero nucleus). In the PMR pyrrol spectra (see Experimental Part) the acetyl proton signals in compounds IIa, c, d appear as singlets in the 1.50-1.60 ppm range, while the propionyl in compounds IIB-c appears as a multiplet with centers at 0.93 and 1.73 ppm. The N-methyl protons of pyrrols IIa, b appear in the 3.73-3.83 ppm region, those of the 4-methyl in compounds IIB appear at 2.00 ppm, while 4-H proton of the pyrrol ring in compounds IIa, c, d appears as a singlet in the 6.00-6.27 interval; this agrees with the data for 4-substituted pyrrols [7, p. 436]. In the substituted pyrrols IIa-d the protons of the phenyl substituents appear as multiplets in the 6.83-7.50 ppm region.

When we go to the other azolium salts such as 2,3,5-tri-phenyl-1,3,4-thiadiazolium or 2,3,5-triphenyl-1,3,4-selenadiazolium perchlorates (Ie, f), we might expect two routes for the reaction in acetic anhydride in the presence of base; either by analogy with oxazolium salt recyclization described above, or by analogy with cycloconversion of 1,3,4-oxazolium salt to acylhydrazones IIIa, b described in [6]. We established that when 1,3,4-thiadiazolium (Ie) or 1,3,4-selenadiazolium salts (If) are heated in acetic anhydride with potassium acetate they are recyclized to pyrazoles IVa and Va. But in contrast to the pyrrol ring formation described above (Scheme 1, Y = CH) that takes place with separation of water, recyclization to pyrazole systems, as follows from Scheme 1 (Y = N), can take place only with detachment of acetic acid. When thiadiazolium salt (Ie) recyclizes, first pyrazolone IVa separates; when heated to 180°C it isomerizes to pyrazole (Vb). The recyclization of salt (If) is completed directly by formation of pyrazole (Va), whereas intermediate (IVb) could not be separated. This interesting case of easy acyl migration from nitrogen to oxygen is apparently due to the tendency of the pyrazolone system to undergo aromatization.

Compounds (IVa) and (Va, b) are crystalline orange-brown materials. Their structures were established on the basis of spectral data. The IR spectrum of (IVa), which we identified as 1,5-diphenyl-2-thiobenzoyl-4-pyrazolin-3-one, clearly shows a carbonyl band at 1750 cm^{-1} . Its PMR spectrum shows a singlet at 5.66 ppm that is assigned to the 4-H proton of the pyrazoline ring, and aromatic proton signals as a multiplet with center at 7.33 ppm. The ratio of the integrated intensities of these signals is 1:15. Additional confirmation of the structure of pyrazolone (IVa) is its conversion by acid hydrolysis to 1,5-diphenylpyrazolone-3 [8, p. 1041].

The IR spectra of pyrazoles (Va, b) contain aromatic ring bands in the 1610-1590 and 1510 cm^{-1} regions, and C-O-C bands in the 1230-1205 cm^{-1} region (see Experimental). The PMR spectra of (Va, b) show only multiplet signals with center at 7.4 ppm that belong to the protons of the phenyl substituents and the 4-H proton of the pyrazole nucleus.

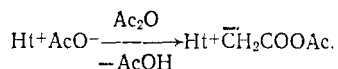
For comparison we studied other 6π -isoelectronic heterocyclic salts in an analogous reaction. Here 2,4,6-triphenylpyrilium perchlorate (VIa) when heated in acetic anhydride with potassium acetate recyclizes to the known 1,3,5-triphenylbenzene (VII) [9, p. 885]:



1,2,4,6-Tetraphenylpyridinium perchlorate did not react after 20 min boiling in acetic anhydride with potassium acetate. Here the reaction is apparently hindered for steric reasons; under the same conditions 1-methylpyridinium iodide is quite reactive, but forms dark-colored resinous products that we were unable to identify.

We would like to turn our attention to an interesting circumstance during the recyclization of heteroarene salts of weak acids such as alkanooates, specifically the way in which a C-nucleophile is generated from anhydride. The recyclization of heteroarene salts Ia, c-f, and VI perchlorates shows that at the start of the reaction of equimolar amounts of heteroarene perchlorate and base (KOAc), potassium perchlorate separates from the solution, i.e., there is a fast exchange of anions, and naturally heteroarene salt Ig, i-l or VIb alkanooate is left in solution. Under such conditions the C-nucleophile should be generated from the anhydride autocatalytically, due to the alkanooate counter ions of the substrate itself (as free counter ions

or bound in ion pairs: predominantly separate, less probably closely bound, much less probably at HtOAc type adducts):

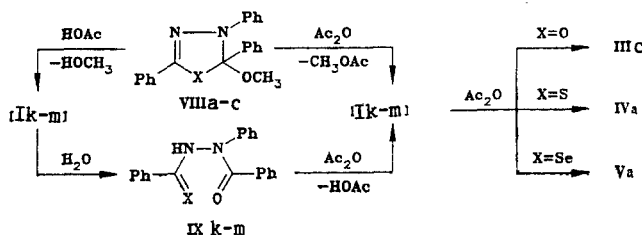


When a tertiary amine is present as generating agent of C-nucleophile, the alkanoate counter ion, which forms by reaction of tertiary amine with anhydride, $[\text{Et}_3\text{NAC}]^+ \text{AcO}^-$, also takes part.

In this way, in our opinion, these recyclizations of heteroarenium salts under the action of anhydride are different from recyclization by reaction with other C-nucleophiles, such as malonates, cyanoacetates, and β -ketoesters, which take place in the presence of a twofold or threefold excess of tertiary amine or alcoholate in acetonitrile, ether, or alcohol [5]. The latter reactions, as follows from the procedures of [5], are distinctly slower. This is completely understandable: under these conditions first the cryptosalts Ht-OR or $\text{Ht-NEt}_3\text{X}$ form quickly; subsequent cycloconversion in a weakly polar medium can take place as a replacement of OR or NEt_3 in these substrates by C-nucleophile (generated by excess base), but not as a direct addition of the latter to heteroarenium cation.

With cryptosalts VIIIa-c (Scheme 2), separately synthesized (see Experimental Part), as examples, we showed that in acetic anhydride they undergo conversion just like the respective perchlorates in acetic anhydride with potassium acetate (but slower); oxadiazoline (VIIIa) is converted to triacylhydrazine IIIc (analogous to the corresponding oxadiazolinium perchlorate [6]), while thia- and selenadiazolines VIIIb, c are converted to derivatives of pyrazole IVa and Va respectively. It can be assumed that these recyclizations proceed via the intermediate heteroarenium acetates Ik-m, which are converted in excess anhydride to the final reaction products as shown in the discussion of Scheme 1. However these compounds (IIIc, IVa, Va) are obtained from hydrazines (IXa-c) in acetic anhydride apparently also via the respectively acetates (Ik-m).

Scheme 2



Im. IIIc, VIIIa, IXa X=O; VIIIb, IXb X=S; VIIIc IXc X=Se

Apparently by the action of acetic acid on cryptosalts VIIIa-c acetates Ik-m are formed; these are then slowly converted to hydrazines IXa-c by water which has separated by esterification of acetic acid by alcohol, catalyzed by the heteroarenium salt (Scheme 2).

Recyclization of heteroarenium salts such as Ia-f and VIa begins with the reaction of C-nucleophile at the heteronucleus $\text{C}(2)$; according to [10] this atom is distinguished by the highest residual positive charge of all the ring carbons, and is always the center of primary nucleophilic attack in recyclization. Then apparently the hetero ring opens to form intermediates Xa-f or XI respectively. Here the generality of these cycloconversions for five-membered and six-membered rings is apparently limited. The new cycloformation in these intermediates is determined by the participation of a different kind of nucleophilic center; in the first case, nucleophilic center Y (CH^- in Xa-d or N^- in Xe, f); in the second case, CH_2 groups (CH^- in XI). With detachment of water (XIIa-d) or acid (XIIe, f, XIII) these intermediates go over to the final products, viz., to pyrrols IIa-d, pyrazoles IVa, Va, and benzene derivatives VII respectively. The interesting feature of the recyclization of pyrilium salt (VI) by reaction with anhydride is that in the last step aromatization of ring of XIII is accompanied by scission of a C-C bond (with detachment of carboxyacetate, see Scheme 2). But this does not exclude another possible path, viz., detachment of water to form the corresponding functional benzene derivative; this might agree with the known cases [11] of recyclization by the action of other C-nucleophiles.

EXPERIMENTAL

IR spectra were obtained in mineral oil with a UR-20 instrument; PMR spectra, in CCl₄ with a Tesla BS-487 instrument (60 MHz), with TMS internal standard.

2,3,5-Triphenyl-1,3,4-thiadiazolium perchlorate (Ie) was synthesized according to [12]; 2,3,5-triphenyl-1,3,4-selenadiazolium perchlorate (If) and 5-methoxy-2,4,6-triphenyl- Δ^2 -1,3,4-selenadiazolium perchlorate (VIIIc), according to [1]; pyrilium salts (VIa) and pyridinium salts, according to [13, 14].

3-Methyl-5-phenyl-2-(4-biphenyl)oxazolium perchlorate (Ia) was obtained from the respective tosylate (Ib) (synthesized according to [15]) by the action of HClO₄ in acetic acid. Yield 97%. mp 246-247°C (dec., from acetic acid).

2,3,5-Triphenyloxazolium perchlorate (Ic) was obtained from phenacylaniline by benzoylation in pyridine and acid cyclization as for other oxazolium salts [16]. Yield 90%. mp 237-239°C (dec., from ethanol).

2,5-Diphenyl-3-(3-tolyl)oxazolium perchlorate (Id) was obtained analogously to compound (Ic). Yield 85%. mp 235-236°C (dec., from acetic acid).

1-Methyl-3-acetoxy-5-(4-biphenyl)-2-benzoylpyrrol (IIa). Potassium acetate, 0.2 g, (2 mmole) was dissolved in 80 ml of acetic anhydride with heating; to the hot solution was added 0.8 g (2 mmole) of Ia perchlorate. The reaction mixture was boiled for 20 min. Acetic anhydride was evaporated in vacuum, and 20 ml of benzene was added to the residue and heated to boiling. Potassium perchlorate was filtered off (0.28 g, quantitative yield). The benzene filtrate was evaporated, and the residue was washed with 3 ml of ether and recrystallized from ethanol. Yield 0.5 g (63%). mp 146-147°C. IR spectrum: 1760 (C=O), 1625 cm⁻¹ (C=O). PMR spectrum: 1.50 (3H, s, CH₃), 3.83 (3H, s, CH₃), 6.03 (1H, s, 4-H), 7.46 ppm (14H, m, aromatic protons). Found: C 79.1; H 5.2; N 3.5%. C₂₆H₂₁NO₃. Calculated: C 79.0; H 5.3; N 3.5%.

1,4-Dimethyl-3-propionyloxy-5-(4-biphenyl)-2-benzoylpyrrol (IIb). KOH, 0.15 g (2 mmole), was dissolved in 50 ml of propionic anhydride with heating. To the hot solution was added 1 g (2 mmole) of 3-methyl-5-phenyl-2-(4-biphenyl)oxazolium tosylate (Ib). The mixture was boiled for 2 h, and the anhydride was distilled off in vacuum. The reaction product was extracted from the residue with 10 ml of hot benzene, and the solvent was evaporated. The solid was washed with 3 ml of ether and crystallized from aqueous alcohol. Yield 0.2 g (23%). mp 126-127°C. IR spectrum: 1760 (C=O), 1615 cm⁻¹ (C=O). PMR spectrum: 0.93 (3H, t, CH₃), 1.73 (2H, m, CH₂), 2.00 (3H, s, CH₃), 3.73 (3H, s, CH₃), 7.50 ppm (14H, m, aromatic protons). Found: C 79.4; H 5.7; N 3.4%. C₂₈H₂₅NO₃. Calculated: C 79.4; H 5.9; N 3.3%.

3-Acetoxy-1,5-diphenyl-2-benzoylpyrrol (IIc) was obtained analogously to compound (IIa) from 0.8 g (2 mmole) of Ic perchlorate, 0.2 g (2 mmole) of potassium acetate, and 80 ml of acetic anhydride. Yield 0.2 g (26%). mp 167-168°C (from alcohol). IR spectrum: 1750 (C=O), 1625 cm⁻¹ (C=O). PMR spectrum: 1.53 (3H, s, CH₃), 6.00 (1H, s, 4-H), 6.83 ppm (15H, m, aromatic protons). Found: C 78.6; H 4.8; N 3.4%. C₂₅H₁₉NO₃. Calculated C 78.7; H 5.0; N 3.7%.

3-Acetoxy-5-phenyl-1-(3-tolyl)-2-benzoylpyrrol (IIId) was obtained analogously to compound (IIa) from 2 g (5 mmole) of Id perchlorate and 0.5 g (5 mmole) of potassium acetate in 120 ml of acetic anhydride. Yield 0.2 g (10%). mp 123-124°C (from alcohol). IR spectrum: 1760 (C=O), 1630 cm⁻¹ (C=O). PMR spectrum: 1.60 (3H, s, CH₃), 2.27 (3H, s, CH₃), 6.27 (1H, s, 4-H), 7.10 ppm (14H, m, aromatic protons). Found: C 79.1; H 5.2; N 3.5%. C₂₆H₂₁NO₃. Calculated 79.0; H 5.3; N 3.5%.

1-Acetyl-2-phenyl-1,2-dibenzoylhydrazine (IIIc). Compound VIIIa, 0.33 mole (1 mmole) (for synthesis see below) was boiled with 5 ml of acetic anhydride for 3 h. The acetic anhydride was then boiled off, 2 ml of methanol was added to the residue, and the resulting crystals were filtered off. Yield 0.15 g (42%). mp 140-145°C (from methanol). The melting point of a mixture with a sample obtained as in [6] was not depressed.

1,5-Diphenyl-2-thiobenzoyl-4-pyrazolin-3-one (IVa). A). Perchlorate (Ie), 0.2 g (0.5 mmole), was dissolved in 10 ml of acetic anhydride; to the solution a separately prepared hot solution of 0.05 g (0.5 mmole) of potassium acetate in 20 ml of acetic anhydride was added in two portions. The mixture was boiled for 5 min. The potassium perchlorate precipitate was filtered off, the filtrate was evaporated in vacuum, and 3 ml of methanol was added to the residue. The resulting crystals were filtered off, washed with hexane, and dried. Yield

0.15 g (83%). mp 162-165°C (dec., from methanol). IR spectrum: 1590, 1615 (C=C), 1750 cm⁻¹ (C=O). PMR spectrum: 5.66 (1H, s, 4-H); 7.33 ppm (15H, m, aromatic protons). Found: C 74.3; H 4.7; N 8.1; S 8.6%. C₂₂H₁₆N₂OS. Calculated: C 74.2; H 4.5; N 7.9; S 9.0%.

B). Compound (VIIIb), 0.35 g (1 mmole) (for synthesis see below), was dissolved with heating in 4 ml of acetic anhydride and boiled for 1.5 h. Acetic anhydride was evaporated in vacuum. To the residue was added 3 ml of methanol, the precipitated crystals were filtered off and dried. Yield 0.07 g (19%). mp 162-165°C (with decomposition).

C). Hydrazine IXb, 0.33 g (1 mmole) (for synthesis see below), was boiled with 5 ml of acetic anhydride for 1 h. Acetic anhydride was evaporated in vacuum. To the residue was added 2 ml of methanol and the resulting crystals were filtered off and dried. Yield 0.2 g (56%). mp 162-165°C (dec., from methanol).

The melting points of mixtures of samples obtained by methods A), B), and C) were not depressed.

1,5-Diphenylpyrazolone-3. Compound IVa, 0.1 g (0.28 mmole) was dissolved in 2 ml of ethanol, 2 ml of conc. HCl was added, and the mixture was boiled for 1 h. Hydrogen sulfide was evolved. After cooling the mixture was diluted with water to 20 ml, and the precipitate was filtered off and dried. Yield 0.05 g (75%). mp 254-255°C (purified by vacuum distillation). According to [8], mp is 255-256°C.

1,5-Diphenyl-3-selenobenzoyloxy pyrazole (Va). Potassium acetate, 0.2 g (2 mmole), was dissolved in 80 ml of acetic anhydride. To the hot solution was added 0.9 g (2 mmole) of compound (If). The mixture was boiled for 15 min. Potassium perchlorate separated from the solution; it was filtered off; KClO₄ yield 0.2 g (72%). The filtrate was evaporated in vacuum; the residue was extracted with 10 ml of hot toluene. Precipitation with hexane gave brown crystals. Yield Va 0.25 g (31%). mp 223-224°C (dec., from ethanol). IR spectrum: 1600, 1580, 1565 (C=C, C=N), 1510 (C=N), 1335, 1225, 1210, 1180, 1130 cm⁻¹ (C-O-C). PMR spectrum: 7.40 ppm (m, aromatic protons). Found: C 66.1; H 4.2; N 7.1; Se 19.0%. C₂₂H₁₆N₂OSe. Calculated C 65.5; H 4.0; N 6.9; Se 19.6%.

1,5-Diphenyl-3-thiobenzoyloxy pyrazole (Vb). Compound IVa, 0.2 g (0.6 mmole), was heated for 10 min at 180°C. After cooling the melt was crystallized from 2:1 benzene-hexane. Yield 0.05 g (25%). mp 223-225°C. IR spectrum: 1610, 1590, 1575 (C=C, C=N), 1510 (C=N), 1340, 1230, 1210, 1180, 1140 cm⁻¹ (C-O-C). PMR spectrum: 7.40 ppm (m, aromatic protons). Found: C 74.2; H 4.5; N 8.1; S 8.7%. C₂₂H₁₆N₂OS. Calculated: C 74.2; H 4.5; N 7.9; S 9.0%.

1,3,5-Triphenylbenzene (VII) was obtained analogously to compound (IIa) from 2.0 g (5 mmole) of potassium acetate, and 125 ml of acetic anhydride by boiling for 1 h. Yield 0.55 g (36%). mp 170-172°C (from acetic acid). According to [9], mp 172°C. Mixed melting point with an authentic sample gave no depression.

5-Methoxy-2,4,5-triphenyl-Δ²-1,3,4-oxadiazoline (VIIIa) was obtained analogously to compound (VIIIc) [1] from 2,3,5-triphenyl-1,3,4-oxadiazolinium perchlorate (synthesized as in [17]) and sodium methylate. Yield 80%. mp 93-94°C (from methanol). Found: N 8.7%. C₂₁H₁₈-N₂O₂. Calculated: N 8.5%.

5-Methoxy-2,4,5-triphenyl-Δ²-1,3,4-thiadiazoline (VIIIb) was obtained from perchlorate Ie and sodium methylate analogously to (VIIIa). Yield 98%. mp 119-120°C (from hexane). Found N 7.9%. C₂₁H₁₈N₂OS. Calculated: N 8.1%.

1-Phenyl-1,2-dibenzoylhydrazine (IXa). Compound VIIIa, 0.33 g (1 mmole) was dissolved in 4 ml of acetic acid with stirring; after 2 h the precipitate was filtered off. Yield 0.25 g (66%). mp 175-178°C. Mixed melting point with a known sample [18] gave no depression.

1-Phenyl-1-benzoyl-2-thiobenzoylhydrazine (IXb). With heating, 0.35 of compound VIIIb (1 mmole) was dissolved in 5 ml of acetic acid. The solution was kept at 20°C for 17 h. The mixture turned yellow and a precipitate formed. The desired material was precipitated with 50 ml of water and filtered off. Yield 0.3 g (90%). mp 168-170°C (dec., from ethanol). Mixed melting point with a known sample [19] gave no depression.

1-Phenyl-1-benzoyl-2-selenobenzoylhydrazine (IXc) was obtained analogously to hydrazine (IXb) from 0.25 g (0.6 mmole) of compound VIIIc in 5 ml of acetic acid. Yield 0.24 g (99%). mp 120-125°C (dec., from benzene-hexane). IR spectrum: 1650 (C=O), 3140 cm⁻¹ (NH). Found: N 7.3%. C₂₀H₁₆N₂OSe. Calculated: N 7.4%.

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