Selenium-Containing Heterocycles from Isoselenocyanates: Cycloaddition of Carbodiimides to Selenazetidines

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The reactions of isoselenocyanates with carbodiimides in refluxing hexane afforded 1,3-selenazetidine-2,4-diimines **3a**–**3h** by a [2 + 2] cycloaddition in moderate-to-good yields. The molecular structure of **3a** has been established by X-ray crystallography.

Introduction. – In recent years, many exciting research results have indicated that selenium (Se) is a very important element, which has attracted the attention of scientists working in a variety of fields. In molecular biology, the study of Se is adding a new and unexpected chapter to the story of the genetic code and its translation. In biochemistry, selenoproteins are being recognized as the mediators of the biological effects of the element, and new selenoproteins are being studied. Finally, in medicine and public health, Se has implications that range from effects on heart disease to relationships with cancer. For this reasons, organoselenium chemistry has undergone a spectacular mutation during the last fifteen years: from an exotic area of science practiced by a few specialists it became a relatively well-mastered and widely used methodology by synthetic organic chemists. The key to this success is that a fair number of Se-based reagents and reactions have been discovered, which are able to perform specific transformations selectively and often under very mild conditions. The interest in Se-containing compounds has increased not only because of their reactivities and chemical properties [1–7] but also because of their pharmaceutical applications [8–14]. Organoselenium compounds have proven to be an important class of biologically active products [15][16] as antioxidants [15], antibacterial agents [17], and catalysts, which mimic the activity of glutathione peroxydase (GP_x) [18]. *Shamberger* [19] widely evidenced the important utility of Se in biology and in human health [20], in cancer chemoprevention [21], in foods (garlic [22], cereals [23], and potatoes [24]), and plants [25][26].

On the other hand, syntheses of Se-containing heterocycles often involve the use of toxic Se reagents, which are often difficult to handle and to store. Isoselenocyanates have less unpleasant chemical properties [27–29] and are very useful as starting materials in heterocyclic chemistry [30–32] since they can easily be prepared [33] and safely stored and handled. Therefore, they are reagents of choice for the preparation of selenoformamides [34], selenocarbonic acids [35], selenazoles [36], semicarbazides [37], and carbodiimides [38].

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Though syntheses of S-containing heterocyclic compounds have been studied extensively, those of Se analogues have not been investigated appreciably, and a comparison of the reactivity and properties of Se-containing heterocyclic systems with the S-containing analogues is still a challenging subject. The larger size of the Se-atom could explain why Se-containing heterocycles show an increased polarizability and, therefore, that they are, in general, less stable than their sulfur analogues.

Very few four-membered Se-containing heterocycles are known [4][32c]. To the best of our knowledge, only three papers deal with N,Se heterocyclic systems [39][40]. For this reason, we focused our attention on the synthesis of selenazetidine derivatives. In the present paper, we report on the easy preparation of 1,3-selenazetidine-2,4-diimines from isoselenocyanates and carbodiimides. The analogous reaction of phenyl isothiocyanate to give thiazetidine derivatives has already been described [41–43].

Results and Discussion. – A series of aromatic and aliphatic isoselenocyanates **1** were prepared according to [33]. The reactions with equimolar amounts of dicyclohexyl and diisopropyl carbodiimide (**2a** and **2b**, resp.) were carried out in refluxing hexane and gave the corresponding 1:1 adducts **3** in good-to-excellent yields (*Scheme*, *Table 1*). In general, the crude products were purified by chromatography (SiO_2, hexane) and recrystallization. All products were stable and could be stored at room temperature.

Scheme

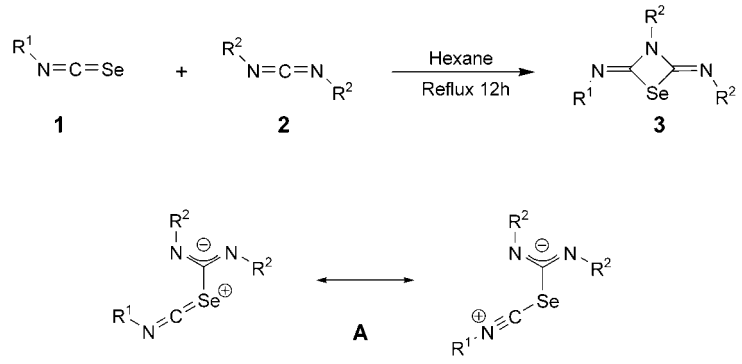

Table 1. *Preparation of Selenazetidines* **3a**–**3h** *from Isoselenocyanates* **1**

Compound	R^1	R^2	Yield [%]
3a	Ph	Cyclohexyl	98
3b	Ph	i-Pr	84
3c	4-ClC_6H_4	Cyclohexyl	88
3d	4-ClC_6H_4	i-Pr	97
3e	4-BrC_6H_4	Cyclohexyl	98
3f	4-BrC_6H_4	i-Pr	99
3g	Cyclohexyl	Cyclohexyl	99
3h	Cyclohexyl	i-Pr	88

Based on the elemental analyses and spectroscopic data, the structure of the formal [2+2] cycloadducts, *i.e.*, 1,3-selenazetidine-2,4-diimines **3**, was assigned to the products. For example, in the IR spectrum (KBr) of **3a**, strong absorptions for the C=N groups appear at *ca.* 1690 and 1674 cm^{-1}. The ^{13}C-NMR spectrum ($CDCl_3$) shows two signals for C=N at 133.1 and 137.1 ppm, and in the CI-MS (NH_3), *m/z* 390 ($[M+1]^+$) and 207 ($[(C_6H_{11})_2CN_2]^{+\cdot}$) are characteristic. On the other hand, no information concerning the relative configuration of the C=N groups could be obtained. Therefore, the molecular structure of **3a** was established by X-ray crystallography (*Figure*).

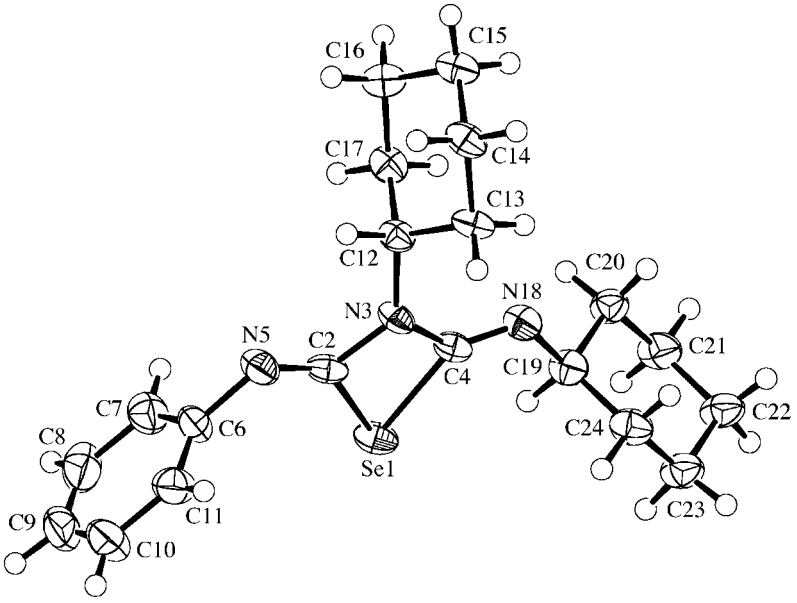

Fig. 1. ORTEP *Plot* [44] *of the molecular structure of* **3a** (arbitrary numbering of the atoms, 50% probability ellipsoids)

Both imino groups of **3a** are (*Z*)-configured. The selenazetidine is planar with the maximum deviation from the four-atom plane being 0.010(4) Å for N(3). The planarity extends through both imino groups to the *ipso*-C-atoms of the attached rings with the maximum deviation from the plane defined by these eight atoms being 0.036(5) Å for C(6). In contrast, C(12), which is attached to N(3), lies 0.327(5) Å from the plane of the selenazetidine ring, which demonstrates significant pyramidalization at N(3) and, therefore, only weak conjugation between N(3) and the imino groups. The sum of the bond angles at N(3) is 357.3°. The Ph ring at N(5) makes an angle of 73.2(3)° with the plane of the heterocyclic ring, thereby precluding conjugation between the Ph and C=N groups. This is also reflected by the length of the N(5)–C(6) bond (1.422(6) Å). The inner-annular angles of the selenazetidine ring are characteristic for four-membered Se-heterocycles (*cf.* [32c]): C(2)–Se(1)–C(4) 67.4(2)°, Se(1)–C(2)–N(3) 94.9(3)°, Se(1)–C(4)–N(3) 92.9(3)°, and C(2)–N(3)–C(4) 104.8(4)°. The two cyclohexane rings show the expected chair conformations.

By analogy to the structure of **3a**, we propose that **3b**–**3f**, which each bear an arylimino group, also have the (*Z,Z*)-configuration. However, the 'symmetrically substituted' **3g**, with two cyclohexylimino moieties, shows three signals for CH and twelve signals for CH_2 groups of the cyclohexyl substituents. Therefore, the structure cannot be symmetric. Furthermore, the relative intensities of the ^{1}H *multiplets* at δ 2.45–2.60 and 3.15–3.24 ppm in different batches vary from 2:1 to 1:3. A similar observation was made with respect to the ^{13}C-NMR signals at δ 55.6 and 55.7/65.7 ppm. A likely interpretation is that mixtures of (*Z,Z*)- and (*E,Z*)-**3g** are present in varying ratios. A similar observation has been made in the case of **3h**, which, in the NMR-spectra, also shows some doubling of signals.

Although compounds **3** are formal [2 + 2] cycloadducts, it is most likely that a two-step mechanism is responsible for their formation: the nucleophilic attack of the Se-atom at the carbodiimide C-atom leads to a zwitterion of type **A**, which, *via* ring closure, yields the final product (*Scheme*).

We thank the analytical units of our institute for spectra and analyses. Financial support of this work by the *Dr. Helmut Legerlotz Foundation* and *F. Hoffmann-La Roche AG,* Basel, is gratefully acknowledged.

Experimental Part

General. See [31a]. TLC: silica gel *60 F_{254}* plates (0.25 mm; *Merck*). Column chromatography (CC): silica gel *60* (0.040–0.063 mesh; *Merck*). M.p.: *Büchi B-540* apparatus, in capillaries; uncorrected. ^{1}H-NMR (300 MHz) and ^{13}C-NMR (75.5 MHz) spectra: *Bruker ARX-300* instrument, in $CDCl_3$ at 300 K, unless otherwise specified; chemical shifts δ in ppm, coupling constants *J* in Hz. EI-MS and CI-MS: *Finnigan SSQ-700* or *MAT-90* instrument; EI mode: 70 eV; CI mode: NH_3 as carrier gas.

Preparation of Selenazetidines. – General Procedure. A 50-ml round-bottomed flask equipped with a magnetic stirrer and a condenser was charged with a soln. of the respective isoselenocyanate **1** [33] (1.0 mmol) in hexane (15 ml). Then, carbodiimide **2** (1 equiv., 1.0 mmol) was added, and the stirred mixture was heated to reflux for 12 h. The mixture was cooled to r.t. and concentrated *in vacuo* to give the crude product **3**. The latter was then purified by chromatography on silica gel using hexane as eluant. Solid products were recrystallized from appropriate solvents.

N-*[3-Cyclohexyl-4-(cyclohexylimino)-1,3-selenazetidin-2-ylidene]benzenamine* (**3a**). Yield: 217 mg (98%). Colorless crystals. M.p. 90–92° (CH_2Cl_2). IR (KBr): 2935*m*, 2922*m*, 2852*m*, 1690*s* (br.), 1674*s* (br.), 1590*m*, 1486*w*, 1445*w*, 1364*w*, 1321*s*, 1272*w*, 1236*w*, 1165*w*, 1126*w*, 1053*w*, 894*w*, 772*w*, 693*w*, 614*w*. ^{1}H-NMR: 1.14–1.40 (*m*, 6 H); 1.46–1.66 (*m*, 4 H); 1.72–1.83 (*m*, 6 H); 1.96–2.01 (*m*, 2 H); 2.12–2.25 (*m*, 2 H); 2.55–2.64, 3.98–4.08 (2*m*, 2 CH); 7.00 (*d*, *J* = 7.5, 2 arom. H); 7.13 (*t*, *J* = 7.4, 1 arom. H); 7.33 (*t*, *J* = 7.4, 2 arom. H). ^{13}C-NMR: 24.4 (2 CH_2); 25.0 (CH_2); 25.4 (CH_2); 25.6 (2 CH_2); 30.1 (2 CH_2); 39.9 (2 CH_2); 56.5, 66.6 (2 CH); 120.9 (2 arom. CH); 124.6 (1 arom. CH); 129.1 (2 arom. CH); 133.1, 137.1, 147.8 (C(2), C(4), 1 arom. C). CI-MS: 390 (8, [*M* + 1]$^+$), 207 (100), 201 (18). Anal. calc. for $C_{20}H_{27}N_3Se$ (388.41): C 61.85, H 7.01, N 10.82; found: C 61.57, H 6.77, N 10.68.

Crystals suitable for the X-ray crystal-structure determination were grown from CH_2Cl_2 by slow evaporation of the solvent.

N-*[3-Isopropyl-4-(isopropylimino)-1,3-selenazetidin-2-ylidene]benzenamine* (**3b**). Yield: 259 mg (84%). Yellow oil. IR (neat): 2970*m*, 2933*w*, 2828*w*, 1694*s*, 1675*s*, 1593*s*, 1488*w*, 1455*w*, 1318*s*, 1264*w*, 1193*w*, 1169*w*, 1131*w*, 1060*w*, 837*w*, 805*w*, 759*m*, 708*w*, 693*m*. ^{1}H-NMR: 1.19 (*d*, *J* = 6.3, 2 Me); 1.51 (*d*, *J* = 6.3, 2 Me); 2.86, 4.36 (2*sept.*, *J* = 6.8, 2 CH); 7.01 (*d*, *J* = 7.2, 2 arom. H); 7.11 (*t*, *J* = 7.4, 1 arom. H); 7.28 (*t*, *J* = 7.3, 2 arom. H). ^{13}C-NMR: 20.2 (2 Me); 24.0 (2 Me); 48.7, 59.2 (2 CH); 120.9 (2 arom. CH); 124.6 (1 arom. CH); 129.1 (2 arom. CH); 133.0, 136.6, 147.8 (C(2), C(4), 1 arom. C). CI-MS: 310 (57, [*M* + 1]$^+$), 230 (7), 161 (41), 127 (100). Anal. calc. for $C_{14}H_{19}N_3Se$ (308.28): C 54.54, H 6.21, N 13.63; found: C 54.53, H 5.95, N 13.58.

4-Chloro-N-*[3-cyclohexyl-4-(cyclohexylimino)-1,3-selenazetidin-2-ylidene]benzenamine* (**3c**). Yield: 372 mg (88%). Yellowish crystals. M.p. 82–84° (CH_2Cl_2). IR (KBr): 2930*s*, 2852*m*, 1680*s* (br.), 1675*s* (br.), 1588*m*, 1486*s*, 1446*m*, 1364*w*, 1319*s*, 1258*w*, 1237*w*, 1164*m*, 1089*m*, 1052*w*, 1010*w*, 959*w*, 886*w*, 828*m*, 683*w*, 642*w*. ^{1}H-NMR: 1.13–1.37 (*m*, 6 H); 1.47–1.65 (*m*, 4 H); 1.71–1.83 (*m*, 6 H); 1.93–1.98 (*m*, 2 H); 2.09–2.23 (*m*,

2 H); 2.53–2.62, 3.90–4.00 (2*m*, 2 CH); 6.94 (*AA′*, *J* = 7.4, 2 arom. H); 7.26 (*BB′*, *J* = 7.4, 2 arom. H). ^{13}C-NMR: 24.3 (2 CH_2); 25.0, 25.4 (2 CH_2); 25.6 (2 CH_2); 30.1 (2 CH_2); 39.9 (2 CH_2); 56.5, 66.6 (2 CH); 122.2 (2 arom. CH); 129.2 (2 arom. CH); 129.9, 132.3, 137.9, 146.5 (C(2), C(4), 2 arom. C). CI-MS: 424 (1, $[M+1]^+$), 235 (16), 207 (100). Anal. calc. for $C_{20}H_{26}ClN_3Se$ (422.85): C 56.81, H 6.20, N 9.94; found: C 56.74, H 6.10, N 9.81.

4-Chloro-N-*3-[isopropyl-4-(isopropylimino)-1,3-selenazetidin-2-ylidene]benzenamine* (**3d**). Yield: 332 mg (97%). Yellow oil. IR (neat): 2971*s*, 2934*m*, 2870*w*, 2828*w*, 1690*s* (br.), 1672*s* (br.), 1589*s*, 1487*s*, 1456*m*, 1365*m*, 1320*s*, 1265*m*, 1193*m*, 1167*m*, 1130*m*, 1091*m*, 844*s*, 830*m*, 727*w*. ^{1}H-NMR: 1.20 (*d*, *J* = 6.4, 2 Me); 1.50 (*d*, *J* = 6.3, 2 Me); 2.87, 4.33 (2*sept.*, *J* = 6.8, 2 CH); 6.95 (*AA′*, *J* = 7.2, 2 arom. H); 7.27 (*BB′*, *J* = 7.4, 2 arom. H). ^{13}C-NMR: 21.1 (2 Me); 24.0 (2 Me); 48.8, 59.2 (2 CH); 122.2 (2 arom. CH); 129.2 (2 arom. CH); 130.0, 132.2, 137.5, 146.4 (C(2), C(4), 2 arom. C). CI-MS: 344 (100, $[M+1]^+$), 264 (7), 195 (33), 127 (65). Anal. calc. for $C_{14}H_{18}ClN_3Se$ (342.73): C 49.06, H 5.29, N 12.26; found: C 49.31, H 5.21, N 12.26.

4-Bromo-N-*[3-cyclohexyl-4-(cyclohexylimino)-1,3-selenazetidin-2-ylidene]benzenamine* (**3e**). Yield: 458 mg (98%). Colorless plates. M.p. 78–80° (CH_2Cl_2). IR (KBr): 2933*m*, 2852*m*, 1677*s* (br.), 1580*m*, 1483*m*, 1449*m*, 1367*w*, 1344*w*, 1325*s*, 1260*w*, 1236*w*, 1168*w*, 1069*w*, 1008*w*, 891*w*, 848*w*, 812*w*, 722*w*, 706*w*. ^{1}H-NMR: 1.20–1.38 (*m*, 6 H); 1.43–1.62 (*m*, 4 H); 1.66–1.83 (*m*, 6 H); 1.94–1.98 (*m*, 2 H); 2.09–2.22 (*m*, 2 H); 2.53–2.61, 3.98–4.08 (2*m*, 2 CH); 6.89 (*AA′*, *J* = 8.6, 2 arom. H); 7.41 (*BB′*, *J* = 8.6, 2 arom. H). ^{13}C-NMR: 24.4 (2 CH_2); 25.0, 25.4 (2 CH_2); 25.6 (2 CH_2); 30.1 (2 CH_2); 39.9 (2 CH_2); 56.5, 66.6 (2 CH); 120.9 (2 arom. CH); 124.6 (1 arom. C); 129.1 (2 arom. CH); 133.1, 137.1, 147.8 (C(2), C(4), 1 arom. C). CI-MS: 468 (6, $[M+1]^+$), 207 (100), 225 (13), 281 (6). Anal. calc. for $C_{20}H_{26}BrN_3Se$ (467.30): C 51.40, H 6.61, N 8.99; found: C 51.63, H 5.66, N 9.05.

4-Bromo-N-*[3-isopropyl-4-(isopropylimino)-1,3-selenazetidin-2-ylidene]benzenamine* (**3f**). Yield: 383 mg (99%). Yellowish crystals. M.p. 49–51° (hexane). IR (KBr): 2974*w*, 2933*w*, 1686*s*, 1674*s*, 1582*w*, 1482*m*, 1388*w*, 1320*s*, 1264*m*, 1189*w*, 1170*w*, 1126*m*, 1066*w*, 1007*w*, 843*s*, 828*m*, 699*w*. ^{1}H-NMR: 1.20 (*d*, *J* = 6.4, 2 Me); 1.50 (*d*, *J* = 6.3, 2 Me); 2.88, 4.31 (2*sept.*, *J* = 6.8, 2 CH); 6.89 (*AA′*, *J* = 7.2, 2 arom. H); 7.42 (*BB′*, *J* = 7.4, 2 arom. H). ^{13}C-NMR: 20.1 (2 Me); 24.0 (2 Me); 48.8, 59.2 (2 CH); 117.7 (1 arom. C); 122.6 (2 arom. CH); 132.1 (2 arom. CH); 129.9, 137.5, 146.5 (C(2), C(4), 1 arom. C). CI-MS: 388 (14 $[M+1]^+$), 241 (16), 127 (100). Anal. calc. for $C_{14}H_{18}BrN_3Se$ (387.18): C 43.43, H 4.69, N 10.85; found: C 43.54, H 4.52, N 10.63.

Cyclohexyl-N-*[3-cyclohexyl-4-(cyclohexylimino)-1,3-selenazetidin-2-ylidene]benzenamine* (**3g**). Yield: 390 mg (99%). Yellowish oil. IR (neat): 2927*s*, 2856*s*, 1688*s* (br.), 1651*s* (br.), 1592*m*, 1488*w*, 1449*m*, 1363*m*, 1318*s*, 1259*m*, 1085*w*, 1049*w*, 1026*w*, 976*w*, 909*m*, 892*m*, 804*w*, 764*w*, 733*s*, 695*w*, 615*m*. ^{1}H-NMR (*ca.* 2:1 mixture of isomers[2]): 1.16–1.58 (*m*, *ca.* 18 H); 1.70–1.75 (*m*, *ca.* 8 H); 1.89–1.94 (*m*, *ca.* 4 H); 2.00–2.15 (*m*, *ca.* 1 H); 2.45–2.60 (*m*, *ca.* 1 H); 3.15–3.24 (*m*, *ca.* 0.5 H); 3.75–3.85 (*m*, *ca.* 1 H). ^{13}C-NMR[2]): 22.9 (2 CH_2); 24.4 (2 CH_2); 24.5 (2 CH_2); 24.8 (2 CH_2); 25.1 (2 CH_2); 25.3 (2 CH_2); 25.5 (2 CH_2); 29.9 (2 CH_2); 32.6 (CH_2); 33.8 (CH_2); 34.0 (CH_2); 34.8 (CH_2); 55.5 (CH); 55.7 (CH); 55.8 (CH); 66.6 (CH); 132.5, 139.6 (C(2), C(4)). CI-MS: 396 (5, $[M+1]^+$), 207 (100). Anal. calc. for $C_{20}H_{33}N_3Se$ (394.46): C 69.90, H 8.43, N 10.65; found: C 69.58, H 8.15, N 10.59.

Cyclohexyl-N-*[3-isopropyl-4-(isopropylimino)-1,3-selenazetidin-2-ylidene]benzenamine* (**3h**). Yield: 277 mg (88%). Yellowish oil. IR (neat): 2933*s*, 2856*s*, 1694*s*, 1683*s*, 1450*m*, 1362*m*, 1350*m*, 1312*s*, 1128*w*, 1048*w*, 891*w*, 718*w*, 672*w*, 626*w*. ^{1}H-NMR (mixture of 2 isomers): 1.17 (*d*, *J* = 6.4, 2 Me); 1.25–1.50 (*m*, 8 H); 1.64–1.78 (*m*, 6 H); 1.88–1.95 (*m*, 2 H); 2.84 (*sept.*, *J* = 6.8, 1 CH); 3.76–3.83 (*m*, 1 H); 4.16 (*sept.*, *J* = 6.8, 1 CH). ^{13}C-NMR (mixture of 2 isomers): 14.0 (2 Me); 20.0 (2 Me); 22.9 (4 Me); 24.0 (2 CH_2); 24.4 (2 CH_2); 24.8 (2 CH_2); 25.5 (2 CH_2); 32.6 (CH_2); 34.0 (CH_2); 48.2 (CH); 55.8 (2 CH); 59.1 (CH); 66.5 (CH); 131.8, 132.7 (C(2), C(4)). CI-MS: 316 (100, $[M+1]^+$). Anal. calc. for $C_{14}H_{25}N_3Se$ (314.33): C 53.49, H 8.02, N 13.37; found: C 53.65, H 7.98, N 13.18.

X-Ray Crystal-Structure Determination of **3a** (see *Table 2* and *Fig.*[3]). All measurements were made on a *Nonius KappaCCD* diffractometer [45] using graphite-monochromated MoK_α radiation (λ 0.71073 Å) and an *Oxford Cryosystems Cryostream 700* cooler. Data reduction was performed with *HKL Denzo* and *Scalepack* [46]. The intensities were corrected for *Lorentz* and polarization effects, and an absorption correction based on the multi-scan method [47] was applied. Equivalent reflections were merged. Data collection and refinement

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[2]) The material from another reaction contained the isomers in a ratio of 1:3.

[3]) CCDC-256793 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge *via* www.ccdc.cam.ac.uk/conts/retrieving.html (or from the *Cambridge Crystallographic Data Centre*, 12 Union Road, Cambridge CB2 1EZ, U.K.; fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk).

parameters are given in *Table 2*, and a view of the molecule is shown in the *Fig.* The structure was solved by direct methods using SIR92 [48], which revealed the positions of all non-H-atoms. The non-H-atoms were refined anisotropically. All of the H-atoms were placed in geometrically calculated positions and refined using a riding model where each H-atom was assigned a fixed isotropic displacement parameter with a value equal to $1.2U_{eq}$ of its parent C-atom. Refinement of the structure was carried out on F^2 using full-matrix least-squares procedures, which minimized the function $\Sigma w(F_o^2 - F_c^2)^2$. A correction for secondary extinction was not applied. Neutral-atom scattering factors for non-H-atoms were taken from [49a], and the scattering factors for H-atoms were taken from [50]. Anomalous dispersion effects were included in F_c [51]; the values for f' and f'' were those of [49b]. The values of the mass attenuation coefficients are those of [49c]. All calculations were performed using the SHELXL97 [52] program.

Table 2. *Crystallographic Data of Compound* **3a**

Crystallized from	CH_2Cl_2
Empirical formula	$C_{20}H_{27}N_3Se$
Formula weight [g mol^{-1}]	388.35
Crystal color, habit	colorless, plate
Crystal dimensions [mm]	$0.02 \times 0.12 \times 0.12$
Temperature [K]	160(1)
Crystal system	orthorhombic
Space group	*Pbca*
Z	8
Reflections for cell determination	116658
2θ range for cell determination [°]	4–50
Unit cell parameters: a [Å]	13.1982(3)
b [Å]	9.9459(2)
c [Å]	29.1231(6)
V [Å^3]	3822.9(1)
D_x [g cm^{-3}]	1.349
μ(MoK_α) [mm^{-1}]	1.969
Scan type	ϕ and ω
$2\theta_{max}$ [°]	50
Transmission factors (min; max)	0.839; 0.968
Total reflections measured	63265
Symmetry independent reflections	3379
Reflections with $I > 2\sigma(I)$	2415
Reflections used in refinement	3379
Parameters refined	217
Final $R(F)$ [$I > 2\sigma(I)$ reflections]	0.0587
$wR(F^2)$ (all data)	0.1101
Weights: $w = [\sigma^2(F_o^2) + (0.0146P)^2 + 16.9985P]^{-1}$ where $P = (F_o^2 + 2F_c^2)/3$	
Goodness-of-fit	1.054
Final Δ_{max}/σ	0.001
$\Delta\rho$ (max; min) [e Å^{-3}]	1.35; −0.76

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