Novel Heterocyclic Systems. Part 28.[1] Preparation and Characterization of the 1,6-, 1,7-, 1,8-, and 1,9-Diazaphenoxaselenines: an Unexpected Divergence Between Closely Related Sulphur and Selenium Systems

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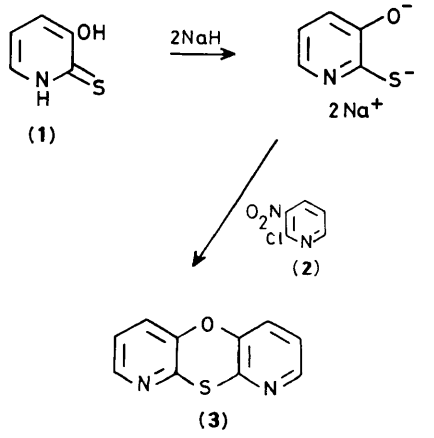
Preparations are reported of the parent members of four new heterocyclic systems, the 1,6-, 1,7-, 1,8-, and 1,9-diazaphenoxaselenines. Whereas the reaction of the dianion of 3-hydroxypyridine-2(1*H*)-thione with 2-chloro-3-nitropyridine gives only 1,9-diazaphenoxathiine, the corresponding reaction of the dianion of 3-hydroxypyridine-2(1*H*)-selone (**4**) gives a mixture of 1,6- and 1,9-diazaphenoxaselenines. Furthermore, the 1,6-isomer, presumably formed *via* a Smiles rearrangement which does not occur in the phenoxathiine series, predominates. Reaction of the dianion of (**4**) with 3-chloro-4-nitropyridine 1-oxide gives a mixture of the *N*-oxides of the 1,7- and 1,8-diazaphenoxaselenines, the 1,8-isomer predominating. This mixture is separated by flash-column chromatography and the individual isomers are reduced to their respective diazaphenoxaselenines with phosphorus trichloride.

As part of our ongoing research into the chemistry of tricyclic systems related to phenothiazines[2] we recently synthesized the first examples of the 1-azaphenoxaselenine† ring system.[3] In the phenoxathiine series the 1,7- and 1,9-diaza analogues have been reported previously,[4,5] and the 1,7-isomer showed significant central nervous system activity, comparable to that of chlorpromazine,[6] in a preliminary screen.[4] We have also recently prepared 1,8-diazaphenoxathiine,[7] but the methods tried to date have been unsuccessful for the synthesis of the 1,6-isomer. It was of interest to compare the corresponding selenium compounds and we now report the preparation and properties of the 1,6-, 1,7-, 1,8-, and 1,9-diazaphenoxaselenines. Surprisingly, the 1,6-isomer was obtained as the major product, along with a minor amount of the 1,9-isomer, by a reaction which gave solely the 1,9-isomer in the isosterically related phenoxathiine system.

Preparation of 1,6-*Diazaphenoxaselenine* (**6**) *and* 1,9-*Diazaphenoxaselenine* (**5**).—The synthesis of 1,9-diazaphenoxathiine (**3**) had previously been achieved regiospecifically by the reaction of the dianion of 3-hydroxypyridine-2(1*H*)-thione (**1**) with 2-chloro-3-nitropyridine (**2**) (Scheme 1).[4,5] Since the syntheses of 1-azaphenoxaselenines from 3-hydroxypyridine-2(1*H*)-selone (**4**) proceed smoothly[3] and in a manner very comparable to the syntheses[8] of the corresponding 1-azaphenoxathiines from (**1**), we expected to be able to synthesize 1,9-diazaphenoxaselenine (**5**) regiospecifically by an analogous route.

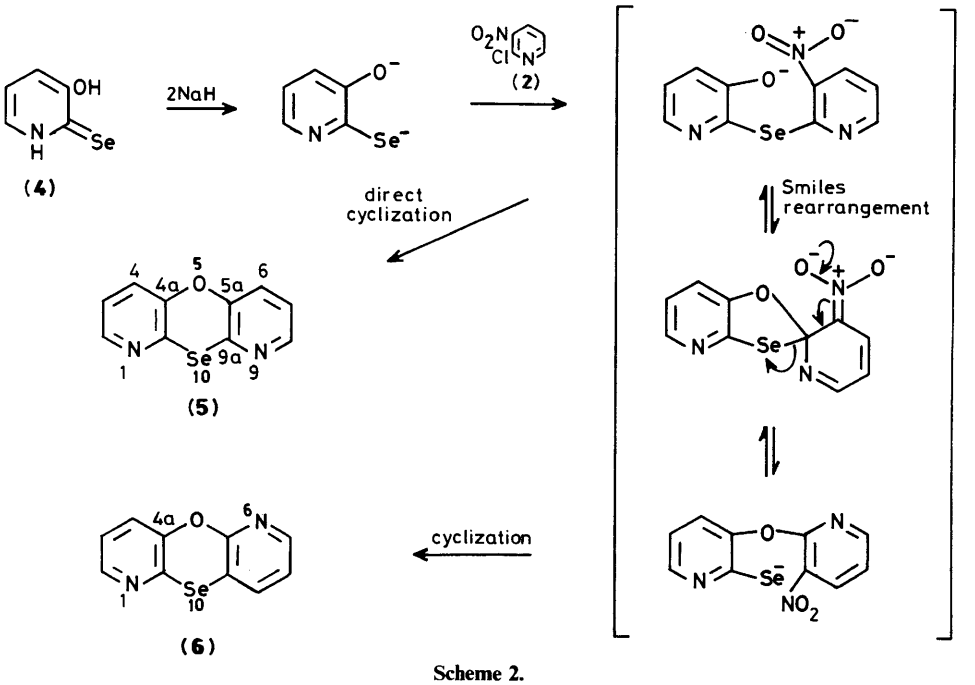


Scheme 1.

Surprisingly, however, the reaction of the dianion of (**4**) with (**2**) was not regiospecific. Rather, a mixture of two isomers was obtained (Scheme 2). These were separated by flash-column chromatography on silica and identified as the 1,6- and 1,9-diazaphenoxaselenines. Furthermore, the 1,9-isomer (**5**) which was expected to be the sole or at least major product, was isolated in only 25% yield, and was the minor product. The major product, isolated in 46% yield, was the 1,6-isomer (**6**), for which there is as yet no counterpart in the phenoxathiine series.

The proportion of the two products (**5**):(**6**) remained constant within experimental error (by h.p.l.c.) during the entire course of the reaction and also during prolonged heating of the final product mixture. Furthermore, there is no evidence for equilibration of the isolated products. Thus, it appears that the product mixture obtained is a result of kinetic rather than thermodynamic factors. Although we have not attempted to establish the fact unequivocally, the most likely mechanism for the formation of (**6**) involves a Smiles rearrangement[9] (Scheme 2). Smiles rearrangements do not generally occur in the phenoxathiine series. Indeed, the only known example of such a rearrangement during the synthesis of an azaphenoxathiine involves an oxo derivative[10] in which the carbonyl group presumably facilitates formation of the spirocyclic Smiles intermediate. There is, however, no such encouragement in the case of the formation of (**6**). Thus, pending further studies, there is as yet no definitive explanation for the marked difference in this case between the sulphur and selenium compounds. The fact that C–Se bonds are longer and weaker than their C–S counterparts may, however, serve to rationalize the observations.

† The nomenclature system currently recommended by the I.U.P.A.C. is used throughout this article; thus, phenoxaselenine rather than phenoxaselenin is used.



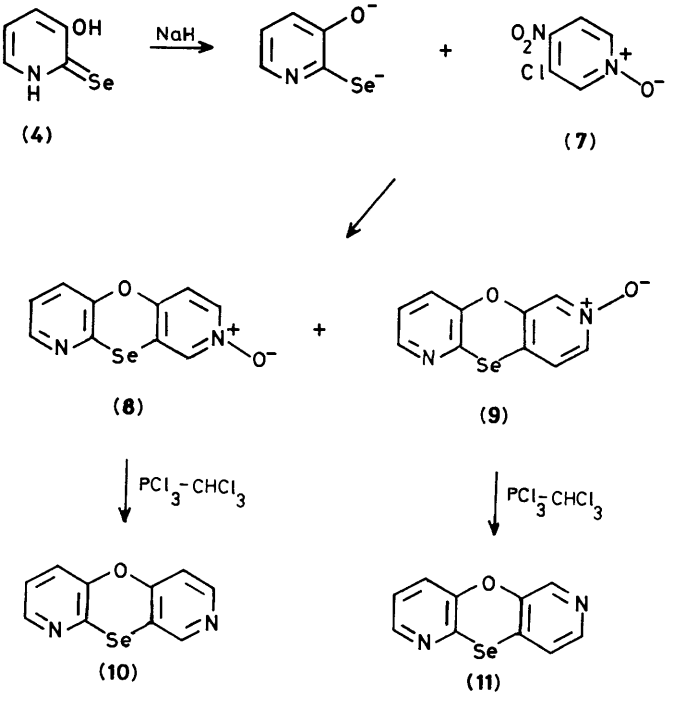
Scheme 2.

The structures of compounds (**5**) and (**6**) were confirmed by mass spectrometry and ^{1}H and ^{13}C n.m.r. spectroscopy. Both compounds showed a substantial molecular ion cluster with a characteristic selenium isotope distribution, and a base peak corresponding to loss of Se from the molecular ion, in their electron-impact mass spectra. Distinction of the two isomers by n.m.r. spectroscopy was relatively easy because of the higher order of symmetry of compound (**5**). Thus, (**5**) showed only three ^{1}H n.m.r. signals and five ^{13}C n.m.r. signals, whereas (**6**) showed six and ten signals respectively. Because of the very close correlations between signals in the phenoxathiine and phenoxaselenine series,[3] and the availability of assigned spectra for 1,9-diazaphenoxathiine,[5] assignment (see the Experimental section) of the spectra for (**5**) was straightforward.

Unfortunately, 1,6-diazaphenoxathiine has never been prepared, so unequivocal assignment of all signals in the ^{13}C n.m.r. spectrum of (**6**) from a simple noise or off-resonance proton-decoupled spectrum was not possible. Only the quaternary carbons (C-4a, C-5a, C-9a, and C-10a) and one of the protonated carbons (C-9) could readily be assigned, whilst there was some ambiguity of assignment between C-2 and C-7 and between C-3, C-4, and C-8. However, assignment of the spectrum is important for future identification of substituted and/or analogous compounds. Thus, we carried out a 300 MHz two-dimensional proton–carbon chemical shift correlation experiment (CSCM)[11] and a two-dimensional long range optimized proton–carbon heteronuclear chemical shift correlation experiment (LROCSCM)[12] in order to ascertain all one-bond and three-bond connectivities as well as some two-bond couplings in the system. From the long range connectivities observed it was possible to assign with certainty all of the remaining carbon and proton resonances (see the Experimental section).

Preparation of 1,7-*Diazaphenoxaselenine* (**11**) *and* 1,8-*Diazaphenoxaselenine* (**10**).—1,7-Diazaphenoxathiine was synthesized previously by the reaction of the dianion of (**1**) with 4-chloro-3-nitropyridine.[4] The report of this work does not mention production of any 1,8-diazaphenoxathiine as a contaminant. We have recently synthesized the latter compound *via* reaction of the dianion of (**1**) with 3-chloro-4-nitropyridine 1-oxide (**7**) and then reduction of the *N*-oxide, and in this case the product was accompanied by 7% of the 1,7-isomer.[7] Thus, the utilization of the latter approach was favoured for preparation of the corresponding diazaphenoxaselenines because it appeared more likely that both compounds could be obtained from a single reaction. A further advantage of this approach is that (**7**) is a stable, crystalline, easily prepared starting material, whereas 4-chloro-3-nitropyridine is difficult to prepare and to handle.

The reaction of the dianion of (**4**) with (**7**) (Scheme 3) did



Scheme 3.

Table. ^{13}C N.m.r. chemical shifts[a] of compounds (**5**), (**6**), (**8**), (**9**), (**10**), and (**11**)

	Compound					
Carbon[b]	(**5**)[c]	(**6**)	(**8**)	(**9**)	(**10**)	(**11**)
2	145.9	146.0	147.1	146.8	146.4	146.2[A]
3	122.6	122.9	123.9	123.5	122.9	123.3[B]
4	124.2	125.2	125.2	124.9[A]	124.4	124.8[B]
6	124.2		115.2	130.3	113.3	145.6[A]
7	122.6	146.4	138.6[A]		150.1[A]	
8	145.9	121.8		136.1		139.4
9		139.1	139.1[A]	125.0[A]	150.2[A]	124.0[B]
4a	147.6	148.8	147.9	147.7	147.9	147.7
5a	147.6	156.2	149.9	149.6	157.1	148.5
9a	140.8	110.8	116.0	114.6	112.5	126.7
10a	140.8	140.0	137.8	138.8	139.5	139.1

[a] Spectra were recorded for deuteriochloroform solutions, with the exception of (**8**) which was recorded for a deuteriochloroform–deuteriomethanol solution. [b] Where assignments may be permutable, the same superscript capital letter denotes possibly permutable signals. [c] The symmetry inherent in this compound means that there are only five distinct signals.

indeed produce both the 1,7- and 1,8-diazaphenoxaselenine *N*-oxides [(**9**) and (**8**) respectively], with the 1,8-isomer predominating. However, despite our efforts to improve the yields by variation of the reaction conditions, the yields obtained were rather poor, *viz.* 44% for (**8**) and 4% for (**9**) after separation by flash-column chromatography. The separated isomers were readily converted into the parent systems in high yield by treatment with phosphorus trichloride in chloroform. In one experiment the mixture of (**8**) and (**9**) was converted directly into a mixture of (**10**) and (**11**). However, separation of (**10**) from (**11**) proved to be more difficult than separation of (**8**) from (**9**). Thus, separation at the intermediate stage is preferable.

The electron impact mass spectra characterized the compounds as diazaphenoxaselenines or their *N*-oxides respectively. The diazaphenoxaselenines (**10**) and (**11**) showed major molecular ion clusters with a characteristic selenium isotope pattern and base peaks corresponding to loss of Se. The *N*-oxides (**8**) and (**9**) showed selenium isotope patterns for the molecular ion clusters and for the fragments corresponding to loss of O to give the parent diazaphenoxaselenines, and substantial peaks at *m/z* 170 corresponding to a further loss of Se. Full identification of the 1,8-isomers, (**10**) and (**8**), was relatively straightforward because of the availability of the corresponding phenoxathiine compounds, the n.m.r. spectra of which have been assigned.[7] The only resonances which could not readily be assigned unequivocally from the simple spectra were the C-7 and C-9 carbon resonances. These were so similar in chemical shift [0.1 p.p.m. difference for (**10**), 0.6 p.p.m. for (**8**)] that changes in concentration or solvent could easily invert the positions. Thus, it was not felt to be worthwhile to carry out T_1 relaxation time measurements in order to provide unequivocal assignments.

Although in the case of the 1,7-compounds, (**11**) and (**9**), there were rather more n.m.r. resonances which could not be unambiguously assigned, the small amount of material available prevented the use of heteronuclear two-dimensional n.m.r. techniques for clarification. Nevertheless, the close similarity of the spectra of (**11**) to those of the corresponding 1,7-diazaphenoxathiine clearly established the identity of the compound.

Conclusion

The reaction of the dianion of 3-hydroxypyridine-2(1*H*)-selone (**4**) with 2-chloro-3-nitropyridine (**2**) takes a surprising course. The major product is not 1,9-diazaphenoxaselenine (**5**), the only product expected by analogy with the synthesis of the comparable 1,9-diazaphenoxathiine (**3**), but is 1,6-diazaphenoxaselenine (**6**), which has no counterpart as yet in the phenoxathiine series. Further studies are in hand to try and establish the basis of the difference between the two systems, and also to investigate the possibility of the conversion of (**6**) into its phenoxathiine analogue which has so far not been obtainable.

The reaction of the dianion of (**4**) with 3-chloro-4-nitropyridine 1-oxide (**7**) gives 1,8-diazaphenoxaselenine 8-oxide (**8**) in 44% yield and 1,7-diazaphenoxaselenine 7-oxide (**9**) in 4% yield. These compounds are separable by flash-column chromatography on alumina and can then be individually reduced in high yield with phosphorus trichloride to give the corresponding diazaphenoxaselenines (**10**) and (**11**). Thus, the first and parent examples of four new heterocyclic systems, the 1,6-, 1,7-, 1,8-, and 1,9-diazaphenoxaselenines, have been characterized. The investigation of their molecular structures and pharmacological properties in comparison to those of their sulphur-containing counterparts will be of particular interest.

Experimental

2-Chloro-3-nitropyridine, 3-chloropyridine, and phosphorus trichloride were purchased from Aldrich and used without further purification. Solvents were purified and dried by standard procedures.[13] Sodium hydride was purchased as a dispersion in oil (80%); the solid was washed with pentane under nitrogen to remove the oil, and aliquots of the oil-free solid were weighed for individual reactions. 3-Chloro-4-nitropyridine 1-oxide was prepared by the procedure of Talik and Talik.[14]

Melting points were determined using a Gallenkamp hot-stage apparatus and are reported uncorrected. ^1H N.m.r. spectra were recorded on a Varian HA100 spectrometer. ^{13}C N.m.r. spectra were generally recorded on a Varian XL-100 spectrometer, but for the 2D studies of compound (**6**) a Nicolet NT-300 wide bore spectrometer controlled by a Model 293-C pulse programmer was used. The conventional proton–carbon CSCM experiment employed the pulse sequence of Bax and Morris,[11] the deviations of the Δ_1 and Δ_2 delays set to 3.0 and 2.0 ms, respectively. The LROCSCM involved the same sequence with Δ_1 and Δ_2 set to 50.0 and 33.3 ms, respectively, to provide a 10 Hz transfer of magnetization.[12] Data for both experiments were collected as 140 blocks, zero filled to provide an initial 256 × 1 K point matrix. The data were processed using double exponential apodization prior to both Fourier transforms, giving a final 256 × 512 point matrix. All chemical shifts are

reported in parts per million (δ) downfield from tetramethylsilane. U.v. spectra were recorded on a Perkin-Elmer UV402 instrument and i.r. spectra were recorded as KBr discs on a Pye-Unicam SP1050 instrument. Mass spectra were run on a modified KRATOS MS9 mass spectrometer and elemental analyses were obtained using a Perkin-Elmer 185 C, H, N analyser.

^{13}C N.m.r. chemical shifts for the compounds prepared in this study are recorded in the Table.

Preparation of 1,6-*Diazaphenoxaselenine* (**6**) *and* 1,9-*Diazaphenoxaselenine* (**5**).—A two-necked, round-bottomed flask was charged rapidly with oil-free sodium hydride (0.19 g, 7.9 mmol). It was then fitted with a septum on the side-arm and a septum-capped reflux condenser, cooled in ice, and flushed with argon. Dry *N*,*N*-dimethylformamide (DMF) (20 ml) was added and the suspension was stirred during the addition of a solution of 3-hydroxypyridine-2(1*H*)-selone (**4**) (0.66 g, 3.8 mmol) in dry DMF (20 ml). The mixture was stirred for 45 min at 0 °C and then overnight at 20 °C. It was then recooled to 0 °C and a solution of 2-chloro-3-nitropyridine (**2**) (0.60 g, 3.8 mmol) in dry DMF (20 ml) was added over 15 min, during which the mixture darkened. The reaction mixture was maintained at 0—5 °C for 1 h, allowed to warm to ambient and then refluxed for 4 h. The cooled mixture was poured into distilled water (100 ml), which was then extracted with diethyl ether (4 × 100 ml). The combined extracts were washed with aqueous sodium carbonate (1M; 2 × 100 ml) and distilled water (2 × 80 ml), dried ($MgSO_4$), and then evaporated under reduced pressure to yield a yellow solid consisting of three components according to t.l.c. Flash-column chromatography [silica; cyclohexane–ethyl acetate (6:4)] gave a yellow oil (0.02 g), which remains uncharacterized, followed by (**6**) (0.43 g, 46%), and (**5**) (0.24 g, 25%), both as off-white solids.

Recrystallization of (**6**) from ethyl acetate–cyclohexane (6:4) gave off-white crystals, m.p. 125—127 °C; $\nu_{max.}$ 3 060, 1 580, 1 560, 1 450, 1 410, 1 280, 1 200, 1 085, 810, and 790 cm^{-1}; $\lambda_{max.}$ (0.095mM in methanol) 222 (ε 9 900 dm^3 mol^{-1} cm^{-1}) and 305 nm (8 200); $\delta_H(CDCl_3)$ 8.16 (1 H, dd, *J* 2 and 5 Hz, 2-H), 8.05 (1 H, dd, *J* 2 and 5 Hz, 7-H), 7.57 (1 H, dd, *J* 2 and 8 Hz, 9-H), 7.29 (1 H, dd, *J* 2 and 8 Hz, 4-H), 7.05 (1 H, dd, *J* 5 and 8 Hz, 3-H), and 6.97 (1 H, dd, *J* 5 and 8 Hz, 8-H); *m*/*z* 250 (45%), 248 (22), 171 (12), 170 (100), 142 (11), 130 (10), 115 (12), and 39 (64) (Found: C, 48.3; H, 2.3; N, 11.1%; M^+, 249.9644. $C_{10}H_6N_2O^{80}Se$ requires C, 48.2; H, 2.4; N, 11.3%; *M*, 249.9645).

Recrystallization of (**5**) from ethyl acetate–cyclohexane (6:4) gave off-white crystals, m.p. 104.5—105.5 °C; $\nu_{max.}$ 3 060, 1 585, 1 450, 1 400, 1 265, 1 205, 1 090, 820, 795, and 730 cm^{-1}; $\lambda_{max.}$ (0.097mM in methanol) 218 (ε 9 800), 233 (10 400), and 310 nm (7 730); $\delta_H(CDCl_3)$ 8.14 (2 H, dd, *J* 2 and 5 Hz, 2- and 8-H), 7.18 (2 H, dd, *J* 2 and 8 Hz, 4- and 6-H), and 7.00 (2 H, dd, *J* 5 and 8 Hz, 3- and 7-H); *m*/*z* 252 (10%), 250 (56), 248 (27), 247 (10), 246 (10), 171 (10), 170 (100), 169 (18), 130 (10), 115 (12), and 39 (83) (Found: C, 48.5; H, 2.3; N, 11.2%; M^+, 249.9644. $C_{10}H_6N_2O^{80}Se$ requires C, 48.2; H, 2.4; N, 11.3%; *M*, 249.9645).

Preparation of 1,8-*Diazaphenoxaselenine* 8-*Oxide* (**8**) *and* 1,7-*Diazaphenoxaselenine* 7-*Oxide* (**9**).—The apparatus was set up and charged with oil-free sodium hydride (0.63 g, 26.4 mmol) and dry DMF (20 ml) at 0 °C as described above. Compound (**4**) (1.93 g, 11.1 mmol) in dry DMF (20 ml) was added and the mixture was then allowed to warm to 20 °C, stirred for 3 h, and then recooled to 0 °C. A solution of 3-chloro-4-nitropyridine 1-oxide (**7**) (2.09 g, 11.1 mmol) in dry DMF (20 ml) was added dropwise, with stirring, over 15 min, during which time the mixture darkened. The mixture was stirred overnight at 20 °C then refluxed for 5 h, after which it was cooled and poured into distilled water (100 ml). The resultant dark brown mixture was brought to pH 9 with aqueous NaOH (5M) and extracted with chloroform (6 × 50 ml). The combined extracts were washed with aqueous sodium carbonate (1M; 2 × 50 ml) and water (2 × 50 ml), dried ($MgSO_4$), and evaporated under reduced pressure to yield a brown solid consisting of three components according to t.l.c. Flash-column chromatography (alumina; chloroform) gave a brown oil (0.16 g), which remains uncharacterized, and (**8**) (1.30 g, 44%) and (**9**) (0.11 g, 4%) as off-white solids.

Recrystallization of (**8**) from chloroform–hexane gave off-white crystals, m.p. 223—224 °C; $\nu_{max.}$ 3 040, 1 470, 1 420, 1 280, 1 205, 1 075, 870, 835, 805, and 780 cm^{-1}; $\lambda_{max.}$ (0.061mM in methanol) 219 (ε 14 000) and 271 nm (22 900); $\delta_H(CDCl_3$–$CD_3OD)$ 8.21 (1 H, dd, *J* 2 and 4.5 Hz, 2-H), 8.12 (1 H, d, *J* 2 Hz, 9-H), 7.98 (1 H, dd, *J* 2 and 7 Hz, 7-H), 7.27 (1 H, dd, *J* 2 and 8.5 Hz, 4-H), 7.12 (1 H, dd, *J* 4.5 and 8.5 Hz, 3-H), and 6.96 (1 H, d, *J* 7 Hz, 6-H); *m*/*z* 268 (13%), 266 (84), 264 (34), 263 (13), 262 (16), 250 (37), 248 (16), 170 (59), 169 (17), 158 (19), 130 (12), 103 (12), and 39 (100) (Found: C, 45.6; H, 2.1; N, 10.3%; M^+, 265.9600. $C_{10}H_6N_2O_2{}^{80}Se$ requires C, 45.3; H, 2.3; N, 10.6%; *M*, 265.9594).

Recrystallization of (**9**) from chloroform–hexane gave off-white crystals, m.p. 187—189 °C; $\nu_{max.}$ 3 480br, 3 020, 1 470, 1 440, 1 410, 1 230, 1 200, 1 170, 1 060, and 800 cm^{-1}; $\lambda_{max.}$ (0.096mM in methanol) 227 (13 300), 266 (14 800), and 332 nm (6 240); $\delta_H(CDCl_3)$ 8.25 (1 H, dd, *J* 1.5 and 4.5 Hz, 2-H), 8.02 (1 H, d, *J* 1.5 Hz, 6-H), 7.92 (1 H, dd, *J* 1.5 and 6.5 Hz, 8-H), and 7.23 (3 H, m, 3-, 4-, and 9-H); *m*/*z* 268 (20%), 266 (100), 264 (47), 263 (16), 262 (15), 250 (29), 186 (48), 170 (50), 130 (11), 103 (36), and 39 (86) (Found: C, 45.3; H, 2.3; N, 10.2%; M^+, 265.9601. $C_{10}H_6N_2O_2{}^{80}Se$ requires C, 45.3; H, 2.3; N, 10.6%; *M*, 265.9594).

Synthesis of 1,8-*Diazaphenoxaselenine* (**10**).—Compound (**8**) (0.35 g, 1.3 mmol) was dissolved in chloroform (12 ml), PCl_3 (0.16 ml, excess) was added, and the mixture was brought to reflux for 2 h, after which t.l.c. [alumina; MeOH–$CHCl_3$ (5:95)] indicated that no compound (**8**) remained. The mixture was cooled and poured cautiously into distilled water (15 ml) and then neutralized with aqueous NaOH (5M). The chloroform layer was separated and the aqueous layer was extracted with more chloroform (15 ml). The combined chloroform extracts were dried ($MgSO_4$) and evaporated under reduced pressure to yield (**10**) (0.32 g, 98%) as a pale yellow oil which solidified when allowed to stand. Recrystallization of the solid from diethyl ether gave a white crystalline solid, m.p. 96—97 °C; $\nu_{max.}$ 1 580, 1 480, 1 445, 1 415, 1 270, 1 200, 890, 840, 810, 795, and 720 cm^{-1}; $\lambda_{max.}$ (0.06mM in methanol) 214sh, 237 (20 200), and 303 nm (7 500); $\delta_H(CDCl_3)$ 8.32 (1 H, s, 9-H), 8.27 (1 H, d, *J* 6 Hz, 7-H), 8.15 (1 H, dd, *J* 2 and 4.5 Hz, 2-H), 7.17 (1 H, dd, *J* 2 and 8 Hz, 4-H), 7.02 (1 H, dd, *J* 4.5 and 8 Hz, 3-H), and 6.85 (1 H, d, *J* 6 Hz, 6-H); *m*/*z* 252 (12%), 250 (74), 248 (34), 247 (11), 246 (12), 170 (100), 169 (29), 130 (12), 115 (13), and 39 (49) (Found: C, 48.3; H, 2.4; N, 11.3%; M^+, 249.9653. $C_{10}H_6N_2O^{80}Se$ requires C, 48.2; H, 2.4; N, 11.3%; *M*, 249.9645).

Synthesis of 1,7-*Diazaphenoxaselenine* (**11**).—Compound (**9**) (0.1082 g, 0.41 mmol) was reduced with phosphorus trichloride in chloroform in exactly the same manner as described for the reduction of (**8**) to (**10**). The product (**11**) (0.0944 g, 93%) was obtained as a pale yellow solid, recrystallization of which from diethyl ether gave white crystals, m.p. 125—126 °C; $\nu_{max.}$ 1 585, 1 445, 1 420, 1 270, 1 220, 830, and 790 cm^{-1}; $\lambda_{max.}$ (0.088mM in methanol) 233 (16 400), 245 (15 700), and 307 nm (7 390); $\delta_H(CDCl_3)$ 8.05—8.35 (3 H, m, 2-, 6-, and 8-H) and 7.0—7.3 (3 H, m, 3-, 4-, and 9-H); *m*/*z* 252 (14%), 250 (86), 248 (41), 247 (14), 246 (16), 170 (100), 169 (28), 142 (14), 130 (17), 115 (19), and 39 (81)

(Found: C, 48.8; H, 2.3; N, 11.7%; M^+, 249.9651. $C_{10}H_6N_2O^{80}Se$ requires C, 48.2; H, 2.4; N, 11.3%; M, 249.9645).

Acknowledgements

K. S. and G. E. M. thank the North Atlantic Treaty Organization for grant 019.81 which enabled the collaboration leading to the initiation of this work. The partial support of this work through Grant No. E-792 by the Robert A. Welch Foundation to G. E. M. is also acknowledged.

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Received 22*nd December* 1986; *Paper* 6/2449