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STRUCTURAL STUDIES OF HALOGEN ADDUCTS OF SOME CYCLIC SELENIDES AND TELLURIDES BY ^{1}H, ^{13}C, ^{77}Se AND ^{125}Te NMR. EVIDENCE FOR THE FORMATION OF MOLECULAR COMPLEXES OF SELENOXANTHONE AND SELENANTHRENE WITH BROMINE*

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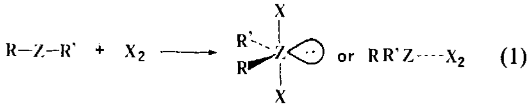
^{1}H, ^{13}C, ^{77}Se and ^{125}Te NMR spectra were recorded for selenoxanthone (1a), telluroxanthone (2a), selenanthrene (3a), related compounds and their halogen adducts to elucidate the structure of the adducts in solutions. Although selenides react with bromine to give trigonal bipyramidal (TB) hypervalent complexes in general, 1a reacted with bromine to yield a molecular complex (MC). Its chlorine adduct was found to be a TB. The effective electronegativity of selenium in 1a may be modulated by conjugation with the strongly electron-withdrawing carbonyl group. In the case of 2a, its halogen adducts were TBs. The structure of the bromine adduct of 3a was also demonstrated to be an MC from NMR data.

INTRODUCTION

Ethers, sulphides, selenides and tellurides react with halogens to give trigonal bipyramidal (TB) adducts or molecular complexes (MCs).[2,3] TBs are formed, in general, if the electronegativity of the halogen ligands (χ_X) is larger than that of the Group VIB elements (χ_Z) in the compounds. The adducts are MCs when χ_X is less than χ_Z [equation (1)].[4]

This generalization holds good in most cases, but there are some exceptions. Selenides react with bromine to give TBs,[2,3c,e] but the bromine adducts of sulphides are well demonstrated to be MCs;[2,3b] the MC structure of sulphide dibromides is the opposite of expectation, because the electronegativity[4,5] of bromine ($\chi_{Br} = 2{\cdot}74$) is larger than those of sulphur ($\chi_S = 2{\cdot}44$) and selenium ($\chi_{Se} = 2{\cdot}48$).



TB($\chi_X > \chi_Z$)　　MC($\chi_X < \chi_Z$)

M (χ_X)	F(4·10)	Cl(2·83)	Br(2·74)	I(2·21)
O (3·50)		MC	MC	MC
S (2·44)	TB	TB	MC	MC
Se (2·48)	TB	TB	TB	MC
Te (2·01)	TB	TB	TB	TB

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* For a preliminary contribution, see Ref. 1.

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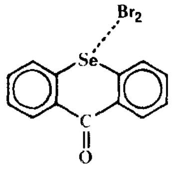
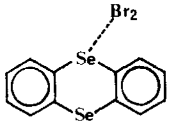
Since the electronegativity of selenium is very close to that of sulphur[4,5] and the bromine adducts of sulphur are MCs,[2,3b] MCs of selenides with bromine should exist if the effective electronegativity of the selenium atom in a given compound is high with respect to the neighbouring substituents. This idea led us to look for some selenides that react with bromine to yield MCs.

Here we report that (i) selenoxanthone (**1a**) reacts with bromine to give an MC (**1d**), although telluroxanthone (**2a**) gives TBs with chlorine, bromine and iodine, and (ii) selenanthrene (**3a**) also yields an MC (**3d**) with bromine.



1d



3d

RESULTS AND DISCUSSION

^{13}C chemical shifts

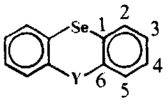
Before discussing the structures of the halogen adducts, it is instructive to extend the scope of the criteria obtained in the preceding paper[2] for distinguishing MCs from TBs for acyclic systems on the basis of the NMR data.

^{13}C NMR spectra were measured for selenoxanthone (**1a**), telluroxanthone (**2a**), selenanthrene (**3a**), phenoxselenine (**4a**), phenoxtellurine (**5a**) and their chlorine, bromine and iodine adducts (e.g. **1c**, **1d** and **1e**, respectively) and selenoxanthone selenium oxide (**1f**) and selenanthrene diselenium dioxide (**3f**). ^{77}Se and ^{125}Te NMR spectra were also recorded for **1a**, **2a**, **4a**, **5a** and their halogen adducts. The results are shown in Tables 1–4.

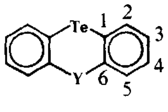
Table 2. ^{13}C NMR chemical shifts of selenanthrene (**3a**), its halogen adducts and selenanthrene diselenium dioxide (**3f**)[a,b]

Compound	C-1	C-2	C-3	Structure
3a	134·4	131·1	127·9	Selenide
	0·0	0·0	0·0	
3d	−2·1	−0·2	1·9	MC
3e	−0·5	0·1	0·3	MC
3f	10·3	−2·6	3·3	Selenoxide

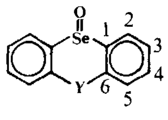
[a] In $CDCl_3$.
[b] Chemical shifts (ppm) of **3a** are given from TMS and those of the derivatives from **3a**.



1a (Y=C=O)
3a (Y=Se)
4a (Y=O)



2a (Y=C=O)
5a (Y=O)



1f (Y=C=O)
3f (Y=Se=O)

Ar_2ZX_2: X = — Cl Br I
a c d e

As shown in Table 3, large downfield shifts were observed for C-4 carbons of the chlorine and bromine adducts (**4c** and **4d**, respectively) of phenoxselenine (**4a** of 6·0–6·2 ppm and of chlorine, bromine and iodine adducts (**5c**, **5d**, and **5e**, respectively) of phenoxtellurine (**5a**) of 4·2–4·5 ppm relative to those of **4a** and **5a**, respectively. These results correspond with a TB structure of the adducts, although the downfield shifts of the C-1 carbons are smaller than those in diphenyl derivatives (by 7–9 ppm).[2] Such smaller shifts for the C-1 carbons may be due to the cyclic structure of **4a** and **5a**, since a similar trend has been observed in TB

Table 1. ^{13}C NMR chemical shifts of selenoxanthone (**1a**), telluroxanthone (**2a**), their halogen adducts and selenoxanthone selenium oxide (**1f**)[a,b]

Compound	C-1	C-2	C-3	C-4	C-5	C-6	C=O	Structure
1a	134·9	128·1	132·0	126·6	131·2	130·8	181·7	Selenide
	0·0	0·0	0·0	0·0	0·0	0·0	0·0	
1c	7·8	0·4	2·7	6·1	0·9	−2·1	−1·9	TB
1d	[c]	0·2	0·1	0·1	0·2	[c]	[c]	MC
1e	−0·1	0·1	0·1	0·1	0·1	0·0	0·1	MC
1f	7·7	1·4	2·0	5·4	−0·9	0·1	0·3	Selenoxide
2a	121·8	134·2	131·7	127·4	131·7	133·6	185·7	Telluride
	0·0	0·0	0·0	0·0	0·0	0·0	0·0	
2c	16·4	−0·6	1·2	4·4	−1·7	0·1	2·8	TB
2d	12·7	−0·1	1·4	3·8	−1·5	0·5	2·4	TB
2e	8·7	0·3	2·9	3·4	1·2	1·0	1·7	TB

[a] In $CDCl_3$ for selenoxanthones and in DMSO-d_6 for telluroxanthones.
[b] Chemical shifts (ppm) are given from TMS for **1a** and **2a** and from their parent compounds for the derivatives.
[c] Not observed owing to broadening.

Table 3. ^{13}C NMR chemical shifts of phenoxselenine (**4a**), phenoxtellurine (**5a**) and their halogen adducts[a,b]

Compound	C-1	C-2	C-3	C-4	C-5	C-6	Structure
4a	116·2	129·3	124·9	128·1	118·6	152·9	Selenide
	0·0	0·0	0·0	0·0	0·0	0·0	
4c	4·4	−0·1	0·8	6·2	1·3	−5·3	TB
4d	0·7	0·5	1·1	6·0	1·3	−4·8	TB
4e	−0·9	0·1	0·1	0·5	0·1	−0·3	MC
5a	103·9	135·1	125·2	128·7	118·9	155·2	Telluride
	0·0	0·0	0·0	0·0	0·0	0·0	
5c	15·3	−1·3	−0·7	4·5	0·0	−4·1	TB
5d	11·3	−0·7	−0·5	4·5	−0·1	−3·7	TB
5e	5·5	0·2	−0·3	4·2	−0·2	−2·8	TB

[a] In $CDCl_3$ for phenoxselenines and in DMSO-d_6 for phenoxtellurines.
[b] Chemical shifts (ppm) are given from TMS for **4a** and **5a**, and from their parent compounds for the adducts.

Table 4. ^{77}Se and ^{125}Te NMR chemical shifts of **1a**, **2a**, **4a**, **5a** and their halogen adducts

Compound	$\delta(^{77}Se)^a$	$\Delta\delta$	Compound	$\delta(^{125}Te)^b$	$\Delta\delta$
1a[c]	334·7	0·0	**2a**[c]	471·5	0·0
1d	336·7	2·0	**2d**	572·4	100·9
4a[c]	260·6	0·0	**5a**[c]	432·3	0·0
4c[c]	381·7	121·1	**5c**[c]	628·6	196·3
			5d[c]	598·4	166·1
4e[c]	263·4	2·8	**5e**[c]	549·1	116·8

[a] In $CDCl_3$ (reference: MeSeMe in $CDCl_3$).
[b] In DMSO-d_6 (reference: MeTeMe in C_6D_6).
[c] Ref. 2.

formation from selenoxanthone (**1a**) and telluroxanthone (**2a**), as will be described later.

Large upfield shifts for C-6 carbons are characteristic of TB formation from **4a** and **5a**. The strong electronegative selenodihalo and tellurodihalo groups may contribute to the upfield shifts since the chemical shifts of *ortho* carbons in substituted benzenes bearing electronegative groups usually move upfield.[6] The extra interaction between the selenodihalo or tellurodihalo group and the oxygen atom must also play an important role.

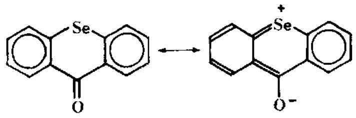
On the other hand, chemical shifts of the iodine adduct (**4e**) are not very different from those of **4a**; the chemical shifts for C-1 and C-4 in **4e** are 0·9 ppm upfield and 0·5 ppm downfield respectively, relative to those of **4a**. The chemical shift data reflect the MC structure of **4e**.

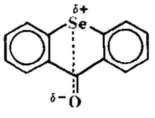
These results show that the criteria obtained in the preceding paper[2] for distinguishing MCs from TBs on the basis of NMR data are also valid for cyclic systems. The chemical shifts of C-4 carbons serve as good measures of the distinction; those of C-1 carbons are also useful if they are examined carefully.

We are now ready to discuss the structure of the halogen adducts of **1a**–**3a**. As shown in Table 1, the signals of C-1 and C-4 in the chlorine adduct (**1c**) of selenoxanthone (**1a**) appeared 7·8 and 6·1 ppm, respectively, downfield from those of **1a**, indicating the TB structure of **1c**. The strong electron-withdrawing ability of the selenodichloro group in **1c** becomes apparent if the chemical shifts of **1c** are compared with those of the selenoxide **1f**. The C-1 and C-4 chemical shifts of **1f** from those of **1a** are 7·7 and 5·4 ppm, respectively, which are very close to those for **1c**, although the structure of **1c** is planar (or TB), but **1f** is tetrahedral. The structure of the iodine adduct (**1e**) must be an MC, since the chemical shifts of **1e** are almost equal to those of **1a**. In particular, the chemical shifts of C-1 and C-4 in **1e** are only 0·1 ppm upfield and downfield, respectively, of those of **1a**. Such small shift differences between **1e** and **1a** should correspond to the large dissociation constant for **1e**.[2,7] The electron transfer from the lone pair of electrons of the selenium atom to the σ^*-orbital of iodine in **1e** may not be so effective.[8]

In the case of the bromine adduct **1d**, the C-4 chemical shift is only 0·1 ppm downfield of that of **1a** and other shift values are almost equal to those of **1a** and **1e**, although the signals of C-1, C-6 and carbonyl carbons could not be detected owing to broadening. These results show that the structure of the bromine adduct **1d** must be an MC. The signals of C-2, C-3, C-4 and C-5 were also broad; the half-width values were 1·6, 3·0, 3·1 and 2·0 Hz, respectively, at 45 °C. The rate of the dissociation process may not be very fast relative to the ^{13}C NMR time scale.

When two or more atoms of initially different electronegativity are combined chemically, the electronegativities will be more or less neutralized within the molecule.[5b] The effective (or adjusted) electronegativity of selenium in **1a** becomes larger, because the electronegativity of oxygen ($\chi_O = 3{\cdot}50$) is much larger than that of selenium ($\chi_{Se} = 2{\cdot}48$).[5] The electron density of the lone pairs on the selenium atom in **1a** is expected to be smaller and its ionization potential[9] larger, since the carbonyl group withdraws electron strongly by both through-bond and through-space mechanisms. The effective electronegativity of selenium in **1a** may no longer be small enough to give a TB in the reaction with bromine.[1,10] The MC structure of **1d** is consistent with the ineffective electron transfer in **1e** discussed above.





It is worth noting that on heating, the dark-red needles of **1d** decolorized at ca 125 °C and melted at 188–190 °C. The melting point is similar to that of the parent, **1a** (191–192 °C). The decolorized material was identified with **1a** by NMR, IR and m.p. data. These results may support the MC structure of **1d**, and its behaviour is different from that of diphenyl selenide dibromide (TB), in which bromination of benzene rings occurs on heating.[11]

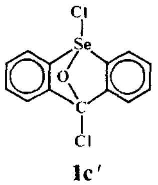
The alternative structure (**1c′**) for **1c** is very unlikely because the observed ^{13}C chemical shift at δ 179·8 and the IR spectrum at 1662 cm^{-1} should be assigned to the carbonyl group of **1c** (1634 cm^{-1} for **1a**).

The structure of halogen adducts of telluroxanthone (**2a**) was also examined. Large downfield shifts were observed for C-1 (8·7–16·4 ppm) and C-4 (3·4–4·4 ppm) of the chlorine, bromine and iodine adducts (**2c**, **2d** and **2e**, respectively) relative to those of **2a**. These observations show that the halogen adducts, **2c**, **2d** and **2e**, are all TBs. The shifts of the C-1 carbons depend on the electronegativity of the halogens and they are smaller than those of diphenyl derivatives by ca 7 ppm, which was also observed in the formation of TBs from **4a** and **5a**.

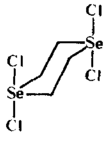
The structure of the bromine adduct (**3d**) of selenanthrene (**3a**) has also been demonstrated to be an MC in solutions. As shown in Table 2, the signals for C-1 and C-3 of the iodine adduct **3e**, which is an MC, appeared 0·5 ppm upfield and 0·3 ppm, downfield, respectively, relative to those of **3a**. The chemical shift differences between **3d** and **3a** are −2·1 and 1·9 ppm, respectively, which indicates an MC structure for **3d**. The larger shift differences of **3d** from **3a** may be due to the smaller dissociation constant and the larger electron transfer in **3d**.[2]

Although ^{13}C NMR spectra could not be recorded for the tetrachloride **3c** since the chlorine adduct of **3a** was very sparingly soluble in typical organic solvents (the chlorine adduct obtained may not be **3c**), the chemical shifts of **3c**, if it existed, could be estimated from those of selenanthrene diselenium dioxide (**3f**). The chemical shifts of the *ipso* and *para* carbons of diphenyl selenoxide are very close to those of diphenyl selenide dichloride,[12] and must be so in compounds **1c** and **1f** (Table 1). The chemical shifts of C-1 and C-4 of **3f** are 10·3 and 3·3 ppm downfield of those of **3a**, i.e. different from the case of **3d** and **3e**. These results are well explained by assuming that the structure of the bromine adduct **3d** is an MC, in addition to that of **3e**.

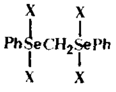
The TB structure has been demonstrated for the tetrachlorides, **6c**,[13] **7c**[14] and **8c**,[15,16] respectively, of 1,4-diselenane (**6a**), bis(phenylseleno)methane (**7a**) and 1,2-bis(phenylseleno)benzene (**8a**). The dibromide (**8d′**)[16] of **8a** is also proposed to be a TB. Since **6a** assumes a chair conformation, the distances between the chlorine atoms in **6c** may be long enough for the formation of a di-TB. The four C—Se bonds in **7a** and **8a** rotate easily to escape the steric repulsion when the halogen atoms approach the selenium atoms. In the case of selenanthrene (**3a**), it cannot offer an appropriate conformation for a TB structure when halogen atoms attack the selenium atom(s) potentially to give a TB, because of its rigid boat conformation.[17]



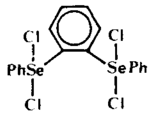
1c′



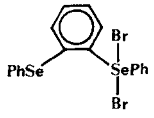
6c


$$\mathrm{Ph}\overset{\overset{\textstyle X}{|}}{\underset{\underset{\textstyle X}{|}}{\mathrm{Se}}}\mathrm{CH_2}\overset{\overset{\textstyle X}{|}}{\underset{\underset{\textstyle X}{|}}{\mathrm{Se}}}\mathrm{Ph}$$

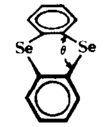
7c (X = Cl)



8c



8d'



3a

^{77}Se and ^{125}Te chemical shifts

While the formation of TBs in the reaction of the usual selenides or tellurides with halogens causes large downfield shifts[2,3c,e] in the ^{77}Se (ca 100–450 ppm) and ^{125}Te (ca 100–750 ppm) NMR spectra, the values are small if the adducts are MCs (less than ca 10 ppm).[2] As shown in Table 4, the ^{77}Se chemical shift of **1d** is only 2·0 ppm larger (downfield) than that of **1a**, similarly to the case of the MC of **4e**, the chemical shift of which is 2·8 ppm larger than that of **4a**.

An MC structure of the bromine adduct **1d** is confirmed, again based on the ^{77}Se chemical shifts. The chemical shift of **1d** relative to **1a** is less than 10 ppm, in addition to the case of **4e**. The ^{77}Se and ^{125}Te chemical shifts support the conclusion that other halogen adducts in Table 4, **2d**, **4c**, **5c**, **5d** and **5e**, are TBs.

It is noteworthy that the ^{125}Te chemical shift difference between the bromine adduct **2d** and **2a** ($\Delta\delta$ 101 ppm) is significantly smaller than that between the bromine adduct **5d** and **5a** ($\Delta\delta$ 166 ppm). This can be explained by assuming that the electron transfer from the tellurium atom to bromine is more difficult in **2d** than in **5d**. This observation is consistent with the fact that the bromine adduct **1d** is no longer a TB but an MC.

CONCLUSION

This present work has illustrated how a subtle structural change controls the stability of either a TB or an MC over the other in solution. A study of the structures of bromine adducts such as **1d** and **3d** and the chlorine adduct of **3a** in solids is in progress.

EXPERIMENTAL

Spectral measurement. The ^{1}H and ^{13}C NMR spectra were measured on a JEOL FX-60Q spectrometer operating at 60 and 15 MHz, respectively. The ^{77}Se and ^{125}Te NMR spectra were recorded on a JNM GX-400 spectrometer (JEOL) operated at 76 and 126 MHz, respectively. The conditions for measurements were almost identical with those in the preceding paper.[2] IR spectra were recorded on a Hitachi 295 infrared spectrometer.

Compounds. Selenoxanthone (**1a**),[18] its selenium oxide (**1f**),[19] telluroxanthone (**2a**),[20] selenanthrene diselenium dioxide (**3f**),[21] phenoxselenine (**4a**),[22] phenoxtellurine (**5a**)[23] and their halogen adducts[18,20,22] were prepared following literature procedures. The physical properties agreed well with those in the literature.

Selenoxanthone dichloride (**1c**). Excess of chlorine gas was bubbled through a dichloromethane solution (30 ml) of **1a**[18] (500 mg, 1·9 mmol) to give a pale yellow precipitate. Recrystallization from dechloromethane–hexane yielded pale yellow prisms, almost quantitatively; m.p. (decomp.) 205–220 °C. Found, C 47·07, H 2·24; calculated for $C_{13}H_8Cl_2OSe$, C 47·30, H 2·44%. ^{1}H NMR, $\delta(CDCl_3)$ 7·6–8·0 (6H, m), 8·5–8·8 (2H, m); IR (KBr), 1662 cm^{-1} ($\nu_{C=O}$).

Selenoxanthone dibromide (**1d**). To a dichloromethane solution (30 ml) of **1a**[18] (500 mg, 1·9 mmol) was added bromine (340 mg, 2·1 mmol) in carbon tetrachloride (2·5 ml), giving a dark red precipitate. Careful recrystallization from dichloromethane–hexane yielded dark red needles (83%), which decolorized at ca 125 °C and melted at 188–190 °C (cf. m.p. of **1a**, 191–192 °C). Found, C 37·31, H 1·56; calculated for $C_{13}H_8Br_2OSe$, C 37·27, H 1·92%. ^{1}H NMR, $\delta(CDCl_3)$ 7·3—7·7 (6H, m), 8·5–8·8 (2H, m); IR (KBr), 1658 cm^{-1} ($\nu_{C=O}$). The decolorized material was **1a** and the ^{1}H NMR spectrum of **1d** was almost identical with that of **1a**.

Telluroxanthone diiodide (**2e**). To a chloroform solution (10 ml) of **2a**[20] (100 mg, 0·33 mmol) was added iodine (99 mg, 0·39 mmol) in chloroform (5 ml), giving a reddish brown precipitate. After washing with hexane several times, **2e** (95%) was obtained, m.p. (decomp.) 215–216 °C. Found, C 27·5, H 1·6; calculated for $C_{13}H_8I_2OTe$, C 27·8, H 1·4%. ^{1}H NMR, δ(DMSO-d_6) 7·5–7·9 (4H, m), 8·2–8·6 (4H, m).

Selenanthrene (**3a**) was prepared by an improved method based on the literature.[24] In the course of the preparation, sodium diselenide was prepared from selenium and sodium borohydride in ethanol[25] containing sufficient aqueous sodium hydroxide not to evolve H_2Se.[12,25] (An adequate amount of sodium borohydride should be used; there is a typographical error in the amount reported earlier.[12])

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