Registry No. 1, 136847-07-9; 2, 136847-08-0; 2-PP, 7032-25-9; 2-PC, 7166-35-0; ClRe(CO)$_5$, 14099-01-5; 2-pyridinecarboxaldehyde, 1121-60-4; aniline, 62-53-3; cyclohexylamine, 108-91-8.

Contribution from the Departments of Chemistry, Oregon State University, Corvallis, Oregon 97331, and University of Reading, Whiteknights Park, Reading, England

Gas-Phase Electron-Diffraction Studies of the Molecular Structures of Tetrabromo(sulfido)tungsten(VI), WSBr$_4$, and Tetrabromo(seleno)tungsten(VI), WSeBr$_4$

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The molecular structures of WSBr$_4$ and WSeBr$_4$ at 220 and 250 °C, respectively, have been determined by gas-phase electron diffraction. The experimental data for both compounds are fitted by square-pyramidal models of C_{4v} symmetry in which the tungsten atom is above the plane of the bromine atoms. The bond lengths (r_a/Å) and bond angles ($\angle$/deg) with estimated 2σ uncertainties are as follows. WSBr$_4$: r(W=S) = 2.109 (11), r(W—Br) = 2.433 (3), $\angle$(S=W—Br) = 103.5 (7), $\angle$(Br—W—Br)$_{cis}$ = 86.9 (3), and $\angle$(Br—W—Br)$_{trans}$ = 153.0 (14). WSeBr$_4$: r(W=Se) = 2.220 (22), r(W—Br) = 2.427 (9), $\angle$(Se=W—Br) = 102.5 (9), $\angle$(Br—W—Br)$_{cis}$ = 87.3 (4), and $\angle$(Br—W—Br)$_{trans}$ = 154.9 (19). The structures of the series of nine WYX$_4$ molecules (Y = O, S, Se; X = F, Cl, Br) are discussed.

Introduction

We have previously determined the structures of WOF$_4$,[2] WSF$_4$,[3] WSeF$_4$,[4] WSCl$_4$,[5] WSeCl$_4$,[5] and WOBr$_4$[6,7] by gas-phase electron diffraction; other workers have measured WOCl$_4$.[8,9] In each case the data have been consistent with C_{4v} symmetry for the free molecules, although the experimental uncertainties would also allow small distortions from that symmetry. There are only limited single-crystal X-ray data for the compounds[10,11] because the crystals tend to be twinned or the structures disordered. Tetrabromo(sulfido)tungsten(VI) (WSBr$_4$) and tetrabromo(seleno)tungsten(VI) (WSeBr$_4$) remain the only compounds in the WYX$_4$ (X = F, Cl, Br; Y = O, S, Se) series for which gas-phase structures have not yet been reported. This article is an account of our electron-diffraction investigation.

Experimental Section

Samples of WSBr$_4$ and WSeBr$_4$ were prepared at Reading University as previously described.[12] Small quantities were loaded under an inert atmosphere into glass ampules fitted with break-seals. The ampules were evacuated, sealed, and taken to Oregon State University for the diffraction experiments. For an experiment an ampule was connected via glass tubing and a Swagelok coupling to the diffraction apparatus. The tubing between the break-seal and the apparatus was heated to ca. 150 °C under vacuum for 1.5 h before the break-seal was opened. At the end of the experiments no evidence for the formation of tungsten blue (an indication that the compounds had reacted or decomposed) was observed.

The diffraction photographs were made at nozzle temperatures of 220 °C (WSBr$_4$) and 250 °C (WSeBr$_4$). Other experimental conditions, the various analytical procedures, and the source of the electron-scattering amplitudes and phases were similar to those adopted for WSF$_4$.[3] The scattered intensity data (four plates each from the long and intermediate camera distances for both compounds) covered the ranges $2.00 \leq s/\text{Å}^{-1} \leq 13.75$ (long) and $6.00 \leq s/\text{Å}^{-1} \leq 33.75$ (intermediate) at intervals $\Delta s = 0.25$ Å^{-1}. The total intensity curves (s^4I_t), the calculated backgrounds, the average intensity curves ($sI_m(s)$), the theoretical curve that gives the best fit to the data, and the difference curves for the compounds are shown in Figures 1 and 2. The total and averaged intensity data and the calculated backgrounds are available as supplementary material.

Experimental radial distribution curves, calculated in the usual manner from $I'(s) = sI_m(s)Z_WZ_{Br}(A_WA_{Br})^{-1} \exp(-0.0025s^2)$, are shown in Figure 3. These curves are consistent with C_{4v} symmetry for the molecules, as is the case for the other WYX$_4$ compounds.

Results from preliminary refinements of the structures of both compounds suggested that the samples of each contained an impurity. In view of the known propensity of sulfido and seleno halides to react with oxygenated species, the most likely impurity was judged to be WOBr$_4$. This conclusion is supported by IR matrix studies carried out on samples of WSBr$_4$ and WSeBr$_4$[13] from the same preparations used for the electron-diffraction experiments. Accordingly, models that allowed for the presence of WOBr$_4$ in each of our materials were formulated. In each case the parameters chosen to define the system were the bond lengths r(W=Y) and r(W—Br), the bond angle $\angle$(Y=W—Br) (Y = S, Se), the five vibrational amplitudes corresponding to the interatomic distances in molecules of assumed C_{4v} symmetry, and a composition parameter to represent the presumed WOBr$_4$ impurity (which was assigned the structure and vibrational amplitudes previously determined[6]). The

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Table I. Structural Results for WSBr₄, WSeBr₄, and Similar Molecules[a]

	WSeBr₄	WSBr₄	WOBr₄	WSeCl₄	WSCl₄	WOCl₄	WSeF₄	WSF₄	WOF₄
$r(W=Y)$	2.220 (22)	2.109 (11)	1.684 (6)	2.203 (4)	2.086 (6)	1.685 (15)	2.226 (7)	2.104 (7)	1.666 (7)
$r(W—X)$	2.427 (9)	2.433 (3)	2.437 (3)	2.284 (3)	2.277 (3)	2.280 (3)	1.853 (5)	1.847 (3)	1.847 (2)
$r(Y·X)$	3.623 (32)	3.568 (19)	3.255 (24)	3.545 (9)	3.445 (12)	3.117 (35)	3.239 (11)	3.128 (22)	2.786 (12)
$r(X·X)_{cis}$	3.346 (15)	3.343 (10)	3.361 (14)	3.129 (6)	3.121 (8)	3.146 (15)	2.526 (10)	2.528 (13)	2.525 (8)
$r(X·X)_{trans}$	4.736 (21)	4.730 (14)	4.754 (20)	4.425 (9)	4.414 (11)	4.449 (21)	3.572 (11)	3.575 (18)	3.570 (11)
$\angle Y=W—X$	102.5 (9)	103.5 (7)	102.8 (11)	104.4 (3)	104.2 (5)	102.4 (13)	105.0 (7)	104.5 (11)	104.5 (6)
$\angle(X—W—X)_{cis}$	87.3 (4)	86.9 (3)	87.2 (5)	86.5 (2)	86.5 (2)	87.3 (5)	86.1 (4)	86.6 (5)	86.2 (3)
$\angle(X—W—X)_{trans}$	154.9 (19)	153.0 (14)	154.4 (22)	151.2 (6)	151.6 (10)	155.2 (26)	150.0 (14)	151.0 (22)	151.0 (12)
$l(W=Y)$	0.065 (23)	0.045 (8)	0.039 (5)	0.055 (4)	0.031 (6)	0.045 (11)	0.050 (8)	[0.033][b]	[0.036][b]
$l(W—X)$	0.078 (9)	0.073 (5)	0.074 (5)	0.060 (3)	0.058 (4)	0.052 (4)	0.037 (6)	0.055 (4)	0.041 (4)
$l(Y·X)$	0.169 (42)	0.155 (41)	0.137 (34)	0.147 (9)	0.147 (17)	0.095 (38)	0.109 (9)	0.121 (19)	0.097 (17)
$l(X·X)_{cis}$	0.126 (19)	0.129 (13)	0.144 (8)	0.166 (8)	0.114 (10)	0.122 (28)	0.107 (24)	0.107 (20)	0.094 (14)
$l(X·X)_{trans}$	0.171 (64)	0.141 (23)	0.148 (15)	0.116 (19)	0.120 (25)	0.118 (20)	0.064 (27)	0.091 (45)	0.083 (29)
X	0.61 (11)[c]	0.35 (11)[c]						0.23 (6)[d]	
R[e]	0.098	0.081		0.064	0.112			0.109	
ref	this work	this work	6	5	5	8	4	3	2

[a] Distances (r_a) and amplitudes of vibration (l) in angstroms; bond angles ($\angle$) in degrees. [b] Assumed values. [c] Mole fraction of WOBr₄ as impurity. [d] Mole fraction of WOF₄ as impurity. [e] Quality of agreement factor. $R = [\sum_i w_i \Delta_i^2 / \sum_i w_i (s_i I_i(\text{obsd}))^2]^{1/2}$, where $\Delta_i = s_i I_i(\text{obsd}) - s_i I_i(\text{calcd})$.
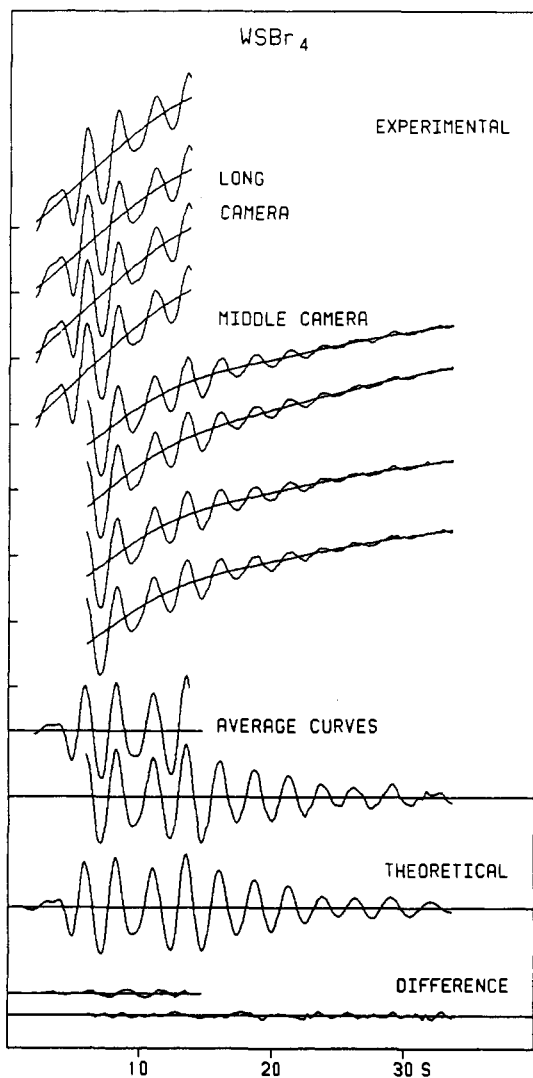

Figure 1. Intensity curves for the WSBr₄/WOBr₄ mixture. The $s^4 I_t$ experimental curves are shown magnified 7 times with respect to the backgrounds on which they are superimposed. The average curves are $s[s^4 I_t - \text{bkgd}]$. The theoretical curve is calculated from parameter values given in Table I.

Figure 2. Intensity curves for the WSeBr₄/WOBr₄ mixture. See caption to Figure 1.

structures were defined in terms of r_a-type distances; i.e., the effects of vibrational averaging were assumed to be negligible. The results of the refinements are shown as the first two data columns of Table I, and the correlation matrices are found in Table II.
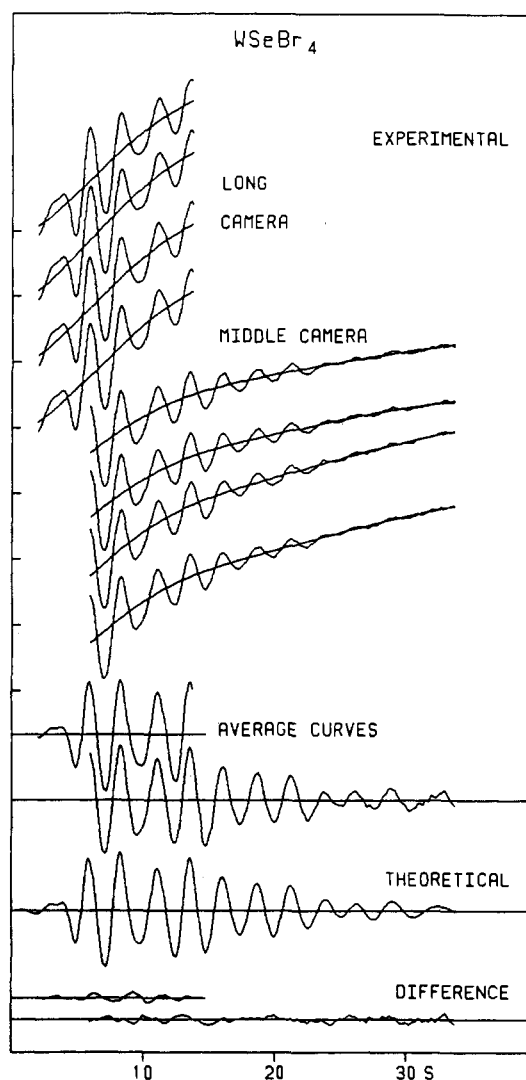
A surprising feature of the results was the apparently large amount of WOBr₄ found in the samples. Unfortunately, the bromine and tungsten analyses to which the samples were subjected do not provide an accurate measure of the amount of an impurity such as WOBr₄. In the matrix-isolation experiments mentioned above the presence of the oxo-bromide was detected, but it would appear from examination of the spectra that the amounts were small. We conclude that some oxo-

Table II. Correlation Matrices ($\times 100$) for Parameters of $WSBr_4$ and $WSeBr_4{}^a$

no.	param	$\sigma_{LS}(S)^b$	$\sigma_{LS}(Se)^b$	r_1	r_2	$\angle_3$	l_4	l_5	l_6	l_7	l_8	$X_9{}^c$
1	$r(W{=}Y)$	0.371	0.785	100	16	−11	3	−24	1	−6	−2	1
2	$r(W{-}Br)$	0.087	0.293	56	100	19	17	−5	4	−7	−2	9
3	$\angle(Y{=}W{-}Br)$	24.4	32.7	−14	27	100	4	4	−59	−53	8	1
4	$l(W{=}Y)$	0.292	0.803	2	39	24	100	4	12	9	4	33
5	$l(W{-}Br)$	0.137	0.296	−46	−24	11	28	100	14	26	11	34
6	$l(Y{\cdot}Br)$	1.42	1.45	13	34	<1	30	−10	100	81	<1	38
7	$l(Br{\cdot}Br)_{cis}$	0.428	0.654	−6	9	−3	18	12	69	100	1	32
8	$l(Br{\cdot}Br)_{trans}$	0.797	2.26	−7	−11	2	−5	8	−9	−4	100	11
9	X^c	3.54	3.77	13	63	33	62	−5	50	29	−10	100

^{a} Correlation coefficients for the sulfur and selenium compounds are respectively above and below the diagonal. ^{b} Standard deviations ($\times 100$) for the sulfur and selenium compounds. ^{c} Mole fractions of $WOBr_4$.

Table III. Steric and Electrostatic Effects in WYX_4 Molecules

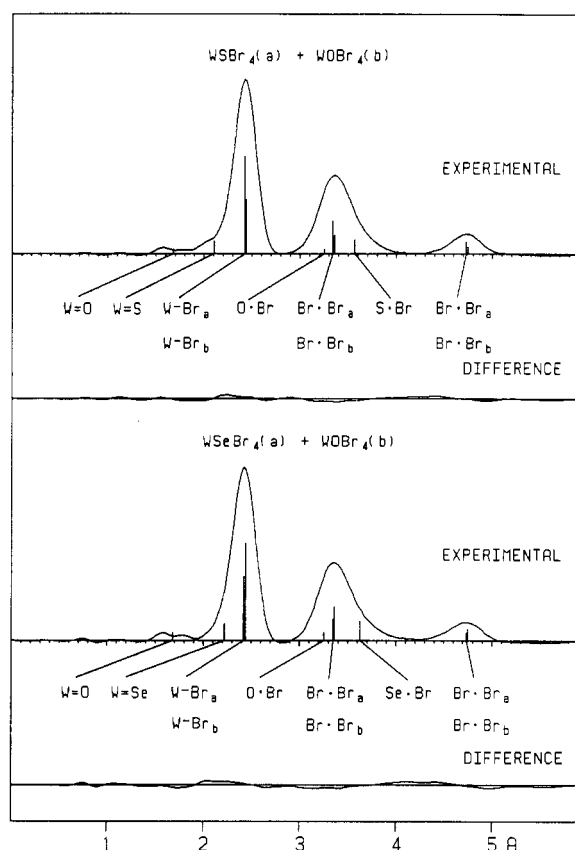|	$WSeBr_4$	$WSBr_4$	$WOBr_4$	$WSeCl_4$	$WSCl_4$	$WOCl_4$	$WSeF_4$	WSF_4	WOF_4
				Steric Effects^a					
$X{\cdot}Y$	0.35	0.25	0.10	0.25	0.20	0.10	0.20	0.05	−0.05
$X{\cdot}X_{cis}$	0.55	0.55	0.55	0.45	0.45	0.45	0.15	0.15	0.15
				Electrostatic Effects^b					
$X{\cdot}Y$	1.0	1.1	4.4	1.2	1.3	6.2	2.7	3.5	14.4
$X{\cdot}X_{cis}$	2.0	2.0	2.0	3.9	3.9	3.9	21	21	21

^{a} Values (in Å) are r(van der Waals) $- r_a$. $^b Q_iQ_j/r_{ij}$, where electronic charges Q are estimates from electronegativity differences.[15]

bromide must have been formed as the sample passed into the diffraction apparatus. We have found that $WOBr_4$ has volatility similar to those of $WSBr_4$ and $WSeBr_4$, whereas in the case of $WSCl_4$ and $WOCl_4$ the latter is much the more volatile.

Discussion

Although our assumption of C_{4v} symmetry for both our molecules is consistent with the diffraction data, small deviations from this symmetry cannot be ruled out. The allowable magnitudes of such possible deviations were not explored, but in a recent similar case, $CrOF_4$,[14] distortions from C_{4v} to C_{2v} represented by differences in the angles O=Cr—F of up to about 8° gave acceptable fits. Similar deviations in $WSBr_4$ and $WSeBr_4$ must also be regarded as likely to give acceptable fits. The following discussion is based on the assumption of C_{4v} symmetry for all WYX_4 molecules but is not dependent upon it. Were some of the structures to be slightly distorted, the parameter values listed for them would represent averages of those corresponding to the lower symmetry and our remarks would apply equally well.

A study of Table I reveals some noteworthy features of the structures of the nine WYX_4 molecules. First, there is the remarkable similarity of all bond angles of a given type throughout the group of nine molecules: The average value of $\angle Y{=}W{-}X$ is equal to 103.8° with an average (maximum) deviation of 0.8° (1.4°); corresponding values for $\angle(X{-}W{-}X)_{cis}$ are 86.7° and 0.4° (0.6°), and for $\angle(X{-}W{-}X)_{trans}$ the values are 152.5° and 1.7° (2.8°). These bond-angle similarities are at first glance surprising since, due to the quite different sizes of the ligand atoms, nonbond steric repulsions should be quite different from molecule to molecule. For example, in each of the three molecules in a group characterized by halogen ligands of a given type, the nonbond repulsions between halogens are equal, but those between the Y atom and the halogens vary significantly in a way that should lead to a decrease in the Y=W—X bond angle from Y = Se → S → O if steric effects were the only nonbond effects to operate. However, the electronegativity differences between tungsten and the ligand atoms leads to residual negative charges on the latter. We believe that the near constancy of the Y=W—X angle under these circumstances is due to a balancing of steric and electrostatic repulsion among the ligands. Table III provides a rough indication of the relative magnitudes of each of these types of interaction for the nine molecules. It is seen that, in each of

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Figure 3. Radial distribution curves for $WSBr_4/WOBr_4$ and $WSeBr_4/WOBr_4$. The experimental curves were calculated from composites of the average intensities with use of theoretical data for the region $0 \le s/\text{Å}^{-1} \le 1.75$, and $B/\text{Å}^2 = 0.0025$. The vertical lines indicate the interatomic distances and have lengths proportional to the distance weights.

the groups characterized by a given halogen ligand, the decrease in X·Y steric repulsion is accompanied by an increase in electrostatic repulsion. A similar correspondence between these effects is seen for the X·X terms for the groups of molecules characterized by a given Y atom: as the steric repulsion decreases in the order Br → Cl → F in such a group, the electrostatic repulsion correspondingly increases.

The W—X bond lengths are largely unaffected by changes in the Y ligand. Throughout the three groups of three compounds

each, these distances have average (maximum) deviations of 0.002 Å (0.005 Å) from their average values. On the other hand, the W=Y distances vary over a rather wider range: the average (maximum) deviations from their respective averages are 0.008 Å (0.013 Å). For reasons that are unclear, the W=Y variation is unsystematic. In WSF_4 and $WSeF_4$, for example, the W=S and W=Se distances are longer than in the corresponding chloro compounds, but the W=O distance in WOF_4 is shorter than it is in $WOBr_4$ and $WOCl_4$. Unfortunately, our measurements of the W=S and W=Se distances in $WSBr_4$ and $WSeBr_4$ have relatively large uncertainties that render the distances statistically indistinguishable from those in the related fluoro and chloro compounds.

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Molecular Structure of CrO₂(NO₃)₂ in the Gas Phase: A Novel Form of Coordination for Chromium?

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The gas-phase molecular structure of chromyl nitrate, $CrO_2(NO_3)_2$, has been studied by electron diffraction at a temperature of 50 °C and by ab initio methods at the HF level. The diffraction data are consistent with C_2 symmetry for the molecule. The coordination about Cr is best described as derived from a severely distorted octahedron, since the nitrate groups act as bidentate ligands which are asymmetrically bonded to Cr. Chromium forms two multiple bonds to O atoms (Cr=O), two nominal single bonds to O_N atoms (Cr—O), and two very long, presumably weak (estimated bond order 0.19–0.29) bonds (Cr←O) to other O_N atoms on each of the nitrate groups. Given the doubt about the existence of CrF_6, chromyl nitrate may be the only Cr(VI) species known to be six-coordinate. Principal distances ($r_g/Å$) and angles ($\angle/deg$), followed by estimated 2σ uncertainties in parentheses, are r(Cr=O) = 1.586 (2), r(Cr—O) = 1.957 (5), r(Cr←O) = 2.254 (20), r(N—O) = 1.341 (4), r(N=O$_{Cr}$) = 1.254 (4), r(N=O) = 1.193 (4), $\angle$(O=Cr=O) = 112.6 (35), $\angle$(O—Cr—O) = 140.4 (33), $\angle$(O=Cr—O$_4$) = 97.2 (18), $\angle$(O=Cr—O$_8$) = 104.5 (9), $\angle$(O=Cr←O) = 83.7 (34), $\angle$(Cr—O—N) = 97.5 (5), $\angle$(O—N=O$_{Cr}$) = 112.2 (1), $\angle$(O—N=O) = 119.7 (40), and $\angle$(O=N=O) = 128.1 (36). The Cr—O—NO₂ group is only slightly nonplanar with the dihedral angle of the planes Cr—O—N and NO₂ equal to 16 (3)°. It appears that there is relatively little torsional motion around either of the single bonds in the –O—NO₂ groups. Results of geometry optimization at the SCF level with a large all-electron basis agree reasonably well with those from the electron-diffraction experiment and strongly support the conclusions about the nature of the coordination of the Cr atom. Tests with a pseudopotential for Cr, however, led to the unreasonable value 1.40 Å for r(C=O).

Introduction

The set of rules known as valence-shell electron-pair repulsion theory (VSEPR)[2] is one of the great organizing themes of structural chemistry. While the rules are simple, they provide a foundation for predictions of molecular shapes in organic and main-group inorganic chemistry which are almost always reliable. Compounds for which the VSEPR rules fail are therefore of unusual interest. Recent work from this laboratory has shown that the bond angles in some d⁰ transition-metal compounds such as CrO_2F_2[3] and CrO_2Cl_2[4] do not obey the VSEPR rules. In both these molecules, the Cr=O (formally) double bonds are less sterically demanding than the Cr—F or Cr—Cl single bonds, whereas in the analogous sulfuryl halides the O=S=O angles exceed the X–S–X angles (X = F, Cl) by more than 20°.[5,6] However, it should not be assumed that the VSEPR rules always fail for transition-metal derivatives, as $TiCl_4$ is tetrahedral[7] and MoF_6

is octahedral;[8] both of these compounds also contain d⁰ metals.

Since nitrate groups have an unusually versatile coordination chemistry (no fewer than nine different coordination modes were identified in a review of nitrate complexes[9]), chromyl nitrate, $CrO_2(NO_3)_2$, poses particularly interesting structural problems over and above those associated with the VSEPR-like angular question already mentioned for the chromyl halides. In general,[9] nitrate acts as a bidentate ligand to metal ions, and its small "bite" of less than 2.2 Å allows metals to display unusually high coordination numbers; some examples include $[Co(NO_3)_4]^-$[10] and $Sn(NO_3)_4$,[11] which both have dodecahedral structures with 8-coordinate metals, and $[Y(NO_3)_5]^-$[12] with 10-coordinate[12] and $[La(NO_3)_6]^-$[13] with 12-coordinate metals. There are compounds in which all the coordinated nitrates act as monodentate ligands, such as tetrahedral $[Au(NO_3)_4]^-$[14] and others in which some of the nitrate groups are bidentate and some monodentate, such as $[Sc(NO_3)_5]_2^-$,[13] in which Sc is 9-coordinate. In bidentate examples

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