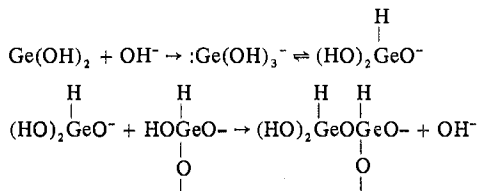correlated with the solubility—the dark material having a lower solubility than the light material.

These results indicate that ordinary germanium(II) hydroxide reacts with the hydroxide ion to form a soluble species, perhaps a complex such as $OGeH(OH)_2^-$. The conversion of $Ge(OH)_2$ to the relatively insoluble $(HGe)_2O_3$ appears to be catalyzed by this dissolved species or by hydroxide ions, perhaps through a mechanism such as the following.[12-15]

$$Ge(OH)_2 + OH^- \rightarrow :Ge(OH)_3^- \rightleftharpoons \overset{\overset{\text{H}}{|}}{(HO)_2GeO^-}$$

$$\overset{\overset{\text{H}}{|}}{(HO)_2GeO^-} + \overset{\overset{\text{H}}{|}}{HOGeO-} \rightarrow \overset{\overset{\text{H}}{|}}{(HO)_2}\overset{\overset{\text{H}}{|}}{GeOGeO-} + OH^-$$
$$\underset{|}{O} \qquad\qquad \underset{|}{O}$$

Experimental Section

Germanium(II) hydroxide was precipitated by the addition of aqueous ammonia to a solution of Ge(II) in hydrochloric acid.[16] This and all subsequent operations were carried out under a nitrogen atmosphere. The $Ge(OH)_2$ was collected on a sintered-glass funnel and washed with deoxygenated water. The conversion to $(HGe)_2O_3$ was effected most conveniently by stirring damp $Ge(OH)_2$ (prepared from 5 g of GeO_2) with 35 mL of 10 M NaOH at 70 °C for 1 h. The resulting dark brown solid was washed on a coarse sintered-glass funnel with deoxygenated water until the filtrate was neutral. The sample was dried to constant weight by pumping through a liquid-nitrogen trap (20 h generally sufficing). To avoid splattering of the sample, it was cooled to 0 °C during the initial pumping; yield 1.2 g (26%).

The purity of the $Ge(OH)_2$ was established by determining the equivalents of reducing power per mole of germanium. The hydrogen evolved during digestion of the material in 10 M NaOH at 120 °C for 3 days, followed by analysis for Ge by the mannitol method,[17] corresponded to 1.996 equiv/mol (theory, 2.000 equiv/mol). The $(HGe)_2O_3$ was analyzed for Ge by the mannitol titration method (after treatment with acid and peroxide). Anal. Calcd: Ge, 74.4. Found: Ge, 75.7.

Infrared spectra were obtained using mulls in Nujol or chlorofluorocarbon oil (Series 14–25, Halocarbon Products Corp., Hackensack, N.J.) between NaCl or KBr plates and a Perkin-Elmer Model 337 grating infrared spectrophotometer. Raman spectra were obtained using a Coherent Radiation Co. spectrometer (Model 1401).

Pyrolyses of damp samples of $Ge(OH)_2$ and $(HGe)_2O_3$ were carried out in a glass tube in a tube furnace. The samples were heated while continuously Toepler-pumping the evolved hydrogen through liquid nitrogen cooled traps. Germanium in the pyrolyzed residues was determined as described above. The results are presented in Table II.

All manipulations involved in the solubility measurements were carried out under nitrogen. Each $Ge(OH)_2$ slurry was stirred for a measured time; a portion of the slurry was centrifuged, and an aliquot of the clear supernatant was acidified, treated with excess standard triiodide solution, and titrated with thiosulfate.

Registry No. $(HGe)_2O_3$, 63937-01-9; $Ge(OH)_2$, 12024-99-6.

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(25) less three times the inductive factor for oxygen, i.e., $25 - (3 \times 4) = 13$. The pK of an O–H proton of $HGe(OH)_3$ can be estimated to be equal to the pK of $Ge(OH)_4$[15] (8.6) plus a correction of 4/2.8 for the replacement of an OH group by H, i.e., 8.6 + 1.4 = 10.0. Although these estimates are very rough, they do indicate that the $OGeH(OH)_2^-$ tautomer should have a stability at least comparable to that of the $Ge(OH)_3^-$ tautomer.
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X-Ray Crystal and Molecular Structure of Tetrakis(N,N-dimethyldithiocarbamato)-μ-nitrido-μ-(N,N-dimethyldithiocarbamato)-diosmium(IV)

K. W. Given and L. H. Pignolet*

There have been only a few reports of dithiocarbamato complexes of osmium.[1-4] Of these, the ones which are well characterized generally contain tertiary phosphine ligands[1,2] and in one case a tris(dithiocarbamato)osmium(III) complex has been claimed;[3] however, this compound is not well characterized. We report here the synthesis, characterization, and x-ray structure of a novel symmetric μ-nitrido-diosmium(IV) complex with the formula $Os_2N[S_2CN(CH_3)_2]_5$ where one dtc ligand also bridges the two osmium atoms. Only a few nitrido complexes of osmium are known[5-7] and none with the Os–N–Os bridge has been structurally characterized. This complex is the first well-characterized osmium–nitrido complex which contains dithio-acid ligands and it is the only example of a M–N–M complex which contains a second bridging ligand.

Experimental Section

Spectra. ^{1}H NMR spectra were recorded at 79.54 MHz using a Varian CFT 20 spectrometer. IR spectra were recorded in KBr disks with a Perkin-Elmer Model 237 grating spectrophotometer. Electronic absorption spectra were obtained in CH_2Cl_2 solution at 25 °C with a Cary Model 14 spectrophotometer. Magnetic susceptibilities were determined by the Faraday method at 25 °C using $Hg[Co(SCN)_4]$ as calibrant. Conductivity experiments were carried out using a Yellow Springs Instrument Co. Model 31 conductivity bridge. Reagent grade nitromethane was purified by double distillation from anhydrous $CaCl_2$ onto anhydrous $CaSO_4$ under nitrogen. Mass spectra were obtained using an AEI MS-30 mass spectrometer and samples were introduced using the solid-sample probe. All spectra were measured at an ion source temperature of 200 °C or less and an ionization energy of 70 eV.

Preparation and Characterization of the Compounds. Os_2N-$(S_2CNR_2)_5$, **Where R = CH_3 or C_2H_5.** In an attempt to synthesize new osmium dithiocarbamato complexes, the reaction between $OsCl_3\cdot xH_2O$ (ROC/RIC) and $Na(R_2dtc)$, where R = CH_3 or C_2H_5, was carried out in refluxing CH_3CN solution for 1 h under a N_2 atmosphere. Column chromatography of the residue using alumina for R = CH_3 and silica gel for R = C_2H_5 and CH_2Cl_2 as eluent gave a 70% yield of an orange-brown complex. Crystallization was achieved from CH_2Cl_2–heptane. The compounds are diamagnetic in solid and solution, nonconducting in nitromethane at 25 °C (Λ = 0.35 and 0.56 Ω^{-1} cm^2 mol^{-1} for R = CH_3 and C_2H_5, respectively), and benzene soluble and the R = CH_3 compound has five equally intense singlet ^{1}H NMR signals (τ (in CD_2Cl_2 solvent) 6.25, 6.68, 6.73, 6.74, 6.79).

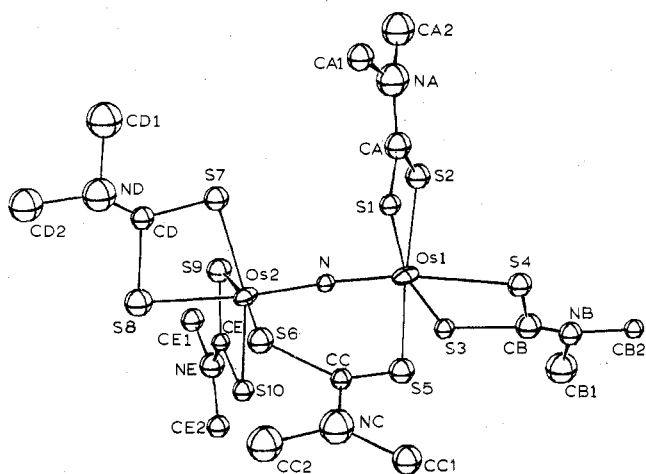**Figure 1.** ORTEP drawing of the molecule $Os_2N[S_2CN(CH_3)_2]_5$. Ellipsoids are drawn at the 30% probability level.

The R = C_2H_5 compound has a complex ^1H NMR spectrum. Elemental analyses of vacuum-dried samples are consistent with the formulation $Os_2N(S_2CNR_2)_5$. Anal. Calcd for $C_{15}H_{30}N_6Os_2S_{10}$: C, 18.11; H, 3.00; N, 8.45. Found for R = CH_3: C, 18.25; H, 3.04; N, 8.45. Anal. Calcd for $C_{25}H_{50}N_6Os_2S_{10}$: C, 26.44; H, 4.44; N, 7.40. Found for R = C_2H_5: C, 26.40; H, 4.78; N, 7.42. Infrared spectra recorded in a KBr disk showed the presence of a strong band ν_{Os_2N}(asym) 1052 cm^{-1} which is expected for a μ-nitrido bridge.[5,8] Mass spectral analysis is entirely consistent with the above formulation with the major fragments being $OsN[S_2CN(C_2H_5)_2]_2^+$ (m/e 502), $Os[S_2CN(C_2H_5)_2]_2[SCN(C_2H_5)_2]^+$ (m/e 604), and $Os[S_2CN(C_2H_5)_2]_3^+$ (m/e 636).[9] The m/e values reported correspond to the ^{192}Os isotope (see ref 9 for a discussion of the mass spectra of dtc complexes). The solution molecular weight determined by osmometry using CHCl$_3$ at 37 °C is consistent with the above formulation, (found, 987 ± 30; required for R = CH$_3$, 995). Electronic absorption spectra (λ_{max}, nm (log ϵ)) for R = CH$_3$ 252 (4.88), 330 (4.48), 361 sh (4.41), 495 sh (3.05); for R = C$_2$H$_5$ 254 (4.82), 331 (4.40), 363 sh (4.33), 496 sh (2.99).

Structure Determination of $Os_2N[S_2CN(CH_3)_2]_5 \cdot C_7H_{16}$. Suitable crystals always contained heptane of crystallization which rapidly evaporated upon removal from the mother liquor thus destroying the crystals. Therefore, it was necessary to quickly seal crystals in epoxy before x-ray analysis. Great difficulty was experienced in finding a large suitable crystal so a nonideal tiny crystal was finally used for data collection. The dimensions of the small needle used for data collection were 0.03 × 0.10 × 0.04 mm. The crystal was mounted with the *b* axis parallel to the spindle axis. The cell constants, a = 17.294 (6) Å, b = 10.062 (3) Å, c = 21.539 (9) Å, β = 95.54 (4)°, and V = 3731 Å^3, were determined by a least-squares refinement using θ-angle values for 12 Mo Kα (λ 0.707 Å) peaks carefully centered on the diffractometer. The measured density, 2.02 g/cm^3, agrees with the calculated density, 1.95 g/cm^3, for Z = 4. Systematic extinctions indicated the space group to be $P2_1/c$.

Intensity data were collected on a four-circle Hilger and Watts automatic diffractometer using Zr-filtered Mo Kα radiation. Of the 3515 independent reflections collected out to θ = 20°, 1130 with F_o^2 > 3$\sigma(F_o^2)$ were used in solution and refinement of the structure. Two standard reflection intensities were checked at intervals of 20 sequential reflections and no changes greater than 4% from the average value and no trends with time were noted.

The data were processed in the manner described by Corfield, Doedens, and Ibers,[10] using 0.04 for p in the $\sigma(I)$ equation. The intensity data were corrected for Lorentz and polarization effects as well as for absorption (μ = 90.9 cm^{-1}). Conventional heavy-atom techniques, used to solve the structure and refinement[11] with isotropic temperature factors for all nonhydrogen atoms except the osmiums which were refined anisotropically by full-matrix least-squares methods, converged R and r to their final values of 0.10 and 0.032, respectively.[12] The standard deviation of an observation of unit weight is 1.56. Six disordered carbons of the heptane of crystallization were located and refined in all but the last three cycles. In the final difference Fourier, the highest peak was 1.9 Å^{-3} in the vicinity of the osmium atoms. The next highest peak was 0.9 Å^{-3} in the vicinity of the disordered heptane

Table I

Final Atomic Fractional Coordinates and Thermal Parameters with Standard Deviations

Atom	x	y	z	B, Å^2
Os(1)	0.1220 (3)	0.1857 (3)	−0.1230 (2)	4.5^a
Os(2)	0.2629 (3)	0.2232 (3)	−0.2269 (2)	4.4^a
N	0.1829 (18)	0.2077 (20)	−0.1833 (12)	3.0 (10)
S(1)	0.1800 (14)	−0.0235 (21)	−0.0934 (10)	3.9 (6)
S(2)	0.0388 (16)	0.0212 (25)	−0.1725 (11)	5.7 (7)
S(3)	0.0344 (13)	0.3699 (21)	−0.1432 (10)	3.9 (6)
S(4)	0.0405 (13)	0.1971 (24)	−0.0358 (9)	4.9 (6)
S(5)	0.2074 (15)	0.3214 (25)	−0.0590 (10)	5.6 (6)
S(6)	0.3472 (14)	0.2198 (25)	−0.1301 (10)	5.6 (6)
S(7)	0.2848 (15)	−0.0083 (25)	−0.2421 (11)	5.4 (7)
S(8)	0.3847 (15)	0.1989 (27)	−0.2689 (11)	6.7 (7)
S(9)	0.1969 (15)	0.2571 (25)	−0.3482 (11)	5.7 (7)
S(10)	0.2559 (16)	0.4626 (22)	−0.2433 (11)	4.5 (6)
N(A)	0.0895 (54)	−0.2237 (98)	−0.1611 (37)	9.5 (24)
N(B)	−0.0770 (47)	0.3895 (67)	−0.0590 (30)	4.6 (18)
N(C)	0.3563 (52)	0.3274 (82)	−0.0162 (37)	8.8 (23)
N(D)	0.4109 (71)	−0.0522 (99)	−0.2895 (42)	10.0 (29)
N(E)	0.1835 (44)	0.5190 (77)	−0.3596 (33)	6.4 (20)
C(A)	0.1066 (58)	−0.0927 (99)	−0.1422 (41)	6.5 (25)
C(B)	−0.0193 (64)	0.3455 (97)	−0.0715 (40)	7.1 (27)
C(C)	0.3033 (45)	0.2852 (79)	−0.0650 (31)	4.0 (18)
C(D)	0.3571 (54)	0.0236 (85)	−0.2823 (34)	4.3 (21)
C(E)	0.2099 (45)	0.4180 (73)	−0.3148 (34)	3.1 (19)
C(A1)	0.1684 (48)	−0.3225 (89)	−0.1365 (35)	6.2 (23)
C(A2)	0.0339 (65)	−0.2984 (110)	−0.2007 (48)	10.0 (31)
C(B1)	−0.1117 (67)	0.5147 (113)	−0.0963 (50)	9.8 (31)
C(B2)	−0.1069 (45)	0.3586 (73)	0.0016 (35)	4.2 (20)
C(C1)	0.3262 (44)	0.3844 (71)	0.0443 (31)	3.9 (18)
C(C2)	0.4477 (119)	0.3259 (215)	−0.0368 (82)	23.6 (79)
C(D1)	0.3988 (63)	−0.2163 (120)	−0.2879 (45)	10.5 (33)
D(D2)	0.4921 (94)	−0.0545 (140)	−0.3163 (64)	14.0 (47)
C(E1)	0.1405 (58)	0.4687 (89)	−0.4255 (43)	6.4 (25)
C(E2)	0.1966 (47)	0.6552 (86)	−0.3424 (35)	5.1 (21)
C(S1)^b	0.6302	0.0413	−0.0392	8.7
C(S2)^b	0.5825	−0.0130	−0.1103	10.6
C(S3)^b	0.5054	−0.0119	−0.1283	11.9
C(S4)^b	0.4578	−0.1558	−0.1254	13.5
C(S5)^b	0.5844	−0.0038	−0.1821	11.3
C(S6)^b	0.6297	0.1934	−0.4222	14.2

Anisotropic Thermal Parameters ($\times 10^4$)^c

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Os(1)	60 (3)	49 (5)	23 (1)	13 (3)	0 (1)	5 (2)
Os(2)	55 (2)	67 (5)	22 (1)	5 (1)	4 (1)	−3 (2)

^{a} Refined anisotropically; equivalent isotropic B is listed. ^{b} Heptane solvent carbon atoms not refined during final least-squares cycles due to disorder. ^{c} The form of the anisotropic thermal ellipsoid is $\exp[-(\beta_{11}h^2 + \ldots + 2\beta_{12}hk + \ldots)]$.

carbon atoms, compared with 4 Å^{-3} for a terminal methyl carbon atom in a Fourier map.

The final atomic coordinates with their standard deviations and the final thermal parameters with their standard deviations are given in Table I. Figure 1 presents an ORTEP perspective of the molecular structure and shows the labeling scheme. A table of observed and calculated structure factors is available (supplementary material).

Results and Discussion.

The analytical and spectroscopic characterization data presented in the Experimental Section clearly establish the molecular formula to be $Os_2N(S_2CNR_2)_5$ for R = CH$_3$ or C$_2$H$_5$. An x-ray diffraction study of the R = CH$_3$ complex was undertaken because the exact stereochemistry could not be determined by conventional spectroscopy and because no μ-nitrido-diosmium(IV) compounds have previously been characterized. The structure consists of well-separated bimetallic molecules each possessing the Os–N–Os linkage, four terminal dtc chelating ligands, and a novel μ-dtc ligand which bridges the two osmium atoms. The stereochemistry of the molecule is best viewed in the stereoscopic drawing shown in Figure 2. Selected bond lengths and angles in the Os_2NS_{10}

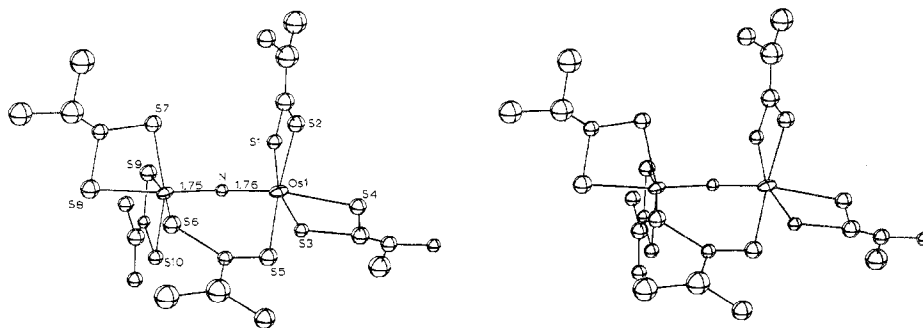**Figure 2.** ORTEP stereoview of the molecule $Os_2N[S_2CN(CH_3)_2]_5$. Ellipsoids are drawn at the 30% probability level.

Table II. Interatomic Distances and Angles in the Os_2NS_{10} Core

Distances, Å

Os(1)-N	1.76 (3)	S(1)-S(2)	2.87 (3)
Os(2)-N	1.75 (3)	S(3)-S(4)	2.89 (3)
Os(1)-Os(2)	3.485 (5)	S(5)-S(6)	3.15 (4)
Os(1)-S(1)	2.39 (2)	S(7)-S(8)	2.80 (4)
Os(1)-S(2)	2.38 (2)	S(9)-S(10)	2.88 (3)
Os(1)-S(3)	2.41 (2)	N-S(1)	3.03 (4)
Os(1)-S(4)	2.46 (2)	N-S(2)	3.15 (4)
Os(1)-S(5)	2.36 (2)	N-S(3)	3.23 (4)
Os(2)-S(6)	2.43 (2)	N-S(5)	2.91 (4)
Os(2)-S(7)	2.39 (3)	N-S(6)	2.96 (4)
Os(2)-S(8)	2.38 (3)	N-S(7)	3.14 (4)
Os(2)-S(9)	2.39 (2)	N-S(9)	3.20 (3)
Os(2)-S(10)	2.44 (2)	N-S(10)	3.19 (4)

Angles, Deg

Os(1)-N-Os(2)	164.6 (18)	N-Os(1)-S(4)	170.0 (11)
S(1)-Os(1)-S(2)	74.2 (8)	N-Os(2)-S(8)	165.1 (11)
S(3)-Os(1)-S(4)	72.8 (7)	S(1)-Os(1)-S(3)	165.1 (8)
S(7)-Os(2)-S(8)	72.0 (8)	S(7)-Os(2)-S(10)	162.1 (8)
S(9)-Os(2)-S(10)	73.3 (8)	S(2)-Os(1)-S(5)	169.8 (8)
S(5)-Os(1)-N	88.5 (11)	S(6)-Os(2)-S(9)	169.1 (8)
S(6)-Os(2)-N	88.7 (11)		

core and their estimated standard deviations are listed in Table II. The bond lengths and angles in the ligands are in good agreement with those of other dtc complexes although sizeable standard deviations in these parameters were observed due to a combination of the small crystal size and the presence of the heavy osmium atoms which dominated the x-ray scattering.

The two osmium atoms have distorted octahedral OsS_5N coordination cores which are structurally similar to the $RuCl_4ON$ and $OsCl_5N$ cores in $[Ru_2NCl_8(H_2O)_2]^{3-}$ [13,14] and $K_2[OsCl_5N]$,[15] respectively. A major distortion is an opening up (from 90°) of the $N-Os-S_{CNB}$ (CNB = cis nonbridging) angles (average = 98°) caused by nonbonded $N-S_{CNB}$ repulsions. The short Os–N distance of 1.75 Å would result in close $N-S_{CNB}$ distances < 3.0 Å if the $N-Os-S_{CNB}$ angles were 90°; hence the larger angles result in an average $N-S_{CNB}$ distance of 3.16 Å. See ref 15 for a complete discussion of this phenomenon in various nitrido complexes. The bridging dtc ligand imposes an additional more obvious distortion not found in other μ-nitrido complexes[5] and results in a nonlinear Os–N–Os linkage (Os–N–Os angle = 165 (2)°). The bridging dtc ligand which has a S–S intraligand bite distance of 3.15 Å (compared to the average value of 2.96 Å for the other dtc ligands) in effect forces the molecule to fold around the nitrido atom. This prevents the bridging S_B atoms from being repelled by N and results in an average $N-S_B$ distance of only 2.94 Å and an average $N-Os-S_B$ angle of 89°.

The short Os–N distances (1.75 Å) indicate significant multiple bonding as expected in nitrido complexes[5] (Ru–N distance of 1.72 Å and Os–N distance of 1.61 Å in the above examples[13-15]). The distances and angles in the ligands are not exceptional except that the bridging dtc ligand has an unusually large S–S bite distance of 3.15 Å. It is surprising that the dtc ligand can span the two osmium atoms which are

separated by 3.49 Å. This is the longest span distance yet observed for 1,1-dithiolate ligands and is an example of the great bonding flexibility of sulfur donor ligands. All of the Os–S distances are within 2σ of the mean (2.40 Å) and compare favorably with the mean Ru–S distances found in various dtc complexes of ruthenium (2.40, 2.38, and 2.41 Å in $ClRu(Et_2dtc)_3$,[16] $Ru(Et_2dtc)_3$, and β-$[Ru_2(Et_2dtc)_5]BF_4$,[18] respectively.

The bridging dtc chelate ring is puckered; however, the molecule has an approximate C_2 symmetry axis which contains NC, CC, and N and accounts for the five-line ^1H NMR spectrum (S_2C-N bond rotation is slow).

The source of the nitrido N atom is of some concern and interest. It most likely comes from the excess dtc$^-$ ligand used in the preparation of the complexes. Work in progress is directed to this question.

Registry No. $Os_2N(S_2CN(CH_3)_2)_5$, 63866-82-0; $Os_2N(S_2C-N(C_2H_5)_2)_5$, 63866-81-9.

Supplementary Material Available: Observed and calculated structure factor amplitudes (9 pages). Ordering information is given on any current masthead page.

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(12) $R = \sum||F_o| - |F_c||/\sum|F_o|$, $r = \sum w(|F_o|^2 - |F_c|^2)^2/\sum w|F_o|^4$; the numerator of r was the function minimized; the weights were $1/(\sigma(F^2))^2$ where $\sigma(F_o^2) = \sigma(I)/Lp$. Atomic scattering factors were taken from "International Tables for X-Ray Crystallography", Vol. III, Kynoch Press, Birmingham, England, 1962, Table 3.3.1A (S, C, N) and Table 3.3.1B (Os). Anomalous dispersion corrections from the same source (Table 3.3.2C) were used for Os and S.
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