

resolved into its two half-components and had to be placed at the mean position. Consequently, the listed values for distances and angles involving C(1) have little physical significance and certainly cannot be used to infer bond orders. We expect that C-O(*n*0) will have a lower bond order than the other two (O(*n*0) is involved in two Cu-O bonds rather than just one) and therefore will be longer. Valence shell repulsion furthermore predicts that angle O(*n*1)-C-O(*n*2) will be larger O(*n*0)-C-O(*n*1)/O(*n*2)⁷ unless there are external forces acting upon the group such as stereochemical requirements or hydrogen bonding.^{4,14}

As can be seen in Figure 1, the major vibration axes of Cl(10) and Cl(20) are not normal to the Cu-Cl-Cu plane, as would be expected, but lie in plane. This suggests that the Cl atoms might be disordered to a small extent, probably in concert with the orientation of the carbonate. Cl(10) would be expected to be located further from the center for carbonate

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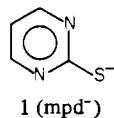
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A Dodecahedral, Eight-Coordinate Chelate Complex, Tetrakis(2-mercaptopyrimidinato)tungsten(IV)

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Considerable effort has been devoted to understanding the factors that govern ligand arrangements in eight-coordinate complexes.¹⁻³ One of the most common and most intensively studied polyhedra is the dodecahedron. When the ligands are chelating, and especially when they have a small bite, they tend to span the *m* edges,⁴ giving a structure of the *mmmm* type, although this is not invariably the case.⁵ If the chelating ligands are identical but unsymmetrical, there are six possible stereoisomers.¹ A consideration of metal-ligand σ - and π -bonding factors suggests that the π bonding will predominate³ in determining the preferences for sites A and B.⁴ It is not always easy to decide which of two ligating atoms has the greater π -donating or π -accepting ability, but for the ligand used in this work, the anion of 2-mercaptopyrimidine, **1**, it



seems clear that the mercaptide sulfur atom would be the better π donor and the aromatic nitrogen atom the better π

acceptor. For a d^2 metal ion, the arguments of Burdett, Hoffmann, and Fay³ suggest that the nitrogen atoms should be found in the B positions and the sulfur atoms in the A positions.

We have prepared and structurally characterized an M-(mpd)₄ molecule, where M is the d^2 species W^{IV}, and we find that the implied prediction of Burdett, Hoffmann, and Fay is obeyed.

Registry No. 1, 12329-65-6.

Supplementary Material Available: Tables of calculated H coordinates, deviations of Cu atoms from the planes through N atoms of dpt ligands, and structure factor amplitudes (43 pages). Ordering information is given on any current masthead page.

Table I. Crystallographic Parameters

space group	C2/c	cryst size, mm	0.30 × 0.30 × 0.30
<i>a</i> , Å	24.991 (3)	μ , cm ⁻¹	54.47
<i>b</i> , Å	14.611 (2)	2 θ range, deg	0-45
<i>c</i> , Å	14.112 (2)	no. of data	2285
$\alpha = \gamma$, deg	90.0	$F_o^2 > 2.5\sigma(F_o^2)$	2008
β , deg	111.52 (1)	no. of variables	279
<i>V</i> , Å ³	4794 (2)	<i>R</i> ₁	0.028
<i>d</i> _x , g/cm ³	1.639	<i>R</i> ₂	0.040
<i>Z</i>	8	esd	0.944
fw	591.53		

acceptor. For a d^2 metal ion, the arguments of Burdett, Hoffmann, and Fay³ suggest that the nitrogen atoms should be found in the B positions and the sulfur atoms in the A positions.

We have prepared and structurally characterized an M-(mpd)₄ molecule, where M is the d^2 species W^{IV}, and we find that the implied prediction of Burdett, Hoffmann, and Fay is obeyed.

Experimental Section

Materials. Tungsten carbonyl was purchased from Pressure Chemical Co., and 2-mercaptopyrimidine, Hmpd, was purchased from Aldrich Chemical Co., Inc. All other compounds and solvents were of reagent grade or better from commercial sources. Diglyme was dried over sodium and deoxygenated prior to use. Reactions were performed under argon by using standard Schlenk techniques.

Preparation of W(mpd)₄·1/2(diglyme). To 30 mL of dry diglyme were added 0.352 g (1 mM) of W(CO)₆ and 0.444 g (4 mM) of 2-mercaptopyrimidine. The mixture was then heated under reflux for ~16 h to give a brown-violet solution. The mixture was filtered, and slow evaporation of the mother liquor gave an essentially quantitative yield of mostly microcrystalline product in which there were one or two crystals suitable for X-ray diffraction.

X-ray Crystallography. A suitable crystal was secured to the end of a thin glass fiber with epoxy cement and mounted on a Syntex P1 automatic diffractometer. Procedures used for data collection as well as solution and refinement of the structure have been described

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Table II. Table of Positional and Thermal Parameters and Their Estimated Standard Deviations

atom	x	y	z	B ₁₁	B ₂₂	B ₃₃	B ₁₂	B ₁₃	B ₂₃
W(1)	0.07845 (1)	0.24688 (2)	0.03753 (2)	2.31 (1)	1.64 (1)	1.89 (1)	0.00 (1)	0.666 (8)	-0.24 (1)
S(1)	0.02025 (9)	0.3666 (1)	0.0855 (2)	3.09 (8)	2.33 (9)	3.54 (8)	0.01 (8)	1.29 (6)	-0.95 (8)
S(2)	0.14801 (9)	0.3525 (2)	0.1647 (2)	2.92 (9)	2.94 (9)	3.50 (9)	-0.22 (9)	0.62 (7)	-1.23 (9)
S(3)	0.07351 (10)	0.0746 (2)	0.0349 (1)	4.7 (1)	1.91 (8)	2.31 (8)	0.10 (8)	1.30 (7)	-0.05 (7)
S(4)	0.07086 (9)	0.1940 (1)	-0.1369 (2)	3.65 (8)	2.14 (8)	2.25 (7)	-0.04 (8)	1.21 (6)	-0.00 (7)
N(1)	-0.0141 (3)	0.2331 (4)	-0.0318 (4)	2.8 (2)	1.6 (3)	2.0 (2)	-0.3 (2)	0.8 (2)	-0.2 (2)
N(2)	-0.0913 (3)	0.3224 (5)	-0.0223 (6)	3.6 (3)	4.3 (4)	4.3 (3)	-0.0 (3)	2.2 (2)	-0.2 (3)
N(3)	0.1686 (3)	0.2180 (5)	0.0733 (5)	2.9 (3)	3.2 (3)	2.1 (3)	1.1 (3)	0.5 (2)	0.4 (2)
N(4)	0.2533 (3)	0.2887 (6)	0.1876 (6)	3.0 (3)	5.4 (4)	4.4 (3)	-0.3 (3)	1.1 (2)	-1.4 (3)
N(5)	0.0774 (3)	0.1816 (5)	0.1744 (5)	4.4 (3)	2.7 (2)	1.7 (2)	-0.3 (3)	1.6 (2)	-0.5 (2)
N(6)	0.0712 (4)	0.0269 (5)	0.2184 (5)	10.7 (5)	2.6 (3)	3.9 (3)	0.7 (3)	4.2 (3)	1.0 (3)
N(7)	0.0801 (3)	0.3528 (4)	-0.0678 (5)	2.6 (2)	1.7 (3)	2.5 (3)	-0.0 (2)	0.6 (2)	-0.0 (2)
N(8)	0.0716 (3)	0.3485 (5)	-0.2425 (5)	4.9 (3)	2.6 (3)	3.4 (3)	0.8 (3)	2.2 (2)	0.4 (3)
C(1)	-0.0357 (3)	0.3048 (6)	0.0036 (6)	2.9 (3)	2.1 (3)	2.9 (3)	0.5 (3)	1.4 (2)	0.1 (3)
C(2)	-0.1283 (4)	0.2648 (6)	-0.0870 (7)	3.4 (3)	4.3 (5)	3.3 (3)	-0.8 (4)	1.1 (3)	0.0 (4)
C(3)	-0.1101 (4)	0.1865 (6)	-0.1228 (6)	2.9 (3)	4.1 (4)	2.7 (3)	-1.0 (3)	0.9 (3)	0.6 (3)
C(4)	-0.0511 (4)	0.1734 (6)	-0.0965 (6)	3.6 (3)	3.0 (4)	2.0 (3)	-1.2 (3)	1.2 (2)	-0.1 (3)
C(5)	0.1983 (3)	0.2819 (6)	0.1443 (6)	2.6 (3)	2.9 (4)	2.8 (3)	-0.1 (3)	0.8 (3)	0.1 (3)
C(6)	0.2846 (4)	0.2259 (9)	0.1585 (8)	3.6 (4)	7.8 (7)	4.6 (5)	0.4 (5)	1.2 (3)	-0.1 (5)
C(7)	0.2586 (4)	0.1570 (8)	0.0880 (8)	3.7 (4)	5.9 (6)	5.1 (5)	0.9 (4)	1.3 (4)	-0.5 (5)
C(8)	0.1999 (4)	0.1556 (7)	0.0461 (7)	2.8 (3)	4.3 (4)	4.0 (4)	0.7 (3)	1.6 (3)	-0.5 (4)
C(9)	0.0736 (4)	0.0906 (6)	0.1540 (6)	4.0 (4)	1.9 (3)	3.0 (3)	0.1 (3)	1.4 (3)	0.1 (3)
C(10)	0.0710 (5)	0.0530 (7)	0.3086 (7)	11.1 (6)	3.6 (5)	4.8 (4)	-0.1 (5)	4.8 (4)	0.1 (4)
C(11)	0.0748 (5)	0.1442 (8)	0.3343 (7)	10.9 (7)	3.9 (5)	3.2 (4)	-0.3 (5)	3.4 (4)	-0.1 (4)
C(12)	0.0786 (4)	0.2104 (7)	0.2669 (7)	6.3 (5)	4.2 (5)	3.1 (4)	0.2 (4)	2.2 (3)	-0.2 (4)
C(13)	0.0740 (3)	0.3100 (6)	-0.1563 (6)	2.6 (3)	2.8 (4)	3.1 (3)	0.4 (3)	1.0 (3)	0.0 (3)
C(14)	0.0765 (4)	0.4389 (7)	-0.2406 (7)	5.5 (4)	3.8 (5)	4.8 (4)	0.9 (4)	2.5 (3)	1.0 (4)
C(15)	0.0838 (4)	0.4902 (6)	-0.1545 (7)	6.1 (4)	2.7 (4)	5.8 (4)	0.9 (4)	3.7 (3)	1.6 (3)
C(16)	0.0848 (4)	0.4457 (6)	-0.0654 (7)	3.9 (3)	2.1 (4)	4.1 (4)	-0.1 (3)	1.9 (3)	0.8 (3)

atom	x	y	z	B, Å ²	atom	x	y	z	B, Å ²
C(21)	0.2465 (10)	0.338 (2)	0.458 (2)	16.3 (8)	O(20)	0.2500 (0)	0.250 (0)	0.500 (0)	20.3 (11)
C(22)	0.2459 (10)	0.415 (2)	0.506 (2)	18.7 (11)	O(23)	0.2597 (9)	0.390 (2)	0.624 (2)	11.5 (7)
C(24)	0.2680 (9)	0.458 (2)	0.675 (2)	13.8 (7)	O(23')	0.2606 (13)	0.462 (2)	0.561 (3)	17.4 (11)

^a The form of the anisotropic thermal parameter is $\exp[-1/4(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}k lb^*c^*)]$.

Table III. Bond Distances for W(mpd)₄·1/2(diglyme)

atoms	dist, Å	atoms	dist, Å
W(1)-S(1)	2.521 (3)	N(6)-C(9)	1.32 (1)
-S(2)	2.517 (3)	-C(10)	1.33 (1)
-S(3)	2.520 (3)	N(7)-C(13)	1.35 (1)
-S(4)	2.520 (3)	-C(16)	1.36 (1)
-N(1)	2.166 (7)	N(8)-C(13)	1.32 (1)
-N(3)	2.162 (8)	-C(14)	1.32 (1)
-N(5)	2.164 (7)	C(2)-C(3)	1.39 (2)
-N(7)	2.156 (7)	C(3)-C(4)	1.40 (1)
S(1)-C(1)	1.71 (1)	C(6)-C(7)	1.40 (2)
S(2)-C(5)	1.73 (1)	C(7)-C(8)	1.37 (2)
S(3)-C(9)	1.697 (9)	C(10)-C(11)	1.38 (2)
S(4)-C(13)	1.72 (1)	C(11)-C(12)	1.38 (2)
N(1)-C(1)	1.36 (1)	C(14)-C(15)	1.38 (2)
-C(4)	1.35 (1)	C(15)-C(16)	1.41 (2)
N(2)-C(1)	1.32 (1)	O(23)-O(23)'	1.37 (5)
-C(2)	1.33 (1)	-C(22)	1.61 (4)
N(3)-C(5)	1.37 (1)	-C(24)	1.20 (3)
-C(8)	1.34 (1)	O(23)''-C(22)	1.00 (6)
N(4)-C(5)	1.29 (1)	-C(24)	1.55 (5)
-C(6)	1.36 (2)	O(20)-C(21)	1.40 (4)
N(5)-C(9)	1.36 (1)	C(21)-C(22)	1.32 (5)
-C(12)	1.36 (1)		

Table IV. Bond Angles for W(mpd)₄·1/2(diglyme)

atoms	angle, deg	atoms	angle, deg
S(1)-W(1)-S(2)	72.5 (1)	W(1)-N(7)-C(13)	106.3 (6)
-N(1)	63.9 (2)	-C(16)	136.2 (7)
-N(5)	82.5 (2)	C(13)-N(8)-C(14)	115.6 (9)
-N(7)	81.2 (2)	S(1)-C(1)-N(1)	108.6 (7)
S(2)-W(1)-N(3)	64.2 (2)	-N(2)	126.8 (8)
-N(5)	82.3 (2)	N(1)-C(1)-N(2)	124.6 (9)
-N(7)	82.5 (2)	N(2)-C(2)-C(3)	122 (1)
S(3)-W(1)-S(4)	72.1 (1)	C(2)-C(3)-C(4)	118.1 (9)
-N(1)	82.1 (2)	C(3)-C(4)-N(1)	119.0 (9)
-N(3)	81.4 (2)	S(2)-C(5)-N(3)	107.2 (7)
-N(5)	63.7 (2)	-N(4)	126.1 (8)
S(4)-W(1)-N(1)	81.1 (2)	N(3)-C(5)-N(4)	126.6 (9)
-N(3)	82.3 (2)	N(4)-C(6)-C(7)	122 (1)
-N(7)	63.9 (2)	C(6)-C(7)-C(8)	118 (1)
N(1)-W(1)-N(3)	159.5 (3)	C(7)-C(8)-N(3)	121 (1)
-N(5)	90.1 (3)	S(3)-C(9)-N(5)	108.7 (7)
-N(7)	92.5 (3)	-N(6)	127.0 (8)
N(3)-W(1)-N(5)	93.4 (3)	N(5)-C(9)-N(6)	124.3 (9)
-N(7)	91.1 (3)	N(6)-C(10)-C(11)	120 (1)
N(5)-W(1)-N(7)	160.3 (3)	C(10)-C(11)-C(12)	121 (1)
W(1)-S(1)-C(1)	82.1 (2)	C(11)-C(12)-N(5)	117 (1)
W(1)-S(2)-C(5)	82.5 (2)	S(4)-C(13)-N(7)	107.7 (7)
W(1)-S(3)-C(9)	82.2 (2)	-N(8)	125.2 (8)
W(1)-S(4)-C(13)	82.1 (2)	N(7)-C(13)-N(8)	127.2 (9)
W(1)-N(1)-C(1)	105.4 (6)	N(8)-C(14)-C(15)	123 (1)
-C(4)	135.8 (6)	C(14)-C(15)-C(16)	119 (1)
C(1)-N(2)-C(2)	117.4 (9)	C(15)-C(16)-N(7)	118 (1)
W(1)-N(3)-C(5)	106.0 (6)	C(21)-O(20)-C(21)'	180 (4)
-C(8)	136.9 (7)	O(20)-C(21)-C(22)	125 (3)
C(5)-N(4)-C(6)	116 (1)	C(21)-C(22)-O(23)	107 (4)
W(1)-N(5)-C(9)	105.4 (6)	-O(23)'	156 (6)
-C(12)	135.8 (7)	C(22)-O(23)-C(24)	112 (3)
C(9)-N(6)-C(10)	118.3 (9)	C(22)-O(23)''-C(24)	131 (6)

previously.⁶ The standard corrections were made to the data as well as an empirical absorption correction based on ψ scans at $\chi = 90^\circ$. The minimum, maximum, and average transmission factors are 0.938, 0.999, and 0.975, respectively. Pertinent crystallographic parameters for the compound are summarized in Table I.

Results and Discussion

Atomic positional and thermal parameters for W(mpd)₄·1/2(diglyme) are listed in Table II. Figure 1 shows the mo-

lecular structure and labeling scheme for the compound. Bond distances and angles are presented in Tables III and IV, re-

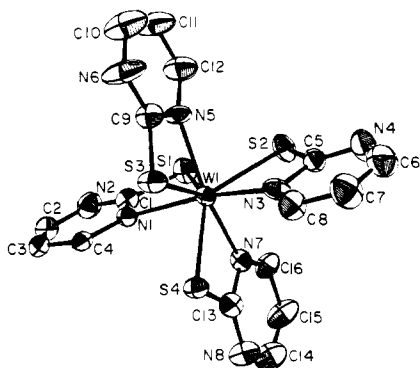


Figure 1. ORTEP drawing of the $W(mpd)_4$ molecules showing the numbering used in the tables. Each atom is represented by its ellipsoid of thermal vibration, scaled to enclose 30% of the electron density.

Table V

Equations of Planes ^a for $W(mpd)_4 \cdot 1/2$ (diglyme)					
P_1	S(3), W(1), N(5)	-0.9328	0.0456	-0.3576	-1.6592
P_2	S(1), W(1), N(1)	0.2987	0.5673	-0.7674	2.1958
P_3	S(2), W(1), N(3)	0.2916	0.6009	-0.7442	2.3160
P_4	S(4), W(1), N(7)	-0.9449	0.0731	-0.3190	-1.5626

Dihedral Angles, Deg			
P_1/P_2	88.8	P_2/P_3	2.4
P_1/P_3	88.8	P_2/P_4	89.8
P_1/P_4	2.8	P_3/P_4	89.7

^a The equation of the plane is of the form $AX + BY + CZ - D = 0$, where A , B , C , and D are constants and X , Y , and Z are orthogonalized coordinates.

Table VI. Lengths (Å) of m Edges

S(1)-N(1)	2.498 (7)	S(4)-N(7)	2.495 (8)
S(2)-N(3)	2.505 (8)	av	2.497 (5)
S(3)-N(5)	2.488 (8)		

spectively. Dihedral angles and equations for selected planes are presented in Table V.

The structure of $W(mpd)_4 \cdot 1/2$ (diglyme) was solved in the monoclinic space group $C2/c$. Each $W(mpd)_4$ molecule occupies a general position within the unit cell. The diglyme molecules sit on the inversion centers at $(1/4, 1/4, 1/2)$, $(3/4, 3/4, 1/2)$, $(3/4, 1/4, 0)$, and $(1/4, 3/4, 0)$ and show disordering of the O-CH₃ oxygen atoms. The tungsten atom is, of course, bonded to four mercaptopyrimidine ligands. Within experimental error the four ligands occupy two planes that are mutually perpendicular (see Table V) and contain the tungsten atom. The m edges⁴ are equal within experimental error (Table VI) with a mean value of 2.497 (5) Å.

The structure of the $W(mpd)_4$ molecule has D_{2d} virtual symmetry and can be described as a dodecahedron with the $mmmm$ arrangement of ligands. The A vertices are all occupied by sulfur atoms and the B vertices by ring nitrogen atoms. The angles⁴ θ_A and θ_B have the mean values 36.2 ± 0.1 and $80.0 \pm 0.2^\circ$. The former is very typical for all dodecahedral complexes while the latter is somewhat greater than the usual values, which are more commonly around 72° .

The excellent agreement of the observed ligand arrangement with that expected from the analysis of Burdett, Hoffmann, and Fay³ is very gratifying. It would be interesting to know if there would be a reversal of site preferences for a d^0 complex such as $Zr(mpd)_4$. If so, it might also be expected that the wrapping pattern would change to $g g g g$, as in the case⁵ of $Zr(8\text{-oxoquinoline})_4$, to minimize nonbonded contacts between the aromatic rings. We do not, however, plan such a study at present.

Acknowledgment. We are grateful to the Robert A. Welch Foundation for support under Grant No. A-494.

Registry No. $W(mpd)_4 \cdot 1/2$ (diglyme), 75701-22-3; $W(CO)_6$, 14040-11-0.

Supplementary Material Available: A table of observed and calculated structure factors (9 pages). Ordering information is given on any current masthead page.

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Stability Quotients of Some Lanthanide Cryptates in Aqueous Solutions

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The ability of certain diazapolyoxabicyclic ligands ("cryptands") to form inclusion complexes ("cryptates") containing various alkali, alkaline-earth, transition-metal, and heavy-metal ions has been established by structural and thermodynamic studies.¹ The early reports² noted a variation in the stability of cryptates formed with a series of ions, e.g., the alkalis, which reaches a maximum when the estimated sizes of the cryptand cavity and the included ion are nearly the same. As more measurements were made,³ including calorimetry,⁴ other factors began to appear relevant for explaining the observations. Among these were (1) the relative importance of enthalpy and entropy in complex formation and cation solvation, (2) the cation charge, and (3) the nature of the complex formed in solution, i.e., whether the cation is completely enclosed, is partly enclosed with small anions also included, or perhaps is even an exterior complex.⁵

Lanthanide elements also form cryptates,⁶⁻⁸ and crystal structures have been determined⁸⁻¹⁰ in which each of the ions La^{3+} , Sm^{3+} , and Eu^{3+} are enclosed in the cryptand called [2.2.2] (see ref 11 for nomenclature). Electrochemical methods have been used to measure the effect of the metal

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- (11) The shorthand nomenclature for cryptands was originated by Lehn.¹ The three numbers denote the ether oxygen in each strand of the bicyclic molecule. The cryptands used in this work are designated as follows: [2.1.1] represents 4,7,13,18-tetraoxa-1,10-diazabicyclo[8.5.5]eicosane; [2.2.1] represents 4,7,13,16,21-pentaoxa-1,10-diazabicyclo[8.8.5]tricosane; [2.2.2] represents 4,7,13,16,21,24-hexaoxa-1,10-diazabicyclo[8.8.8]hexacosane. The mathematical symbol for inclusion is used in naming cryptates.