Fig. 1. A stereoview of the cubic environment of thorium(IV) (Johnson, 1965).

The structure is in fact close to that of the tetragonal form: the Th atom lies in a strictly cubic configuration with eight linear N—C—S groups along the diagonals (Fig.1). The cubic symmetry also implies equivalence of the three tetraethylammonium groups sandwiched between the faces of adjacent cubes. As in the tetragonal form, there are two carbon (CH_2) positions related by a $\pi/2$ rotation around the respective fourfold axes at $\frac{1}{2},0,0$; $0,\frac{1}{2},0$ and $0,0,\frac{1}{2}$ which have an occupancy factor of $\frac{1}{2}$. Such rotation leaves unchanged the C atom, but not the H atoms, of the CH_3 group.

The center of the cell is occupied by a fourth tetraethylammonium anion lying in one of the six equiprobable positions (occupancy factor $= \frac{1}{6}$) related by rotations around the three fourfold axes.

The essential difference between the cubic and tetragonal crystalline forms is a different packing of

identical layers of cubic anions, the latter being obtained from the former by a $(\frac{1}{2},\frac{1}{2},0)$ translation of consecutive layers. No evidence was found that the U compound exists in the cubic form: possibly, the variation of the c/a ratio with the size of the metal ion, and its value very close to 2 for the Th complex, provides justification (Table 4 gives cell dimensions and c/a ratios in the tetragonal series).*

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* See deposition footnote.

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(η-Allyl)bromodicarbonyl(N,N'-di-tert-butyl-ethanediimine)molybdenum(II), MoBr(C3H5)(C10H20N2)(CO)2

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Abstract. $C_{15}H_{25}BrMoN_2O_2$, $M_r = 441.22$, $P2_12_12_1$; $a = 19.850$ (7), $b = 13.266$ (4), $c = 7.044$ (3) Å; $V = 1854.89$ Å^3; $Z = 4$; $D_m = 1.574$, $D_c = 1.580$ Mg m^{-3}; monochromatic (graphite crystal) Cu $K\alpha_1$, $\lambda = 1.54051$ Å, $\mu = 79.71$ mm^{-1}, $F(000) = 888$, 293 K, $R = 0.0789$ for 1443 measured reflections.

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Introduction. This compound is the fifth in a series of the types (I) $MX(CO)_3(LN=CHCH=NL)$ and (II) $MX(CO)_2A$ (LN=CHCH=NL), where $X =$ Br, Cl; $M =$ Mn, Re, W, Mo; $L =$ cyclohexyl, tert-butyl, isopropyl; $A = \eta$-allyl, η-2-methylallyl, as prepared by Hsieh & West (1976); it has $M =$ Mo, $X =$ Br, $A = \eta$-allyl and $L =$ tert-butyl. The previous compounds in the series had $L =$ cyclohexyl (three com-

pounds) and L = isopropyl (one compound). This is the first in the series to have L = *tert*-butyl.

Experimental. Needle-shaped, 0·015 × 0·05 × 0·64 mm̄, Enraf–Nonius CAD-4, moving crystal/moving counter technique, $6 \le 2\theta \le 140°$, fifteen reflections for measuring lattice parameters, no absorption corrections, $h = 0$ to 24, $k = 0$ to 16, $l = 0$ to 8, reflection 520 as intensity standard, which had an average count of 7550 with $\sigma = 98$, 2041 reflections measured, 1443 non-zero, 291 excluded [$F < 3\sigma(F)$]; Mo and Br positions from Patterson function, non-hydrogen atoms from Fourier syntheses, based on the phases defined by Mo and Br; least squares with isotropic and then anisotropic temperature factors gave $R = 0·096$; overall scale factor refined anisotropically (Shakked & Rabinovich, 1977) gave $R = 0·0793$. C(21), one of the *tert*-butyl atoms, had a non-positive-definite temperature factor and U_{13} of this atom was fixed at 0·01 Å² for one cycle of refinement. H atoms were included in calculated positions. The final R, including weak reflections, all of which gave low calculated F's, was 0·0789. In the final cycle the maximum LS shift/error was 0·646 and the average shift/error was 0·078. Unit weights were used. The final difference synthesis showed maximum and minimum peaks of 1·52 and −0·90e Å⁻³ respectively. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

Calculations were carried out using XRAY 72 (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) and *SHELX* 76 (Sheldrick, 1976).*

Discussion. The configuration of this molecule is the same as the others in the series, in particular Re(CO)₃-Cl(C₃H₇NCHCHNC₃H₇), which has isopropyl groups where the present molecule has *tert*-butyl groups. The Mo—C(carbonyl) bond distances average 1·930 (24) Å and the Mo—C—O bond angles average 173·1 (20)°. In the diimine moiety the mean C=N distance is 1·316 (23) Å, the C—C (bridging) distance is 1·358 (35) Å. The mean C—C bond distance in the *tert*-butyl group is 1·534 (16) Å. The interplanar angle between the α-diimine and dicarbonyl group (*trans* to the diimine) is 2·95 (25)°. Table 1 gives the atom coordinates and equivalent U_{iso}'s and Table 2 has the intramolecular bond distances and angles.

This configuration, the bond distances and the angles are consistent with those in similar complexes, in particular those described by Fenn & Graham (1972), Graham & Fenn (1969, 1970), Graham, Akrigg & Sheldrick (1976, 1977a, b) and Griffiths (1971).

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* Lists of structure factors, anisotropic thermal parameters and selected torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38224 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The molecule is shown in Fig. 1; the packing in Fig. 2.

Table 1. *Atomic coordinates* (×10⁴) *and equivalent isotropic temperature factors* (Å² ×10⁴)

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Br	2038 (2)	1124 (2)	2990 (5)	582
Mo	1473 (1)	2568 (2)	960 (3)	376
C(10)	1653 (10)	1013 (17)	−2097 (38)	376
C(20)	2245 (12)	1505 (24)	−2162 (43)	637
O(1)	347 (11)	2666 (19)	4013 (36)	1030
C(1)	752 (13)	2662 (24)	2935 (42)	650
C(2)	1770 (21)	3552 (26)	2666 (45)	945
O(2)	1968 (12)	4075 (16)	4020 (30)	842
C(3)	1102 (13)	3458 (18)	−1454 (29)	355
C(4)	497 (15)	3260 (23)	−335 (42)	656
C(5)	1526 (14)	4172 (17)	−451 (38)	489
N(10)	1196 (9)	1189 (15)	−875 (31)	371
C(11)	595 (14)	481 (18)	−924 (45)	560
C(12)	844 (17)	−590 (20)	−844 (57)	893
C(13)	190 (17)	650 (27)	−2688 (48)	806
C(14)	182 (15)	691 (23)	843 (44)	712
N(20)	2384 (8)	2263 (15)	−886 (32)	451
C(21)	3114 (10)	2688 (20)	−853 (38)	517
C(22)	3576 (15)	1788 (23)	−663 (50)	757
C(23)	3177 (14)	3367 (24)	808 (50)	726
C(24)	3225 (19)	3237 (28)	−2723 (47)	844

Table 2. *Bond lengths* (Å) *and angles* (°)

Mo—Br	2·639 (4)	C(20)—C(10)	1·34 (3)	C(5)—C(3)	1·45 (3)
Mo—C(1)	2·00 (3)	N(10)—C(10)	1·27 (3)	C(11)—N(10)	1·52 (3)
Mo—C(2)	1·87 (4)	N(20)—C(20)	1·38 (4)	C(12)—C(11)	1·50 (4)
Mo—C(3)	2·20 (2)	C(22)—C(21)	1·51 (4)	C(13)—C(11)	1·50 (4)
Mo—C(4)	2·33 (3)	C(24)—C(21)	1·52 (4)	C(11)—C(14)	1·52 (4)
Mo—C(5)	2·35 (2)	C(1)—O(1)	1·10 (3)	C(21)—N(20)	1·56 (3)
Mo—N(10)	2·30 (2)	C(2)—O(2)	1·24 (4)	C(23)—C(21)	1·48 (4)
Mo—N(20)	2·26 (2)	C(4)—C(3)	1·46 (4)		

| C(1)—Mo—Br	88·4 (9)	C(3)—Mo—Br	162·0 (6)
C(2)—Mo—C(1)	75 (2)	C(3)—Mo—C(2)	103 (1)
C(3)—Mo—C(1)	105 (1)	N(10)—Mo—C(1)	106 (1)
N(10)—Mo—Br	80·2 (5)	N(10)—Mo—C(3)	85 (1)
N(10)—Mo—C(2)	172 (1)	N(20)—Mo—C(1)	169 (1)
N(20)—Mo—Br	80·9 (5)	N(20)—Mo—C(3)	85 (1)
N(20)—Mo—N(10)	74·2 (7)	C(24)—C(21)—C(22)	112 (2)
N(20)—C(20)—C(10)	121 (2)	N(10)—C(10)—C(20)	124 (2)
O(2)—C(2)—Mo	170 (3)	O(1)—C(1)—Mo	177 (3)
C(5)—C(3)—Mo	77 (1)	C(4)—C(3)—Mo	76 (1)
C(3)—C(4)—Mo	66 (1)	C(5)—C(3)—C(4)	109 (2)
C(10)—N(10)—Mo	134 (2)	C(3)—C(5)—Mo	66 (1)
C(11)—N(10)—C(10)	116 (2)	C(11)—N(10)—Mo	134 (2)
N(20)—Mo—C(2)	104 (1)	C(13)—C(11)—C(12)	110 (3)
C(13)—C(11)—N(10)	110 (2)	C(14)—C(11)—C(12)	109 (3)
C(14)—C(11)—N(10)	107 (2)	C(20)—N(20)—Mo	110 (2)
C(14)—C(11)—C(13)	111 (2)	C(21)—N(20)—C(20)	118 (2)
C(21)—N(20)—Mo	132 (2)	C(23)—C(21)—N(20)	108 (2)
C(22)—C(21)—N(20)	106 (2)	C(24)—C(21)—N(20)	107 (2)
C(23)—C(21)—C(22)	111 (2)	C(24)—C(21)—C(23)	112 (2)
C(2)—Mo—Br	91·4 (9)	C(12)—C(11)—N(10)	109 (2)
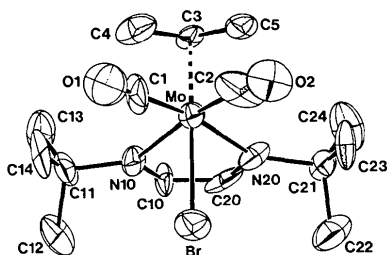
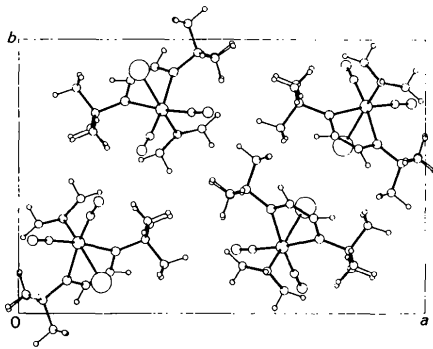Fig. 1. *ORTEP* (Johnson, 1976) plot showing 50% probability thermal ellipsoids.

Fig. 2. *c*-axis projection packing diagram (*PLUTO*, Motherwell, 1978).

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The Structure of Dichloro(2,2':6',2''-terpyridyl)copper(II) Monohydrate, [Cu($C_{15}H_{11}N_3$)Cl_2].H_2O

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Abstract. $M_r = 385.7$, monoclinic, space group $C2/c$, $a = 17.254$ (8), $b = 9.594$ (5), $c = 18.779$ (9) Å, $\beta = 96.81$ (9)°, $Z = 8$, $d_m = 1.651$ (9), $d_x = 1.659$ Mg m^{-3}, $V = 3086.6$ Å^3. The crystal structure of Cu(tpy)Cl$_2$.H$_2$O has been determined using three-dimensional single-crystal X-ray data. The material crystallizes as heavy green needles. Least-squares anisotropic refinement led to $R = 6.5\%$ for 2614 reflexions. The crystal consists of discrete monomeric molecules in which the Cu atom is pentacoordinated. The configuration approaches that of a square pyramid. The various theories concerning pentacoordinated 3*d* elements are described and the square-pyramidal environment is justified.

Introduction. Cu^{II} is a typical metal ion with respect to the formation of coordination complexes, but is a special one in its reluctance to adopt regular octahedral