1.1 times the average of the bound C atoms, and repositioned twice during refinement. The H atoms of the water molecules were ignored. Modeling the disordered water molecules was difficult and a three-site model was eventually adopted. The corresponding O atoms were assigned populations based on their heights in a Fourier map with fixed isotropic B values. The maximum ($2.06\,e\,Å^{-3}$) and minimum ($-1.86\,e\,Å^{-3}$) peaks in the final difference map are within 0.4 Å of the Re2 atom. Individual variances $\sigma^2(I)$ were derived from counting statistics plus an additional term $(0.014I)^2$, while variances of the merged intensities included propagation of error plus another additional term $(0.014\langle I\rangle)^2$.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *CRYM* (Duchamp, 1964). Program(s) used to solve structure: *CRYM*. Program(s) used to refine structure: *CRYM*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *CRYM*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1449). Services for accessing these data are described at the back of the journal.

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Dibenzylbis(*tert*-butylimido)molybdenum(VI), containing both η^1- and η^2-benzyl ligands

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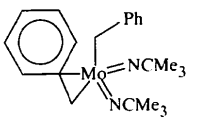
Abstract

The title compound, $[Mo(CH_2Ph)_2(C_4H_9N)_2]$, contains two slightly bent imido ligands and two benzyl ligands. One of these benzyl ligands is coordinated as a simple η^1-alkyl ligand, while the other is η^2-coordinated through the CH_2 group and the *ipso*-C atom, *i.e.* dibenzyl-κC^{α}; $\kappa^2 C^1, C^{\alpha}$-bis(*tert*-butylimido-κN)molybdenum(VI). Considering the η^2-benzyl ligand as occupying one coordination site, this gives a tetrahedral geometry around molybdenum, the main angular distortion being a mutual repulsion of the two strongly π-donating imido ligands. This complex is not crystallographically isostructural with its chromium analogue, but the two molecular structures are very similar.

Comment

As part of our work directed towards the development of new alkene polymerization catalysts based on complexes of imido ligands (Coles & Gibson, 1994), we have already reported the synthesis and crystallographic characterization of the chromium dibenzyl complex $[Cr(CH_2Ph)_2(N^tBu)_2]$, (1) (Coles *et al.*, 1995). Although the possibility of α-agostic interactions between chromium and the CH_2 H atoms of the benzyl ligands was envisaged in this study, the structure revealed instead the presence of one η^1- and one η^2-benzyl ligand. In subsequent investigations of related complexes of chromium and molybdenum with different imido and alkyl ligands, we have prepared the molybdenum analogue of (1), complex (2), and report here its structure.



(2)

Although complexes (1) and (2) are not crystallographically isostructural, belonging to completely different space groups, their molecular structures are very similar (Fig. 1), differing mainly in the sizes of the Cr and Mo atoms, and hence in bond lengths involving these atoms. The Mo—N and Mo—C bonds in (2) are consistently 0.10–0.12 Å longer than the corresponding Cr—N and Cr—C bonds in (1). The coordination geometry of the two metals, and even the orientations of the ligand substituents, are almost identical in the two molecules, with no important differences caused by the different crystalline environments. In (2), as in (1), both *M*—N—C imido linkages depart somewhat from linearity; this structural parameter is known to be quite variable in imido complexes and can differ markedly even for two ligands in the same complex or for two crystallographically independent molecules in the asymmetric unit (Bell *et al.*, 1994*a*; Gibson *et al.*, 1996), but the total range for the four ligands in these two complexes is only 6°.

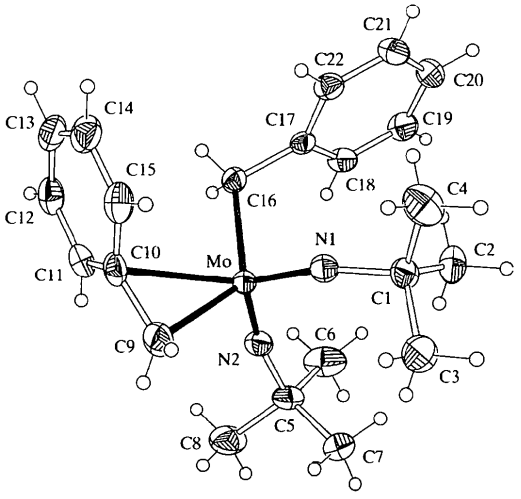


Fig. 1. The molecular structure of (2) with atom labels and 50% probability ellipsoids for non-H atoms.

The coordination geometry of molybdenum can be described as approximately tetrahedral if the η^2-benzyl ligand is considered to occupy one coordination site; the main distortion from ideal tetrahedral angles is a widening of the N—Mo—N angle, as is expected for the strongly π-donating imido ligands. The η^2-coordination of one benzyl ligand is associated with some multiple-bond character of C9—C10. This is shorter than C16—C17 by almost 0.03 Å, and concomitantly the adjacent C_{ipso}—C_{ortho} bonds are lengthened by around 0.02–0.03 Å relative to the other ring C—C bonds in the two benzyl ligands [mean of 1.383 (4) Å for four bonds and of 1.390 (5) Å for six bonds]. The mean plane of the η^2-benzyl ligand is approximately perpendicular to the Mo—C9—C10 triangle [dihedral angle 85.91 (7)°]. The freely refined H atoms on C9 are bent significantly out of the plane of the ligand, away from molybdenum, as is usual for η^2-alkene coordination to a metal.

The H atoms on C16 were also freely refined. Within experimental error, they are symmetrically disposed about the Mo—C16—C17 linkage, indicating no α-agostic interaction with the metal atom for either of the CH_2 groups. In structural studies of a number of bis(imido)bis(alkyl) complexes of chromium and molybdenum, we have found that α-agostic interactions may be evident or absent in each individual case, with no obvious pattern (Bell *et al.*, 1994*a*,*b*; Coles *et al.*, 1996, 1998).

Experimental

$[Mo(N^tBu)_2Cl_2]$·dme (2.0 g, 5.03 mmol; dme is dimethoxyethane) was dissolved in diethyl ether (75 ml) and cooled in a dry-ice/acetone slush bath. To this deep-yellow solution was then added 2 equivalents of $PhCH_2MgCl$ (10.1 ml, 1.0 *M* solution in diethyl ether; 10.1 mmol). The mixture was allowed to warm to room temperature and was stirred for 18 h. The white precipitate which formed was filtered off and the volatile components were removed from the resultant deep-orange solution under reduced pressure. Extraction and recrystallization from acetonitrile at 243 K afforded the title complex [yield 1.62 g (77%)].

Crystal data

$[Mo(C_7H_7)_2(C_4H_9N)_2]$	Mo $K\alpha$ radiation
M_r = 420.44	λ = 0.71073 Å
Monoclinic	Cell parameters from 28 reflections
$P2_1/n$	θ = 11.29–12.51°
a = 9.733 (3) Å	μ = 0.600 mm^{-1}
b = 17.579 (4) Å	T = 160 (2) K
c = 13.016 (3) Å	Prism
β = 97.12 (2)°	0.61 × 0.44 × 0.29 mm
V = 2209.8 (10) Å^3	Yellow
Z = 4	
D_x = 1.264 Mg m^{-3}	
D_m not measured	

Data collection

Stoe–Siemens diffractometer with Cryostream cooler (Cosier & Glazer, 1986)	3695 reflections with $I > 2\sigma(I)$
ω/θ scans with on-line profile fitting (Clegg, 1981)	R_{int} = 0.010
Absorption correction: multi-scan (Sheldrick; 1994)	θ_{max} = 25.01°
T_{min} = 0.781, T_{max} = 0.840	$h = -11 \rightarrow 11$
5099 measured reflections	$k = -4 \rightarrow 20$
3903 independent reflections	$l = -15 \rightarrow 15$
	5 standard reflections frequency: 60 min intensity decay: 1%

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max}$ = 0.001
$R[F^2 > 2\sigma(F^2)]$ = 0.018	$\Delta\rho_{max}$ = 0.234 e Å^{-3}
$wR(F^2)$ = 0.051	$\Delta\rho_{min}$ = −0.275 e Å^{-3}

S = 1.086
3903 reflections
245 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0208P)^2 + 0.9976P]$
where $P = (F_o^2 + 2F_c^2)/3$
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0031 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Mo—N2	1.7420 (15)	N2—C5	1.456 (2)
Mo—N1	1.7437 (14)	C9—C10	1.459 (2)
Mo—C9	2.1866 (17)	C10—C15	1.412 (2)
Mo—C16	2.1958 (16)	C10—C11	1.412 (2)
Mo—C10	2.4673 (16)	C16—C17	1.488 (2)
N1—C1	1.455 (2)		
N2—Mo—N1	113.64 (7)	N1—Mo—C10	118.71 (6)
N2—Mo—C9	103.17 (7)	C9—Mo—C10	35.89 (6)
N1—Mo—C9	105.75 (7)	C16—Mo—C10	91.92 (6)
N2—Mo—C16	104.01 (7)	C1—N1—Mo	160.00 (12)
N1—Mo—C16	102.82 (6)	C5—N2—Mo	162.83 (12)
C9—Mo—C16	127.77 (6)	C10—C9—Mo	82.61 (10)
N2—Mo—C10	119.65 (6)	C9—C10—Mo	61.50 (9)

H atoms were placed geometrically and refined with a riding model (including free rotation about C—C bonds), and with U_{iso} constrained to be 1.2 (1.5 for methyl and methylene groups) times U_{eq} of the carrier atom, except for those of the benzyl CH_2 groups, the positions of which were refined freely.

Data collection: *DIF*4 (Stoe & Cie, 1988). Cell refinement: *DIF*4. Data reduction: local programs. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1994). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and local programs.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1081). Services for accessing these data are described at the back of the journal.

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Dipotassium naphthalene-1,8-dicarboxylate–potassium bicarbonate

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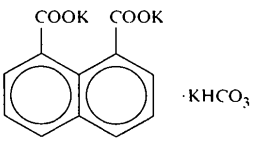
Abstract

The title substance, tripotassium naphthalene-1,8-dicarboxylate bicarbonate, $3K^+\cdot C_{12}H_6O_4^{2-}\cdot HCO_3^-$, crystallized in the centrosymmetric space group *Pbca*. There is a single hydrogen bond. In it, the O and H atoms are ordered and $O_D\cdots O_A$ is 2.651 (3) Å. There is a single significant intermolecular C—H···O interaction, with $C\cdots O_A$ = 3.480 (5) Å. Each potassium ion is coordinated by an irregular polyhedron of O atoms. These three polyhedra contain eight, eight and five members; each O atom is a member of at least two polyhedra. Structural comparisons are made with tetrapotassium naphthalene-1,4,5,8-tetracarboxylate hexahydrate.

Comment

This study of the title compound, (I), follows a study of tetrapotassium, tetrarubidium and tetracesium



(I)

naphthalene-1,4,5,8-tetracarboxylate hexahydrates (Fitzgerald *et al.*, 1993) in a series on hydrogen bonding in organic solids. The title compound crystallized in the centrosymmetric space group *Pbca* with one dipotassium naphthalene-1,8-dicarboxylate and one potassium bicarbonate in the asymmetric unit. The refined asymmetric unit, with our numbering scheme, is shown in Fig. 1. In the single hydrogen bond, O6—H8···O2^{vii} [symmetry code: (vii) $-\frac{1}{2}-x, 1-y, z-\frac{1}{2}$], the O and H atoms are ordered, with O—H = 1.00 (5), H···O = 1.68 (5) and $O_D\cdots O_A$ = 2.651 (3) Å, and with O—H···O = 165 (5)°. In addition, there is a single significant intermolecular C—H···O interaction, C2—H2···O3^{viii} [symmetry code: (viii) $-\frac{1}{2}+x, y, \frac{3}{2}-z$], which has H···O = 2.50, C···O = 3.480 (5) Å and C—H···O = 176° (no s.u.'s are given for quantities involv-