Refinement

Refinement on F
Final $R = 0.033$
$wR = 0.031$
$S = 1.854$
6516 reflections
367 parameters
H-atom parameters not refined, fixed at idealized positions with common $U_{\text{iso}} = 0.08$ Å^2; phenyl groups treated as rigid bodies (C—C 1.395, C—H 0.96 Å)
Calculated weights $w = 1/[\sigma^2(F)+0.0001F^2]$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.73$ e Å^{-3}
$\Delta\rho_{\min} = -0.52$ e Å^{-3}

Scattering factors, structure solution and refinement, and all calculations: *SHELXTL-Plus* (Sheldrick, 1990). Other programs: *PARST* (Nardelli, 1983); *MISSYM* (Le Page, 1987).

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	U_{eq}
Mo	0.2231 (1)	0.1128 (1)	0.9243 (1)	0.035 (1)
Zn	0.4419 (1)	0.3657 (1)	0.5127 (1)	0.048 (1)
Cl1	0.5283 (1)	0.4578 (1)	0.6524 (1)	0.070 (1)
Cl2	0.2658 (1)	0.3816 (1)	0.4364 (1)	0.069 (1)
P1	0.3131 (1)	0.2535 (1)	0.8780 (1)	0.033 (1)
P2	0.1631 (1)	0.1745 (1)	0.7094 (1)	0.035 (1)
C1	0.3025 (3)	0.0845 (2)	1.0924 (3)	0.051 (2)
O1	0.3496 (2)	0.0672 (2)	1.1883 (3)	0.082 (2)
C2	0.1007 (3)	0.1947 (2)	1.0089 (3)	0.045 (2)
O2	0.0330 (2)	0.2360 (2)	1.0591 (2)	0.068 (2)
C3	0.3384 (2)	0.0195 (2)	0.8528 (3)	0.044 (2)
O3	0.3999 (2)	−0.0387 (2)	0.8145 (2)	0.068 (2)
C4	0.1344 (3)	0.0061 (2)	0.9218 (3)	0.051 (2)
O4	0.0858 (2)	−0.0570 (2)	0.9180 (3)	0.085 (2)
C5	0.2837 (2)	0.2420 (2)	0.7037 (2)	0.034 (1)
C6	0.3762 (2)	0.1859 (2)	0.6377 (3)	0.041 (2)
C7	0.5616 (3)	0.1840 (2)	0.5734 (3)	0.056 (2)
C8	0.5451 (3)	0.1860 (2)	0.4349 (3)	0.061 (2)
C9	0.6478 (3)	0.3139 (3)	0.3896 (4)	0.078 (3)
C10	0.4834 (3)	0.2866 (3)	0.2619 (3)	0.068 (3)
N1	0.4672 (2)	0.2415 (2)	0.6255 (2)	0.040 (1)
N2	0.5382 (2)	0.2852 (2)	0.3898 (2)	0.047 (2)
C11	0.4597 (1)	0.2646 (1)	0.9191 (2)	0.035 (1)
C12	0.5336	0.1822	0.9341	0.047 (2)
C13	0.6462	0.1871	0.9663	0.056 (2)
C14	0.6849	0.2744	0.9834	0.056 (2)
C15	0.6110	0.3568	0.9684	0.057 (2)
C16	0.4984	0.3519	0.9362	0.046 (2)
C21	0.2444 (2)	0.3696 (1)	0.9190 (2)	0.039 (2)
C22	0.2109	0.4413	0.8346	0.054 (2)
C23	0.1541	0.5263	0.8737	0.065 (3)
C24	0.1308	0.5395	0.9972	0.061 (2)
C25	0.1642	0.4677	1.0815	0.070 (2)
C26	0.2210	0.3828	1.0424	0.053 (2)
C31	0.1528 (2)	0.1007 (1)	0.5671 (2)	0.039 (2)
C32	0.1580	0.0038	0.5778	0.048 (2)
C33	0.1483	−0.0543	0.4709	0.064 (2)
C34	0.1335	−0.0154	0.3532	0.075 (3)
C35	0.1283	0.0816	0.3424	0.074 (3)
C36	0.1379	0.1397	0.4494	0.061 (2)
C41	0.0398 (1)	0.2613 (1)	0.6838 (2)	0.037 (2)
C42	0.0319	0.3375	0.6062	0.052 (2)
C43	−0.0672	0.3976	0.5867	0.060 (2)
C44	−0.1583	0.3816	0.6449	0.055 (2)
C45	−0.1504	0.3054	0.7225	0.050 (2)
C46	−0.0514	0.2453	0.7420	0.044 (2)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Mo—P1	2.522 (1)	Zn—N2	2.087 (2)
Mo—P2	2.515 (1)	P1—C5	1.878 (3)
Mo—C1	1.990 (3)	P2—C5	1.882 (3)
Mo—C2	2.056 (3)	C5—C6	1.530 (4)
Mo—C3	2.011 (3)	C6—N1	1.478 (3)
Mo—C4	1.992 (3)	C7—C8	1.487 (5)
Zn—Cl1	2.226 (1)	C7—N1	1.481 (4)
Zn—Cl2	2.207 (1)	C8—N2	1.516 (4)
Zn—N1	2.174 (2)		
P1—Mo—P2	66.2 (1)	Mo—P1—C5	95.9 (1)
Cl1—Zn—Cl2	128.5 (1)	Mo—P2—C5	96.0 (1)
Cl1—Zn—N1	96.4 (1)	P1—C5—P2	94.0 (1)
Cl1—Zn—N2	117.1 (1)	C8—C7—N1	109.7 (3)
Cl2—Zn—N1	105.8 (1)	C7—C8—N2	111.4 (3)
Cl2—Zn—N2	110.4 (1)	Zn—N1—C7	102.4 (2)
N1—Zn—N2	86.0 (1)	Zn—N2—C8	103.7 (2)

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55850 (38 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1029]

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2-η^3-Allyl-1,1,1-tricarbonyl-μ-(η^1:η^6-diphenylmethyl)chromiumpalladium(*Pd—Cr*) at 130 K

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Abstract

The introduction of the phenyl substituent at the benzyl C atom of the arene, π-coordinated by the Cr atom, does not cause any significant changes in the geometry of the binuclear complex which contains a direct Pd—Cr bond [average length 2.768(1) Å for the two independent molecules]. The Pd—C$_{\text{benzyl}}$, Pd—C$_{ipso}$ and C$_{\text{benzyl}}$—C$_{ipso}$ bond lengths [averages 2.138(3), 2.547(2) and

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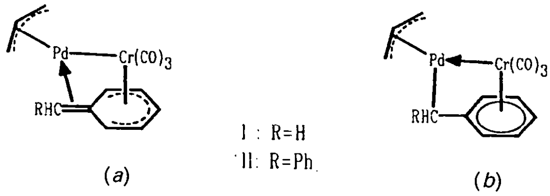
1.452(3) Å, respectively] and other geometric features indicate that the title compound may be treated as a superposition of two canonical structures, one of which implies π coordination of the Pd atom by the exocylic C_{benzyl}—C_{ipso} bond whereas the second involves only Pd—C_{benzyl} σ bonding.

Comment

We have reported recently (Kalinin, Cherepanov, Moiseev, Batsanov & Struchkov, 1991) the synthesis and unusual structure of the complex (η^3-C_3H_5)Pd(CH_2Ph-η^6)Cr(CO)$_3$ (I) which has a direct Pd—Cr bond. The geometry of the bridging benzyl substituent suggests that the bonding in (I) should be described in terms of a superposition of the two canonical forms (*a*) and (*b*).



The introduction of a phenyl substituent on the benzyl C atom of (I) might be expected to contribute to the stabilization of the exocyclic double bond [*i.e.* structure (*a*)]. Accordingly, we have undertaken the synthesis and X-ray diffraction study of the complex (η^3-C_3H_5)Pd[(CH(Ph)Ph-η^6)]Cr(CO)$_3$ (II), a close analogue of (I) but with a diphenylmethyl group as a bridging ligand. The preparation of (II), by reaction of (OC)$_3$Cr[η^6-PhCH(Ph)ZnCl] and (η^3-C_3H_5PdCl)$_2$, was performed in the same manner as the synthesis of (I) (Kalinin, Cherepanov, Moiseev, Batsanov & Struchkov, 1991). The molecular structure of (II) is shown in Fig. 1.

The obvious first result of the introduction of a Ph group is the possibility of diastereomerism in (II) as a result of the presence of two chirality centres in the molecule (the overall chirality of the Pd coordination centre and the asymmetric centre at the atom C7). Indeed, (II) turned out to be a racemic mixture of two diastereomers in the ratio 4:1 as revealed by disorder in the π-allyl groups of both independent molecules of (II) (see experimental details).

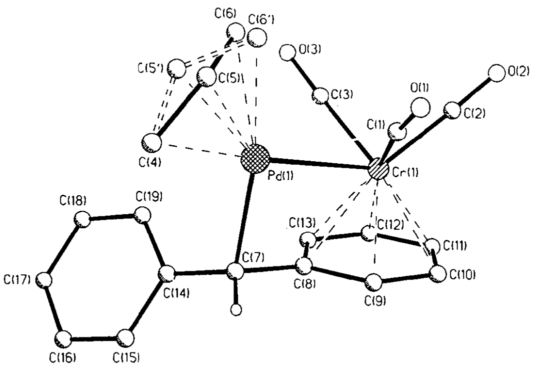


Fig. 1. View of $C_{19}H_{16}CrO_3Pd$ showing the labelling of the non-H atoms. The alternative position of the disordered allyl group (with atomic s.o.f.'s equal to 0.2) is shown with the dashed bonds. Phenyl and allyl H atoms are omitted.

Both molecules of (II) have very similar geometric parameters which are quite close to those found for (I). The Pd—Cr distances in (II) [2.766(1) and 2.770(1) Å] correspond to direct metal-metal bonds and are nearly the same length as in (I) (2.764 Å) and in $Pd_2Cr_2(\eta^5\text{-}C_5H_5)_2(\mu_3\text{-}CO)_2(\mu\text{-}CO)_4(PEt_3)_2$ (2.741 and 2.777 Å), the only other structurally characterized complex with Pd—Cr bonds (Bender, Braunstein, Jud & Dusausoy, 1983).

As pointed out above, the Pd-benzyl-group bonding may be treated as intermediate between the π-olefin coordination [structure (*a*)] and normal metal-carbon σ bonding [structure (*b*)]. The former is consistent with the rather short Pd1—C8 distances [2.551(2) and 2.544(2) Å in (II), 2.53 Å in (I)] as well as with the short C7—C8 bonds [1.451(3) and 1.453(3) Å in (II), 1.42 Å in (I)] and the slight elongation of the C8—C9 and C8—C13 bonds in the benzene ring (see Table 2), whereas the shortness of the Pd1—C7 bonds [2.141(3) and 2.136(3) Å in (II), 2.10(1) Å in (I)] compared with the Pd1—C8 distances, the almost symmetrical Cr—C(arene) coordination and the lack of significant folding of the arene ring along the C9···C13 line [1.0(3) and 1.5(3)° in (II)] are more in accord with the bonding scheme of formula (*b*). A similar kind of bonding dualism is typical of the long-known and thoroughly studied series of π complexes with carbenium centres in the α position of the π ligands (see *e.g.* Watts, 1979; Rybinskaya, Kreindlin, Struchkov & Yanovsky, 1989).

A comparison of the geometries of molecules (I) and (II) indicates that the Ph substituent has only a minor influence on the mode of organometallic coordination. The most significant structural difference between the two complexes is associated with the slight asymmetry of the semi-bridging carbonyl groups C1O1 and C3O3 in both independent molecules of (II), evident from the unequal Pd···C distances [Pd1···C1 2.513(3), 2.528(3); Pd1···C3 2.746(3), 2.755(3) Å] and Cr—C—O bond angles [Cr1—C1—O1 171.3(2), 171.2(2); Cr1—C3—O3 175.1(3), 174.8(2)°]. It is noteworthy that in both molecules, the Ph substituent and the semi-bridging carbonyl (which is more strongly bonded to the Pd atom) lie on opposite sides of the plane passing through the atoms Cr1, Pd1, O2, C2, C7, C8, and C11.

Experimental

Crystal data

$C_{19}H_{16}CrO_3Pd$	D_x = 1.800 Mg m^{-3}
M_r = 450.72	Mo $K\alpha$ radiation

Triclinic
$P\bar{1}$
a = 11.470 (2) Å
b = 12.645 (3) Å
c = 13.027 (3) Å
α = 118.18 (1)°
β = 90.26 (2)°
γ = 92.37 (2)°
V = 1663.4 (6) Å^3
Z = 4

λ = 0.71073 Å
Cell parameters from 24 reflections
θ = 13–13.5°
μ = 1.747 mm^{-1}
T = 133.0 (10) K
Well formed plates
0.4 × 0.2 × 0.2 mm
Light brown

Data collection

Siemens *P*3/*PC* diffractometer
$\theta/2\theta$ scans
Absorption correction: none
7169 measured reflections
6796 independent reflections
6032 observed reflections [$I>2\sigma(I)$]

$R_{\rm int}$ = 0.0134
$\theta_{\rm max}$ = 27.06°
$h = 0 \rightarrow 14$
$k = -16 \rightarrow 16$
$l = -16 \rightarrow 16$
2 standard reflections monitored every 98 reflections
intensity variation: 1.5%

Refinement

Refinement on F^2
Final R = 0.030 (on F)
wR = 0.063 (on F^2)
S = 1.063
6781 reflections
597 parameters
All H-atom parameters refined
Calculated weights $w = 1/[\sigma^2(F_o^2)+(0.0310P)^2 + 1.0337P]$, where $P = (F_o^2+2F_c^2)/3$

$(\Delta/\sigma)_{\rm max}$ = 0.054
$\Delta\rho_{\rm max}$ = 0.30 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.24 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Program(s) used to solve structure: *SHELXTL-Plus*, PC Version (Robinson & Sheldrick, 1988). Program(s) used to refine structure: *SHELXL*92 (Sheldrick, 1992).

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Molecule *A*				
Pd1	0.04823 (2)	0.37447 (2)	0.70467 (2)	0.02118 (9)
Cr1	0.15534 (3)	0.23324 (3)	0.49735 (3)	0.0200 (2)
O1	0.1197 (2)	0.4866 (2)	0.5439 (2)	0.0347 (11)
O2	0.1205 (2)	0.1853 (2)	0.2507 (2)	0.0486 (12)
O3	−0.0957 (2)	0.1442 (2)	0.4759 (2)	0.0404 (10)
C1	0.1275 (2)	0.3910 (2)	0.5330 (2)	0.0266 (12)
C2	0.1351 (2)	0.2029 (3)	0.3452 (2)	0.0308 (13)
C3	−0.0004 (2)	0.1832 (2)	0.4883 (2)	0.0295 (14)
C4	−0.0384 (3)	0.4923 (3)	0.8600 (3)	0.034 (2)
C5*	−0.0743 (3)	0.5135 (4)	0.7671 (4)	0.039 (2)
C5′†	−0.1173 (10)	0.4534 (11)	0.7675 (11)	0.019 (5)
C6*	−0.1218 (4)	0.4264 (6)	0.6643 (4)	0.039 (2)
C6′†	−0.0892 (14)	0.4801 (17)	0.6718 (16)	0.028 (7)
C7	0.1766 (2)	0.3143 (2)	0.7825 (2)	0.0246 (12)
C8	0.2261 (2)	0.2480 (2)	0.6684 (2)	0.0216 (11)
C9	0.3054 (2)	0.3076 (2)	0.6245 (2)	0.0235 (11)
C10	0.3501 (2)	0.2493 (2)	0.5127 (2)	0.0268 (11)
C11	0.3140 (2)	0.1292 (2)	0.4366 (2)	0.0294 (13)
C12	0.2383 (2)	0.0672 (2)	0.4761 (2)	0.0283 (14)
C13	0.1931 (2)	0.1254 (2)	0.5880 (2)	0.0242 (12)
C14	0.1203 (2)	0.2490 (2)	0.8412 (2)	0.0247 (13)
C15	0.1724 (3)	0.2562 (3)	0.9413 (2)	0.032 (2)
C16	0.1198 (3)	0.2009 (3)	1.0009 (3)	0.043 (2)
C17	0.0149 (3)	0.1370 (3)	0.9617 (3)	0.042 (2)
C18	−0.0385 (3)	0.1274 (3)	0.8622 (3)	0.041 (2)
C19	0.0141 (3)	0.1825 (3)	0.8027 (3)	0.0339 (14)
Molecule *B*				
Pd1	0.45675 (2)	0.63076 (2)	0.82447 (2)	0.02128 (9)
Cr1	0.35119 (3)	0.78131 (3)	0.75591 (3)	0.0195 (2)
O1	0.3853 (2)	0.5328 (2)	0.5602 (2)	0.0363 (10)
O2	0.3837 (2)	0.8437 (2)	0.5621 (2)	0.0433 (12)
O3	0.6021 (2)	0.8692 (2)	0.8224 (2)	0.0388 (10)
C1	0.3784 (2)	0.6264 (2)	0.6407 (2)	0.0268 (11)
C2	0.3705 (2)	0.8210 (2)	0.6370 (2)	0.0277 (12)
C3	0.5070 (2)	0.8303 (2)	0.7963 (2)	0.0278 (14)
C4	0.5461 (3)	0.5098 (3)	0.8661 (3)	0.036 (2)
C5*	0.5795 (3)	0.4925 (4)	0.7549 (4)	0.036 (2)
C5′†	0.6203 (14)	0.5479 (17)	0.8091 (13)	0.036 (8)
C6*	0.6283 (4)	0.5842 (5)	0.7365 (5)	0.034 (2)
C6′†	0.5963 (14)	0.5338 (17)	0.6970 (15)	0.028 (8)
C7	0.3275 (2)	0.6831 (2)	0.9548 (2)	0.0228 (12)
C8	0.2793 (2)	0.7555 (2)	0.9071 (2)	0.0201 (10)
C9	0.2002 (2)	0.7014 (2)	0.8073 (2)	0.0235 (11)
C10	0.1569 (2)	0.7671 (2)	0.7553 (2)	0.0271 (11)
C11	0.1936 (2)	0.8881 (2)	0.7964 (2)	0.0284 (13)
C12	0.2699 (2)	0.9438 (2)	0.8937 (2)	0.0271 (13)
C13	0.3142 (2)	0.8787 (2)	0.9464 (2)	0.0234 (12)
C14	0.3791 (2)	0.7448 (2)	1.0767 (2)	0.0224 (12)
C15	0.3169 (2)	0.7410 (2)	1.1675 (2)	0.0271 (13)
C16	0.3628 (3)	0.7954 (2)	1.2808 (2)	0.032 (2)
C17	0.4711 (3)	0.8547 (2)	1.3067 (2)	0.030 (2)
C18	0.5343 (2)	0.8599 (2)	1.2185 (2)	0.0301 (14)
C19	0.4886 (2)	0.8051 (2)	1.1048 (2)	0.0272 (13)

* s.o.f. G = 0.8.
† s.o.f. G = 0.2.

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

	Molecule *A*	Molecule *B*
Pd1—Cr1	2.766 (1)	2.770 (1)
Pd1—C4	2.143 (3)	2.143 (3)
Pd1—C5	2.143 (3)	2.140 (4)
Pd1—C5′	2.161 (10)	2.151 (12)
Pd1—C6	2.222 (4)	2.238 (4)
Pd1—C6′	2.273 (16)	2.256 (16)
Pd1—C7	2.141 (3)	2.136 (3)
Pd1—C8	2.551 (2)	2.544 (2)
Cr1—C1	1.865 (3)	1.861 (3)
Cr1—C2	1.844 (3)	1.852 (3)
Cr1—C3	1.854 (3)	1.856 (3)
Cr1—C8	2.288 (2)	2.294 (2)
Cr1—C9	2.228 (3)	2.230 (3)
Cr1—C10	2.235 (3)	2.228 (3)
Cr1—C11	2.213 (3)	2.215 (3)
Cr1—C12	2.238 (3)	2.237 (3)
Cr1—C13	2.237 (2)	2.241 (2)
O1—C1	1.156 (3)	1.159 (3)
O2—C2	1.152 (3)	1.149 (3)
O3—C3	1.160 (3)	1.159 (3)
C4—C5	1.420 (5)	1.417 (6)
C4—C5′	1.380 (14)	1.348 (18)
C5—C6	1.362 (7)	1.387 (7)
C5′—C6′	1.472 (20)	1.409 (22)
C7—C8	1.451 (3)	1.453 (3)
C7—C14	1.496 (3)	1.504 (3)
C8—C9	1.441 (3)	1.442 (3)
C8—C13	1.434 (3)	1.429 (3)
C9—C10	1.398 (4)	1.399 (4)
C10—C11	1.408 (4)	1.405 (4)
C11—C12	1.402 (4)	1.401 (4)
C12—C13	1.400 (4)	1.406 (3)
C14—C15	1.394 (4)	1.404 (3)
C14—C19	1.398 (4)	1.394 (4)
C15—C16	1.393 (4)	1.391 (4)
C16—C17	1.373 (5)	1.378 (4)
C17—C18	1.382 (5)	1.387 (4)
C18—C19	1.390 (4)	1.394 (4)

C7—Pd1—C8	34.65 (8)	34.80 (8)
C7—Pd1—Cr1	85.41 (7)	85.70 (7)
C8—Pd1—Cr1	50.79 (5)	50.92 (5)
C2—Cr1—C3	86.21 (12)	86.84 (11)
C2—Cr1—C1	84.14 (12)	84.18 (12)
C3—Cr1—C1	95.87 (12)	96.17 (11)
C2—Cr1—Pd1	134.95 (8)	135.75 (8)
C3—Cr1—Pd1	69.77 (9)	69.92 (8)
C1—Cr1—Pd1	62.19 (8)	62.60 (8)
C8—Cr1—Pd1	59.75 (6)	59.43 (6)
O1—C1—Cr1	171.3 (2)	171.2 (2)
O2—C2—Cr1	178.6 (2)	178.7 (2)
O3—C3—Cr1	175.1 (3)	174.8 (2)
C6—C5—C4	123.6 (5)	122.9 (4)
C4—C5′—C6′	118.3 (12)	123.5 (17)
C8—C7—C14	120.4 (2)	118.9 (2)
C8—C7—Pd1	88.34 (14)	88.13 (14)
C14—C7—Pd1	111.0 (2)	112.9 (2)
C13—C8—C9	115.4 (2)	115.9 (2)
C13—C8—C7	123.9 (2)	123.5 (2)
C9—C8—C7	120.4 (2)	120.3 (2)
C13—C8—Pd1	108.8 (2)	108.09 (15)
C9—C8—Pd1	100.37 (15)	100.65 (15)
C7—C8—Pd1	57.02 (12)	57.07 (12)
C10—C9—C8	122.5 (2)	121.8 (2)
C9—C10—C11	119.9 (2)	120.6 (2)
C12—C11—C10	119.5 (2)	119.1 (2)
C13—C12—C11	120.6 (2)	120.8 (2)
C12—C13—C8	121.9 (2)	121.7 (2)
C15—C14—C19	117.0 (2)	117.3 (2)
C15—C14—C7	119.8 (2)	123.1 (2)
C19—C14—C7	123.2 (2)	119.6 (2)
C16—C15—C14	121.3 (3)	121.1 (3)
C17—C16—C15	120.5 (3)	120.6 (2)
C16—C17—C18	119.7 (3)	119.4 (2)
C17—C18—C19	119.8 (3)	120.1 (3)
C18—C19—C14	121.7 (3)	121.5 (2)

The structure was solved by direct methods. The Pd and Mo atoms were located in the *E* map; all other atoms were found in difference Fourier syntheses. Careful inspection of the difference map and subsequent refinement of the site occupation factors (s.o.f.) revealed disorder of the allyl-group C atoms over two positions. Constrained refinement of the s.o.f.'s yielded values of 0.81(2) and 0.78(2) for the prevailing isomer for each of the independent molecules. In the final stages of refinement, these values were fixed at 0.8 for both independent molecules; the s.o.f.'s of the alternative isomer were fixed at 0.2. The H atoms corresponding only to the prevailing diastereomer were located and refined, although some of their positions seem to be biased as a result of the contribution of the H atoms of the alternative isomer.

The final refinement was performed on F^2 for all reflections, including those generally believed to be unobserved [$F^2 < 2\sigma(F^2)$] and even those having negative F^2 but larger than $-3\sigma(F^2)$ (no reflections were rejected according to the latter criterion); 15 reflections obscured by the primary beam stop or thought to be affected by temperature instability were omitted. The final conventional *R* factor was 0.0302 for all reflections and 0.0237 for the 6032 observed reflections with $F > 4\sigma(F)$. The weighted *wR*2 factor (calculated on F^2) was 0.0632 for all data and 0.0567 for the observed data.

Both independent molecules are virtually geometrically identical (see *Comment*) and the coordinates of the corresponding atoms satisfy the following approximate relationship:

$$x(A) + x(B) = 0.5; \quad y(A) + y(B) = 1.$$

However, careful geometric analysis of the unit-cell parameters leaves no possibility for metric symmetry higher than triclinic.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55783 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1029]

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Barium Bis(trimethylacetate)–18-crown-6 (1/1)

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Abstract

The (1,4,7,10,13,16-hexaoxacyclooctadecane)bis(trimethylacetato)barium molecule is contained in a $P\overline{1}$ triclinic cell. The Ba atom is located on an inversion center. There are two trimethylacetate ligands coordinated to the Ba atom which is encapsulated by an 18-crown-6 ether ring. The Ba atom is ten coordinate with an average Ba—O(18-crown-6) bond distance of 2.823 (6) Å and an average Ba—O(trimethylacetate) bond distance of 2.835 (8) Å.

Comment

This compound is the first barium crown ether useful for superconductor fabrication. The six crown-ether O atoms bonded to the Ba atom assume an almost

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