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Dicarbonylbis(1,4-difluoro-2,3,5,6-tetramethyl-1,4-diboracyclohexa-2,5-diene)molybdenum

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The structure of the title compound, $[\mathrm{Mo}(\eta^6\text{-}\mathrm{C_4Me_4B_2F_2})_2(\mathrm{CO})_2]$, (II), was determined by X-ray crystallography at 173 K and has two independent molecules of (II) in the asymmetric unit. The six-membered diboracycles are η^6-coordinated [$\mathrm{Mo-C_{alkene}}$ = 2.4157 (18)–2.6008 (19); Mo—B = 2.538 (2)–2.621 (2) Å].

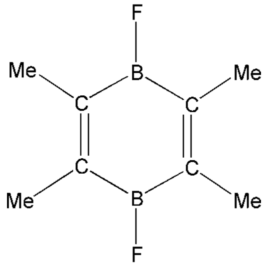
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.023
wR factor = 0.053
Data-to-parameter ratio = 16.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

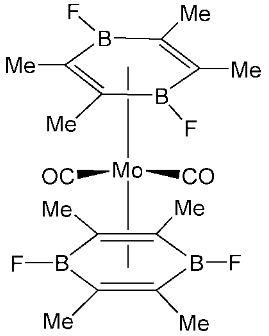
Comment

The reaction of boron monofluoride with but-2-yne at low temperatures yields 1,4-difluoro-2,3,5,6-tetramethyl-1,4-diboracyclohexa-2,5-diene, (I) (Timms, 1968).



(I)

Reaction of (I), which is an analogue of duroquinone, with metal–carbonyl complexes displaces carbon monoxide thermally or under photolysis to yield complexes such as $[\mathrm{Ni(CO)_2}(\eta^6\text{-}\mathrm{C_4Me_4B_2F_2})]$ and $[\mathrm{Ni}(\eta^6\text{-}\mathrm{C_4Me_4B_2F_2})_2]$ (Maddren *et al.*, 1975). The title compound, $[\mathrm{Mo(CO)_2(C_4Me_4B_2F_2)_2}]$, (II) (Fig. 1), was prepared by a sequential photolysis from $[\mathrm{Mo(CO)_6}]$ and (I); photolysis of a mixture of $[\mathrm{Mo(CO)_6}]$ with 1.5 equivalents of (I) yielded $[\mathrm{Mo(CO)_4}(\eta^6\text{-}\mathrm{C_4Me_4B_2F_2})]$ (Hawker, 1981) which, after further photolysis with another equivalent of (I), gave (II). Single crystals were grown from a solution of (II) in dichloromethane at 279 K.



(II)

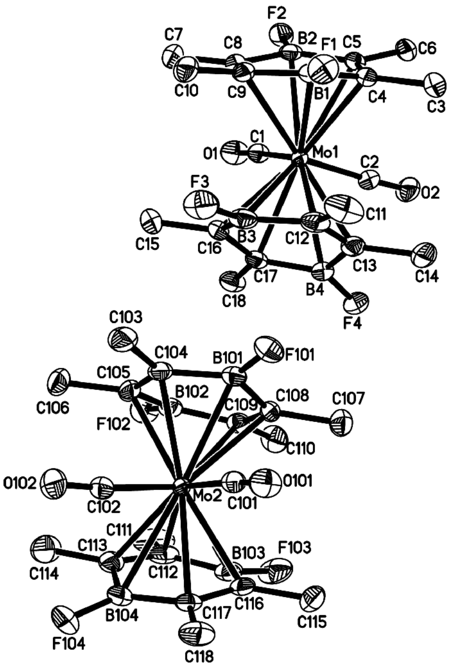


Figure 1

The structure of (II) showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

Compound (II) crystallizes in space group $P2_1/n$ with the asymmetric unit containing two independent molecules, each having C_2 symmetry (Fig. 1). The complex consists of one Mo atom in a distorted pseudo-tetrahedral environment bonded to two carbonyl units and two conversely oriented η^6-rings (angles between the MoB$_2$ units = 45.4/70.7°). Each six-membered diboracycle acts as a 4 e$^-$ donor through two alkene entities, with Mo—C$_{\rm alkene}$ bond lengths ranging between 2.415 (2) and 2.601 (2) Å. Back donation to the B—F units also occurs, leading to Mo—B contacts in the range 2.538 (2)–2.621 (2) Å. The rings are almost flat with the B atoms deviating from the plane by between −0.172 and −0.165 Å (Mo deviations between 1.986 and 1.994 Å).

Experimental

Analysis of compound (II) showed the following: IR (CD$_2$Cl$_2$ solution, cm^{-1}): 2001 (*s*), 1955 (*s*), ν(CO); Mass spectrum (EI, *m*/*z*): M^+ 481–493 (487, most intense); NMR ^{1}H (300 MHz, CD$_2$Cl$_2$, p.p.m.): δ 1.59; ^{11}B (96 MHz, CD$_2$Cl$_2$, p.p.m.): δ 21.6; ^{19}F (282 MHz, CD$_2$Cl$_2$, p.p.m.): δ −152.5 (very broad signal); ^{13}C (75 MHz, CD$_2$Cl$_2$, p.p.m.): δ 13.7 (CH$_3$); ^{13}C signals for the CO and ring *C* atoms not observed.

Crystal data

[Mo(C$_8$H$_{12}$B$_2$F$_2$)$_2$(CO)$_2$]
M_r = 487.55
Monoclinic, $P2_1/n$
a = 14.9106 (10) Å
b = 8.9361 (6) Å
c = 30.252 (2) Å
β = 102.074 (9)°
V = 3941.7 (5) Å^3
Z = 8
D_x = 1.643 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 494 reflections
θ = 3–50°
μ = 0.71 mm^{-1}
T = 173 (2) K
Block, yellow
0.5 × 0.3 × 0.3 mm

Data collection

Siemens CCD area-detector diffractometer
ω scans with narrow frames
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\rm min}$ = 0.573, $T_{\rm max}$ = 0.730
24 739 measured reflections
9039 independent reflections
7446 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.026
$\theta_{\rm max}$ = 27.5°
$h = -19 \rightarrow 19$
$k = -8 \rightarrow 11$
$l = -39 \rightarrow 38$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.053
S = 0.98
9039 reflections
540 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0259P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max}$ = 0.004
$\Delta\rho_{\rm max}$ = 0.50 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.61 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.00059 (5)

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXTL* (Bruker, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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