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Solid solution of *cis*-1,2-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1,2-bis[4-(trifluoromethyl)phenyl]-ethene–*cis*-2-(1,3,2-benzodioxaborolan-2-yl)-1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1,2-bis[4-(trifluoromethyl)phenyl]ethene–hexane solvate (1/1/1)

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Key indicators

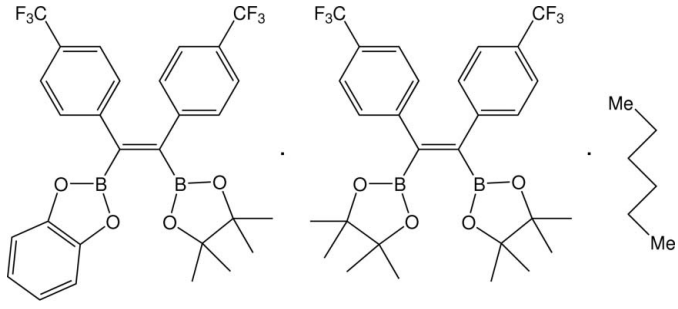
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
Some non-H atoms missing
Disorder in main residue
R factor = 0.074
wR factor = 0.257
Data-to-parameter ratio = 16.5

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

In the title adduct, $C_{28}H_{24}B_2F_6O_4\cdot C_{28}H_{32}B_2F_6O_4\cdot C_6H_{14}$, *cis* isomers of 2-catecholboryl-1-pinacolboryl- and bis(1,2-pinacolboryl)-1,2-bis[4-(trifluoromethyl)phenyl]ethene substitute one another isomorphously in a 1:1 ratio. The molecule has no crystallographic symmetry, with one olefinic site shared equally between superimposed pinacol- and catecholboryls, and the other occupied solely by a (disordered) pinacolboryl. CF_3 groups and hexane solvent molecules are also disordered.

Comment

The title material, (I)·(II)·C_6H_{14}, was obtained as a concomitant by-product during recrystallization of *cis*-1,2-bis-[4-(trifluoromethyl)phenyl]-1-pinacolboryl-2-catecholboryl-ethene, (I), prepared from *cis*-1,2-bis[4-(trifluoromethyl)-phenyl]-1,2-bis(catecholboryl)ethene by treatment with pinacol (Adams *et al.*, 2006).



(I) (II)

The molecules of (I) and *cis*-1,2-bis[4-(trifluoromethyl)-phenyl]-1,2-bis(pinacolboryl)ethene, (II), share the same general position in the crystal structure, whereas a hexane molecule of crystallization is chaotically disordered in a cavity around the inversion centre $(0, \frac{1}{2}, \frac{1}{2})$. Thus, the asymmetric unit comprises half a molecule of each component. The molecular structures of (I) and (II) are shown in Fig. 1, and the way these molecules are superimposed in the crystal structure is illustrated in Fig. 2.

The diphenylethene units of (I) and (II) coincide strictly. The boryl substituent at C13 is a superposition of pinacolboryl (atoms labelled *A*) and catecholboryl (atoms labelled *B*) with equal probability, atoms B1 and O1 having the same positions in either case. Methyl atoms C3*A* and C5*A* form impossibly short intermolecular contacts [2.01 (1) and 2.03 (1) Å, respectively] with their equivalents related by the rotation

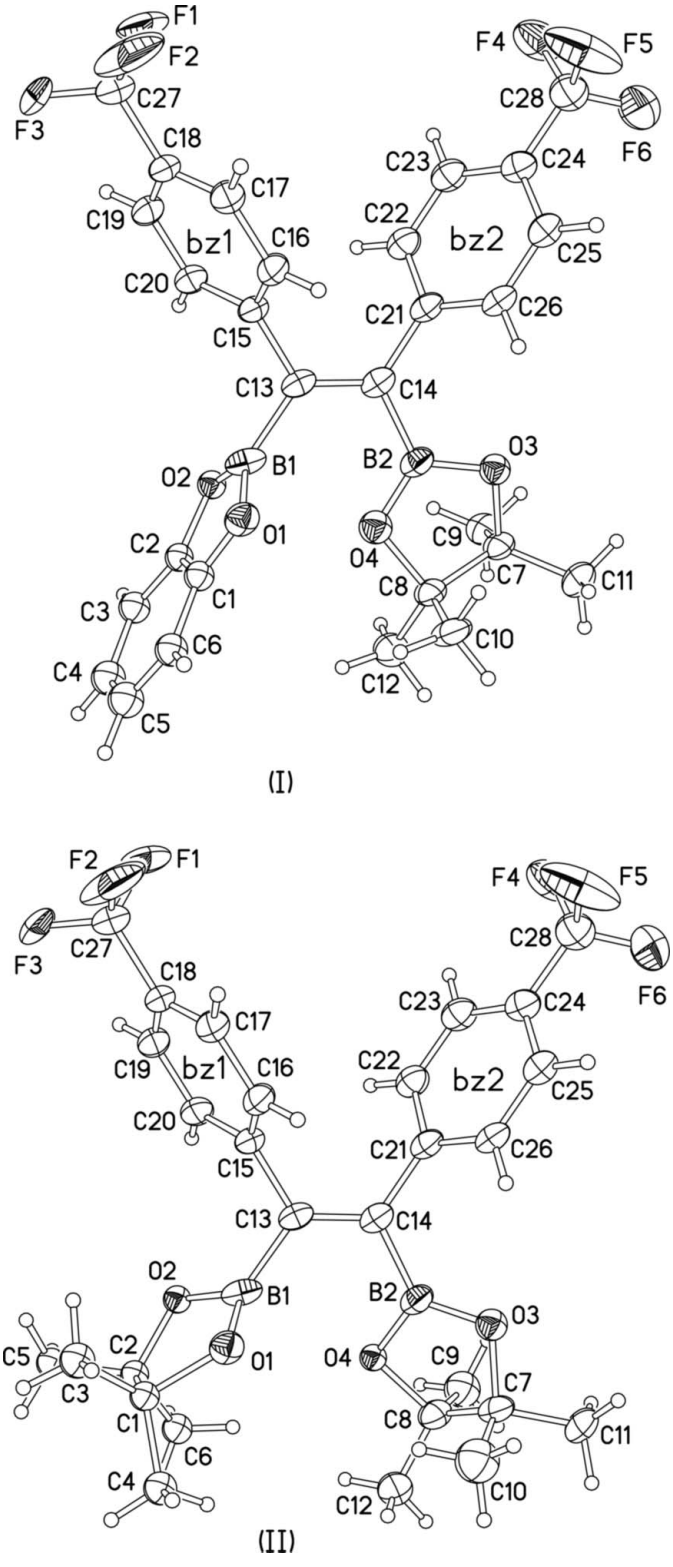


Figure 1
The molecular structures of (I) (component *B*) and (II) (component *A*) in the crystal structure of (I)·(II)·C_6H_{14}. Displacement ellipsoids are drawn at the 30% probability level. The minor positions of atoms F1, F2 and F3 have been omitted.

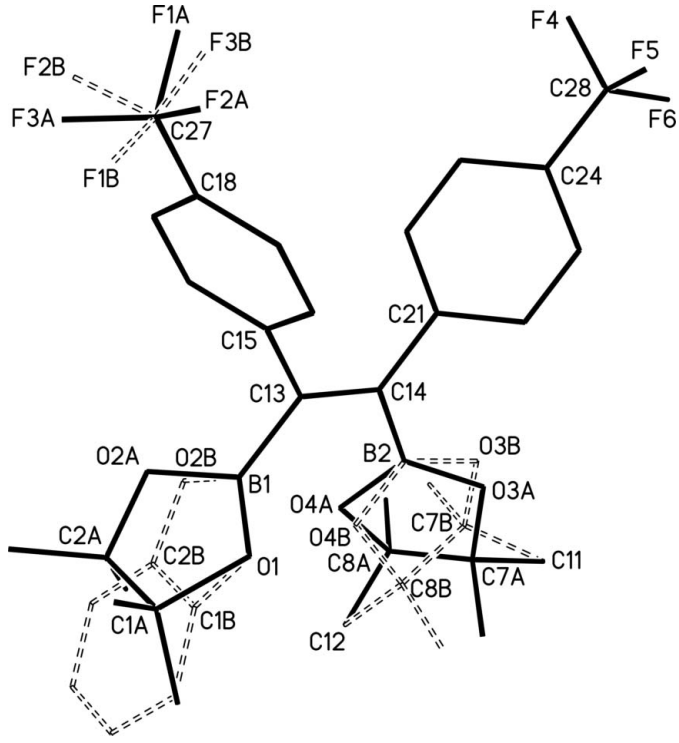


Figure 2
Superposition and disorder of the molecules of (I) and (II). H atoms have been omitted.

($\frac{1}{2}-x, y, \frac{3}{2}-z$). Thus two molecules of (II) cannot occur next to each other across this twofold axis, *i.e.* the disorder is not completely random.. However, refinement of the structure in space group *Pn* was unstable. In fact, sharing of the same crystal site by pinacol- and catecholboryl groups has been observed in the structure of pure compound (I), where one of the independent molecules has crystallographic C_2 symmetry (Adams *et al.*, 2006).

The substituent at C14 is entirely pinacolboryl, but all of its atoms except B2, C11 and C12 are disordered with 1:2 probability between positions *A* and *B*, which correspond to oppositely twisted conformations of the heterocycle. Either conformation is sterically compatible with the presence of the catecholboryl group in the same molecule, but the *B* conformation is probably more so, since the intramolecular contact O4*A*···O2*B* = 2.770 (7) Å is somewhat shorter than O4*B*···O2*B* = 2.880 (5) Å.

The CF_3 substituent at C18 was treated as disordered in a 4:1 ratio between two orientations (*A* and *B*) differing by a rotation around the C18—C27 bond. The CF_3 substituent at C24 may also experience a rotational disorder (indicated by large displacement ellipsoids of atoms F3, F4 and F5), although it was impossible to resolve. The disorder of the hexane molecule was also impossible to rationalize. It was approximated by arbitrary atoms C1*S* at the centre, and C2*S* and C3*S* in general positions (with occupancies of 0.5).

The molecular geometry is similar to those of pure (I) and other 1,2-diaryl-1,2-diboryl-substituted ethenes (Clegg *et al.*, 1996*a*,*b*; Lesley *et al.*, 1996). The olefinic unit C13/C14/B1/B2/C15/C21 is planar within experimental error, in contrast with *cis*-1,2-bis(4-methoxyphenyl)-1,2-bis(pinacolboryl)ethene,

which shows a twist of 9° around the C=C bond (Clegg *et al.*, 1996*a*). Arene rings bz1 and bz2 (Fig. 1) are inclined to the olefinic plane by 64.3 (1) and 58.4 (1)°, respectively, the planar catecholboryl group by 88.3 (1)°, the coordination plane of B1 in the pinacolboryl group by 70.2 (3)°, and the coordination plane of B2 in the *A* conformation by 31.0 (3)°, and in the *B* conformation by 1.3 (3)°.

Experimental

Treatment of *cis*-1,2-bis[4-(trifluoromethyl)phenyl]-1,2-bis-(catecholboryl)ethene with pinacol (Adams *et al.*, 2006) resulted in exchange of catecholate for pinacolate on the boryl units, but to a varying extent. Thus, a mixture of (I) and (II) was present in the *n*-hexane solution from which the title crystals were grown.

Crystal data

$C_{28}H_{24}B_2F_6O_4\cdot C_{28}H_{32}B_2F_6O_4\cdot C_6H_{14}$
M_r = 1214.42
Monoclinic, $P2_1/n$
a = 16.598 (2) Å
b = 6.4631 (5) Å
c = 28.535 (3) Å
β = 104.25 (1)°
V = 2966.9 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 120 (2) K
0.22 × 0.15 × 0.15 mm

Data collection

Bruker SMART 6000 CCD area-detector diffractometer
Absorption correction: none
31871 measured reflections
6813 independent reflections
3598 reflections with $I > 2\sigma(I)$
R_{int} = 0.053

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.258
S = 1.07
6812 reflections
414 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

Reflection 002 was partly obscured by the primary beam stopper. All H atoms were placed in geometrically calculated positions and treated as riding on the corresponding C atoms, assuming C—H = 0.98 Å for methyl groups and 0.95 Å for benzene rings, and with U_{iso}(H) = 1.5 or 1.2 times U_{eq}(C), respectively. Methyl atom C11 was refined in a single position with two alternative sets of attached H atoms, and atom C12 was treated likewise (the site-occupancy factors were 0.67 and 0.33).

All disordered non-H atoms were located in a difference Fourier map and refined without constraints (C7*A* and C8*A* with site-occupancy factors 0.33, C7*B*, C8*B* and C9*B* with site-occupancy factors 0.67 and the major positions, site-occupancy factors 0.80, of F1, F2 and F3 in anisotropic, the rest in isotropic approximations). Their occupancies were optimized by trial and error, least-squares refinement of occupancy factors being unstable.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXTL* (Bruker, 2003); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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References

Adams, C. J., Baber, R. A., Batsanov, A. S., Bramham, G., Charmant, J. P. H., Haddow, M. F., Howard, J. A. K., Lam, W. H., Lin, Zh., Marder, T. B., Norman, N. C. & Orpen, A. G. (2006). *Dalton Trans.* pp. 1370–1373.
Bruker (2001). *SMART* (Version 5.625) and *SAINT* (Version 6.02a). Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2003). *SHELXTL*. Version 6.14. Bruker AXS Inc., Madison, Wisconsin, USA.
Clegg, W., Scott, A. J., Lesley, G., Marder, T. B. & Norman, N. C. (1996*a*). *Acta Cryst.* C**52**, 1989–1991.
Clegg, W., Scott, A. J., Lesley, G., Marder, T. B. & Norman, N. C. (1996*b*). *Acta Cryst.* C**52**, 1991–1995.
Lesley, G., Nguyen, P., Taylor, N. J., Marder, T. B., Scott, A. J., Clegg, W. & Norman, N. C. (1996). *Organometallics*, **15**, 5137–5154.