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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GS1035). Services for accessing these data are described at the back of the journal.

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(PMePh₃)(7,8-Et₂-7,8-*nido*-C₂B₉H₁₀)

REIJO SILLANPÄÄ,^a JOSEFINA PEDRAJAS,^b CLARA VIÑAS,^b
 FRANCÉS TEIXIDOR^b AND RAIKKO KIVEKÄS^c

^aDepartment of Chemistry, University of Turku, FIN-20014 Turku, Finland, ^bInstitut de Ciència de Materials de Barcelona (CSIC), Campus UAB, 08193 Bellaterra, Spain, and ^cDepartment of Chemistry, University of Helsinki, FIN-00014 Helsinki, Finland. E-mail: sillanpa@utu.fi

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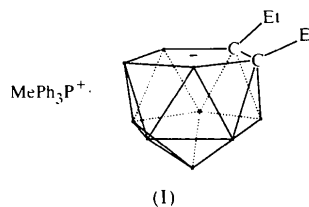
Abstract

Methyltriphenylphosphonium 7,8-diethyl-7,8-dicarba-*nido*-undecaborate(1⁻), C₁₉H₁₈P⁺·C₆H₂₀B₉⁻, is a salt which is formed from the cationic phosphonium ion and anionic *nido* carborane cage. The C—C distance in the boron cage is 1.567 (4) Å.

Comment

Our studies concerning *closo*-1,2-dicarbadodecaborane(12)s have led us to conclude that the C—C distance in the cage can be modified (Sillanpää *et al.*, 1996). In the case of relevant *nido* cages, the elongation seems to be less striking. For example, in *nido*-(7,8-C₂B₉H₁₂)⁻ the C—C distance is 1.542 (3) Å (Buchanan *et al.*, 1990) and in *nido*-(7,8-Ph₂-7,8-C₂B₉H₁₀)⁻ (NH₄⁺ salt) 1.590 (5) Å and for [NMe₃(CH₂Ph)]⁺ salt 1.602 (3) Å (Cowie *et al.*, 1993). In order to get more

information on the dependence of the C—C distance as a function of the C substituent, the crystal structure of (PMePh₃)(7,8-Et₂-7,8-C₂B₉H₁₀), (I), is now reported.



An anionic structural unit of the salt is shown in Fig. 1. The methyl group of one ethyl arm bonded to the cage C atom is orientated up from the upper belt (atoms C7, C8, B9, B10 and B11) of the boron cage. The methyl group of the other ethyl group is approximately in the same plane of the upper-belt atoms. The bond angles and distances in the anion and cation are normal, but some distortions are found in the anion.

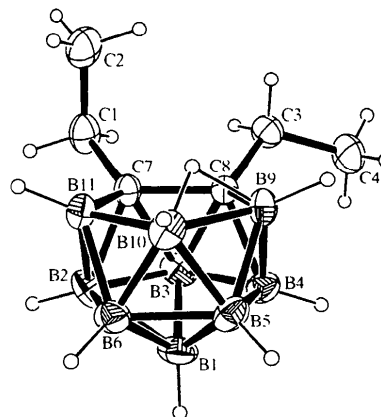


Fig. 1. ORTEPII (Johnson, 1976) plot of the anionic part of (I) in the asymmetric unit. Only the heavy atoms have been labelled. Displacement ellipsoids are drawn at the 20% probability level.

In the lower belt (B2, B3, B4, B5 and B6), the B—B distances are in the range 1.730 (7)–1.806 (7) Å (Table 1), while in the upper belt the C7—C8 bond length is 1.567 (4) Å (the shortest bond) and the B9—B10 bond length is 1.843 (7) Å (the longest bond). Bond angles in the lower belt range from 106.0 (3) to 109.8 (3)° and in the upper belt from 101.5 (3) to 114.5 (3)°.

Alkyl substituents on C atoms in 7,8-dicarboranes seem to have quite a small effect on the C—C bond distance. Aromatic substituents, compared with H substituents, lengthen the C—C distance by about 0.05 Å. Generally this follows with that which has been found for relevant *closo* cages, but in the *nido* case this effect is much weaker.

Experimental

The title compound was prepared using a method described by Viñas *et al.* (1997).

Crystal data

C₁₉H₁₈P*·C₆H₂₀B₉⁻

M_r = 466.81

Monoclinic

*P*2₁/*c*

a = 11.6141 (15) Å

b = 15.9238 (14) Å

c = 15.115 (2) Å

β = 100.31 (1)°

V = 2750.3 (6) Å³

Z = 4

D_x = 1.127 Mg m⁻³

D_m not measured

Mo *K*α radiation

λ = 0.71069 Å

Cell parameters from 20 reflections

θ = 6.6–9.2°

μ = 0.114 mm⁻¹

T = 294 (2) K

Prism

0.38 × 0.36 × 0.26 mm

Colourless

Data collection

Rigaku AFC-5S diffractometer

ω–2θ scans

Absorption correction: none

5449 measured reflections

4836 independent reflections

2427 reflections with

I > 2σ(*I*)

*R*_{int} = 0.139

θ_{max} = 25.09°

h = 0 → 13

k = 0 → 18

l = –18 → 18

3 standard reflections

every 150 reflections

intensity decay: none

Refinement

Refinement on *F*²

R[*F*² > 2σ(*F*²)] = 0.066

wR(*F*²) = 0.149

S = 1.006

4836 reflections

356 parameters

H atoms treated by a

mixture of independent

and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0485P)^2 + 0.9040P]$

where $P = (F_o^2 + 2F_c^2)/3$

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.231 e Å⁻³

Δρ_{min} = –0.274 e Å⁻³

Extinction correction: none

Scattering factors from

International Tables for Crystallography (Vol. C)

Table 1. Selected geometric parameters (Å, °)

P—C19	1.780 (3)	B2—B6	1.734 (7)
P—C25	1.782 (3)	B2—B3	1.744 (7)
P—C12	1.787 (3)	B3—B4	1.730 (7)
P—C13	1.795 (3)	B4—B5	1.745 (6)
C7—C8	1.567 (4)	B5—B6	1.807 (7)
C7—B11	1.610 (5)	B9—B10	1.843 (7)
C8—B9	1.620 (5)	B10—B11	1.784 (6)
C1—C7—C8	119.1 (3)	B3—B4—B5	109.8 (3)
C8—C7—B11	114.5 (3)	B4—B5—B6	106.0 (3)
C3—C8—C7	118.3 (3)	B2—B6—B5	107.3 (3)
C7—C8—B9	110.6 (3)	C8—B9—B10	106.7 (3)
B6—B2—B3	109.1 (4)	C7—B11—B10	106.5 (3)
B4—B3—B2	107.7 (3)		

H atoms bonded to C were fixed and the H atoms bonded to B were refined with isotropic displacement parameters.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1995). Cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1995). Data reduction: *TEXSAN* (Molecular Structure Corporation, 1989). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used

to refine structure: *SHELXL97* (Sheldrick, 1997. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL97*.

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(1*R*,2*R*,3*S*,4*S*,5*S*,6*S*)-exo-2-Cyano-exo-3-[(*S*)-1,2-dibenzyloxyethyl]-exo-5-iodobicyclo[2.2.1]heptane-endo-2,6-carbolactone

MIRIAM ALÍAS, ELENA BUÑUEL, CARLOS CATIVIOLA, MARÍA D. DÍAZ-DE-VILLEGAS AND JOSÉ A. GÁLVEZ

Departamento de Química Orgánica, Instituto de Ciencia de Materiales de Aragón, Universidad de Zaragoza—CSIC, 50009 Zaragoza, Spain. E-mail: jagl@posta.unizar.es

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Abstract

In the title compound (C₂₅H₂₄INO₄), an enantiomerically pure iodolactone, the absolute configurations at the chiral C1, C2, C3, C4, C5 and C6 centres have been unambiguously assigned as 1*R*, 2*R*, 3*S*, 4*S*, 5*S* and 6*S*, respectively. In the bicyclo[2.2.1]heptane (norbornane) unit, the six-membered ring presents a boat conformation, and is fused with a five-membered lactone ring which adopts an envelope conformation.