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

Te—C1	2.132 (7)	Te—Cl1	2.469 (5)
Te—Cl3	2.280 (2)	Cl4—C2	1.763 (8)
Te—Cl2	2.463 (5)	C1—C2	1.307 (10)
C1—Te—Cl3	97.8 (2)	C1—Te—Cl1	86.8 (5)
C1—Te—Cl2	88.4 (5)	Cl3—Te—Cl1	87.9 (2)
Cl3—Te—Cl2	89.4 (2)	Cl2—Te—Cl1	174.15 (12)

The phenyl rings were refined as rigid groups with their H atoms placed in calculated positions at fixed C—H distances of 0.93 Å, each riding on a carrier atom, and with isotropic displacement parameters amounting to 1.2 times the value of the equivalent isotropic displacement parameter of the atom to which they were attached. The Flack (1983) parameter is 0.02 (5).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ZORTEP* (Zsolnai, 1995). Software used to prepare material for publication: *SHELXL97*.

This work has received partial support from FAPESP (Proc. 94/1213-5), CNPq and CAPES. RLC thanks FAPESP for a fellowship (98/05787-7). The X-ray facility at the Instituto de Química–USP was established with the assistance of FAPESP (94/2061-4).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GS1044). Services for accessing these data are described at the back of the journal.

References

Chakravorty, R., Irgolic, K. J. & Meyers, E. A. (1985). *Acta Cryst.* C**41**, 1545–1547.
Chieffi, A., Menezes, P. H. & Comasseto, J. V. (1997). *Organometallics*, **16**, 809–811.
Ciunik, Z., Berski, S., Latajka, Z. & Leszczynski, J. (1998). *J. Mol. Struct.* **442**, 125–134.
Comasseto, J. V. (1993). *Rev. Heteroatom. Chem.* **9**, 61–86.
Comasseto, J. V., Ling, L. V., Petragnani, N. & Stefani, H. A. (1997). *Synthesis*, 373–403.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
Gillespie, R. J. (1972). In *Molecular Geometry*, pp. 6–21. London: Van Nostrand Reinhold.
Hargittai, I. & Rozsnodai, B. (1986). *The Chemistry of Organic Selenium and Tellurium Compounds*, Vol. 1, edited by S. Patai & Z. Rappoport, pp. 63–155. New York: John Wiley.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Pauling, L. (1960). *The Nature of the Chemical Bond*, 3rd ed. Ithaca: Cornell University Press.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473
Sheldrick, G. M. (1997). *SHELXL97. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Ziolo, R. F. & Troup, J. M. (1983). *J. Am. Chem. Soc.* **105**, 229–235.
Zsolnai, L. (1995). *ZORTEP. An Interactive Molecular Graphics Program*. University of Heidelberg, Germany.
Zukerman-Schpector, J., Comasseto, J. V. & Stefani, H. A. (1995). *Acta Cryst.* C**51**, 861–863.

© 1999 International Union of Crystallography
Printed in Great Britain – all rights reserved

Acta Cryst. (1999). C**55**, 1579–1581

1,3a,5-Triphenyl-3a,4,5,6-tetrahydro-1,2,4-oxadiazolo[4,5-*a*][1,5]benzodiazepine

Jiaxi Xu and Sheng Jin

College of Chemistry and Molecular Engineering, Peking University, Beijing 100871, People's Republic of China. E-mail: jxxu@chemms.chem.pku.edu.cn

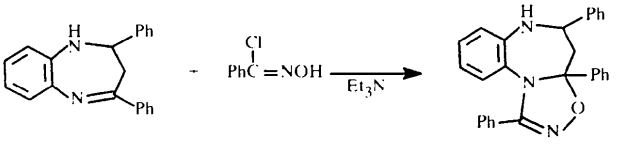
(Received 31 March 1999; accepted 10 May 1999)

Abstract

The title compound, $C_{28}H_{23}N_3O$, has a *cis* ring-fusion tricyclic structure, which is formed from a benzene ring, a seven-membered heterocyclic diazepine ring and a 1,2,4-oxadiazole ring. The 1,5-diazepine ring has a slightly distorted boat-like conformation, whereas the 1,2,4-oxadiazole ring adopts an envelope conformation.

Comment

Benzodiazepine derivatives containing additional rings are potential pharmaceutical agents (Sternbach, 1979; Xu & Jin, 1994; Bock *et al.*, 1989). In connection with investigations on possible approaches to novel benzodiazepine derivatives with additional fused heterocyclic rings (Xu & Jin, 1992, 1999; Xu *et al.*, 1998), we have tested the C=N double bond of the 2,3-dihydro-1*H*-1,5-benzodiazepines as a dipolarophile in the 1,3-dipolar cycloaddition of nitrile oxides (Xu *et al.*, 1999) and obtained some 3a,4,5,6-tetrahydro-1,2,4-oxadiazolo[4,5-*a*][1,5]benzodiazepine derivatives with potential central nervous system depressant and anti-HIV activities. In order to elucidate the conformation of the 1,3-cycloadduct of 2,3-dihydro-1*H*-1,5-benzodiazepine with nitrile oxide, the crystal structure of the title compound, (I), was determined.



(I)

The molecular backbone of (I) is a tricyclic system, formed from a benzene ring, a seven-membered heterocyclic 1,5-diazepine ring and a 1,2,4-oxadiazole ring. The central seven-membered heterocycle is in a slightly distorted boat-like conformation and is *cis*-fused to the 1,2,4-oxadiazole ring at N2 and C9, while the latter moiety is in an envelope conformation. The phenyl group on C7 is equatorial to the central ring, but that on C9 is axial (Fig. 1), hence the cycloaddition reaction is a *cis*-addition reaction. The molecule contains two chiral

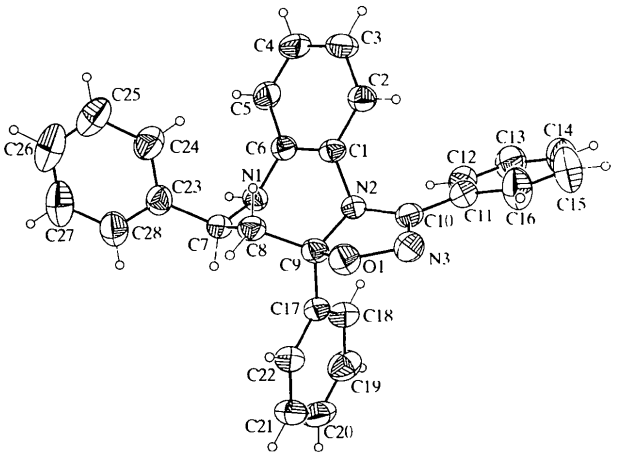


Fig. 1. Perspective view (*XP*; Siemens, 1990) of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as circles of arbitrary radii.

carbon atoms, C7 and C9, with a *rel*-(*RS*) configuration.

The conformation of the central seven-membered ring was determined using the least-squares plane passing through atoms N1, N2 and C9. Atoms C1, C6, C7 and C8 lie 0.868 (3), 0.833 (2), 0.462 (2) and 0.958 (3) Å, respectively, above this plane. The dihedral angle of 18.0 (2)° between the N1/N2/C9 and N1/N2/C7 planes in the seven-membered diazepine ring also shows it to be in a twisted-boat conformation. The 1,2,4-oxadiazoline ring adopts an envelope conformation with atoms N2, C10, N3 and O1 in a common plane, and the smallest torsion angle of the oxadiazoline ring is O1—N3—C10—N2 [−4.2 (2)°]. Atom C9 is 0.431 (3) Å out of this plane. The phenyl ring (C11–C16) makes an angle of only 18.8 (2)° with the common plane passing through atoms N2, C10, N3 and O1.

Experimental

To a stirred solution of 2,4-diphenyl-2,3-dihydro-1*H*-1,5-benzodiazepine (2.5 mmol) and triethylamine (2.5 mmol) in a mixture of anhydrous diethyl ether (25 ml) and tetrahydrofuran (10 ml), a solution of α-chlorobenzaldehyde oxime (2.5 mmol) in anhydrous diethyl ether (5 ml) was added slowly dropwise. The mixture was stirred for 3 h at room temperature, washed twice with water and dried over anhydrous $MgSO_4$. The solvent was then evaporated at reduced pressure to give a brown oil. This material was separated by silica-gel chromatography and then recrystallized from a mixture of diethyl ether and cyclohexane to yield colourless crystals of (I) (m.p. 486–487 K). Single crystals for X-ray analysis were obtained by evaporation from a saturated ethyl acetate solution.

Crystal data

$C_{28}H_{23}N_3O$	Mo $K\alpha$ radiation
M_r = 417.49	λ = 0.71073 Å
Triclinic	Cell parameters from 25 reflections
$P\bar{1}$	θ = 5–10°
a = 10.961 (2) Å	μ = 0.077 mm^{-1}
b = 11.775 (2) Å	T = 293 (2) K
c = 10.452 (2) Å	Block
α = 112.60 (3)°	0.40 × 0.25 × 0.20 mm
β = 108.47 (3)°	Colourless
γ = 65.02 (3)°	
V = 1109.5 (3) Å^3	
Z = 2	
D_x = 1.250 Mg m^{-3}	
D_m not measured	

Data collection

Rigaku AFC-6*S* diffractometer	R_{int} = 0.035
$\omega/2\theta$ scans	θ_{max} = 25.01°
Absorption correction: none	h = 0 → 11
3775 measured reflections	k = −12 → 13
3553 independent reflections	l = −12 → 11
2292 reflections with $I > 2\sigma(I)$	3 standard reflections every 150 reflections intensity decay: 1%

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max}$ < 0.001
$R[F^2 > 2\sigma(F^2)]$ = 0.043	$\Delta\rho_{max}$ = 0.173 e Å^{-3}
$wR(F^2)$ = 0.134	$\Delta\rho_{min}$ = −0.209 e Å^{-3}
S = 0.941	Extinction correction: none
3553 reflections	Scattering factors from *International Tables for Crystallography* (Vol. C)
289 parameters	
H-atom parameters constrained	
$w = 1/[\sigma^2(F_o^2) + (0.0775P)^2 + 0.1352P]$ where $P = (F_o^2 + 2F_c^2)/3$	

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

O1—N3	1.434 (2)	N3—C10	1.286 (3)
O1—C9	1.450 (2)	C1—C6	1.391 (3)
N1—C6	1.417 (2)	C7—C8	1.515 (3)
N1—C7	1.495 (3)	C7—C23	1.516 (3)
N2—C10	1.401 (3)	C8—C9	1.515 (3)
N2—C1	1.430 (3)	C9—C17	1.523 (3)
N2—C9	1.473 (2)		
N3—O1—C9	106.08 (14)	C8—C7—C23	113.87 (18)
C6—N1—C7	116.12 (15)	C7—C8—C9	113.07 (17)
C10—N2—C1	121.71 (15)	O1—C9—N2	101.78 (15)
C10—N2—C9	103.22 (16)	O1—C9—C8	108.85 (16)
C1—N2—C9	120.96 (16)	N2—C9—C8	113.32 (17)
C10—N3—O1	106.22 (18)	O1—C9—C17	107.29 (16)
C6—C1—N2	118.70 (16)	N2—C9—C17	111.84 (16)
C2—C1—N2	121.44 (19)	C8—C9—C17	112.95 (17)
C5—C6—N1	121.9 (2)	N3—C10—N2	114.35 (19)
C1—C6—N1	119.27 (18)	N3—C10—C11	122.5 (2)
N1—C7—C8	109.97 (16)	N2—C10—C11	123.07 (19)
N1—C7—C23	111.96 (18)		

H atoms were generated by *HFIX* instructions. H-atom coordinates were allowed to ride on the coordinates of the parent atom, with isotropic U values 1.2 or 1.5 times the equivalent U values of the parent atom.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *SHELXS*97 (Sheldrick, 1997*a*). Program(s) used to solve structure: *SHELXS*97. Program(s) used to refine struc-

ture: *SHELXL*97 (Sheldrick, 1997*b*). Molecular graphics: *XP* (Siemens, 1990). Software used to prepare material for publication: *SHELXL*97.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1252). Services for accessing these data are described at the back of the journal.

References

Bock, M. G., Dipardo, R. M., Evans, B. E., Rittle, K. E., Whitter, W. L., Veber, D. F., Anderson, P. S. & Freidinger, R. M. (1989). *J. Med. Chem.* **32**, 13–16.

Molecular Structure Corporation (1994). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Sheldrick, G. M. (1997*a*). *SHELXS*97. *Program for the Solution of Crystal Structures*. University of Göttingen, Germany.

Sheldrick, G. M. (1997*b*). *SHELXL*97. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Siemens (1990). *XP. Molecular Graphics Program*. Version 4.2 for MS-DOS. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sternbach, L. H. (1979). *J. Med. Chem.* **22**, 1–7.

Xu, J. X. & Jin, S. (1992). *Chin. Chem. Lett.* **3**, 181.

Xu, J. X. & Jin, S. (1994). *Chin. Chem. Lett.* **5**, 557–560.

Xu, J. X. & Jin, S. (1999). *Heteroatom Chem.* **10**, 35–41.

Xu, J. X., Jin, S., Zhang, Z. Y. & Mak, T. C. W. (1998). *Chem. J. Chin. Univ.* **19**, 1070–1073.

Xu, J. X., Wu, H. T. & Jin, S. (1999). *Chin. J. Chem.* **17**, 84–91.

Acta Cryst. (1999). C**55**, 1581–1583

Methyltriphenylphosphonium 2-methyl-1-sulfido-1,2-dicarba-*closo*-dodecaborane(12)

RAIKKO KIVEKÄS,[a] RAJAE BENAKKI,[b] CLARA VIÑAS[b] AND REIJO SILLANPÄÄ[c]

[a]*Department of Chemistry, PO Box 55, FIN-00014 University of Helsinki, Finland,* [b]*Institut de Ciència de Materials de Barcelona, CSCI, Campus UAB, 08193 Bellaterra, Spain, and* [c]*Department of Chemistry, University of Turku, FIN-20014 Turku, Finland. E-mail: kivekas@cc.helsinki.fi*

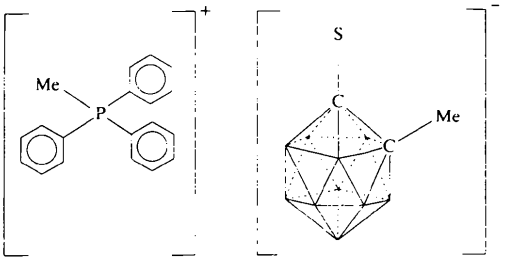
(*Received 15 February 1999; accepted 5 May 1999*)

Abstract

In the title compound, $CH_3(C_6H_5)_3P^+\cdot C_3H_{13}B_{10}S^-$, the negative charge of the sulfido group connected to the carborane cage is balanced by the phosphonium cation. In the cation, one of the three phenyl groups is disordered, exhibiting two orientations. In the anion, the C_{cage}—C_{cage} distance is 1.792 (5) Å.

Comment

In 1,2-dicarba-*closo*-dodecaboranes, the C_{cage}—C_{cage} or C1—C2 bond length varies considerably, depending on the number of substituents and atom species connected to the cluster C atoms. Thus, values of 1.57 (1)–1.634 (3) Å are reported for the 1,2-H_2 compounds (Šubrtová *et al.*, 1980; Novák *et al.*, 1983), and much larger values of 1.816 (6)–1.858 (5) Å are observed for compounds in which S atoms are connected to both cluster C atoms (Teixidor, Romerosa *et al.*, 1990; Teixidor, Viñas *et al.*, 1990). In recent papers, we have studied the elongation of the C1—C2 distance and suggested an empirically derived equation to calculate this distance (Kivekäs *et al.*, 1994, 1995). In order to synthesize new ligands for complexation and to study further the contribution of different types of C_{cage} substituents on the lengthening of the C1—C2 bond, we have now synthesized the title compound, (I), and determined its crystal structure.



(I)

The structure of (I) consists of discrete $CH_3(C_6H_5)_3P^+$ and $C_3H_{13}B_{10}S^-$ ions without any close contact between them. In the $CH_3(C_6H_5)_3P^+$ cation, one of the phenyl groups is disordered, exhibiting two orientations, with a dihedral angle between these two phenyl orientations of 33.0 (12)°. The bond parameters of the cation are as expected.

In the $C_3H_{13}B_{10}S^-$ anion, the sulfido and methyl groups are connected to the cluster C atoms of

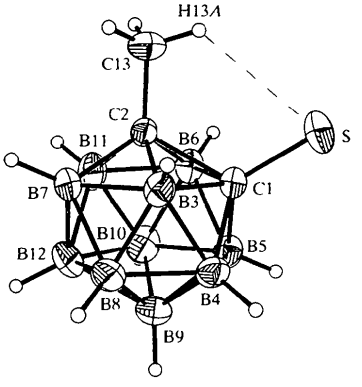


Fig. 1. A simplified view of the anion of (I) with displacement ellipsoids at the 20% probability level and H atoms shown as spheres of an arbitrary radius.

© 1999 International Union of Crystallography
Printed in Great Britain – all rights reserved