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1,6-Bis(diphenylarsino)hexane

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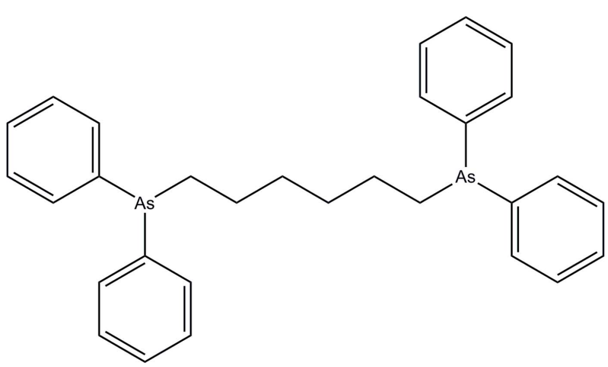
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.035; wR factor = 0.084; data-to-parameter ratio = 26.7.

The title diphenylarsino compound, $C_{30}H_{32}As_2$ or $Ph_2As(CH_2)_6AsPh_2$, lies about a crystallographic inversion centre located at the mid-point of the central $Csp^3—Csp^3$ bond of the methylene chain. The two benzene rings bonded to As are inclined to one another at a dihedral angle of 75.98 (8)°. In the crystal structure, weak intermolecular C—H$\cdots\pi$ interactions stack the molecules down the b axis.

Related literature

For general background to and applications of diphenylarsino derivatives, see: Hill *et al.* (1983); Song *et al.* (2005). For the preparation of the title compound, see: Aguiar & Archibald (1967); Burfield *et al.* (1977, 1978); Tzschach & Lange (1962). For closely related structures, see: Hill *et al.* (2001); Shawkataly *et al.* (2005). For information on the Cambridge Structural Database, see: Allen (2002). For bond-length data, see: Allen *et al.* (1987). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).



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Experimental

Crystal data

$C_{30}H_{32}As_2$
M_r = 542.40
Monoclinic, $P2_1/c$
a = 12.3774 (2) Å
b = 5.7145 (1) Å
c = 18.1263 (3) Å
β = 101.076 (1)°
V = 1258.20 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.67 mm^{-1}
T = 100 K
0.44 × 0.29 × 0.03 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005) T_{min} = 0.384, T_{max} = 0.919
24043 measured reflections
5572 independent reflections
4144 reflections with $I > 2\sigma(I)$
R_{int} = 0.052

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.084
S = 1.03
5572 reflections
209 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C15—H15$B\cdots Cg1^{i}$	0.97 (3)	2.81 (3)	3.776 (2)	169.9 (18)
C4—H4$\cdots Cg2^{ii}$	0.91 (2)	2.80 (2)	3.708 (2)	173.2 (19)
C9—H9$\cdots Cg2^{iii}$	0.91 (2)	2.97 (2)	3.617 (2)	129.5 (16)

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x, -y+\frac{1}{2}, z-\frac{3}{2}$; (iii) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$. $Cg1$ and $Cg2$ are the centroids of the C1–C6 and C7–C12 benzene rings, respectively.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2648).

References

Aguiar, A. M. & Archibald, T. G. (1967). *J. Org. Chem.* **32**, 2627–2628.
Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.
Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Burfield, D. R., Gan, G. H. & Smithers, R. H. (1978). *J. Appl. Chem. Biotech.* **28**, 23–30.
Burfield, D. R., Lee, K.-H. & Smithers, R. H. (1977). *J. Org. Chem.* **42**, 3060–3065.

Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
Hill, N. J., Levason, W., Reid, G. & Webster, M. (2001). *Acta Cryst.* E**57**, o700–o701.
Hill, W. E., Minahan, D. M. A. & McAuliffe, C. A. (1983). *Inorg. Chem.* **22**, 3382–3387.
Shawkataly, O., Chong, M.-L., Fun, H.-K., Didierjean, C. & Aubry, A. (2005). *Acta Cryst.* E**61**, 3351–3352.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Song, L.-C., Jin, G.-X., Wang, H.-T., Zhang, W.-X. & Hu, Q.-M. (2005). *Organometallics* **24**, 6464–6471.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Tzschach, A. & Lange, W. (1962). *Chem. Ber.* **95**, 1360–1366.

supplementary materials

Acta Cryst. (2009). E**65**, o2591-o2592 [**doi:10.1107/S1600536809036757**]

1,6-Bis(diphenylarsino)hexane

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Comment

1,6-Bis(diphenylarsino)hexane has been used for trans chelation in transition metal complexes (Hill *et al.*, 1983). A search of the November 2008 release of the Cambridge Structural Database (Allen, 2002) revealed no structures of complexes containing the above ligand. Among bis(diphenylarsino)alkanes, only the structure of 1,2-bis(diphenylarsino)ethane (Hill *et al.*, 2001) and complexes of the ligand 1,3-bis(diphenylarsino)propane (Song *et al.*, 2005) are known.

The title compound (Fig. 1), contains a crystallographic inversion centre at the mid-point of the central Csp^3—Csp^3 (C15—C15A) bond [symmetry code of atoms labellel with suffix A: -x+1, -y+1, -z+1]. The C1-C6 and C7-C12 benzene rings are inclined to one another, with a dihedral angle of 75.98 (8)°. The bond lengths (Allen *et al.*, 1987) are comparable to those found in closely related structures (Hill *et al.*, 2001; Shawkataly *et al.*, 2005). In the crystal structure (Fig. 2), the molecules are stacked down the *b* axis. The crystal structure is consolidated by intermolecular C15—H15B···*Cg*1, C4—H4···*Cg*2 and C9—H9···*Cg*2 interactions (Table 1).

Experimental

Solvents were dried by recommended literature routes (Burfield *et al.*, 1977, 1978) and the title compound was prepared by the reaction of diphenylarsino lithium with 1,6-dibromohexane in dry THF at 273 K under nitrogen atmosphere (Aguiar *et al.*, 1967; Tzschach & Lange, 1962). Colourless plates of suitable quality for single crystal X-ray diffraction studies were obtained by slow evaporation from ethanol solution.

Refinement

All the H atoms were located from difference Fourier map and allowed to refine freely [range of C—H = 0.89 (2) – 0.99 (3) Å].

Figures

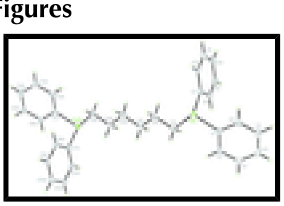

Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids for non-H atoms and the atom-numbering scheme. The suffix A corresponds to the symmetry code [-x+1, -y+1, -z+1].

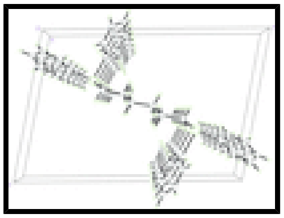

Fig. 2. The crystal structure of the title compound viewed along the *b* axis, showing stacking of molecules along the *b* axis.

1,6-Bis(diphenylarsino)hexane

Crystal data

$C_{30}H_{32}As_2$	$F_{000} = 556$
$M_r = 542.40$	$D_x = 1.432$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 5477 reflections
a = 12.3774 (2) Å	θ = 3.1–32.2°
b = 5.7145 (1) Å	μ = 2.67 mm^{-1}
c = 18.1263 (3) Å	*T* = 100 K
β = 101.076 (1)°	Plate, colourless
V = 1258.20 (4) Å^3	0.44 × 0.29 × 0.03 mm
Z = 2	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	5572 independent reflections
Radiation source: fine-focus sealed tube	4144 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.052$
T = 100 K	$\theta_{max} = 35.1°$
φ and ω scans	$\theta_{min} = 2.3°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	$h = -19 \rightarrow 20$
$T_{min} = 0.384$, $T_{max} = 0.919$	$k = -9 \rightarrow 8$
24043 measured reflections	$l = -29 \rightarrow 26$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.035$	All H-atom parameters refined
$wR(F^2) = 0.084$	$w = 1/[\sigma^2(F_o^2) + (0.0344P)^2 + 0.3375P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
5572 reflections	$\Delta\rho_{max} = 0.58$ e Å^{-3}
209 parameters	$\Delta\rho_{min} = -0.47$ e Å^{-3}

Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cyrosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1)K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
As1	0.351354 (13)	0.98218 (3)	0.284872 (8)	0.01668 (5)
C1	0.29473 (13)	0.8690 (3)	0.18275 (8)	0.0183 (3)
C2	0.32226 (14)	0.6566 (3)	0.15347 (9)	0.0226 (3)
C3	0.27896 (15)	0.5965 (4)	0.07878 (10)	0.0264 (4)
C4	0.20763 (15)	0.7469 (4)	0.03339 (10)	0.0299 (4)
C5	0.17979 (17)	0.9578 (4)	0.06212 (10)	0.0313 (4)
C6	0.22374 (16)	1.0190 (3)	0.13583 (10)	0.0257 (3)
C7	0.20975 (13)	0.9750 (3)	0.31860 (8)	0.0165 (3)
C8	0.13587 (13)	0.7891 (3)	0.30118 (9)	0.0192 (3)
C9	0.03431 (14)	0.7930 (3)	0.32299 (9)	0.0224 (3)
C10	0.00492 (14)	0.9838 (3)	0.36256 (9)	0.0225 (3)
C11	0.07749 (14)	1.1692 (3)	0.38019 (9)	0.0235 (3)
C12	0.17944 (14)	1.1653 (3)	0.35879 (9)	0.0208 (3)
C13	0.41464 (14)	0.6910 (3)	0.33284 (9)	0.0195 (3)
C14	0.43828 (15)	0.7124 (3)	0.41818 (9)	0.0218 (3)
C15	0.48951 (15)	0.4927 (3)	0.45720 (9)	0.0234 (3)
H2	0.3700 (19)	0.552 (4)	0.1820 (12)	0.026 (6)*
H3	0.303 (2)	0.458 (4)	0.0593 (13)	0.031 (6)*
H4	0.1799 (19)	0.704 (4)	−0.0149 (13)	0.040 (6)*
H5	0.128 (2)	1.066 (5)	0.0299 (15)	0.048 (7)*
H6	0.2055 (18)	1.172 (4)	0.1569 (12)	0.034 (6)*
H8	0.1550 (16)	0.654 (4)	0.2729 (11)	0.026 (5)*
H9	−0.0121 (17)	0.669 (4)	0.3102 (12)	0.033 (6)*
H10	−0.063 (2)	0.989 (4)	0.3763 (14)	0.033 (7)*
H11	0.0573 (18)	1.300 (4)	0.4072 (12)	0.036 (6)*
H12	0.2278 (16)	1.291 (4)	0.3710 (11)	0.024 (5)*
H13A	0.4768 (18)	0.668 (4)	0.3164 (12)	0.033 (6)*
H13B	0.364 (2)	0.570 (4)	0.3189 (13)	0.032 (6)*
H14A	0.370 (2)	0.752 (5)	0.4355 (13)	0.043 (7)*

H14B	0.4872 (17)	0.844 (4)	0.4344 (11)	0.026 (5)*
H15A	0.4390 (19)	0.362 (5)	0.4414 (13)	0.037 (6)*
H15B	0.556 (2)	0.465 (4)	0.4373 (14)	0.032 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
As1	0.01574 (8)	0.01865 (8)	0.01533 (7)	−0.00094 (6)	0.00222 (5)	0.00114 (6)
C1	0.0172 (6)	0.0221 (7)	0.0160 (6)	−0.0017 (6)	0.0044 (5)	0.0026 (5)
C2	0.0218 (7)	0.0269 (8)	0.0194 (7)	0.0002 (7)	0.0050 (6)	0.0007 (6)
C3	0.0285 (9)	0.0296 (9)	0.0225 (8)	−0.0069 (7)	0.0087 (7)	−0.0053 (7)
C4	0.0283 (9)	0.0462 (11)	0.0147 (7)	−0.0115 (8)	0.0028 (6)	−0.0013 (7)
C5	0.0299 (9)	0.0430 (11)	0.0185 (7)	0.0012 (8)	−0.0011 (7)	0.0079 (7)
C6	0.0269 (8)	0.0299 (9)	0.0195 (7)	0.0042 (7)	0.0027 (6)	0.0046 (6)
C7	0.0169 (6)	0.0182 (7)	0.0137 (6)	0.0008 (5)	0.0014 (5)	0.0017 (5)
C8	0.0195 (7)	0.0187 (7)	0.0188 (7)	0.0003 (6)	0.0022 (5)	−0.0016 (5)
C9	0.0204 (7)	0.0243 (8)	0.0217 (7)	−0.0025 (6)	0.0018 (6)	−0.0010 (6)
C10	0.0173 (7)	0.0303 (9)	0.0198 (7)	0.0033 (6)	0.0035 (6)	0.0008 (6)
C11	0.0258 (8)	0.0243 (8)	0.0200 (7)	0.0046 (7)	0.0034 (6)	−0.0030 (6)
C12	0.0219 (7)	0.0195 (7)	0.0200 (7)	−0.0005 (6)	0.0013 (6)	−0.0013 (6)
C13	0.0186 (7)	0.0239 (8)	0.0156 (6)	0.0047 (6)	0.0022 (5)	0.0022 (6)
C14	0.0235 (8)	0.0263 (8)	0.0149 (6)	0.0071 (7)	0.0016 (6)	0.0015 (6)
C15	0.0250 (8)	0.0283 (9)	0.0154 (6)	0.0096 (7)	0.0003 (6)	0.0008 (6)

Geometric parameters (Å, °)

As1—C1	1.9590 (15)	C8—H8	0.98 (2)
As1—C7	1.9644 (16)	C9—C10	1.391 (2)
As1—C13	1.9688 (16)	C9—H9	0.91 (2)
C1—C2	1.393 (2)	C10—C11	1.386 (3)
C1—C6	1.394 (2)	C10—H10	0.93 (3)
C2—C3	1.400 (2)	C11—C12	1.390 (2)
C2—H2	0.93 (2)	C11—H11	0.95 (2)
C3—C4	1.384 (3)	C12—H12	0.94 (2)
C3—H3	0.94 (2)	C13—C14	1.523 (2)
C4—C5	1.383 (3)	C13—H13A	0.89 (2)
C4—H4	0.91 (2)	C13—H13B	0.93 (3)
C5—C6	1.387 (3)	C14—C15	1.519 (2)
C5—H5	0.99 (3)	C14—H14A	0.98 (2)
C6—H6	0.99 (2)	C14—H14B	0.98 (2)
C7—C8	1.398 (2)	C15—C15^{i}	1.525 (3)
C7—C12	1.399 (2)	C15—H15A	0.98 (3)
C8—C9	1.388 (2)	C15—H15B	0.97 (3)
C1—As1—C7	96.24 (6)	C10—C9—H9	121.6 (14)
C1—As1—C13	100.26 (7)	C11—C10—C9	119.72 (16)
C7—As1—C13	98.51 (7)	C11—C10—H10	119.9 (13)
C2—C1—C6	118.37 (15)	C9—C10—H10	120.4 (14)
C2—C1—As1	125.33 (12)	C10—C11—C12	120.41 (16)

C6—C1—As1	116.28 (13)	C10—C11—H11	119.8 (14)
C1—C2—C3	120.38 (17)	C12—C11—H11	119.8 (14)
C1—C2—H2	121.8 (14)	C11—C12—C7	120.44 (16)
C3—C2—H2	117.8 (14)	C11—C12—H12	119.7 (12)
C4—C3—C2	120.32 (18)	C7—C12—H12	119.9 (13)
C4—C3—H3	120.8 (14)	C14—C13—As1	111.29 (11)
C2—C3—H3	118.7 (15)	C14—C13—H13A	110.1 (14)
C5—C4—C3	119.60 (16)	As1—C13—H13A	105.9 (15)
C5—C4—H4	121.5 (16)	C14—C13—H13B	108.8 (15)
C3—C4—H4	118.9 (16)	As1—C13—H13B	108.4 (15)
C4—C5—C6	120.16 (18)	H13A—C13—H13B	112 (2)
C4—C5—H5	119.9 (16)	C15—C14—C13	112.84 (14)
C6—C5—H5	119.9 (16)	C15—C14—H14A	110.5 (15)
C5—C6—C1	121.15 (18)	C13—C14—H14A	109.6 (13)
C5—C6—H6	121.0 (13)	C15—C14—H14B	108.6 (12)
C1—C6—H6	117.9 (12)	C13—C14—H14B	110.8 (12)
C8—C7—C12	118.61 (15)	H14A—C14—H14B	104.2 (19)
C8—C7—As1	121.98 (12)	C14—C15—C15^{i}	113.73 (18)
C12—C7—As1	119.36 (12)	C14—C15—H15A	107.9 (14)
C9—C8—C7	120.81 (15)	C15^{i}—C15—H15A	108.3 (14)
C9—C8—H8	119.0 (12)	C14—C15—H15B	105.7 (14)
C7—C8—H8	120.2 (12)	C15^{i}—C15—H15B	113.3 (15)
C8—C9—C10	120.01 (16)	H15A—C15—H15B	107.6 (19)
C8—C9—H9	118.4 (14)		
C7—As1—C1—C2	−114.45 (14)	C1—As1—C7—C12	−134.94 (13)
C13—As1—C1—C2	−14.60 (15)	C13—As1—C7—C12	123.67 (13)
C7—As1—C1—C6	67.51 (14)	C12—C7—C8—C9	0.2 (2)
C13—As1—C1—C6	167.36 (13)	As1—C7—C8—C9	−177.24 (12)
C6—C1—C2—C3	−0.2 (2)	C7—C8—C9—C10	0.1 (2)
As1—C1—C2—C3	−178.21 (13)	C8—C9—C10—C11	−0.1 (2)
C1—C2—C3—C4	−0.5 (3)	C9—C10—C11—C12	−0.2 (3)
C2—C3—C4—C5	0.4 (3)	C10—C11—C12—C7	0.6 (2)
C3—C4—C5—C6	0.5 (3)	C8—C7—C12—C11	−0.6 (2)
C4—C5—C6—C1	−1.2 (3)	As1—C7—C12—C11	176.96 (12)
C2—C1—C6—C5	1.0 (3)	C1—As1—C13—C14	−165.02 (12)
As1—C1—C6—C5	179.24 (15)	C7—As1—C13—C14	−67.05 (13)
C1—As1—C7—C8	42.50 (13)	As1—C13—C14—C15	−178.78 (13)
C13—As1—C7—C8	−58.90 (13)	C13—C14—C15—C15^{i}	−178.14 (19)

Symmetry codes: (i) $-x+1, -y+1, -z+1$.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H15B···Cg1^{ii}	0.97 (3)	2.81 (3)	3.776 (2)	169.9 (18)
C4—H4···Cg2^{iii}	0.91 (2)	2.80 (2)	3.708 (2)	173.2 (19)
C9—H9···Cg2^{iv}	0.91 (2)	2.97 (2)	3.617 (2)	129.5 (16)

Symmetry codes: (ii) $-x+1, y-1/2, -z+1/2$; (iii) $x, -y+1/2, z-3/2$; (iv) $-x, y-1/2, -z+1/2$.

Fig. 1

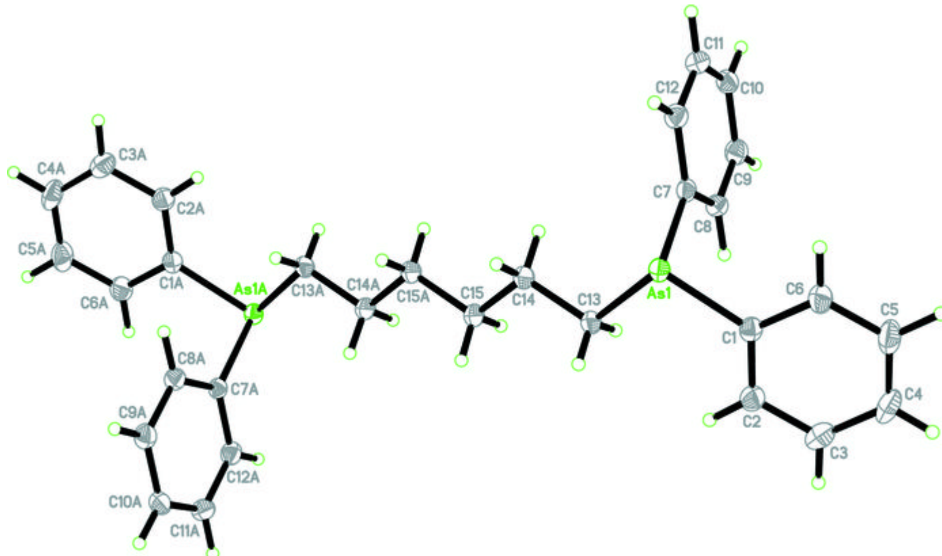
C11
C10
C12
C4A
C3A
C9
C7
C2A
C8
C5A
As1A
C1A
C13A
C14A
C15A
C15
C14
C13
As1
C6
C5
C6A
C1
C8A
C7A
C9A
C4
C2
C3
C12A
C10A
C11A

Fig. 2