Table 2. *Selected geometric parameters* (Å, °)

C1—C2	1.518 (3)	C7—C8	1.501 (3)
C1—C14	1.522 (4)	C8—C9	1.530 (4)
C1—N	1.470 (3)	C9—C10	1.518 (4)
C2—C3	1.388 (3)	C9—N	1.481 (3)
C2—C7	1.394 (3)	C10—C11	1.506 (5)
C3—C4	1.384 (4)	C11—C12	1.504 (4)
C4—C5	1.375 (4)	C12 C13	1.513 (4)
C5—C6	1.372 (4)	C13—N	1.343 (3)
C6—C7	1.399 (3)	C13—O	1.232 (3)
C2—C1—C14	113.0 (2)	C7—C8—C9	112.5 (2)
C2—C1—N	110.8 (2)	C8—C9—C10	113.3 (2)
C14—C1—N	111.3 (2)	C8—C9—N	108.1 (2)
C1—C2—C3	118.8 (2)	C10—C9—N	112.4 (2)
C1—C2—C7	121.9 (2)	C9—C10—C11	111.5 (2)
C3—C2—C7	119.3 (2)	C10—C11—C12	109.3 (2)
C2—C3—C4	121.2 (2)	C11—C12—C13	114.1 (2)
C3—C4—C5	119.9 (3)	C12—C13—N	118.2 (2)
C4—C5—C6	119.3 (3)	C12—C13—O	119.8 (2)
C5—C6—C7	122.0 (2)	N—C13—O	122.0 (2)
C2—C7—C6	118.3 (2)	C1—N—C9	114.6 (2)
C2—C7—C8	121.4 (2)	C1—N—C13	119.3 (2)
C6—C7—C8	120.3 (2)	C9—N—C13	126.1 (2)
N—C1—C2—C7	13.2 (3)	C10—C9—N—C13	10.2 (3)
C2—C1—N—C9	−47.0 (3)	C9—C10—C11—C12	61.0 (3)
C1—C2—C7—C8	0.4 (3)	C10—C11—C12—C13	−50.4 (3)
C2—C7—C8—C9	17.4 (3)	C11—C12—C13—N	20.4 (4)
C7—C8—C9—N	−47.4 (3)	C11—C12—C13—O	−161.3 (2)
N—C9—C10—C11	−40.7 (3)	C12—C13—N—C9	0.3 (3)
C8—C9—N—C1	65.3 (2)	O—C13—N—C9	−178.0 (2)

The θ-scan width was $(1.3 + 0.346\tan\theta)°$, with θ-scan rate 32° min^{-1}; up to four scans on weak reflections and background counts for one quarter of the scan time on each end of every scan were made. H atoms were refined with one common isotropic displacement factor.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *Xtal*3.0 *DIFDAT*, *ABSORB*, *SORTRF* and *ADDREF* (Hall & Stewart, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *Xtal*3.0 *CRYLSQ*. Molecular graphics: *Xtal*3.0. Software used to prepare material for publication: *Xtal*3.0 *BONDLA* and *CIFIO*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1038). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Screw-Chain Structure of 1,10-Phenanthroline Hydrate, $C_{12}H_8N_2.H_2O$

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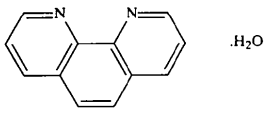
Abstract

The X-ray analysis revealed that the title compound is an infinite 3_1 screw-chain structure with a repeat distance of 8.453 Å. The O atoms of the water connect to one another around the screw axis to form the core of the chain and the *o*-phenanthroline molecules connect through intermolecular hydrogen bonds to these water molecules. The structure can be regarded as a screw supermolecule assembled by hydrogen bonds and is a displacive modulation of an idealized $P3_121$ parent structure.

Comment

o-Phenanthroline (*o*-phen) is well known as a bidentate ligand in coordination chemistry. Many metal complexes involving *o*-phen as a ligand have special properties and in order to obtain a better understanding of structures of the transition metal to *o*-phen chelating systems, many authors have investigated the crystal structures of *o*-phen and *o*-phen.H_2O (Donnay, Donnay & Harding, 1965; Sen, 1974; Nishigaki, Yoshioka & Nakatsu, 1975, 1978). However, no detailed structure determination of *o*-phen.H_2O has been reported. Moreover, it is of interest to compare the crystal and molecular structure of *o*-phen.H_2O with that of *o*-phen and to study the effect of hydrogen bonding on the crystal structure. In this paper, an unrestrained crystal structure analysis is reported. The molecular geometry and unit cell are illustrated in Figs. 1 and 2, respectively.



o-phen

There are three differently orientated molecules [(I), (II) and (III)] in the asymmetric unit (Fig. 1). It can be seen from Tables 2 and 3 that chemically equivalent bond lengths and angles vary greatly, not only between *o*-phen molecules, but also between sides of individual molecules. The most conspicuous differences are N(1)—C(2) 1.358, N(12)—C(11) 1.258 Å in (I); N(15)—C(16) 1.326, N(26)—C(25) 1.293 Å in (II); and N(29)—C(30) 1.343, N(40)—C(39) 1.309 Å in (III). The 1.00 Å spread of pseudo-equivalent distances is not necessarily significant since there are large standard deviations associated with these parameters despite the reasonable value of $R(F)$. Table 3 gives average values for the geometric parameters and these are very reasonable, and can be compared with values for *o*-phen (Nishigaki *et al.*, 1978) and *o*-phen.2HNO$_3$ (Thevenet & Rodier, 1978).

The large calculated errors are a result of pseudo-symmetry. The errors in mean values of $N = 6$ (or 3) pseudo-equivalent values [approximated by $1/(N)^{1/2}$ times the individual errors in Table 3] are probably less than approximated whereas deviations from the mean have effectively the same error as the error on individual values.

From Table 1 it can be seen that the three molecules have the approximate relationship x, y, z; $x + 1/3, y - 1/3, z$; $x - 1/3, y - 2/3, z$. If this symmetry was exact then data with $h - k = 3n \pm 1$ would be unobserved. There is also the approximate relationship $x, x - y, 0.130 - z$ between the two halves of individual molecules. For the polar space group $P3_1$, there is an arbitrary choice of origin along **c** and the relationship corresponds to a twofold-rotation axis passing through 0, 0, 0.065. The structure can be regarded as a displacive modulation of an idealized $P3_121$ parent structure with $Z = 3$ and axes $2a/3 + b/3$, $-a/3 + b/3$, c relative to a, b, c of the final structure. The O atoms of the water molecules would lie on the twofold axis in the parent structure. The hydrogen bonding destroys the twofold symmetry and is the probable cause of the structure modulation.

The structure can be described in terms of pseudo-equivalent 3_1 chains parallel to **c**. The O atoms are connected by O···H—O hydrogen bonds and effectively lie along the 3_1 screw axes. The distances between adjacent O atoms are 3.010, 2.991 and 3.000 Å for chains involving molecules (I), (II) and (III), respectively. Phenanthroline molecules are connected by hydrogen bonds to these core O atoms. The planes of the phenanthroline are at angles of 32.1, 32.3 and 32.5°, respectively, to the screw axes.

Clearly, the extensive hydrogen bonds play a key role in determining the packing geometry of the crystal. Donnay *et al.* (1965) proposed three possible configurations for the interaction between the water and the phenanthroline molecules. However, the actual configuration shows soft intermolecular forces with two N atoms of the phenanthroline molecule and one O atom of the water sharing one H atom. The distances between the O atom and the N atoms are 2.969, 3.145 Å for (I), 3.125, 2.991 Å for (II), and 3.120, 2.999 Å for (III). Each chain can be thought of as a screw supermolecule (Lehn *et al.*, 1987) assembled by hydrogen-bond interactions.

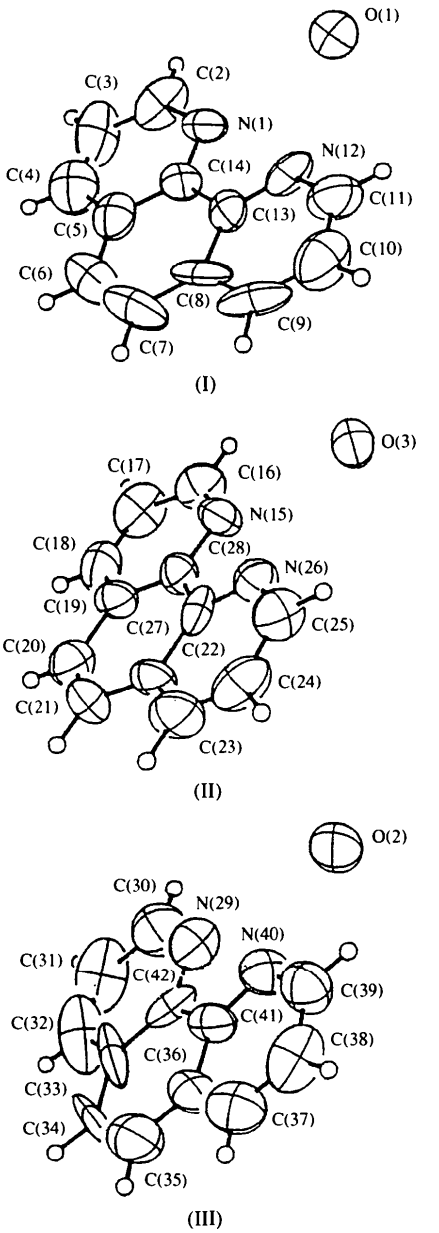


Fig. 1. The three differently orientated molecular configurations. Displacement ellipsoids are plotted at the 50% probability level.

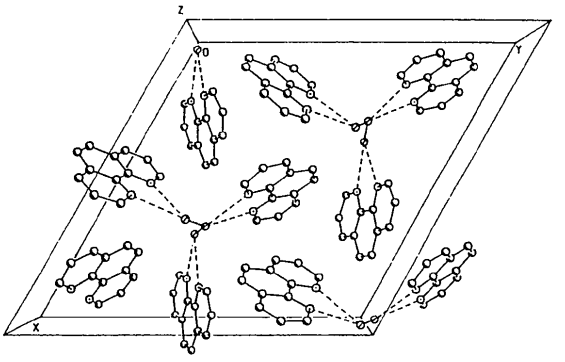


Fig. 2. The unit cell of the title compound.

Experimental

The title compound was recrystallized from water.

Crystal data

$C_{12}H_8N_2.H_2O$
M_r = 198.2
Trigonal
$P3_1$
a = 17.833 (3) Å
c = 8.5430 (10) Å
V = 2352.9 (7) Å^3
Z = 9
D_x = 1.259 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 13–15°
μ = 0.077 mm^{-1}
T = 296 K
Prism
0.45 × 0.35 × 0.30 mm
Pale yellow

Data collection

Siemens $R3m/V$ diffractometer
ω scans
Absorption correction: by integration from crystal shape
T_{min} = 0.923, T_{max} = 0.978
3081 measured reflections
3067 independent reflections
1315 observed reflections [$F > 4\sigma(F)$]
R_{int} = 0.008
θ_{max} = 25.0°
$h = -21 \rightarrow 0$
$k = 0 \rightarrow 18$
$l = -10 \rightarrow 10$
10 standard reflections monitored every 100 reflections
intensity decay: 2.7%

Refinement

Refinement on F^2
$R(F)$ = 0.07
$wR(F^2)$ = 0.061
S = 1.77
1315 reflections
405 parameters
H atoms refined as riding model, fixed U_{iso}
$w = 1/[\sigma^2(F) + 0.0002F^2]$
$(\Delta/\sigma)_{max}$ = 0.048
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2*B*)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
N(1)	0.5481 (7)	0.7688 (7)	−0.0869 (18)	0.064 (6)
C(2)	0.5617 (14)	0.7448 (10)	−0.230 (2)	0.086 (12)
C(3)	0.6438 (15)	0.7639 (14)	−0.282 (3)	0.107 (16)
C(4)	0.7132 (13)	0.8080 (12)	−0.189 (3)	0.101 (14)
C(5)	0.7057 (12)	0.8359 (10)	−0.038 (3)	0.075 (11)
C(6)	0.7786 (12)	0.8859 (13)	0.063 (3)	0.097 (12)
C(7)	0.7682 (13)	0.9121 (12)	0.204 (3)	0.109 (12)
C(8)	0.6859 (12)	0.8877 (9)	0.257 (3)	0.081 (10)
C(9)	0.6682 (15)	0.9100 (10)	0.412 (3)	0.100 (12)
C(10)	0.5851 (16)	0.8847 (13)	0.451 (3)	0.104 (14)
C(11)	0.5203 (12)	0.8372 (10)	0.344 (2)	0.095 (12)
N(12)	0.5306 (9)	0.8143 (7)	0.2107	0.072 (8)
C(13)	0.6109 (11)	0.8413 (9)	0.167 (2)	0.058 (9)
C(14)	0.6202 (10)	0.8135 (9)	0.005 (2)	0.062 (9)
N(15)	0.8636 (7)	0.3837 (8)	−0.111 (2)	0.067 (7)
C(16)	0.8561 (11)	0.3518 (9)	−0.254 (2)	0.075 (10)
C(17)	0.9280 (13)	0.3637 (11)	−0.344 (2)	0.083 (12)
C(18)	1.0071 (12)	0.4115 (11)	−0.282 (3)	0.082 (12)
C(19)	1.0167 (10)	0.4435 (9)	−0.131 (2)	0.063 (9)
C(20)	1.1022 (10)	0.4953 (9)	−0.067 (2)	0.072 (9)
C(21)	1.1131 (10)	0.5298 (9)	0.077 (2)	0.082 (10)
C(22)	1.0407 (10)	0.5174 (9)	0.172 (2)	0.065 (9)
C(23)	1.0456 (11)	0.5502 (10)	0.319 (2)	0.086 (11)
C(24)	0.9705 (16)	0.5307 (12)	0.397 (2)	0.094 (13)
C(25)	0.8903 (11)	0.4760 (12)	0.328 (2)	0.078 (11)
N(26)	0.8828 (7)	0.4436 (7)	0.190 (2)	0.062 (7)
C(27)	0.9541 (11)	0.4636 (9)	0.110 (2)	0.055 (9)
C(28)	0.9428 (10)	0.4289 (9)	−0.046 (2)	0.063 (9)
N(29)	0.2151 (9)	0.0801 (9)	−0.052 (2)	0.081 (9)
C(30)	0.2192 (14)	0.0551 (12)	−0.199 (3)	0.105 (13)
C(31)	0.301 (2)	0.0908 (17)	−0.281 (3)	0.117 (19)
C(32)	0.3734 (16)	0.1511 (18)	−0.209 (3)	0.13 (2)
C(33)	0.3750 (13)	0.1809 (14)	−0.063 (3)	0.096 (14)
C(34)	0.4494 (11)	0.2454 (14)	0.019 (3)	0.109 (14)
C(35)	0.4399 (14)	0.2713 (12)	0.163 (3)	0.113 (14)
C(36)	0.3631 (12)	0.2343 (11)	0.247 (3)	0.076 (10)
C(37)	0.3528 (13)	0.2587 (11)	0.392 (3)	0.092 (12)
C(38)	0.2752 (16)	0.2228 (14)	0.461 (2)	0.100 (14)
C(39)	0.2030 (11)	0.1548 (11)	0.381 (3)	0.086 (11)
N(40)	0.2095 (9)	0.1285 (8)	0.241 (2)	0.072 (8)
C(41)	0.2853 (11)	0.1689 (10)	0.169 (2)	0.068 (9)
C(42)	0.2923 (13)	0.1422 (10)	0.013 (2)	0.073 (11)
O(1)	0.3649 (6)	0.6982 (6)	0.007 (2)	0.092 (6)
O(2)	0.0341 (6)	0.0165 (6)	0.102 (2)	0.103 (6)
O(3)	0.6981 (5)	0.3352 (5)	0.086 (2)	0.081 (5)

Table 2. *Selected geometric parameters* (Å, °)

N(1)—C(2)	1.36 (2)	N(1)—C(14)	1.37 (2)
C(2)—C(3)	1.40 (4)	C(3)—C(4)	1.34 (3)
C(4)—C(5)	1.42 (3)	C(5)—C(6)	1.44 (3)
C(5)—C(14)	1.42 (3)	C(6)—C(7)	1.34 (4)
C(7)—C(8)	1.38 (3)	C(8)—C(9)	1.46 (3)
C(8)—C(13)	1.40 (3)	C(9)—C(10)	1.36 (4)
C(10)—C(11)	1.38 (3)	C(11)—N(12)	1.26 (2)
N(12) C(13)	1.32 (2)	C(13) C(14)	1.51 (3)
N(15)—C(16)	1.33 (2)	N(15)—C(28)	1.35 (2)
C(16)—C(17)	1.42 (3)	C(17)—C(18)	1.34 (3)
C(18)—C(19)	1.38 (3)	C(19)—C(20)	1.44 (2)
C(19)—C(28)	1.41 (3)	C(20)—C(21)	1.35 (3)
C(21)—C(22)	1.45 (3)	C(22)—C(23)	1.37 (3)
C(22)—C(27)	1.45 (2)	C(23)—C(24)	1.38 (3)
C(24)—C(25)	1.40 (3)	C(25)—N(26)	1.29 (3)
N(26)—C(27)	1.32 (2)	C(27)—C(28)	1.44 (3)
N(29)—C(30)	1.34 (3)	N(29)—C(42)	1.38 (2)
C(30)—C(31)	1.45 (4)	C(31)—C(32)	1.35 (3)
C(32)—C(33)	1.35 (4)	C(33)—C(34)	1.43 (3)
C(33)—C(42)	1.43 (3)	C(34)—C(35)	1.35 (4)
C(35)—C(36)	1.39 (3)	C(36)—C(37)	1.35 (3)
C(36)—C(41)	1.45 (2)	C(37)—C(38)	1.34 (3)
C(38)—C(39)	1.43 (3)	C(39)—N(40)	1.31 (3)
N(40)—C(41)	1.33 (2)	C(41)—C(42)	1.44 (3)
C(2)—N(1)—C(14)	116.0 (16)	N(1)—C(2)—C(3)	122.7 (17)
C(2)—C(3)—C(4)	120 (2)	C(3)—C(4)—C(5)	122 (2)
C(4)—C(5)—C(6)	123 (2)	C(4)—C(5)—C(14)	114.5 (17)
C(6)—C(5)—C(14)	122 (2)	C(5)—C(6)—C(7)	121 (2)
C(6)—C(7)—C(8)	119.7 (19)	C(7)—C(8)—C(9)	123.4 (19)
C(7)—C(8)—C(13)	124 (2)	C(9)—C(8)—C(13)	112.6 (19)
C(8)—C(9)—C(10)	118.7 (19)	C(9)—C(10)—C(11)	119 (2)
C(10)—C(11)—N(12)	126 (2)	C(11)—N(12)—C(13)	116.4 (14)
C(8)—C(13)—N(12)	127.4 (17)	C(8)—C(13)—C(14)	117.9 (17)

N(12)—C(13)—C(14)	114.6 (13)	N(1)—C(14)—C(5)	125.2 (17)
N(1)—C(14)—C(13)	119.6 (16)	C(5)—C(14)—C(13)	115.2 (15)
C(16)—N(15)—C(28)	119.2 (16)	N(15)—C(16)—C(17)	123.3 (15)
C(16)—C(17)—C(18)	117.5 (18)	C(17)—C(18)—C(19)	120 (2)
C(18)—C(19)—C(20)	119.3 (17)	C(18)—C(19)—C(28)	119.6 (15)
C(20)—C(19)—C(28)	121.0 (17)	C(19)—C(20)—C(21)	120.3 (17)
C(20)—C(21)—C(22)	122.0 (14)	C(21)—C(22)—C(23)	126.0 (15)
C(21)—C(22)—C(27)	118.0 (16)	C(23)—C(22)—C(27)	115.9 (17)
C(22)—C(23)—C(24)	119.3 (16)	C(23)—C(24)—C(25)	120.0 (19)
C(24)—C(25)—N(26)	122.7 (19)	C(25)—N(26)—C(27)	118.5 (13)
C(22)—C(27)—N(26)	123.5 (17)	C(22)—C(27)—C(28)	119.7 (16)
N(26)—C(27)—C(28)	116.8 (14)	N(15)—C(28)—C(19)	119.9 (17)
N(15)—C(28)—C(27)	121.2 (17)	C(19)—C(28)—C(27)	118.9 (15)
C(30)—N(29)—C(42)	116.2 (16)	N(29)—C(30)—C(31)	121.1 (17)
C(30)—C(31)—C(32)	119 (2)	C(31)—C(32)—C(33)	124 (2)
C(32)—C(33)—C(34)	127 (2)	C(32)—C(33)—C(42)	114.4 (18)
C(34)—C(33)—C(42)	119 (2)	C(33)—C(34)—C(35)	119.6 (19)
C(34)—C(35)—C(36)	124.8 (18)	C(35)—C(36)—C(37)	125.5 (17)
C(35)—C(36)—C(41)	118 (2)	C(37)—C(36)—C(41)	116.7 (18)
C(36)—C(37)—C(38)	121.8 (17)	C(37)—C(38)—C(39)	118 (2)
C(38)—C(39)—N(40)	122.6 (17)	C(39)—N(40)—C(41)	118.8 (13)
C(36)—C(41)—N(40)	121.7 (18)	C(36)—C(41)—C(42)	119.0 (17)
N(40)—C(41)—C(42)	119.1 (14)	N(29)—C(42)—C(33)	125.5 (19)
N(29)—C(42)—C(41)	114.8 (17)	C(33)—C(42)—C(41)	119.7 (16)

Table 3. *Average geometric values of the pseudo-equivalent bonds* (Å, °)

N(1)—C(2)	1.315 (11)	N(1)—C(14)	1.345 (9)
C(2)—C(3)	1.412 (13)	C(3)—C(4)	1.352 (13)
C(4)—C(5)	1.388 (13)	C(5)—C(14)	1.426 (11)
C(5)—C(6)	1.421 (11)	C(14)—C(13)	1.462 (16)
C(6)—C(7)	1.35 (2)		
C(2)—N(1)—C(14)	117.5 (6)	N(1)—C(2)—C(3)	123.0 (7)
C(2)—C(3)—C(4)	118.8 (8)	C(3)—C(4)—C(5)	121.1 (8)
C(4)—C(5)—C(14)	115.6 (7)	C(4)—C(5)—C(6)	125.5 (7)
C(14)—C(5)—C(6)	120.3 (8)	N(1)—C(14)—C(5)	123.9 (7)
N(1)—C(14)—C(13)	117.7 (6)	C(5)—C(14)—C(13)	118.4 (7)
C(5)—C(6)—C(7)	121.2 (7)		

The ω scan width was $(0.85 + 0.35\tan\theta)°$ and the scan speed was 5.45–29.30° min^{-1}. The structure was solved by direct methods and difference Fourier synthesis, refined with unit weights by full-matrix least-squares methods with anisotropic displacement parameters for all non-H atoms.

Data collection: Siemens *R3m/V* software. *PLUTO* (Motherwell & Clegg, 1978) and all calculations were performed on a VMX computer with the *SHELXTL-Plus* (VMS) (Sheldrick, 1987) program package.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: TA1000). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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An $As_2Si_3O_6$ Cage Compound, 3,3,7,7,10,10-Hexaphenyl-2,4,6,8,9,11-hexaoxa-1,5-diarsa-3,7,10-trisilabicyclo-[3.3.3]undecane

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Abstract

The title compound, $C_{36}H_{30}As_2O_6Si_3$, is the first arsenosilicate compound containing tricoordinate arsenic(III) to be studied using X-ray crystallography. This is also the first structural determination of an arsenosilicate compound with the siloxy moiety acting as a bridging group. Two trigonal pyramidal AsO_3 units are joined by three tetrahedral —$OSiPh_2O$— bridges to form an 11-atom $As_2Si_3O_6$ cage with mean dimensions As—O 1.760 (15), Si—O 1.637 (5) Å, O—As—O 98.9 (10), O—Si—O 111.1 (4), As—O—Si 139.1 (13)°.

Comment

A small number of compounds containing terminal As—O—$SiMe_3$ units have been reported recently (Baier, Bissinger & Schmidbaur, 1992, 1993; Baier, Paul & Schmidbaur, 1993), but compounds with As—O—$Si(R)_2$—O—As bridging units have not been characterized previously. Additionally, all the reported As—O—$SiMe_3$ compounds contained arsenic in the +V oxidation state with tetra-, penta- or hexacoordination. We report here the structure of 3,3,7,7,10,10-hexa-