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cyclo-Tetra-μ-oxido-tetrakis[3-nitro-4-hydroxyphenylarsenic(III)]

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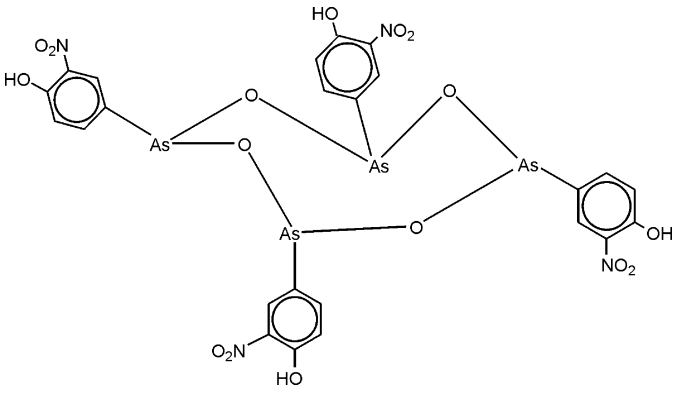
Key indicators: single-crystal X-ray study; T = 93 K; mean σ(C–C) = 0.014 Å; R factor = 0.068; wR factor = 0.131; data-to-parameter ratio = 12.7.

The title compound, $[As_4O_4(C_6H_4NO_3)_4]$, has an eight-membered As_4O_4 ring with a slightly twisted boat–chair conformation. The aryl groups complete the threefold coordination for each As atom. Each OH group forms a strong intramolecular O—H···O hydrogen bond to the adjacent NO_2 group, with only weak C—H···O, O···As [3.036 (6)–3.184 (6) Å] and O···O [2.921 (10)–2.930 (10) Å] interactions between tetramers.

Related literature

Other examples of cyclic $(RAsO)_n$ are n = 4 for R = Me (DiMaio & Rheingold, 1991), Ph (Muller & Muhle, 1999), mesityl (Arif *et al.*, 1987) or 3-$F_3CC_6H_4$ (Sun *et al.*, 2005), and n = 5 for R = Et (Hausler & Sheldrick, 1997). A related compound, (4-$H_2NC_6H_4$)As$(OH)_2$, was reported by Knoch *et al.* (1995).



Experimental

Crystal data

$[As_4O_4(C_6H_4NO_3)_4]$
M_r = 916.09
Monoclinic, $P2_1/c$
a = 7.1289 (2) Å
b = 31.6743 (9) Å
c = 13.0217 (4) Å
β = 98.286 (1)°
V = 2909.64 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.64 mm^{-1}
T = 93 (2) K
0.16 × 0.10 × 0.05 mm

Data collection

Siemens SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.524, T_{max} = 0.801
16032 measured reflections
5490 independent reflections
3294 reflections with $I > 2\sigma(I)$
R_{int} = 0.114

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.131
S = 1.08
5490 reflections
433 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.03 e Å^{-3}
$\Delta\rho_{min}$ = −0.91 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O11—H11···O12	0.84	1.91	2.614 (9)	140
O21—H21···O22	0.84	1.88	2.579 (11)	141
O31—H31···O33	0.84	1.86	2.572 (11)	142
O41—H41···O43	0.84	1.86	2.569 (10)	142
O11—H11···O1^{i}	0.84	2.65	3.260 (8)	131
C15—H15···O42^{ii}	0.95	2.47	3.191 (12)	132
C25—H25···O43^{iii}	0.95	2.47	3.151 (12)	128
C15—H15···O13^{i}	0.95	2.70	3.539 (12)	148
C12—H12···O11^{iv}	0.95	2.61	3.340 (11)	134
C42—H42···O32^{iv}	0.95	2.48	3.355 (13)	154

Symmetry codes: (i) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (ii) $x-1, y, z-1$; (iii) $-x+1, -y+1, -z+1$; (iv) $x, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *SMART* (Bruker 2001); cell refinement: *SAINT* (Bruker 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

We thank Dr Tania Groutso, University of Auckland, for the collection of the X-ray intensity data. We also thank the Marsden Fund, administered by the Royal Society of New Zealand, for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2082).

References

Arif, A. M., Cowley, A. H. & Pakulski, M. (1987). *Chem. Commun.* pp. 165–166.
Bruker (2001). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
DiMaio, A. J. & Rheingold, A. L. (1991). *Organometallics*, **10**, 3764–3746.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Hausler, T. & Sheldrick, W. S. (1997). *Chem. Ber.* **130**, 371–376.
Knoch, F., Schmachtel, T. & Ullrich, V. (1995). *Z. Kristallogr.* **210**, 642.
Muller, I. M. & Muhle, J. (1999). *Z. Anorg. Allg. Chem.* **625**, 336–340.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sun, H., Patrick, B. O. & Cullen, W. R. (2005). *Appl. Organomet. Chem.* **19**, 129–131.

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supplementary materials

Acta Cryst. (2007). E**63**, m2573 [**doi:10.1107/S1600536807044728**]

cyclo-Tetra-μ-oxido-tetrakis[3-nitro-4-hydroxyphenylarsenic(III)]

N. C. Lloyd and B. K. Nicholson

Comment

Aryl arsenoxides of empirical formula RAsO exist as either hydrates $RAs(OH)_2$ as for the 4-$H_2NC_6H_4$ example (Knock *et al.*, 1995), or as cyclic $(RAsO)_n$ where n = 4 for *R* = Me (DiMaio & Rheingold, 1991), Ph (Muller & Muhle, 1999), mesityl (Arif *et al.*, 1987), or 3-$F_3CC_6H_4$ (Sun *et al.*, 2005) and n = 5 for *R* = Et (Hausler & Sheldrick, 1997). The title compound, $[3\text{-}O_2N\text{-}4\text{-}HOC_6H_3AsO]_4$, also forms an eight-membered As_4O_4 ring which has a slightly twisted boat-chair conformation. The aryl groups complete the 3-coordination for each As atom. Average parameters are: As—O = 1.801 (6) Å, O—As—O = 98.8 (3)° and As—O—As = 121.3 (4)°. The OH group is internally H-bonded to the adjacent NO_2 group, so the intermolecular interactions between tetramers are weak C—H···O and O···As ones. There are also some short intermolecular O···O interactions involving the NO_2 groups.

Experimental

The title compound was prepared by hydrolysis of the dichloride $3\text{-}O_2N\text{-}4\text{-}HOC_6H_3AsCl_2$, which in turn was prepared by reduction of $3\text{-}O_2N\text{-}4\text{-}HOC_6H_3AsO_3H_2$ with SO_2 in conc HCl. Crystals suitable for X-ray analysis were obtained from an aqueous solution.

Refinement

All H-atoms were positioned geometrically and refined using a riding model with C—H = 0.95 Å, $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic and O—H = 0.84 Å, $U_{iso}(H) = 1.5U_{eq}(O)$ for the OH groups. As only very small needle crystals were available, the data set was weak and so R_{int} and the final agreement factors are higher than usual. The highest residual electron density was 0.87Å from atom As1.

Figures

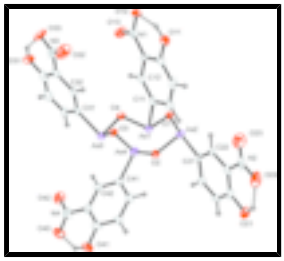

Fig. 1. The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. Dotted lines denote hydrogen bonds.

cyclo-Tetra-μ-oxido-tetra[3-nitro-4-hydroxyphenylarsenic(III)]

Crystal data

$[As_4O_4(C_6H_4NO_3)_4]$

$M_r = 916.09$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 7.1289\ (2)$ Å

$b = 31.6743\ (9)$ Å

$c = 13.0217\ (4)$ Å

$\beta = 98.286\ (1)^o$

$V = 2909.64\ (15)$ Å^3

$Z = 4$

$F_{000} = 1792$

$D_x = 2.091$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 4805 reflections

$\theta = 2–25^o$

$\mu = 4.64$ mm^{-1}

$T = 93\ (2)$ K

Needle, yellow

0.16 × 0.10 × 0.05 mm

Data collection

Siemens SMART CCD diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 93(2)$ K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{min} = 0.524$, $T_{max} = 0.801$

16032 measured reflections

5490 independent reflections

3294 reflections with $I > 2\sigma(I)$

$R_{int} = 0.114$

$\theta_{max} = 25.7^o$

$\theta_{min} = 1.7^o$

$h = -8\rightarrow8$

$k = -38\rightarrow38$

$l = -11\rightarrow15$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.068$

$wR(F^2) = 0.131$

$S = 1.08$

5490 reflections

433 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0328P)^2 + 6.0469P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 1.03$ e Å^{-3}

$\Delta\rho_{min} = -0.91$ e Å^{-3}

Extinction correction: none

Special details

Refinement. As only small needle crystals were available, the data set was weak and so the final agreement factors are higher than usual.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
As1	0.02562 (13)	0.64613 (3)	0.12402 (7)	0.0189 (2)
As2	−0.24608 (13)	0.60197 (3)	0.25665 (8)	0.0204 (2)
As3	0.29993 (13)	0.67460 (3)	0.31865 (8)	0.0212 (3)
As4	0.07624 (14)	0.61371 (3)	0.45063 (8)	0.0236 (3)
N1	−0.1904 (10)	0.7965 (2)	−0.0349 (7)	0.0242 (19)
N2	−0.3338 (12)	0.5235 (3)	−0.1123 (7)	0.034 (2)
N3	0.2923 (12)	0.8406 (3)	0.2570 (7)	0.039 (2)
N4	0.7762 (13)	0.5809 (3)	0.6360 (7)	0.034 (2)
O1	−0.2016 (8)	0.64360 (17)	0.1704 (5)	0.0197 (14)
O2	−0.0071 (8)	0.59444 (18)	0.3221 (5)	0.0216 (15)
O3	0.1553 (8)	0.66539 (18)	0.4188 (5)	0.0221 (15)
O4	0.1206 (8)	0.68753 (17)	0.2095 (5)	0.0197 (15)
O11	−0.2457 (8)	0.76047 (19)	−0.2455 (5)	0.0241 (15)
H11	−0.2551	0.7859	−0.2288	0.036*
O12	−0.2549 (8)	0.82023 (19)	−0.1079 (5)	0.0262 (16)
O13	−0.1622 (10)	0.8085 (2)	0.0547 (5)	0.0325 (18)
O21	−0.2054 (9)	0.4438 (2)	−0.0092 (6)	0.0377 (19)
H21	−0.2416	0.4477	−0.0728	0.057*
O22	−0.3260 (10)	0.4912 (2)	−0.1659 (6)	0.043 (2)
O23	−0.3879 (12)	0.5576 (2)	−0.1491 (6)	0.049 (2)
O31	0.4044 (10)	0.8558 (2)	0.4784 (6)	0.048 (2)
H31	0.3830	0.8736	0.4303	0.072*
O32	0.2570 (13)	0.8322 (2)	0.1640 (6)	0.057 (2)
O33	0.2920 (11)	0.8781 (2)	0.2897 (6)	0.052 (2)
O41	0.8297 (9)	0.5182 (2)	0.4778 (5)	0.0338 (18)
H41	0.9070	0.5263	0.5288	0.051*
O42	0.7476 (10)	0.6104 (2)	0.6930 (5)	0.040 (2)
O43	0.9213 (10)	0.5587 (2)	0.6485 (6)	0.044 (2)
C11	−0.0641 (12)	0.6837 (3)	0.0105 (7)	0.019 (2)
C12	−0.0898 (12)	0.7266 (3)	0.0266 (7)	0.020 (2)
H12	−0.0634	0.7383	0.0944	0.024*
C13	−0.1554 (12)	0.7524 (3)	−0.0587 (7)	0.017 (2)
C14	−0.1879 (12)	0.7370 (3)	−0.1586 (7)	0.021 (2)
C15	−0.1662 (12)	0.6946 (3)	−0.1739 (7)	0.021 (2)
H15	−0.1932	0.6832	−0.2420	0.026*
C16	−0.1051 (12)	0.6679 (3)	−0.0911 (7)	0.022 (2)
H16	−0.0908	0.6385	−0.1032	0.026*
C21	−0.2211 (12)	0.5540 (3)	0.1643 (7)	0.019 (2)
C22	−0.2801 (12)	0.5560 (3)	0.0605 (7)	0.022 (2)
H22	−0.3236	0.5820	0.0293	0.027*

C23	−0.2766 (12)	0.5194 (3)	−0.0008 (7)	0.021 (2)
C24	−0.2126 (13)	0.4805 (3)	0.0424 (8)	0.028 (2)
C25	−0.1569 (13)	0.4790 (3)	0.1489 (8)	0.027 (2)
H25	−0.1170	0.4529	0.1811	0.032*
C26	−0.1591 (13)	0.5148 (3)	0.2082 (8)	0.025 (2)
H26	−0.1177	0.5132	0.2808	0.030*
C31	0.3370 (12)	0.7330 (3)	0.3635 (8)	0.026 (2)
C32	0.3039 (12)	0.7657 (3)	0.2948 (9)	0.028 (3)
H32	0.2640	0.7603	0.2232	0.034*
C33	0.3301 (13)	0.8084 (3)	0.3322 (9)	0.034 (3)
C34	0.3886 (13)	0.8159 (4)	0.4376 (8)	0.033 (3)
C35	0.4198 (13)	0.7831 (4)	0.5059 (9)	0.038 (3)
H35	0.4586	0.7880	0.5778	0.045*
C36	0.3934 (12)	0.7424 (3)	0.4678 (8)	0.027 (2)
H36	0.4151	0.7196	0.5154	0.032*
C41	0.3192 (13)	0.5837 (3)	0.4575 (7)	0.022 (2)
C42	0.4625 (13)	0.5922 (3)	0.5381 (7)	0.024 (2)
H42	0.4426	0.6128	0.5886	0.029*
C43	0.6336 (14)	0.5715 (3)	0.5465 (7)	0.024 (2)
C44	0.6650 (14)	0.5406 (3)	0.4736 (7)	0.025 (2)
C45	0.5206 (12)	0.5315 (3)	0.3945 (7)	0.023 (2)
H45	0.5392	0.5102	0.3456	0.027*
C46	0.3488 (13)	0.5527 (3)	0.3846 (8)	0.023 (2)
H46	0.2518	0.5463	0.3288	0.028*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
As1	0.0183 (5)	0.0151 (5)	0.0225 (6)	0.0018 (4)	0.0000 (4)	−0.0001 (4)
As2	0.0208 (5)	0.0148 (5)	0.0253 (6)	−0.0013 (4)	0.0022 (4)	−0.0004 (4)
As3	0.0167 (5)	0.0169 (5)	0.0281 (6)	0.0021 (4)	−0.0030 (4)	−0.0031 (5)
As4	0.0259 (5)	0.0211 (6)	0.0227 (6)	0.0022 (4)	0.0002 (4)	−0.0007 (4)
N1	0.016 (4)	0.027 (5)	0.029 (5)	−0.003 (4)	0.001 (4)	−0.001 (4)
N2	0.024 (5)	0.047 (6)	0.031 (6)	−0.019 (4)	−0.003 (4)	−0.001 (5)
N3	0.040 (6)	0.038 (6)	0.034 (6)	−0.010 (5)	−0.012 (5)	0.008 (5)
N4	0.037 (5)	0.032 (5)	0.029 (5)	−0.002 (4)	−0.007 (4)	0.009 (5)
O1	0.017 (3)	0.012 (3)	0.031 (4)	−0.002 (3)	0.005 (3)	0.002 (3)
O2	0.024 (3)	0.018 (3)	0.020 (4)	0.001 (3)	−0.006 (3)	−0.005 (3)
O3	0.026 (4)	0.016 (3)	0.023 (4)	0.004 (3)	0.001 (3)	−0.004 (3)
O4	0.023 (3)	0.014 (3)	0.020 (4)	0.003 (3)	−0.003 (3)	−0.002 (3)
O11	0.021 (3)	0.026 (4)	0.025 (4)	−0.002 (3)	0.000 (3)	0.005 (3)
O12	0.021 (3)	0.019 (4)	0.036 (4)	0.004 (3)	−0.003 (3)	0.013 (3)
O13	0.049 (5)	0.023 (4)	0.026 (4)	0.002 (3)	0.004 (4)	−0.002 (3)
O21	0.039 (4)	0.034 (4)	0.038 (5)	−0.002 (3)	−0.001 (4)	−0.019 (4)
O22	0.045 (5)	0.056 (5)	0.029 (5)	−0.015 (4)	0.008 (4)	−0.023 (4)
O23	0.066 (6)	0.046 (5)	0.031 (5)	−0.009 (4)	−0.010 (4)	0.008 (4)
O31	0.039 (4)	0.019 (4)	0.081 (6)	0.007 (4)	−0.005 (4)	−0.017 (4)
O32	0.087 (7)	0.041 (5)	0.041 (6)	−0.009 (5)	0.001 (5)	0.004 (4)

O33	0.067 (6)	0.013 (4)	0.073 (6)	−0.009 (4)	−0.005 (5)	−0.001 (4)
O41	0.026 (4)	0.035 (4)	0.037 (5)	0.009 (3)	−0.005 (3)	0.002 (4)
O42	0.052 (5)	0.041 (5)	0.025 (4)	−0.004 (4)	−0.002 (4)	−0.010 (4)
O43	0.035 (4)	0.049 (5)	0.043 (5)	0.011 (4)	−0.016 (4)	0.010 (4)
C11	0.017 (5)	0.025 (6)	0.016 (5)	−0.003 (4)	0.002 (4)	0.005 (4)
C12	0.016 (5)	0.028 (6)	0.015 (5)	−0.001 (4)	0.004 (4)	0.004 (4)
C13	0.019 (5)	0.010 (5)	0.022 (6)	0.000 (4)	0.007 (4)	−0.001 (4)
C14	0.012 (5)	0.034 (6)	0.016 (6)	−0.004 (4)	0.001 (4)	0.008 (4)
C15	0.017 (5)	0.029 (6)	0.019 (5)	−0.010 (4)	0.005 (4)	−0.007 (5)
C16	0.014 (5)	0.024 (6)	0.028 (6)	−0.001 (4)	0.004 (4)	−0.001 (5)
C21	0.018 (5)	0.018 (5)	0.019 (6)	−0.008 (4)	−0.001 (4)	−0.008 (4)
C22	0.008 (4)	0.024 (5)	0.033 (6)	−0.006 (4)	0.001 (4)	−0.005 (5)
C23	0.020 (5)	0.032 (6)	0.011 (5)	−0.005 (4)	−0.001 (4)	0.001 (4)
C24	0.016 (5)	0.031 (6)	0.035 (7)	−0.006 (4)	0.003 (5)	−0.012 (5)
C25	0.028 (6)	0.020 (5)	0.030 (6)	0.009 (4)	−0.005 (5)	−0.001 (5)
C26	0.025 (5)	0.024 (6)	0.023 (6)	0.001 (4)	−0.003 (4)	−0.007 (5)
C31	0.009 (5)	0.019 (5)	0.049 (7)	−0.003 (4)	0.005 (5)	−0.008 (5)
C32	0.010 (5)	0.026 (6)	0.048 (7)	−0.008 (4)	0.003 (5)	−0.008 (5)
C33	0.012 (5)	0.030 (6)	0.060 (8)	0.000 (4)	0.005 (5)	0.013 (6)
C34	0.014 (5)	0.050 (8)	0.034 (7)	−0.003 (5)	0.004 (5)	0.000 (6)
C35	0.015 (5)	0.055 (8)	0.046 (8)	−0.003 (5)	0.013 (5)	−0.003 (6)
C36	0.013 (5)	0.027 (6)	0.041 (7)	−0.001 (4)	0.003 (5)	−0.010 (5)
C41	0.030 (6)	0.016 (5)	0.020 (5)	0.004 (4)	−0.003 (4)	0.006 (4)
C42	0.029 (6)	0.019 (5)	0.025 (6)	0.006 (4)	0.004 (5)	0.006 (4)
C43	0.030 (6)	0.022 (6)	0.017 (5)	−0.008 (4)	−0.003 (5)	0.003 (4)
C44	0.037 (6)	0.021 (6)	0.018 (6)	0.005 (5)	0.002 (5)	0.009 (4)
C45	0.023 (5)	0.022 (5)	0.021 (6)	−0.002 (4)	−0.002 (4)	−0.001 (4)
C46	0.023 (5)	0.021 (5)	0.025 (6)	−0.004 (4)	−0.001 (4)	0.006 (5)

Geometric parameters (Å, °)

As1—O4	1.789 (6)	C12—C13	1.403 (12)
As1—O1	1.811 (6)	C12—H12	0.9500
As1—C11	1.934 (9)	C13—C14	1.377 (12)
As2—O1	1.790 (6)	C14—C15	1.371 (13)
As2—O2	1.807 (6)	C15—C16	1.391 (12)
As2—C21	1.962 (9)	C15—H15	0.9500
As3—O3	1.799 (6)	C16—H16	0.9500
As3—O4	1.816 (6)	C21—C22	1.357 (12)
As3—C31	1.948 (9)	C21—C26	1.411 (12)
As4—O3	1.799 (6)	C22—C23	1.411 (12)
As4—O2	1.800 (6)	C22—H22	0.9500
As4—C41	1.966 (9)	C23—C24	1.402 (13)
N1—O13	1.216 (9)	C24—C25	1.387 (13)
N1—O12	1.247 (9)	C25—C26	1.376 (12)
N1—C13	1.461 (11)	C25—H25	0.9500
N2—O23	1.219 (10)	C26—H26	0.9500
N2—O22	1.243 (10)	C31—C32	1.365 (13)
N2—C23	1.457 (12)	C31—C36	1.391 (13)

N3—O32	1.229 (11)	C32—C33	1.441 (14)
N3—O33	1.262 (10)	C32—H32	0.9500
N3—C33	1.412 (13)	C33—C34	1.396 (14)
N4—O42	1.227 (10)	C34—C35	1.367 (14)
N4—O43	1.242 (10)	C35—C36	1.383 (14)
N4—C43	1.463 (12)	C35—H35	0.9500
O11—C14	1.367 (10)	C36—H36	0.9500
O11—H11	0.8400	C41—C42	1.382 (12)
O21—C24	1.349 (11)	C41—C46	1.402 (13)
O21—H21	0.8400	C42—C43	1.376 (13)
O31—C34	1.370 (12)	C42—H42	0.9500
O31—H31	0.8400	C43—C44	1.402 (13)
O41—C44	1.367 (11)	C44—C45	1.378 (12)
O41—H41	0.8400	C45—C46	1.387 (12)
C11—C12	1.391 (12)	C45—H45	0.9500
C11—C16	1.405 (12)	C46—H46	0.9500
O4—As1—O1	95.8 (3)	C26—C21—As2	118.9 (7)
O4—As1—C11	94.5 (3)	C21—C22—C23	119.7 (9)
O1—As1—C11	94.0 (3)	C21—C22—H22	120.1
O1—As2—O2	98.8 (3)	C23—C22—H22	120.1
O1—As2—C21	98.4 (3)	C24—C23—C22	121.8 (9)
O2—As2—C21	91.4 (3)	C24—C23—N2	120.5 (9)
O3—As3—O4	101.0 (3)	C22—C23—N2	117.7 (9)
O3—As3—C31	90.2 (4)	O21—C24—C25	116.2 (9)
O4—As3—C31	94.0 (3)	O21—C24—C23	126.2 (9)
O3—As4—O2	99.6 (3)	C25—C24—C23	117.5 (9)
O3—As4—C41	98.4 (3)	C26—C25—C24	120.5 (9)
O2—As4—C41	92.8 (3)	C26—C25—H25	119.7
O13—N1—O12	122.3 (8)	C24—C25—H25	119.7
O13—N1—C13	119.7 (8)	C25—C26—C21	121.8 (9)
O12—N1—C13	118.0 (8)	C25—C26—H26	119.1
O23—N2—O22	123.0 (9)	C21—C26—H26	119.1
O23—N2—C23	119.9 (9)	C32—C31—C36	118.5 (9)
O22—N2—C23	117.1 (9)	C32—C31—As3	121.3 (8)
O32—N3—O33	121.7 (9)	C36—C31—As3	120.2 (8)
O32—N3—C33	121.3 (9)	C31—C32—C33	119.2 (10)
O33—N3—C33	117.0 (9)	C31—C32—H32	120.4
O42—N4—O43	124.0 (9)	C33—C32—H32	120.4
O42—N4—C43	118.7 (8)	C34—C33—N3	124.0 (10)
O43—N4—C43	117.2 (9)	C34—C33—C32	119.9 (10)
As2—O1—As1	119.3 (3)	N3—C33—C32	116.1 (10)
As4—O2—As2	123.8 (3)	C35—C34—O31	117.0 (10)
As3—O3—As4	123.1 (3)	C35—C34—C33	120.5 (10)
As1—O4—As3	118.8 (3)	O31—C34—C33	122.4 (10)
C14—O11—H11	109.5	C34—C35—C36	118.3 (11)
C24—O21—H21	109.5	C34—C35—H35	120.8
C34—O31—H31	109.5	C36—C35—H35	120.8
C44—O41—H41	109.5	C35—C36—C31	123.6 (10)
C12—C11—C16	118.5 (8)	C35—C36—H36	118.2

C12—C11—As1	121.5 (7)	C31—C36—H36	118.2
C16—C11—As1	120.0 (7)	C42—C41—C46	119.0 (9)
C11—C12—C13	118.9 (8)	C42—C41—As4	119.4 (7)
C11—C12—H12	120.5	C46—C41—As4	121.6 (7)
C13—C12—H12	120.5	C43—C42—C41	121.1 (9)
C14—C13—C12	122.3 (8)	C43—C42—H42	119.5
C14—C13—N1	121.9 (8)	C41—C42—H42	119.5
C12—C13—N1	115.8 (8)	C42—C43—C44	120.4 (9)
O11—C14—C15	116.1 (8)	C42—C43—N4	118.1 (9)
O11—C14—C13	125.4 (9)	C44—C43—N4	121.5 (9)
C15—C14—C13	118.5 (9)	O41—C44—C45	117.7 (9)
C14—C15—C16	120.8 (9)	O41—C44—C43	123.7 (9)
C14—C15—H15	119.6	C45—C44—C43	118.5 (9)
C16—C15—H15	119.6	C44—C45—C46	121.5 (9)
C15—C16—C11	120.8 (9)	C44—C45—H45	119.3
C15—C16—H16	119.6	C46—C45—H45	119.3
C11—C16—H16	119.6	C45—C46—C41	119.6 (9)
C22—C21—C26	118.7 (8)	C45—C46—H46	120.2
C22—C21—As2	122.1 (7)	C41—C46—H46	120.2

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O11—H11···O12	0.84	1.91	2.614 (9)	140
O21—H21···O22	0.84	1.88	2.579 (11)	141
O31—H31···O33	0.84	1.86	2.572 (11)	142
O41—H41···O43	0.84	1.86	2.569 (10)	142
O11—H11···O1^{i}	0.84	2.65	3.260 (8)	131
C15—H15···O42^{ii}	0.95	2.47	3.191 (12)	132
C25—H25···O43^{iii}	0.95	2.47	3.151 (12)	128
C15—H15···O13^{i}	0.95	2.70	3.539 (12)	148
C12—H12···O11^{iv}	0.95	2.61	3.340 (11)	134
C42—H42···O32^{iv}	0.95	2.48	3.355 (13)	154

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

Fig. 1