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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ (C–C) = 0.007 Å
R factor = 0.038
wR factor = 0.088
Data-to-parameter ratio = 12.1

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

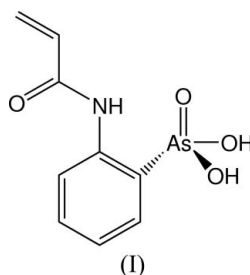
(2-Acryloylaminophenyl)arsonic acid

The geometrical parameters for the title compound, C₉H₁₀AsNO₄ or [As(C₉H₈NO)O(OH)₂], are normal. The dihedral angle between the benzene ring and the acrylamide group is 12.2 (2)°. A network of hydrogen bonds leads to a chain structure.

Received 24 October 2005
Accepted 23 November 2005
Online 30 November 2005

Comment

N-Substituted methacryloyl derivatives of arsanilic acid are widely used as monomers for the synthesis of linear polymers (Percino *et al.*, 2000) with many subsequent applications in analytical chemistry (Hirsch *et al.*, 1970; Cheng *et al.*, 1982). Recently, the synthesis of low molecular weight polymers using *o*- and *p*-methacryloylaminophenylarsonic acid has been reported (Zayas *et al.*, 2000). We now report the crystal structure of the title compound, (I).



The geometrical parameters for (I) are normal and consistent with the equivalent data for similar compounds (Bel'skii,

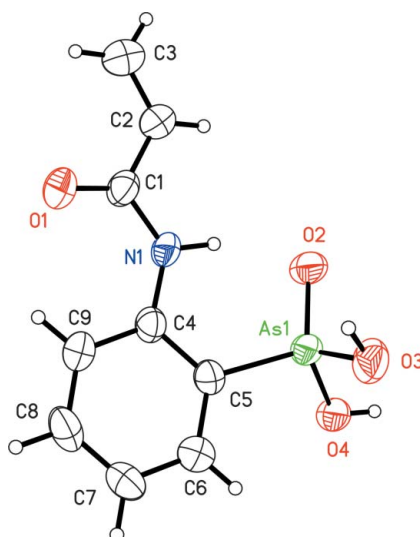


Figure 1
The structure of (I), with displacement ellipsoids drawn at the 50% probability level for non-H atoms.

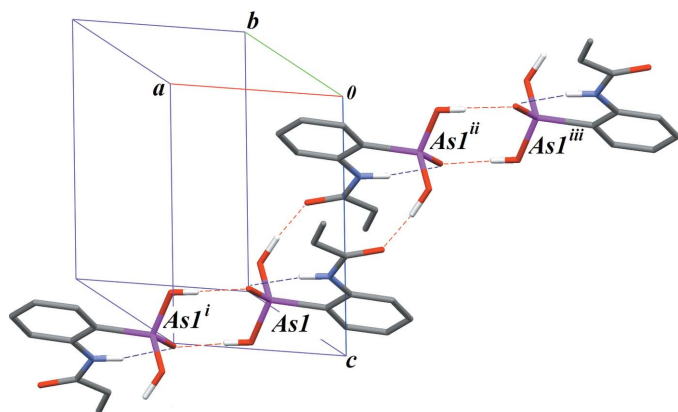


Figure 2
Part of the crystal structure of (I), showing the hydrogen-bonding scheme (dashed lines). Symmetry codes: (i) $1 - x, 1 - y, 2 - z$; (ii) $-x, -y, 1 - z$; (iii) $-1 + x, -1 + y, -1 + z$.

1982; Chatterjee, 1977). The As atom in (I) is tetrahedrally coordinated by three O atoms and one C atom of the substituted benzene ring, with the As—OH bonds being longer than the As=O bond (Table 1). The acyloyl substituent is stabilized in an *s-cis* conformation. The vinyl group is not sterically hindered, and the dihedral angle between the C4-benzene ring and the (C1/C2/C3/N1/O1) acrylamide group is $12.2(2)^\circ$. (I) is thus a good candidate for the synthesis of polymers with high molecular weights, as this conformation allows the formation of an intramolecular hydrogen bond between the NH group of the amide functionality and the oxo O atom (Table 2).

The crystal structure of (I) is built up from classical centrosymmetric double hydrogen bonds involving the oxo and hydroxo functionalities (O2 and O4, respectively) of the arsonic acid group. These dimers are in turn connected through a hydrogen bond involving the hydroxo group O3 as donor and the carbonyl O atom of the amide functionality, O1, as acceptor. The resulting network in the crystal structure is a one-dimensional chain running along [111] (Fig. 2).

Experimental

o-Arsanic acid (1 g, 4.61 mmol) was dissolved in 10 ml of ethanol in a 100 ml flask with mechanical stirring. The reaction flask was then cooled to 278 K in an ice bath, followed by the dropwise addition of freshly distilled acryloyl chloride (0.416 g, 4.60 mmol). The temperature of the exothermic reaction was maintained at 278 K for 8 h with stirring. The resulting mixture (colourless liquid) was precipitated with water, giving a white solid (98% yield). Finally the compound was recrystallized from ethanol/water (4:1) at 278 K. Single crystals of (I) formed almost immediately after cooling the concentrated solution at 278 K, resulting in rather small crystals for the diffraction experiment. Analysis found: C 39.89, H 3.73, N 4.22%; calculated for $C_9H_{10}AsNO_4$: C 39.87, H 3.71, N 5.16%. 1H NMR (400 MHz, d_6 -dimethylsulfoxide): δ 5.80 (*t*, $J = 9.8$ Hz, 1H, CH), 6.25 (*m*, $J = 7.32, 9.8$ Hz, 1H, CH_2), 6.31 (*m*, $J = 8.32, 8.8$ Hz, 1H, CH_2), 7.27 (*t*, $J = 7.32, 7.36$ Hz, 1H, Ph), 7.62 (*t*, $J = 7.32, 8.8$ Hz, 1H, Ph), 7.70 (*d*, $J = 7.34, 6.36$ Hz, 1H, Ph), 8.57 (*t*, $J = 8.8$ Hz, 1H, Ph), 11.96 (*s*, 1H, NH).

Crystal data

[As(C₉H₈NO)O(OH)₂]
 $M_r = 271.10$
Triclinic, $P\bar{1}$
 $a = 8.2088(4) \text{ \AA}$
 $b = 8.3132(4) \text{ \AA}$
 $c = 8.5259(5) \text{ \AA}$
 $\alpha = 99.432(1)^\circ$
 $\beta = 97.766(1)^\circ$
 $\gamma = 111.390(1)^\circ$
 $V = 522.25(5) \text{ \AA}^3$

$Z = 2$
 $D_x = 1.724 \text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 1641 reflections
 $\theta = 2.5\text{--}24.0^\circ$
 $\mu = 3.25 \text{ mm}^{-1}$
 $T = 293(2) \text{ K}$
Prism, colorless
 $0.14 \times 0.11 \times 0.07 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.579, T_{\max} = 0.800$
2862 measured reflections

1641 independent reflections
1322 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$
 $\theta_{\text{max}} = 24.0^\circ$
 $h = -7 \rightarrow 9$
 $k = -9 \rightarrow 9$
 $l = -9 \rightarrow 9$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.088$
 $S = 0.94$
1641 reflections
136 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0492P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.55 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.54 \text{ e \AA}^{-3}$

Table 1

Selected bond lengths (\AA).

As1—O2	1.643 (3)	As1—O3	1.720 (3)
As1—O4	1.687 (3)	As1—C5	1.892 (4)

Table 2

Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 \cdots O2	0.93	1.93	2.814 (4)	160
O3—H3 \cdots O1 ⁱ	1.03	1.65	2.669 (5)	172
O4—H4 \cdots O2 ⁱⁱ	0.84	1.77	2.561 (4)	157

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

Data collection was limited to $\theta_{\text{max}} = 24^\circ$ due to the small size of available single crystals. H atoms bonded to N and O atoms were located in difference maps and refined as riding in their as-found relative positions with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{carrier})$. Other H atoms were placed in idealized positions ($C-H = 0.93 \text{ \AA}$) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXTL-Plus (Bruker, 1999); program(s) used to refine structure: SHELXTL-Plus; molecular graphics: SHELXTL-Plus; software used to prepare material for publication: SHELXTL-Plus.

We thank Dr Sylvain Bernès (Instituto de Ciencias, BUAP) for his valuable teaching and discussions. J. García-Serrano acknowledges PROMEP, Mexico, for a graduate fellowship.

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supporting information

Acta Cryst. (2005). E61, m2752–m2754 [https://doi.org/10.1107/S1600536805038857]

(2-Acryloylaminophenyl)arsonic acid

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(I)

*Crystal data*C₉H₁₀AsNO₄ $M_r = 271.10$ Triclinic, $P\bar{1}$

Hall symbol: -P 1

 $a = 8.2088$ (4) Å $b = 8.3132$ (4) Å $c = 8.5259$ (5) Å $\alpha = 99.432$ (1)° $\beta = 97.766$ (1)° $\gamma = 111.390$ (1)° $V = 522.25$ (5) Å³ $Z = 2$ $F(000) = 272$ $D_x = 1.724$ Mg m⁻³

Melting point: > 573 K K

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

Cell parameters from 1641 reflections

 $\theta = 2.5$ – 24.0 ° $\mu = 3.25$ mm⁻¹ $T = 293$ K

Prism, colorless

 $0.14 \times 0.11 \times 0.07$ mm*Data collection*Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 φ and ω scansAbsorption correction: multi-scan
(SADABS; Sheldrick, 1996) $T_{\min} = 0.579$, $T_{\max} = 0.800$

2862 measured reflections

1641 independent reflections

1322 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.025$ $\theta_{\max} = 24.0$ °, $\theta_{\min} = 2.5$ ° $h = -7 \rightarrow 9$ $k = -9 \rightarrow 9$ $l = -9 \rightarrow 9$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.038$ $wR(F^2) = 0.088$ $S = 0.94$

1641 reflections

136 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
map

Hydrogen site location: difmap and geom

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0492P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.55$ e Å⁻³ $\Delta\rho_{\min} = -0.54$ e Å⁻³*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
As1	0.23569 (6)	0.36189 (6)	0.88492 (6)	0.0429 (2)

O1	-0.0461 (5)	-0.2986 (4)	0.5143 (4)	0.0600 (9)
O2	0.3923 (4)	0.2978 (4)	0.8343 (4)	0.0566 (9)
O3	0.2054 (5)	0.5123 (4)	0.7801 (4)	0.0587 (9)
H3	0.1471	0.4397	0.6629	0.088*
O4	0.2825 (4)	0.4761 (4)	1.0795 (4)	0.0481 (8)
H4	0.3751	0.5699	1.1097	0.072*
N1	0.1084 (5)	-0.0319 (4)	0.6902 (4)	0.0425 (9)
H1	0.2159	0.0665	0.7211	0.064*
C1	0.0941 (6)	-0.1725 (6)	0.5760 (5)	0.0421 (11)
C2	0.2690 (7)	-0.1596 (6)	0.5366 (6)	0.0517 (12)
H2B	0.3711	-0.0581	0.5865	0.062*
C3	0.2838 (8)	-0.2876 (7)	0.4332 (6)	0.0636 (15)
H3A	0.1830	-0.3899	0.3824	0.076*
H3B	0.3950	-0.2755	0.4111	0.076*
C4	-0.0273 (6)	-0.0015 (5)	0.7599 (5)	0.0384 (10)
C5	0.0138 (6)	0.1684 (5)	0.8569 (5)	0.0399 (10)
C6	-0.1141 (6)	0.2023 (6)	0.9324 (6)	0.0517 (12)
H6A	-0.0855	0.3146	0.9968	0.062*
C7	-0.2806 (6)	0.0738 (6)	0.9136 (6)	0.0539 (13)
H7A	-0.3654	0.0979	0.9642	0.065*
C8	-0.3219 (6)	-0.0937 (6)	0.8177 (6)	0.0551 (13)
H8A	-0.4355	-0.1820	0.8039	0.066*
C9	-0.1969 (6)	-0.1310 (6)	0.7429 (6)	0.0493 (12)
H9A	-0.2267	-0.2444	0.6803	0.059*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
As1	0.0354 (3)	0.0349 (3)	0.0460 (3)	0.0056 (2)	0.0113 (2)	-0.0056 (2)
O1	0.054 (2)	0.0424 (19)	0.058 (2)	-0.0001 (19)	0.0107 (18)	-0.0097 (18)
O2	0.0332 (17)	0.0496 (19)	0.066 (2)	0.0057 (16)	0.0108 (16)	-0.0174 (17)
O3	0.068 (2)	0.0400 (18)	0.050 (2)	0.0062 (18)	0.0118 (18)	0.0010 (17)
O4	0.0334 (16)	0.0419 (17)	0.049 (2)	0.0032 (15)	0.0047 (14)	-0.0125 (15)
N1	0.034 (2)	0.0306 (19)	0.048 (2)	0.0030 (17)	0.0055 (18)	-0.0064 (18)
C1	0.050 (3)	0.033 (2)	0.035 (3)	0.011 (2)	0.006 (2)	0.002 (2)
C2	0.053 (3)	0.044 (3)	0.050 (3)	0.015 (3)	0.015 (3)	0.000 (2)
C3	0.067 (4)	0.060 (3)	0.064 (4)	0.027 (3)	0.023 (3)	0.003 (3)
C4	0.035 (2)	0.034 (2)	0.039 (3)	0.008 (2)	0.005 (2)	0.005 (2)
C5	0.034 (2)	0.036 (2)	0.043 (3)	0.007 (2)	0.009 (2)	0.005 (2)
C6	0.046 (3)	0.047 (3)	0.060 (3)	0.017 (3)	0.018 (3)	0.007 (3)
C7	0.038 (3)	0.058 (3)	0.066 (4)	0.016 (3)	0.020 (3)	0.016 (3)
C8	0.034 (3)	0.052 (3)	0.072 (4)	0.006 (2)	0.013 (3)	0.020 (3)
C9	0.047 (3)	0.035 (2)	0.054 (3)	0.007 (2)	0.010 (2)	0.001 (2)

Geometric parameters (Å, °)

As1—O2	1.643 (3)	C3—H3A	0.9300
As1—O4	1.687 (3)	C3—H3B	0.9300

As1—O3	1.720 (3)	C4—C9	1.385 (6)
As1—C5	1.892 (4)	C4—C5	1.410 (6)
O1—C1	1.215 (5)	C5—C6	1.391 (6)
O3—H3	1.0267	C6—C7	1.361 (6)
O4—H4	0.8373	C6—H6A	0.9300
N1—C1	1.350 (5)	C7—C8	1.389 (6)
N1—C4	1.412 (5)	C7—H7A	0.9300
N1—H1	0.9258	C8—C9	1.379 (6)
C1—C2	1.488 (6)	C8—H8A	0.9300
C2—C3	1.319 (6)	C9—H9A	0.9300
C2—H2B	0.9300		
O2—As1—O4	114.58 (15)	H3A—C3—H3B	120.0
O2—As1—O3	112.57 (17)	C9—C4—C5	118.1 (4)
O4—As1—O3	101.50 (15)	C9—C4—N1	123.7 (4)
O2—As1—C5	112.33 (17)	C5—C4—N1	118.2 (4)
O4—As1—C5	105.84 (16)	C6—C5—C4	120.1 (4)
O3—As1—C5	109.27 (17)	C6—C5—As1	116.7 (3)
As1—O3—H3	104.6	C4—C5—As1	123.2 (3)
As1—O4—H4	117.5	C7—C6—C5	121.1 (4)
C1—N1—C4	128.6 (4)	C7—C6—H6A	119.4
C1—N1—H1	118.9	C5—C6—H6A	119.4
C4—N1—H1	112.0	C6—C7—C8	119.0 (4)
O1—C1—N1	123.6 (4)	C6—C7—H7A	120.5
O1—C1—C2	123.3 (4)	C8—C7—H7A	120.5
N1—C1—C2	113.0 (4)	C9—C8—C7	120.9 (4)
C3—C2—C1	122.1 (5)	C9—C8—H8A	119.5
C3—C2—H2B	119.0	C7—C8—H8A	119.5
C1—C2—H2B	119.0	C8—C9—C4	120.8 (4)
C2—C3—H3A	120.0	C8—C9—H9A	119.6
C2—C3—H3B	120.0	C4—C9—H9A	119.6
C4—N1—C1—O1	0.3 (7)	O3—As1—C5—C6	-69.7 (4)
C4—N1—C1—C2	-178.2 (4)	O2—As1—C5—C4	-17.8 (4)
O1—C1—C2—C3	-1.4 (8)	O4—As1—C5—C4	-143.6 (4)
N1—C1—C2—C3	177.0 (4)	O3—As1—C5—C4	107.8 (4)
C1—N1—C4—C9	12.8 (7)	C4—C5—C6—C7	-0.5 (7)
C1—N1—C4—C5	-169.4 (4)	As1—C5—C6—C7	177.1 (4)
C9—C4—C5—C6	0.1 (6)	C5—C6—C7—C8	0.3 (8)
N1—C4—C5—C6	-177.8 (4)	C6—C7—C8—C9	0.3 (8)
C9—C4—C5—As1	-177.4 (3)	C7—C8—C9—C4	-0.8 (7)
N1—C4—C5—As1	4.8 (6)	C5—C4—C9—C8	0.5 (7)
O2—As1—C5—C6	164.7 (3)	N1—C4—C9—C8	178.3 (4)
O4—As1—C5—C6	38.9 (4)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1 \cdots O2	0.93	1.93	2.814 (4)	160
O3—H3 \cdots O1 ⁱ	1.03	1.65	2.669 (5)	172
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