

Structure of (+)-L-Arginine Diarsenate

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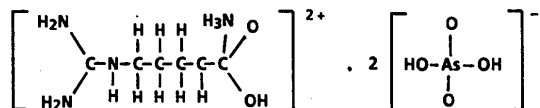
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Abstract. $C_6H_{16}N_4O_{22}^{2+} \cdot 2[AsO_2(OH)_2]^-$, $M_r = 458.09$, orthorhombic, $P2_12_12_1$, $a = 9.787$ (2), $b = 22.024$ (2), $c = 7.134$ (3) Å, $V = 1380.6$ Å³, $Z = 4$, $D_x = 2.20$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 48.87$ cm⁻¹, $F(000) = 920$, $T = 296$ K, $R = 0.021$ for 2563 [$F^2 > \sigma(F^2)$] of 2732 total unique data. L-Arginine²⁺ cations and $[AsO_2(OH)_2]^-$ anions are connected by a network of hydrogen bonds.

Experimental. (+)-L-Arginine diarsenate was investigated for its second-harmonic-generating properties,



and the crystal structure was determined to study the relationship of the structure to the optical properties. Crystals of the title compound were grown by evapora-

tion from a solution of L-arginine and arsenic acid. Colorless crystal, $0.08 \times 0.21 \times 0.37$ mm; modified Picker automatic diffractometer, graphite monochromator; cell dimensions from 27 reflections, $20 < 2\theta < 35^\circ$; 2θ scan width $(1.5 + 0.693 \tan\theta)^\circ$, 2θ scan speed $2-8^\circ \text{ min}^{-1}$; analytical absorption correction, range 1.34 to 2.02; max. $(\sin\theta)/\lambda = 0.60$ Å⁻¹, h 0 to 11, k 0 to 26, l -8 to 8; three standard reflections (002, 400, 060), $\sigma = 1.0, 1.1, 1.3\%$, data corrected for variations; 2963 data, 2732 unique (Friedel pairs not combined), $R_{\text{int}} = 0.014$; As-atom positions from Patterson function, remaining atomic positions from ΔF maps; refinement on F , 2563 $F^2 > \sigma(F^2)$ data, non-H

Table 1. Atomic parameters

$$B_{\text{eq}} = \sum_i \sum_j B_{ij} a_i^* a_j^* a_i a_j / 3.$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B/B_{eq}</i> (Å ²)
As1	0.41438 (3)	0.13418 (1)	0.63720 (4)	1.79
As2	0.62409 (3)	0.03247 (2)	0.19905 (4)	1.94
O1	0.29049 (25)	0.18053 (10)	0.7049 (4)	2.62
O2	0.53642 (27)	0.13154 (14)	0.8102 (4)	2.88
O3	0.36559 (26)	0.06496 (10)	0.5730 (3)	2.20
O4	0.4980 (3)	0.16905 (13)	0.4565 (4)	2.99
O5	0.7629 (3)	-0.01398 (14)	0.2007 (4)	3.99
O6	0.65874 (26)	0.10229 (11)	0.2667 (3)	2.87
O7	0.5058 (3)	-0.00248 (13)	0.3377 (4)	3.21
O8	0.57244 (24)	0.02764 (11)	-0.02289 (29)	2.20
O9	0.12115 (29)	0.14474 (12)	-0.0455 (4)	2.93
O10	0.27861 (28)	0.07575 (13)	0.0336 (4)	3.55
N1	0.1412 (3)	0.03598 (14)	0.3315 (4)	2.11
N2	-0.1695 (3)	0.18172 (14)	0.7664 (4)	2.50
N3	-0.0990 (4)	0.27239 (15)	0.8918 (5)	3.06
N4	-0.2289 (4)	0.20860 (18)	1.0677 (5)	2.99
C1	0.1708 (4)	0.10114 (15)	0.0591 (5)	2.17
C2	0.0777 (4)	0.08641 (14)	0.2218 (5)	1.94
C3	0.0484 (4)	0.14115 (16)	0.3454 (5)	2.15
C4	-0.0466 (4)	0.12924 (17)	0.5107 (5)	2.25
C5	-0.0871 (5)	0.18935 (16)	0.5962 (5)	2.84
C6	-0.1665 (3)	0.22090 (16)	0.9084 (5)	2.29

Table 2. Selected bond distances (Å) and angles (°) and hydrogen-bond distances (Å)

O1-As1	1.6569 (23)	C2-N1	1.494 (4)
O2-As1	1.7183 (26)	C6-N2	1.331 (4)
O3-As1	1.6619 (22)	C5-N2	1.467 (4)
O4-As1	1.7094 (29)	C6-N3	1.318 (5)
O5-As2	1.7010 (26)	C6-N4	1.318 (5)
O6-As2	1.6469 (24)	C2-C1	1.511 (5)
O7-As2	1.7063 (28)	C3-C2	1.521 (5)
O8-As2	1.6654 (22)	C4-C3	1.525 (5)
C1-O9	1.310 (4)	C5-C4	1.510 (5)
C1-O10	1.208 (4)		
O1-As1-O2	108.67 (14)	O9-C1-O10	125.3 (3)
O1-As1-O3	115.80 (13)	O9-C1-C2	111.8 (3)
O1-As1-O4	107.06 (14)	O10-C1-C2	122.9 (3)
O2-As1-O3	111.51 (13)	N1-C2-C1	108.12 (28)
O3-As1-O4	109.99 (13)	C1-C2-C3	112.86 (28)
O2-As1-O4	102.95 (14)	N1-C2-C3	111.36 (28)
O5-As2-O6	113.27 (15)	C2-C3-C4	115.3 (3)
O5-As2-O7	105.48 (17)	C3-C4-C5	108.8 (3)
O5-As2-O8	102.15 (13)	N2-C5-C4	112.22 (29)
O6-As2-O7	113.03 (14)	N2-C6-N3	120.0 (3)
O6-As2-O8	113.63 (12)	N2-C6-N4	120.8 (3)
O7-As2-O8	108.44 (14)	N3-C6-N4	119.1 (3)
C5-N2-C6	122.9 (3)		
N1-H8...O1 ⁱⁱ	2.723 (4)	N4-H15...O1 ⁱⁱ	2.938 (4)
N1-H9...O3 ⁱ	2.889 (4)	O2-H16...O8	2.604 (4)
N1-H10...O3	2.864 (3)	O4-H17...O6	2.534 (4)
N2-H11...O2	3.099 (4)	O5-H18...O8	2.564 (4)
N3-H13...O4 ⁱⁱ	2.956 (3)	O7-H19...O3	2.628 (4)
N4-H14...O6	2.951 (4)	O9-H20...O1	2.557 (4)

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

atoms anisotropic, H atoms isotropic; 280 parameters [1 scale factor, 22 anisotropic atoms, 20 isotropic atoms, and a Rogers (1981) parameter η (an indicator of absolute configuration and twinning in non-centrosymmetric structures)]; $R = 0.021$ (non-zero-weighted data); $R(\text{all data}) = 0.026$; $wR = 0.025$; $S = 1.01$; $w = 4F^2/[\sigma^2(F^2) + (0.035F^2)^2]$; $\eta = 1.02$ (2) (theoretically, $\eta = 1.0$ for correct absolute configuration and no twinning); max. $\Delta/\sigma < 0.02$; max. empirical isotropic correction for extinction 12% of F ; max. and min. of ΔF synthesis 0.6 and $-0.5 \text{ e } \text{\AA}^{-3}$; scattering factors from *International Tables for X-ray Crystallography* (1974); local unpublished programs and ORTEP (Johnson, 1976). Atomic coordinates are listed in Table 1,* with the atomic numbering scheme shown in Fig. 1.

Related literature. The structure consists of [L-arginine]²⁺ cations and [AsO₂(OH)₂]⁻ anions interconnected by a network of hydrogen bonds that involve all of the H atoms bonded to the N and O atoms with the exception of H12. Distances and angles are given in Table 2. The optical properties of the title compound are reported elsewhere (Velsko, 1986; Monaco, Davis, Velsko, Wang, Eimerl & Zalkin, 1987).

*Lists of structure factors, anisotropic thermal parameters, H-atom parameters and interatomic distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51557 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

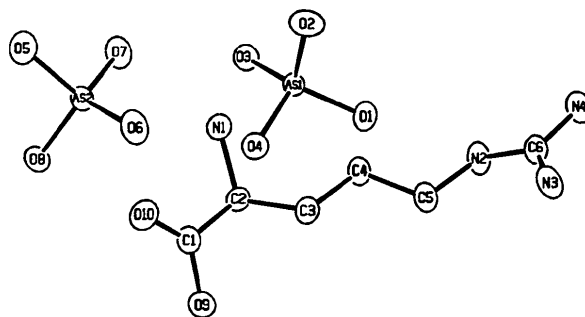


Fig. 1. ORTEP drawing showing the atomic numbering scheme; 50% probability ellipsoids are shown.

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Structure of the 1/1 Complex Between Chromium Tricarbonyl and (–)-Canadine

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Abstract. Tricarbonyl{8a–12a- η -5,6,13,13a-tetrahydro-9,10-dimethoxy-2,3-(methylenedioxy)-8H-dibenzo[*a,g*]quinolizine}chromium. [Cr(C₂₀H₂₁NO₄)(CO)₃], $M_r = 475.4$, orthorhombic, $P2_12_12_1$, $a = 8.061$ (2), $b = 14.658$ (1), $c = 18.001$ (1) Å, $V = 2127.06$ Å³, $Z = 4$, $D_x = 1.485$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.54148$ Å, $\mu = 4.88$ mm⁻¹, $F(000) = 984$, $T = 293$ K, $R = 0.045$ for 1681 observed reflections with $I > 3\sigma(I)$. The molecule is nearly planar with the two aromatic rings inclined at an angle of 150.1° to each other. The

chromium atom is 1.74 Å from the best plane calculated through the aromatic ring to which it is complexed.

Experimental. Columnar yellow crystals (0.20 × 0.20 × 0.58 mm); Enraf–Nonius CAD-4F diffractometer with graphite monochromator; $\omega/2\theta$ scans; lattice parameters from least-squares fits of 25 reflections ($25 < \theta < 35^\circ$); semi-empirical absorption correction (transmission factors 1.00–1.15); $\theta_{\text{max}} = 75^\circ$ ($h =$

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