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Acta Cryst. (1988). **C44**, 2024–2025

ortho-(Benzoyl)phenylarsonic Acid

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(Received 20 May 1988; accepted 21 June 1988)

Abstract. C₁₃H₁₁AsO₄, $M_r = 306.15$, orthorhombic, $P2_12_12_1$, $a = 6.1611$ (3), $b = 13.6596$ (7), $c = 14.7371$ (11) Å, $V = 1240.26$ Å³, $Z = 4$, $D_x = 1.639$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 2.73$ mm⁻¹, $F(000) = 616$, $T = 298$ K, $R = 0.0178$ for 2125 observed reflections. There is a short non-bonded interaction [2.741 (2) Å] between the carbonyl oxygen [O(4)] and As, and both OH groups make hydrogen bonds [O(2)–O(1') 2.619 (3) and O(3)–O(1') 2.607 (3) Å] to the relatively charged terminal oxygens of symmetry-related arsonate groups.

Experimental. Crystal size 0.5 × 0.3 × 0.3 mm. Stoe-Siemens four-circle diffractometer, monochromated Mo $K\alpha$ radiation, profile-fitting mode involving variable scan width and speed (Clegg, 1981). 3654 reflections measured, $2\theta_{\text{max}} 50^\circ$, three check reflections with no significant intensity variation. 2174 unique reflections ($R_{\text{int}} = 0.0168$), of which 2125 with $F > 3\sigma(F)$ were used for all calculations (*SHELXS86*, Sheldrick, 1985; *SHELX76*, Sheldrick, 1976). Cell constants refined from $\pm 2\theta$ values of 54 reflections in the range 20–25°. 283 azimuthal scan reflections were used to correct the data for absorption (range of transmission factors: 0.736–0.869). An extinction correction was not necessary. Structure solution by Patterson interpretation. Refinement on F to $R = 0.0178$, $wR = 0.0231$; all non-H atoms anisotropic, H atoms were included using a riding model, except for those attached to O which were refined with restrained O–H bond lengths [C–H 0.96, O–H 0.90 Å, $U(\text{H}) = 0.08$ Å²]. 169 parameters refined, $S = 1.37$, weighting scheme $w^{-1} = \sigma^2(F) + 0.00015F^2$ which led to a featureless analysis of variance in terms of $\sin\theta$ and F_o , max. $\Delta/\sigma = 0.001$, max. and min. height in final $\Delta\rho$ map 0.39 and

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (Å² $\times 10^3$)

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}^*
As(1)	664 (1)	8384 (1)	692 (1)	27 (1)
O(1)	740 (2)	7177 (1)	634 (1)	31 (1)
O(2)	3078 (3)	8875 (1)	351 (1)	40 (1)
O(3)	–1123 (3)	8929 (1)	–36 (1)	35 (1)
O(4)	–3214 (3)	7775 (1)	1408 (1)	44 (1)
C(1)	403 (4)	8944 (1)	1888 (1)	30 (1)
C(2)	1982 (4)	9610 (2)	2145 (2)	40 (1)
C(3)	1826 (5)	10079 (2)	2984 (2)	50 (1)
C(4)	64 (5)	9900 (2)	3535 (2)	51 (1)
C(5)	–1458 (5)	9217 (2)	3294 (2)	43 (1)
C(6)	–1311 (4)	8710 (1)	2472 (1)	31 (1)
C(7)	–2915 (4)	7945 (2)	2211 (1)	34 (1)
C(8)	–4072 (4)	7366 (1)	2913 (1)	33 (1)
C(9)	–3179 (5)	7166 (2)	3765 (2)	44 (1)
C(10)	–4275 (5)	6561 (2)	4368 (2)	58 (1)
C(11)	–6243 (6)	6163 (2)	4139 (2)	60 (1)
C(12)	–7130 (5)	6355 (2)	3302 (2)	54 (1)
C(13)	–6069 (4)	6944 (2)	2688 (2)	42 (1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

–0.31 e Å⁻³ respectively. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The absolute structure of the crystal actually chosen was determined by Rogers' (1981) η -refinement [$\eta = 1.03$ (2)].

Atomic parameters are given in Table 1, bond distances and angles in Table 2.* Fig. 1 shows a thermal ellipsoid plot with the atom numbering.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51173 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (Å) and angles (°)

As(1)—O(1)	1.652 (1)	As(1)—O(2)	1.707 (2)
As(1)—O(3)	1.708 (2)	As(1)—C(1)	1.928 (2)
O(4)—C(7)	1.220 (3)	C(1)—C(2)	1.385 (3)
C(1)—C(6)	1.399 (3)	C(2)—C(3)	1.396 (3)
C(3)—C(4)	1.378 (4)	C(4)—C(5)	1.370 (4)
C(5)—C(6)	1.398 (3)	C(6)—C(7)	1.489 (3)
C(7)—C(8)	1.484 (3)	C(8)—C(9)	1.398 (3)
C(8)—C(13)	1.399 (3)	C(9)—C(10)	1.388 (4)
C(10)—C(11)	1.371 (5)	C(11)—C(12)	1.374 (4)
C(12)—C(13)	1.375 (4)		
O(1)—As(1)—O(2)	110.6 (1)	O(1)—As(1)—O(3)	114.9 (1)
O(2)—As(1)—O(3)	101.9 (1)	O(1)—As(1)—C(1)	116.5 (1)
O(2)—As(1)—C(1)	100.7 (1)	O(3)—As(1)—C(1)	110.3 (1)
As(1)—C(1)—C(2)	116.9 (2)	As(1)—C(1)—C(6)	122.3 (2)
C(2)—C(1)—C(6)	120.8 (2)	C(1)—C(2)—C(3)	119.7 (2)
C(2)—C(3)—C(4)	119.7 (2)	C(3)—C(4)—C(5)	120.5 (2)
C(4)—C(5)—C(6)	121.2 (2)	C(1)—C(6)—C(5)	118.0 (2)
C(1)—C(6)—C(7)	120.2 (2)	C(5)—C(6)—C(7)	121.9 (2)
O(4)—C(7)—C(6)	118.9 (2)	O(4)—C(7)—C(8)	120.1 (2)
C(6)—C(7)—C(8)	120.9 (2)	C(7)—C(8)—C(9)	122.8 (2)
C(7)—C(8)—C(13)	118.5 (2)	C(9)—C(8)—C(13)	118.5 (2)
C(8)—C(9)—C(10)	120.0 (3)	C(9)—C(10)—C(11)	120.6 (2)
C(10)—C(11)—C(12)	119.8 (3)	C(11)—C(12)—C(13)	120.9 (3)
C(8)—C(13)—C(12)	120.2 (2)		

Related literature. For the preparation of the compound see Aeschlimann & McClelland (1924), who also assigned the structure correctly; for spectroscopic studies see Parmar & Saluja (1988). For spectroscopic studies of related compounds see Parmar, Basra, Malhotra & Sandhu (1980, 1981).

We thank the Deutsche Forschungsgemeinschaft and Fonds der Chemischen Industrie for financial support.

Acta Cryst. (1988). **C44**, 2025–2027

Structure of Pentacarbonyl(cycloheptatrienyl)(cyclopentadienyl)dimolybdenum(Mo—Mo)

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(Received 18 May 1988; accepted 1 August 1988)

Abstract. $[\text{Mo}_2(\text{C}_5\text{H}_5)(\text{C}_7\text{H}_7)(\text{CO})_5]$, $M_r = 488.2$, monoclinic, $P2_1/c$, $a = 9.949(2)$, $b = 8.568(1)$, $c = 20.580(5)$ Å, $\beta = 110.00(3)^\circ$, $V = 1648.5$ Å³, $Z = 4$, $D_x = 1.97$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 1.67$ mm⁻¹, $F(000) = 952$, $T = 294$ K, $R = 0.0222$ for 2393 unique reflections with $F > 3\sigma(F)$. The molecule consists of $\text{Mo}(\eta\text{-C}_7\text{H}_7)(\text{CO})_2$ and $\text{Mo}(\eta\text{-C}_5\text{H}_5)(\text{CO})_3$ fragments linked by an unsupported Mo—Mo bond of length 3.160(1) Å.

Experimental. The title compound was prepared by reaction of $[\text{Mo}(\eta\text{-C}_7\text{H}_7)(\text{NCMe})(\text{CO})_2][\text{BF}_4]$ with $[\text{Mo}(\eta\text{-C}_5\text{H}_5)(\text{CO})_3]$ (Breeze, Ricalton & Whiteley,

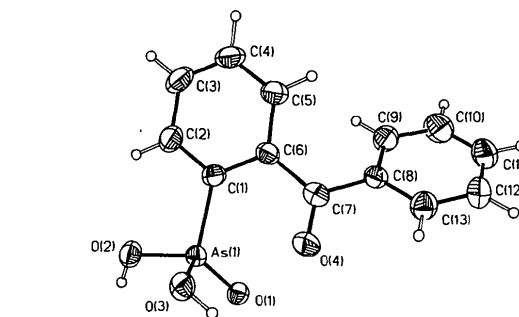


Fig. 1. View of the title molecule with atom numbering.

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1987); crystallization by slow diffusion of hexane into a CH_2Cl_2 solution of the compound afforded dark-green crystals. Crystal size 0.075 × 0.16 × 0.28 mm, CAD-4 diffractometer, cell parameters were derived from the setting angles of 25 reflections ($19 < 2\theta < 25^\circ$), no absorption or extinction correction, no significant systematic drift in one standard reflection measured at $2\frac{1}{2}$ h intervals during data collection. Scan mode $\omega/2\theta$, scan width = $(0.6 + 0.35 \tan\theta)^\circ$, $(\sin\theta/\lambda)_{\text{max}} = 0.595$ Å⁻¹, scan time 7 to 120 s, h 0→11, k 0→10, l -24→22. 3104 measured reflections, all unique, 2393 with $F > 3\sigma(F)$ used in refinement. Structure solved by the heavy-atom method, refinement was by partitioned