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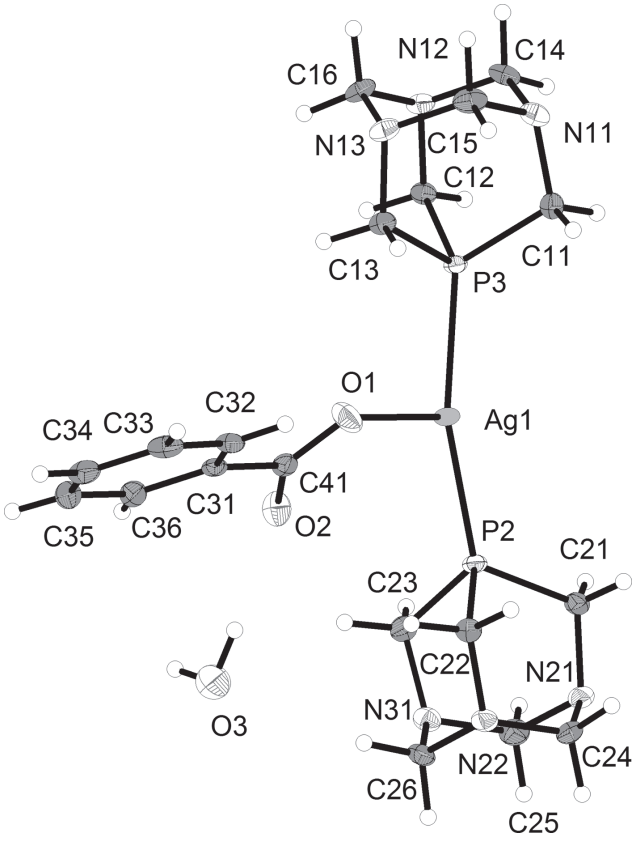
Crystal structure of benzoato-κ*O*-bis(1,3,5-triaza-7-phosphaadamantane-κ*P*)silver(I) monohydrate $C_{19}H_{31}AgN_6O_3P_2$



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Abstract

$C_{19}H_{31}AgN_6O_3P_2$, monoclinic, $P2_1/c$ (no. 14), $a = 16.5661(8)$ Å, $b = 6.1644(3)$ Å, $c = 23.8822(10)$ Å, $\beta = 114.553(3)°$, $V = 2218.32(18)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0188$, $wR_{ref}(F^2) = 0.0525$, $T = 100(2)$ K.

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Table 1: Data collection and handling.

Crystal:	Colourless cuboid
Size:	$0.57 \times 0.28 \times 0.22$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	10.9 cm^{-1}
Diffractometer, scan mode:	Bruker X8 APEX-II, φ and ω
$2\theta_{max}$, completeness:	56°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	43157, 5330, 0.041
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5034
$N(param)_{refined}$:	288
Programs:	Bruker programs [1], SIR97 [2], DIAMOND [3], SHELX [4], WinGX [5]

The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

Silver benzoate (72.4 mg, 0.308 mmol) and 1,3,5-triaza-7-phosphaadamantane (150 mg, 0.955 mmol) were dissolved in acetonitrile (10 mL). The resulting mixture was refluxed for 30 min and filtrated. The filtrate was left to crystallize via slow evaporation. A product of colourless needle-like crystals suitable for X-ray diffraction was obtained.

Experimental details

In the structure all the H atoms were positioned geometrically and refined discernibly using a riding model, with C—$H_{methylene}$ = 1.00 Å; C—$H_{aromatic}$ = 0.95 Å. The H atom isotropic displacement parameters were fixed; $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic, and methylene allowing them to ride on the parent atom. H atoms bonded to O3 were placed according to the electron-density map. To obtain sufficient high angle reflections, the exposure time had to be extended, resulting in the overexposure of some very strong reflections. Hence 26 reflections that are below Theta (min) have been omitted from the refinement. Sufficient unique reflections were obtained (5330) to yield a fully refined structure with 99.5% data completeness.

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	U_{iso}*/U_{eq}
N31	0.55577(8)	0.7589(2)	0.14883(6)	0.0175(2)
O3	0.36317(10)	0.9171(2)	−0.01204(8)	0.0392(3)
H3A	0.338(2)	0.796(5)	−0.0063(14)	0.063(9)*
H3B	0.3304(18)	1.008(5)	−0.0056(12)	0.050(7)*
Ag1	0.32886(2)	0.18657(2)	0.11611(2)	0.01643(4)
C11	0.23860(10)	−0.3315(2)	0.14836(7)	0.0178(3)
H11A	0.2667	−0.4012	0.1236	0.021*
H11B	0.2801	−0.3442	0.1923	0.021*
P3	0.21891(2)	−0.04053(6)	0.12726(2)	0.01245(7)
P2	0.46352(2)	0.37287(6)	0.13372(2)	0.01344(7)
O2	0.27604(8)	0.56894(18)	0.02370(5)	0.0234(2)
O1	0.24712(8)	0.21537(19)	0.00640(5)	0.0259(3)
N11	0.15529(9)	−0.4456(2)	0.13790(6)	0.0196(3)
N13	0.05692(8)	−0.2199(2)	0.05057(6)	0.0182(3)
N22	0.58205(8)	0.4794(2)	0.08441(6)	0.0162(2)
C21	0.57024(10)	0.3052(2)	0.19846(7)	0.0173(3)
H21A	0.5653	0.3323	0.2378	0.021*
H21B	0.5827	0.1489	0.1967	0.021*
C12	0.15119(9)	0.0242(2)	0.17031(6)	0.0146(3)
H12A	0.1895	0.0216	0.215	0.018*
H12B	0.1266	0.1725	0.1594	0.018*
C35	0.10136(11)	0.6980(3)	−0.16082(7)	0.0226(3)
H35	0.0764	0.8375	−0.1741	0.027*
N21	0.64483(8)	0.4325(2)	0.19741(6)	0.0177(2)
C33	0.12189(11)	0.3282(3)	−0.18368(7)	0.0215(3)
H33	0.1124	0.2154	−0.2129	0.026*
N12	0.07790(8)	−0.1307(2)	0.15660(6)	0.0159(2)
C36	0.15246(10)	0.6592(2)	−0.09867(7)	0.0182(3)
H36	0.1639	0.7738	−0.0698	0.022*
C22	0.49987(10)	0.3559(2)	0.07027(6)	0.0150(3)
H22A	0.5098	0.202	0.0631	0.018*
H22B	0.4524	0.4134	0.0321	0.018*
C13	0.12752(9)	−0.0759(2)	0.05010(6)	0.0162(3)
H13A	0.1016	0.0678	0.0339	0.019*
H13B	0.1518	−0.1366	0.0219	0.019*
C41	0.24154(9)	0.4108(2)	−0.01071(7)	0.0162(3)
C15	0.09247(10)	−0.4371(2)	0.07261(7)	0.0218(3)
H15A	0.1227	−0.4916	0.0473	0.026*
H15B	0.0423	−0.5359	0.0662	0.026*
C25	0.63007(11)	0.6659(2)	0.20200(7)	0.0209(3)
H25A	0.6194	0.6911	0.2393	0.025*
H25B	0.685	0.7447	0.2075	0.025*
C26	0.56983(11)	0.7106(2)	0.09305(7)	0.0181(3)
H26A	0.5181	0.7644	0.0567	0.022*
H26B	0.6228	0.7908	0.0952	0.022*
C31	0.18708(9)	0.4531(2)	−0.07848(6)	0.0144(3)
C34	0.08676(10)	0.5331(3)	−0.20356(7)	0.0226(3)
H34	0.0529	0.5602	−0.2461	0.027*
C24	0.65488(10)	0.3944(3)	0.13967(7)	0.0190(3)
H24A	0.711	0.462	0.1432	0.023*
H24B	0.6598	0.2363	0.1345	0.023*
C14	0.11202(11)	−0.3515(2)	0.17480(7)	0.0206(3)
H14A	0.1551	−0.3494	0.2185	0.025*
H14B	0.0621	−0.4466	0.1716	0.025*
C32	0.17084(9)	0.2869(2)	−0.12143(7)	0.0177(3)
H32	0.1933	0.1454	−0.1081	0.021*
C23	0.46995(10)	0.6718(2)	0.14333(7)	0.0170(3)
H23A	0.4215	0.7396	0.1075	0.02*
H23B	0.4612	0.7105	0.1807	0.02*
C16	0.01776(9)	−0.1323(3)	0.09080(7)	0.0191(3)
H16A	−0.0353	−0.2193	0.0851	0.023*
H16B	−0.0022	0.0181	0.0778	0.023*

Comment

Several adamantane based drugs are in use to treat a whole variety of ailments. Some of these include hyperglycaemia [6], Alzheimer's and central nervous disorders [7–9], malaria [10], anti-viral [11] and drug resistanct TB strains [12, 13]. Due to the pharmaceutical advantages gained from these model compounds, 1,3,5-triaza-7-phosphaadamantane (PTA), a derivative of adamantane, has been investigated in a variety of applications such as medicinal chemistry [14, 15], catalysis [16–18] and luminescence [19].

PTA has a high solubility in water and its pharmacological properties make it an ideal candidate in the search for better and more effective pharmaceutical models. Silver is known to be bio-active and silver-organic networks can show antimicrobial properties [20]. Significant antibacterial and antifungal activity has already been reported with silver-PTA networks [21]. Aside from the many pharmaceutical advantages, PTA in conjunction with silver nano-particles can catalyse the hydration of nitriles to amides in water under mild conditions and has been shown to excel at cyanohydrin hydration [22].

The title compound crystallises in the monoclinic space group $P2_1/c$ and consists of a silver metal centre with two PTA ligands bound in a near linear fashion with a P2—Ag—P3 bond angle of 163.54(1)° and similar bond distances (Ag—P2 = 2.3860(4) Å and Ag—P3 = 2.3982(5) Å). The benzoato ligand shows monodentate coordination *via* the oxygen atom. The benzoato ligand occupies the position of least steric interference as indicated by the P3—Ag—O1 bond angle of 93.11(3)°. The position of the benzoato ligand is stabilised by intra- and intermolecular hydrogen bonding with the water molecule (O2···O3, D···A = 2.906(2) Å and O1···O3$^{\#1}$, D···A = 2.824(2) Å, symmetry operator #1 = x, $-1+y$, z).

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