Crystal structure of aluminium cyclononaphosphate, $Al_3P_9O_{27}$

D. Fratzky*,[I], M. Schneider[II] and M. Meisel[I]

[I] Humboldt-Universität zu Berlin, Institut für Anorganische und Allgemeine Chemie, Hessische Straße 1-2, D-10115 Berlin, Germany
[II] Institut für Angewandte Chemie Berlin-Adlershof e.V., Rudower Chaussee 5, D-12484 Berlin, Germany

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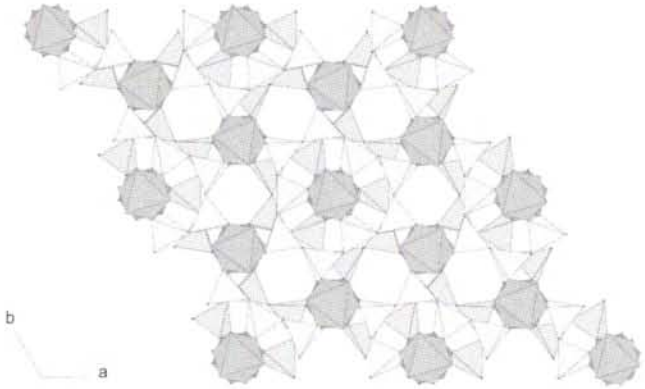

Abstract

$Al_3O_{27}P_9$, trigonal, $P3c1$ (No. 165), a = 10.935(2) Å, c = 9.191(2) A, V = 951.8 Å^3, Z = 8, $R_{gt}(F)$ = 0.037, $wR_{ref}(F^2)$ = 0.094, T = 293 K.

Source of material

2g $AlCl_3 \cdot 6H_2O$ was dissolved in 20 g hot (420 K) $H_4P_2O_7$ in a gold crucible and the mixture is stored for 14 days at 520 K. Then the mixture was dissolved in water and filtered. The residue contains clear white crystals of $Al_3P_9O_{27}$ and a fine second unknown compound.

Discussion

Cyclophosphates $[P_nO_{3n}]^{n-}$ are a common class of compounds. The ring anions are known to exist for n = 3, 4, 5, 6, 8, 9, 10 and 12. For all cyclophosphate anions exist relyable structural data with one exeption, the cyclononaphosphate anion [1]. The first successful attemps to prepare cyclononaphosphates were made by Bagieu [2]. In this work the authors characterized $Al_3P_9O_{27}$, $Fe_3P_9O_{27}$ and a $Cr_3P_9O_{27}$ and estimated the unit cell dimensions. At this state of work they couldn't recognize the chemical nature of the compounds. Later the authors prepared an isotypic $(Fe,V)_3P_9O_{27}$ and made a crystallographic investigation. Because of the inaccuracy of the investigation they didn't report any numerical values. We prepared crystals of $Al_3P_9O_{27}$ by investigations of the system $H_4P_2O_7/Al^{3+}$ and estimated the crystal structure of this compound. The structure of $Al_3P_9O_{27}$ consist of tubes with stacked P_9O_{27}-rings. The Al(1)-atom connects the rings in the tube each other. The tubes are arranged in a hexagonal symmetry and connected via the Al(2)-atom. A remarkable characteristic of the structure is, that all aluminium atoms and all phospate rings form separate layers parallel to the [110] plane. The bond distances are all in a normal range. Al(1) is surrounded by six O(1) with a Al—O distance of 1.874 Å and Al(2) is surrounded by O(2) and O(3) with distances of 1.888 Å and 1.875 Å. Phosphorus forms the typical tetrahedron with two long P—O bonds (1.57 Å) and two short P—O bonds (1.48 Å).

Table 1. Data collection and handling.

Crystal:	white prism, size 0.15 × 0.15 × 0.2 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	6.46 cm^{-1}
Diffractometer, scan mode:	Nonius Turbo CAD4, θ/2θ
$2\theta_{max}$:	63.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5474, 935
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 725
$N(param)_{refined}$:	62
Programs:	DIAMOND [3], SHELXS-90 [4], SHELXL-97 [5]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
P(1)	6*f*	0.2052(1)	0	1/4	0.0063(3)	0.0060(4)	0.0090(5)	$U_{22}/2$	0.0004(2)	$2U_{13}$
Al(1)	2*b*	0	0	0	0.0083(6)	U_{11}	0.006(1)	$U_{11}/2$	0	0
Al(2)	4*d*	2/3	1/3	-0.0021(2)	0.0109(4)	U_{11}	0.0086(7)	$U_{11}/2$	0	0
P(2)	12*g*	0.44583(8)	0.28727(8)	0.24318(8)	0.0060(3)	0.0058(3)	0.0084(3)	0.0032(3)	0.0000(3)	0.0001(3)
O(1)	12*g*	0.1175(2)	-0.0383(2)	0.1165(3)	0.014(1)	0.014(1)	0.012(1)	0.0084(9)	-0.0041(8)	0.0001(8)
O(2)	12*g*	0.5359(2)	0.3541(2)	0.1143(3)	0.016(1)	0.012(1)	0.017(1)	0.0064(9)	0.0093(9)	0.0049(9)
O(3)	12*g*	0.6831(3)	0.4839(3)	-0.1145(3)	0.026(1)	0.020(1)	0.014(1)	0.013(1)	0.005(1)	0.0094(9)
O(4)	12*g*	0.3590(2)	0.1227(2)	0.2150(3)	0.009(1)	0.0047(9)	0.023(1)	0.0025(8)	0.0060(8)	-0.0003(8)
O(5)	6*f*	0.3239(3)	x	1/4	0.008(1)	U_{11}	0.022(2)	0.006(1)	-0.0002(6)	$-U_{13}$

* Correspondence author (e-mail: D_Fratzky@yahoo.de)

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