Crystal structure of tetraaluminium trinitride carbide oxide, Al_4N_3CO

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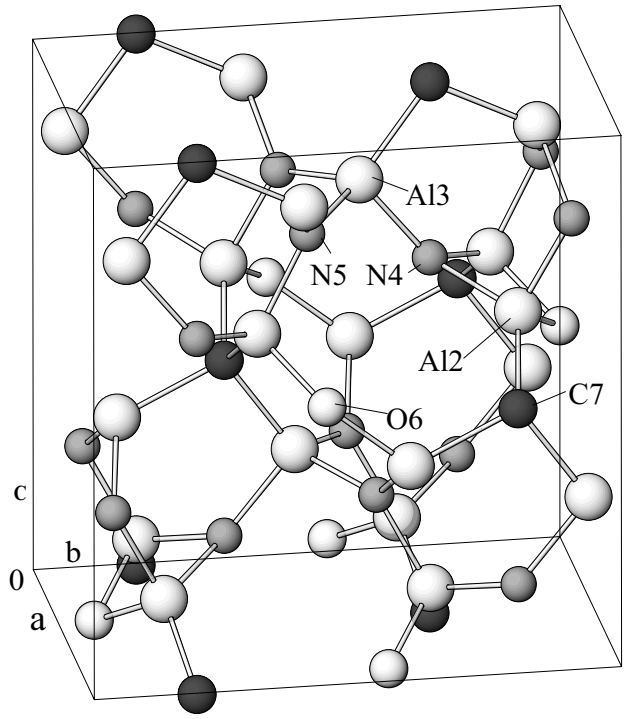

Abstract

Al_4CN_3O, orthorhombic, $Cmc2_1$ (No. 36), a = 5.7431(5) Å, b = 8.528(1) Å, c = 9.094(1) Å, V = 445.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.026, $wR_{ref}(F^2)$ = 0.098, T = 293 K.

Source of material

Mixtured powder of AlN and Al was pressed into pellet. The pellet was put in a BN crucible and then heated with a graphite susceptor in a RF furnace. The mixture was first heated to about 1773 K in flowing nitrogen atmosphere, kept there for 8 to 12 hours, then cooled slowly (about 40 K/h) down to 773 K. Finally, switch off the power and let the melt cooled down to room temperature. Single crystals with needle shape were obtained on the surface of the Al matrix and on the wall of the crucible. The crystal is transparent to light red in colour and from nanometer to several centimeters long in size. EPMA analysis results indicate that the composition of these crystals are near Al_4N_3CO. The elements of C and O came from the environment, such as the graphite crucible, nitrogen gas or inpurity oxygen in the starting material.

Discussion

The obtained Al_4N_3CO single crystal is isostructural with the ternary compound: Al_4O_4C [1]. All Al atoms have four ligands to form distorted tetrahedra, in which atoms of Al1 and Al3 bond with two N atoms, one O and one C atom, Al2 bonds with three N atoms and another C. The average bond length between Al and its ligands are: d(Al—N4) = 1.8360 Å, d(Al—N5) = 1.8116 Å, d(Al—O) = 1.7757 Å and d(Al—C) =1.9226 Å. Every N and O atoms are bonded with three Al atoms, in which O and three ligand atoms of Al are located in one plane. Also, N5 and its three Al ligands are nearly in one plane, but N4 and its three ligands are not in a plane. The C7 is coordinated with four Al atoms and they build up a slightly distorted tetrahedron.

Table 1. Data collection and handling.

Crystal:	transparent needle, size 0.200 × 0.225 × 0.610 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	9.15 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω
$2\theta_{max}$:	89.76°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2030, 1947
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1911
$N(param)_{refined}$:	50
Programs:	SHELXS-97 [2], SHELXL-97 [3]

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}
Al(1)	4*a*	0	0.60105(6)	0.40260(5)	0.0050(2)	0.0052(2)	0.0054(2)	0	0	0.0002(1)
Al(2)	8*b*	0.22883(6)	0.66424(5)	0.11270(4)	0.0044(1)	0.0064(1)	0.0052(1)	–0.00006(9)	0.0004(1)	–0.00001(9)
Al(3)	4*a*	0	0.93855(6)	0.32229(6)	0.0049(2)	0.0051(2)	0.0052(2)	0	0	–0.0000(1)
N(4)	4*a*	0	0.8050(1)	0.1672(1)	0.0039(3)	0.0041(3)	0.0051(4)	0	0	–0.0017(3)
N(5)	8*b*	0.2550(2)	0.5653(1)	0.28823(9)	0.0039(2)	0.0061(2)	0.0045(2)	0.0019(2)	0.0011(2)	0.0015(2)
O(6)	4*a*	0	0.4418(2)	0.5233(1)	0.0060(3)	0.0078(4)	0.0086(4)	0	0	0.0033(3)
C(7)	4*a*	0	0.8046(2)	0.4973(2)	0.0053(4)	0.0068(4)	0.0051(4)	0	0	0.0003(3)

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