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γ-Sodium gallate: a Rietveld refinement using X-ray powder diffraction

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Tetrahedrally coordinated oxides usually present polymorphism, but for $NaGaO_2$, only the β polymorph has been reported. In this work, the synthesis and structural characterization of γ-sodium gallate, γ-$NaGaO_2$, are presented. The crystal structure belongs to the orthorhombic system, space group *Pbca* (No. 61), and has been characterized by a Rietveld refinement of the X-ray powder diffraction pattern. The structure is similar to those exhibited by the γ phases of many tetrahedral oxides.

Comment

$NaGaO_2$ belongs to the group of tetrahedrally coordinated oxides with the general formula ABO_2, where A is Li or Na and B is Al, Ga or Fe (B is Fe only when A is Na). These oxides are usually polymorphic and the polymorphs fall into two groups.

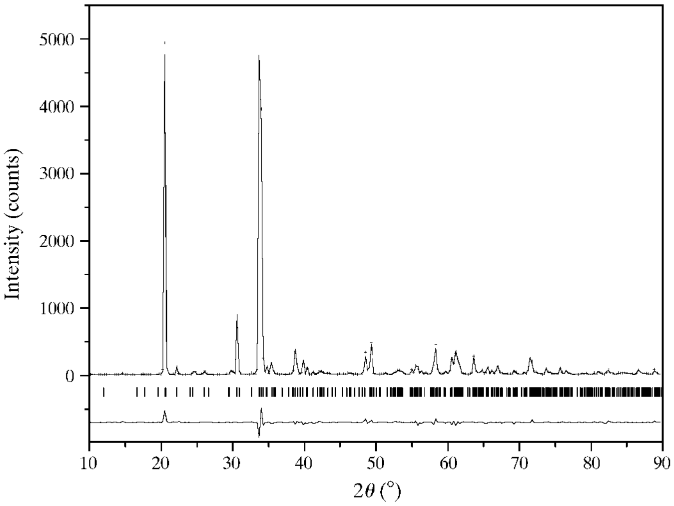

Figure 1
Comparison of the observed (+) and calculated (solid line) intensities for γ-$NaGaO_2$. The difference pattern is given underneath.

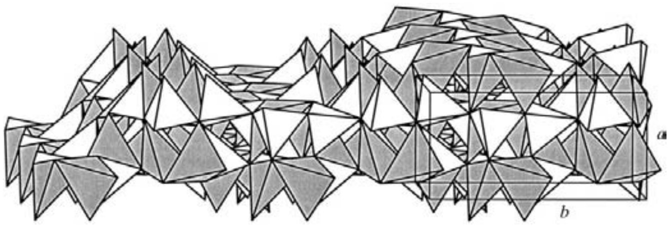

Figure 2
A view of part of the structure of γ-$NaGaO_2$. Note that some tetrahedra point downwards and others point upwards. GaO_4 tetrahedra are white and NaO_4 tetrahedra are grey.

The first group is known as the low-temperature β phase, having the basic wurtzite structure, and the second group is the high-temperature γ phase, where the crystal structure presents the cations distributed over two different sets of available tetrahedral sites.

In addition to these β and γ polymorphic phases, a low-temperature, and sometimes high-pressure, α form occurs (West, 1975).

Vielhaber & Hoppe (1969) and Müller & Hoppe (1992) have reported crystal data for the β phase of $NaGaO_2$, but no reports exist to date concerning the synthesis or crystal characterization of the γ phase of $NaGaO_2$, which is reported here.

Experimental

The γ polymorph of $NaGaO_2$ was synthesized by a solid-state reaction. The starting materials were Na_2CO_3 (99.7%, Baker) and Ga_2O_3 (99.99%, Aldrich). A mixture totalling 10 g was prepared by weighing Na_2CO_3 and Ga_2O_3, and mixing them into a paste with acetone in an agate mortar. The mixture was fired in an electric muffle furnace, whose temperature was controlled and measured. Initial firing was at 873–973 K for a few hours to expel CO_2, followed by 6 d at 1323 K and quenching in ice to room temperature.

Crystal data

$NaGaO_2$
$M_r = 124.71$
Orthorhombic, *Pbca*
$a = 5.3145\ (2)$ Å
$b = 10.6234\ (5)$ Å
$c = 14.7782\ (7)$ Å
$V = 834.36\ (7)$ Å^3
$Z = 16$
$D_x = 3.97$ Mg m^{-3}
$D_m = 3.6$ Mg m^{-3}
Density measured by picnometry
Cu $K\alpha_1$ and Cu $K\alpha_2$ radiation
$\mu = 1.713$ mm^{-1}
$T = 295$ K
Specimen shape: flat sheet
20 × 20 × 0.2 mm
Particle morphology: no specific colour, white
Specimen prepared at 1323 K

Data collection

Siemens *D*5000 diffractometer
Specimen mounting: packed powder sample container
Specimen mounted in reflection mode
$2\theta_{\min} = 10$, $2\theta_{\max} = 90°$
Increment in $2\theta = 0.02°$

Refinement

$R_p = 0.12$
$R_{\rm exp} = 0.11$
$R_B = 0.05$
$S = 1.48$
$2\theta_{\min} = 10$, $2\theta_{\max} = 90°$
Increment in $2\theta = 0.02°$
Wavelength of incident radiation:
Cu $K\alpha_1 = 1.541$ Å
Cu $K\alpha_2 = 1.544$ Å
Profile function: pseudo-Voigt modified by Thompson *et al.* (1987)
363 reflections
47 parameters
Preferred orientation correction: none

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Table 1
Selected interatomic distances (Å).

Ga1—O2	1.84 (2)	Na1—O1	2.39 (2)
Ga1—O3	1.83 (2)	Na1—O2	2.33 (2)
Ga1—O4	1.85 (2)	Na1—O2^{iii}	2.34 (2)
Ga1—O4^{i}	1.81 (2)	Na1—O4	2.29 (2)
Ga2—O1	1.79 (2)	Na2—O1	2.40 (2)
Ga2—O1^{ii}	1.83 (2)	Na2—O3	2.35 (1)
Ga2—O2	1.85 (2)	Na2—O3^{i}	2.29 (2)
Ga2—O3	1.87 (2)	Na2—O4	2.43 (2)

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

The positions of 37 resolvable peaks were input into the *LSUCRI* program for least-squares unit-cell refinement (Garvey, 1986). The starting set of cell parameters for the refinement was taken from the data reported by Grey *et al.* (1990) for the isostructural compound $KGaO_2$. The close values observed for the full width at half-maximum (FWHM; 0.12° at 28.446°), corresponding to the 111 peak of Si (*Natl Bur. Stand.* 640BC) used as external standard, and that for the 231 peak, belonging to the sample (FWHM = 0.124° at 29.962°), indicate a high degree of crystallinity in the specimen. The powder diffraction data were refined by the Rietveld method, using the coordinates reported for $KGaO_2$ as starting parameters. A pseudo-Voigt function modified by Thompson *et al.* (1987) was chosen to generate the line shape of the diffraction peaks. The background was fitted to a polynomial refinable function. The following parameters were refined in space group *Pbca*: zero point, scale factor, six background polynomial coefficients, unit-cell dimensions, half-width, pseudo-Voigt and asymmetry parameters for the peak shape, and positional and isotropic displacement parameters. The *FULLPROF* program for Rietveld refinement and pattern-matching analysis (Juan Rodríguez-Carvajal; unpublished) is a modified version of that described by Wiles & Young (1981).

Data collection: *DIFFRAC/AT* (Siemens, 1993); cell refinement: *LSUCRI* (Garvey, 1986); program(s) used to refine structure: *FULLPROF* (Rodríguez-Carvajal; unpublished); molecular graphics: *ATOMS* (Dowty, 1994); software used to prepare material for publication: *ATOMS*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BR1357). Services for accessing these data are described at the back of the journal.

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