# Redetermination of the crystal structure of sodium trisamarium digermanate dihydroxide, $NaSm_3(GeO_4)_2(OH)_2$

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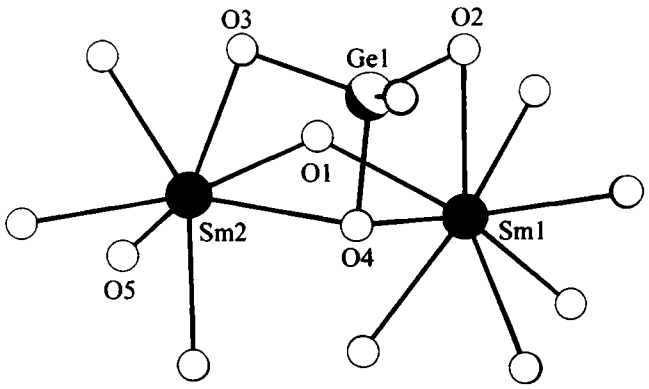


## Abstract

$Ge_2H_2NaO_{10}Sm_3$, monoclinic, $C12/c1$ (No. 15), $a = 18.323(3)$ Å, $b = 5.2369(8)$ Å, $c = 12.108(2)$ Å, $\beta = 131.230(2)°$, $V = 873.8$ Å$^3$, $Z = 4$, $R_{gt}(F) = 0.037$, $wR_{ref}(F^2) = 0.010$, $T = 153$ K.

## Source of material

Yellow crystals of $NaSm_3(GeO_4)_2(OH)_2$ were obtained from a hydrothermal reaction of 0.035 g (0.10 mmol) $Sm_2O_3$ and 0.020 g (0.20 mmol) $GeO_2$ in 0.4 mL of NaOH (10.5 M) solution sealed in a silver tube that was heated at 797 K for 3 days.

## Discussion

Hydrothermal methods have been used successfully to synthesize many compounds [1,2]. The title compound was first synthesized in [3,4]., and the structure was described in the noncentrosymmetric space group *Cc*. However, we believed that the space group determination was not right based on the position of the atoms. In addition, a review article by Demianets gives the space group as *I2/a* for the same compound, but they did not give any explanation or new characterization [5].

Our X-ray single crystal structure determination of the compound was done with a final reliability factor $R = 0.037$ and space group was determined as *C2/c*. The hydrogen atoms were not localized. This structure has an olivine type chain parallel to *a*-axis [6]. The cation polyhedral chain is made up with alternation Na and Sm polyhedra. There are two crystallographically independent Sm atoms. The Sm1 is coordinated by eight oxygen atoms and Sm2 is coordinated by seven oxygen atoms in an irregular polyhedron. The Sm1—O distances range from 2.359(5) Å to 2.644(5) Å. The Sm2—O distances range from 2.353(5) Å to 2.507(5) Å. The Ge atom has four coordinating oxygen atoms forming a distorted tetrahedron. The Ge—O distances range from 1.741(5) Å to 1.786(5) Å.

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | yellow block, size 0.058 × 0.118 × 0.150 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 267.36 cm$^{-1}$ |
| Diffractometer, scan mode: | Bruker SMARTCCD, $\Delta\omega = 0.3°$ |
| $2\theta_{max}$: | 57.62° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 3997, 1023 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\,\sigma(I_{obs})$, 970 |
| $N(param)_{refined}$: | 78 |
| Programs: | SHELXS-97 [7], SHELXL-97 [8] |

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

| Atom | Site | Occ. | *x* | *y* | *z* | $U_{iso}$ |
|---|---|---|---|---|---|---|
| Na(1) | 8*f* | 0.23 | 0.247(3) | 0.246(7) | 0.509(4) | 0.006(4) |
| Na(2) | 8*f* | 0.27 | 0.264(1) | 0.295(3) | 0.480(2) | 0.006 |

**Table 3.** Atomic coordinates and displacement parameters (in Å$^2$).

| Atom | Site | *x* | *y* | *z* | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| Sm(1) | 4*e* | 0 | 0.29016(9) | 1/4 | 0.0066(3) | 0.0027(3) | 0.0031(3) | 0 | 0.0039(3) | 0 |
| Sm(2) | 8*f* | 0.11829(2) | 0.29557(7) | 0.08559(4) | 0.0070(3) | 0.0047(3) | 0.0029(3) | 0.0013(1) | 0.0035(2) | 0.0000(1) |
| Ge(1) | 8*f* | 0.14042(5) | 0.7395(1) | 0.29168(8) | 0.0077(4) | 0.0028(4) | 0.0045(4) | –0.0004(2) | 0.0048(4) | –0.0005(2) |
| O(1) | 8*f* | 0.1258(3) | 0.0696(9) | 0.2718(5) | 0.008(2) | 0.001(2) | 0.004(2) | –0.001(2) | 0.003(2) | –0.000(2) |
| O(2) | 8*f* | 0.1503(3) | 0.5952(9) | 0.4316(5) | 0.011(2) | 0.006(2) | 0.006(2) | –0.002(2) | 0.007(2) | –0.002(2) |
| O(3) | 8*f* | 0.2233(3) | 0.615(1) | 0.2779(5) | 0.008(2) | 0.006(2) | 0.013(2) | 0.001(2) | 0.008(2) | 0.001(2) |
| O(4) | 8*f* | 0.0339(4) | 0.5799(9) | 0.1375(5) | 0.009(2) | 0.005(2) | 0.003(2) | –0.001(2) | 0.004(2) | –0.001(2) |
| O(5) | 8*f* | 0.0876(4) | –0.118(1) | –0.0127(5) | 0.015(2) | 0.006(2) | 0.006(2) | –0.002(2) | 0.007(2) | –0.002(2) |

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