# Gd$_2$OSe$_2$

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Single crystals of digadolinium monooxygen diselenide, Gd$_2$OSe$_2$, have been obtained from a KBr flux. The compound is isostructural with the low-pressure form of Dy$_2$OS$_2$. All atoms lie on the mirror plane of *Pnma*. The Gd environments are a GdO$_3$Se$_5$ bicapped trigonal prism and a GdOSe$_5$ distorted octahedron. The two coordination polyhedra pack by face-sharing and edge-sharing to form a [Gd$_{8/4}$O$_{4/4}$Se$_{8/8}^a$-Se$_{8/8}^i$] fragment, which is the main motif of the structure. These fragments lie in the *ac* plane and form infinite chains parallel to *c* through the sharing of Se atoms around atom Gd1. In the *a* direction, these chains stack through the sharing of an Se atom around atom Gd2, thereby delimiting large pentagonal cavities.

## Comment

Rare-earth oxychalcogenides are of particular interest for their use as phosphors. Suitable material for efficient conversion of X-rays to light are Gd$_2$O$_2$S doped with Tb and Y$_2$O$_2$S doped with Eu.

Rare-earth monooxygen disulfides, *Ln*$_2$OS$_2$ (*Ln* = rare earth), exhibit two different structure types. The first is the Tm$_2$OS$_2$ structure type (monoclinic, *P*2$_1$/*c*) adopted by Er, Tm, Yb (Range *et al.*, 1990), Dy (Schleid, 1991), Y (Schleid, 1992) and Sm (Lissner & Schleid, 1992). The crystal structure is built from the close packing of two mixed *Ln*$Q_7$ ($Q$ = O, S) polyhedra and shows some similarities to the structures of rare-earth sesquichalcogenides. The second is the low-temperature Dy$_2$OS$_2$ structure type (orthorhombic, *Pnma*) (Schleid, 1991). The title compound, Gd$_2$OSe$_2$, is isotypic to this second structure type and represents the first rare-earth monooxygen diselenide to be characterized by single-crystal X-ray crystallography.

A displacement ellipsoid diagram of Gd$_2$OSe$_2$ is shown in Fig. 1. GdO$_3$Se$_5$ bicapped trigonal prisms and GdOSe$_5$ distorted octahedra pack by face-sharing and edge-sharing to form a [Gd$_{8/4}$O$_{4/4}$Se$_{8/8}^a$Se$_{8/8}^i$] (*i* = inner and *a* = apical) fragment that constitutes the main building motif of the structure. These fragments, which lie in the *ac* plane, form infinite chains parallel to the *c* axis by sharing Se atoms around atom Gd1. In the *a* direction, these chains stack by sharing Se atoms around

atom Gd2, delimiting pentagonal cavities. In the *b* direction, the three-dimensional interconnection occurs *via* face-sharing of trigonal prisms and edge-sharing of octahedra.
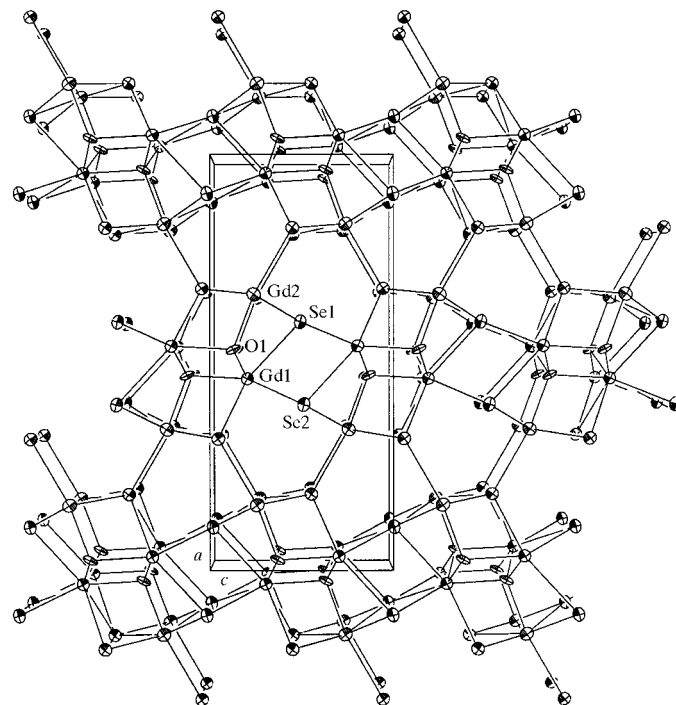


**Figure 1**
View of the structure of Gd$_2$OSe$_2$ down **b**. Displacement ellipsoids are drawn at the 90% probability level.

## Experimental

Single crystals of Gd$_2$OSe$_2$ were obtained as a side product of the reaction of Gd (0.290 g, Alfa, 99.9%), TiO$_2$ (0.029 g, Alfa, 99.9%), Ti (0.017 g, Alfa, 99.9%) and Se (0.228 g, Alfa, 99.5%), with KBr (200 mg, Alfa, 99%) added to promote crystal growth. The materials were mixed and sealed in a fused-silica tube that was then evacuated to 10$^{-4}$ Torr (1 Torr = 133.322 Pa). The tube was heated to 1223 K for 4 d before being cooled to 973 K at the rate of 3 K h$^{-1}$ when the furnace was turned off. The final product was washed with methanol and water, and dried with acetone. Yellow single crystals of Gd$_2$OSe$_2$ of rod-like habit were found. Qualitative energy dispersive spectroscopy (EDS) analysis verified the presence of Gd, Se, and O.

*Crystal data*

| | |
|---|---|
| Gd$_2$OSe$_2$ | Mo $K\alpha$ radiation |
| $M_r = 488.42$ | Cell parameters from 2878 |
| Orthorhombic, *Pnma* | reflections |
| $a = 16.050$ (3) Å | $\theta = 2.54$–28.29° |
| $b = 3.9375$ (8) Å | $\mu = 45.78$ mm$^{-1}$ |
| $c = 7.0309$ (14) Å | $T = 153$ (2) K |
| $V = 444.32$ (15) Å$^3$ | Rod, pale yellow |
| $Z = 4$ | 0.240 × 0.024 × 0.022 mm |
| $D_x = 7.301$ Mg m$^{-3}$ | |

# inorganic compounds

Data collection: *SMART* (Bruker, 1999); cell refinement: *SMART*; data reduction: *SAINT-Plus* (Bruker, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXTL/PC* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC*; software used to prepare material for publication: *SHELXTL/PC*.

**Table 1**
Selected geometric parameters (Å).

| | | | |
|---|---|---|---|
| Gd1—O1 | 2.317 (6) | Gd2—O1 | 2.240 (5) |
| Gd1—O1$^i$ | 2.326 (3) | Gd2—Se2$^{vi}$ | 2.8183 (7) |
| Gd1—O1$^{ii}$ | 2.326 (3) | Gd2—Se2$^{vii}$ | 2.8183 (7) |
| Gd1—Se2$^{iii}$ | 3.0228 (10) | Gd2—Se1$^{iv}$ | 2.8642 (8) |
| Gd1—Se2$^{iv}$ | 3.0932 (8) | Gd2—Se1$^v$ | 2.8642 (8) |
| Gd1—Se2$^v$ | 3.0932 (8) | Gd2—Se1 | 2.9203 (10) |
| Gd1—Se1$^{iv}$ | 3.1726 (8) | Gd2—Gd1$^i$ | 3.7968 (7) |
| Gd1—Se1$^v$ | 3.1726 (8) | | |

Symmetry codes: (i) $-x, -y, 1 - z$; (ii) $-x, 1 - y, 1 - z$; (iii) $x - \frac{1}{2}, y, \frac{3}{2} - z$; (iv) $\frac{1}{2} - x, 1 - y, \frac{1}{2} + z$; (v) $\frac{1}{2} - x, -y, \frac{1}{2} + z$; (vi) $\frac{1}{2} - x, -y, z - \frac{1}{2}$; (vii) $\frac{1}{2} - x, 1 - y, z - \frac{1}{2}$.

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