



# Crystal structure of $\text{Ag}_3\text{Dy}_2(\text{NO}_3)_9$ and quantitative comparison to isotypic compounds

Wilhelm Klein\*

Technical University of Munich, School of Natural Sciences, Department of Chemistry, Lichtenbergstrasse 4, 85747 Garching, Germany. \*Correspondence e-mail: wilhelm.klein@tum.de

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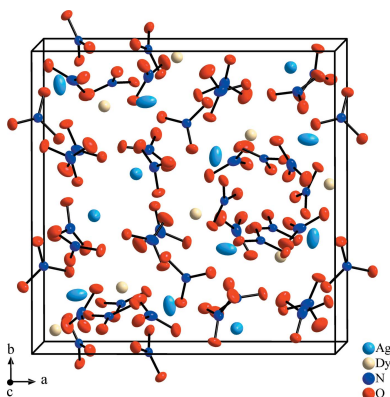
**Supporting information:** this article has supporting information at journals.iucr.org/e

Single crystals of  $\text{Ag}_3\text{Dy}_2(\text{NO}_3)_9$  (trisilver didysprosium nonantrate) were obtained from a mixture of  $\text{AgNO}_3$  and  $\text{Dy}(\text{NO}_3)_3 \cdot 5 \text{H}_2\text{O}$ . The new compound crystallizes in space group  $P4_132$  (No. 213) with  $a = 13.2004(4) \text{ \AA}$ ,  $V = 2300.2(2) \text{ \AA}^3$ ,  $Z = 4$ . The Ag and Dy cations are coordinated by five and six bidentate nitrate anions, respectively.  $\text{Ag}_3\text{Dy}_2(\text{NO}_3)_9$  is isostructural to several compounds that include alkali metals or ammonium and lanthanide cations, but silver and dysprosium are included for the first time and feature the smallest ion radii observed for this structure type to date. Crystal structures of isotypic compounds are compared.

## 1. Chemical context

Double nitrates of alkali metals and lanthanides of the composition  $A_3Ln_2(\text{NO}_3)_9$  have been found to crystallize in the chiral space groups  $P4_132$  and  $P4_332$  (Wickleder, 2002). After the first finding of  $\text{K}_3\text{Pr}_2(\text{NO}_3)_9$  by Carnall *et al.* (1973), this structure type has been observed in several compounds, to date with K (Carnall *et al.*, 1973; Vigdorichik *et al.*, 1992; Guillou *et al.*, 1995; Gobichon *et al.*, 1999), Rb (Vigdorichik *et al.*, 1990; Manek & Meyer, 1993a; Guillou *et al.*, 1996), and  $\text{NH}_4$  (Manek & Meyer, 1992) as 'A' cations for the lighter lanthanides La–Sm, and also detached examples with Na at the 'A' site (Stockhause & Meyer, 1997; Luo & Corruccini, 2004) and Eu (Manek & Meyer, 1992), Gd (Manek & Meyer, 1992; Luo & Corruccini, 2004), and even Bi (Goaz *et al.*, 2012) at the lanthanide site have been reported. The compounds are typically synthesized by dissolving the lanthanide oxides or nitrates in melts of the respective alkali metal or ammonium nitrates under anhydrous atmosphere, while lanthanum and cerium compounds have been crystallized from solutions in  $\text{H}_2\text{O}$  or  $\text{HNO}_3$  (Guillou *et al.*, 1995, 1996; Gobichon *et al.*, 1999). For the heavier lanthanides, usually another structure type with a slightly different composition, namely in an  $A/Ln$  ratio of 2:1 instead of 3:2, is observed under similar reaction conditions (Manek & Meyer, 1992, 1993a), and also for lithium, *e.g.* after the use of  $\text{LiNO}_3$  as a starting material, compounds with 2:1 ratio seem to be favoured (Manek & Meyer, 1993b).

In this work a new member of this group of compounds,  $\text{Ag}_3\text{Dy}_2(\text{NO}_3)_9$ , is presented, the first one containing Ag and Dy, which has been found to crystallize in the above-mentioned structure type.



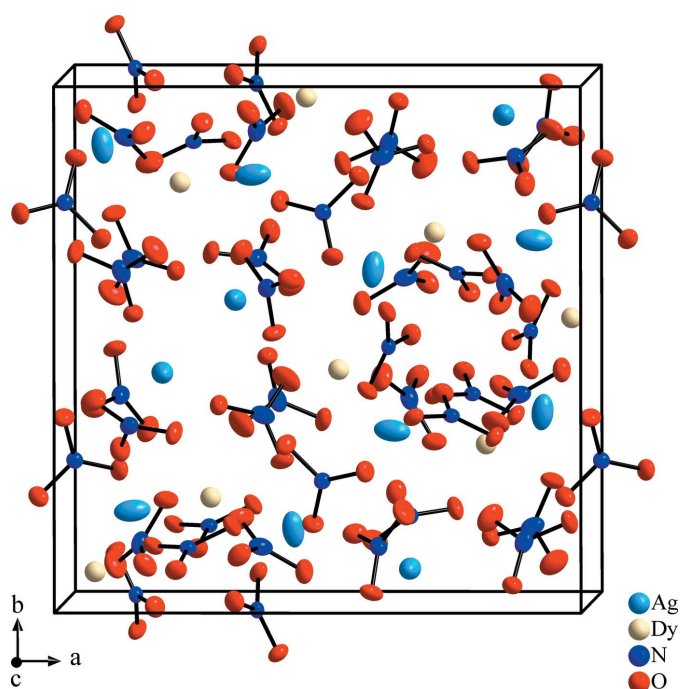
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## 2. Structural commentary

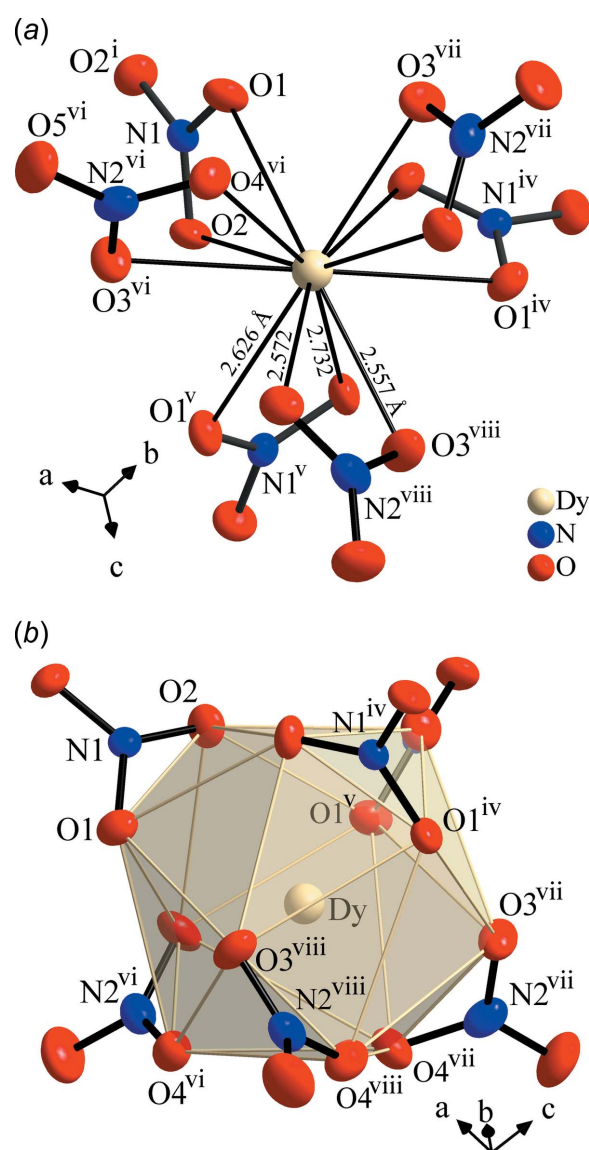
Similar to many related compounds, the title compound was obtained from a melt of nitrates, in this case silver nitrate and dysprosium nitrate pentahydrate. However, while for synthesis of related compounds, oxides are often used as lanthanide sources and the respective alkali metal nitrate or a eutectic combination of nitrates act as solvent as well as nitrate donor, in the present experimental setting the nitrates can be deployed in stoichiometric amounts. The crystals, which were found to be suitable for structure determination were obtained from a 2:1 mixture of Ag and Dy nitrates, *i.e.* a slight excess of  $\text{AgNO}_3$ , as described in the experimental section. The surplus Ag is present as remaining  $\text{AgNO}_3$  as well as elemental silver after partial thermal or light-induced decomposition. So far, no hint of another compound with a 2:1 composition of metals in the Ag/Dy system, as could be expected for smaller lanthanides similar to the alkali metal or ammonium systems (Manek & Meyer, 1992, 1993a), has been observed.

$\text{Ag}_3\text{Dy}_2(\text{NO}_3)_9$  (Fig. 1) crystallizes in space group  $P4_132$  with most atoms at general positions except for Ag, N1 and O1 at  $12d$  and Dy at  $8c$  Wyckoff positions. The asymmetric unit comprises one Ag, one Dy, two N, and five O atoms. The Dy atom, being located on a threefold axis, is coordinated by six bidentate nitrate anions with Dy–O distances of 2.557 (11)–2.732 (11) Å (see Fig. 2a), the surrounding oxygen atoms form a distorted icosahedron (Fig. 2b). The polyhedra are connected to neighbouring icosahedra *via* common vertices, and inside this polyhedron the Dy atom is slightly off-centre, shown by formation of the shortest Dy–O distances to O3

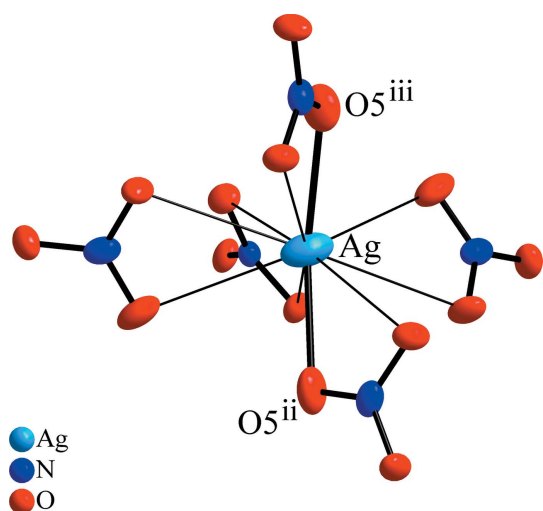


**Figure 1**  
Unit cell of  $\text{Ag}_3\text{Dy}_2(\text{NO}_3)_9$ , view along the  $c$  axis, atomic displacement ellipsoids are drawn with a probability of 60%.

and O4 as part of the same  $\text{NO}_3^-$  anion (the lower one in Fig. 2b), most probably driven by repulsion of next-neighbour Dy atoms. The silver atom is also coordinated by five nitrate ions in exclusively bidentate manner (Fig. 3). The Ag–O distances span quite a large range, so besides eight distances between 2.741 (11) and 3.004 (11) Å two relatively short distances of 2.383 (15) Å are found. These short bonds include oxygen atoms in almost opposite positions, which form an O–Ag–O angle of  $154.7(6)^\circ$ , indicating the preferred formation of  $\text{AgO}_2$  dumbbells even in an environment of quite rigid complex anions, for instance observed in  $\text{Ag}_4\text{SiO}_4$  (Klein & Jansen, 2008), in contrast to a more spherical ‘alkali metal-like’ coordination as in  $\text{Ag}_3\text{SbO}_4$  (distorted rock salt structure; Klein & Jansen, 2010). Consequently, the Ag atom has its



**Figure 2**  
Twelvefold coordination of the  $\text{Dy}^{3+}$  ion by six bidentate nitrate ions in  $\text{Ag}_3\text{Dy}_2(\text{NO}_3)_9$ : (a) view along the threefold symmetry axis; (b) distorted icosahedron around Dy. Atoms are drawn at the 60% probability level. [Symmetry codes: (i)  $z + \frac{1}{4}, -y + \frac{1}{4}, x - \frac{1}{4}$ ; (iv)  $x - \frac{1}{4}, z + \frac{1}{4}, -y + \frac{1}{4}$ ; (v)  $-y + \frac{1}{4}, x - \frac{1}{4}, z + \frac{1}{4}$ ; (vi)  $-y + \frac{1}{2}, -z, x - \frac{1}{2}$ ; (vii)  $x - \frac{1}{2}, -y + \frac{1}{2}, -z$ ; (viii)  $-z, x - \frac{1}{2}, -y + \frac{1}{2}$ .]


**Figure 3**

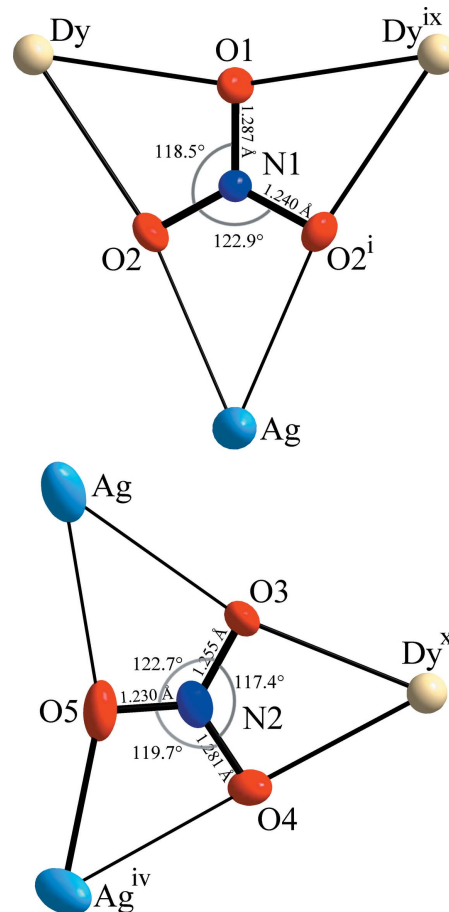
Coordination of the  $\text{Ag}^+$  cation by five bidentate nitrate anions. The shorter  $\text{Ag}-\text{O}$  bonds, which define the  $\text{AgO}_2$  dumbbell, are emphasized, displacement ellipsoids are drawn at the 60% probability level. [Symmetry codes: (ii)  $y, z, x$ ; (iii)  $x + \frac{1}{4}, -z + \frac{1}{4}, y - \frac{1}{4}$ ]

largest axis of the displacement ellipsoid perpendicular to the  $\text{AgO}_2$  dumbbell direction (see Fig. 3), which also represents the largest extension of an anisotropic parameter of all atoms in this structure (see supporting information,  $U_{22}$ ). The two independent nitrate ions are perfectly planar, with  $\text{O}-\text{N}-\text{O}$  angle sums of 360.00 and 359.79° around N1 and N2, respectively. Both the nitrate ions are situated between three bidentately coordinated metal atoms forming almost planar  $\text{AgDy}_2(\text{NO}_3)$  and  $\text{Ag}_2\text{Dy}(\text{NO}_3)$  units, respectively, as illustrated in Fig. 4. The longest  $\text{N}-\text{O}$  distances and the smallest  $\text{O}-\text{N}-\text{O}$  angles are found in the direction of coordinated Dy atoms, and in addition the  $\text{Ag}$  atom coordination, including a short  $\text{Ag}-\text{O}$  distance shows an  $\text{O}-\text{N}-\text{O}$  angle slightly below the mean value.

The appearance of this structure type for the combination  $\text{Ag}-\text{Dy}$  is somewhat remarkable. While silver as an atypical single-charged cation deforms its direct environment slightly to achieve a more convenient coordinative situation as explained above, dysprosium represents the heaviest lanthanide and, thus, the one with the smallest ionic radius observed in this structure type so far (Shannon, 1976), and a twelve-coordinate site seems to be unusual for this small lanthanide. This view is supported by the finding that compounds that include smaller lanthanide cations avoid to adopt this structure type in favour of another structure with a smaller coordination number and even a slightly different composition ( $A/Ln = 2:1$ ; Manek & Meyer, 1992, 1993a). Additionally, this might be confirmed by the ‘underbonding’ of the Dy cation, as the bond-valence sums (Brown & Altermatt, 1985) are calculated to be 2.51 valence units for the threefold positively charged ion, according to the parameters of Brese & O’Keeffe (1991).

The crystal structure has been quantitatively compared to isotopic structures by applying the program *compstru* (de la Flor *et al.*, 2016), available at the Bilbao Crystallographic

Server (Aroyo *et al.*, 2006). With  $\text{Ag}_3\text{Dy}_2(\text{NO}_3)_9$  as the reference structure, Table 1 lists the absolute distances between paired atoms as well as the arithmetic mean of the distance ( $d_{\text{av}}$ ) between paired atoms, the degree of lattice deviation ( $S$ ) and the measure of similarity ( $\Delta$ ). Generally, the low values for  $S$  and  $\Delta$  indicate a close relationship between all phases, including the trend to increasing numbers at larger differences of lattice parameters from Na to Rb compounds. The differences of  $d_{\text{av}}$ ,  $S$ , and  $\Delta$  are of course determined in a higher degree by the more differing radii of the (more frequent) alkali metal cations than by those of the more similar lanthanide ions. Significantly, in all cases the largest displacements between atom pairs are observed for O5, *i.e.* the closest  $\text{Ag}$ -coordinating O atom, confirming the special bonding situation for  $\text{Ag}$  including the above-mentioned  $\text{AgO}_2$  dumbbells. Consequently, the whole  $\text{NO}_3$  anion, of which O5 is a part, is shifted slightly more than the atoms of the other anion. The  $\text{Ag}$  atom is also affected, as indicated by


**Figure 4**

Planar surrounding of the two independent nitrate anions:  $\text{NO}_3(1)$  (upper) coordinating two Dy and one Ag, view perpendicular to the twofold symmetry axis through Ag, N1, and O1;  $\text{NO}_3(2)$  (lower) coordinating one Dy and two Ag, the short  $\text{Ag}-\text{O5}$  bond is drawn thicker than other  $\text{Ag}-\text{O}$  bonds. All atoms are shown at the 60% probability level. [Symmetry codes: (i)  $z + \frac{1}{4}, -y + \frac{1}{4}, x - \frac{1}{4}$ ; (iv)  $x - \frac{1}{4}, z + \frac{1}{4}, -y + \frac{1}{4}$ ; (ix)  $y + \frac{1}{4}, -x + \frac{1}{4}, z - \frac{1}{4}$ ; (x)  $x + \frac{1}{2}, -y + \frac{1}{2}, -z$ .]

**Table 1**

Structure comparison of  $\text{Ag}_3\text{Dy}_2(\text{NO}_3)_9$  with  $\text{Na}_3\text{Nd}_2(\text{NO}_3)_9^a$ ,  $\text{K}_3\text{Ce}_2(\text{NO}_3)_9^b$ ,  $\text{K}_3\text{Pr}_2(\text{NO}_3)_9^{c,g}$ ,  $\text{Rb}_3\text{Ce}_2(\text{NO}_3)_9^d$ ,  $\text{Rb}_3\text{Pr}_2(\text{NO}_3)_9^{e,g}$ , and  $\text{Rb}_3\text{Nd}_2(\text{NO}_3)_9^{f,g}$ , by using the program *Compstru* (de la Flor *et al.*, 2016).

Cubic lattice parameters ( $\text{\AA}$ ), absolute atomic displacements ( $\text{\AA}$ ), arithmetic mean displacements ( $d_{av}$ ;  $\text{\AA}$ ), degree of lattice distortion ( $S$ ), and measure of similarity ( $\Delta$ )<sup>h</sup>.

$A =$	Na	K	K	Rb	Rb	Rb
$Ln =$	Nd	Ce	Pr	Ce	Pr	Nd
$a$	13.1279	13.5975	13.52	13.8411	13.8091	13.759
$A$	0.0035	0.3157	0.3325	0.4725	0.4680	0.4729
$Ln$	0.0151	0.2318	0.2440	0.3670	0.3768	0.3885
N1	0.0261	0.1605	0.1997	0.1848	0.2296	0.2352
O1	0.0187	0.2072	0.2035	0.2576	0.2912	0.3080
O2	0.0170	0.1786	0.1821	0.2763	0.2582	0.2646
N2	0.0223	0.3350	0.3555	0.5195	0.4883	0.4907
O3	0.0577	0.3059	0.3072	0.4321	0.4278	0.4502
O4	0.0346	0.3302	0.3271	0.4027	0.4815	0.4732
O5	0.0577	0.4583	0.4839	0.6160	0.6490	0.6483
$d_{av}$	0.0320	0.2966	0.3080	0.4136	0.4280	0.4338
$S$	0.0032	0.0166	0.0135	0.0261	0.0249	0.0230
$\Delta$	0.003	0.032	0.033	0.044	0.046	0.046

Notes: (a) Stockhause & Meyer (1997); (b) Guillou *et al.* (1995); (c) Carnall *et al.* (1973); (d) Guillou *et al.* (1996); (e) Manek & Meyer (1993a); (f) Vigdorichik *et al.* (1990); (g)  $\text{K}_3\text{Pr}_2(\text{NO}_3)_9$ ,  $\text{Rb}_3\text{Pr}_2(\text{NO}_3)_9$ , and  $\text{Rb}_3\text{Nd}_2(\text{NO}_3)_9$  were originally described in  $P4_332$  and were transformed into  $P4_332$ ; (h) atom displacements are calculated by applying the lattice parameter of  $\text{Ag}_3\text{Dy}_2(\text{NO}_3)_9$  [ $a = 13.2004$  (4)  $\text{\AA}$ ] to the structure models of the listed compounds.

higher Ag—O displacements than those of the lanthanide cation pairs, while the coordination of the  $Ln$  cations remains similar (distortedly icosahedral, slightly off-centered), just accompanied by decreasing  $Ln$ —O distances with decreasing cation radii. An exception represents the, so far, only known Na structure, where the similarity as well as the relative displacements are about one order lower than for all other examples, indicating that the packing is distorted to a similar degree by the small Na cation as in the title compound by the Ag cation. However, the closest Ag—O distance is shorter than all Na—O distances in the related  $\text{Na}_3\text{Nd}_2(\text{NO}_3)_9$ .

### 3. Database survey

Several anhydrous rare-earth double nitrates of the composition  $A_3Ln_2(\text{NO}_3)_9$  have been investigated, mainly including larger lanthanide elements and alkali metals of medium size or ammonium cations, as listed in the *Chemical context*. Obviously, all of them seem to crystallize in the above-mentioned structure type, however, for some of them only the cubic lattice parameter is given. To date, detailed structural data are available for  $\text{K}_3\text{La}_2(\text{NO}_3)_9$  (Gobichon *et al.*, 1999),  $\text{K}_3\text{Ce}_2(\text{NO}_3)_9$  (Guillou *et al.*, 1995),  $\text{Rb}_3\text{Ce}_2(\text{NO}_3)_9$  (Guillou *et al.*, 1996),  $\text{K}_3\text{Pr}_2(\text{NO}_3)_9$  (Carnall *et al.*, 1973),  $\text{Rb}_3\text{Pr}_2(\text{NO}_3)_9$  (Manek & Meyer, 1993a),  $(\text{NH}_4)_3\text{Pr}_2(\text{NO}_3)_9$  (Manek & Meyer, 1992),  $\text{Na}_3\text{Nd}_2(\text{NO}_3)_9$  (Stockhause & Meyer, 1997),  $\text{K}_3\text{Nd}_2(\text{NO}_3)_9$  (Vigdorichik *et al.*, 1992),  $\text{Rb}_3\text{Nd}_2(\text{NO}_3)_9$  (Vigdorichik *et al.*, 1990), and  $\text{K}_3\text{Bi}_2(\text{NO}_3)_9$  (Goaz *et al.*, 2012).

**Table 2**

Experimental details.

Crystal data	
Chemical formula	$\text{Ag}_3\text{Dy}_2(\text{NO}_3)_9$
$M_r$	1206.70
Crystal system, space group	Cubic, $P4_332$
Temperature (K)	223
$a$ ( $\text{\AA}$ )	13.2004 (4)
$V$ ( $\text{\AA}^3$ )	2300.2 (2)
$Z$	4
Radiation type	Mo $K\alpha$
$\mu$ ( $\text{mm}^{-1}$ )	9.07
Crystal size (mm)	$0.4 \times 0.3 \times 0.1$
Data collection	
Diffractometer	Stoe StadiVari
Absorption correction	Empirical (using intensity measurements) ( <i>X-AREA</i> ; Stoe & Cie, 2015)
$T_{\min}$ , $T_{\max}$	0.001, 0.215
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	37326, 763, 715
$R_{\text{int}}$	0.159
$(\sin \theta/\lambda)_{\text{max}}$ ( $\text{\AA}^{-1}$ )	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$ , $wR(F^2)$ , $S$	0.045, 0.122, 1.18
No. of reflections	763
$\Delta\rho_{\text{max}}$ , $\Delta\rho_{\text{min}}$ ( $\text{e \AA}^{-3}$ )	2.01, $-1.07$
Absolute structure	Refined as an inversion twin
Absolute structure parameter	0.18 (6)

Computer programs: *X-AREA* (Stoe & Cie, 2015), *SHELXS97* and *SHELX* (Sheldrick, 2008), *SHELXL2014/7* (Sheldrick, 2015), *DIAMOND* (Brandenburg & Putz (2018) and *publCIF* (Westrip, 2010).

### 4. Synthesis and crystallization

An alumina crucible was charged with 359 mg  $\text{AgNO}_3$  (2.1 mmol; Merck; p.A.) and 495 mg  $\text{Dy}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$  (1.1 mmol; Alfa Aesar; 99.99%). The mixture was melted together at 573 K for 72 h in an Ar atmosphere, and was cooled down to 453 K at a rate of  $0.1 \text{ K min}^{-1}$ . Within an amorphous yellow–grey matrix, pale-yellow plates were found that were hygroscopic. EDX measurements on several crystals confirm the presence of Ag and Dy as the only elements heavier than oxygen. For the X-ray data collection, crystals were immersed into perfluoroalkyl ether, which covers and acts as glue on a glass tip during the data collection at low temperatures.

### 5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The structure was refined as an inversion twin.

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## supporting information

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## Crystal structure of $\text{Ag}_3\text{Dy}_2(\text{NO}_3)_9$ and quantitative comparison to isotopic compounds

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### Computing details

Data collection: *X-AREA* (Stoe & Cie, 2015); cell refinement: *X-AREA* (Stoe & Cie, 2015); data reduction: *X-AREA* (Stoe & Cie, 2015); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014/7* (Sheldrick, 2015); molecular graphics: *DIAMOND 3.2k* (Brandenburg & Putz (2018)); software used to prepare material for publication: *SHELX* (Sheldrick, 2008) and *pubCIF* (Westrip, 2010).

### Trisilver Didysprosium nonanitrate

#### Crystal data

$\text{Ag}_3\text{Dy}_2(\text{NO}_3)_9$   
 $M_r = 1206.70$   
 Cubic,  $P4_32$   
 $a = 13.2004$  (4) Å  
 $V = 2300.2$  (2) Å<sup>3</sup>  
 $Z = 4$   
 $F(000) = 2208$   
 $D_x = 3.485$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å  
 Cell parameters from 63387 reflections  
 $\theta = 2.2$ – $30.7^\circ$   
 $\mu = 9.07$  mm<sup>-1</sup>  
 $T = 223$  K  
 Plate, yellow  
 $0.4 \times 0.3 \times 0.1$  mm

#### Data collection

Stoe StadiVari  
 diffractometer  
 Radiation source: Genix 3D HF Mo  
 Graded multilayer mirror monochromator  
 Detector resolution: 5.81 pixels mm<sup>-1</sup>  
 $\omega$  scans  
 Absorption correction: empirical (using  
 intensity measurements)  
 (X-AREA; Stoe & Cie, 2015)

$T_{\min} = 0.001$ ,  $T_{\max} = 0.215$   
 37326 measured reflections  
 763 independent reflections  
 715 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.159$   
 $\theta_{\max} = 26.0^\circ$ ,  $\theta_{\min} = 2.2^\circ$   
 $h = -16 \rightarrow 16$   
 $k = -16 \rightarrow 16$   
 $l = -16 \rightarrow 16$

#### Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.045$   
 $wR(F^2) = 0.122$   
 $S = 1.18$   
 763 reflections  
 65 parameters  
 0 restraints  
 Primary atom site location: structure-invariant  
 direct methods

Secondary atom site location: difference Fourier  
 map  
 $w = 1/[\sigma^2(F_o^2) + (0.0538P)^2 + 51.0589P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 2.01$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -1.07$  e Å<sup>-3</sup>  
 Absolute structure: Refined as an inversion twin  
 Absolute structure parameter: 0.18 (6)

*Special details*

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

**Refinement.** Refined as a 2-component inversion twin.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Ag	0.42059 (11)	0.1250	0.17059 (11)	0.0411 (6)
Dy	0.03885 (6)	0.03885 (6)	0.03885 (6)	0.0233 (4)
N1	0.2541 (9)	0.1250	0.0041 (9)	0.014 (4)
O1	0.1852 (8)	0.1250	-0.0648 (8)	0.021 (3)
O2	0.2346 (8)	0.0854 (8)	0.0870 (8)	0.022 (2)
N2	0.3881 (11)	0.3676 (11)	0.1011 (10)	0.023 (3)
O3	0.4747 (8)	0.3304 (9)	0.0888 (9)	0.025 (2)
O4	0.3674 (8)	0.4493 (8)	0.0530 (9)	0.024 (2)
O5	0.3217 (10)	0.3253 (11)	0.1508 (9)	0.035 (3)

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Ag	0.0260 (7)	0.0713 (17)	0.0260 (7)	-0.0052 (7)	-0.0006 (8)	0.0052 (7)
Dy	0.0233 (4)	0.0233 (4)	0.0233 (4)	0.0002 (3)	0.0002 (3)	0.0002 (3)
N1	0.015 (5)	0.011 (8)	0.015 (5)	0.000 (4)	-0.001 (7)	0.000 (4)
O1	0.022 (5)	0.017 (7)	0.022 (5)	-0.005 (4)	-0.003 (6)	0.005 (4)
O2	0.026 (6)	0.022 (5)	0.018 (5)	0.001 (4)	0.000 (5)	0.007 (4)
N2	0.019 (7)	0.031 (8)	0.018 (7)	-0.006 (6)	0.001 (5)	-0.004 (6)
O3	0.015 (5)	0.030 (6)	0.029 (6)	0.005 (5)	0.005 (5)	0.008 (5)
O4	0.022 (6)	0.017 (5)	0.033 (7)	0.001 (5)	-0.001 (5)	0.000 (5)
O5	0.036 (7)	0.043 (8)	0.027 (6)	-0.014 (6)	0.008 (6)	0.007 (6)

*Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )*

Ag—O5 <sup>i</sup>	2.383 (15)	Dy—O1 <sup>i</sup>	2.626 (2)
Ag—O5 <sup>ii</sup>	2.383 (15)	Dy—O1 <sup>vii</sup>	2.626 (2)
Ag—O2 <sup>iii</sup>	2.741 (11)	Dy—O1	2.626 (2)
Ag—O2	2.741 (11)	Dy—O2	2.732 (11)
Ag—O4 <sup>ii</sup>	2.793 (11)	Dy—O2 <sup>vii</sup>	2.732 (11)
Ag—O4 <sup>i</sup>	2.793 (11)	Dy—O2 <sup>i</sup>	2.732 (11)
Ag—O5 <sup>iii</sup>	2.960 (15)	N1—O2	1.240 (14)
Ag—O5	2.960 (15)	N1—O2 <sup>iii</sup>	1.240 (14)
Ag—N2 <sup>ii</sup>	2.972 (14)	N1—O1	1.29 (2)
Ag—N2 <sup>i</sup>	2.972 (14)	O1—Dy <sup>viii</sup>	2.626 (2)
Ag—O3	3.004 (11)	N2—O5	1.230 (17)
Ag—O3 <sup>iii</sup>	3.004 (11)	N2—O3	1.255 (18)
Dy—O3 <sup>iv</sup>	2.557 (11)	N2—O4	1.281 (18)

Dy—O3 <sup>v</sup>	2.557 (11)	N2—Ag <sup>vii</sup>	2.972 (14)
Dy—O3 <sup>vi</sup>	2.557 (11)	O3—Dy <sup>ix</sup>	2.557 (11)
Dy—O4 <sup>vi</sup>	2.572 (11)	O4—Dy <sup>ix</sup>	2.572 (11)
Dy—O4 <sup>iv</sup>	2.572 (11)	O4—Ag <sup>vii</sup>	2.793 (11)
Dy—O4 <sup>v</sup>	2.572 (11)	O5—Ag <sup>vii</sup>	2.383 (15)
O5 <sup>i</sup> —Ag—O5 <sup>ii</sup>	154.7 (6)	O3 <sup>vi</sup> —Dy—O4 <sup>v</sup>	118.6 (3)
O5 <sup>i</sup> —Ag—O2 <sup>iii</sup>	120.7 (4)	O4 <sup>vi</sup> —Dy—O4 <sup>v</sup>	70.6 (4)
O5 <sup>ii</sup> —Ag—O2 <sup>iii</sup>	83.8 (4)	O4 <sup>iv</sup> —Dy—O4 <sup>v</sup>	70.6 (4)
O5 <sup>i</sup> —Ag—O2	83.8 (4)	O3 <sup>iv</sup> —Dy—O1 <sup>i</sup>	66.9 (4)
O5 <sup>ii</sup> —Ag—O2	120.7 (4)	O3 <sup>v</sup> —Dy—O1 <sup>i</sup>	67.4 (3)
O2 <sup>iii</sup> —Ag—O2	46.8 (4)	O3 <sup>vi</sup> —Dy—O1 <sup>i</sup>	168.5 (4)
O5 <sup>i</sup> —Ag—O4 <sup>ii</sup>	109.2 (4)	O4 <sup>vi</sup> —Dy—O1 <sup>i</sup>	139.4 (2)
O5 <sup>ii</sup> —Ag—O4 <sup>ii</sup>	48.8 (3)	O4 <sup>iv</sup> —Dy—O1 <sup>i</sup>	112.0 (4)
O2 <sup>iii</sup> —Ag—O4 <sup>ii</sup>	115.5 (3)	O4 <sup>v</sup> —Dy—O1 <sup>i</sup>	72.6 (3)
O2—Ag—O4 <sup>ii</sup>	162.3 (3)	O3 <sup>iv</sup> —Dy—O1 <sup>vii</sup>	168.5 (4)
O5 <sup>i</sup> —Ag—O4 <sup>i</sup>	48.8 (3)	O3 <sup>v</sup> —Dy—O1 <sup>vii</sup>	66.9 (4)
O5 <sup>ii</sup> —Ag—O4 <sup>i</sup>	109.2 (4)	O3 <sup>vi</sup> —Dy—O1 <sup>vii</sup>	67.4 (3)
O2 <sup>iii</sup> —Ag—O4 <sup>i</sup>	162.3 (3)	O4 <sup>vi</sup> —Dy—O1 <sup>vii</sup>	72.6 (3)
O2—Ag—O4 <sup>i</sup>	115.5 (3)	O4 <sup>iv</sup> —Dy—O1 <sup>vii</sup>	139.4 (2)
O4 <sup>ii</sup> —Ag—O4 <sup>i</sup>	82.1 (5)	O4 <sup>v</sup> —Dy—O1 <sup>vii</sup>	112.0 (4)
O5 <sup>i</sup> —Ag—O5 <sup>iii</sup>	116.8 (5)	O1 <sup>i</sup> —Dy—O1 <sup>vii</sup>	106.9 (3)
O5 <sup>ii</sup> —Ag—O5 <sup>iii</sup>	73.3 (5)	O3 <sup>iv</sup> —Dy—O1	67.4 (3)
O2 <sup>iii</sup> —Ag—O5 <sup>iii</sup>	74.9 (3)	O3 <sup>v</sup> —Dy—O1	168.5 (4)
O2—Ag—O5 <sup>iii</sup>	64.8 (3)	O3 <sup>vi</sup> —Dy—O1	66.9 (4)
O4 <sup>ii</sup> —Ag—O5 <sup>iii</sup>	116.5 (3)	O4 <sup>vi</sup> —Dy—O1	112.0 (4)
O4 <sup>i</sup> —Ag—O5 <sup>iii</sup>	96.8 (3)	O4 <sup>iv</sup> —Dy—O1	72.6 (3)
O5 <sup>i</sup> —Ag—O5	73.3 (5)	O4 <sup>v</sup> —Dy—O1	139.4 (2)
O5 <sup>ii</sup> —Ag—O5	116.8 (5)	O1 <sup>i</sup> —Dy—O1	106.9 (3)
O2 <sup>iii</sup> —Ag—O5	64.8 (3)	O1 <sup>vii</sup> —Dy—O1	106.9 (3)
O2—Ag—O5	74.9 (3)	O3 <sup>iv</sup> —Dy—O2	65.6 (4)
O4 <sup>ii</sup> —Ag—O5	96.8 (3)	O3 <sup>v</sup> —Dy—O2	122.7 (4)
O4 <sup>i</sup> —Ag—O5	116.5 (3)	O3 <sup>vi</sup> —Dy—O2	108.3 (3)
O5 <sup>iii</sup> —Ag—O5	136.1 (5)	O4 <sup>vi</sup> —Dy—O2	158.2 (3)
O5 <sup>i</sup> —Ag—N2 <sup>ii</sup>	133.8 (4)	O4 <sup>iv</sup> —Dy—O2	104.7 (3)
O5 <sup>ii</sup> —Ag—N2 <sup>ii</sup>	23.4 (4)	O4 <sup>v</sup> —Dy—O2	129.0 (4)
O2 <sup>iii</sup> —Ag—N2 <sup>ii</sup>	99.1 (3)	O1 <sup>i</sup> —Dy—O2	62.4 (2)
O2—Ag—N2 <sup>ii</sup>	142.2 (4)	O1 <sup>vii</sup> —Dy—O2	103.1 (4)
O4 <sup>ii</sup> —Ag—N2 <sup>ii</sup>	25.4 (3)	O1—Dy—O2	47.8 (4)
O4 <sup>i</sup> —Ag—N2 <sup>ii</sup>	97.1 (3)	O3 <sup>iv</sup> —Dy—O2 <sup>vii</sup>	122.7 (4)
O5 <sup>iii</sup> —Ag—N2 <sup>ii</sup>	94.4 (4)	O3 <sup>v</sup> —Dy—O2 <sup>vii</sup>	108.3 (3)
O5—Ag—N2 <sup>ii</sup>	108.1 (3)	O3 <sup>vi</sup> —Dy—O2 <sup>vii</sup>	65.6 (4)
O5 <sup>i</sup> —Ag—N2 <sup>i</sup>	23.4 (4)	O4 <sup>vi</sup> —Dy—O2 <sup>vii</sup>	104.7 (3)
O5 <sup>ii</sup> —Ag—N2 <sup>i</sup>	133.8 (4)	O4 <sup>iv</sup> —Dy—O2 <sup>vii</sup>	129.0 (4)
O2 <sup>iii</sup> —Ag—N2 <sup>i</sup>	142.2 (4)	O4 <sup>v</sup> —Dy—O2 <sup>vii</sup>	158.2 (3)
O2—Ag—N2 <sup>i</sup>	99.1 (3)	O1 <sup>i</sup> —Dy—O2 <sup>vii</sup>	103.1 (4)
O4 <sup>ii</sup> —Ag—N2 <sup>i</sup>	97.1 (3)	O1 <sup>vii</sup> —Dy—O2 <sup>vii</sup>	47.8 (4)
O4 <sup>i</sup> —Ag—N2 <sup>i</sup>	25.4 (3)	O1—Dy—O2 <sup>vii</sup>	62.4 (2)



O5 <sup>iii</sup> —Ag—N2 <sup>i</sup>	108.1 (3)	O2—Dy—O2 <sup>vii</sup>	60.9 (4)
O5—Ag—N2 <sup>i</sup>	94.4 (4)	O3 <sup>iv</sup> —Dy—O2 <sup>i</sup>	108.3 (3)
N2 <sup>ii</sup> —Ag—N2 <sup>i</sup>	117.8 (5)	O3 <sup>v</sup> —Dy—O2 <sup>i</sup>	65.6 (4)
O5 <sup>i</sup> —Ag—O3	107.3 (4)	O3 <sup>vi</sup> —Dy—O2 <sup>i</sup>	122.7 (4)
O5 <sup>ii</sup> —Ag—O3	75.0 (4)	O4 <sup>vi</sup> —Dy—O2 <sup>i</sup>	129.0 (4)
O2 <sup>iii</sup> —Ag—O3	66.5 (3)	O4 <sup>iv</sup> —Dy—O2 <sup>i</sup>	158.2 (3)
O2—Ag—O3	103.9 (3)	O4 <sup>v</sup> —Dy—O2 <sup>i</sup>	104.7 (3)
O4 <sup>ii</sup> —Ag—O3	61.3 (3)	O1 <sup>i</sup> —Dy—O2 <sup>i</sup>	47.8 (4)
O4 <sup>i</sup> —Ag—O3	127.5 (3)	O1 <sup>vii</sup> —Dy—O2 <sup>i</sup>	62.4 (2)
O5 <sup>iii</sup> —Ag—O3	132.0 (3)	O1—Dy—O2 <sup>i</sup>	103.1 (4)
O5—Ag—O3	42.9 (3)	O2—Dy—O2 <sup>i</sup>	60.9 (4)
N2 <sup>ii</sup> —Ag—O3	65.8 (3)	O2 <sup>vii</sup> —Dy—O2 <sup>i</sup>	60.9 (4)
N2 <sup>i</sup> —Ag—O3	119.9 (4)	O2—N1—O2 <sup>iii</sup>	122.9 (18)
O5 <sup>i</sup> —Ag—O3 <sup>iii</sup>	75.0 (4)	O2—N1—O1	118.5 (9)
O5 <sup>ii</sup> —Ag—O3 <sup>iii</sup>	107.3 (4)	O2 <sup>iii</sup> —N1—O1	118.5 (9)
O2 <sup>iii</sup> —Ag—O3 <sup>iii</sup>	103.9 (3)	N1—O1—Dy	98.7 (3)
O2—Ag—O3 <sup>iii</sup>	66.5 (3)	N1—O1—Dy <sup>viii</sup>	98.7 (3)
O4 <sup>ii</sup> —Ag—O3 <sup>iii</sup>	127.5 (3)	Dy—O1—Dy <sup>viii</sup>	162.6 (6)
O4 <sup>i</sup> —Ag—O3 <sup>iii</sup>	61.3 (3)	N1—O2—Dy	94.9 (9)
O5 <sup>iii</sup> —Ag—O3 <sup>iii</sup>	42.9 (3)	N1—O2—Ag	95.1 (9)
O5—Ag—O3 <sup>iii</sup>	132.0 (3)	Dy—O2—Ag	169.6 (5)
N2 <sup>ii</sup> —Ag—O3 <sup>iii</sup>	119.9 (4)	O5—N2—O3	122.7 (15)
N2 <sup>i</sup> —Ag—O3 <sup>iii</sup>	65.8 (3)	O5—N2—O4	119.7 (14)
O3—Ag—O3 <sup>iii</sup>	170.1 (5)	O3—N2—O4	117.4 (13)
O3 <sup>iv</sup> —Dy—O3 <sup>v</sup>	116.77 (16)	O5—N2—Ag <sup>vii</sup>	50.3 (9)
O3 <sup>iv</sup> —Dy—O3 <sup>vi</sup>	116.77 (16)	O3—N2—Ag <sup>vii</sup>	170.4 (11)
O3 <sup>v</sup> —Dy—O3 <sup>vi</sup>	116.77 (16)	O4—N2—Ag <sup>vii</sup>	69.4 (8)
O3 <sup>iv</sup> —Dy—O4 <sup>vi</sup>	118.6 (3)	N2—O3—Dy <sup>ix</sup>	97.0 (9)
O3 <sup>v</sup> —Dy—O4 <sup>vi</sup>	76.0 (4)	N2—O3—Ag	95.2 (9)
O3 <sup>vi</sup> —Dy—O4 <sup>vi</sup>	50.0 (3)	Dy <sup>ix</sup> —O3—Ag	157.7 (5)
O3 <sup>iv</sup> —Dy—O4 <sup>iv</sup>	50.0 (3)	N2—O4—Dy <sup>ix</sup>	95.6 (8)
O3 <sup>v</sup> —Dy—O4 <sup>iv</sup>	118.6 (3)	N2—O4—Ag <sup>vii</sup>	85.1 (8)
O3 <sup>vi</sup> —Dy—O4 <sup>iv</sup>	76.0 (4)	Dy <sup>ix</sup> —O4—Ag <sup>vii</sup>	170.9 (5)
O4 <sup>vi</sup> —Dy—O4 <sup>iv</sup>	70.6 (4)	N2—O5—Ag <sup>vii</sup>	106.3 (11)
O3 <sup>iv</sup> —Dy—O4 <sup>v</sup>	76.0 (4)	N2—O5—Ag	98.0 (11)
O3 <sup>v</sup> —Dy—O4 <sup>v</sup>	50.0 (3)	Ag <sup>vii</sup> —O5—Ag	148.6 (5)

Symmetry codes: (i)  $y, z, x$ ; (ii)  $x+1/4, -z+1/4, y-1/4$ ; (iii)  $z+1/4, -y+1/4, x-1/4$ ; (iv)  $-y+1/2, -z, x-1/2$ ; (v)  $-z, x-1/2, -y+1/2$ ; (vi)  $x-1/2, -y+1/2, -z$ ; (vii)  $z, x, y$ ; (viii)  $y+1/4, -x+1/4, z-1/4$ ; (ix)  $x+1/2, -y+1/2, -z$ .