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Structure of “bis(methoxymagnesium)diselenide”: A reagent for the introduction of selenium into organic molecules

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The reaction of magnesium and selenium in dry methanol has been reported to yield a reagent tentatively identified as bis(methoxymagnesium) diselenide. The dark red-brown solution obtained from this reaction yielded red crystals that crystallized in the monoclinic space group $C2/c$, $a = 17.391(4)$ Å, $b = 15.823(3)$ Å, $c = 21.626(4)$ Å, $\beta = 110.71(3)^\circ$, $V = 5566.5(19)$ Å^3, $Z = 4$, $R = 0.0350$, $wR_2 = 0.0850$ for 4930 reflections. X-ray crystallographic analysis of this reagent showed it to be dodecamethanol-tetramethoxy-di(μ_4-hydroxy)tetra(μ_3-methoxy)hexamagnesium hexaselenide, $Mg_6(\mu_4\text{-}OH)_2(\mu_3\text{-}OCH_3)_4(OCH_3)_4(CH_3OH)_{12}]Se_6 \cdot 2CH_3OH$ (**1**), not the bis(methoxymagnesium) diselenide as previously described. The structure of **1** is composed of six magnesium ions and six bridging oxygen-containing ligands in a face-sharing cubic arrangement.

KEY WORDS: methoxymagnesium selenide reagent; polyselenide; methoxymagnesium cluster.

Introduction

Many synthetic approaches for the introduction of diselenide into organic molecules are based on converting elemental selenium into a nucleophilic species, a transformation achieved either through the formation of various inorganic selenium compounds such as potassium selenocyanate[1] and potassium selenosulfate,[2] or through the formation of selenium compounds with a protective organic group such as benzyl selenol[3] or selenourea.[4] These methods often involve multiple steps to generate the selenium-containing product and subsequently yield the desired organic diselenide compounds. More recently, a number of synthetic approaches have employed elemental selenium to yield a metal-diselenide species. On further reaction with organic halides, these reagents yield the desired organic diselenides. Typically, these types of reactions involve treating elemental selenium with a Grignard reagent[5] or with alkali metals as shown in Fig. 1.[6,7] These approaches also use highly reactive alkali metals and require multiple steps to isolate and purify the desired diselenide.[6]

However, Günther reported the synthesis of a bis(methoxymagnesium) diselenide reagent that avoids the disadvantages of other diselenide synthesis.[8] This reagent is synthesized by treating elemental selenium with magnesium in dry methanol, and the resulting reagent adds a diselenide moiety directly to organic halides *via* nucleophilic displacement.[8] Thus, with this reagent, organic diselenides can be generated in a

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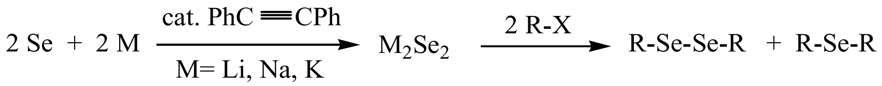

Fig. 1. Reaction of elemental selenium with alkali metals as a precursor to the formation of organic diselenides.

minimum number of steps with good yield (Fig. 2), while avoiding the use of reactive alkali metals. Based on the amount of gas evolved by the reaction of magnesium and methanol in the presence and absence of selenium, and on elemental analysis, this reagent was tentatively identified as a diselenide with the formula $(MeOMg)_2Se_2$. Since this reagent has broad applications in synthesizing organic diselenides and its composition was uncertain, we crystallized this compound to determine its structure. Instead of the reported bis(methoxymagnesium) diselenide, the X-ray structure revealed the compound to be a much more complex methoxymagnesium cluster cation with a hexaselenide anion.

Experimental

Preparation of $[Mg_6(\mu_4\text{-}OH)_2(\mu_3\text{-}OCH_3)_4(OCH_3)_4(CH_3OH)_{12}]Se_6 \cdot 2CH_3OH$ (1). The bis(methoxymagnesium) diselenide reagent (**1**) was synthesized as reported[8] except that the reaction was carried out under argon. Magnesium turnings (3.0 g) and a small crystal of iodine were added to a Schlenk flask and activated by heating briefly over a flame. The flask was flushed with argon, dry methanol (150 mL) was added, and the flask was fitted with a reflux condenser and a magnetic stirrer. After the yellow iodine color had been discharged, selenium powder (7.9 g) was added, and the mixture was stirred overnight until all solids dissolved. The resultant dark red-brown solution was cooled to −20°C for 24 hrs to yield red crystals of **1**.

Synthesis of dibenzyl diselenide. Dibenzyl diselenide was synthesized from **1** as reported by Günther[8] using benzyl chloride (6.3 g, 0.05 mole) and a methanolic solution of **1** (from 0.05 g-atom of selenium). After 10 min stirring, water (100 mL) and concentrated hydrochloric acid (5 mL) were added, the solution was cooled, and the black solid was collected by filtration. This black solid was then extracted with ethanol to yield a bright yellow solution which, upon solvent removal, gave a yellow crystalline product. The ^1H NMR of the yellow solid (in $CDCl_3$) was identical to that of commercially available dibenzyl diselenide.

X-ray structure determination of **1**. A single crystal of **1** was mounted on a glass fiber and cooled to −100°C in a cold nitrogen stream on a Rigaku AFC8 diffractometer. Data were collected with an area detector using the measurement parameters listed in Table 1. Systematic absences for $h\,k\,l\ (h+k \neq 2n)$ and $h\,0\,l\ (l \neq 2n)$ were only consistent with space groups *Cc* and *C2/c*. The centrosymmetric space group *C2/c* was chosen based on the average values of the normalized structure factors. The measured intensities were reduced to structure factor amplitudes and were corrected for absorption using REQAB[9] multiscan technique, background and Lorentz and

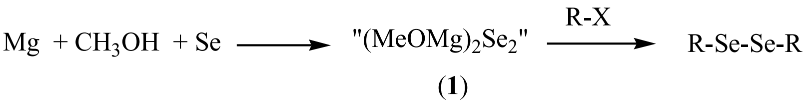

Fig. 2. General reactions of an organic halide with the "$(MeOMg)_2Se_2$" reagent (**1**) to afford organic diselenide compounds (R = benzylic, aliphatic groups; X = Br, Cl; range of yields = 55–85%).[8]

Table 1. Crystallographic data and structure refinement for **1**

Empirical formula	$C_{22}H_{82}O_{24}Mg_6Se_6$
Formula weight (amu)	1350.50
CCDC #	615359
Temperature	173(2) K
Wavelength, λ (Å)	0.71073
Crystal system,	Monoclinic
Space group	*C*2/*c*
a (Å)	17.391(4)
b (Å)	15.823(3)
c (Å)	21.626(4)
β (°)	110.71(3)
V (Å^3)	5566.5(19)
Z	4
Crystal size (mm)	0.31 × 0.26 × 0.24
D_{calc} (g cm^{-3})	1.611
Absorption coefficient, μ (mm^{-1})	4.077
θ range, °	2.76–25.10
Max. and min. transmission	0.4412 and 0.3646
Reflections collected	23234
Reflections unique (R_{int})	0.0421
Data/restraints/parameters	4930/0/281
Goodness of fit on F^2	1.068
$R_1{}^a$, wR_2 [$I > 2\sigma(I)$]	0.0350, 0.0850
$R_1{}^a$ $wR_2{}^b$ (all data)	0.0501, 0.0899

$^a R_1 = \sum \| F_o - |F_c \|) / \sum |F_o|$ for observed data ($I > 2\sigma(I)$).
$^b wR_2 = \{\sum[w(F_o{}^2 - F_c{}^2)^2]/[w(F_o{}^2)^2]\}^{1/2}$ for all data.

polarization effects using Crystal Clear.[10] Systematically absent reflections were removed and symmetry equivalent reflections were averaged to yield the set of unique data. The structure solution, refinement, and calculation of derived results were performed using direct methods with SHELXTL.[11] Subsequent least-squares refinement and difference Fourier calculations revealed the positions of non-hydrogen atoms. The quantity minimized by the least-squares program was $\sum w = (F_o{}^2 - F_c{}^2)^2$, where $w = \{[\sigma^2(F_o{}^2)] + (0.0460P)^2 + 7.7633P\}^{-1}$ and $P = (F_o{}^2 + 2F_c{}^2)/3]$. The analytical approximations to the scattering factors were used, and all structure factors were corrected for both real and imaginary components of anomalous dispersion. In the final cycle of least squares, independent anisotropic displacement factors were refined for the non-hydrogen atoms and the methyl hydrogen atoms were fixed in “idealized” positions with C–H = 0.96 Å. Their isotropic displacement parameters were set equal to 1.5 times U_{eq} of the attached carbon atom. Eight molecules of non-coordinating methanol were also found in the unit cell. Final refinement parameters for the structure of **1** are given in Table 1. The highest peak in the final Fourier difference map (1.08 Å^3) was located 1.19 Å from Se(3) and the lowest peak (-0.48 Å^3) was located at a distance of 0.79 Å from Se(3). The final analysis of variance between observed and calculated structure factors showed no apparent errors.

Results and discussion

In 1967, Günther reported that the reaction of activated magnesium and selenium in dry methanol yielded a reagent tentatively identified as bis(methoxymagnesium) diselenide (**1**).[8] Although use of air-sensitive techniques is not mentioned in the reported synthetic procedure, we repeated the synthesis under argon due to the reported instability of **1** to oxygen and water.[8] Cooling the resultant dark red solution to -20°C yielded red crystals of $[Mg_6(\mu_4\text{–}OH)_2(\mu_3\text{–}OCH_3)_4(OCH_3)_4(CH_3OH)_{12}]Se_6 \cdot 2CH_3OH$ (**1**), the structure of which was solved by X-ray analysis.

The asymmetric unit of **1** is shown in Fig. 3, and selected bond distances and angles are given in Table 2. The structure of the entire ion pair is shown in Fig. 4, and consists of a methoxymagnesium cluster cation, a hexaselenide ($Se_6{}^{2-}$) anion, and two non-coordinating methanol molecules. The methoxymagnesium cluster cation of **1** is unique in that it consists of six magnesium ions and six bridging oxygen ligands in a face-sharing cubic arrangement. In addition, different coordination environments are observed for Mg(1) as compared to Mg(2) and Mg(3) (Fig. 5). Despite the differences in coordination, each magnesium ion has Mg-O coordination in distorted octahedral geometry. To distinguish the methoxy and methanol ligands, hydrogen atoms bonded to oxygen atoms were located based on geometries of peaks found in electron density maps. Mg(1) is bound to two quadruply-bridging OH groups, two

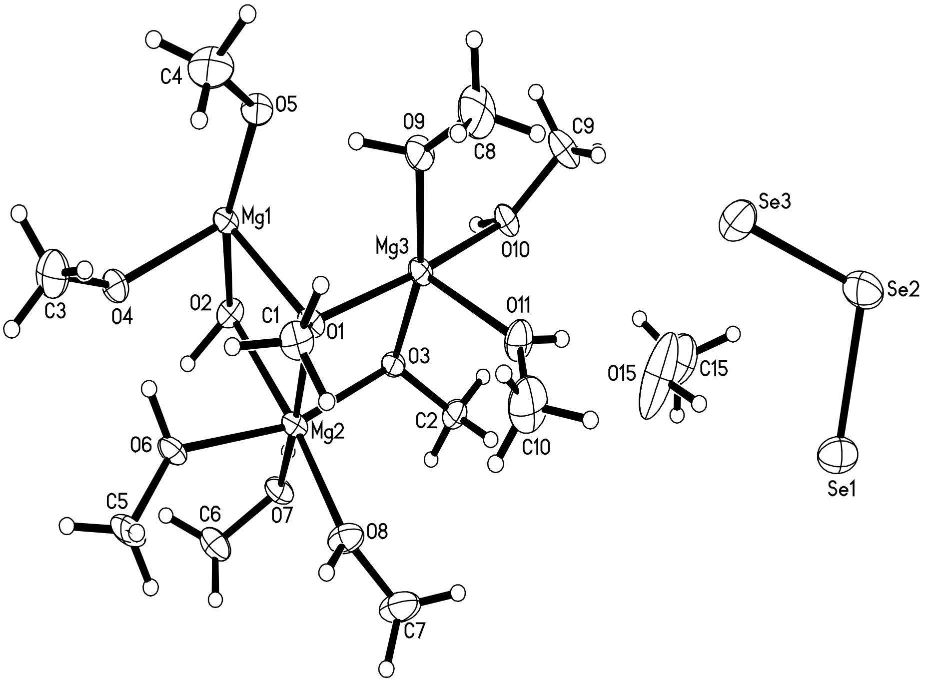

Fig. 3. The asymmetric unit of **1**. The 40% probability density surfaces are shown.

triply-bridging OCH_3 groups, and two terminal OCH_3 groups. Mg(2) and Mg(3) are each coordinated to three terminal CH_3OH groups, one quadruply-bridging OH group, and two triply-bridging OCH_3 groups. Mg(1) differs from the other two magnesium ions in the cation since it has no terminal CH_3OH coordination.

The methoxymagnesium cation of **1** lies around an inversion center at 0, 0.5, 0 as derived from the atomic coordinates. In **1**, the average bond distance of triply bridging methoxy groups, Mg(2)–OCH_3 and Mg(3)–OCH_3 is 2.05 Å, whereas that of Mg(1)–OCH_3 is slightly longer at 2.140(3) Å. The triply bridging Mg–OCH_3 distances observed for **1** are comparable to those in the few reported methoxymagnesium clusters: the average distance of triply-bridging Mg-OCH_3 bonds in tetrakis(μ_3-methoxo)tetrakis (acetylacetonato-O,O'-tetrakis(methanol-O)tetramagnesium reported by Kessler *et al.* is 2.07 Å,[12] in tetrakis(μ_3-methoxo)(2,2,6,6-tetramethylheptane-3,5-dionato)(methanol-O) magnesium this distance is 2.08 Å,[13] and in $Mg(OCH_3)_2 \cdot 3.5CH_3OH$ the distance of triply-bridging Mg–OCH_3 bonds vary from 2.05–2.14 Å.[14] Mg(1) is unique in its coordination to terminal methoxy groups, with an average terminal Mg(1)–OCH_3 bond distance of 2.03 Å. This bond distance is comparable to the 2.01–2.09 Å distances reported for terminal Mg–OCH_3 bonds in $Mg(OCH_3)_2 \cdot 3.5CH_3OH$.[14]

Two quadruply-bridging hydroxyl groups (containing O2 and O2A) lie across the inversion center with Mg-O bond distances from 2.12–2.24 Å. The average distance of the terminal Mg–O(H)CH_3 bonds is 2.06 Å, somewhat less than the 2.15 Å[12] and 2.14 Å[13] average bond distances for terminal Mg–O(H)CH_3 bonds reported for similar magnesium clusters. The Mg-Mg bond distances in **1** range from 3.05 to 3.19 Å.

The structure of the hexaselenide (Se_6^{2-}) anion in **1** has a helical conformation similar to that reported for polyselenide compounds with organic counterions.[15,16] The average Se-Se bond

Table 2. Selected bond lengths (Å) and angles (°) for **1**

Se(2)–Se(3)	2.3111(8)	Se(1)–Se(2)	2.3167(8)
Mg(1)–O(5)	2.024(2)	Mg(1)–O(4)	2.029(2)
Mg(1)–O(3)#1	2.117(2)	Mg(1)–O(2)	2.122(3)
Mg(1)–O(2)#1	2.130(2)	Mg(1)–O(1)	2.138(2)
Mg(1)–Mg(3)#1	3.1481(15)	Mg(1)–Mg(3)	3.1665(15)
Mg(1)–Mg(2)#1	3.1771(19)	Mg(1)–Mg(2)	3.1915(16)
Mg(1)–Mg(1)#1	3.398(2)	Mg(2)–Mg(3)	3.0552(15)
Mg(2)–Mg(1)#1	3.1771(19)	Se(3)–Se(3)#2	2.3657(13)
Mg(3)–O(2)#1	2.188(2)	Mg(3)–Mg(1)#1	3.1481(15)
O(1)–Mg(2)	2.053(2)	O(1)–Mg(3)	2.051(2)
O(2)–Mg(1)#1	2.130(2)	O(2)–Mg(3)#1	2.188(2)
O(2)–Mg(2)	2.238(3)	O(3)–Mg(2)	2.050(2)
O(3)–Mg(3)	2.054(2)	O(3)–Mg(1)#1	2.117(2)
O(6)–Mg(2)	2.066(2)	O(7)–Mg(2)	2.063(3)
O(8)–Mg(2)	2.050(3)	O(9)–Mg(3)	2.071(3)
O(10)–Mg(3)	2.071(2)	O(11)–Mg(3)	2.035(3)
Se(3)–Se(1)–Se(2)	110.07(2)	O(5)–Mg(1)–O(4)	110.73(10)
O(5)–Mg(1)–O(3)#1	94.40(10)	O(4)–Mg(1)–O(3)#1	95.56(9)
O(5)–Mg(1)–O(2)	161.41(10)	O(4)–Mg(1)–O(2)	87.86(10)
O(3)#1–Mg(1)–O(2)	83.06(9)	O(5)–Mg(1)–O(2)#1	87.48(10)
O(4)–Mg(1)–O(2)#1	161.78(11)	O(3)#1–Mg(1)–O(2)#1	83.17(9)
O(2)–Mg(1)–O(2)#1	73.93(10)	O(5)–Mg(1)–O(1)	95.55(10)
O(4)–Mg(1)–O(1)	94.87(10)	O(3)#1–Mg(1)–O(1)	162.02(9)
O(2)–Mg(1)–O(1)	82.73(9)	O(2)#1–Mg(1)–O(1)	82.34(9)
O(5)–Mg(1)–Mg(3)#1	134.27(8)	O(4)–Mg(1)–Mg(3)#1	85.07(7)
O(3)#1–Mg(1)–Mg(3)#1	40.25(6)	O(2)–Mg(1)–Mg(3)#1	43.90(7)
O(2)#1–Mg(1)–Mg(3)#1	82.29(7)	O(1)–Mg(1)–Mg(3)#1	126.63(8)
O(5)–Mg(1)–Mg(3)	84.93(7)	O(4)–Mg(1)–Mg(3)	134.39(8)
O(3)#1–Mg(1)–Mg(3)	126.71(7)	O(2)–Mg(1)–Mg(3)	81.95(7)
O(2)#1–Mg(1)–Mg(3)	43.55(6)	O(1)–Mg(1)–Mg(3)	39.86(6)
Mg(3)#1–Mg(1)–Mg(3)	114.90(4)	Mg(3)#1–Mg(1)–Mg(1)#1	57.71(4)
Mg(3)–Mg(1)–Mg(1)#1	57.19(3)	Mg(2)#1–Mg(1)–Mg(1)#1	57.96(4)
Mg(2)–Mg(1)–Mg(1)#1	57.55(4)	C(1)–O(1)–Mg(2)	121.1(2)
C(1)–O(1)–Mg(3)	124.49(19)	Mg(2)–O(1)–Mg(3)	96.25(10)
C(1)–O(1)–Mg(1)	112.69(19)	Mg(2)–O(1)–Mg(1)	99.18(9)
Mg(3)–O(1)–Mg(1)	98.20(10)	Mg(1)–O(2)–Mg(1)#1	106.07(10)
Mg(1)–O(2)–Mg(3)#1	93.84(10)	Mg(1)#1–O(2)–Mg(3)#1	94.32(9)
Mg(1)–O(2)–Mg(2)	94.07(9)	Mg(1)#1–O(2)–Mg(2)	93.29(9)
Mg(3)#1–O(2)–Mg(2)	167.08(12)	C(2)–O(3)–Mg(2)	122.48(19)
C(2)–O(3)–Mg(3)	120.84(18)	Mg(2)–O(3)–Mg(3)	96.22(9)

Symmetry transformations used to generate atoms #1–x, –y–1, –z #2 –x, –y, –z + 1/2.

distance of 2.331 Å in **1** is comparable to the 2.3351 Å Se-Se distance found in $[NMe_4]_2Se_6$.[17] One characteristic feature of polychalcogenide Se_x^{2-} anions is that the two outer Se-Se bonds are shorter than the innermost Se-Se bond, likely due to $p_\pi - d_\pi$ bonding.[17] Accordingly, the central Se(3)–Se(3A) bond length of 2.3657(13) Å is slightly longer the Se(1)–Se(2) and Se(1)–Se(3) bond distances (2.3111(8) and 2.3167(8) Å, respectively) in **1**. It is interesting to note the Se(2)–Se(3)–Se(3A) bond angle of 105.66(3)° is similar to the bond angles found for known Se_5^{2-} complexes (104.5°–106.6°),[17] but smaller than the corresponding bond angle for the Se_6^{2-} moiety in $[Me_4N]_2Se_6$ (110.1°). However, the bond angle of 110.05(3)° between Se(1)–Se(2)–Se(3) atoms is comparable to that seen for $[Me_4N]_2Se_6$ (109.3°).[17]

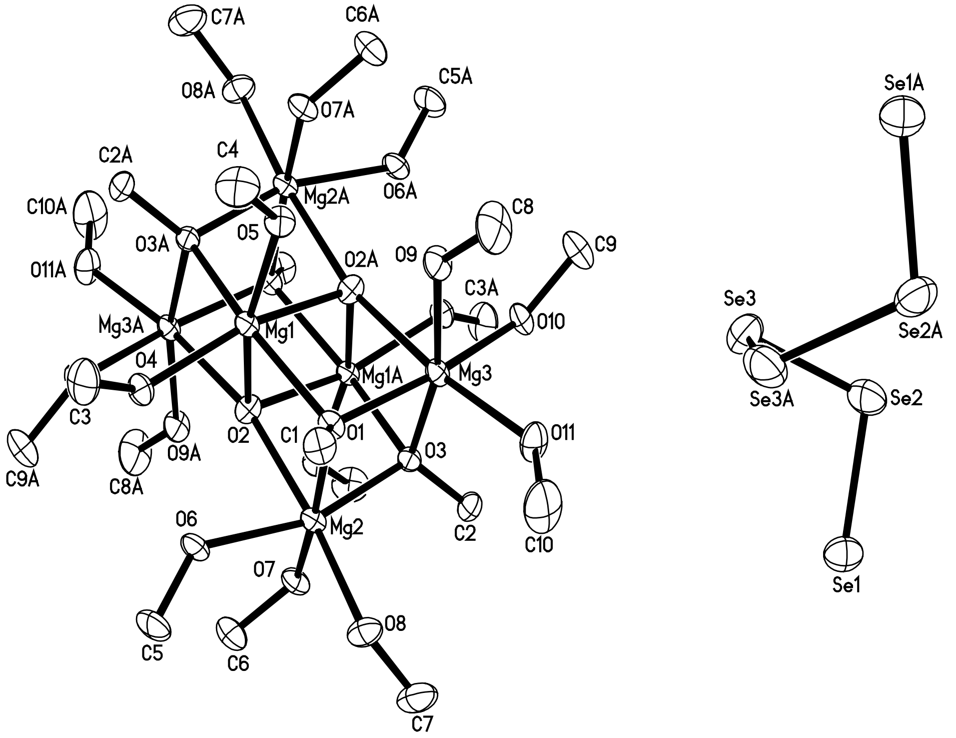

Fig. 4. Structure of **1**. Hydrogen atoms and non-coordinating methanol molecules have been removed for clarity. The 40% probability density surfaces are shown.

The crystal packing of **1** viewed along the *c*-axis is depicted in Fig. 6. The cluster cation, selenide anion, and the methanol of crystallization are connected by three hydrogen bonds: between Se(1) and O(8) from a terminal methanol group (Se-O distance 3.264 Å), Se(1) and O(15) with the non-coordinating methanol molecule (Se-O distance 3.310 Å), and O(15) and O(11) of a coordinating methanol molecule (O-O distance 2.592 Å). When viewed along the *c*-axis, the hexaselenide anions are seen along the four corners of the unit cell at coordinates ($a = 0$, $b = 0$), (0,1), (1,0), and (1,1) and at the middle of the cell ($a = 1/2$, $b = 1/2$). The methoxymagnesium cluster cations are stacked along the center of each side of the unit cell at coordinates (1/2, 0), (0, 1/2), (1, 1/2), and (1/2, 1/2).

Günther likely identified the reagent **1** as a diselenide because it is used to introduce diselenides into organic halides. Although the calculated elemental analysis values for Günther's proposed $(CH_3OMg)_2Se_2 \cdot 4CH_3OH$ formula and the actual structure of **1** are similar, neither correspond exactly to the questionable elemental analysis data reported in his 1967 paper. Despite an excess of selenium found in the elemental analysis, Günther assumed the reagent to be a diselenide, and explained that the higher value of selenium found during analysis was likely due to the formation of triselenide chains in the reagent.[8] Despite the inaccuracy of the reported elemental analysis data, we successfully repeated the synthesis of dibenzyl diselenide from benzyl chloride and **1** according to Günther's reported procedure.

By measuring the amount of gas evolved from the reaction mixture, Günther arrived at the stoichiometry of three molecules of

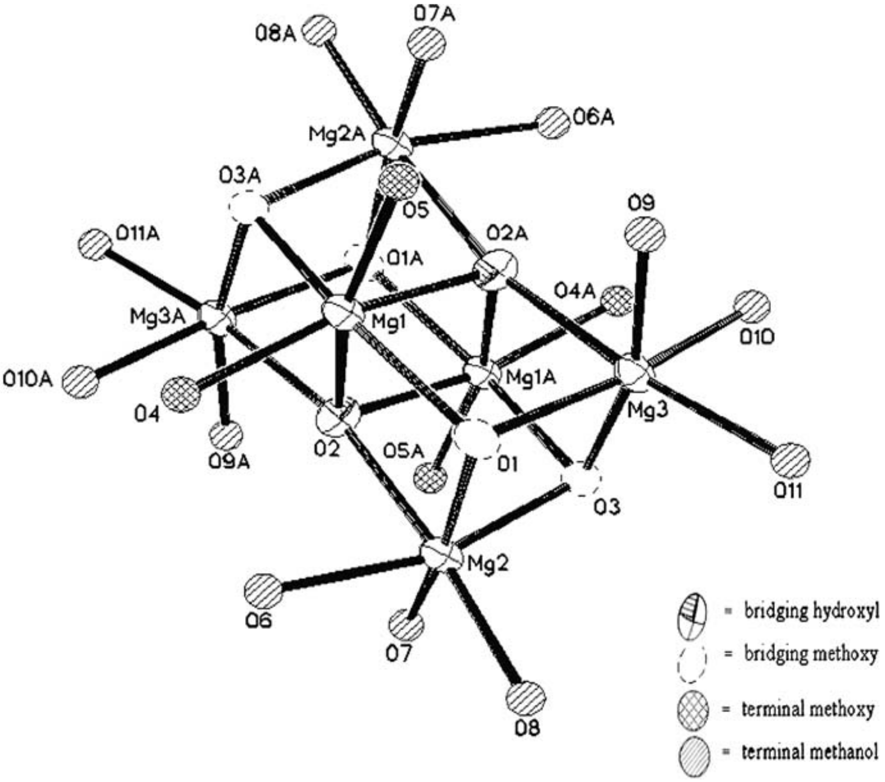

Fig. 5. Structure of the hexamagnesium cluster in **1**, showing the varied magnesium ion coordination.

methanol reacting with two atoms each of magnesium and selenium. As he states, this stoichiometry neither satisfies the simple valence of the elements involved in synthesis of the reagent nor corresponds to his proposed formula for **1**.[8] These results, however, do correspond well to the actual structure of **1**, $[Mg_6(\mu_4–OH)_2(\mu_3–OCH_3)_4(OCH_3)_4(CH_3OH)_{12}]Se_6 \cdot 2CH_3OH$. From this formula, four equivalents of hydrogen (to form the eight methoxide ligands) and one equivalent of ethane (to form the two hydroxide ligands) would be generated during the reaction, requiring ten equivalents of methanol for every six of magnesium or selenium. This 3.3 to 2 ratio found for the formation of **1** is much closer to the reported experimental results than the 1:1 ratio expected for the formation of $(CH_3OMg)_2Se_2$. Thus, upon structural analysis, our reported structure of **1** reveals a much greater degree of complexity than the $(CH_3OMg)_2Se_2$ structure originally proposed by Günther.

Conclusion

Günther reported the reaction of magnesium and selenium in refluxing dry methanol to yield a reagent tentatively identified as bis(methoxymagnesium) diselenide. X-ray crystallographic analysis of this reagent instead showed it to be $[Mg_6(\mu_4–OH)_2(\mu_3–OCH_3)_4(OCH_3)_4(CH_3OH)_{12}]Se_6 \cdot 2CH_3OH$ (**1**). The crystal structure of **1** reveals the compound to be a hexaselenide anion with a cationic methoxymagnesium cluster, and our reported structure better agrees with previous characterization than the structure reported by Günther. In **1**, the structure of the cationic cluster is unique, having a face-sharing cubic

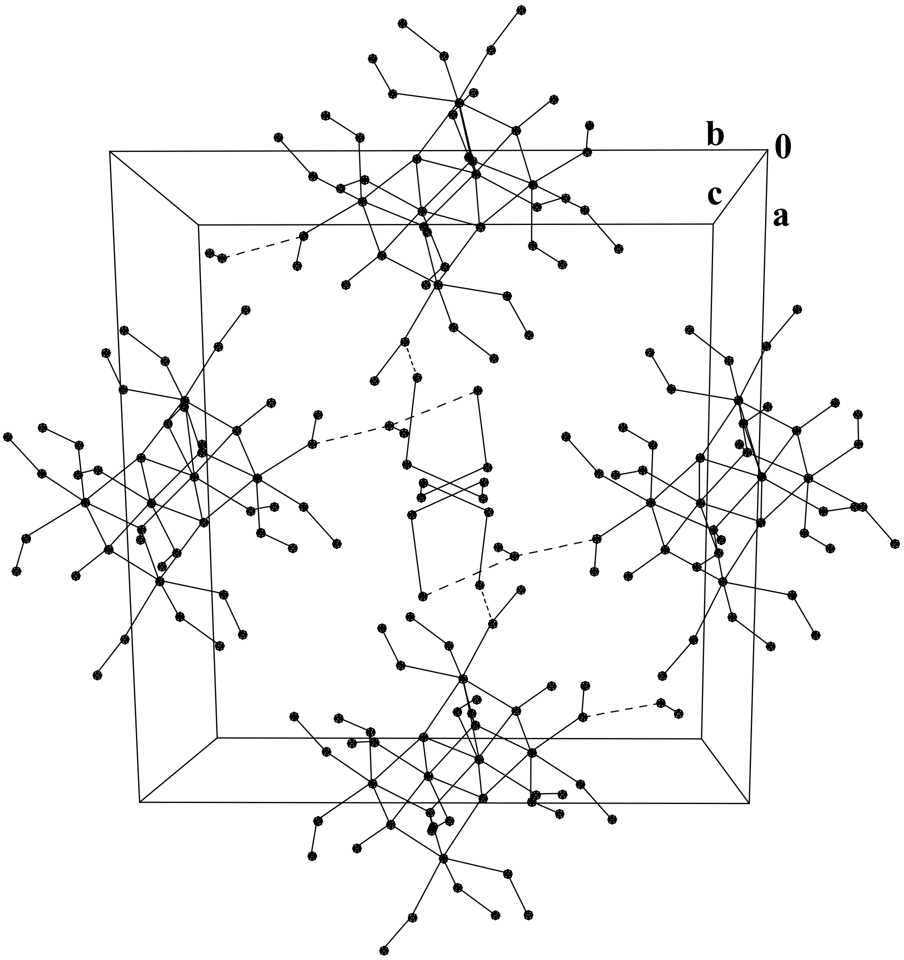

Fig. 6. Packing diagram of **1** viewed along the *c*-axis of the unit cell. Hydrogen atoms are omitted for clarity. Dotted lines show hydrogen bonding between Se(1)–O(15), Se(1)–O(8) and O(11)–O(15).

cation with varied coordination to magnesium ions. The hexaselenide anion has the expected helical conformation. This reagent can be used to synthesize many types of organic diselenides from the corresponding organic halides in good yields.

Supplementary material: Additional data of the X-ray structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publications, CCDC number 615359. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, United Kingdom (fax: +44–1223–336033, email: deposit@ccdc.cam.ac.uk).

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