Crystal Structure of [BeI₂(OEt₂)₂] (OEt₂ = diethyl ether)

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The crystal structure properties of [BeI₂(OEt₂)₂] (OEt₂ = diethyl ether) are: monoclinic, space group *P*2₁/*n* with *a* = 8.7737(18) Å, *b* = 12.596(3) Å, *c* = 26.086(5) Å, β = 93.36(3)°, *V* = 2877.9(10) Å³, *Z* = 8, *R*₁ = 0.0316, *wR*₂ = 0.0640 for 7240 reflections (*I* > 2σ(*I*)). The compound crystallizes with two crystallographically independent molecules in the asymmetric unit and contains the first structurally characterized examples of Be-I bonds in a molecular compound.

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The inorganic and organometallic chemistry of beryllium is poorly developed, mainly because of its high toxicity. A greater understanding of the structural chemistry of beryllium halide complexes would allow chemists to expand the use of such species in, for example, salt elimination reactions. A survey of the Cambridge Crystallographic Database¹ revealed that there are only 60 examples of structurally characterized compounds containing Be-halide bonds and no examples of Be-I bonded

complexes. In this paper we describe the synthesis of the previously unreported compound [BeI₂(OEt₂)₂] (OEt₂ = diethyl ether) and its crystal structure determination.

The compound was prepared by treating a suspension of beryllium metal (0.1 g, 1.1 mmol) with an excess of diiodine (3.8 g, 1.5 mmol) in diethyl ether (50 cm³) at 20°C, followed by stirring for 12 h. Subsequent removal of volatiles *in vacuo* and washing with hexane (40 cm³) yielded the title complex (4.4 g,

Fig. 1 Chemical structure of [BeI₂(OEt₂)₂].

Table 1 Crystal and experimental data

Formula: C ₈ H ₂₀ BeI ₂ O ₂	
Formula weight = 411.05	
Crystal system: monoclinic	
Space group: <i>P</i> 2 ₁ / <i>n</i>	<i>Z</i> = 8
<i>a</i> = 8.7737(18) Å	
<i>b</i> = 12.596(3) Å	β = 93.36(3)°
<i>c</i> = 26.086(5) Å	
<i>V</i> = 2877.9(10) Å ³	
<i>D</i> _x = 1.897 g/cm ³	
<i>T</i> = 150 K	
No. of reflections used = 8299	
2θ _{max} = 60.0° with Mo <i>K</i> _α	
<i>R</i> = 0.032	
(Δσ) _{max} = 0.002	
(Δρ) _{max} = 1.06 e Å ⁻³	
(Δρ) _{min} = -1.18 e Å ⁻³	
Measurement: Enraf-Nonius Kappa CCD	
Program system: Enraf-Nonius Collect	
Structure determination: SHELXL-97	
Refinement: full matrix	

Table 2 Atomic coordinates (× 10⁴) and equivalent isotropic thermal parameters *U*(eq) (Å² × 10³) for non-hydrogen atoms

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> (eq)
Be(1)	8047(4)	2963(3)	6498(1)	20(1)
Be(2)	8555(4)	2216(3)	3898(2)	23(1)
I(1)	6930(1)	4635(1)	6723(1)	28(1)
I(2)	9484(1)	3127(1)	5746(1)	29(1)
I(3)	6689(1)	1098(1)	4278(1)	37(1)
I(4)	9115(1)	1644(1)	3045(1)	30(1)
O(1)	9149(2)	2501(2)	6982(1)	19(1)
O(2)	6677(2)	2066(2)	6403(1)	22(1)
O(3)	10166(2)	2256(2)	4257(1)	22(1)
O(4)	7927(2)	3453(2)	3882(1)	22(1)
C(1)	9701(3)	3092(2)	7437(1)	24(1)
C(2)	8817(4)	2822(3)	7895(1)	33(1)
C(3)	9829(3)	1446(2)	6936(1)	26(1)
C(4)	11508(4)	1492(3)	6841(1)	33(1)
C(5)	5984(4)	1736(3)	5900(1)	29(1)
C(6)	4812(5)	2512(4)	5691(2)	53(1)
C(7)	5698(4)	1888(3)	6832(1)	30(1)
C(8)	5584(4)	727(3)	6963(2)	43(1)
C(9)	11383(4)	2954(3)	4090(1)	32(1)
C(10)	12020(4)	3662(3)	4512(1)	41(1)
C(11)	10730(4)	1389(3)	4595(1)	32(1)
C(12)	11325(7)	472(4)	4310(2)	74(2)
C(13)	7872(4)	4052(3)	4365(1)	27(1)
C(14)	6277(4)	4240(3)	4522(2)	44(1)
C(15)	7119(4)	3953(3)	3439(1)	30(1)
C(16)	7937(6)	4907(4)	3264(2)	59(1)

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Fig. 2 Molecular structure of $[\text{BeI}_2(\text{OEt}_2)_2]$ (30% probability ellipsoids, hydrogen atoms omitted).

Table 3 Selected bond distances (Å) and angles (°)

Bond distance			
I(1)-Be(1)	2.411(4)	O(1)-Be(1)	1.650(4)
I(2)-Be(1)	2.402(4)	O(2)-Be(1)	1.657(4)
Bond angles			
O(1)-Be(1)-O(2)	105.2(2)	O(1)-Be(1)-I(2)	110.05(19)
O(2)-Be(1)-I(2)	110.32(19)	O(1)-Be(1)-I(1)	110.43(19)
O(2)-Be(1)-I(1)	109.29(19)	I(2)-Be(1)-I(1)	111.40(15)

97 %). It was recrystallized as colorless crystals from diethyl ether.

The crystal structure of $[\text{BeI}_2(\text{OEt}_2)_2]$ was solved by direct methods and refined by full-matrix least squares using the program SHELXL-97.² All non-hydrogen atoms were refined anisotropically whilst hydrogen atoms were placed at the calculated positions (riding model). The asymmetric unit of the

crystal structure contains two crystallographically independent molecules with no significant geometric differences between them and thus the molecular structure of only one is depicted in Fig. 2. It is of note that the crystal structures of $[\text{BeCl}_2(\text{OEt}_2)_2]$ ³ and $[\text{BeBr}_2(\text{OEt}_2)_2]$ ⁴ have different cell parameters to each other and those of $[\text{BeI}_2(\text{OEt}_2)_2]$. However, as is the case for the previously reported complexes, the Be centers in the structure of $[\text{BeI}_2(\text{OEt}_2)_2]$ have distorted tetrahedral geometries. There have been no previously structurally characterized examples of Be-I bonds in molecular compounds and thus no comparisons can be drawn with the mean Be-I interaction of 2.411 Å in the structure of $[\text{BeI}_2(\text{OEt}_2)_2]$.

Supplementary Material

Supplementary crystallographic data can be obtained upon request from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK, or [www: http://www.ccdc.cam.ac.uk](http://www.ccdc.cam.ac.uk), quoting CCDC 636942.

Acknowledgements

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