The Molecular Structure of $[InBr_2\{N(Sime_3)_2\}_2][Li(DME)_3]$

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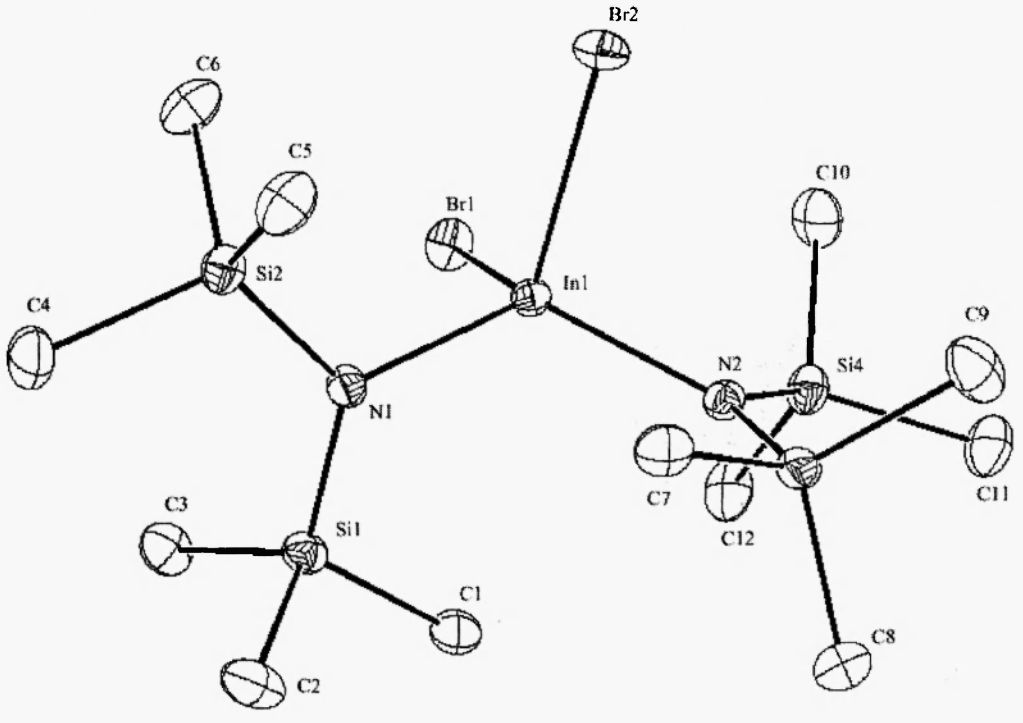
Figure 1. Molecular structure of the anion of $[InBr_2\{N(SiMe_3)_2\}_2][Li(DME)_3]$. Selected bond lengths (Å) and angles(°): In(1)-N(2) 2.085(5), In(1)-N(1) 2.097(4), In(1)-Br(1) 2.5585(8), In(1)-Br(2) 2.5663(7), Si(1)-N(1) 1.710(5), Si(2)-N(1) 1.722(5), Si(3)-N(2) 1.732(5), Si(4)-N(2) 1.712(5), N(2)-In(1)-N(1) 122.52(19), N(2)-In(1)-Br(1) 112.72(13), N(1)-In(1)-Br(1) 101.61(13), N(2)-In(1)-Br(2) 101.25(12), N(1)-In(1)-Br(2) 112.25(12), Br(1)-In(1)-Br(2) 105.71(3), Si(1)-N(1)-Si(2) 123.7(3), Si(1)-N(1)-In(1) 117.3(2), Si(2)-N(1)-In(1) 117.5(2), Si(4)-N(2)-Si(3) 124.9(3), Si(4)-N(2)-In(1) 117.9(3), Si(3)-N(2)-In(1) 116.4(2).



COMMENT

The molecule was found to be a salt with no interaction between the anion and cation. The metal centre of the anion has a distorted tetrahedral coordination environment with In-N and In-Br bond lengths in the normal range [1]. The In-N bond lengths are, however, slightly shorter than those in the closely related salt, $[InF\{N(SiMe_3)_2\}_3]Cs$ (2.126 Å avge.) /2/, presumably to the greater degree of steric crowding about the In centre in that compound. The geometry of the distorted octahedral lithium centre in the $[Li(DME)_3]^+$ cation is similar to that previously observed for this moiety, for example in $[AlH\{N(CH_2Ph)_2\}_3][Li(DME)_3]$ /3/.

EXPERIMENTAL

Preparation of $[InBr_2\{N(SiMe_3)_2\}_2][Li(DME)_3]$:

A solution of $LiN(SiMe_3)_2$ (0.94g, 5.6 mmol) in THF (20 ml) was added to a solution of $InBr_3$ (1.00g, 2.8 mmol) in THF (20 ml) at 25°C. A dark brown solution resulted which was stirred for 1 hour, volatiles removed *in vacuo* and the residue extracted with DME (30 ml). This extract was filtered and the filtrate placed at –35°C overnight to yield the title compound as colourless crystals (1.55g, 61% yield), m.p. 94 - 96°C, ^1H NMR (250MHz, C_6D_6) δ 0.73 (s, 36H, $SiMe_3$), 3.08 (s, 18H, OMe), 3.11 (s, 12H, OCH_2); ^{13}C NMR (100.6 MHz, C_6D_6) δ 6.4 (SiMe), 58.8 (OMe), 70.6 (OCH_2); IR (Nujol) ν/cm^{-1} 1249(m), 1125(m), 1083(m), 932(w), 840(m), 614(w); MS:FAB m/z 435 $[In\{N(SiMe_3)_2\}_2^+$, 7%], 160 $[N(SiMe_3)_2^+$, 100%].

Crystallography:

Table 1. Crystal data for $[InBr_2\{N(SiMe_3)_2\}_2][Li(DME)_3]$

Formula	$C_{24}H_{66}Br_2InLiO_6Si_4$	Formula weight	872.73
Crystal system	monoclinic	Crystal size, mm	0.50x0.50x0.45
Space Group	$P2_1/n$	*a*, Å	10.3302(6)
b, Å	19.5470(11)	*c*, Å	22.0612(12)
β,° 102.2940(10)	*V*, Å^3	4352.5(4)	
Z 4	Diffractometer	SMART CCD	
Temperature, K	223(2)	μ(Mo-K_α), mm^{-1}	2.52
D_{calcd}, g cm^{-3}	1.332	*F*(000)	1800
θ_{max},°	23.26	Reflns meas.	19616
Reflns unique	6241	Reflns with $I > 2\sigma(I)$	4547
$R(F^2)$, $R_w(F^2)$ (all data)	0.046, 0.140	ρ, e Å^{-3}	1.64
G.O.F.	1.035	No. obs/No. para	6241/368
Programs used	SHELX-97 [4], Ortep-3 [5]		
Deposition number	CCDC 195078		

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REFERENCES

1. as determined from a survey of the Cambridge Crystallographic Database.
2. M.R. Kopp and B. Neumuller, *Z. Anorg. Allg. Chem.*, **624**, 361 (1998).
3. J. Pauls and B. Neumuller, *Inorg. Chem.*, **40**, 121 (2001).
4. G.M. Sheldrick, *SHELX-97*, University of Göttingen, Germany, 1997.
5. L.J. Farrugia, *Ortep-3 for Windows*, University of Glasgow, 1998.