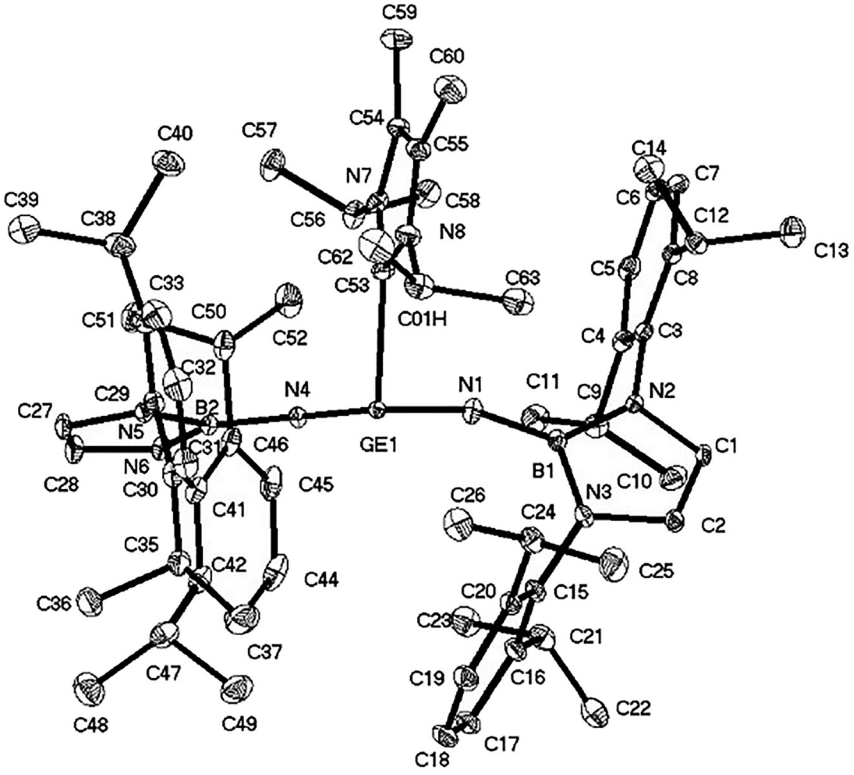
Bing Wang, Yi-Fei Hu and Di Wu*

Crystal structure of 1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene-*N*,*N*′-bis(1,3-bis(2,6-diisopropylphenyl)-1,3-dihydro-2*H*-1,3,2-diazaborol-2-yl)-l^2-germenediamine, $C_{63}H_{94}B_2GeN_8$



https://doi.org/10.1515/ncrs-2023-0300
Received June 26, 2023; accepted July 23, 2023;
published online August 15, 2023

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Abstract

$C_{63}H_{94}B_2GeN_8$, orthorhombic, $P2_12_12_1$ (no. 19), a = 17.1750(7) Å, b = 19.1188(7) Å, c = 19.4359(8) Å, V = 6382.1(4) Å^3, Z = 4, $R_{gt}(F)$ = 0.0539, $wR_{ref}(F^2)$ = 0.1483, T = 170 K.

CCDC no.: 2276415

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of material

Under a nitrogen atmosphere, a solution of *i*PrNHC·$GeCl_2$ [4] (0.32 g, 1 mmol) in 50 ml of THF was slowly added dropwise

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.20 × 0.13 × 0.10 mm
Wavelength:	Ga *K*α radiation (1.34138 Å)
μ:	0.63 mm^{-1}
Diffractometer, scan mode:	Bruker D8 VENTURE, φ and ω
θ_{max}, completeness:	57.1°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	56,045, 13064, 0.060
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 9561
$N(param)_{refined}$:	699
Programs:	Olex2 [1], SHELX [2, 3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
N1	0.4759 (2)	0.56895 (19)	0.4731 (2)	0.0546 (10)
H1A[a]	0.525033	0.582016	0.471225	0.066*
H1B[b]	0.442153	0.538463	0.489140	0.066*
N2	0.43403 (17)	0.65824 (17)	0.38282 (17)	0.0370 (8)
N3	0.34665 (18)	0.63739 (18)	0.46771 (16)	0.0426 (8)
N4	0.5455 (2)	0.4879 (2)	0.57375 (17)	0.0528 (9)
H4A[a]	0.573665	0.525362	0.565176	0.063*
H4B[b]	0.496601	0.493109	0.586266	0.063*
N5	0.5597 (2)	0.37613 (18)	0.64745 (17)	0.0444 (9)
N6	0.6365 (2)	0.46993 (18)	0.67560 (19)	0.0483 (9)
N7	0.5849 (2)	0.42495 (19)	0.3992 (2)	0.0486 (9)
N8	0.4671 (2)	0.3886 (2)	0.3870 (2)	0.0599 (10)
C1	0.3677 (2)	0.7010 (2)	0.3728 (2)	0.0419 (10)
H1	0.360955	0.733436	0.336224	0.050*
C01H	0.3830 (3)	0.3732 (3)	0.4011 (3)	0.0740 (16)
H01H	0.373097	0.385528	0.450320	0.089*
C2	0.3164 (2)	0.6889 (3)	0.4224 (2)	0.0486 (11)
H2	0.267239	0.711089	0.426874	0.058*
C3	0.5030 (2)	0.6636 (2)	0.3420 (2)	0.0397 (10)
C4	0.5671 (2)	0.6997 (2)	0.3685 (2)	0.0460 (10)
C5	0.6352 (2)	0.7016 (2)	0.3281 (3)	0.0539 (12)
H5	0.679833	0.725175	0.345363	0.065*
C6	0.6391 (3)	0.6704 (2)	0.2644 (3)	0.0530 (12)
H6	0.685491	0.672569	0.237839	0.064*
C7	0.5742 (3)	0.6357 (2)	0.2398 (2)	0.0498 (11)
H7	0.576723	0.613834	0.195946	0.060*
C8	0.5055 (2)	0.6319 (2)	0.2772 (2)	0.0438 (10)
C9	0.5630 (3)	0.7369 (3)	0.4373 (3)	0.0553 (12)
H9	0.526357	0.709831	0.467157	0.066*
C10	0.5296 (3)	0.8094 (3)	0.4288 (3)	0.0716 (15)
H10A	0.564954	0.837730	0.400645	0.107*
H10B	0.523524	0.831227	0.474119	0.107*
H10C	0.478758	0.806436	0.406148	0.107*
C11	0.6411 (3)	0.7403 (3)	0.4745 (3)	0.0767 (16)
H11A	0.666188	0.694338	0.472847	0.115*
H11B	0.632630	0.753911	0.522508	0.115*
H11C	0.674525	0.774950	0.451933	0.115*
C12	0.4365 (2)	0.5934 (2)	0.2473 (2)	0.0494 (11)
H12	0.396285	0.589559	0.284295	0.059*
C13	0.4004 (3)	0.6340 (3)	0.1878 (3)	0.0787 (17)
H13A	0.384477	0.680431	0.203788	0.118*
H13B	0.354721	0.608656	0.170572	0.118*
H13C	0.438647	0.638890	0.150698	0.118*
C14	0.4573 (3)	0.5194 (3)	0.2246 (3)	0.0750 (14)
H14A	0.491383	0.521526	0.184271	0.112*
H14B	0.409569	0.493960	0.212864	0.112*
H14C	0.484174	0.495221	0.262178	0.112*
C15	0.3135 (2)	0.6291 (3)	0.5353 (2)	0.0483 (11)
C16	0.3388 (3)	0.6739 (3)	0.5877 (2)	0.0549 (12)
C17	0.3057 (3)	0.6657 (3)	0.6536 (2)	0.0640 (14)
H17	0.322877	0.694211	0.690649	0.077*
C18	0.2483 (3)	0.6161 (3)	0.6643 (3)	0.0716 (16)
H18	0.225814	0.611399	0.708742	0.086*
C19	0.2234 (3)	0.5739 (3)	0.6124 (3)	0.0661 (14)
H19	0.183238	0.540963	0.620983	0.079*
C20	0.2559 (3)	0.5782 (3)	0.5464 (2)	0.0525 (12)
C21	0.3999 (3)	0.7292 (3)	0.5767 (3)	0.0631 (14)
H21	0.412545	0.730154	0.526493	0.076*
C22	0.3695 (4)	0.8012 (4)	0.5962 (3)	0.0835 (18)
H22A	0.324769	0.812959	0.567096	0.125*
H22B	0.410724	0.836029	0.589351	0.125*
H22C	0.353523	0.801140	0.644549	0.125*
C23	0.4753 (3)	0.7128 (4)	0.6156 (3)	0.0851 (19)
H23A	0.466042	0.716339	0.665187	0.128*
H23B	0.515743	0.746352	0.602106	0.128*
H23C	0.492493	0.665324	0.604236	0.128*
C24	0.2283 (3)	0.5302 (3)	0.4898 (3)	0.0611 (13)
H24	0.270421	0.529495	0.454311	0.073*
C25	0.1566 (4)	0.5590 (4)	0.4547 (4)	0.108 (2)
H25A	0.113425	0.560693	0.487705	0.161*
H25B	0.142354	0.528626	0.416073	0.161*
H25C	0.167367	0.606210	0.437676	0.161*
C26	0.2173 (4)	0.4545 (3)	0.5134 (4)	0.0839 (17)
H26A	0.267934	0.434351	0.525527	0.126*
H26B	0.193810	0.427139	0.476141	0.126*
H26C	0.183004	0.453411	0.553734	0.126*
C27	0.6075 (3)	0.3593 (3)	0.7041 (2)	0.0541 (12)
H27	0.608771	0.315299	0.726773	0.065*
C28	0.6505 (3)	0.4145 (2)	0.7208 (3)	0.0594 (13)
H28	0.685861	0.416124	0.758348	0.071*
C29	0.4965 (3)	0.3321 (2)	0.6258 (2)	0.0533 (12)
C30	0.4216 (3)	0.3445 (3)	0.6522 (3)	0.0574 (12)
C31	0.3609 (4)	0.3032 (3)	0.6290 (3)	0.0770 (16)
H31	0.309570	0.311480	0.645209	0.092*
C32	0.3746 (4)	0.2503 (4)	0.5827 (4)	0.086 (2)
H32	0.332339	0.222589	0.566907	0.103*
C33	0.4476 (4)	0.2371 (3)	0.5590 (3)	0.0850 (19)
H33	0.455591	0.199040	0.528323	0.102*
C34	0.5114 (3)	0.2780 (3)	0.5787 (3)	0.0637 (14)
C35	0.4069 (3)	0.4015 (3)	0.7052 (3)	0.0630 (14)
H35	0.446805	0.438738	0.697039	0.076*
C36	0.4223 (4)	0.3720 (4)	0.7773 (3)	0.0871 (19)
H36A	0.380200	0.339966	0.790039	0.131*
H36B	0.424651	0.410510	0.810567	0.131*
H36C	0.471967	0.346755	0.777425	0.131*
C37	0.3296 (4)	0.4356 (5)	0.6992 (4)	0.116 (3)
H37A	0.321709	0.451508	0.651784	0.174*
H37B	0.327125	0.475777	0.730441	0.174*
H37C	0.288767	0.401980	0.711439	0.174*
C38	0.5927 (4)	0.2623 (3)	0.5539 (3)	0.0792 (18)

Table 2: (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H38	0.624433	0.305609	0.560819	0.095*
C39	0.6304 (4)	0.2047 (4)	0.5957 (4)	0.102 (2)
H39A	0.625432	0.215507	0.644825	0.154*
H39B	0.685652	0.201298	0.583567	0.154*
H39C	0.604539	0.160171	0.585868	0.154*
C40	0.5962 (5)	0.2443 (3)	0.4769 (3)	0.102 (2)
H40A	0.573750	0.197777	0.469430	0.153*
H40B	0.650586	0.244450	0.461577	0.153*
H40C	0.566630	0.279040	0.450718	0.153*
C41	0.6562 (3)	0.5414 (2)	0.6896 (3)	0.0547 (12)
C42	0.6150 (3)	0.5780 (3)	0.7396 (3)	0.0640 (14)
C43	0.6332 (3)	0.6489 (3)	0.7489 (4)	0.0761 (17)
H43	0.606948	0.674557	0.783741	0.091*
C44	0.6869 (4)	0.6816 (3)	0.7099 (4)	0.085 (2)
H44	0.696945	0.729961	0.716754	0.102*
C45	0.7276 (3)	0.6448 (3)	0.6598 (4)	0.0736 (17)
H45	0.765365	0.668165	0.632368	0.088*
C46	0.7133 (3)	0.5739 (3)	0.6496 (3)	0.0587 (13)
C47	0.5509 (3)	0.5437 (3)	0.7830 (3)	0.0730 (15)
H47	0.544879	0.494452	0.766621	0.088*
C48	0.5738 (5)	0.5409 (4)	0.8595 (3)	0.107 (2)
H48A	0.619579	0.510935	0.865173	0.161*
H48B	0.530371	0.521851	0.886391	0.161*
H48C	0.585868	0.588220	0.875788	0.161*
C49	0.4719 (4)	0.5815 (4)	0.7713 (4)	0.100 (2)
H49A	0.476471	0.630517	0.785411	0.150*
H49B	0.431322	0.558534	0.798609	0.150*
H49C	0.458078	0.579221	0.722408	0.150*
C50	0.7589 (3)	0.5323 (3)	0.5962 (3)	0.0664 (15)
H50	0.720970	0.500892	0.572363	0.080*
C51	0.8192 (3)	0.4853 (4)	0.6314 (3)	0.0896 (19)
H51A	0.856428	0.514388	0.656749	0.134*
H51B	0.846866	0.457904	0.596572	0.134*
H51C	0.792753	0.453661	0.663473	0.134*
C52	0.7972 (3)	0.5770 (4)	0.5414 (4)	0.090 (2)
H52A	0.757989	0.607134	0.519833	0.134*
H52B	0.820585	0.546665	0.506349	0.134*
H52C	0.837729	0.606000	0.562416	0.134*
C53	0.5130 (3)	0.4263 (3)	0.4296 (2)	0.0530 (12)
C54	0.5810 (3)	0.3857 (3)	0.3389 (2)	0.0535 (12)
C55	0.5083 (3)	0.3648 (3)	0.3318 (2)	0.0550 (12)
C56	0.6511 (3)	0.4627 (3)	0.4296 (3)	0.0620 (13)
H56	0.632727	0.483580	0.473889	0.074*
C57	0.7169 (3)	0.4114 (3)	0.4471 (4)	0.0779 (17)
H57A	0.697400	0.375717	0.478857	0.117*
H57B	0.759959	0.436881	0.468672	0.117*
H57C	0.735342	0.388929	0.404785	0.117*
C58	0.6756 (3)	0.5219 (3)	0.3839 (3)	0.0717 (14)
H58A	0.704030	0.503401	0.344159	0.108*
H58B	0.709469	0.553829	0.409702	0.108*
H58C	0.629446	0.547327	0.368009	0.108*
C59	0.6506 (4)	0.3711 (4)	0.2930 (3)	0.097 (2)
H59A	0.679357	0.414581	0.285065	0.145*
H59B	0.632547	0.352226	0.248921	0.145*
H59C	0.684734	0.336936	0.315455	0.145*
C60	0.4765 (4)	0.3240 (3)	0.2715 (3)	0.088 (2)

Table 2: (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H60A	0.470957	0.274740	0.284436	0.132*
H60B	0.512265	0.328036	0.232487	0.132*
H60C	0.425453	0.342893	0.258585	0.132*
C62	0.3623 (4)	0.2966 (4)	0.3926 (4)	0.107 (2)
H62A	0.351880	0.286771	0.343971	0.160*
H62B	0.315813	0.285932	0.419887	0.160*
H62C	0.405792	0.267574	0.408419	0.160*
C63	0.3351 (3)	0.4256 (4)	0.3561 (3)	0.089 (2)
H63A	0.352226	0.473535	0.365902	0.133*
H63B	0.279653	0.421047	0.366986	0.133*
H63C	0.343529	0.415244	0.307312	0.133*
B1	0.4224 (3)	0.6170 (3)	0.4432 (2)	0.0401 (11)
B2	0.5765 (3)	0.4477 (3)	0.6271 (3)	0.0442 (11)
Ge1[a]	0.45853 (3)	0.48148 (3)	0.51613 (3)	0.03448 (18)
Ge1A[b]	0.54961 (15)	0.53332 (14)	0.49669 (13)	0.0358 (10)

[a]Occupancy: 0.8339 (16), [b]Occupancy: 0.1661 (16).

to a solution of $B(NDipCH)_2NHLi$ [5] (0.82 g, 2 mmol) at −78 °C. The resulting solution was slowly warmed to room temperature and stirred overnight. The solvent was then evaporated under vacuum, and the product was extracted with toluene and washed with *n*-hexane to afford as a light yellow powder (Single crystals were obtained from a 1:10 mixture of hexane/THF).

2 Experimental details

Using Olex2 [1], the structure was solved with the ShelXT [2] structure solution program using Intrinsic Phasing and refined with the ShelXL [3] refinement package.

3 Comment

The compounds of divalent germanium element, called germylenes, are considered as heavier congeners of carbenes due to their similar electronic structure [6]. Germylenes possess a lone pair of electrons and a vacant *p* orbital, which makes them highly reactive and serve as both nucleophiles and electrophiles [7, 8]. We demonstrate a successful application of the salt-metathesis reaction of lithium salt with $GeCl_2$ to produce a linear NHC-stabilized germylene species [9]. This provides a new method for the preparation of germylene species. The treatment of $B(NDipCH)_2NHLi$ (Dip = 2,6-diisopropyl phenyl) with 0.5 equivalents of *i*Pr·$GeCl_2$ [10] (*i*Pr = 1,3-diisopropyl-4,5-dimethyl-imidazolin-2-ylidene) produced the title compound. The X-ray diffraction analysis of the compound revealed that the length of N–Ge bonds is 1.894(4) Å (Ge_1–N_1) and 1.871(4) Å (Ge_1–N_4),

which fall within the range of the typical Ge–N single bonds [10].

Acknowledgments: We thank Mr. J. Liu (Zhejiang University) for X-Ray diffraction analysis.
Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.
Research funding: None declared.
Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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