Convenient Syntheses of Aryl and Perfluoroaryl Trichlorogermanes and Germatranes via an Organotin Route.

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Supporting Information

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NMR spectroscopy data for *n*-BuGeCl₃.

¹H NMR (CDCl₃, 300 MHz): δ 2.11-2.05 (m, 2H, CH₂Ge), 1.80-1.69 (m, 2H, CH₂CH₂Ge), 1.49 (qt, 2H, ³J = 7.2; 7.2, CH₂CH₂CH₃), 0.98 (t, 3H, ³J = 7.2, CH₂CH₃).

¹³C {¹H} NMR (CDCl₃): δ 32.6, 25.1, 24.2, 13.4.

Preparation of germatranes 3a-e. The following procedure for **3c** can serve as a typical preparation: a 100 mL Schlenk flask equipped with a magnetic stirbar was charged with 1.1258 g of **2c** (3.42 mmol) and sealed with a rubber septum. The flask was evacuated and refilled with nitrogen followed by addition of dry toluene (30 mL). To the resulting solution 0.60 mL of absolute ethanol (10.30 mmol) was added followed by 1.43 mL of triethylamine (10.30 mmol) freshly distilled from CaH₂. After 1 h the white precipitate formed was filtered off under nitrogen and washed with 60 mL of dry toluene. The filtrate and washings were combined and added to a 250 mL round-bottom flask containing a magnetic stirbar and 0.5280 g of 99% triethanolamine (3.50 mmol). A reflux condenser was attached to the flask and the toluene solution was heated at 100-110 °C for 14 hrs under nitrogen. After cooling down the solution was filtered followed by removal of toluene on the rotary evaporator. The resulting solid was recrystallized from dichloromethane-hexanes yielding 0.9898 g of **3c** (78%) as a white crystalline solid. Additional amount of **3c** (0.1240 g) was isolated from mother liquor upon slow evaporation of solvents raising the overall yield to 88%.

Structure Determination and Refinement of 3a. Crystals of **3a** were obtained by crystallization from chloroform/ethylcyclohexane. Data were collected on a Nonius Kappa CCD (Mo-K_α radiation) and corrected for absorption. The structure was solved by direct method and refined on *F* for all reflections. Non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms were included at calculated positions. **3a** crystallized in the polar space group *Pna*2₁ and the correct polarity was determined by refining the opposite enantiomer which gave an *R* and *R_w* of 0.0348 and 0.0423 relative to the correct enantiomer which gave improved values of 0.0310 and 0.0380.

X-ray analysis of 3a.**A. Crystal Data**

Empirical Formula	C ₁₂ H ₁₇ GeNO ₃
Formula Weight	295.86
Crystal Color, Habit	Colorless, needle
Crystal Dimensions	0.10 × 0.12 × 0.12 mm
Crystal System	orthorhombic
Lattice Type	Primitive
Lattice Parameters	a = 15.7635(4) Å b = 6.6216(2) Å c = 11.6955(4) Å V = 1220.77(6) Å ³
Space Group	Pna2 ₁ (#33)
Z value	4
D _{calc}	1.610 g/cm ³
F ₀₀₀	608.00
μ(MoKα)	25.04 cm ⁻¹

B. Intensity Measurements

Diffractometer	Nonius KappaCCD
Radiation	MoKα (λ = 0.71069 Å) graphite monochromated
Data Images	172 exposures @ 120.0 seconds

ω oscillation Range	1.0°
Detector Position	35.00 mm
$2\theta_{\text{max}}$	55.0°
No. of Reflections Measured	Total: 2744 Unique: 1645 ($R_{\text{int}} = 0.027$)
Corrections	Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F
Function Minimized	$\Sigma w (F_o - F_c)^2$
Least Squares Weights	$1/\sigma^2(F_o) = 4F_o^2/\sigma^2(F_o^2)$
p-factor	0.0100
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ($I > 3.00\sigma(I)$)	1363
No. Variables	153
Reflection/Parameter Ratio	8.91
Residuals: R; R_w	0.031 ; 0.038
Goodness of Fit Indicator	2.07
Max Shift/Error in Final Cycle	0.00
Maximum peak in Final Diff. Map	0.36 e ⁻ /Å ³
Minimum peak in Final Diff. Map	-0.48 e ⁻ /Å ³

Table 1. Atomic coordinates and Biso/Beq

atom	x	y	z	Beq
Ge(1)	0.320950(13)	0.35415(4)	0.2500	1.141(6)
O(1)	0.36093(12)	0.2256(3)	0.3736(2)	1.73(4)
O(2)	0.23653(11)	0.2313(3)	0.1712(2)	1.89(4)
O(3)	0.33178(11)	0.6252(3)	0.2491(5)	2.05(4)
N(1)	0.2151(1)	0.4299(4)	0.3666(2)	1.40(5)
C(1)	0.4104(2)	0.2862(4)	0.1414(3)	1.33(6)
C(2)	0.4391(2)	0.0884(5)	0.1282(3)	1.72(6)
C(3)	0.4941(2)	0.0359(5)	0.0423(3)	1.83(6)
C(4)	0.5240(2)	0.1783(5)	-0.0342(3)	2.09(7)
C(5)	0.4983(2)	0.3790(5)	-0.0213(3)	2.04(7)
C(6)	0.4432(2)	0.4318(5)	0.0660(3)	1.56(6)
C(7)	0.2254(2)	0.2896(5)	0.4643(3)	1.82(6)
C(8)	0.3199(2)	0.2533(6)	0.4811(3)	1.92(7)
C(9)	0.1389(2)	0.3896(5)	0.2982(3)	1.84(6)
C(10)	0.1551(2)	0.2105(5)	0.2229(3)	1.91(7)
C(11)	0.2288(2)	0.6441(4)	0.3944(3)	1.90(7)
C(12)	0.2656(2)	0.7446(4)	0.2902(3)	2.03(6)
H(1)	0.4201	-0.0125	0.1799	2.063
H(2)	0.5119	-0.1006	0.0352	2.202
H(3)	0.5612	0.1405	-0.0943	2.510
H(4)	0.5187	0.4794	-0.0724	2.451
H(5)	0.4272	0.5692	0.0750	1.870
H(6)	0.2018	0.3478	0.5314	2.190
H(7)	0.1976	0.1655	0.4482	2.190
H(8)	0.3278	0.1359	0.5266	2.305
H(9)	0.3442	0.3665	0.5189	2.305
H(10)	0.0924	0.3620	0.3475	2.207
H(11)	0.1262	0.5040	0.2524	2.207
H(12)	0.1128	0.2037	0.1652	2.298
H(13)	0.1537	0.0905	0.2675	2.298
H(14)	0.1764	0.7057	0.4141	2.282
H(15)	0.2672	0.6556	0.4567	2.282
H(16)	0.2865	0.8746	0.3100	2.441
H(17)	0.2230	0.7577	0.2331	2.441

$$\text{Beq} = 8/3 \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos \gamma + 2U_{13}(aa^*cc^*)\cos \beta + 2U_{23}(bb^*cc^*)\cos \alpha)$$

Table 2. Anisotropic Displacement Parameters

atom	U11	U22	U33	U12	U13	U23
Ge(1)	0.0155(2)	0.0155(2)	0.0124(2)	0.00200(9)	-0.0004(2)	-0.0003(2)
O(1)	0.0227(11)	0.0242(11)	0.0189(11)	0.0075(9)	-0.0009(10)	0.0027(11)
O(2)	0.0166(10)	0.0351(12)	0.0201(11)	0.0009(10)	-0.0003(9)	-0.0113(12)
O(3)	0.0301(10)	0.0175(10)	0.0301(13)	0.0029(7)	0.010(2)	0.006(2)
N(1)	0.0162(11)	0.0222(13)	0.0149(13)	0.0040(10)	-0.0001(11)	0.0006(12)
C(1)	0.0158(13)	0.022(1)	0.013(2)	0.0004(13)	0.0018(12)	-0.006(2)
C(2)	0.021(1)	0.022(1)	0.023(2)	0.0018(13)	-0.001(1)	-0.003(2)
C(3)	0.0175(13)	0.027(2)	0.025(2)	0.0008(13)	-0.0018(13)	-0.009(2)
C(4)	0.0147(13)	0.041(2)	0.024(2)	-0.0002(13)	0.0034(13)	-0.014(2)
C(5)	0.0129(13)	0.040(2)	0.024(2)	-0.0091(13)	0.002(1)	0.009(2)
C(6)	0.0201(13)	0.020(2)	0.019(2)	-0.0019(12)	0.0031(13)	-0.003(2)
C(7)	0.023(2)	0.026(2)	0.020(2)	-0.004(1)	0.0065(13)	0.001(2)
C(8)	0.023(2)	0.030(2)	0.020(2)	0.0037(12)	0.0019(13)	0.006(2)
C(9)	0.014(1)	0.034(2)	0.021(2)	0.0015(12)	0.0014(13)	-0.003(2)
C(10)	0.0148(12)	0.031(2)	0.027(2)	-0.001(1)	-0.0018(12)	-0.005(2)
C(11)	0.025(2)	0.023(2)	0.024(2)	0.0047(12)	0.004(1)	-0.005(2)
C(12)	0.031(2)	0.018(1)	0.029(2)	0.0012(13)	0.0061(13)	-0.003(2)

The general temperature factor expression: $\exp(-2\pi^2(a^2U_{11}h^2 + b^2U_{22}k^2 + c^2U_{33}l^2 + 2a*b*U_{12}hk + 2a*c*U_{13}hl + 2b*c*U_{23}kl))$

Table 3. Bond Lengths (Å)

atom	atom	distance	atom	atom	distance
Ge(1)	O(1)	1.792(3)	Ge(1)	O(2)	1.812(3)
Ge(1)	O(3)	1.803(3)	Ge(1)	N(1)	2.212(3)
Ge(1)	C(1)	1.950(4)	O(1)	C(8)	1.426(6)
O(2)	C(10)	1.426(5)	O(3)	C(12)	1.395(6)
N(1)	C(7)	1.481(6)	N(1)	C(9)	1.468(6)
N(1)	C(11)	1.471(5)	C(1)	C(2)	1.394(6)
C(1)	C(6)	1.405(6)	C(2)	C(3)	1.372(7)
C(3)	C(4)	1.383(7)	C(4)	C(5)	1.398(6)
C(5)	C(6)	1.385(7)	C(7)	C(8)	1.523(5)
C(9)	C(10)	1.499(6)	C(11)	C(12)	1.505(7)

Table 4. Bond Angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
O(1)	Ge(1)	O(2)	117.1(1)	O(1)	Ge(1)	O(3)	116.4(3)
O(1)	Ge(1)	N(1)	82.87(13)	O(1)	Ge(1)	C(1)	99.3(2)
O(2)	Ge(1)	O(3)	120.9(2)	O(2)	Ge(1)	N(1)	82.05(13)
O(2)	Ge(1)	C(1)	95.5(2)	O(3)	Ge(1)	N(1)	81.3(2)
O(3)	Ge(1)	C(1)	99.1(2)	N(1)	Ge(1)	C(1)	177.3(2)
Ge(1)	O(1)	C(8)	119.4(2)	Ge(1)	O(2)	C(10)	119.2(3)
Ge(1)	O(3)	C(12)	119.4(3)	Ge(1)	N(1)	C(7)	104.5(2)
Ge(1)	N(1)	C(9)	103.9(3)	Ge(1)	N(1)	C(11)	104.2(3)
C(7)	N(1)	C(9)	113.3(3)	C(7)	N(1)	C(11)	114.7(4)
C(9)	N(1)	C(11)	114.6(3)	Ge(1)	C(1)	C(2)	121.6(3)
Ge(1)	C(1)	C(6)	121.1(3)	C(2)	C(1)	C(6)	117.1(4)
C(1)	C(2)	C(3)	121.6(4)	C(2)	C(3)	C(4)	121.1(4)
C(3)	C(4)	C(5)	118.7(4)	C(4)	C(5)	C(6)	120.1(4)
C(1)	C(6)	C(5)	121.3(4)	N(1)	C(7)	C(8)	107.8(3)
O(1)	C(8)	C(7)	110.5(4)	N(1)	C(9)	C(10)	108.9(3)
O(2)	C(10)	C(9)	109.0(3)	N(1)	C(11)	C(12)	107.7(4)
O(3)	C(12)	C(11)	108.4(4)				

Table 5. Torsion Angles (°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
Ge(1)	O(1)	C(8)	C(7)	31.6(5)	Ge(1)	O(2)	C(10)	C(9)	33.4(4)
Ge(1)	O(3)	C(12)	C(11)	39.5(7)	Ge(1)	N(1)	C(7)	C(8)	33.2(4)
Ge(1)	N(1)	C(9)	C(10)	35.5(4)	Ge(1)	N(1)	C(11)	C(12)	34.3(4)
Ge(1)	C(1)	C(2)	C(3)	171.7(4)	Ge(1)	C(1)	C(6)	C(5)	-171.3(4)
O(1)	Ge(1)	O(2)	C(10)	67.8(3)	O(1)	Ge(1)	O(3)	C(12)	-92.7(6)
O(1)	Ge(1)	N(1)	C(7)	-15.1(3)	O(1)	Ge(1)	N(1)	C(9)	-134.1(3)
O(1)	Ge(1)	N(1)	C(11)	105.6(3)	O(1)	Ge(1)	C(1)	C(2)	48.7(4)
O(1)	Ge(1)	C(1)	C(6)	-137.2(3)	O(1)	C(8)	C(7)	N(1)	-42.6(5)
O(2)	Ge(1)	O(1)	C(8)	-86.3(3)	O(2)	Ge(1)	O(3)	C(12)	60.1(7)
O(2)	Ge(1)	N(1)	C(7)	103.6(3)	O(2)	Ge(1)	N(1)	C(9)	-15.4(3)
O(2)	Ge(1)	N(1)	C(11)	-135.7(3)	O(2)	Ge(1)	C(1)	C(2)	-70.0(4)
O(2)	Ge(1)	C(1)	C(6)	104.2(4)	O(2)	C(10)	C(9)	N(1)	-45.3(5)
O(3)	Ge(1)	O(1)	C(8)	67.5(4)	O(3)	Ge(1)	O(2)	C(10)	-84.9(4)
O(3)	Ge(1)	N(1)	C(7)	-133.3(3)	O(3)	Ge(1)	N(1)	C(9)	107.7(3)
O(3)	Ge(1)	N(1)	C(11)	-12.6(3)	O(3)	Ge(1)	C(1)	C(2)	167.5(4)
O(3)	Ge(1)	C(1)	C(6)	-18.3(4)	O(3)	C(12)	C(11)	N(1)	-47.6(5)
N(1)	Ge(1)	O(1)	C(8)	-9.1(3)	N(1)	Ge(1)	O(2)	C(10)	-10.0(3)
N(1)	Ge(1)	O(3)	C(12)	-15.1(6)	N(1)	Ge(1)	C(1)	C(2)	-94.5(32)
N(1)	Ge(1)	C(1)	C(6)	79.7(31)	C(1)	Ge(1)	O(1)	C(8)	172.5(3)
C(1)	Ge(1)	O(2)	C(10)	171.1(3)	C(1)	Ge(1)	O(3)	C(12)	162.2(6)

C(1)	Ge(1)	N(1)	C(7)	128.2(31)	C(1)	Ge(1)	N(1)	C(9)	9.2(32)
C(1)	Ge(1)	N(1)	C(11)	-111.1(31)	C(1)	C(2)	C(3)	C(4)	0.7(7)
C(1)	C(6)	C(5)	C(4)	-1.5(7)	C(2)	C(1)	C(6)	C(5)	3.1(7)
C(2)	C(3)	C(4)	C(5)	1.1(7)	C(3)	C(2)	C(1)	C(6)	-2.8(7)
C(3)	C(4)	C(5)	C(6)	-0.7(7)	C(7)	N(1)	C(9)	C(10)	-77.4(5)
C(7)	N(1)	C(11)	C(12)	147.9(4)	C(8)	C(7)	N(1)	C(9)	145.7(4)
C(8)	C(7)	N(1)	C(11)	-80.2(4)	C(9)	N(1)	C(11)	C(12)	-78.6(4)
C(10)	C(9)	N(1)	C(11)	148.4(4)					

Table 6. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	ADC	atom	atom	distance	ADC
O(1)	C(4)	3.407(5)	65502	O(1)	C(3)	3.481(5)	65502
O(2)	C(11)	3.333(7)	54403	O(2)	C(12)	3.540(6)	54501
C(3)	C(8)	3.574(6)	65402				

The ADC (atom designator code) specifies the position of an atom in a crystal. The 5-digit number shown in the table is a composite of three one-digit numbers and one two-digit number: TA (first digit) + TB (second digit) + TC (third digit) + SN (last two digits). TA, TB and TC are the crystal lattice translation digits along cell edges a, b and c. A translation digit of 5 indicates the origin unit cell. If TA = 4, this indicates a translation of one unit cell length along the a-axis in the negative direction. Each translation digit can range in value from 1 to 9 and thus ± 4 lattice translations from the origin (TA=5, TB=5, TC=5) can be represented.

The SN, or symmetry operator number, refers to the number of the symmetry operator used to generate the coordinates of the target atom. A list of symmetry operators relevant to this structure is given below.

For a given intermolecular contact, the first atom (origin atom) is located in the origin unit cell and its position can be generated using the identity operator (SN=1). Thus, the ADC for an origin atom is always 55501. The position of the second atom (target atom) can be generated using the ADC and the coordinates of the atom in the parameter table. For example, an ADC of 47502 refers to the target atom moved through symmetry operator two, then translated -1 cell translations along the a axis, +2 cell translations along the b axis, and 0 cell translations along the c axis.

An ADC of 1 indicates an intermolecular contact between two fragments (eg. cation and anion) that reside in the same asymmetric unit.

Symmetry Operators:

(1)	+X, +Y, +Z	(2)	-X, -Y, 1/2+Z
(3)	1/2-X, 1/2+Y, 1/2+Z	(4)	1/2+X, 1/2-Y, +Z

Table 7. Least Squares Planes

Plane number 1

Atoms defining plane	Distance
C(7)	-0.017(4)
C(8)	0.013(4)
C(9)	0.001(5)
C(10)	-0.011(4)
C(11)	0.005(5)
C(12)	0.009(5)

Additional Atoms	Distance
Ge(1)	-0.252
N(1)	-0.622

Summary

plane	mean deviation	χ^2
1	0.0093	37.0

Figure 1. ORTEP drawing of **3a**.

X-ray analysis of 3b.**A. Crystal Data**

Empirical Formula	C ₁₂ H ₁₂ F ₅ GeNO ₃
Formula Weight	385.81
Crystal Color, Habit	colorless, needle
Crystal Dimensions	0.10 × 0.10 × 0.19 mm
Crystal System	monoclinic
Lattice Type	Primitive
Lattice Parameters	a = 6.7491(2) Å b = 9.6839(3) Å c = 20.7819(7) Å β = 94.713(1) ° V = 1353.66(7) Å ³
Space Group	P2 ₁ /c (#14)
Z value	4
D _{calc}	1.893 g/cm ³
F ₀₀₀	768.00
μ(MoKα)	23.33 cm ⁻¹

B. Intensity Measurements

Diffractometer	Nonius KappaCCD
Radiation	MoKα (λ = 0.71069 Å) graphite monochromated
Data Images	149 exposures @ 34.0 seconds

ω oscillation Range	1.7°
Detector Position	35.00 mm
$2\theta_{\max}$	54.9°
No. of Reflections Measured	Total: 5181 Unique: 3287 ($R_{\text{int}} = 0.037$)
Corrections	Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F
Function Minimized	$\Sigma w (F_o - F_c)^2$
Least Squares Weights	$1/\sigma^2(F_o) = 4F_o^2/\sigma^2(F_o^2)$
p-factor	0.0100
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ($I > 3.00\sigma(I)$)	1939
No. Variables	199
Reflection/Parameter Ratio	9.74
Residuals: R; R_w	0.033 ; 0.033
Goodness of Fit Indicator	1.06
Max Shift/Error in Final Cycle	0.00
Maximum peak in Final Diff. Map	0.39 e ⁻ /Å ³
Minimum peak in Final Diff. Map	-0.44 e ⁻ /Å ³

Table 1. Atomic coordinates and Biso/Beq

atom	x	y	z	Beq
Ge(1)	0.55472(5)	0.09258(3)	0.62666(2)	1.617(8)
F(1)	0.9103(3)	0.1739(2)	0.71860(8)	2.60(5)
F(2)	1.1678(3)	0.3832(2)	0.72144(11)	3.99(6)
F(3)	1.1396(3)	0.5787(2)	0.62697(11)	4.48(6)
F(4)	0.8422(3)	0.5646(2)	0.53129(10)	3.67(5)
F(5)	0.5780(3)	0.3618(2)	0.52835(9)	3.29(5)
O(1)	0.4638(3)	0.1347(2)	0.70280(10)	2.22(5)
O(2)	0.7553(3)	-0.0279(2)	0.62168(10)	1.93(5)
O(3)	0.3962(3)	0.1253(2)	0.55554(10)	2.28(5)
N(1)	0.3782(4)	-0.0916(3)	0.63366(11)	1.64(6)
C(1)	0.7321(5)	0.2561(3)	0.6233(2)	1.68(7)
C(2)	0.8880(5)	0.2691(3)	0.6701(2)	2.00(8)
C(3)	1.0241(5)	0.3750(4)	0.6727(2)	2.68(9)
C(4)	1.0086(5)	0.4737(4)	0.6255(2)	2.84(9)
C(5)	0.8576(6)	0.4671(3)	0.5776(2)	2.53(9)
C(6)	0.7226(5)	0.3597(3)	0.5773(2)	2.16(8)
C(7)	0.2478(5)	-0.0638(3)	0.6860(2)	2.13(8)
C(8)	0.3638(5)	0.0288(4)	0.7350(2)	2.33(8)
C(9)	0.5227(5)	-0.2029(4)	0.6491(2)	2.15(8)
C(10)	0.7068(5)	-0.1712(3)	0.6155(2)	2.19(8)
C(11)	0.2694(5)	-0.1038(4)	0.5691(2)	2.12(8)
C(12)	0.2219(5)	0.0413(4)	0.5458(2)	2.32(8)
H(1)	0.1300	-0.0184	0.6691	2.560
H(2)	0.2138	-0.1479	0.7059	2.560
H(3)	0.4588	-0.0252	0.7601	2.798
H(4)	0.2745	0.0697	0.7624	2.798
H(5)	0.5547	-0.2070	0.6944	2.579
H(6)	0.4683	-0.2888	0.6343	2.579
H(7)	0.6839	-0.1939	0.5710	2.626
H(8)	0.8145	-0.2247	0.6343	2.626
H(9)	0.3499	-0.1490	0.5401	2.539
H(10)	0.1501	-0.1546	0.5720	2.539
H(11)	0.1197	0.0788	0.5694	2.787
H(12)	0.1784	0.0393	0.5012	2.787

$$Beq = 8/3 \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos \gamma + 2U_{13}(aa^*cc^*)\cos \beta + 2U_{23}(bb^*cc^*)\cos \alpha)$$

Table 2. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Ge(1)	0.0186(2)	0.0194(2)	0.0234(2)	-0.0017(2)	0.0014(1)	-0.0010(2)
F(1)	0.0340(13)	0.0291(11)	0.0346(11)	-0.0005(10)	-0.0042(9)	-0.0016(9)
F(2)	0.0342(13)	0.044(1)	0.070(2)	-0.0081(11)	-0.0138(12)	-0.0120(11)
F(3)	0.044(1)	0.036(1)	0.091(2)	-0.0216(12)	0.0109(13)	-0.0020(12)
F(4)	0.061(2)	0.0252(12)	0.055(1)	-0.0034(11)	0.0171(12)	0.0068(10)
F(5)	0.051(2)	0.0290(12)	0.0426(12)	-0.0054(11)	-0.0085(11)	0.0068(10)
O(1)	0.028(1)	0.0296(13)	0.0277(13)	-0.0042(11)	0.0073(11)	-0.0065(10)
O(2)	0.0181(13)	0.0170(12)	0.039(1)	-0.0005(10)	0.0043(11)	-0.0027(10)
O(3)	0.0245(13)	0.030(1)	0.0306(13)	-0.0083(11)	-0.0065(11)	0.0081(10)
N(1)	0.015(1)	0.025(2)	0.023(1)	-0.0022(13)	0.0019(11)	-0.0005(13)
C(1)	0.015(2)	0.022(2)	0.028(2)	0.001(1)	0.002(2)	-0.001(1)
C(2)	0.025(2)	0.019(2)	0.033(2)	0.001(2)	0.008(2)	-0.004(2)
C(3)	0.021(2)	0.036(2)	0.044(2)	-0.001(2)	-0.004(2)	-0.011(2)
C(4)	0.029(2)	0.022(2)	0.058(3)	-0.011(2)	0.011(2)	-0.008(2)
C(5)	0.044(3)	0.019(2)	0.034(2)	-0.000(2)	0.011(2)	0.001(2)
C(6)	0.027(2)	0.027(2)	0.028(2)	0.001(2)	-0.001(2)	-0.006(2)
C(7)	0.023(2)	0.034(2)	0.025(2)	-0.004(2)	0.007(2)	0.005(2)
C(8)	0.024(2)	0.037(2)	0.028(2)	-0.004(2)	0.004(2)	-0.002(2)
C(9)	0.026(2)	0.024(2)	0.031(2)	-0.002(2)	0.002(2)	0.002(2)
C(10)	0.026(2)	0.018(2)	0.039(2)	-0.000(2)	0.003(2)	-0.000(2)
C(11)	0.022(2)	0.032(2)	0.026(2)	-0.007(2)	-0.001(2)	-0.004(2)
C(12)	0.026(2)	0.035(2)	0.027(2)	-0.007(2)	-0.004(2)	0.006(2)

The general temperature factor expression: $\exp(-2\pi^2(a^2U_{11}h^2 + b^2U_{22}k^2 + c^2U_{33}l^2 + 2a*b*U_{12}hk + 2a*c*U_{13}hl + 2b*c*U_{23}kl))$

Table 3. Bond Lengths (Å)

atom	atom	distance	atom	atom	distance
Ge(1)	O(1)	1.791(2)	Ge(1)	O(2)	1.797(2)
Ge(1)	O(3)	1.780(2)	Ge(1)	N(1)	2.156(3)
Ge(1)	C(1)	1.990(3)	F(1)	C(2)	1.365(4)
F(2)	C(3)	1.346(4)	F(3)	C(4)	1.346(4)
F(4)	C(5)	1.346(4)	F(5)	C(6)	1.351(4)
O(1)	C(8)	1.424(4)	O(2)	C(10)	1.429(4)
O(3)	C(12)	1.431(4)	N(1)	C(7)	1.479(4)
N(1)	C(9)	1.471(4)	N(1)	C(11)	1.480(4)
C(1)	C(2)	1.379(4)	C(1)	C(6)	1.384(4)
C(2)	C(3)	1.375(5)	C(3)	C(4)	1.367(5)
C(4)	C(5)	1.368(5)	C(5)	C(6)	1.382(5)
C(7)	C(8)	1.524(5)	C(9)	C(10)	1.507(5)
C(11)	C(12)	1.512(5)			

Table 4. Bond Angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
O(1)	Ge(1)	O(2)	120.89(10)	O(1)	Ge(1)	O(3)	117.83(11)
O(1)	Ge(1)	N(1)	84.12(10)	O(1)	Ge(1)	C(1)	95.76(11)
O(2)	Ge(1)	O(3)	118.16(10)	O(2)	Ge(1)	N(1)	83.68(10)
O(2)	Ge(1)	C(1)	93.24(11)	O(3)	Ge(1)	N(1)	84.57(9)
O(3)	Ge(1)	C(1)	98.73(11)	N(1)	Ge(1)	C(1)	176.29(11)
Ge(1)	O(1)	C(8)	117.4(2)	Ge(1)	O(2)	C(10)	117.9(2)
Ge(1)	O(3)	C(12)	116.6(2)	Ge(1)	N(1)	C(7)	105.3(2)
Ge(1)	N(1)	C(9)	105.1(2)	Ge(1)	N(1)	C(11)	103.9(2)
C(7)	N(1)	C(9)	113.4(3)	C(7)	N(1)	C(11)	113.6(3)
C(9)	N(1)	C(11)	114.2(3)	Ge(1)	C(1)	C(2)	118.3(2)
Ge(1)	C(1)	C(6)	127.4(3)	C(2)	C(1)	C(6)	114.2(3)
F(1)	C(2)	C(1)	119.4(3)	F(1)	C(2)	C(3)	116.2(3)
C(1)	C(2)	C(3)	124.4(3)	F(2)	C(3)	C(2)	120.7(3)
F(2)	C(3)	C(4)	120.4(3)	C(2)	C(3)	C(4)	118.8(3)
F(3)	C(4)	C(3)	120.1(4)	F(3)	C(4)	C(5)	120.0(3)
C(3)	C(4)	C(5)	119.9(3)	F(4)	C(5)	C(4)	119.8(3)
F(4)	C(5)	C(6)	120.9(3)	C(4)	C(5)	C(6)	119.3(3)
F(5)	C(6)	C(1)	121.4(3)	F(5)	C(6)	C(5)	115.2(3)
C(1)	C(6)	C(5)	123.4(3)	N(1)	C(7)	C(8)	107.1(3)
O(1)	C(8)	C(7)	110.3(3)	N(1)	C(9)	C(10)	107.9(3)
O(2)	C(10)	C(9)	110.3(3)	N(1)	C(11)	C(12)	107.0(3)
O(3)	C(12)	C(11)	109.4(3)				

Table 5. Torsion Angles (°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
Ge(1)	O(1)	C(8)	C(7)	-34.0(3)	Ge(1)	O(2)	C(10)	C(9)	-30.9(3)
Ge(1)	O(3)	C(12)	C(11)	-36.7(3)	Ge(1)	N(1)	C(7)	C(8)	-32.7(3)
Ge(1)	N(1)	C(9)	C(10)	-34.1(3)	Ge(1)	N(1)	C(11)	C(12)	-35.4(3)
Ge(1)	C(1)	C(2)	F(1)	-3.0(4)	Ge(1)	C(1)	C(2)	C(3)	178.4(3)
Ge(1)	C(1)	C(6)	F(5)	2.4(5)	Ge(1)	C(1)	C(6)	C(5)	-177.4(3)
F(1)	C(2)	C(1)	C(6)	179.0(3)	F(1)	C(2)	C(3)	F(2)	-1.2(5)
F(1)	C(2)	C(3)	C(4)	-179.7(3)	F(2)	C(3)	C(2)	C(1)	177.4(3)
F(2)	C(3)	C(4)	F(3)	1.2(5)	F(2)	C(3)	C(4)	C(5)	-177.5(3)
F(3)	C(4)	C(3)	C(2)	179.8(3)	F(3)	C(4)	C(5)	F(4)	1.1(5)
F(3)	C(4)	C(5)	C(6)	-179.0(3)	F(4)	C(5)	C(4)	C(3)	179.8(3)
F(4)	C(5)	C(6)	F(5)	-0.4(5)	F(4)	C(5)	C(6)	C(1)	179.5(3)
F(5)	C(6)	C(1)	C(2)	-179.8(3)	F(5)	C(6)	C(5)	C(4)	179.7(3)
O(1)	Ge(1)	O(2)	C(10)	87.6(2)	O(1)	Ge(1)	O(3)	C(12)	-68.3(2)
O(1)	Ge(1)	N(1)	C(7)	13.7(2)	O(1)	Ge(1)	N(1)	C(9)	-106.3(2)
O(1)	Ge(1)	N(1)	C(11)	133.4(2)	O(1)	Ge(1)	C(1)	C(2)	63.2(3)
O(1)	Ge(1)	C(1)	C(6)	-119.1(3)	O(1)	C(8)	C(7)	N(1)	43.5(4)
O(2)	Ge(1)	O(1)	C(8)	-67.7(2)	O(2)	Ge(1)	O(3)	C(12)	91.9(2)

O(2)	Ge(1)	N(1)	C(7)	135.7(2)	O(2)	Ge(1)	N(1)	C(9)	15.7(2)
O(2)	Ge(1)	N(1)	C(11)	-104.6(2)	O(2)	Ge(1)	C(1)	C(2)	-58.3(3)
O(2)	Ge(1)	C(1)	C(6)	119.4(3)	O(2)	C(10)	C(9)	N(1)	42.7(3)
O(3)	Ge(1)	O(1)	C(8)	92.0(2)	O(3)	Ge(1)	O(2)	C(10)	-72.0(2)
O(3)	Ge(1)	N(1)	C(7)	-105.1(2)	O(3)	Ge(1)	N(1)	C(9)	134.9(2)
O(3)	Ge(1)	N(1)	C(11)	14.6(2)	O(3)	Ge(1)	C(1)	C(2)	-177.5(3)
O(3)	Ge(1)	C(1)	C(6)	0.2(3)	O(3)	C(12)	C(11)	N(1)	47.3(3)
N(1)	Ge(1)	O(1)	C(8)	11.3(2)	N(1)	Ge(1)	O(2)	C(10)	8.4(2)
N(1)	Ge(1)	O(3)	C(12)	12.1(2)	N(1)	Ge(1)	C(1)	C(2)	-24.7(18)
N(1)	Ge(1)	C(1)	C(6)	153.0(15)	C(1)	Ge(1)	O(1)	C(8)	-165.0(2)
C(1)	Ge(1)	O(2)	C(10)	-173.7(2)	C(1)	Ge(1)	O(3)	C(12)	-169.6(2)
C(1)	Ge(1)	N(1)	C(7)	102.0(17)	C(1)	Ge(1)	N(1)	C(9)	-18.0(17)
C(1)	Ge(1)	N(1)	C(11)	-138.3(16)	C(1)	C(2)	C(3)	C(4)	-1.1(5)
C(1)	C(6)	C(5)	C(4)	-0.4(5)	C(2)	C(1)	C(6)	C(5)	0.3(5)
C(2)	C(3)	C(4)	C(5)	1.0(5)	C(3)	C(2)	C(1)	C(6)	0.4(5)
C(3)	C(4)	C(5)	C(6)	-0.3(5)	C(7)	N(1)	C(9)	C(10)	-148.6(3)
C(7)	N(1)	C(11)	C(12)	78.5(3)	C(8)	C(7)	N(1)	C(9)	81.7(3)
C(8)	C(7)	N(1)	C(11)	-145.7(3)	C(9)	N(1)	C(11)	C(12)	-149.3(3)
C(10)	C(9)	N(1)	C(11)	79.1(3)					

Table 6. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	ADC	atom	atom	distance	ADC
F(1)	F(2)	3.141(3)	74602	F(1)	C(7)	3.346(4)	65501
F(1)	C(8)	3.359(4)	65501	F(1)	F(3)	3.382(3)	74602
F(1)	C(7)	3.445(4)	65602	F(2)	O(1)	3.172(3)	65501
F(2)	O(2)	3.371(3)	75602	F(2)	C(9)	3.373(4)	75602
F(2)	C(10)	3.466(4)	75602	F(2)	C(8)	3.512(4)	75602
F(2)	C(7)	3.566(4)	65602	F(3)	C(9)	3.343(4)	66501
F(3)	C(11)	3.441(4)	66501	F(3)	N(1)	3.574(3)	66501
F(3)	F(4)	3.581(3)	76603	F(4)	F(4)	2.872(4)	76603
F(4)	F(5)	3.084(3)	66603	F(4)	C(5)	3.173(4)	76603
F(4)	C(10)	3.271(4)	56501	F(4)	C(4)	3.509(4)	76603
F(5)	F(5)	3.075(4)	66603	F(5)	C(11)	3.428(4)	65603
O(1)	C(9)	3.451(4)	65602	O(2)	C(7)	3.496(4)	65501
O(2)	C(12)	3.500(4)	65603	O(3)	C(11)	3.579(4)	65603
O(3)	C(10)	3.592(4)	65603	C(2)	C(7)	3.585(5)	65602
C(8)	C(9)	3.584(5)	65602	C(12)	C(12)	3.507(7)	55603

The ADC (atom designator code) specifies the position of an atom in a crystal. The 5-digit number shown in the table is a composite of three one-digit numbers and one two-digit number: TA (first digit) + TB (second digit) + TC (third digit) + SN (last two digits). TA, TB and TC are the crystal lattice translation digits along cell edges a, b and c. A translation digit of 5 indicates the origin unit cell. If TA = 4, this indicates a translation of one unit cell length along the a-axis

in the negative direction. Each translation digit can range in value from 1 to 9 and thus ± 4 lattice translations from the origin (TA=5, TB=5, TC=5) can be represented.

The SN, or symmetry operator number, refers to the number of the symmetry operator used to generate the coordinates of the target atom. A list of symmetry operators relevant to this structure is given below.

For a given intermolecular contact, the first atom (origin atom) is located in the origin unit cell and its position can be generated using the identity operator (SN=1). Thus, the ADC for an origin atom is always 55501. The position of the second atom (target atom) can be generated using the ADC and the coordinates of the atom in the parameter table. For example, an ADC of 47502 refers to the target atom moved through symmetry operator two, then translated -1 cell translations along the a axis, +2 cell translations along the b axis, and 0 cell translations along the c axis.

An ADC of 1 indicates an intermolecular contact between two fragments (eg. cation and anion) that reside in the same asymmetric unit.

Symmetry Operators:

(1)	+X,	+Y,	+Z	(2)	-X,	1/2+Y,	1/2-Z
(3)	-X,	-Y,	-Z	(4)	+X,	1/2-Y,	1/2+Z

Table 7. Least Squares Planes

Plane number 1	
Atoms defining plane	Distance
C(1)	-0.000(3)
C(2)	0.004(3)
C(3)	-0.004(4)
C(5)	0.004(4)
C(6)	-0.004(3)

Additional Atoms	Distance
Ge(1)	0.061
N(1)	0.188

Summary		
plane	mean deviation	χ^2
1	0.0031	4.9