

Inorganic Chemistry

including bioinorganic chemistry

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STRUCTURE DETERMINATION SUMMARYCrystal Data

Empirical Formula	$C_{13} H_{35} Al_2 Cl_2 N_3$
Color; Habit	Colorless Needle
Crystal size (mm)	0.8 x 0.3 x 0.3
Crystal System	Triclinic
Space Group	$P\bar{1}$
Unit Cell Dimensions	$a = 6.9542(6) \text{ \AA}$
	$b = 12.2058(10) \text{ \AA}$
	$c = 13.2417(11) \text{ \AA}$
	$\alpha = 106.236(2)^\circ$
	$\beta = 98.885(2)^\circ$
	$\gamma = 93.807(2)^\circ$
Volume	$1059.06(15) \text{ \AA}^3$
Z	2
Formula weight	358.3
Density(calc.)	1.124 Mg/m^3
Absorption Coefficient	0.386 mm^{-1}
F(000)	388

Data Collection

Diffractionmeter Used	Siemens R3m/V
Radiation	MoK α ($\lambda = 0.71073 \text{ \AA}$)
Temperature (K)	298
Monochromator	Highly oriented graphite crystal
2θ Range	0.0 to 55.0 $^\circ$
Scan Type	$2\theta-\theta$
Scan Speed	Constant; 0.00 $^\circ$ /min. in ω
Scan Range (ω)	110.00 $^\circ$ plus K α -separation
Background Measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 0.0% of total scan time
Index Ranges	$-8 \leq h \leq 9, -15 \leq k \leq 16$ $-17 \leq l \leq 11$
Reflections Collected	6098
Independent Reflections	6098 ($R_{\text{int}} = 0.00\%$)
Observed Reflections	4358 ($F > 6.0\sigma(F)$)
Absorption Correction	N/A

Solution and Refinement

System Used	Siemens SHELXTL PLUS (PC Version)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Quantity Minimized	$\sum w(F_o - F_c)^2$
Absolute Structure	N/A
Extinction Correction	N/A
Hydrogen Atoms	Riding model, fixed isotropic U
Weighting Scheme	$w^{-1} = \sigma^2(F) + 99.0000F^2$
Number of Parameters Refined	181
Final R Indices (obs. data)	R = 6.97 %, wR = 6.97 %
R Indices (all data)	R = 9.52 %, wR = 8.34 %
Goodness-of-Fit	1.17
Largest and Mean Δ/σ	0.001, 0.000
Data-to-Parameter Ratio	24.1:1
Largest Difference Peak	0.63 eÅ ⁻³
Largest Difference Hole	-0.48 eÅ ⁻³

Table 1. Atomic coordinates ($\times 10^5$) and equivalent isotropic displacement coefficients ($\text{\AA}^2 \times 10^4$)

	x	y	z	U(eq)
Cl(1)	54410(20)	13365(11)	143257(9)	671(5)
Cl(2)	34613(21)	6106(11)	116226(10)	704(5)
Al(1)	6528(19)	30768(10)	80060(10)	430(5)
Al(2)	51050(20)	20444(11)	129469(10)	469(5)
N(1)	9775(56)	34332(28)	64316(27)	496(15)
N(2)	12093(50)	14833(26)	71207(24)	407(13)
N(3)	16031(54)	23105(29)	93261(26)	490(14)
C(1)	-21790(75)	30149(50)	79350(43)	835(28)
C(2)	20590(98)	45941(36)	88311(39)	893(28)
C(3)	-6438(87)	40060(44)	60438(43)	813(27)
C(4)	28406(86)	41645(43)	65368(45)	852(27)
C(5)	10011(72)	23076(34)	56363(31)	528(18)
C(6)	21493(70)	15766(36)	61956(32)	503(17)
C(7)	-6398(74)	6828(36)	66960(36)	656(21)
C(8)	25764(76)	9796(39)	78136(33)	596(20)
C(9)	18151(78)	10910(36)	88357(34)	597(20)
C(10)	2078(90)	23905(51)	100640(40)	870(29)
C(11)	35513(84)	28843(46)	99611(41)	860(26)
C(12)	35584(73)	33379(38)	132508(40)	653(22)
C(13)	76983(68)	23723(38)	126165(41)	648(22)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2. Bond lengths (Å)

Cl(1)-Al(2)	2.216 (2)	Cl(2)-Al(2)	2.201 (2)
Al(1)-N(1)	2.286 (4)	Al(1)-N(2)	2.067 (3)
Al(1)-N(3)	2.238 (4)	Al(1)-C(1)	1.952 (5)
Al(1)-C(2)	1.969 (4)	Al(2)-C(12)	1.953 (5)
Al(2)-C(13)	1.962 (5)	N(1)-C(3)	1.466 (7)
N(1)-C(4)	1.489 (7)	N(1)-C(5)	1.482 (5)
N(2)-C(6)	1.502 (6)	N(2)-C(7)	1.492 (5)
N(2)-C(8)	1.499 (6)	N(3)-C(9)	1.476 (5)
N(3)-C(10)	1.469 (7)	N(3)-C(11)	1.492 (6)
C(1)-H(1A)	0.960	C(1)-H(1B)	0.960
C(1)-H(1C)	0.960	C(2)-H(2A)	0.960
C(2)-H(2B)	0.960	C(2)-H(2C)	0.960
C(3)-H(3A)	0.960	C(3)-H(3B)	0.960
C(3)-H(3C)	0.960	C(4)-H(4A)	0.960
C(4)-H(4B)	0.960	C(4)-H(4C)	0.960
C(5)-H(5A)	0.960	C(5)-H(5B)	0.960
C(5)-C(6)	1.505 (7)	C(6)-H(6A)	0.960
C(6)-H(6B)	0.960	C(7)-H(7A)	0.960
C(7)-H(7B)	0.960	C(7)-H(7C)	0.960
C(8)-H(8A)	0.960	C(8)-H(8B)	0.960
C(8)-C(9)	1.503 (7)	C(9)-H(9A)	0.960
C(9)-H(9B)	0.960	C(10)-H(10A)	0.960
C(10)-H(10B)	0.960	C(10)-H(10C)	0.960
C(11)-H(11A)	0.960	C(11)-H(11B)	0.960
C(11)-H(11C)	0.960	C(12)-H(12A)	0.960
C(12)-H(12B)	0.960	C(12)-H(12C)	0.960
C(13)-H(13A)	0.960	C(13)-H(13B)	0.960
C(13)-H(13C)	0.960		

Table 3. Bond angles ($^{\circ}$)

N(1)-Al(1)-N(2)	79.3(1)	N(1)-Al(1)-N(3)	151.9(2)
N(2)-Al(1)-N(3)	80.1(1)	N(1)-Al(1)-C(1)	101.5(2)
N(2)-Al(1)-C(1)	107.1(2)	N(3)-Al(1)-C(1)	102.7(2)
N(1)-Al(1)-C(2)	91.1(2)	N(2)-Al(1)-C(2)	139.9(2)
N(3)-Al(1)-C(2)	92.5(2)	C(1)-Al(1)-C(2)	113.0(3)
Cl(1)-Al(2)-Cl(2)	103.5(1)	Cl(1)-Al(2)-C(12)	108.7(2)
Cl(2)-Al(2)-C(12)	110.3(1)	Cl(1)-Al(2)-C(13)	109.2(2)
Cl(2)-Al(2)-C(13)	108.4(1)	C(12)-Al(2)-C(13)	116.0(2)
Al(1)-N(1)-C(3)	112.2(3)	Al(1)-N(1)-C(4)	111.6(3)
C(3)-N(1)-C(4)	108.0(4)	Al(1)-N(1)-C(5)	107.0(3)
C(3)-N(1)-C(5)	109.2(3)	C(4)-N(1)-C(5)	108.7(4)
Al(1)-N(2)-C(6)	111.0(2)	Al(1)-N(2)-C(7)	110.8(3)
C(6)-N(2)-C(7)	108.6(3)	Al(1)-N(2)-C(8)	109.6(2)
C(6)-N(2)-C(8)	108.3(3)	C(7)-N(2)-C(8)	108.5(3)
Al(1)-N(3)-C(9)	107.9(2)	Al(1)-N(3)-C(10)	112.9(3)
C(9)-N(3)-C(10)	109.3(4)	Al(1)-N(3)-C(11)	110.9(3)
C(9)-N(3)-C(11)	107.7(4)	C(10)-N(3)-C(11)	108.1(4)
Al(1)-C(1)-H(1A)	109.6(2)	Al(1)-C(1)-H(1B)	109.4(2)
H(1A)-C(1)-H(1B)	109.5(1)	Al(1)-C(1)-H(1C)	109.4(1)
H(1A)-C(1)-H(1C)	109.5(1)	H(1B)-C(1)-H(1C)	109.5(1)
Al(1)-C(2)-H(2A)	109.9(2)	Al(1)-C(2)-H(2B)	108.7(2)
H(2A)-C(2)-H(2B)	109.5(1)	Al(1)-C(2)-H(2C)	109.8(2)
H(2A)-C(2)-H(2C)	109.5(1)	H(2B)-C(2)-H(2C)	109.5(1)
N(1)-C(3)-H(3A)	110.2(3)	N(1)-C(3)-H(3B)	108.2(3)
H(3A)-C(3)-H(3B)	109.5(1)	N(1)-C(3)-H(3C)	110.0(2)
H(3A)-C(3)-H(3C)	109.5(1)	H(3B)-C(3)-H(3C)	109.5(1)
N(1)-C(4)-H(4A)	109.7(3)	N(1)-C(4)-H(4B)	109.9(2)
H(4A)-C(4)-H(4B)	109.5(1)	N(1)-C(4)-H(4C)	108.8(3)
H(4A)-C(4)-H(4C)	109.5(1)	H(4B)-C(4)-H(4C)	109.5(1)
N(1)-C(5)-H(5A)	110.6(2)	N(1)-C(5)-H(5B)	109.9(2)
H(5A)-C(5)-H(5B)	108.7(1)	N(1)-C(5)-C(6)	107.8(3)
H(5A)-C(5)-C(6)	110.9(3)	H(5B)-C(5)-C(6)	108.9(2)
N(2)-C(6)-C(5)	108.2(4)	N(2)-C(6)-H(6A)	110.5(2)
C(5)-C(6)-H(6A)	111.6(2)	N(2)-C(6)-H(6B)	109.2(2)
C(5)-C(6)-H(6B)	108.7(2)	H(6A)-C(6)-H(6B)	108.5(1)
N(2)-C(7)-H(7A)	109.6(2)	N(2)-C(7)-H(7B)	109.8(2)
H(7A)-C(7)-H(7B)	109.5(1)	N(2)-C(7)-H(7C)	109.1(2)
H(7A)-C(7)-H(7C)	109.5(1)	H(7B)-C(7)-H(7C)	109.5(1)
N(2)-C(8)-H(8A)	109.2(2)	N(2)-C(8)-H(8B)	110.7(2)
H(8A)-C(8)-H(8B)	108.6(1)	N(2)-C(8)-C(9)	108.1(4)
H(8A)-C(8)-C(9)	108.7(2)	H(8B)-C(8)-C(9)	111.5(2)
N(3)-C(9)-C(8)	108.6(4)	N(3)-C(9)-H(9A)	109.7(3)
C(8)-C(9)-H(9A)	108.6(2)	N(3)-C(9)-H(9B)	110.3(2)
C(8)-C(9)-H(9B)	111.1(3)	H(9A)-C(9)-H(9B)	108.6(1)
N(3)-C(10)-H(10A)	110.1(3)	N(3)-C(10)-H(10B)	109.3(3)
H(10A)-C(10)-H(10B)	109.5(1)	N(3)-C(10)-H(10C)	108.9(2)
H(10A)-C(10)-H(10C)	109.5(1)	H(10B)-C(10)-H(10C)	109.5(1)
N(3)-C(11)-H(11A)	109.7(3)	N(3)-C(11)-H(11B)	108.9(3)
H(11A)-C(11)-H(11B)	109.5(1)	N(3)-C(11)-H(11C)	109.7(3)
H(11A)-C(11)-H(11C)	109.5(1)	H(11B)-C(11)-H(11C)	109.5(1)
Al(2)-C(12)-H(12A)	109.5(2)	Al(2)-C(12)-H(12B)	109.3(2)
H(12A)-C(12)-H(12B)	109.5(1)	Al(2)-C(12)-H(12C)	109.6(1)
H(12A)-C(12)-H(12C)	109.5(1)	H(12B)-C(12)-H(12C)	109.5(1)
Al(2)-C(13)-H(13A)	109.3(2)	Al(2)-C(13)-H(13B)	109.5(1)
H(13A)-C(13)-H(13B)	109.5(1)	Al(2)-C(13)-H(13C)	109.6(1)
H(13A)-C(13)-H(13C)	109.5(1)	H(13B)-C(13)-H(13C)	109.5(1)

Table 4. Anisotropic displacement coefficients ($\text{\AA}^2 \times 10^4$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl(1)	714(9)	837(9)	550(7)	140(7)	141(6)	322(6)
Cl(2)	803(10)	657(8)	611(8)	-134(7)	-23(7)	249(6)
Al(1)	452(8)	384(7)	434(7)	72(5)	128(6)	54(5)
Al(2)	433(8)	474(7)	538(8)	6(6)	117(6)	204(6)
N(1)	582(25)	407(19)	547(22)	58(17)	164(18)	185(17)
N(2)	495(22)	381(18)	347(18)	41(16)	94(15)	104(14)
N(3)	560(25)	529(22)	363(19)	14(18)	89(17)	112(16)
C(1)	621(38)	1219(48)	814(39)	298(35)	264(30)	422(35)
C(2)	1548(63)	427(28)	587(33)	-178(32)	16(34)	121(24)
C(3)	1057(49)	721(35)	840(39)	306(34)	263(34)	423(30)
C(4)	959(47)	674(35)	919(42)	-225(31)	311(35)	225(30)
C(5)	733(34)	489(25)	382(23)	54(23)	150(22)	140(19)
C(6)	669(32)	502(25)	412(23)	178(23)	231(22)	157(20)
C(7)	824(39)	497(27)	556(29)	-215(25)	55(26)	113(22)
C(8)	790(37)	573(28)	488(27)	297(26)	127(24)	203(22)
C(9)	855(39)	510(27)	466(27)	76(25)	81(25)	231(22)
C(10)	1051(50)	1181(49)	570(33)	353(40)	426(33)	365(32)
C(11)	839(45)	919(40)	704(36)	-199(33)	-189(31)	295(31)
C(12)	620(34)	600(30)	843(36)	137(25)	254(27)	300(26)
C(13)	535(32)	523(27)	965(39)	-14(23)	320(28)	264(26)

The anisotropic displacement exponent takes the form:

$$-2\pi^2 (h^2 a^2 U_{11} + \dots + 2hka*b*U_{12})$$

Table 5. H-Atom coordinates ($\times 10^4$) and isotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)

	x	y	z	U
H(1A)	-2512	2865	8565	80
H(1B)	-2595	3737	7890	80
H(1C)	-2823	2413	7314	80
H(2A)	2157	4690	9583	80
H(2B)	3349	4642	8661	80
H(2C)	1371	5187	8647	80
H(3A)	-464	4145	5384	80
H(3B)	-1840	3512	5928	80
H(3C)	-709	4722	6566	80
H(4A)	2934	4300	5865	80
H(4B)	2880	4885	7074	80
H(4C)	3919	3772	6740	80
H(5A)	1577	2406	5049	80
H(5B)	-313	1937	5365	80
H(6A)	2196	828	5720	80
H(6B)	3465	1947	6460	80
H(7A)	-351	-51	6288	80
H(7B)	-1241	593	7278	80
H(7C)	-1520	994	6244	80
H(8A)	3851	1408	7981	80
H(8B)	2685	193	7450	80
H(9A)	560	641	8670	80
H(9B)	2682	813	9322	80
H(10A)	675	2056	10618	80
H(10B)	59	3183	10378	80
H(10C)	-1035	1986	9671	80
H(11A)	3933	2547	10525	80
H(11B)	4501	2783	9498	80
H(11C)	3478	3689	10264	80
H(12A)	3403	3652	12659	80
H(12B)	2293	3077	13357	80
H(12C)	4215	3919	13885	80
H(13A)	7556	2679	12019	80
H(13B)	8500	2923	13226	80
H(13C)	8307	1677	12441	80

