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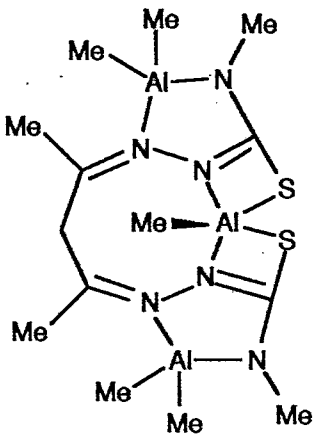
X-ray Structure Determination of Compound 3081



Compound 3081, $Al_3C_{14}H_{29}N_6S_2$, crystallizes in the orthorhombic space group $Cmc2_1$ (systematic absences hkl: h+k=odd and h0l: l=odd) with a=24.956(3)Å, b=7.1212(8)Å, c=12.744(2)Å, V=2264.8(4)Å^3, Z=4 and d_{calc}=1.251 g/cm^3. X-ray intensity data were collected on an Rigaku/RAXIS IIc area detector employing graphite-monochromated Mo-K_α radiation (λ=0.71069 Å) at a temperature of 228°K. Indexing was performed from a series of 1° oscillation images with exposures of 5 minutes per frame. A hemispere of data was collected using 8° oscillation angles with exposures of 3 minutes per frame and a crystal-to-detector distance of 82 mm. Oscillation images were processed using bioteX [1], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the teXsan[2] program package for further processing and structure solution and refinement on a Silicon Graphics Indigo R4000 computer. A total of 9353 reflections were measured over the ranges: $0.0 \le 2\theta \le 49.9°$, $-29 \le h \le 29$, $0 \le k \le 8$, $-15 \le l \le 15$.

The intensity data were corrected for Lorentz and polarization effects but not for absorption. Of the reflections measured a total of 974 unique reflections with $F^2 > 3.0\sigma(F^2)$ were used during subsequent structure refinement.

The structure was solved by direct methods (SIR92[3]). Refinement was by full-matrix least squares techniques based on F to minimize the quantity $\Sigma w(|F_o|-|F_c|)^2$ with $w=1/\sigma^2(F)$. Non-hydrogen

atoms were refined anisotropically and hydrogen atoms were included as constant contributions to the structure factors and were not refined. Refinement converged to R_1=0.0333 and R_2=0.0401.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Table 2. Anisotropic thermal parameters are in Table 3. Tables 4. and 5. list bond distances and bond angles. Figure 1. is an ORTEP[4] representation of the molecule with 30% probability thermal ellipsoids displayed.

References

1. bioteX: A suite of Programs for the Collection, Reduction and Interpretation of Imaging Plate Data, Chen, D., C.L. Day, J.D. Ferrara,T.L. Higashi, J.W. Pflugrath, B.D. Santarsiero, P.N. Swepston, J.M. Troup, B.R. Vincent and L. Xiong (1995).

2. teXsan: Single Crystal Structure Analysis Software, Version 1.7 (1995), Molecular Structure Corporation, The Woodlands, TX 77381.

3. "SIR92": Altomare, A., Burla, M.C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A., Polidoro, G. (1994). *J. Appl. Cryst.*, in preparation.

4. "ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

Table 1. Summary of Structure Determination of Compound 3081

Formula:	$Al_3C_{14}H_{29}N_6S_2$
Formula weight:	426.49
Crystal class:	orthorhombic
Space group:	$Cmc2_1$ (#36)
Z	4
Cell constants:	
a	24.956(3)Å
b	7.1212(8)Å
c	12.744(2)Å
V	2264.8(4)Å^3
μ	3.62 cm^{-1}
crystal size, mm	0.28 x 0.25 x 0.17
D_{calc}	1.251 g/cm^3
F(000)	118
Radiation:	Mo-K_α (λ=0.71069Å)
2θ range	0.0–49.9°
hkl collected	±29, +8, ±15
No. reflections measured:	9353
No. unique reflections:	1158 (R_{merge}=0.0518)
No. reflections used in refinement	974 ($F^2>3.0\sigma$)
No. parameters	118
Data/Parameter Ratio	8.3
R_1:	0.0333
R_2:	0.0401
GOF:	1.80
Final Difference Peaks, e/Å^3	+.18, -.21

Table 2. Refined Positional Parameters for Compound 3081

Atom	x	y	z	B_{eq}, Å^2
S6	0.07186(4)	0.0238(1)	0.6231	3.34(2)
Al1	0.0000	0.0441(2)	0.7495(1)	2.60(3)
Al2	0.15446(4)	0.4058(2)	0.8409(1)	3.25(2)
N3	0.0786(1)	0.3293(4)	0.8687(3)	2.84(7)
N4	0.0597(1)	0.2081(4)	0.7912(3)	2.79(6)
N7	0.1458(1)	0.2440(5)	0.7239(3)	3.11(7)
C1	0.0000	0.2580(8)	0.9766(4)	2.8(1)
C2	0.0512(2)	0.3595(5)	0.9525(3)	2.75(8)
C5	0.0979(2)	0.1708(5)	0.7173(3)	2.89(8)
C8	0.0724(2)	0.4932(7)	1.0314(4)	3.92(10)
C9	0.1602(2)	0.6673(7)	0.8011(4)	4.9(1)
C10	0.1995(2)	0.2997(8)	0.9504(4)	4.7(1)
C11	0.0000	-0.1882(8)	0.8284(5)	3.5(1)
C12	0.1860(2)	0.1967(7)	0.6444(4)	4.3(1)
H1a	0.0000	0.1169	0.9374	5.1961
H1b	0.0000	0.2483	1.0569	5.1961
H8a	0.0469	0.5916	1.0435	4.8464
H8b	0.0797	0.4312	1.0949	4.8464
H8c	0.1048	0.5503	1.0056	4.8464
H9a	0.1300	0.7052	0.7593	5.8192
H9b	0.1920	0.6903	0.7614	5.8192
H9c	0.1611	0.7469	0.8621	5.8192
H10a	0.1802	0.3025	1.0160	5.8859

H10b	0.2312	0.3705	0.9576	5.8859
H10c	0.2078	0.1729	0.9344	5.8859
H11a	0.0000	-0.2941	0.7842	4.6342
H11b	-0.0317	-0.1967	0.8719	4.6342
H12c	0.1718	0.2179	0.5760	5.1493
H12a	0.2169	0.2732	0.6540	5.1493
H12b	0.1959	0.0682	0.6509	5.1493

Hydrogen atoms were not refined.

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

Table 3. Refined Thermal Parameters (U's) for Compound 3081

Name	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S6	0.0400(5)	0.0478(6)	0.0391(5)	-0.0038(4)	0.0033(5)	-0.0069(5)
Al1	0.0323(8)	0.0321(8)	0.0344(9)	0.0000	0.0000	-0.0005(7)
Al2	0.0317(6)	0.0472(6)	0.0447(7)	-0.0059(5)	-0.0052(6)	0.0015(6)
N3	0.036(2)	0.036(2)	0.036(2)	-0.002(1)	-0.003(1)	-0.001(1)
N4	0.033(2)	0.039(2)	0.034(2)	-0.001(1)	0.002(1)	-0.006(1)
N7	0.027(2)	0.051(2)	0.040(2)	0.001(1)	0.002(1)	0.000(1)
C1	0.036(3)	0.041(3)	0.029(3)	0.0000	0.0000	0.003(2)
C2	0.038(2)	0.032(2)	0.034(2)	0.003(2)	-0.005(2)	-0.002(2)
C5	0.033(2)	0.035(2)	0.042(2)	0.001(2)	0.000(2)	0.004(2)
C8	0.050(2)	0.054(3)	0.045(2)	-0.005(2)	-0.005(2)	-0.013(2)
C9	0.060(3)	0.056(3)	0.069(3)	-0.014(2)	-0.006(3)	0.006(2)
C10	0.044(3)	0.079(4)	0.056(3)	0.006(2)	-0.007(2)	0.001(3)
C11	0.044(3)	0.037(3)	0.053(4)	0.0000	0.0000	0.000(3)
C12	0.035(2)	0.077(3)	0.052(3)	-0.003(2)	0.010(2)	-0.005(2)

The form of the anisotropic displacement parameter is:
$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+ 2a^*b^*U_{12}hk+2a^*c^*U_{13}hl+2b^*c^*U_{23}kl)]$.

Table 4. Bond Distances in Compound 3081, Å

S6—Al1	2.414(1)	S6—C5	1.720(4)	Al1—N4	1.967(3)
Al1—N4	1.967(3)	Al1—C11	1.936(6)	Al2—N3	2.001(3)
Al2—N7	1.897(4)	Al2—C9	1.936(5)	Al2—C10	1.944(5)
N3—N4	1.394(4)	N3—C2	1.287(5)	N4—C5	1.364(5)
N7—C5	1.307(5)	N7—C12	1.465(6)	C1—C2	1.499(5)
C1—C2	1.499(5)	C2—C8	1.483(6)		

Table 5. Bond Angles in Compound 3081, °

Al1—S6—C5	77.2(1)	S6—Al1—S6	95.9(1)	S6—Al1—N4	69.7(1)
S6—Al1—N4	141.1(1)	S6—Al1—C11	107.2(1)	S6—Al1—N4	141.1(1)
S6—Al1—N4	69.7(1)	S6—Al1—C11	107.2(1)	N4—Al1—N4	98.6(2)
N4—Al1—C11	111.5(2)	N4—Al1—C11	111.5(2)	N3—Al2—N7	82.3(1)
N3—Al2—C9	112.2(2)	N3—Al2—C10	108.3(2)	N7—Al2—C9	112.8(2)
N7—Al2—C10	113.2(2)	C9—Al2—C10	121.2(2)	Al2—N3—N4	111.3(2)
Al2—N3—C2	127.2(3)	N4—N3—C2	120.8(3)	Al1—N4—N3	144.6(2)
Al1—N4—C5	103.1(2)	N3—N4—C5	111.9(3)	Al2—N7—C5	113.4(3)
Al2—N7—C12	127.2(3)	C5—N7—C12	119.4(3)	C2—C1—C2	116.8(5)
N3—C2—C1	122.9(4)	N3—C2—C8	118.6(4)	C1—C2—C8	118.4(4)
S6—C5—N4	109.7(3)	S6—C5—N7	129.2(3)	N4—C5—N7	121.1(3)