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Tris(6-*tert*-butyl-4-methylpyridazine-3-thiolato-κ^2N,S)aluminium

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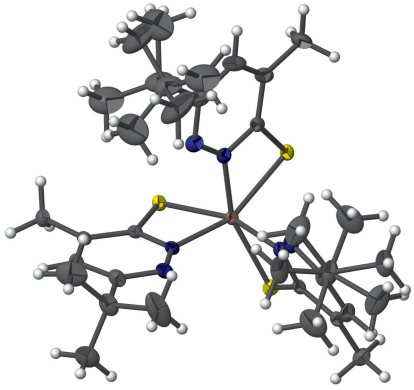
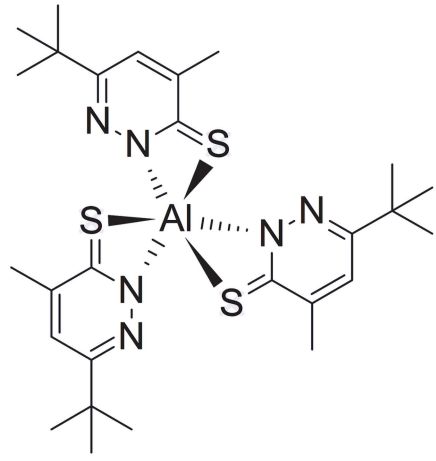
Structural data: full structural data are available from iucrdata.iucr.org

The title complex, $[Al(C_9H_{13}N_2S)_3]$, is composed of an aluminium atom coordinated by three bidentate thiopyridazine ligands in an octahedral environment. It has approximate C_3 symmetry, with Al—N distances in the range 1.9732 (17)–1.9794 (17) Å and three Al—S distances in the range 2.3961 (8)–2.4354 (8) Å. In the crystal, there are no significant intermolecular interactions present.

3D view



Chemical scheme



Structure description

The crystal structure analysis of the title compound, Fig. 1, confirmed it to be tris(6-*tert*-butyl-4-methylpyridazine-3-thiolato-*N*,*S*)-aluminium. It is the first crystal structure determination of a compound where an Al atom is surrounded by three S and three N atoms (Fig. 1 and Table 1). All atoms lie on general positions. In the octahedral environment [the *trans* N—Al—S bond angles vary between 158.12 (6) and 161.99 (6)°] small bond angles at the S atoms are observed [76.12 (7)–76.53 (7)°]. The sums of the three bond angles around the coordinating N atoms are in the range 358.97–360.00°. The S—C—N angles [110.84 (14)–111.06 (15)°] are much smaller than the S—C—C angles [128.35 (16)–128.78 (16)°]. The mean planes through the pyridazine rings enclose angles of 63.23 (9), 81.68 (10) and 89.86 (10)°. Further geometrical parameters are available in the archived CIF.

In the crystal, there are no significant intermolecular interactions present.

Synthesis and crystallization

To a solution of lithiumaluminium hydride in THF (12 ml, 0.15 *M*, 1.8 mmol) a solution of 6-(*tert*-butyl)-4-methylpyridazine-3(2*H*)-thione (1.00 g, 5.48 mmol), prepared by a reported procedure (Holler *et al.*, 2016), in 5 ml THF was added dropwise using a syringe

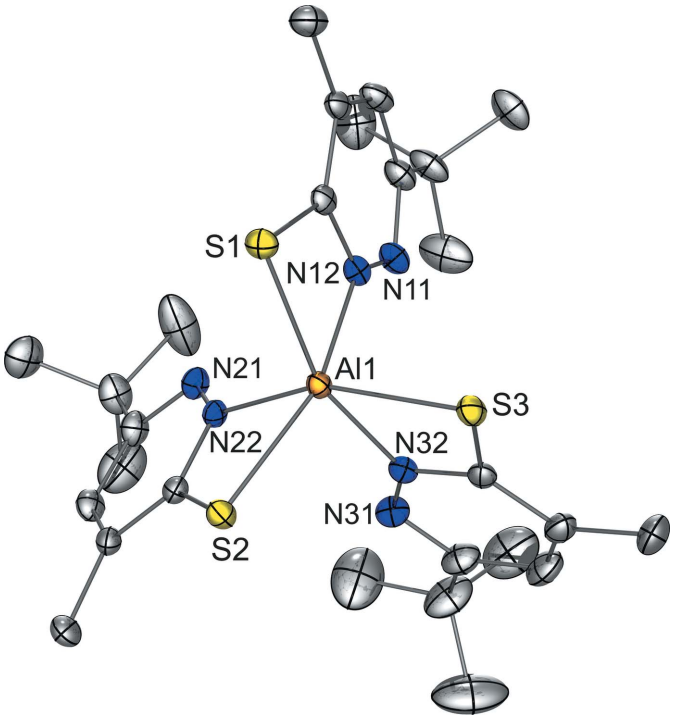


Figure 1
The molecular structure of the title compound, with atom labelling and displacement ellipsoids drawn at the 50% probability level. The H atoms and the minor occupancy orientations (16.2%) of the disordered *tert*-butyl groups have been omitted for clarity.

under a nitrogen atmosphere over a period of 30 min, whereupon gas evolution was observed. After 1 h of stirring gas evolution ceased. The reaction mixture was concentrated to 10 ml, frozen with liquid nitrogen and layered with heptane. Upon warming to room temperature pale-yellow crystals formed which were isolated *via* inert filtration and dried *in vacuo* (yield 501 mg, 49%). Single crystals suitable for X-ray diffraction analysis were obtained by layering of a THF solution with heptane. ^{1}H NMR (300 MHz, CD_2Cl_2) δ 7.26 (*d*, J = 1.0 Hz, 3H), 2.31 (*d*, J = 1.0 Hz, 9H), 2.31 (*s*, 27H) p.p.m.; ^{13}C NMR (75 MHz, CD_2Cl_2) δ 175.8, 165.0, 141.1, 126.3, 36.6, 29.8, 19.5 p.p.m. Elemental analysis: calculated for $C_{27}H_{39}AlN_6S_3$: C: 56.81 H: 6.89 N: 14.72; found: C: 56.67 H: 6.53 N: 14.65.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Two of the three *tert*-butyl groups are disordered over two orientations (C30/C37–C39: C60/C67–C69 and C17–C19: C47–C49), and both have a refined occupancy ratio of 0.838 (2): 0.162 (2). Their C—C bond lengths were refined with distance restraints [C—C = 1.540 (4) Å] and the same anisotropic displacement parameters were used for equivalent C atoms (using restraint EADP).

Table 1
Selected geometric parameters (Å, °).

Al1—N12	1.9732 (17)	Al1—S1	2.3961 (8)
Al1—N32	1.9750 (18)	Al1—S2	2.4029 (8)
Al1—N22	1.9794 (17)	Al1—S3	2.4354 (8)
N12—Al1—N32	95.97 (7)	N22—Al1—S2	70.15 (5)
N12—Al1—N22	94.89 (7)	N12—Al1—S3	97.64 (5)
N32—Al1—N22	91.45 (7)	N32—Al1—S3	69.49 (5)
N12—Al1—S1	70.44 (5)	N22—Al1—S3	158.12 (6)
N32—Al1—S1	160.48 (6)	S1—Al1—S3	97.76 (3)
N22—Al1—S1	103.33 (5)	S1—Al1—S2	102.49 (3)
N12—Al1—S2	161.99 (6)	S2—Al1—S3	99.76 (3)
N32—Al1—S2	94.43 (5)		

Table 2
Experimental details.

Crystal data	
Chemical formula	$[Al(C_9H_{13}N_2S)_3]$
M_r	570.80
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	100
a, b, c (Å)	15.988 (1), 12.2027 (8), 17.2571 (11)
β (°)	102.5536 (19)
V (Å^3)	3286.3 (4)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.28
Crystal size (mm)	0.35 × 0.26 × 0.19
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2012)
T_{min}, T_{max}	0.732, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	55131, 6448, 5208
R_{int}	0.064
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.117, 1.06
No. of reflections	6448
No. of parameters	368
No. of restraints	14
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.56, −0.40

Computer programs: *APEX2* (Bruker, 2012), *SAINT* (Bruker, 2012), *SHELXS97* (Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 2012), *SHELXL2014* (Sheldrick, 2015) and *publCIF* (Westrip, 2010).

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161986 [https://doi.org/10.1107/S2414314616019866]

Tris(6-*tert*-butyl-4-methylpyridazine-3-thiolato-κ^2N,S)aluminium

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Tris(6-*tert*-butyl-4-methylpyridazine-3-thiolato-κ^2N,S)aluminium

Crystal data

$[Al(C_9H_{13}N_2S)_3]$
M_r = 570.80
Monoclinic, $P2_1/n$
a = 15.988 (1) Å
b = 12.2027 (8) Å
c = 17.2571 (11) Å
β = 102.5536 (19)°
V = 3286.3 (4) Å^3
Z = 4
$F(000)$ = 1216
D_x = 1.154 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9768 reflections
θ = 2.4–30.6°
μ = 0.28 mm^{-1}
T = 100 K
Needle, colourless
0.35 × 0.26 × 0.19 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: Incoatec microfocus sealed tube
Multilayer monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
T_{min} = 0.732, T_{max} = 1.000
55131 measured reflections
6448 independent reflections
5208 reflections with $I > 2\sigma(I)$
R_{int} = 0.064
θ_{max} = 26.0°, θ_{min} = 2.4°
h = −19→19
k = −15→15
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.117
S = 1.06
6448 reflections
368 parameters
14 restraints
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.061P)^2 + 1.2153P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Al1	0.76332 (4)	0.58841 (5)	0.56278 (3)	0.01481 (15)	
S1	0.71695 (3)	0.40203 (4)	0.54122 (3)	0.01897 (13)	
N11	0.61059 (11)	0.65871 (15)	0.43242 (10)	0.0211 (4)	
N12	0.65544 (10)	0.58308 (13)	0.48155 (10)	0.0168 (4)	
C13	0.63753 (12)	0.47544 (17)	0.47782 (11)	0.0165 (4)	
C14	0.56245 (13)	0.43596 (17)	0.42610 (12)	0.0187 (4)	
C15	0.51718 (13)	0.51276 (18)	0.37640 (12)	0.0230 (5)	
H15	0.466487	0.491430	0.339704	0.028*	
C16	0.54405 (13)	0.62286 (18)	0.37839 (12)	0.0227 (5)	
C11	0.53782 (14)	0.31762 (18)	0.42748 (13)	0.0254 (5)	
H111	0.486454	0.303984	0.385928	0.038*	
H112	0.525967	0.299701	0.479406	0.038*	
H113	0.584933	0.271781	0.418022	0.038*	
C10	0.50024 (13)	0.70679 (18)	0.31680 (13)	0.0297 (5)	
C17	0.5436 (2)	0.8191 (2)	0.3309 (2)	0.0461 (9)	0.841 (2)
H171	0.516889	0.869349	0.288415	0.069*	0.841 (2)
H172	0.604651	0.811556	0.331211	0.069*	0.841 (2)
H173	0.536856	0.848137	0.382151	0.069*	0.841 (2)
C18	0.5080 (2)	0.6643 (3)	0.23447 (15)	0.0458 (9)	0.841 (2)
H181	0.480933	0.592046	0.225120	0.069*	0.841 (2)
H182	0.568625	0.658483	0.232571	0.069*	0.841 (2)
H183	0.479266	0.715445	0.193422	0.069*	0.841 (2)
C19	0.40598 (15)	0.7183 (3)	0.31842 (18)	0.0337 (7)	0.841 (2)
H191	0.400604	0.746885	0.370168	0.051*	0.841 (2)
H192	0.378161	0.646396	0.309624	0.051*	0.841 (2)
H193	0.378406	0.768867	0.276506	0.051*	0.841 (2)
C47	0.4114 (7)	0.6673 (14)	0.2741 (11)	0.0461 (9)	0.159 (2)
H471	0.383749	0.629699	0.312043	0.069*	0.159 (2)
H472	0.376560	0.730360	0.251510	0.069*	0.159 (2)
H473	0.417055	0.616632	0.231530	0.069*	0.159 (2)
C48	0.4688 (12)	0.7973 (10)	0.3655 (8)	0.0458 (9)	0.159 (2)
H481	0.424967	0.767495	0.391379	0.069*	0.159 (2)
H482	0.517120	0.824740	0.405868	0.069*	0.159 (2)
H483	0.444309	0.857543	0.330163	0.069*	0.159 (2)
C49	0.5662 (7)	0.7688 (13)	0.2824 (9)	0.0337 (7)	0.159 (2)
H491	0.617163	0.782787	0.324368	0.051*	0.159 (2)
H492	0.582293	0.724979	0.240311	0.051*	0.159 (2)
H493	0.541798	0.838707	0.260291	0.051*	0.159 (2)
S2	0.91122 (3)	0.56837 (4)	0.62863 (3)	0.01773 (13)	
N21	0.82111 (11)	0.63942 (14)	0.40872 (10)	0.0197 (4)	
N22	0.83734 (10)	0.60975 (13)	0.48577 (9)	0.0153 (4)	
C23	0.91589 (12)	0.60785 (16)	0.53315 (11)	0.0157 (4)	
C24	0.98881 (13)	0.63743 (17)	0.50370 (12)	0.0180 (4)	
C25	0.97254 (13)	0.66536 (17)	0.42486 (12)	0.0215 (5)	
H25	1.018864	0.684156	0.401036	0.026*	

C26	0.88824 (14)	0.66664 (18)	0.37860 (12)	0.0223 (5)	
C21	1.07636 (13)	0.64025 (19)	0.55763 (13)	0.0238 (5)	
H211	1.120008	0.643103	0.525645	0.036*	
H212	1.084765	0.574212	0.590736	0.036*	
H213	1.081369	0.705266	0.591666	0.036*	
C20	0.86934 (15)	0.6991 (2)	0.29062 (13)	0.0289 (5)	
C27	0.77298 (18)	0.7129 (3)	0.25814 (16)	0.0581 (9)	
H271	0.743738	0.643695	0.263592	0.087*	
H272	0.762459	0.733537	0.201961	0.087*	
H273	0.751017	0.770406	0.288048	0.087*	
C28	0.9142 (2)	0.8085 (2)	0.28145 (16)	0.0516 (8)	
H281	0.896815	0.863687	0.316089	0.077*	
H282	0.897826	0.832997	0.226138	0.077*	
H283	0.976418	0.798378	0.296271	0.077*	
C29	0.90403 (17)	0.6095 (2)	0.24429 (14)	0.0371 (6)	
H291	0.965598	0.600283	0.265780	0.056*	
H292	0.894380	0.630428	0.188213	0.056*	
H293	0.874374	0.540429	0.249164	0.056*	
S3	0.70105 (3)	0.62550 (4)	0.67696 (3)	0.01938 (14)	
N31	0.78657 (11)	0.83478 (14)	0.54601 (10)	0.0220 (4)	
N32	0.76034 (10)	0.74769 (14)	0.58233 (10)	0.0175 (4)	
C33	0.72259 (12)	0.75455 (17)	0.64423 (12)	0.0181 (4)	
C34	0.70612 (14)	0.85842 (19)	0.67489 (13)	0.0251 (5)	
C35	0.73226 (16)	0.94609 (19)	0.63753 (14)	0.0314 (5)	
H35	0.722866	1.017935	0.655067	0.038*	
C36	0.77299 (15)	0.93278 (17)	0.57335 (13)	0.0286 (5)	
C31	0.66316 (16)	0.8660 (2)	0.74358 (14)	0.0350 (6)	
H311	0.651416	0.943024	0.753331	0.053*	
H312	0.700757	0.834945	0.790982	0.053*	
H313	0.609169	0.825038	0.731419	0.053*	
C30	0.7984 (2)	1.0306 (3)	0.5282 (2)	0.0423 (9)	0.841 (2)
C37	0.8417 (3)	0.9935 (3)	0.4620 (3)	0.0714 (13)	0.841 (2)
H371	0.895018	0.954864	0.485083	0.107*	0.841 (2)
H372	0.854526	1.057562	0.432372	0.107*	0.841 (2)
H373	0.803220	0.944145	0.425965	0.107*	0.841 (2)
C38	0.7172 (3)	1.0936 (3)	0.4887 (2)	0.0601 (11)	0.841 (2)
H381	0.679118	1.045180	0.451616	0.090*	0.841 (2)
H382	0.732891	1.156789	0.459852	0.090*	0.841 (2)
H383	0.687599	1.119089	0.529545	0.090*	0.841 (2)
C39	0.8542 (3)	1.1073 (4)	0.5868 (3)	0.0766 (18)	0.841 (2)
H391	0.824783	1.125702	0.629389	0.115*	0.841 (2)
H392	0.864916	1.174456	0.559405	0.115*	0.841 (2)
H393	0.908767	1.071294	0.609538	0.115*	0.841 (2)
C60	0.8167 (11)	1.0288 (11)	0.5409 (12)	0.0423 (9)	0.159 (2)
C67	0.822 (2)	1.130 (2)	0.594 (2)	0.0714 (13)	0.159 (2)
H671	0.849940	1.189730	0.571655	0.107*	0.159 (2)
H672	0.763756	1.152641	0.597168	0.107*	0.159 (2)
H673	0.854754	1.112339	0.647364	0.107*	0.159 (2)

C68	0.9072 (12)	1.0019 (16)	0.5295 (12)	0.0601 (11)	0.159 (2)
H681	0.904846	0.937163	0.495481	0.090*	0.159 (2)
H682	0.929525	1.064330	0.504427	0.090*	0.159 (2)
H683	0.945048	0.987172	0.581214	0.090*	0.159 (2)
C69	0.7560 (18)	1.049 (2)	0.4597 (13)	0.0766 (18)	0.159 (2)
H691	0.753774	0.983398	0.426662	0.115*	0.159 (2)
H692	0.698470	1.065984	0.467373	0.115*	0.159 (2)
H693	0.777346	1.111005	0.433271	0.115*	0.159 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Al1	0.0136 (3)	0.0174 (3)	0.0134 (3)	0.0000 (2)	0.0030 (2)	0.0004 (2)
S1	0.0190 (3)	0.0186 (3)	0.0182 (3)	0.0000 (2)	0.0015 (2)	0.0020 (2)
N11	0.0169 (9)	0.0245 (10)	0.0213 (9)	0.0018 (7)	0.0028 (7)	0.0052 (8)
N12	0.0156 (8)	0.0198 (9)	0.0151 (9)	0.0011 (7)	0.0037 (7)	0.0028 (7)
C13	0.0166 (10)	0.0214 (11)	0.0131 (10)	−0.0004 (8)	0.0066 (8)	0.0002 (8)
C14	0.0163 (10)	0.0252 (11)	0.0161 (10)	−0.0029 (8)	0.0068 (8)	−0.0032 (8)
C15	0.0161 (10)	0.0338 (13)	0.0177 (11)	−0.0038 (9)	0.0007 (8)	0.0007 (9)
C16	0.0163 (10)	0.0299 (12)	0.0212 (11)	0.0000 (9)	0.0024 (9)	0.0047 (9)
C11	0.0252 (11)	0.0268 (12)	0.0234 (11)	−0.0061 (10)	0.0038 (9)	−0.0018 (9)
C10	0.0171 (11)	0.0379 (14)	0.0314 (13)	−0.0005 (10)	−0.0008 (9)	0.0127 (11)
C17	0.0374 (17)	0.0386 (18)	0.053 (2)	−0.0008 (14)	−0.0113 (15)	0.0233 (16)
C18	0.055 (2)	0.058 (2)	0.0260 (16)	0.0179 (17)	0.0122 (14)	0.0197 (15)
C19	0.0200 (14)	0.0457 (19)	0.0333 (16)	0.0078 (12)	0.0010 (11)	0.0162 (14)
C47	0.0374 (17)	0.0386 (18)	0.053 (2)	−0.0008 (14)	−0.0113 (15)	0.0233 (16)
C48	0.055 (2)	0.058 (2)	0.0260 (16)	0.0179 (17)	0.0122 (14)	0.0197 (15)
C49	0.0200 (14)	0.0457 (19)	0.0333 (16)	0.0078 (12)	0.0010 (11)	0.0162 (14)
S2	0.0156 (3)	0.0247 (3)	0.0127 (2)	−0.0001 (2)	0.00274 (19)	0.00076 (19)
N21	0.0235 (9)	0.0218 (9)	0.0144 (9)	0.0025 (7)	0.0054 (7)	0.0007 (7)
N22	0.0165 (8)	0.0174 (9)	0.0124 (8)	0.0012 (7)	0.0042 (6)	−0.0005 (7)
C23	0.0175 (10)	0.0146 (10)	0.0153 (10)	0.0012 (8)	0.0041 (8)	−0.0013 (8)
C24	0.0176 (10)	0.0177 (11)	0.0198 (10)	0.0002 (8)	0.0067 (8)	−0.0029 (8)
C25	0.0215 (11)	0.0215 (11)	0.0244 (11)	−0.0018 (9)	0.0113 (9)	0.0018 (9)
C26	0.0264 (11)	0.0234 (11)	0.0191 (11)	0.0025 (9)	0.0095 (9)	0.0030 (9)
C21	0.0174 (10)	0.0270 (12)	0.0278 (12)	−0.0024 (9)	0.0072 (9)	−0.0020 (9)
C20	0.0308 (13)	0.0380 (14)	0.0193 (11)	0.0061 (10)	0.0090 (9)	0.0103 (10)
C27	0.0426 (17)	0.107 (3)	0.0257 (14)	0.0300 (17)	0.0104 (12)	0.0270 (16)
C28	0.086 (2)	0.0422 (17)	0.0306 (15)	0.0010 (16)	0.0209 (15)	0.0120 (12)
C29	0.0476 (16)	0.0438 (15)	0.0207 (12)	0.0038 (12)	0.0090 (11)	0.0023 (11)
S3	0.0174 (3)	0.0247 (3)	0.0172 (3)	−0.0027 (2)	0.0061 (2)	−0.0011 (2)
N31	0.0249 (10)	0.0188 (9)	0.0217 (9)	−0.0006 (7)	0.0035 (7)	0.0031 (7)
N32	0.0160 (8)	0.0185 (9)	0.0172 (9)	−0.0009 (7)	0.0024 (7)	0.0004 (7)
C33	0.0135 (10)	0.0242 (11)	0.0150 (10)	0.0010 (8)	−0.0006 (8)	−0.0030 (8)
C34	0.0236 (11)	0.0288 (12)	0.0209 (11)	0.0022 (9)	0.0002 (9)	−0.0077 (9)
C35	0.0397 (14)	0.0217 (12)	0.0307 (13)	0.0048 (10)	0.0030 (11)	−0.0071 (10)
C36	0.0354 (13)	0.0199 (12)	0.0285 (13)	−0.0008 (10)	0.0023 (10)	−0.0003 (9)
C31	0.0364 (14)	0.0395 (15)	0.0316 (14)	0.0002 (11)	0.0127 (11)	−0.0158 (11)

C30	0.069 (2)	0.0206 (13)	0.036 (2)	−0.0055 (14)	0.0095 (19)	0.0035 (12)
C37	0.104 (3)	0.045 (2)	0.081 (3)	−0.008 (2)	0.053 (3)	0.017 (2)
C38	0.090 (3)	0.037 (2)	0.048 (2)	0.0076 (19)	0.004 (2)	0.0146 (17)
C39	0.099 (4)	0.062 (3)	0.060 (3)	−0.051 (3)	−0.003 (3)	0.011 (2)
C60	0.069 (2)	0.0206 (13)	0.036 (2)	−0.0055 (14)	0.0095 (19)	0.0035 (12)
C67	0.104 (3)	0.045 (2)	0.081 (3)	−0.008 (2)	0.053 (3)	0.017 (2)
C68	0.090 (3)	0.037 (2)	0.048 (2)	0.0076 (19)	0.004 (2)	0.0146 (17)
C69	0.099 (4)	0.062 (3)	0.060 (3)	−0.051 (3)	−0.003 (3)	0.011 (2)

Geometric parameters (Å, º)

Al1—N12	1.9732 (17)	C21—H211	0.9800
Al1—N32	1.9750 (18)	C21—H212	0.9800
Al1—N22	1.9794 (17)	C21—H213	0.9800
Al1—S1	2.3961 (8)	C20—C29	1.528 (3)
Al1—S2	2.4029 (8)	C20—C27	1.530 (3)
Al1—S3	2.4354 (8)	C20—C28	1.539 (4)
S1—C13	1.734 (2)	C27—H271	0.9800
N11—C16	1.327 (3)	C27—H272	0.9800
N11—N12	1.349 (2)	C27—H273	0.9800
N12—C13	1.343 (3)	C28—H281	0.9800
C13—C14	1.416 (3)	C28—H282	0.9800
C14—C15	1.367 (3)	C28—H283	0.9800
C14—C11	1.498 (3)	C29—H291	0.9800
C15—C16	1.409 (3)	C29—H292	0.9800
C15—H15	0.9500	C29—H293	0.9800
C16—C10	1.532 (3)	S3—C33	1.733 (2)
C11—H111	0.9800	N31—C36	1.321 (3)
C11—H112	0.9800	N31—N32	1.346 (2)
C11—H113	0.9800	N32—C33	1.339 (3)
C10—C19	1.520 (3)	C33—C34	1.420 (3)
C10—C49	1.520 (4)	C34—C35	1.361 (3)
C10—C47	1.528 (4)	C34—C31	1.497 (3)
C10—C17	1.530 (3)	C35—C36	1.411 (3)
C10—C48	1.537 (4)	C35—H35	0.9500
C10—C18	1.542 (3)	C36—C30	1.528 (3)
C17—H171	0.9800	C36—C60	1.531 (4)
C17—H172	0.9800	C31—H311	0.9800
C17—H173	0.9800	C31—H312	0.9800
C18—H181	0.9800	C31—H313	0.9800
C18—H182	0.9800	C30—C39	1.516 (3)
C18—H183	0.9800	C30—C37	1.528 (3)
C19—H191	0.9800	C30—C38	1.536 (3)
C19—H192	0.9800	C37—H371	0.9800
C19—H193	0.9800	C37—H372	0.9800
C47—H471	0.9800	C37—H373	0.9800
C47—H472	0.9800	C38—H381	0.9800
C47—H473	0.9800	C38—H382	0.9800

C48—H481	0.9800	C38—H383	0.9800
C48—H482	0.9800	C39—H391	0.9800
C48—H483	0.9800	C39—H392	0.9800
C49—H491	0.9800	C39—H393	0.9800
C49—H492	0.9800	C60—C67	1.529 (4)
C49—H493	0.9800	C60—C68	1.539 (4)
S2—C23	1.734 (2)	C60—C69	1.542 (4)
N21—C26	1.332 (3)	C67—H671	0.9800
N21—N22	1.348 (2)	C67—H672	0.9800
N22—C23	1.342 (3)	C67—H673	0.9800
C23—C24	1.416 (3)	C68—H681	0.9800
C24—C25	1.372 (3)	C68—H682	0.9800
C24—C21	1.503 (3)	C68—H683	0.9800
C25—C26	1.409 (3)	C69—H691	0.9800
C25—H25	0.9500	C69—H692	0.9800
C26—C20	1.534 (3)	C69—H693	0.9800

N12—Al1—N32	95.97 (7)	C24—C21—H211	109.5
N12—Al1—N22	94.89 (7)	C24—C21—H212	109.5
N32—Al1—N22	91.45 (7)	H211—C21—H212	109.5
N12—Al1—S1	70.44 (5)	C24—C21—H213	109.5
N32—Al1—S1	160.48 (6)	H211—C21—H213	109.5
N22—Al1—S1	103.33 (5)	H212—C21—H213	109.5
N12—Al1—S2	161.99 (6)	C29—C20—C27	109.8 (2)
N32—Al1—S2	94.43 (5)	C29—C20—C26	108.49 (19)
N22—Al1—S2	70.15 (5)	C27—C20—C26	110.92 (18)
N12—Al1—S3	97.64 (5)	C29—C20—C28	109.7 (2)
N32—Al1—S3	69.49 (5)	C27—C20—C28	108.7 (2)
N22—Al1—S3	158.12 (6)	C26—C20—C28	109.3 (2)
S1—Al1—S3	97.76 (3)	C20—C27—H271	109.5
S1—Al1—S2	102.49 (3)	C20—C27—H272	109.5
S2—Al1—S3	99.76 (3)	H271—C27—H272	109.5
C13—S1—Al1	76.53 (7)	C20—C27—H273	109.5
C16—N11—N12	116.84 (18)	H271—C27—H273	109.5
C13—N12—N11	124.07 (17)	H272—C27—H273	109.5
C13—N12—Al1	102.16 (13)	C20—C28—H281	109.5
N11—N12—Al1	133.40 (13)	C20—C28—H282	109.5
N12—C13—C14	120.39 (18)	H281—C28—H282	109.5
N12—C13—S1	110.84 (14)	C20—C28—H283	109.5
C14—C13—S1	128.76 (16)	H281—C28—H283	109.5
C15—C14—C13	115.03 (19)	H282—C28—H283	109.5
C15—C14—C11	124.45 (19)	C20—C29—H291	109.5
C13—C14—C11	120.52 (19)	C20—C29—H292	109.5
C14—C15—C16	121.44 (19)	H291—C29—H292	109.5
C14—C15—H15	119.3	C20—C29—H293	109.5
C16—C15—H15	119.3	H291—C29—H293	109.5
N11—C16—C15	121.68 (19)	H292—C29—H293	109.5
N11—C16—C10	116.30 (19)	C33—S3—Al1	76.12 (7)

C15—C16—C10	121.99 (18)	C36—N31—N32	117.15 (18)
C14—C11—H111	109.5	C33—N32—N31	124.20 (17)
C14—C11—H112	109.5	C33—N32—Al1	103.28 (13)
H111—C11—H112	109.5	N31—N32—Al1	132.51 (13)
C14—C11—H113	109.5	N32—C33—C34	120.35 (19)
H111—C11—H113	109.5	N32—C33—S3	111.05 (15)
H112—C11—H113	109.5	C34—C33—S3	128.59 (16)
C49—C10—C47	128.3 (10)	C35—C34—C33	115.1 (2)
C19—C10—C17	109.3 (2)	C35—C34—C31	124.6 (2)
C19—C10—C16	110.77 (18)	C33—C34—C31	120.3 (2)
C49—C10—C16	110.6 (6)	C34—C35—C36	121.5 (2)
C47—C10—C16	111.1 (6)	C34—C35—H35	119.2
C17—C10—C16	111.42 (19)	C36—C35—H35	119.2
C49—C10—C48	101.1 (10)	N31—C36—C35	121.6 (2)
C47—C10—C48	96.3 (11)	N31—C36—C30	116.3 (2)
C16—C10—C48	104.8 (6)	C35—C36—C30	122.0 (2)
C19—C10—C18	109.0 (2)	N31—C36—C60	116.0 (8)
C17—C10—C18	108.9 (2)	C35—C36—C60	121.6 (8)
C16—C10—C18	107.4 (2)	C34—C31—H311	109.5
C10—C17—H171	109.5	C34—C31—H312	109.5
C10—C17—H172	109.5	H311—C31—H312	109.5
H171—C17—H172	109.5	C34—C31—H313	109.5
C10—C17—H173	109.5	H311—C31—H313	109.5
H171—C17—H173	109.5	H312—C31—H313	109.5
H172—C17—H173	109.5	C39—C30—C36	109.0 (3)
C10—C18—H181	109.5	C39—C30—C37	112.7 (3)
C10—C18—H182	109.5	C36—C30—C37	111.3 (3)
H181—C18—H182	109.5	C39—C30—C38	107.8 (3)
C10—C18—H183	109.5	C36—C30—C38	108.9 (3)
H181—C18—H183	109.5	C37—C30—C38	107.0 (3)
H182—C18—H183	109.5	C30—C37—H371	109.5
C10—C19—H191	109.5	C30—C37—H372	109.5
C10—C19—H192	109.5	H371—C37—H372	109.5
H191—C19—H192	109.5	C30—C37—H373	109.5
C10—C19—H193	109.5	H371—C37—H373	109.5
H191—C19—H193	109.5	H372—C37—H373	109.5
H192—C19—H193	109.5	C30—C38—H381	109.5
C10—C47—H471	109.5	C30—C38—H382	109.5
C10—C47—H472	109.5	H381—C38—H382	109.5
H471—C47—H472	109.5	C30—C38—H383	109.5
C10—C47—H473	109.5	H381—C38—H383	109.5
H471—C47—H473	109.5	H382—C38—H383	109.5
H472—C47—H473	109.5	C30—C39—H391	109.5
C10—C48—H481	109.5	C30—C39—H392	109.5
C10—C48—H482	109.5	H391—C39—H392	109.5
H481—C48—H482	109.5	C30—C39—H393	109.5
C10—C48—H483	109.5	H391—C39—H393	109.5
H481—C48—H483	109.5	H392—C39—H393	109.5

H482—C48—H483	109.5	C67—C60—C36	111.6 (17)
C10—C49—H491	109.5	C67—C60—C68	108.7 (17)
C10—C49—H492	109.5	C36—C60—C68	113.6 (13)
H491—C49—H492	109.5	C67—C60—C69	111 (2)
C10—C49—H493	109.5	C36—C60—C69	102.1 (13)
H491—C49—H493	109.5	C68—C60—C69	109.9 (19)
H492—C49—H493	109.5	C60—C67—H671	109.5
C23—S2—Al1	76.32 (7)	C60—C67—H672	109.5
C26—N21—N22	116.95 (17)	H671—C67—H672	109.5
C23—N22—N21	124.06 (16)	C60—C67—H673	109.5
C23—N22—Al1	101.85 (12)	H671—C67—H673	109.5
N21—N22—Al1	133.06 (13)	H672—C67—H673	109.5
N22—C23—C24	120.76 (18)	C60—C68—H681	109.5
N22—C23—S2	110.86 (14)	C60—C68—H682	109.5
C24—C23—S2	128.37 (16)	H681—C68—H682	109.5
C25—C24—C23	115.21 (19)	C60—C68—H683	109.5
C25—C24—C21	123.96 (18)	H681—C68—H683	109.5
C23—C24—C21	120.80 (18)	H682—C68—H683	109.5
C24—C25—C26	121.09 (19)	C60—C69—H691	109.5
C24—C25—H25	119.5	C60—C69—H692	109.5
C26—C25—H25	119.5	H691—C69—H692	109.5
N21—C26—C25	121.90 (19)	C60—C69—H693	109.5
N21—C26—C20	116.65 (19)	H691—C69—H693	109.5
C25—C26—C20	121.45 (19)	H692—C69—H693	109.5

C16—N11—N12—C13	1.2 (3)	C21—C24—C25—C26	−176.5 (2)
C16—N11—N12—Al1	172.83 (15)	N22—N21—C26—C25	−0.4 (3)
N11—N12—C13—C14	−7.2 (3)	N22—N21—C26—C20	179.36 (18)
Al1—N12—C13—C14	178.94 (15)	C24—C25—C26—N21	−1.0 (3)
N11—N12—C13—S1	172.02 (14)	C24—C25—C26—C20	179.2 (2)
Al1—N12—C13—S1	−1.79 (15)	N21—C26—C20—C29	−110.6 (2)
Al1—S1—C13—N12	1.48 (12)	C25—C26—C20—C29	69.1 (3)
Al1—S1—C13—C14	−179.33 (19)	N21—C26—C20—C27	10.0 (3)
N12—C13—C14—C15	6.7 (3)	C25—C26—C20—C27	−170.2 (2)
S1—C13—C14—C15	−172.47 (15)	N21—C26—C20—C28	129.9 (2)
N12—C13—C14—C11	−174.01 (18)	C25—C26—C20—C28	−50.4 (3)
S1—C13—C14—C11	6.9 (3)	C36—N31—N32—C33	−0.7 (3)
C13—C14—C15—C16	−0.8 (3)	C36—N31—N32—Al1	178.37 (16)
C11—C14—C15—C16	179.9 (2)	N31—N32—C33—C34	1.3 (3)
N12—N11—C16—C15	5.0 (3)	Al1—N32—C33—C34	−177.98 (16)
N12—N11—C16—C10	−172.97 (17)	N31—N32—C33—S3	−178.08 (14)
C14—C15—C16—N11	−5.2 (3)	Al1—N32—C33—S3	2.63 (15)
C14—C15—C16—C10	172.66 (19)	Al1—S3—C33—N32	−2.14 (13)
N11—C16—C10—C19	−122.2 (2)	Al1—S3—C33—C34	178.5 (2)
C15—C16—C10—C19	59.9 (3)	N32—C33—C34—C35	−0.8 (3)
N11—C16—C10—C49	47.7 (8)	S3—C33—C34—C35	178.52 (17)
C15—C16—C10—C49	−130.2 (8)	N32—C33—C34—C31	179.41 (19)
N11—C16—C10—C47	−163.4 (10)	S3—C33—C34—C31	−1.3 (3)

C15—C16—C10—C47	18.6 (10)	C33—C34—C35—C36	−0.3 (3)
N11—C16—C10—C17	−0.2 (3)	C31—C34—C35—C36	179.5 (2)
C15—C16—C10—C17	−178.2 (2)	N32—N31—C36—C35	−0.4 (3)
N11—C16—C10—C48	−60.5 (8)	N32—N31—C36—C30	−176.5 (2)
C15—C16—C10—C48	121.6 (8)	N32—N31—C36—C60	169.9 (9)
N11—C16—C10—C18	118.9 (2)	C34—C35—C36—N31	0.9 (4)
C15—C16—C10—C18	−59.1 (3)	C34—C35—C36—C30	176.8 (2)
C26—N21—N22—C23	1.1 (3)	C34—C35—C36—C60	−168.9 (9)
C26—N21—N22—Al1	167.23 (15)	N31—C36—C30—C39	−129.1 (3)
N21—N22—C23—C24	−0.3 (3)	C35—C36—C30—C39	54.8 (4)
Al1—N22—C23—C24	−170.06 (16)	N31—C36—C30—C37	−4.1 (4)
N21—N22—C23—S2	178.69 (14)	C35—C36—C30—C37	179.8 (3)
Al1—N22—C23—S2	8.97 (15)	N31—C36—C30—C38	113.6 (3)
Al1—S2—C23—N22	−7.43 (12)	C35—C36—C30—C38	−62.5 (4)
Al1—S2—C23—C24	171.5 (2)	N31—C36—C60—C67	−161.5 (18)
N22—C23—C24—C25	−1.0 (3)	C35—C36—C60—C67	9 (2)
S2—C23—C24—C25	−179.88 (16)	N31—C36—C60—C68	−38.2 (18)
N22—C23—C24—C21	177.21 (18)	C35—C36—C60—C68	132.1 (13)
S2—C23—C24—C21	−1.6 (3)	N31—C36—C60—C69	80.1 (17)
C23—C24—C25—C26	1.7 (3)	C35—C36—C60—C69	−109.6 (18)