JOURNAL OF THE AMERICAN CHEMICAL SOCIETY
J. Am. Chem. Soc., 1996, 118(24), 5822-5823, DOI:10.1021/ja953700q

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'Norbert Emig'
'Regis Reau'
'Harald Krautscheid'
'Dieter Fenske'
'Guy Bertrand'

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_publ_contact_author_phone        '(33) 61333124'
_publ_contact_author_fax          '(33) 61553003'
_publ_contact_author_email        gbertran@lcctoul.lcc-toulouse.fr
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The publication belonging to this cif-File is
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H10B H 0.5296(15) 0.2236(24) 0.4540(21) 0.080(7) Uiso 1 d . .
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H11B H 0.8418(19) -0.1737(29) 0.2992(23) 0.097(10) Uiso 1 d . .
H11C H 0.7507(15) -0.2148(24) 0.2807(19) 0.071(7) Uiso 1 d . .
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_diffrn_radiation_source          'fine-focus sealed tube'
_diffrn_radiation_monochromator   graphite
_diffrn_measurement_device        'Stoe-Stadi IV'
_diffrn_measurement_method        'omega-scan'
_diffrn_standards_number          3
_diffrn_standards_interval_time   '1 h'
_diffrn_standards_decay_%         3
_diffrn_reflns_number             4567
_diffrn_reflns_av_R_equivalents   0.0125
_diffrn_reflns_av_sigmaI/netI     0.0269
_diffrn_reflns_limit_h_min        0
_diffrn_reflns_limit_h_max        9
_diffrn_reflns_limit_k_min        -13
_diffrn_reflns_limit_k_max        13
_diffrn_reflns_limit_l_min        -14
_diffrn_reflns_limit_l_max        15
_diffrn_reflns_theta_min          1.67
_diffrn_reflns_theta_max          24.00
_reflns_number_total              4267
_reflns_number_observed           3312
_reflns_observed_criterion        >2sigma(I)

_computing_data_collection        'STADI IV (Stoe, Darmstadt)'
_computing_cell_refinement        'STADI IV (Stoe, Darmstadt)'
_computing_data_reduction         'XRED (Stoe, Darmstadt)'
_computing_structure_solution     'SHELXS-86 (Sheldrick, 1990)'
_computing_structure_refinement   'SHELXL-92 (Sheldrick, 1993)'
_computing_molecular_graphics     'CRYSTALS User Guide (Watkin et al., 1985)'
_computing_publication_material   'SHELXL-92 (Sheldrick, 1993)'

_refine_special_details
;
 Refinement on F^2^ for ALL reflections except for 2 with very negative F^2^
 or flagged by the user for potential systematic errors.  Weighted R-factors
 wR and all goodnesses of fit S are based on F^2^, conventional R-factors R
```

are based on F, with F set to zero for negative F^2^. The observed criterion of F^2^ > 2sigma(F^2^) is used only for calculating _R_factor_obs etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2^ are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef	Fsqd
_refine_ls_matrix_type	full
_refine_ls_weighting_scheme	'calc w=1/[s^2^(Fo^2^)+(0.0437P)^2^+1.4439P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary	direct
_atom_sites_solution_secondary	difmap
_atom_sites_solution_hydrogens	geom
_refine_ls_hydrogen_treatment	H14 located and refined, other hydrogens placed in estimated positions
_refine_ls_extinction_method	none
_refine_ls_extinction_coef	*
_refine_ls_number_reflns	4265
_refine_ls_number_parameters	248
_refine_ls_number_restraints	0
_refine_ls_R_factor_all	0.0559
_refine_ls_R_factor_obs	0.0374
_refine_ls_wR_factor_all	0.1026
_refine_ls_wR_factor_obs	0.0916
_refine_ls_goodness_of_fit_all	1.034
_refine_ls_goodness_of_fit_obs	1.060
_refine_ls_restrained_S_all	1.037
_refine_ls_restrained_S_obs	1.060
_refine_ls_shift/esd_max	0.012
_refine_ls_shift/esd_mean	0.001

loop_
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 _atom_site_refinement_flags
 _atom_site_disorder_group

Al1	Al	0.04011(10)	0.24922(7)	0.26601(7)	0.0319(2)	Uani	1	d	.	.
Cl1	Cl	0.08631(9)	0.19625(7)	0.40820(6)	0.0474(2)	Uani	1	d	.	.
N1	N	0.0830(3)	0.1296(2)	0.1729(2)	0.0347(6)	Uani	1	d	.	.
H14	H	0.1096(37)	0.1658(29)	0.1089(30)	0.051(10)	Uiso	1	d	.	.
C1	C	-0.0614(3)	0.0796(3)	0.1887(3)	0.0394(8)	Uani	1	d	.	.
H1A	H	-0.0946(3)	0.0247(3)	0.2564(3)	0.047	Uiso	1	calc	R	.
H1B	H	-0.0538(3)	0.0393(3)	0.1258(3)	0.047	Uiso	1	calc	R	.
C2	C	-0.1682(3)	0.1719(3)	0.1973(2)	0.0366(7)	Uani	1	d	.	.
H2A	H	-0.1467(3)	0.2170(3)	0.1244(2)	0.044	Uiso	1	calc	R	.
H2B	H	-0.2662(3)	0.1380(3)	0.2210(2)	0.044	Uiso	1	calc	R	.
N2	N	-0.1618(3)	0.2478(2)	0.2789(2)	0.0314(6)	Uani	1	d	.	.
C3	C	-0.1781(3)	0.3694(2)	0.2366(3)	0.0381(7)	Uani	1	d	.	.
H3A	H	-0.2103(3)	0.4144(2)	0.2987(3)	0.046	Uiso	1	calc	R	.
H3B	H	-0.2520(3)	0.3729(2)	0.2024(3)	0.046	Uiso	1	calc	R	.
C4	C	-0.0363(4)	0.4189(3)	0.1525(3)	0.0417(8)	Uani	1	d	.	.

```
H4A H -0.0174(4) 0.3891(3) 0.0820(3) 0.050 Uiso 1 calc R .
H4B H -0.0408(4) 0.5019(3) 0.1394(3) 0.050 Uiso 1 calc R .
N3 N 0.0807(3) 0.3884(2) 0.1940(2) 0.0382(6) Uani 1 d . .
Si1 Si 0.21623(11) 0.02338(8) 0.17401(7) 0.0437(2) Uani 1 d . .
C5 C 0.1357(5) -0.0795(3) 0.2976(3) 0.0661(11) Uani 1 d . .
H5A H 0.2092(8) -0.1307(15) 0.3067(13) 0.099 Uiso 1 calc R .
H5B H 0.0584(20) -0.1229(17) 0.2898(10) 0.099 Uiso 1 calc R .
H5C H 0.0967(28) -0.0396(3) 0.3621(4) 0.099 Uiso 1 calc R .
C6 C 0.3733(4) 0.1011(4) 0.1750(4) 0.0667(11) Uani 1 d . .
H6A H 0.4518(11) 0.0493(6) 0.1693(24) 0.100 Uiso 1 calc R .
H6B H 0.3478(10) 0.1341(21) 0.2436(11) 0.100 Uiso 1 calc R .
H6C H 0.4040(19) 0.1612(16) 0.1126(14) 0.100 Uiso 1 calc R .
C7 C 0.2561(4) -0.0443(3) 0.0481(3) 0.0595(10) Uani 1 d . .
H7A H 0.3257(22) -0.1032(15) 0.0466(11) 0.089 Uiso 1 calc R .
H7B H 0.2962(27) 0.0123(5) -0.0166(3) 0.089 Uiso 1 calc R .
H7C H 0.1675(7) -0.0780(19) 0.0476(11) 0.089 Uiso 1 calc R .
Si2 Si -0.30295(10) 0.21124(7) 0.41602(7) 0.0376(2) Uani 1 d . .
C8 C -0.2819(4) 0.0631(3) 0.4654(3) 0.0486(9) Uani 1 d . .
H8A H -0.3012(24) 0.0138(3) 0.4179(11) 0.073 Uiso 1 calc R .
H8B H -0.3496(17) 0.0437(6) 0.5408(7) 0.073 Uiso 1 calc R .
H8C H -0.1836(8) 0.0530(5) 0.4636(18) 0.073 Uiso 1 calc R .
C9 C -0.2758(4) 0.3079(3) 0.5064(3) 0.0520(9) Uani 1 d . .
H9A H -0.1754(7) 0.3072(16) 0.5010(15) 0.078 Uiso 1 calc R .
H9B H -0.3383(19) 0.2833(12) 0.5825(4) 0.078 Uiso 1 calc R .
H9C H -0.2994(25) 0.3845(5) 0.4835(13) 0.078 Uiso 1 calc R .
C10 C -0.4779(4) 0.2308(3) 0.4002(3) 0.0581(10) Uani 1 d . .
H10A H -0.4881(12) 0.1800(16) 0.3505(17) 0.087 Uiso 1 calc R .
H10B H -0.4838(12) 0.3090(6) 0.3697(20) 0.087 Uiso 1 calc R .
H10C H -0.5550(4) 0.2137(22) 0.4721(4) 0.087 Uiso 1 calc R .
Si3 Si 0.20450(11) 0.49259(8) 0.18238(8) 0.0478(3) Uani 1 d . .
C11 C 0.3527(5) 0.4263(4) 0.2173(4) 0.0794(14) Uani 1 d . .
H11A H 0.4202(19) 0.4849(4) 0.2173(27) 0.119 Uiso 1 calc R .
H11B H 0.4027(22) 0.3766(21) 0.1629(15) 0.119 Uiso 1 calc R .
H11C H 0.3142(6) 0.3822(23) 0.2903(12) 0.119 Uiso 1 calc R .
C12 C 0.2702(5) 0.5682(4) 0.0401(4) 0.0809(14) Uani 1 d . .
H12A H 0.3461(26) 0.6234(20) 0.0319(9) 0.121 Uiso 1 calc R .
H12B H 0.1907(10) 0.6071(23) 0.0255(10) 0.121 Uiso 1 calc R .
H12C H 0.3085(33) 0.5140(5) -0.0122(4) 0.121 Uiso 1 calc R .
C13 C 0.1210(6) 0.5962(4) 0.2776(5) 0.096(2) Uani 1 d . .
H13A H 0.1924(13) 0.6551(18) 0.2699(23) 0.145 Uiso 1 calc R .
H13B H 0.0853(37) 0.5581(7) 0.3532(5) 0.145 Uiso 1 calc R .
H13C H 0.0409(27) 0.6304(24) 0.2599(21) 0.145 Uiso 1 calc R .
Al2 Al 0.31430(12) 0.24664(8) 0.80017(8) 0.0491(3) Uani 1 d . .
Cl2 Cl 0.38581(11) 0.09820(7) 0.72722(8) 0.0606(3) Uani 1 d . .
Cl3 Cl 0.09373(11) 0.21864(9) 0.90555(8) 0.0712(3) Uani 1 d . .
Cl4 Cl 0.33977(15) 0.38320(9) 0.67696(10) 0.0993(5) Uani 1 d . .
Cl5 Cl 0.4325(2) 0.27202(14) 0.90072(15) 0.1293(7) Uani 1 d . .

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 _atom_site_aniso_U_22
 _atom_site_aniso_U_33
 _atom_site_aniso_U_23
 _atom_site_aniso_U_13
 _atom_site_aniso_U_12
Al1 0.0319(6) 0.0326(5) 0.0313(5) -0.0017(4) -0.0126(4) 0.0012(4)
Cl1 0.0505(6) 0.0584(5) 0.0374(4) -0.0009(4) -0.0226(4) 0.0045(4)
N1 0.034(2) 0.0356(14) 0.0310(14) -0.0015(11) -0.0089(12) 0.0023(11)
C1 0.041(2) 0.038(2) 0.040(2) -0.0108(14) -0.0135(15) -0.0009(14)
```

```
C2 0.037(2) 0.041(2) 0.033(2) -0.0066(13) -0.0137(14) -0.0001(14)
N2 0.034(2) 0.0291(12) 0.0315(13) -0.0038(10) -0.0122(11) 0.0033(10)
C3 0.039(2) 0.035(2) 0.041(2) -0.0002(13) -0.0166(14) 0.0058(14)
C4 0.048(2) 0.033(2) 0.044(2) 0.0047(13) -0.020(2) -0.0024(14)
N3 0.038(2) 0.0345(14) 0.0430(15) 0.0029(11) -0.0182(12) -0.0029(11)
Si1 0.0464(6) 0.0413(5) 0.0424(5) -0.0059(4) -0.0151(4) 0.0147(4)
C5 0.095(3) 0.045(2) 0.058(2) 0.004(2) -0.032(2) 0.014(2)
C6 0.043(2) 0.082(3) 0.079(3) -0.021(2) -0.024(2) 0.018(2)
C7 0.064(3) 0.056(2) 0.056(2) -0.019(2) -0.015(2) 0.023(2)
Si2 0.0331(5) 0.0449(5) 0.0318(4) -0.0049(4) -0.0083(4) 0.0014(4)
C8 0.051(2) 0.051(2) 0.037(2) 0.0030(15) -0.010(2) -0.007(2)
C9 0.053(2) 0.060(2) 0.041(2) -0.017(2) -0.012(2) 0.007(2)
C10 0.040(2) 0.079(3) 0.049(2) -0.005(2) -0.010(2) 0.002(2)
Si3 0.0536(7) 0.0366(5) 0.0592(6) 0.0034(4) -0.0304(5) -0.0096(4)
C11 0.065(3) 0.064(3) 0.116(4) 0.020(3) -0.051(3) -0.021(2)
C12 0.076(3) 0.082(3) 0.082(3) 0.028(2) -0.037(3) -0.037(3)
C13 0.124(5) 0.073(3) 0.120(4) -0.044(3) -0.067(4) 0.009(3)
Al2 0.0547(7) 0.0384(5) 0.0460(6) -0.0051(4) -0.0086(5) 0.0004(5)
Cl2 0.0625(6) 0.0435(5) 0.0618(6) -0.0103(4) -0.0048(5) 0.0066(4)
Cl3 0.0600(7) 0.0799(7) 0.0507(5) -0.0019(5) 0.0050(5) 0.0136(5)
Cl4 0.1110(10) 0.0585(6) 0.0768(8) 0.0194(5) 0.0172(7) 0.0362(6)
Cl5 0.168(2) 0.1135(12) 0.1440(14) -0.0125(10) -0.1010(13) -0.0496(11)

_geom_special_details
;
 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.
;

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Al1 N3 1.782(3) .
Al1 N1 1.947(3) .
Al1 N2 1.963(3) .
Al1 Cl1 2.1034(14) .
N1 C1 1.504(4) .
N1 Si1 1.830(3) .
C1 C2 1.511(4) .
C2 N2 1.511(4) .
N2 C3 1.519(4) .
N2 Si2 1.850(3) .
C3 C4 1.521(4) .
C4 N3 1.481(4) .
N3 Si3 1.724(3) .
Si1 C5 1.839(4) .
Si1 C6 1.841(4) .
Si1 C7 1.850(4) .
Si2 C10 1.841(4) .
Si2 C8 1.845(3) .
Si2 C9 1.850(3) .
Si3 C11 1.843(4) .
Si3 C13 1.850(5) .
Si3 C12 1.859(4) .
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Al2 Cl4 2.098(2) .
Al2 Cl5 2.116(2) .
Al2 Cl2 2.121(2) .
Al2 Cl3 2.144(2) .

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 _geom_angle_atom_site_label_3
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 _geom_angle_site_symmetry_1
 _geom_angle_site_symmetry_3
N3 Al1 N1 114.88(12) . .
N3 Al1 N2 95.01(12) . .
N1 Al1 N2 91.81(11) . .
N3 Al1 Cl1 123.02(10) . .
N1 Al1 Cl1 109.41(9) . .
N2 Al1 Cl1 117.87(9) . .
C1 N1 Si1 113.2(2) . .
C1 N1 Al1 104.5(2) . .
Si1 N1 Al1 123.19(15) . .
N1 C1 C2 109.8(2) . .
N2 C2 C1 110.5(2) . .
C2 N2 C3 109.7(2) . .
C2 N2 Si2 112.7(2) . .
C3 N2 Si2 108.6(2) . .
C2 N2 Al1 104.5(2) . .
C3 N2 Al1 101.5(2) . .
Si2 N2 Al1 119.08(13) . .
N2 C3 C4 110.5(2) . .
N3 C4 C3 109.3(2) . .
C4 N3 Si3 117.9(2) . .
C4 N3 Al1 107.3(2) . .
Si3 N3 Al1 134.0(2) . .
N1 Si1 C5 107.6(2) . .
N1 Si1 C6 105.7(2) . .
C5 Si1 C6 112.9(2) . .
N1 Si1 C7 107.0(2) . .
C5 Si1 C7 110.8(2) . .
C6 Si1 C7 112.4(2) . .
C10 Si2 C8 111.2(2) . .
C10 Si2 C9 111.4(2) . .
C8 Si2 C9 111.4(2) . .
C10 Si2 N2 107.64(14) . .
C8 Si2 N2 107.84(14) . .
C9 Si2 N2 107.11(14) . .
N3 Si3 Cl1 108.3(2) . .
N3 Si3 Cl3 110.4(2) . .
Cl1 Si3 Cl3 109.4(2) . .
N3 Si3 Cl2 109.5(2) . .
Cl1 Si3 Cl2 111.2(2) . .
Cl3 Si3 Cl2 108.0(2) . .
Cl4 Al2 Cl5 112.13(9) . .
Cl4 Al2 Cl2 109.59(7) . .
Cl5 Al2 Cl2 108.19(8) . .
Cl4 Al2 Cl3 111.37(8) . .
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Cl2 Al2 Cl3 108.14(7) . .

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