Supporting Information

Computational Study

All calculations have been performed using the Gaussian98 package.[1] Geometries of 3,4-di-*tert*-butylthiophene 1-oxide (**1a)** and 3,4-di-*tert*-butylthiophene 1-(*p*-toluenesulfonyl)imide (**3a)** have been optimized at the B3LYP levels with the 6-31G(d) basis set. Molden[2] was used for visualizing the orbitals of **1a**. GaussView (Gaussian, Inc.) was used for visualizing the molecular structure of **3a**.

PDB-file of 3,4-di-*tert*-butylthiophene_1-oxide (**1a)**

COMPND 3,4-di-*tert*-butylthiophene_1-oxide.PDB

HETATM	1	S	-0.224	-3.346	1.119
HETATM	2	O	0.036	-3.993	2.465
HETATM	3	C	-0.834	-0.847	0.533
HETATM	4	C	0.611	-1.072	0.089
HETATM	5	C	-1.309	-1.949	1.160
HETATM	6	C	1.074	-0.322	-2.283
HETATM	7	C	-1.738	0.412	0.465
HETATM	8	C	1.049	-2.289	0.495
HETATM	9	C	1.545	-0.221	-0.810
HETATM	10	C	-1.235	1.460	1.491
HETATM	11	C	1.672	1.256	-0.375
HETATM	12	C	2.990	-0.781	-0.775
HETATM	13	C	-1.854	1.027	-0.947
HETATM	14	C	-3.189	0.062	0.882
HETATM	20	H	-2.287	-2.100	1.588

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HETATM   16  H                    0.059   0.048  -2.435
HETATM   17  H                    1.102  -1.364  -2.620
HETATM   24  H                    1.743   0.260  -2.929
HETATM   19  H                    2.015  -2.736   0.314
HETATM   28  H                   -0.207   1.777   1.315
HETATM   30  H                   -1.872   2.352   1.452
HETATM   33  H                   -1.287   1.050   2.505
HETATM   18  H                    2.006   1.331   0.666
HETATM   21  H                    2.423   1.751  -1.000
HETATM   31  H                    0.748   1.823  -0.477
HETATM   23  H                    3.391  -0.812   0.244
HETATM   27  H                    3.060  -1.784  -1.207
HETATM   29  H                    3.640  -0.131  -1.370
HETATM   15  H                   -2.200   0.282  -1.672
HETATM   22  H                   -0.924   1.454  -1.318
HETATM   32  H                   -2.593   1.837  -0.929
HETATM   25  H                   -3.810   0.960   0.797
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HETATM   34  H                   -3.254  -0.277   1.920
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CONECT    2    1
CONECT    3    4    5    7
CONECT    4    3    8    9
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CONECT    8    1    4   19
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CONECT   32   13
CONECT   25   14
CONECT   26   14

CONECT   34   14
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PDB-file of 3,4-di-*tert*-butylthiophene 1-(*p*-toluenesulfonyl)imide (**3a)**

COMPND	3,4-di-*tert*-butylthiophene 1-(*p*-toluenesulfonyl)imide.PDB				
HETATM	1	S	-0.654	-1.708	-2.043
HETATM	2	S	1.236	-2.603	-0.098
HETATM	3	O	2.105	-3.780	-0.026
HETATM	4	O	0.144	-2.425	0.881
HETATM	5	N	0.677	-2.584	-1.689
HETATM	6	C	2.293	-1.149	0.032
HETATM	7	C	-0.679	0.002	-1.588
HETATM	8	C	-2.592	-0.870	-0.474
HETATM	9	C	3.472	-1.092	-0.715
HETATM	10	C	4.291	0.029	-0.612
HETATM	11	C	-3.757	-1.032	0.536
HETATM	12	C	-2.052	-1.991	-0.997
HETATM	13	C	-2.089	1.869	-0.733
HETATM	14	C	1.940	-0.105	0.885
HETATM	15	C	-1.783	0.357	-0.887
HETATM	16	C	3.958	1.097	0.237
HETATM	17	C	2.775	1.011	0.981
HETATM	18	C	-3.276	2.241	-1.658
HETATM	19	C	4.871	2.294	0.360
HETATM	20	C	-2.363	2.307	0.723
HETATM	21	C	-3.190	-0.836	1.967
HETATM	22	C	-4.968	-0.109	0.278
HETATM	23	C	-4.318	-2.476	0.487

HETATM	24	C	-0.883	2.722	-1.200
HETATM	50	H	0.064	0.642	-2.037
HETATM	38	H	3.742	-1.919	-1.364
HETATM	37	H	5.208	0.075	-1.194
HETATM	51	H	-2.353	-3.017	-0.865
HETATM	40	H	1.029	-0.178	1.467
HETATM	39	H	2.505	1.823	1.652
HETATM	47	H	-3.497	3.311	-1.564
HETATM	48	H	-4.188	1.689	-1.429
HETATM	49	H	-3.019	2.041	-2.704
HETATM	34	H	4.370	3.134	0.851
HETATM	35	H	5.221	2.634	-0.622
HETATM	36	H	5.763	2.051	0.952
HETATM	44	H	-2.468	3.398	0.755
HETATM	45	H	-1.527	2.036	1.377
HETATM	46	H	-3.273	1.885	1.144
HETATM	25	H	-3.979	-1.030	2.704
HETATM	26	H	-2.811	0.171	2.144
HETATM	27	H	-2.369	-1.537	2.143
HETATM	28	H	-5.763	-0.354	0.990
HETATM	29	H	-5.370	-0.261	-0.731
HETATM	30	H	-4.748	0.951	0.400
HETATM	31	H	-5.167	-2.549	1.174
HETATM	32	H	-3.581	-3.218	0.809
HETATM	33	H	-4.674	-2.743	-0.514
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CONECT 45 20

CONECT 46 20

CONECT 25 21

CONECT 26 21

CONECT 27 21

CONECT 28 22

CONECT 29 22

CONECT 30 22

CONECT 31 23

CONECT 32 23

CONECT 33 23

CONECT 41 24

CONECT 42 24

CONECT 43 24

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X-Ray Crystallographic Analysis of 3a, 7a, 7b, 7k, 9a, 12a, and 17. The crystal data for **9a** were recorded on a Mac Science MXC3K diffractometer, Cu-*K*α radiation (λ = 1.54178 Å) with a graphite-monochromater. The unit cell dimensions were obtained by a least-squares fit of 22 automatically centered reflections. The structure was solved by direct methods using SIR97[3] and refined with full-matrix least-squares[4] using all independent reflections. Absorption corrections were done by a Psi-scan method. The non-hydrogen atoms were refined anisotropically, and hydrogen atoms were placed at calculated positions. The crystal data for **3a**, **7a**,**b**,**k**, **12a**, and **17** were recorded on a Mac Science DIP3000 diffractometer with a graphite-monochromater. Oscillation and nonscreen Weissenberg photographs were recorded on the imaging plates of the diffractometer by using Mo-*K*α (λ = 0.71073 Å), and the data reduction was made by the MAC DENZO program system. The cell parameters were determined and refined by using MAC DENZO for all observed reflections. The structure was solved by direct methods using SIR97[3] and refined with full-matrix least-squares[4] using all independent reflections. Absorption corrections were done by a multi-scan method.[5] The non-hydrogen atoms were refined anisotropically, and hydrogen atoms were placed at calculated positions. The analyses of **3a** and **7a** were performed on two independent molecules respectively.

(1) CIF file of **3a**

CIF Copied by cif2cif, version 0.0.8 - beta (2 Apr 98)

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_publ_contact_author_address

;

Department of Chemistry, Faculty of Science, Saitama University

Saitama, Saitama 338-8570, Japan

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_publ_contact_author_email 'nakaj@post.saitama-u.ac.jp'

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Crystal and molecular structure

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The study of the titled structure was undertaken to establish its three dimensional structure. Geometries are tabulated below. All diagrams and calculations were performed using maXus (Bruker Nonius, Delft & MacScience,

Japan).

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Sortav (Blessing 1995)

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Refinement of F^2^ against ALL reflections. The weighted R-factor wR and goodness 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 threshold expression of F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) 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.

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S3 S 0.23967(16) 0.72714(13) 0.71590(10) 0.0597(5) Uani 1 1 d . . .

S4 S 0.23823(17) 0.93364(14) 0.78133(12) 0.0666(5) Uani 1 1 d . . .

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C7 C 0.1090(5) 0.3084(4) 0.6075(4) 0.0481(14) Uani 1 1 d . . .

C8 C 0.2790(6) 0.3214(4) 0.4017(4) 0.0449(13) Uani 1 1 d . . .

O9 O 0.1455(5) 0.9951(3) 0.7525(3) 0.0903(15) Uani 1 1 d . . .

N10 N 0.1878(5) 0.8265(4) 0.7035(3) 0.0596(14) Uani 1 1 d . . .

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N12 N 0.2796(5) 0.5086(3) 0.5133(3) 0.0531(12) Uani 1 1 d . . .

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C15 C 0.3691(7) 0.1376(5) 0.3834(4) 0.0584(16) Uani 1 1 d . . .

C16 C 0.4237(6) 0.3184(5) 0.4216(4) 0.0566(16) Uani 1 1 d . . .

C17 C 0.4175(6) 0.6948(5) 0.8434(4) 0.0553(16) Uani 1 1 d . . .

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C24 C 0.0347(7) 0.8859(5) 0.9725(5) 0.077(2) Uani 1 1 d . . .

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C30 C 0.2146(7) 0.5944(5) 0.9112(5) 0.0676(18) Uani 1 1 d . . .

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C32 C 0.2857(8) 0.1216(5) 0.6135(5) 0.091(2) Uani 1 1 d . . .

C33 C 0.4637(7) 0.2290(5) 0.4115(4) 0.0628(17) Uani 1 1 d . . .

C34 C 0.4169(7) 0.0407(5) 0.3743(5) 0.089(2) Uani 1 1 d . . .

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C43 C 0.6792(7) 0.7193(7) 0.8456(5) 0.109(3) Uani 1 1 d . . .

C44 C 0.5787(9) 0.5858(6) 0.8988(6) 0.126(3) Uani 1 1 d . . .

C45 C -0.0266(8) 0.2086(8) 0.6896(6) 0.155(5) Uani 1 1 d . . .

C46 C -0.1601(7) 0.2737(7) 0.5793(7) 0.143(4) Uani 1 1 d . . .

C47 C 0.1102(10) 0.8802(7) 1.1334(5) 0.135(4) Uani 1 1 d . . .

C48 C -0.0496(8) 0.1391(6) 0.5236(7) 0.141(4) Uani 1 1 d . . .

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H20 H 0.0035 0.4068 0.5568 0.063 Uiso 1 1 d R . .

H21 H 0.0663 0.6696 0.7899 0.066 Uiso 1 1 d R . .

H22 H 0.4486 0.3942 0.6231 0.062 Uiso 1 1 d R . .

H23 H 0.0780 0.2333 0.3595 0.064 Uiso 1 1 d R . .

H24 H -0.0627 0.8716 0.9779 0.092 Uiso 1 1 d R . .

H25 H -0.0158 0.8884 0.8412 0.080 Uiso 1 1 d R . .

H27 H 0.4108 0.9427 0.9504 0.084 Uiso 1 1 d R . .

H28 H 0.3625 0.9240 1.0879 0.094 Uiso 1 1 d R . .

H29 H 0.1547 0.0808 0.3447 0.072 Uiso 1 1 d R . .

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H31B H 0.0019 0.5722 0.8494 0.126 Uiso 1 1 d R . .

H31C H 0.0484 0.6665 0.9367 0.126 Uiso 1 1 d R . .

H32A H 0.3356 0.0786 0.6399 0.109 Uiso 1 1 d R . .

H32B H 0.2997 0.1133 0.5527 0.109 Uiso 1 1 d R . .

H32C H 0.1855 0.1044 0.6113 0.109 Uiso 1 1 d R . .

H33 H 0.5646 0.2297 0.4240 0.075 Uiso 1 1 d R . .

H34A H 0.5195 0.0529 0.3903 0.107 Uiso 1 1 d R . .

H34B H 0.3833 -0.0014 0.3121 0.107 Uiso 1 1 d R . .

H34C H 0.3789 0.0080 0.4139 0.107 Uiso 1 1 d R . .

H35A H 0.3726 0.1950 0.7915 0.114 Uiso 1 1 d R . .

H35B H 0.2267 0.2293 0.7700 0.114 Uiso 1 1 d R . .

H35C H 0.3702 0.3091 0.8064 0.114 Uiso 1 1 d R . .

H38 H 0.4913 0.7778 0.7646 0.072 Uiso 1 1 d R . .

H39A H 0.6978 0.7682 1.0229 0.125 Uiso 1 1 d R . .

H39B H 0.5344 0.7561 1.0229 0.125 Uiso 1 1 d R . .

H39C H 0.6058 0.8354 0.9823 0.125 Uiso 1 1 d R . .

H40A H 0.5438 0.2024 0.6963 0.168 Uiso 1 1 d R . .

H40B H 0.5421 0.3167 0.7118 0.168 Uiso 1 1 d R . .

H40C H 0.5133 0.2431 0.6123 0.168 Uiso 1 1 d R . .

H41A H 0.2569 0.5913 1.0457 0.114 Uiso 1 1 d R . .

H41B H 0.2817 0.7007 1.0385 0.114 Uiso 1 1 d R . .

H41C H 0.3934 0.6327 1.0159 0.114 Uiso 1 1 d R . .

H42A H 0.1894 0.4464 0.9079 0.123 Uiso 1 1 d R . .

H42B H 0.3208 0.4797 0.8694 0.123 Uiso 1 1 d R . .

H42C H 0.1650 0.4592 0.8100 0.123 Uiso 1 1 d R . .

H43A H 0.7730 0.7172 0.8767 0.131 Uiso 1 1 d R . .

H43B H 0.6769 0.7861 0.8426 0.131 Uiso 1 1 d R . .

H43C H 0.6568 0.6732 0.7848 0.131 Uiso 1 1 d R . .

H44A H 0.6729 0.5868 0.9316 0.151 Uiso 1 1 d R . .

H44B H 0.5608 0.5400 0.8381 0.151 Uiso 1 1 d R . .

H44C H 0.5092 0.5644 0.9300 0.151 Uiso 1 1 d R . .

H45A H -0.1133 0.1627 0.6840 0.186 Uiso 1 1 d R . .

H45B H -0.0170 0.2680 0.7399 0.186 Uiso 1 1 d R . .

H45C H 0.0537 0.1772 0.7003 0.186 Uiso 1 1 d R . .

H46A H -0.2431 0.2271 0.5784 0.172 Uiso 1 1 d R . .

H46B H -0.1648 0.2807 0.5195 0.172 Uiso 1 1 d R . .

H46C H -0.1570 0.3379 0.6220 0.172 Uiso 1 1 d R . .

H47A H 0.1962 0.8884 1.1784 0.162 Uiso 1 1 d R . .

H47B H 0.0537 0.8142 1.1203 0.162 Uiso 1 1 d R . .

H47C H 0.0563 0.9297 1.1565 0.162 Uiso 1 1 d R . .

H48A H -0.1333 0.0923 0.5214 0.169 Uiso 1 1 d R . .

H48B H 0.0322 0.1080 0.5284 0.169 Uiso 1 1 d R . .

H48C H -0.0623 0.1586 0.4689 0.169 Uiso 1 1 d R . .

loop_

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_atom_site_aniso_U_22

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_atom_site_aniso_U_23

_atom_site_aniso_U_13

_atom_site_aniso_U_12

S1 0.0769(11) 0.0541(11) 0.0551(11) 0.0259(9) 0.0082(8) 0.0171(8)

S2 0.0752(11) 0.0422(10) 0.0547(11) 0.0197(8) 0.0137(8) 0.0069(8)

S3 0.0624(10) 0.0747(12) 0.0442(10) 0.0252(9) 0.0030(7) 0.0181(8)

S4 0.0672(11) 0.0704(13) 0.0751(13) 0.0416(11) 0.0195(9) 0.0121(9)

O5 0.068(3) 0.075(3) 0.085(3) 0.015(3) -0.017(2) 0.028(2)

C6 0.044(3) 0.048(4) 0.043(3) 0.016(3) 0.002(2) 0.016(3)

C7 0.054(3) 0.047(4) 0.049(4) 0.022(3) 0.013(3) 0.014(3)

C8 0.057(3) 0.044(4) 0.039(3) 0.018(3) 0.012(3) 0.014(3)

O9 0.097(3) 0.095(4) 0.112(4) 0.069(3) 0.032(3) 0.044(3)

N10 0.068(3) 0.066(4) 0.052(3) 0.030(3) 0.004(2) 0.024(3)

O11 0.150(4) 0.078(3) 0.079(3) 0.059(3) 0.051(3) 0.034(3)

N12 0.069(3) 0.044(3) 0.050(3) 0.025(3) 0.008(2) 0.006(2)

C13 0.063(4) 0.044(4) 0.049(4) 0.017(3) 0.009(3) 0.008(3)

C14 0.070(4) 0.046(4) 0.043(4) 0.014(3) 0.013(3) 0.015(3)

C15 0.073(4) 0.062(5) 0.055(4) 0.027(4) 0.022(3) 0.033(4)

C16 0.044(4) 0.073(5) 0.056(4) 0.029(4) 0.013(3) 0.002(3)

C17 0.060(4) 0.070(4) 0.039(4) 0.019(3) 0.004(3) 0.024(3)

C18 0.053(4) 0.057(4) 0.061(4) 0.032(4) 0.015(3) 0.003(3)

O19 0.062(3) 0.100(4) 0.104(4) 0.035(3) 0.024(3) -0.008(2)

C20 0.061(4) 0.050(4) 0.064(4) 0.034(3) 0.023(3) 0.025(3)

C21 0.068(4) 0.062(4) 0.042(4) 0.026(3) 0.014(3) 0.015(3)

C22 0.046(3) 0.062(4) 0.053(4) 0.028(3) 0.007(3) 0.006(3)

C23 0.058(4) 0.052(4) 0.053(4) 0.021(3) 0.010(3) 0.022(3)

C24 0.079(5) 0.085(6) 0.069(5) 0.022(4) 0.027(4) 0.015(4)

C25 0.064(4) 0.078(5) 0.063(5) 0.029(4) 0.014(3) 0.015(3)

C26 0.056(4) 0.059(4) 0.063(4) 0.032(4) 0.015(3) 0.022(3)

C27 0.071(4) 0.070(5) 0.066(5) 0.025(4) 0.008(4) 0.005(3)

C28 0.089(5) 0.071(5) 0.061(5) 0.016(4) -0.007(4) 0.011(4)

C29 0.074(4) 0.053(4) 0.051(4) 0.015(3) 0.010(3) 0.012(3)

C30 0.088(5) 0.061(5) 0.066(5) 0.034(4) 0.021(4) 0.022(4)

C31 0.125(7) 0.110(7) 0.116(7) 0.075(6) 0.059(5) 0.019(5)

C32 0.127(6) 0.087(6) 0.078(5) 0.035(5) 0.025(4) 0.066(5)

C33 0.068(4) 0.077(5) 0.059(4) 0.033(4) 0.023(3) 0.034(4)

C34 0.104(6) 0.084(6) 0.102(6) 0.034(5) 0.045(5) 0.052(5)

C35 0.146(7) 0.104(6) 0.052(5) 0.035(4) 0.015(4) 0.067(5)

C36 0.107(6) 0.072(5) 0.055(5) 0.021(4) 0.028(4) 0.015(4)

C37 0.070(4) 0.103(6) 0.049(4) 0.025(4) 0.011(3) 0.036(4)

C38 0.065(4) 0.072(5) 0.047(4) 0.026(4) 0.004(3) 0.019(3)

C39 0.084(5) 0.155(8) 0.065(5) 0.031(6) -0.002(4) 0.026(5)

C40 0.082(6) 0.196(10) 0.211(11) 0.151(9) 0.043(6) 0.066(6)

C41 0.142(7) 0.096(6) 0.062(5) 0.044(5) 0.026(5) 0.025(5)

C42 0.151(7) 0.063(5) 0.104(7) 0.036(5) 0.040(5) 0.019(5)

C43 0.071(5) 0.180(9) 0.078(6) 0.039(6) 0.004(4) 0.048(5)

C44 0.124(7) 0.130(8) 0.146(9) 0.058(7) 0.017(6) 0.078(6)

C45 0.101(7) 0.248(12) 0.123(8) 0.116(8) 0.018(5) -0.050(7)

C46 0.048(5) 0.172(9) 0.263(13) 0.142(9) 0.039(6) 0.036(5)

C47 0.214(10) 0.135(8) 0.075(6) 0.040(6) 0.058(7) 0.051(7)

C48 0.085(6) 0.099(7) 0.199(11) 0.011(7) 0.024(6) -0.031(5)

_geom_special_details

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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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loop_

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_geom_bond_atom_site_label_2

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S1 O5 1.441(4) . ?

S1 O11 1.442(4) . ?

S1 N12 1.606(5) . ?

S1 C8 1.759(5) . ?

S2 N12 1.606(4) . ?

S2 C20 1.724(6) . ?

S2 C22 1.738(5) . ?

S3 N10 1.603(5) . ?

S3 C38 1.744(5) . ?

S3 C21 1.748(6) . ?

S4 O9 1.432(4) . ?

S4 O19 1.438(4) . ?

S4 N10 1.607(5) . ?

S4 C13 1.766(6) . ?

C6 C22 1.333(7) . ?

C6 C7 1.522(7) . ?

C6 C26 1.545(7) . ?

C7 C20 1.346(7) . ?

C7 C18 1.549(7) . ?

C8 C16 1.390(7) . ?

C8 C23 1.393(7) . ?

C13 C25 1.376(8) . ?

C13 C27 1.401(8) . ?

C14 C21 1.345(7) . ?

C14 C17 1.519(8) . ?

C14 C30 1.537(8) . ?

C15 C33 1.384(8) . ?

C15 C29 1.390(8) . ?

C15 C34 1.483(8) . ?

C16 C33 1.349(8) . ?

C17 C38 1.333(7) . ?

C17 C37 1.543(7) . ?

C18 C45 1.469(9) . ?

C18 C46 1.505(8) . ?

C18 C48 1.530(10) . ?

C23 C29 1.386(7) . ?

C24 C25 1.366(8) . ?

C24 C36 1.376(9) . ?

C26 C35 1.516(8) . ?

C26 C40 1.519(8) . ?

C26 C32 1.524(9) . ?

C27 C28 1.385(8) . ?

C28 C36 1.371(9) . ?

C30 C42 1.523(9) . ?

C30 C31 1.533(9) . ?

C30 C41 1.538(9) . ?

C36 C47 1.544(9) . ?

C37 C43 1.516(9) . ?

C37 C44 1.522(9) . ?

C37 C39 1.529(9) . ?

C16 H16 0.9600 . ?

C20 H20 0.9599 . ?

C21 H21 0.9599 . ?

C22 H22 0.9600 . ?

C23 H23 0.9600 . ?

C24 H24 0.9600 . ?

C25 H25 0.9600 . ?

C27 H27 0.9600 . ?

C28 H28 0.9601 . ?

C29 H29 0.9600 . ?

C31 H31A 0.9599 . ?

C31 H31B 0.9600 . ?

C31 H31C 0.9600 . ?

C32 H32A 0.9600 . ?

C32 H32B 0.9600 . ?

C32 H32C 0.9601 . ?

C33 H33 0.9599 . ?

C34 H34A 0.9600 . ?

C34 H34B 0.9599 . ?

C34 H34C 0.9602 . ?

C35 H35A 0.9601 . ?

C35 H35B 0.9600 . ?

C35 H35C 0.9599 . ?

C38 H38 0.9601 . ?

C39 H39A 0.9599 . ?

C39 H39B 0.9600 . ?

C39 H39C 0.9601 . ?

C40 H40A 0.9600 . ?

C40 H40B 0.9599 . ?

C40 H40C 0.9600 . ?

C41 H41A 0.9598 . ?

C41 H41B 0.9601 . ?

C41 H41C 0.9601 . ?

C42 H42A 0.9601 . ?

C42 H42B 0.9600 . ?

C42 H42C 0.9600 . ?

C43 H43A 0.9600 . ?

C43 H43B 0.9599 . ?

C43 H43C 0.9600 . ?

C44 H44A 0.9600 . ?

C44 H44B 0.9600 . ?

C44 H44C 0.9599 . ?

C45 H45A 0.9600 . ?

C45 H45B 0.9599 . ?

C45 H45C 0.9602 . ?

C46 H46A 0.9599 . ?

C46 H46B 0.9601 . ?

C46 H46C 0.9600 . ?

C47 H47A 0.9600 . ?

C47 H47B 0.9600 . ?

C47 H47C 0.9600 . ?

C48 H48A 0.9600 . ?

C48 H48B 0.9600 . ?

C48 H48C 0.9597 . ?

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O5 S1 O11 117.5(3) . . ?

O5 S1 N12 111.2(3) . . ?

O11 S1 N12 103.2(3) . . ?

O5 S1 C8 107.1(3) . . ?

O11 S1 C8 108.7(3) . . ?

N12 S1 C8 108.8(2) . . ?

N12 S2 C20 117.2(3) . . ?

N12 S2 C22 115.0(3) . . ?

C20 S2 C22 91.3(3) . . ?

N10 S3 C38 117.8(3) . . ?

N10 S3 C21 114.7(3) . . ?

C38 S3 C21 90.8(3) . . ?

O9 S4 O19 117.9(3) . . ?

O9 S4 N10 104.8(3) . . ?

O19 S4 N10 112.1(3) . . ?

O9 S4 C13 107.4(3) . . ?

O19 S4 C13 106.7(3) . . ?

N10 S4 C13 107.5(3) . . ?

C22 C6 C7 111.0(5) . . ?

C22 C6 C26 118.9(5) . . ?

C7 C6 C26 130.1(5) . . ?

C20 C7 C6 110.7(5) . . ?

C20 C7 C18 117.9(5) . . ?

C6 C7 C18 131.4(5) . . ?

C16 C8 C23 119.3(5) . . ?

C16 C8 S1 120.6(5) . . ?

C23 C8 S1 120.1(4) . . ?

S3 N10 S4 122.7(3) . . ?

S2 N12 S1 121.6(3) . . ?

C25 C13 C27 120.2(6) . . ?

C25 C13 S4 120.9(5) . . ?

C27 C13 S4 118.9(5) . . ?

C21 C14 C17 111.4(5) . . ?

C21 C14 C30 116.7(5) . . ?

C17 C14 C30 131.6(5) . . ?

C33 C15 C29 115.6(6) . . ?

C33 C15 C34 122.7(6) . . ?

C29 C15 C34 121.7(6) . . ?

C33 C16 C8 119.5(6) . . ?

C38 C17 C14 110.9(5) . . ?

C38 C17 C37 117.3(5) . . ?

C14 C17 C37 131.8(5) . . ?

C45 C18 C46 109.3(6) . . ?

C45 C18 C48 110.5(7) . . ?

C46 C18 C48 101.3(6) . . ?

C45 C18 C7 114.1(5) . . ?

C46 C18 C7 111.1(5) . . ?

C48 C18 C7 109.7(5) . . ?

C7 C20 S2 113.0(4) . . ?

C14 C21 S3 112.3(5) . . ?

C6 C22 S2 112.7(4) . . ?

C29 C23 C8 119.0(5) . . ?

C25 C24 C36 120.7(6) . . ?

C24 C25 C13 119.7(6) . . ?

C35 C26 C40 107.6(6) . . ?

C35 C26 C32 109.2(5) . . ?

C40 C26 C32 104.4(6) . . ?

C35 C26 C6 111.1(5) . . ?

C40 C26 C6 110.7(5) . . ?

C32 C26 C6 113.6(5) . . ?

C28 C27 C13 119.0(6) . . ?

C36 C28 C27 120.0(6) . . ?

C23 C29 C15 122.5(6) . . ?

C42 C30 C31 107.6(6) . . ?

C42 C30 C14 110.6(6) . . ?

C31 C30 C14 109.4(5) . . ?

C42 C30 C41 109.8(6) . . ?

C31 C30 C41 105.4(6) . . ?

C14 C30 C41 113.8(6) . . ?

C16 C33 C15 124.1(6) . . ?

C28 C36 C24 120.4(7) . . ?

C28 C36 C47 120.8(7) . . ?

C24 C36 C47 118.8(7) . . ?

C43 C37 C44 105.1(6) . . ?

C43 C37 C39 106.1(6) . . ?

C44 C37 C39 113.5(6) . . ?

C43 C37 C17 109.8(5) . . ?

C44 C37 C17 112.2(6) . . ?

C39 C37 C17 109.8(5) . . ?

C17 C38 S3 113.2(5) . . ?

C33 C16 H16 121.6 . . ?

C8 C16 H16 118.9 . . ?

C7 C20 H20 127.7 . . ?

S2 C20 H20 119.4 . . ?

C14 C21 H21 128.1 . . ?

S3 C21 H21 119.6 . . ?

C6 C22 H22 127.9 . . ?

S2 C22 H22 119.4 . . ?

C29 C23 H23 121.0 . . ?

C8 C23 H23 120.0 . . ?

C25 C24 H24 119.0 . . ?

C36 C24 H24 120.3 . . ?

C24 C25 H25 120.9 . . ?

C13 C25 H25 119.4 . . ?

C28 C27 H27 121.1 . . ?

C13 C27 H27 119.8 . . ?

C36 C28 H28 120.8 . . ?

C27 C28 H28 119.2 . . ?

C23 C29 H29 118.6 . . ?

C15 C29 H29 119.0 . . ?

C30 C31 H31A 110.0 . . ?

C30 C31 H31B 109.0 . . ?

H31A C31 H31B 109.5 . . ?

C30 C31 H31C 109.5 . . ?

H31A C31 H31C 109.5 . . ?

H31B C31 H31C 109.5 . . ?

C26 C32 H32A 110.2 . . ?

C26 C32 H32B 109.8 . . ?

H32A C32 H32B 109.5 . . ?

C26 C32 H32C 108.5 . . ?

H32A C32 H32C 109.5 . . ?

H32B C32 H32C 109.5 . . ?

C16 C33 H33 117.0 . . ?

C15 C33 H33 118.8 . . ?

C15 C34 H34A 109.4 . . ?

C15 C34 H34B 109.0 . . ?

H34A C34 H34B 109.5 . . ?

C15 C34 H34C 110.0 . . ?

H34A C34 H34C 109.5 . . ?

H34B C34 H34C 109.5 . . ?

C26 C35 H35A 110.0 . . ?

C26 C35 H35B 109.6 . . ?

H35A C35 H35B 109.5 . . ?

C26 C35 H35C 108.9 . . ?

H35A C35 H35C 109.5 . . ?

H35B C35 H35C 109.5 . . ?

C17 C38 H38 127.7 . . ?

S3 C38 H38 119.2 . . ?

C37 C39 H39A 110.6 . . ?

C37 C39 H39B 108.7 . . ?

H39A C39 H39B 109.5 . . ?

C37 C39 H39C 109.1 . . ?

H39A C39 H39C 109.5 . . ?

H39B C39 H39C 109.5 . . ?

C26 C40 H40A 109.8 . . ?

C26 C40 H40B 108.5 . . ?

H40A C40 H40B 109.5 . . ?

C26 C40 H40C 110.1 . . ?

H40A C40 H40C 109.5 . . ?

H40B C40 H40C 109.5 . . ?

C30 C41 H41A 110.7 . . ?

C30 C41 H41B 109.4 . . ?

H41A C41 H41B 109.5 . . ?

C30 C41 H41C 108.3 . . ?

H41A C41 H41C 109.5 . . ?

H41B C41 H41C 109.5 . . ?

C30 C42 H42A 109.4 . . ?

C30 C42 H42B 109.8 . . ?

H42A C42 H42B 109.5 . . ?

C30 C42 H42C 109.1 . . ?

H42A C42 H42C 109.5 . . ?

H42B C42 H42C 109.5 . . ?

C37 C43 H43A 111.2 . . ?

C37 C43 H43B 108.2 . . ?

H43A C43 H43B 109.5 . . ?

C37 C43 H43C 109.0 . . ?

H43A C43 H43C 109.5 . . ?

H43B C43 H43C 109.5 . . ?

C37 C44 H44A 109.6 . . ?

C37 C44 H44B 110.5 . . ?

H44A C44 H44B 109.5 . . ?

C37 C44 H44C 108.3 . . ?

H44A C44 H44C 109.5 . . ?

H44B C44 H44C 109.5 . . ?

C18 C45 H45A 110.5 . . ?

C18 C45 H45B 110.6 . . ?

H45A C45 H45B 109.5 . . ?

C18 C45 H45C 107.3 . . ?

H45A C45 H45C 109.5 . . ?

H45B C45 H45C 109.5 . . ?

C18 C46 H46A 109.9 . . ?

C18 C46 H46B 107.6 . . ?

H46A C46 H46B 109.5 . . ?

C18 C46 H46C 110.9 . . ?

H46A C46 H46C 109.5 . . ?

H46B C46 H46C 109.5 . . ?

C36 C47 H47A 110.8 . . ?

C36 C47 H47B 109.4 . . ?

H47A C47 H47B 109.5 . . ?

C36 C47 H47C 108.2 . . ?

H47A C47 H47C 109.5 . . ?

H47B C47 H47C 109.5 . . ?

C18 C48 H48A 110.4 . . ?

C18 C48 H48B 110.5 . . ?

H48A C48 H48B 109.5 . . ?

C18 C48 H48C 107.5 . . ?

H48A C48 H48C 109.5 . . ?

H48B C48 H48C 109.5 . . ?

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_refine_diff_density_min -0.364

_refine_diff_density_rms 0.103

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#===END

(1) ORTEP drawing (Fig. S1) and CIF file of **7a**

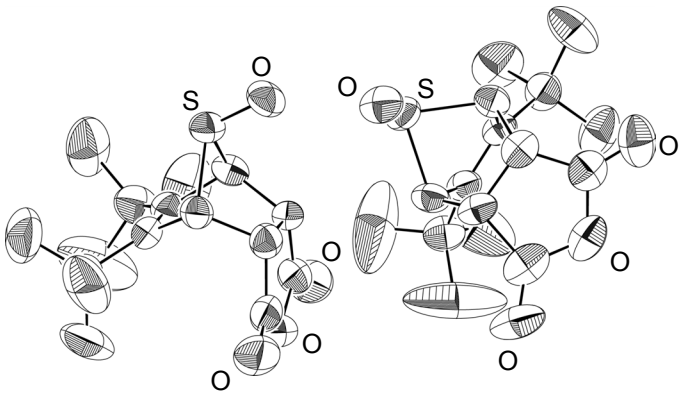

Figure S1. Molecular Sturucture of **7a** (the analysis was performed on two independent molecules)

CIF Copied by cif2cif, version 0.0.8 - beta (2 Apr 98)

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_publ_contact_author_name 'Dr. Juzo Nakayama'

_publ_contact_author_address

;

Department of Chemistry, Faculty of Science, Saitama University

Saitama, Saitama 338-8570, Japan

;

_publ_contact_author_email 'nakaj@post.saitama-u.ac.jp'

_publ_contact_author_fax '+81-48-858-3700'

_publ_contact_author_phone '+81-48-858-3390'

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Crystal and molecular structure

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The study of the titled structure was undertaken to establish its three dimensional structure. Geometries are tabulated below. All diagrams and calculations were performed using maXus (Bruker Nonius, Delft & MacScience, Japan).

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multi-scan from symmetry-related measurements

Sortav (Blessing 1995)

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 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
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O3 O 0.19732(18) 0.0140(2) 0.0327(3) 0.0663(12) Uani 1 1 d . . .

C4 C 0.1757(2) 0.0106(3) 0.5164(4) 0.0470(14) Uani 1 1 d . . .

C5 C 0.3400(2) 0.0756(3) 0.0035(4) 0.0479(14) Uani 1 1 d . . .

O6 O 0.28298(18) 0.0764(2) 0.3389(3) 0.0682(13) Uani 1 1 d . . .

C7 C 0.2073(3) 0.0684(2) 0.4818(4) 0.0488(15) Uani 1 1 d . . .

C8 C 0.3378(3) 0.0252(2) 0.1381(4) 0.0511(15) Uani 1 1 d . . .

C9 C 0.2239(3) 0.0631(4) -0.0112(5) 0.0648(19) Uani 1 1 d . . .

O10 O 0.2049(2) 0.0772(3) -0.0817(3) 0.0928(17) Uani 1 1 d . . .

C11 C 0.1586(2) -0.0258(3) 0.4487(4) 0.0488(15) Uani 1 1 d . . .

C12 C 0.2752(3) 0.0899(3) 0.0409(4) 0.0518(15) Uani 1 1 d . . .

O13 O 0.37620(18) 0.14659(17) 0.1400(3) 0.0641(12) Uani 1 1 d . . .

O14 O 0.0421(2) 0.0478(3) 0.2927(4) 0.0951(18) Uani 1 1 d . . .

C15 C 0.2723(2) 0.0561(2) 0.1272(4) 0.0463(15) Uani 1 1 d . . .

O16 O 0.2021(2) -0.0279(2) 0.1640(3) 0.0826(15) Uani 1 1 d . . .

C17 C 0.1469(2) 0.0686(3) 0.3476(4) 0.0521(16) Uani 1 1 d . . .

C18 C 0.3517(2) 0.0059(3) -0.0104(4) 0.0521(16) Uani 1 1 d . . .

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C22 C 0.0763(3) 0.0665(3) 0.3474(6) 0.071(2) Uani 1 1 d . . .

C23 C 0.1781(3) 0.0063(3) 0.3645(4) 0.0530(16) Uani 1 1 d . . .

C24 C 0.3651(3) -0.0164(3) -0.1033(5) 0.0674(19) Uani 1 1 d . . .

O25 O 0.0915(2) 0.1460(2) 0.5357(4) 0.0866(16) Uani 1 1 d . . .

C26 C 0.1618(3) 0.1069(3) 0.4257(4) 0.0539(16) Uani 1 1 d . . .

C27 C 0.2219(3) 0.0100(3) 0.1148(4) 0.0577(16) Uani 1 1 d . . .

C28 C 0.1696(3) 0.0034(3) 0.6163(4) 0.0611(17) Uani 1 1 d . . .

C29 C 0.1026(3) 0.1195(3) 0.4706(5) 0.0603(18) Uani 1 1 d . . .

C30 C 0.1043(3) -0.0019(4) 0.6447(5) 0.088(2) Uani 1 1 d . . .

C31 C 0.3462(6) -0.1406(4) 0.0400(7) 0.168(5) Uani 1 1 d . . .

C32 C 0.3134(4) -0.0570(5) -0.1381(6) 0.127(4) Uani 1 1 d . . .

C33 C 0.2106(4) -0.0541(4) 0.6468(5) 0.097(3) Uani 1 1 d . . .

C34 C 0.4354(4) -0.0953(4) 0.1116(6) 0.118(3) Uani 1 1 d . . .

C35 C 0.3639(5) 0.0381(5) -0.1691(5) 0.124(3) Uani 1 1 d . . .

C36 C 0.3645(4) -0.0885(3) 0.0992(5) 0.075(2) Uani 1 1 d . . .

C37 C 0.3407(4) -0.0996(4) 0.1906(7) 0.119(3) Uani 1 1 d . . .

C38 C 0.0811(6) -0.0898(5) 0.3754(9) 0.212(8) Uani 1 1 d . . .

C42 C 0.1090(6) -0.1244(4) 0.5150(7) 0.159(5) Uani 1 1 d . . .

C48 C 0.4288(4) -0.0432(5) -0.1117(6) 0.127(4) Uani 1 1 d . . .

C49 C 0.1971(4) 0.0601(4) 0.6633(5) 0.103(3) Uani 1 1 d . . .

C50 C 0.1785(5) -0.1309(4) 0.4001(11) 0.243(10) Uani 1 1 d . . .

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H7 H 0.2254 0.0906 0.5298 0.059 Uiso 1 1 d R . .

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H12 H 0.2691 0.1341 0.0455 0.062 Uiso 1 1 d R . .

H15 H 0.2635 0.0854 0.1727 0.056 Uiso 1 1 d R . .

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H23 H 0.1719 -0.0221 0.3170 0.064 Uiso 1 1 d R . .

H26 H 0.1807 0.1454 0.4080 0.065 Uiso 1 1 d R . .

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H30B H 0.0851 -0.0373 0.6177 0.106 Uiso 1 1 d R . .

H30C H 0.0825 0.0353 0.6280 0.106 Uiso 1 1 d R . .

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H31B H 0.3668 -0.1363 -0.0152 0.202 Uiso 1 1 d R . .

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H32B H 0.2751 -0.0338 -0.1385 0.153 Uiso 1 1 d R . .

H32C H 0.3089 -0.0927 -0.1011 0.153 Uiso 1 1 d R . .

H33A H 0.2072 -0.0589 0.7087 0.116 Uiso 1 1 d R . .

H33B H 0.2534 -0.0471 0.6315 0.116 Uiso 1 1 d R . .

H33C H 0.1960 -0.0913 0.6187 0.116 Uiso 1 1 d R . .

H34A H 0.4460 -0.1363 0.1315 0.141 Uiso 1 1 d R . .

H34B H 0.4496 -0.0651 0.1531 0.141 Uiso 1 1 d R . .

H34C H 0.4550 -0.0877 0.0564 0.141 Uiso 1 1 d R . .

H35A H 0.3724 0.0233 -0.2269 0.149 Uiso 1 1 d R . .

H35B H 0.3953 0.0676 -0.1525 0.149 Uiso 1 1 d R . .

H35C H 0.3238 0.0578 -0.1679 0.149 Uiso 1 1 d R . .

H37A H 0.3504 -0.1413 0.2087 0.142 Uiso 1 1 d R . .

H37B H 0.2964 -0.0936 0.1918 0.142 Uiso 1 1 d R . .

H37C H 0.3603 -0.0705 0.2294 0.142 Uiso 1 1 d R . .

H38A H 0.0636 -0.1305 0.3673 0.255 Uiso 1 1 d R . .

H38B H 0.0932 -0.0730 0.3200 0.255 Uiso 1 1 d R . .

H38C H 0.0507 -0.0631 0.4019 0.255 Uiso 1 1 d R . .

H42A H 0.0937 -0.1648 0.4989 0.191 Uiso 1 1 d R . .

H42B H 0.0756 -0.1001 0.5387 0.191 Uiso 1 1 d R . .

H42C H 0.1413 -0.1288 0.5578 0.191 Uiso 1 1 d R . .

H48A H 0.4362 -0.0570 -0.1702 0.153 Uiso 1 1 d R . .

H48B H 0.4326 -0.0779 -0.0726 0.153 Uiso 1 1 d R . .

H48C H 0.4588 -0.0119 -0.0965 0.153 Uiso 1 1 d R . .

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H49B H 0.1743 0.0964 0.6458 0.123 Uiso 1 1 d R . .

H49C H 0.2403 0.0652 0.6486 0.123 Uiso 1 1 d R . .

H50A H 0.1633 -0.1726 0.3918 0.291 Uiso 1 1 d R . .

H50B H 0.2129 -0.1315 0.4401 0.291 Uiso 1 1 d R . .

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C4 0.039(3) 0.053(3) 0.048(4) 0.009(3) -0.003(3) 0.000(3)

C5 0.047(3) 0.055(4) 0.041(4) 0.000(3) 0.005(3) -0.006(3)

O6 0.058(3) 0.095(3) 0.052(3) 0.015(2) 0.006(2) -0.021(2)

C7 0.056(3) 0.051(3) 0.040(4) 0.005(3) -0.005(3) 0.000(3)

C8 0.060(4) 0.052(3) 0.042(4) 0.000(3) -0.008(3) -0.005(3)

C9 0.048(4) 0.090(5) 0.057(5) -0.009(4) -0.001(4) 0.017(4)

O10 0.080(3) 0.134(5) 0.065(4) 0.004(3) -0.022(3) 0.018(3)

C11 0.040(3) 0.059(4) 0.048(4) 0.002(3) -0.005(3) -0.002(3)

C12 0.053(4) 0.054(3) 0.049(4) 0.001(3) -0.001(3) 0.008(3)

O13 0.072(3) 0.051(2) 0.069(3) -0.016(2) 0.000(2) -0.012(2)

O14 0.062(3) 0.128(4) 0.096(4) 0.022(3) -0.031(3) -0.017(3)

C15 0.040(3) 0.049(3) 0.050(4) -0.009(3) -0.003(3) -0.002(3)

O16 0.080(3) 0.093(4) 0.075(4) 0.005(3) 0.013(3) -0.026(3)

C17 0.042(3) 0.069(4) 0.046(4) 0.018(3) -0.004(3) -0.011(3)

C18 0.038(3) 0.063(4) 0.055(4) -0.008(3) 0.001(3) 0.003(3)

C19 0.061(4) 0.053(4) 0.067(5) 0.008(3) -0.006(4) -0.010(3)

C20 0.047(3) 0.047(3) 0.058(4) -0.004(3) -0.007(3) 0.005(3)

O21 0.056(3) 0.077(3) 0.088(4) 0.020(3) 0.009(3) 0.014(2)

C22 0.061(5) 0.070(5) 0.083(6) 0.034(4) -0.006(4) 0.000(4)

C23 0.053(3) 0.068(4) 0.037(4) 0.001(3) -0.008(3) -0.012(3)

C24 0.055(4) 0.093(5) 0.054(4) -0.018(4) 0.006(3) -0.001(3)

O25 0.104(4) 0.071(3) 0.085(4) 0.001(3) 0.016(3) 0.028(3)

C26 0.053(3) 0.048(3) 0.062(4) 0.014(3) 0.004(3) -0.004(3)

C27 0.059(4) 0.070(4) 0.044(5) -0.004(4) 0.002(3) 0.002(3)

C28 0.060(4) 0.079(5) 0.044(4) 0.009(3) 0.002(3) 0.008(3)

C29 0.066(5) 0.044(4) 0.071(5) 0.013(4) -0.004(4) 0.009(3)

C30 0.069(5) 0.112(6) 0.084(6) 0.025(5) 0.025(4) 0.009(4)

C31 0.283(15) 0.062(5) 0.160(11) 0.008(6) -0.086(10) -0.028(7)

C32 0.122(7) 0.179(9) 0.080(7) -0.057(6) -0.017(6) -0.038(7)

C33 0.094(6) 0.126(7) 0.070(6) 0.038(5) -0.013(5) 0.013(5)

C34 0.133(8) 0.130(7) 0.090(7) 0.016(6) -0.004(6) 0.074(6)

C35 0.173(10) 0.148(9) 0.051(6) -0.008(6) 0.019(6) 0.014(7)

C36 0.091(5) 0.055(4) 0.079(6) -0.003(4) -0.020(5) 0.007(4)

C37 0.123(7) 0.076(5) 0.157(10) 0.048(6) 0.031(7) 0.024(5)

C38 0.256(15) 0.127(9) 0.254(15) 0.069(10) -0.181(13) -0.112(10)

C42 0.272(14) 0.092(6) 0.114(9) 0.014(6) -0.010(9) -0.090(8)

C48 0.095(6) 0.208(11) 0.079(7) -0.017(7) 0.023(5) 0.046(7)

C49 0.156(8) 0.109(6) 0.043(5) -0.010(4) -0.009(5) -0.003(6)

C50 0.163(11) 0.072(6) 0.49(3) -0.086(11) 0.175(15) -0.034(6)

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All esds (except the esd in the dihedral angle between two l.s. planes)

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  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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S2 C5 1.822(6) . ?
S2 C8 1.822(6) . ?
O3 C27 1.370(7) . ?
O3 C9 1.377(8) . ?
C4 C11 1.352(8) . ?

C4 C7 1.510(7) . ?
C4 C28 1.549(8) . ?
C5 C18 1.532(8) . ?
C5 C12 1.537(7) . ?
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C7 C26 1.544(8) . ?

C8 C20 1.502(8) . ?

C8 C15 1.562(7) . ?

C9 O10 1.197(7) . ?

C9 C12 1.478(8) . ?

C11 C23 1.525(8) . ?

C11 C19 1.537(8) . ?

C12 C15 1.513(8) . ?

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C15 C27 1.480(8) . ?

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S1 0.0216(3) 0.0242(2) 0.0157(2) 0.00274(15) -0.00021(15) 0.00134(17)

N2 0.0260(8) 0.0272(8) 0.0159(6) 0.0008(5) 0.0017(5) -0.0003(6)

O3 0.0378(8) 0.0401(8) 0.0248(6) 0.0093(6) 0.0094(5) -0.0098(6)

O4 0.0360(8) 0.0370(8) 0.0271(7) -0.0032(6) -0.0003(5) -0.0134(6)

C5 0.0189(9) 0.0188(8) 0.0149(7) -0.0013(6) 0.0028(6) 0.0015(6)

C6 0.0184(9) 0.0169(8) 0.0159(7) 0.0013(6) 0.0035(6) -0.0008(6)

C7 0.0184(9) 0.0177(8) 0.0142(7) -0.0008(6) 0.0035(6) 0.0005(6)

O8 0.0243(7) 0.0339(8) 0.0319(7) 0.0102(6) 0.0010(5) 0.0097(6)

C9 0.0165(9) 0.0204(8) 0.0164(7) 0.0017(6) 0.0010(6) -0.0017(6)

C10 0.0203(9) 0.0240(9) 0.0297(9) 0.0005(7) 0.0110(7) 0.0018(7)

C11 0.0177(9) 0.0236(9) 0.0178(7) 0.0008(6) 0.0059(6) 0.0020(7)

C12 0.0270(10) 0.0274(10) 0.0158(7) 0.0000(6) 0.0084(6) 0.0006(7)

C13 0.0279(10) 0.0216(9) 0.0177(8) -0.0040(6) 0.0052(7) -0.0016(7)

C14 0.0219(9) 0.0162(8) 0.0187(8) 0.0001(6) 0.0048(6) 0.0017(6)

C15 0.0255(10) 0.0179(9) 0.0274(9) 0.0014(7) 0.0050(7) 0.0011(7)

C16 0.0345(12) 0.0435(12) 0.0228(9) 0.0057(8) -0.0044(8) -0.0019(9)

C17 0.0393(12) 0.0291(11) 0.0351(10) 0.0107(8) 0.0004(9) 0.0051(9)

C18 0.0429(13) 0.0447(13) 0.0378(11) -0.0034(9) 0.0273(9) -0.0022(10)

C19 0.0326(12) 0.0202(10) 0.0512(12) 0.0047(8) 0.0072(9) -0.0037(8)

C20 0.0300(12) 0.0311(11) 0.0591(13) -0.0047(9) 0.0211(10) -0.0091(9)

C21 0.0450(13) 0.0196(10) 0.0460(12) -0.0058(8) 0.0166(9) 0.0021(8)

C22 0.0195(11) 0.0457(13) 0.0624(14) 0.0176(11) 0.0033(9) 0.0011(9)

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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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C5 C7 1.366(2) . ?

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C5 C15 1.543(2) . ?

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C10 C18 1.540(3) . ?

C10 C20 1.541(3) . ?

C11 C12 1.507(2) . ?

C11 C14 1.521(2) . ?

C13 C14 1.512(2) . ?

C15 C21 1.533(3) . ?

C15 C19 1.534(3) . ?

C15 C17 1.542(2) . ?

C6 H6 0.9601 . ?

C9 H9 0.9639 . ?

C11 H11 0.9667 . ?

C14 H14 0.9193 . ?

C16 H16A 0.9383 . ?

C16 H16B 0.9429 . ?

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C17 H17B 0.9910 . ?

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C18 H18B 1.0185 . ?

C18 H18C 0.9913 . ?

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C7 C6 C14 112.31(12) . . ?

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C22 C10 C18 112.05(17) . . ?

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C21 C15 C17 110.38(16) . . ?

C19 C15 C17 106.54(15) . . ?

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C17 C15 C5 110.36(14) . . ?

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S1 C6 H6 110.7 . . ?

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C6 C14 H14 110.2 . . ?

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N2 C16 H16B 107.6 . . ?

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N2 C16 H16C 109.5 . . ?

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C15 C17 H17A 108.9 . . ?

C15 C17 H17B 113.3 . . ?

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C15 C17 H17C 109.8 . . ?

H17A C17 H17C 106.3 . . ?

H17B C17 H17C 107.9 . . ?

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C15 C21 H21B 112.3 . . ?

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C15 C21 H21C 112.5 . . ?

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Sortav (Blessing 1995)

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C6 C 0.7061(3) 0.17654(17) 0.4843(2) 0.0431(6) Uani 1 1 d . . .

C7 C 0.8291(3) 0.24413(18) 0.4844(2) 0.0424(6) Uani 1 1 d . . .

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C9 C 0.6352(3) 0.06016(18) 0.2114(2) 0.0454(6) Uani 1 1 d . . .

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H13B H 0.7351 0.3514 0.2555 0.063 Uiso 1 1 d R . .

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C3 0.0387(14) 0.0433(15) 0.0441(13) 0.0019(11) 0.0175(12) 0.0088(11)

C4 0.0370(14) 0.0403(14) 0.0471(14) -0.0002(12) 0.0181(12) -0.0004(11)

C5 0.0306(13) 0.0486(16) 0.0446(13) -0.0046(12) 0.0101(11) 0.0044(12)

C6 0.0412(15) 0.0475(16) 0.0443(13) 0.0009(12) 0.0217(12) 0.0045(12)

C7 0.0350(13) 0.0482(16) 0.0386(13) -0.0063(11) 0.0100(11) 0.0015(11)

O8 0.0761(15) 0.0901(18) 0.0437(11) 0.0141(11) 0.0104(11) 0.0280(13)

C9 0.0483(15) 0.0432(16) 0.0444(14) -0.0027(11) 0.0190(13) -0.0024(12)

C10 0.0431(16) 0.0437(17) 0.0584(16) -0.0027(13) 0.0128(13) 0.0038(12)

C11 0.0495(16) 0.0555(19) 0.0496(15) -0.0061(13) 0.0231(14) -0.0086(14)

C12 0.0339(15) 0.070(2) 0.0733(19) -0.0118(17) 0.0219(15) -0.0028(14)

C13 0.0540(17) 0.0424(16) 0.0530(16) 0.0076(13) 0.0133(14) -0.0055(13)

C14 0.079(2) 0.072(2) 0.0573(17) -0.0178(16) 0.0410(18) -0.0140(18)

C15 0.067(2) 0.0462(19) 0.072(2) -0.0148(16) 0.0247(17) -0.0088(15)

C16 0.071(2) 0.0485(19) 0.068(2) -0.0073(15) 0.0179(18) -0.0130(16)

C17 0.058(2) 0.068(2) 0.079(2) -0.0084(18) 0.0280(18) -0.0192(17)

C18 0.0464(19) 0.077(3) 0.152(4) 0.006(3) 0.030(2) 0.0167(19)

C19 0.091(3) 0.095(3) 0.0464(16) -0.0045(18) 0.0181(18) 0.026(2)

C20 0.0520(19) 0.091(3) 0.110(3) -0.011(2) 0.036(2) -0.0212(19)

C23 0.051(2) 0.194(6) 0.114(3) -0.056(3) 0.049(2) -0.014(3)

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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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C7 C10 1.521(4) . ?

C9 C19 1.506(4) . ?

C9 C15 1.517(4) . ?

C9 C14 1.542(4) . ?

C10 C16 1.533(4) . ?

C10 C13 1.534(4) . ?

C11 C13 1.505(4) . ?

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C11 H11 0.9599 . ?

C13 H13A 0.9600 . ?

C13 H13B 0.9600 . ?

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C15 H15B 0.9599 . ?

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C16 H16B 0.9602 . ?

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C18 H18A 0.9600 . ?

C18 H18B 0.9599 . ?

C18 H18C 0.9600 . ?

C19 H19A 0.9601 . ?

C19 H19B 0.9599 . ?

C19 H19C 0.9600 . ?

C20 H20A 0.9600 . ?

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C3 C5 C11 121.1(2) . . ?

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C7 C6 S1 102.68(17) . . ?

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C11 C13 H13B 116.3 . . ?

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C9 C14 H14B 109.5 . . ?

H14A C14 H14B 109.5 . . ?

C9 C14 H14C 109.5 . . ?

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C9 C15 H15B 109.6 . . ?

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C9 C15 H15C 108.8 . . ?

H15A C15 H15C 109.5 . . ?

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C11 C17 H17B 109.5 . . ?

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 Refinement of F^2^ against ALL reflections.    The weighted R-factor wR and
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on F, with F set to zero for negative F^2^. The threshold expression of F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) 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.

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O3 O 0.3929(5) 0.14582(16) 0.8842(3) 0.0741(10) Uani 1 1 d . . .
O4 O 0.1718(6) 0.06624(16) 0.8057(2) 0.0764(10) Uani 1 1 d . . .
C5 C -0.1098(7) 0.2649(2) 0.8366(3) 0.0595(12) Uani 1 1 d . . .
C6 C 0.2624(7) 0.0665(2) 0.9790(3) 0.0563(11) Uani 1 1 d . . .
C7 C 0.3177(7) -0.0004(2) 1.1380(3) 0.0627(12) Uani 1 1 d . . .
N8 N 0.0423(6) 0.15045(16) 0.8780(3) 0.0604(10) Uani 1 1 d . . .
C9 C -0.0651(7) 0.3247(2) 0.8888(3) 0.0565(11) Uani 1 1 d . . .
C10 C 0.3176(9) 0.0954(2) 1.0611(4) 0.0745(15) Uani 1 1 d . . .
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C13 C 0.2340(8) 0.0043(2) 0.9764(4) 0.0712(14) Uani 1 1 d . . .

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H26B H 0.2240 0.3892 0.8507 0.207 Uiso 1 1 d R . .

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C6 0.059(3) 0.047(2) 0.062(3) -0.004(2) 0.015(2) 0.001(2)

C7 0.061(3) 0.060(3) 0.068(3) 0.009(2) 0.018(2) 0.007(2)

N8 0.059(2) 0.0431(19) 0.078(3) -0.0023(18) 0.0158(19) 0.0030(17)

C9 0.062(3) 0.044(2) 0.065(3) 0.003(2) 0.018(2) 0.0003(19)

C10 0.102(4) 0.046(3) 0.069(3) -0.003(2) 0.008(3) -0.006(3)

C11 0.059(3) 0.049(2) 0.083(4) -0.005(2) 0.013(2) 0.001(2)

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C13 0.096(4) 0.046(2) 0.068(3) -0.008(2) 0.014(3) 0.000(2)

C14 0.086(4) 0.048(3) 0.088(4) 0.003(2) 0.040(3) 0.001(2)

C15 0.082(3) 0.051(3) 0.056(3) 0.004(2) 0.012(2) 0.002(2)

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C22 0.098(4) 0.049(3) 0.091(4) 0.018(3) 0.029(3) 0.009(3)

C23 0.091(4) 0.083(4) 0.078(4) 0.015(3) 0.023(3) 0.009(3)

O24 0.100(4) 0.104(4) 0.184(6) -0.002(4) -0.029(4) 0.032(3)

O25 0.219(7) 0.127(5) 0.187(6) 0.002(4) 0.155(6) 0.010(5)

C26 0.141(8) 0.105(6) 0.282(14) 0.088(8) 0.071(8) -0.020(6)

C27 0.088(8) 0.165(15) 0.105(10) -0.055(10) -0.017(7) -0.011(9)

C28 0.139(6) 0.067(4) 0.137(6) 0.021(4) 0.031(5) 0.036(4)

C30 0.240(19) 0.136(12) 0.067(7) -0.029(7) 0.064(10) -0.067(13)

C33 0.43(2) 0.074(5) 0.085(5) 0.037(4) 0.084(8) 0.036(8)

C34 0.140(12) 0.079(7) 0.102(9) -0.021(6) 0.029(8) 0.029(8)

C36 0.149(19) 0.109(15) 0.054(9) -0.024(9) -0.010(10) 0.019(14)

C35 0.32(4) 0.16(2) 0.099(17) -0.036(16) -0.01(2) -0.15(3)

C37 0.17(3) 0.27(4) 0.16(3) -0.15(3) 0.02(2) 0.07(3)

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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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S2 C6 1.750(5) . ?

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O16 C20 1.370(9) . ?

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C18 C35 1.725(7) . ?

C20 O24 1.179(9) . ?

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C26 H26B 0.9599 . ?

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C5 S1 C15 79.3(2) . . ?

O4 S2 O3 117.6(2) . . ?

O4 S2 N8 107.7(2) . . ?

O3 S2 N8 110.6(2) . . ?

O4 S2 C6 107.5(2) . . ?

O3 S2 C6 108.4(2) . . ?

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C9 C5 C11 113.0(4) . . ?

C9 C5 S1 97.8(3) . . ?

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C10 C6 S2 120.4(4) . . ?

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C17 C7 C19 117.2(5) . . ?

C17 C7 C23 120.6(5) . . ?

C19 C7 C23 122.2(5) . . ?

S1 N8 S2 115.3(2) . . ?

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Sortav (Blessing 1995)

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C6 C 0.6387(6) 0.10089(14) 1.0057(4) 0.0497(11) Uani 1 1 d . . .

N7 N 0.7124(5) 0.16745(12) 0.8265(3) 0.0576(10) Uani 1 1 d . . .

C8 C 0.5204(6) 0.12397(13) 0.9187(4) 0.0475(11) Uani 1 1 d . . .

C9 C 0.7544(6) 0.06945(14) 0.9734(3) 0.0478(11) Uani 1 1 d . . .

C10 C 0.8443(6) 0.01794(15) 0.8252(4) 0.0486(11) Uani 1 1 d . . .

C11 C 0.6069(5) 0.12440(13) 0.8087(4) 0.0471(11) Uani 1 1 d . . .

C12 C 0.4239(7) 0.23472(17) 0.6748(5) 0.0727(15) Uani 1 1 d . . .

O13 O 0.6380(4) 0.07173(12) 0.6029(3) 0.0704(10) Uani 1 1 d . . .

N14 N 0.7007(5) 0.08391(12) 0.7845(3) 0.0598(11) Uani 1 1 d . . .

C15 C 0.8395(6) -0.01544(15) 0.6392(4) 0.0521(11) Uani 1 1 d . . .

C16 C 0.3283(6) 0.10748(17) 0.9050(4) 0.0607(13) Uani 1 1 d . . .

C17 C 0.7031(6) 0.06049(16) 0.6892(4) 0.0549(12) Uani 1 1 d . . .

C18 C 0.6478(7) 0.11205(16) 1.1282(4) 0.0599(13) Uani 1 1 d . . .

C19 C 1.0091(6) -0.02925(16) 0.6326(4) 0.0578(12) Uani 1 1 d . . .

C20 C 0.7072(7) -0.03577(17) 0.5760(4) 0.0683(14) Uani 1 1 d . . .

C21 C 0.9189(9) -0.08447(18) 0.4982(5) 0.0799(17) Uani 1 1 d . . .

C22 C 0.5562(6) 0.20658(17) 0.6391(5) 0.0633(14) Uani 1 1 d . . .

O23 O 0.8701(5) 0.17377(17) 0.6574(4) 0.1159(17) Uani 1 1 d . . .

O24 O 0.7983(6) 0.24445(14) 0.7645(4) 0.130(2) Uani 1 1 d . . .

C25 C 0.5360(8) 0.18419(19) 0.5427(5) 0.0799(16) Uani 1 1 d . . .

C26 C 1.0473(8) -0.06379(19) 0.5621(4) 0.0733(15) Uani 1 1 d . . .

C27 C 0.6010(8) 0.06839(19) 1.1927(4) 0.0888(18) Uani 1 1 d . . .

C28 C 0.7505(8) -0.07057(19) 0.5052(5) 0.0821(17) Uani 1 1 d . . .

C29 C 0.2722(8) 0.2388(2) 0.6114(6) 0.0850(18) Uani 1 1 d . . .

C30 C 0.8365(8) 0.1253(2) 1.1641(5) 0.098(2) Uani 1 1 d . . .

C31 C 0.2529(9) 0.2167(2) 0.5139(6) 0.091(2) Uani 1 1 d . . .

C33 C 0.2286(7) 0.1297(3) 0.8098(6) 0.131(3) Uani 1 1 d . . .

C34 C 0.3843(10) 0.1898(2) 0.4797(5) 0.0907(19) Uani 1 1 d . . .

C35 C 0.5357(9) 0.1516(2) 1.1673(5) 0.113(2) Uani 1 1 d . . .

C36 C 0.2250(8) 0.1158(3) 1.0031(5) 0.129(3) Uani 1 1 d . . .

C40 C 0.3324(8) 0.0551(2) 0.8862(7) 0.134(3) Uani 1 1 d . . .

C41 C 0.0842(8) 0.2227(3) 0.4450(6) 0.136(3) Uani 1 1 d . . .

H9 H 0.8212 0.0540 1.0307 0.057 Uiso 1 1 d R . .

H11 H 0.5185 0.1272 0.7512 0.056 Uiso 1 1 d R . .

H12 H 0.4414 0.2501 0.7439 0.087 Uiso 1 1 d R . .

H19 H 1.0985 -0.0147 0.6781 0.069 Uiso 1 1 d R . .

H20 H 0.5896 -0.0253 0.5828 0.082 Uiso 1 1 d R . .

H21 H 0.9481 -0.1093 0.4503 0.096 Uiso 1 1 d R . .

H25 H 0.6266 0.1638 0.5208 0.096 Uiso 1 1 d R . .

H26 H 1.1657 -0.0735 0.5548 0.088 Uiso 1 1 d R . .

H27A H 0.6071 0.0750 1.2695 0.107 Uiso 1 1 d R . .

H27B H 0.6813 0.0439 1.1775 0.107 Uiso 1 1 d R . .

H27C H 0.4850 0.0588 1.1707 0.107 Uiso 1 1 d R . .

H28 H 0.6621 -0.0854 0.4595 0.099 Uiso 1 1 d R . .

H29 H 0.1802 0.2581 0.6359 0.102 Uiso 1 1 d R . .

H30A H 0.8436 0.1323 1.2406 0.118 Uiso 1 1 d R . .

H30B H 0.8710 0.1520 1.1236 0.118 Uiso 1 1 d R . .

H30C H 0.9124 0.0997 1.1498 0.118 Uiso 1 1 d R . .

H33A H 0.1109 0.1184 0.8047 0.157 Uiso 1 1 d R . .

H33B H 0.2854 0.1220 0.7442 0.157 Uiso 1 1 d R . .

H33C H 0.2279 0.1628 0.8194 0.157 Uiso 1 1 d R . .

H34 H 0.3715 0.1751 0.4095 0.109 Uiso 1 1 d R . .

H35A H 0.5527 0.1555 1.2448 0.135 Uiso 1 1 d R . .

H35B H 0.4156 0.1445 1.1496 0.135 Uiso 1 1 d R . .

H35C H 0.5668 0.1798 1.1313 0.135 Uiso 1 1 d R . .

H36A H 0.1080 0.1048 0.9905 0.154 Uiso 1 1 d R . .

H36B H 0.2232 0.1485 1.0187 0.154 Uiso 1 1 d R . .

H36C H 0.2793 0.0995 1.0639 0.154 Uiso 1 1 d R . .

H40A H 0.2154 0.0435 0.8778 0.161 Uiso 1 1 d R . .

H40B H 0.3913 0.0403 0.9475 0.161 Uiso 1 1 d R . .

H40C H 0.3930 0.0486 0.8214 0.161 Uiso 1 1 d R . .

H41A H 0.0914 0.2047 0.3795 0.163 Uiso 1 1 d R . .

H41B H 0.0680 0.2549 0.4266 0.163 Uiso 1 1 d R . .

H41C H -0.0124 0.2120 0.4850 0.163 Uiso 1 1 d R . .

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S2 0.0566(8) 0.0786(10) 0.1088(13) 0.0429(9) -0.0163(8) -0.0177(8)

O3 0.079(2) 0.054(2) 0.065(2) 0.0028(17) -0.0093(18) 0.0249(18)

N4 0.056(2) 0.046(2) 0.055(2) -0.0016(18) 0.004(2) 0.0142(18)

N5 0.063(2) 0.046(2) 0.048(2) 0.0018(18) -0.0031(19) 0.0126(19)

C6 0.055(3) 0.043(2) 0.051(3) 0.002(2) 0.006(2) 0.002(2)

N7 0.049(2) 0.049(2) 0.074(3) 0.008(2) -0.007(2) -0.0038(18)

C8 0.052(3) 0.031(2) 0.059(3) 0.002(2) 0.004(2) 0.001(2)

C9 0.058(3) 0.042(2) 0.043(3) 0.003(2) 0.000(2) 0.006(2)

C10 0.052(3) 0.047(3) 0.047(3) 0.001(2) -0.001(2) 0.006(2)

C11 0.046(3) 0.038(2) 0.057(3) 0.001(2) -0.003(2) 0.006(2)

C12 0.073(4) 0.057(3) 0.087(4) 0.015(3) -0.013(3) -0.005(3)

O13 0.081(2) 0.075(2) 0.055(2) 0.0065(18) 0.0001(19) 0.0257(19)

N14 0.081(3) 0.051(2) 0.048(2) 0.0035(19) 0.001(2) 0.026(2)

C15 0.062(3) 0.041(2) 0.053(3) 0.005(2) 0.004(2) 0.007(2)

C16 0.047(3) 0.060(3) 0.075(4) 0.004(3) -0.001(3) -0.003(2)

C17 0.056(3) 0.056(3) 0.052(3) 0.007(3) 0.002(2) 0.015(2)

C18 0.074(3) 0.049(3) 0.057(3) 0.000(2) 0.007(3) 0.013(3)

C19 0.055(3) 0.060(3) 0.058(3) -0.007(2) -0.003(2) 0.012(2)

C20 0.060(3) 0.060(3) 0.084(4) -0.004(3) -0.004(3) -0.001(3)

C21 0.110(5) 0.061(3) 0.069(4) -0.011(3) 0.005(4) 0.019(4)

C22 0.055(3) 0.049(3) 0.084(4) 0.024(3) -0.012(3) -0.010(3)

O23 0.056(2) 0.159(4) 0.135(4) 0.070(3) 0.030(3) 0.027(3)

O24 0.117(3) 0.081(3) 0.182(5) 0.059(3) -0.080(3) -0.061(3)

C25 0.092(4) 0.069(4) 0.078(4) 0.022(3) -0.004(4) 0.002(3)

C26 0.075(4) 0.076(4) 0.070(4) 0.003(3) 0.011(3) 0.021(3)

C27 0.115(5) 0.082(4) 0.071(4) 0.009(3) 0.022(4) 0.005(4)

C28 0.090(5) 0.067(3) 0.088(4) -0.023(3) -0.016(3) -0.004(3)

C29 0.065(4) 0.071(4) 0.118(5) 0.036(4) -0.001(4) 0.005(3)

C30 0.103(5) 0.110(5) 0.081(4) -0.027(4) 0.003(4) -0.017(4)

C31 0.077(4) 0.092(5) 0.101(5) 0.042(4) -0.021(4) -0.028(4)

C33 0.055(4) 0.178(8) 0.156(7) 0.060(6) -0.027(4) -0.013(4)

C34 0.119(6) 0.077(4) 0.075(4) 0.016(3) -0.015(4) -0.020(4)

C35 0.153(6) 0.095(5) 0.089(5) -0.021(4) -0.003(4) 0.053(5)

C36 0.065(4) 0.191(8) 0.131(6) -0.011(6) 0.028(4) -0.028(5)

C40 0.077(4) 0.076(4) 0.247(9) -0.011(5) -0.016(5) -0.034(4)

C41 0.089(5) 0.166(7) 0.146(7) 0.069(6) -0.059(5) -0.032(5)

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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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S1 C8 1.874(4) . ?

S2 O23 1.425(4) . ?

S2 O24 1.429(4) . ?

S2 N7 1.671(4) . ?

S2 C22 1.759(5) . ?

O3 C10 1.195(5) . ?

N4 C10 1.375(5) . ?

N4 C17 1.403(5) . ?

N4 C15 1.435(5) . ?

N5 C9 1.358(5) . ?

N5 C10 1.381(5) . ?

N5 N14 1.382(5) . ?

C6 C9 1.342(6) . ?

C6 C8 1.522(6) . ?

C6 C18 1.536(6) . ?

N7 C11 1.490(5) . ?

C8 C11 1.533(6) . ?

C8 C16 1.553(6) . ?

C11 N14 1.409(5) . ?

C12 C29 1.376(7) . ?

C12 C22 1.387(7) . ?

O13 C17 1.194(5) . ?

N14 C17 1.351(6) . ?

C15 C19 1.369(6) . ?

C15 C20 1.379(6) . ?

C16 C36 1.494(7) . ?

C16 C33 1.507(7) . ?

C16 C40 1.524(7) . ?

C18 C35 1.519(7) . ?

C18 C27 1.536(7) . ?

C18 C30 1.543(7) . ?

C19 C26 1.359(6) . ?

C20 C28 1.377(7) . ?

C21 C28 1.362(8) . ?

C21 C26 1.367(7) . ?

C22 C25 1.350(7) . ?

C25 C34 1.377(8) . ?

C29 C31 1.356(8) . ?

C31 C34 1.356(9) . ?

C31 C41 1.523(8) . ?

C9 H9 0.9599 . ?

C11 H11 0.9601 . ?

C12 H12 0.9600 . ?

C19 H19 0.9599 . ?

C20 H20 0.9600 . ?

C21 H21 0.9599 . ?

C25 H25 0.9601 . ?

C26 H26 0.9600 . ?

C27 H27A 0.9600 . ?

C27 H27B 0.9600 . ?

C27 H27C 0.9600 . ?

C28 H28 0.9599 . ?

C29 H29 0.9599 . ?

C30 H30A 0.9600 . ?

C30 H30B 0.9601 . ?

C30 H30C 0.9600 . ?

C33 H33A 0.9601 . ?

C33 H33B 0.9601 . ?

C33 H33C 0.9597 . ?

C34 H34 0.9600 . ?

C35 H35A 0.9599 . ?

C35 H35B 0.9600 . ?

C35 H35C 0.9600 . ?

C36 H36A 0.9598 . ?

C36 H36B 0.9603 . ?

C36 H36C 0.9597 . ?

C40 H40A 0.9600 . ?

C40 H40B 0.9600 . ?

C40 H40C 0.9600 . ?

C41 H41A 0.9599 . ?

C41 H41B 0.9600 . ?

C41 H41C 0.9599 . ?

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N7 S1 C8 76.78(18) . . ?

O23 S2 O24 122.0(3) . . ?

O23 S2 N7 105.0(2) . . ?

O24 S2 N7 103.7(3) . . ?

O23 S2 C22 108.4(3) . . ?

O24 S2 C22 108.6(3) . . ?

N7 S2 C22 108.3(2) . . ?

C10 N4 C17 111.8(4) . . ?

C10 N4 C15 123.6(4) . . ?

C17 N4 C15 124.5(4) . . ?

C9 N5 C10 128.8(4) . . ?

C9 N5 N14 119.9(4) . . ?

C10 N5 N14 108.3(3) . . ?

C9 C6 C8 117.9(4) . . ?

C9 C6 C18 115.5(4) . . ?

C8 C6 C18 126.5(4) . . ?

C11 N7 S2 118.1(3) . . ?

C11 N7 S1 90.7(3) . . ?

S2 N7 S1 119.2(2) . . ?

C6 C8 C11 110.6(3) . . ?

C6 C8 C16 118.4(4) . . ?

C11 C8 C16 111.0(4) . . ?

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C11 C8 S1 86.3(2) . . ?

C16 C8 S1 118.1(3) . . ?

C6 C9 N5 125.0(4) . . ?

O3 C10 N4 128.2(4) . . ?

O3 C10 N5 126.9(4) . . ?

N4 C10 N5 104.9(4) . . ?

N14 C11 N7 115.9(4) . . ?

N14 C11 C8 115.3(3) . . ?

N7 C11 C8 97.7(3) . . ?

C29 C12 C22 119.0(6) . . ?

C17 N14 N5 111.0(3) . . ?

C17 N14 C11 128.6(4) . . ?

N5 N14 C11 119.7(4) . . ?

C19 C15 C20 121.5(4) . . ?

C19 C15 N4 118.8(4) . . ?

C20 C15 N4 119.7(4) . . ?

C36 C16 C33 106.7(5) . . ?

C36 C16 C40 107.2(5) . . ?

C33 C16 C40 108.3(5) . . ?

C36 C16 C8 113.8(4) . . ?

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C40 C16 C8 106.9(4) . . ?

O13 C17 N14 127.6(4) . . ?

O13 C17 N4 128.5(5) . . ?

N14 C17 N4 103.9(4) . . ?

C35 C18 C6 117.8(4) . . ?

C35 C18 C27 107.3(4) . . ?

C6 C18 C27 109.5(4) . . ?

C35 C18 C30 105.4(5) . . ?

C6 C18 C30 109.2(4) . . ?

C27 C18 C30 107.1(4) . . ?

C26 C19 C15 119.0(5) . . ?

C28 C20 C15 117.9(5) . . ?

C28 C21 C26 119.9(5) . . ?

C25 C22 C12 120.0(5) . . ?

C25 C22 S2 119.6(5) . . ?

C12 C22 S2 120.4(5) . . ?

C22 C25 C34 119.7(6) . . ?

C19 C26 C21 120.7(5) . . ?

C21 C28 C20 120.8(5) . . ?

C31 C29 C12 120.9(6) . . ?

C34 C31 C29 119.3(6) . . ?

C34 C31 C41 121.3(8) . . ?

C29 C31 C41 119.4(8) . . ?

C31 C34 C25 121.1(6) . . ?

C6 C9 H9 115.7 . . ?

N5 C9 H9 119.3 . . ?

N14 C11 H11 105.3 . . ?

N7 C11 H11 113.6 . . ?

C8 C11 H11 109.1 . . ?

C29 C12 H12 122.7 . . ?

C22 C12 H12 118.3 . . ?

C26 C19 H19 121.3 . . ?

C15 C19 H19 119.7 . . ?

C28 C20 H20 122.7 . . ?

C15 C20 H20 119.4 . . ?

C28 C21 H21 120.5 . . ?

C26 C21 H21 119.5 . . ?

C22 C25 H25 118.9 . . ?

C34 C25 H25 121.3 . . ?

C19 C26 H26 120.4 . . ?

C21 C26 H26 118.8 . . ?

C18 C27 H27A 110.1 . . ?

C18 C27 H27B 109.2 . . ?

H27A C27 H27B 109.5 . . ?

C18 C27 H27C 109.0 . . ?

H27A C27 H27C 109.5 . . ?

H27B C27 H27C 109.5 . . ?

C21 C28 H28 118.8 . . ?

C20 C28 H28 120.4 . . ?

C31 C29 H29 119.8 . . ?

C12 C29 H29 119.3 . . ?

C18 C30 H30A 109.9 . . ?

C18 C30 H30B 109.3 . . ?

H30A C30 H30B 109.5 . . ?

C18 C30 H30C 109.3 . . ?

H30A C30 H30C 109.5 . . ?

H30B C30 H30C 109.5 . . ?

C16 C33 H33A 110.5 . . ?

C16 C33 H33B 108.6 . . ?

H33A C33 H33B 109.5 . . ?

C16 C33 H33C 109.3 . . ?

H33A C33 H33C 109.5 . . ?

H33B C33 H33C 109.5 . . ?

C31 C34 H34 118.9 . . ?

C25 C34 H34 120.1 . . ?

C18 C35 H35A 110.4 . . ?

C18 C35 H35B 108.9 . . ?

H35A C35 H35B 109.5 . . ?

C18 C35 H35C 109.2 . . ?

H35A C35 H35C 109.5 . . ?

H35B C35 H35C 109.5 . . ?

C16 C36 H36A 110.3 . . ?

C16 C36 H36B 109.4 . . ?

H36A C36 H36B 109.5 . . ?

C16 C36 H36C 108.7 . . ?

H36A C36 H36C 109.5 . . ?

H36B C36 H36C 109.5 . . ?

C16 C40 H40A 109.5 . . ?

C16 C40 H40B 109.5 . . ?

H40A C40 H40B 109.5 . . ?

C16 C40 H40C 109.4 . . ?

H40A C40 H40C 109.5 . . ?

H40B C40 H40C 109.5 . . ?

C31 C41 H41A 108.8 . . ?

C31 C41 H41B 109.7 . . ?

H41A C41 H41B 109.5 . . ?

C31 C41 H41C 109.9 . . ?

H41A C41 H41C 109.5 . . ?

H41B C41 H41C 109.5 . . ?

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_cell_measurement_theta_max         ?
_diffrn_radiation_source    'fine-focus sealed tube'
_diffrn_reflns_limit_h_min         -9
_diffrn_reflns_limit_h_max          9
_diffrn_reflns_limit_k_min        -12
_diffrn_reflns_limit_k_max         13
_diffrn_reflns_limit_l_min          0
_diffrn_reflns_limit_l_max         17


loop_
 _diffrn_radiation_wavelength         0.71073
_diffrn_radiation_wavelength_id all


_cell_formula_units_Z         2
_exptl_crystal_density_diffrn       1.295
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_chemical_formula_weight        428.555
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  _symmetry_equiv_pos_as_xyz
'+X,+Y,+Z'
'-X,-Y,-Z'

_symmetry_space_group_name_H-M 'P -1                    '
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_chemical_formula_moiety 'C22 H28 N4 O3 S '
_chemical_formula_sum        'C22 H28 N4 O3 S '
_chemical_name_systematic
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  ?
;
_cell_length_a                    7.8070(7)
_cell_length_b                   10.9520(6)
_cell_length_c                   14.1090(11)
_cell_angle_alpha                   66.974(5)
_cell_angle_beta                    82.138(4)
_cell_angle_gamma                    85.308(6)
_cell_volume                     1099.27(14)
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_diffrn_reflns_number 4037

_diffrn_reflns_theta_max 27.00

_diffrn_reflns_theta_min 0.00

_diffrn_reflns_theta_full 27.00

_diffrn_measurement_method 'IP'

_computing_cell_refinement 'HKL Scalepack'

Absorption correction

_exptl_absorpt_correction_type 'multi-scan'

_exptl_absorpt_correction_T_min 0.855

_exptl_absorpt_correction_T_max 0.985

_exptl_absorpt_process_details

;

multi-scan from symmetry-related measurements

Sortav (Blessing 1995)

;

_diffrn_reflns_av_R_equivalents 0.052

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_atom_type_scat_dispersion_imag

_atom_type_scat_source

'C' 'C' 0.0033 0.0016

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'H' 'H' 0.0000 0.0000

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'N' 'N' 0.0061 0.0033

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'O' 'O' 0.0106 0.0060

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'S' 'S' 0.1246 0.1234

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_exptl_absorpt_coefficient_mu 0.178

_reflns_number_total 4037

_reflns_number_gt 2247

_reflns_threshold_expression >2sigma(I)

_computing_structure_refinement 'SHELXL-97 (Sheldrick, 1997)'

_refine_special_details

;

Refinement of F^2^ against ALL reflections. The weighted R-factor wR and goodness 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 threshold expression of F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) 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

_refine_ls_weighting_details

'calc w=1/[¥s^2^(Fo^2^)+(0.0080P)^2^+0.8000P] where P=(Fo^2^+2Fc^2^)/3'

_refine_ls_extinction_method SHELXL

_refine_ls_extinction_coef 0.0225(18)

_refine_ls_extinction_expression

'Fc^*^=kFc[1+0.001xFc^2^¥l^3^/sin(2¥q)]^-1/4^'

_refine_ls_number_reflns 4037

_refine_ls_number_parameters 272

_refine_ls_number_restraints 0

_refine_ls_R_factor_all 0.1129

_refine_ls_R_factor_gt 0.0593

_refine_ls_wR_factor_ref 0.1447

_refine_ls_wR_factor_gt 0.1209

_refine_ls_goodness_of_fit_ref 1.012

_refine_ls_restrained_S_all 1.012

_refine_ls_shift/su_max 0.000

_refine_ls_shift/su_mean 0.000

loop_

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_atom_site_refinement_flags

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_atom_site_disorder_group

S1 S 0.41546(11) 0.04483(9) 0.71919(8) 0.0452(3) Uani 1 1 d . . .

O2 O 0.1688(3) 0.1502(2) 0.85519(19) 0.0454(6) Uani 1 1 d . . .

O3 O -0.3819(3) 0.2925(2) 0.7865(2) 0.0587(8) Uani 1 1 d . . .

C4 C 0.2043(4) 0.2318(3) 0.7461(3) 0.0387(8) Uani 1 1 d . . .

N5 N -0.1558(3) 0.4163(3) 0.7922(2) 0.0444(8) Uani 1 1 d . . .

C6 C 0.0532(4) 0.0759(3) 0.7009(3) 0.0346(8) Uani 1 1 d . . .

C7 C 0.2204(4) 0.1510(3) 0.6780(3) 0.0380(8) Uani 1 1 d . . .

N8 N 0.0591(3) 0.3228(3) 0.7238(2) 0.0415(7) Uani 1 1 d . . .

O9 O 0.1238(3) 0.4818(3) 0.7811(2) 0.0644(8) Uani 1 1 d . . .

N10 N -0.0972(3) 0.2619(3) 0.7292(2) 0.0428(8) Uani 1 1 d . . .

N11 N 0.3990(4) 0.0019(3) 0.8481(2) 0.0487(8) Uani 1 1 d . . .

C12 C -0.0913(4) 0.1389(3) 0.7226(3) 0.0396(8) Uani 1 1 d . . .

C13 C -0.2531(5) 0.5027(3) 0.8374(3) 0.0458(9) Uani 1 1 d . . .

C14 C 0.0201(5) 0.4155(3) 0.7691(3) 0.0438(9) Uani 1 1 d . . .

C15 C 0.2885(4) 0.0500(4) 0.8970(3) 0.0468(9) Uani 1 1 d . . .

C16 C -0.2308(5) 0.3188(3) 0.7715(3) 0.0426(9) Uani 1 1 d . . .

C17 C 0.0348(4) -0.0692(3) 0.7128(3) 0.0408(8) Uani 1 1 d . . .

C18 C 0.0779(5) -0.1639(4) 0.8221(3) 0.0527(10) Uani 1 1 d . . .

C19 C 0.2721(5) 0.2431(4) 0.5587(3) 0.0474(9) Uani 1 1 d . . .

C20 C 0.1528(5) -0.1109(4) 0.6332(3) 0.0553(11) Uani 1 1 d . . .

C21 C -0.1979(6) 0.5163(4) 0.9210(3) 0.0619(11) Uani 1 1 d . . .

C22 C 0.3844(6) 0.3572(4) 0.5488(3) 0.0745(14) Uani 1 1 d . . .

C23 C 0.1137(6) 0.3015(4) 0.5032(3) 0.0690(13) Uani 1 1 d . . .

C24 C -0.1487(5) -0.0974(4) 0.7050(3) 0.0552(11) Uani 1 1 d . . .

C25 C 0.3797(6) 0.1643(4) 0.4981(3) 0.0680(12) Uani 1 1 d . . .

C26 C -0.3972(5) 0.5683(4) 0.7965(3) 0.0624(12) Uani 1 1 d . . .

C27 C 0.2652(6) 0.0016(5) 1.0119(3) 0.0670(12) Uani 1 1 d . . .

C29 C -0.4354(7) 0.6668(4) 0.9218(5) 0.0832(17) Uani 1 1 d . . .

C30 C -0.4897(6) 0.6512(4) 0.8388(4) 0.0806(16) Uani 1 1 d . . .

C31 C -0.2890(6) 0.5996(5) 0.9632(4) 0.0740(14) Uani 1 1 d . . .

H4 H 0.3088 0.2788 0.7339 0.046 Uiso 1 1 d R . .

H12 H -0.2007 0.0984 0.7338 0.048 Uiso 1 1 d R . .

H18A H 0.0668 -0.2543 0.8303 0.063 Uiso 1 1 d R . .

H18B H -0.0019 -0.1449 0.8731 0.063 Uiso 1 1 d R . .

H18C H 0.1939 -0.1510 0.8311 0.063 Uiso 1 1 d R . .

H20A H 0.1351 -0.2019 0.6459 0.066 Uiso 1 1 d R . .

H20B H 0.2719 -0.1001 0.6378 0.066 Uiso 1 1 d R . .

H20C H 0.1241 -0.0552 0.5652 0.066 Uiso 1 1 d R . .

H21 H -0.0966 0.4668 0.9487 0.074 Uiso 1 1 d R . .

H22A H 0.4118 0.4115 0.4767 0.089 Uiso 1 1 d R . .

H22B H 0.4894 0.3219 0.5794 0.089 Uiso 1 1 d R . .

H22C H 0.3221 0.4099 0.5836 0.089 Uiso 1 1 d R . .

H23A H 0.1484 0.3573 0.4323 0.083 Uiso 1 1 d R . .

H23B H 0.0449 0.3529 0.5368 0.083 Uiso 1 1 d R . .

H23C H 0.0470 0.2304 0.5054 0.083 Uiso 1 1 d R . .

H24A H -0.1572 -0.1880 0.7132 0.066 Uiso 1 1 d R . .

H24B H -0.1785 -0.0395 0.6377 0.066 Uiso 1 1 d R . .

H24C H -0.2267 -0.0802 0.7574 0.066 Uiso 1 1 d R . .

H25A H 0.4079 0.2228 0.4271 0.082 Uiso 1 1 d R . .

H25B H 0.3124 0.0932 0.5008 0.082 Uiso 1 1 d R . .

H25C H 0.4843 0.1285 0.5289 0.082 Uiso 1 1 d R . .

H26 H -0.4325 0.5571 0.7379 0.075 Uiso 1 1 d R . .

H27A H 0.3486 -0.0684 1.0403 0.080 Uiso 1 1 d R . .

H27B H 0.1506 -0.0309 1.0380 0.080 Uiso 1 1 d R . .

H27C H 0.2815 0.0740 1.0316 0.080 Uiso 1 1 d R . .

H29 H -0.4986 0.7258 0.9505 0.100 Uiso 1 1 d R . .

H30 H -0.5916 0.6987 0.8102 0.097 Uiso 1 1 d R . .

H31 H -0.2506 0.6071 1.0225 0.089 Uiso 1 1 d R . .

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S1 0.0285(4) 0.0543(6) 0.0615(7) -0.0318(5) -0.0093(4) 0.0076(4)

O2 0.0352(13) 0.0566(15) 0.0495(16) -0.0258(14) -0.0114(12) 0.0089(11)

O3 0.0325(15) 0.0592(16) 0.096(2) -0.0428(17) -0.0132(14) 0.0076(12)

C4 0.0303(17) 0.0410(18) 0.052(2) -0.0269(19) -0.0056(16) 0.0058(14)

N5 0.0345(16) 0.0430(16) 0.060(2) -0.0245(16) -0.0083(15) 0.0075(13)

C6 0.0295(17) 0.0354(17) 0.044(2) -0.0196(17) -0.0108(15) 0.0024(13)

C7 0.0293(17) 0.0405(18) 0.047(2) -0.0188(18) -0.0071(16) -0.0002(14)

N8 0.0342(16) 0.0377(15) 0.059(2) -0.0254(16) -0.0087(14) 0.0009(12)

O9 0.0532(17) 0.0590(16) 0.103(2) -0.0549(18) -0.0071(16) -0.0055(13)

N10 0.0261(15) 0.0418(15) 0.068(2) -0.0286(17) -0.0138(14) 0.0071(12)

N11 0.0364(17) 0.0532(18) 0.059(2) -0.0231(18) -0.0168(15) 0.0102(14)

C12 0.0357(18) 0.0383(18) 0.055(2) -0.0266(19) -0.0124(17) -0.0002(14)

C13 0.043(2) 0.0316(17) 0.062(3) -0.021(2) -0.0022(19) 0.0067(15)

C14 0.044(2) 0.0330(18) 0.060(2) -0.023(2) -0.0116(18) 0.0051(16)

C15 0.035(2) 0.055(2) 0.056(3) -0.024(2) -0.0176(18) 0.0041(17)

C16 0.039(2) 0.0363(18) 0.057(2) -0.0214(19) -0.0156(18) 0.0074(15)

C17 0.0337(18) 0.0413(18) 0.052(2) -0.0227(19) -0.0057(17) -0.0006(15)

C18 0.052(2) 0.0416(19) 0.064(3) -0.019(2) -0.010(2) 0.0047(17)

C19 0.048(2) 0.046(2) 0.045(2) -0.0133(19) -0.0015(18) -0.0098(17)

C20 0.049(2) 0.058(2) 0.071(3) -0.038(2) -0.006(2) -0.0016(19)

C21 0.060(3) 0.064(3) 0.071(3) -0.035(3) -0.006(2) -0.008(2)

C22 0.094(4) 0.062(3) 0.063(3) -0.017(2) 0.005(3) -0.037(3)

C23 0.081(3) 0.068(3) 0.053(3) -0.015(2) -0.022(2) 0.008(2)

C24 0.043(2) 0.049(2) 0.085(3) -0.035(2) -0.015(2) -0.0011(17)

C25 0.070(3) 0.077(3) 0.055(3) -0.030(3) 0.009(2) -0.002(2)

C26 0.061(3) 0.055(2) 0.058(3) -0.013(2) -0.003(2) 0.018(2)

C27 0.057(3) 0.084(3) 0.059(3) -0.024(3) -0.019(2) 0.007(2)

C29 0.090(4) 0.048(2) 0.107(5) -0.040(3) 0.041(3) -0.011(3)

C30 0.070(3) 0.059(3) 0.095(4) -0.024(3) 0.010(3) 0.030(2)

C31 0.073(3) 0.074(3) 0.093(4) -0.056(3) 0.009(3) -0.009(3)

_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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S1 N11 1.680(3) . ?
S1 C7 1.852(3) . ?
O2 C15 1.379(4) . ?
O2 C4 1.446(4) . ?
O3 C16 1.208(4) . ?
C4 N8 1.430(4) . ?
C4 C7 1.528(5) . ?
N5 C14 1.367(4) . ?
N5 C16 1.402(4) . ?
N5 C13 1.450(4) . ?
C6 C12 1.337(4) . ?
C6 C7 1.529(4) . ?
C6 C17 1.548(4) . ?
C7 C19 1.602(5) . ?

N8 C14 1.392(4) . ?

N8 N10 1.416(3) . ?

O9 C14 1.201(4) . ?

N10 C16 1.358(4) . ?

N10 C12 1.383(4) . ?

N11 C15 1.244(5) . ?

C13 C26 1.355(5) . ?

C13 C21 1.373(5) . ?

C15 C27 1.484(5) . ?

C17 C24 1.518(4) . ?

C17 C20 1.538(5) . ?

C17 C18 1.550(5) . ?

C19 C23 1.516(5) . ?

C19 C22 1.536(5) . ?

C19 C25 1.564(6) . ?

C21 C31 1.378(6) . ?

C26 C30 1.379(6) . ?

C29 C30 1.373(7) . ?

C29 C31 1.378(7) . ?

C4 H4 0.9600 . ?

C12 H12 0.9600 . ?

C18 H18A 0.9601 . ?

C18 H18B 0.9599 . ?

C18 H18C 0.9600 . ?

C20 H20A 0.9600 . ?

C20 H20B 0.9600 . ?

C20 H20C 0.9601 . ?

C21 H21 0.9600 . ?

C22 H22A 0.9600 . ?

C22 H22B 0.9602 . ?

C22 H22C 0.9600 . ?

C23 H23A 0.9601 . ?

C23 H23B 0.9598 . ?

C23 H23C 0.9601 . ?

C24 H24A 0.9600 . ?

C24 H24B 0.9600 . ?

C24 H24C 0.9599 . ?

C25 H25A 0.9600 . ?

C25 H25B 0.9599 . ?

C25 H25C 0.9601 . ?

C26 H26 0.9601 . ?

C27 H27A 0.9600 . ?

C27 H27B 0.9600 . ?

C27 H27C 0.9600 . ?

C29 H29 0.9598 . ?

C30 H30 0.9600 . ?

C31 H31 0.9599 . ?

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N11 S1 C7 104.81(16) . . ?

C15 O2 C4 116.6(3) . . ?

N8 C4 O2 104.5(3) . . ?

N8 C4 C7 110.1(3) . . ?

O2 C4 C7 112.5(3) . . ?

C14 N5 C16 111.8(3) . . ?

C14 N5 C13 124.5(3) . . ?

C16 N5 C13 123.7(3) . . ?

C12 C6 C7 115.8(3) . . ?

C12 C6 C17 116.5(3) . . ?

C7 C6 C17 127.4(3) . . ?

C4 C7 C6 107.8(3) . . ?

C4 C7 C19 111.0(3) . . ?

C6 C7 C19 115.3(3) . . ?

C4 C7 S1 102.0(2) . . ?

C6 C7 S1 114.9(2) . . ?

C19 C7 S1 105.1(2) . . ?

C14 N8 N10 107.3(3) . . ?

C14 N8 C4 122.6(3) . . ?

N10 N8 C4 113.6(2) . . ?

C16 N10 C12 127.6(3) . . ?

C16 N10 N8 109.8(3) . . ?

C12 N10 N8 119.5(3) . . ?

C15 N11 S1 124.0(3) . . ?

C6 C12 N10 124.8(3) . . ?

C26 C13 C21 121.3(4) . . ?

C26 C13 N5 119.3(4) . . ?

C21 C13 N5 119.4(3) . . ?

O9 C14 N5 129.0(3) . . ?

O9 C14 N8 125.1(3) . . ?

N5 C14 N8 105.8(3) . . ?

N11 C15 O2 126.3(4) . . ?

N11 C15 C27 123.1(4) . . ?

O2 C15 C27 110.6(3) . . ?

O3 C16 N10 127.6(3) . . ?

O3 C16 N5 127.4(3) . . ?

N10 C16 N5 105.0(3) . . ?

C24 C17 C20 106.5(3) . . ?

C24 C17 C6 112.1(3) . . ?

C20 C17 C6 114.9(3) . . ?

C24 C17 C18 106.5(3) . . ?

C20 C17 C18 107.5(3) . . ?

C6 C17 C18 108.9(3) . . ?

C23 C19 C22 108.7(3) . . ?

C23 C19 C25 106.8(3) . . ?

C22 C19 C25 106.6(3) . . ?

C23 C19 C7 111.7(3) . . ?

C22 C19 C7 110.9(3) . . ?

C25 C19 C7 112.0(3) . . ?

C13 C21 C31 119.5(4) . . ?

C13 C26 C30 119.5(5) . . ?

C30 C29 C31 120.2(5) . . ?

C29 C30 C26 119.9(5) . . ?

C29 C31 C21 119.5(5) . . ?

N8 C4 H4 110.4 . . ?

O2 C4 H4 110.0 . . ?

C7 C4 H4 109.3 . . ?

C6 C12 H12 119.3 . . ?

N10 C12 H12 115.9 . . ?

C17 C18 H18A 109.6 . . ?

C17 C18 H18B 108.7 . . ?

H18A C18 H18B 109.5 . . ?

C17 C18 H18C 110.1 . . ?

H18A C18 H18C 109.5 . . ?

H18B C18 H18C 109.5 . . ?

C17 C20 H20A 110.1 . . ?

C17 C20 H20B 110.0 . . ?

H20A C20 H20B 109.5 . . ?

C17 C20 H20C 108.3 . . ?

H20A C20 H20C 109.5 . . ?

H20B C20 H20C 109.5 . . ?

C13 C21 H21 118.8 . . ?

C31 C21 H21 121.7 . . ?

C19 C22 H22A 109.1 . . ?

C19 C22 H22B 109.8 . . ?

H22A C22 H22B 109.5 . . ?

C19 C22 H22C 109.6 . . ?

H22A C22 H22C 109.5 . . ?

H22B C22 H22C 109.5 . . ?

C19 C23 H23A 109.9 . . ?

C19 C23 H23B 109.7 . . ?

H23A C23 H23B 109.5 . . ?

C19 C23 H23C 108.8 . . ?

H23A C23 H23C 109.5 . . ?

H23B C23 H23C 109.5 . . ?

C17 C24 H24A 110.6 . . ?

C17 C24 H24B 108.0 . . ?

H24A C24 H24B 109.5 . . ?

C17 C24 H24C 109.8 . . ?

H24A C24 H24C 109.5 . . ?

H24B C24 H24C 109.5 . . ?

C19 C25 H25A 109.4 . . ?

C19 C25 H25B 109.2 . . ?

H25A C25 H25B 109.5 . . ?

C19 C25 H25C 109.7 . . ?

H25A C25 H25C 109.5 . . ?

H25B C25 H25C 109.5 . . ?

C13 C26 H26 119.5 . . ?

C30 C26 H26 121.0 . . ?

C15 C27 H27A 109.9 . . ?

C15 C27 H27B 109.8 . . ?

H27A C27 H27B 109.5 . . ?

C15 C27 H27C 108.7 . . ?

H27A C27 H27C 109.5 . . ?

H27B C27 H27C 109.5 . . ?

C30 C29 H29 120.0 . . ?

C31 C29 H29 119.8 . . ?

C29 C30 H30 119.7 . . ?

C26 C30 H30 120.4 . . ?

C29 C31 H31 121.7 . . ?

C21 C31 H31 118.8 . . ?

_diffrn_measured_fraction_theta_max 0.845

_diffrn_measured_fraction_theta_full 0.845

_refine_diff_density_max 0.240

_refine_diff_density_min -0.304

_refine_diff_density_rms 0.053

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