Supplementary material for publication on line

Synthesis, in vitro screening and docking studies of new thiosemicarbazide derivatives as antitubercular agents

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Experimental data for 1-(pyridine-2,3,4-yl)carbonyl-4-substituted thiosemicarbazide (1-19)

4-(2-Fluorophenyl)-1-(pyridin-2 yl)carbonylthiosemicarbazide (1)
Yield 87% (white powder); m.p. 182–184°C. ¹H NMR (DMSO-d₆) δ (ppm): 7.10–7.70 (m, 4H, CHphenyl), 7.97–8.72 (m, 3H, CPyridine), 9.48 (s, 1H, NH), 9.81 (s, 1H, NH), 10.78 (s, 1H, NH) [S1].

4-(2-Chlorophenyl)-1-(pyridin-2 yl)carbonylthiosemicarbazide (2)
Yield 83% (yellow powder); m.p. 172–174°C. ¹H NMR (DMSO-d₆) δ (ppm): 7.25–7.65 (m, 4H, CHphenyl), 8.03–8.71 (m, 4H, CPyridine), 9.58 (s, 1H, NH), 9.90 (s, 1H, NH), 10.84 (s, 1H, NH) [S1].

4-(4-Methylthiophenyl)-1-(pyridin-2-yl)carbonylthiosemicarbazide (3)
Yield 88% (yellow powder); m.p. 184–186°C. ¹H NMR (DMSO-d₆) δ (ppm): 2.28 (s, 3H, CH₃), 7.01–7.25 (m, 4H, CHphenyl), 7.51–8.25 (m, 4H, CPyridine), 8.66 (1s, 1H, NH), 10.53 (s, 1H, NH), 12.01 (s, 1H, NH) [S1].

4-(2,4-Dichlorophenyl)-1-(pyridin-2-yl)carbonylthiosemicarbazide (4)
Yield 91% (white needles); m.p. 158–160°C. ¹H NMR (DMSO-d₆) δ (ppm): 7.02–7.53 (m, 3H, CHphenyl), 7.93–8.49 (m,4H, CPyridine), 9.12 (s, 1H, NH), 10.56 (s, 1H, NH), 11.89 (s, 1H, NH) [S1].

4-(3,4-Dichlorophenyl)-1-(pyridin-2-yl)carbonylthiosemicarbazide (5)
Yield 78% (white powder); m.p. 160–161°C. ¹H NMR (DMSO-d₆) δ (ppm): 7.56–7.84 (m, 3H, CHphenyl), 8.06–8.70 (m,4H, CPyridine), 9.85 (s, 1H, NH), 10.00 (s, 1H, NH), 10.81 (s, 1H, NH). ¹³C NMR (DMSO-d₆) δ (ppm): 123, 125, 127, 129, 130, 138, 139, 148, 149, 164, 181. IR γmax (cm⁻¹): 3318, 3205, 3143, 1685, 1654, 1400 [S2].

4-(2-Chlorophenyl)-1-(pyridin-3-yl)carbonylthiosemicarbazide (6)
Yield 83% (yellow powder); m.p. 185–186°C. ¹H NMR (DMSO-d₆) δ (ppm): 7.28–7.57 (m, 4H, CHphenyl), 8.29–9.12 (m,4H, CPyridine), 9.76 (s, 1H, NH), 9.95 (s, 1H, NH), 10.85 (s, 1H, NH). ¹³C NMR (DMSO-d₆) δ (ppm): 123, 127, 128, 129, 131, 132, 136, 137, 149, 152, 165, 182. IR γmax (cm⁻¹): 3250, 3064, 2969, 1739, 1682, 1592 [S3].
4-(4-Methylthiophenyl)-1-(pyridin-3-yl)carbonylthiosemicarbazide (7)
Yield 89 % (transparent needles); m.p. 176–177 °C. 1H NMR (DMSO-d6) δ (ppm): 2.47 (s, 3H, CH3), 7.23–7.57 (m, 4H, CHphenyl), 8.27–8.76 (m, 4H, CHpyridine), 9.11 (s, 1H, NH), 9.81 (s, 1H, NH), 10.76 (s, 1H, NH) [S1].

4-(4-Nitrophenyl)-1-(pyridin-3-yl)carbonylthiosemicarbazide (8)
Yield: 72% (orange powder); m.p. 220–222°C. 1H NMR (DMSO-d6) δ ppm: 7.55-7.91 (m, 4H), 8.21-9.11 (m, 4H), 10.16 (s, 1H), 10.24 (m, 1H), 10.89 (s, 1H) [S4].

4-[4-(2-Morpholinoethyl)]-1-(pyridin-3-yl)carbonylthiosemicarbazide (9)
Yield: 71% (white powder); m.p. 128–129°C. 1H NMR (DMSO-d6) δ: 2.10-2.48 (m, 4H, 2xCH2morpholina); 3.25-3.50 (4H, 2xCH3morpholina); 3.56-4.17 (4H, 4H, NH-CH2-CH2-); 7.48-8.97 (m, 4H, CHpyridina); 9.55 (s, 1H, NH); 10.00 (s, 1H, NH); 10.55 (s, 1H, NH). 13C NMR (DMSO-d6) δ: 53, 55, 57, 66, 123, 124, 128, 129, 135, 136, 148, 149, 150, 152, 164, 165, 167, 181. IR νmax (cm⁻¹): 3153, 2962, 1662, 1589, 1407. LC/MS (m/z): Calcd. for C13H12N4O5S, Monoisotopic Mass 309.1260 Da, [M+H]+ 310.1332 Da, Measured Mass 309.1264.

4-[Methoxy carbonylmethyl]-1-(pyridin-3-yl)carbonylthiosemicarbazide (10)
Yield: 74% (white powder); 178-179°C. 1H NMR (DMSO-d6) δ ppm: 3.63 (s, 3H, CH3), 4.20 (d, 2H, CH2), 5.61 (m, 1H, CH2morpholine); 7.82–7.84 (m, 2H, CHarom); 8.54 (s, 1H, NH); 8.76-8.78 (m, 2H, CHarom); 9.76 (s, 1H, NH); 10.83 (s, 1H, NH). 13C NMR (DMSO-d6) δ: 45, 52, 122, 139, 150, 164, 170, 183. IR νmax (cm⁻¹): 3101, 2929, 1662, 1586, 1421. LC/MS (m/z): Calcd. for C13H12N4O5S, Monoisotopic Mass 268.0630 Da, [M+H]+ 269.070287 Da, Measured Mass 268.0633.

4-(2,4-Dichlorophenyl)-1-(pyridin-3-yl)carbonyl thiosemicarbazide (11)
Yield 92 % (transparent needles); m.p. 196–197°C. 1H NMR (DMSO-d6) δ ppm: 7.37–7.68 (m, 3H, CHphenyl), 8.14–9.11 (m, 4H, CHpyridine), 9.95 (s, 1H, NH), 10.08 (s, 1H, NH), 10.83 (s, 1H, NH). 13C NMR (DMSO-d6) δ ppm: 123, 126, 127, 128, 129, 130, 135, 136, 139, 148, 149, 152, 165, 181 [S1].

4-(3,4-Dichlorophenyl)-1-(pyridin-3-yl)carbonylthiosemicarbazide (12)
Yield 75% ((yellow needles); m.p. 146-147°C. 1H NMR (DMSO-d6) δ ppm: 7.53–7.81 (m, 3H, CHphenyl), 8.14–9.11 (m, 4H, CHpyridine), 9.94 (s, 1H, NH), 10.08 (s, 1H, NH), 10.86 (s, 1H, NH). 13C NMR (DMSO-d6) δ: 123, 127, 128, 129, 132, 133, 136, 149, 152, 165, 182. IR νmax (cm⁻¹): 3356, 3077, 1654, 1590, 1420 [S5].

4-(2-Fluorophenyl)-1-(pyridin-4-yl)carbonylthiosemicarbazide (13)
Yield 78% (yellow powder); m.p. 202–204°C. 1H NMR (DMSO-d6) δ ppm: 7.18–7.31 (m, 4H, CHphenyl), 7.86–8.78 (m, 4H, CHpyridine), 9.70 (s, 1H, NH), 9.99 (s, 1H, NH), 10.94 (s, 1H, NH) [S1].

4-(2-Chlorophenyl)-1-(pyridin-4-yl)carbonylthiosemicarbazide (14)
Yield 74 % (yellow powder); m.p. 224–226°C. 1H NMR (DMSO-d6) δ ppm: 7.28–7.87 (m, 4H, CHphenyl), 8.77–8.78 (m, 4H, CHpyridine), 9.75 (s, 1H, NH), 9.96 (s, 1H, NH), 10.93 (s, 1H, NH). 13C NMR (DMSO-d6) δ: 122, 127, 128, 129, 131, 132, 137, 140, 150, 165, 182. IR νmax (cm⁻¹): 3257, 3109, 1739, 1677, 1406 [S6].

4-(2-Nitrophenyl)-1-(pyridin-4-yl)carbonylthiosemicarbazide (15)
Yield: 62% (orange powder); m.p. 134–136°C. 1H NMR (DMSO-d6) δ ppm: 7.44-8.04 (m, 4H); 8.19-8.39 (m, 4H); 10.18 (s, 1H); 10.20 (s, 1H); 11.07 (s, 1H) [S4].
4-(4-Methylthiophenyl)-1-(pyridin-4-yl)carbonylthiosemicarbazide (16)
Yield 86% (yellow powder); m.p. 197–198°C. 1H NMR (DMSO-d6) δ ppm: 2.47 (s, 3H, CH3), 7.23–7.86 (m, 4H, CHphenyl), 8.77–8.78 (m, 4H, CHpyridine), 9.83 (s, 2H, NH), 10.86 (s, 1H, NH) [S1].

4-[4-(2-Morpholinoethyl)-1-(pyridin-4-yl)carbonylthiosemicarbazide (17)
Yield: 70% (yellow powder); m.p. 176–178°C. 1H NMR (DMSO-d6) δ: 2.38–2.44 (m, 4H, 2xCH2morpholina); 3.35–3.36 (m, 4H, 2xCH2morpholina); 3.51–3.57 (m, 4H, NH-CH2-CH2-); 7.81–8.77 (m, 4H, CHpyridine); 8.01 (s, 1H, NH); 9.46 (s, 1H, NH); 10.71 (s, 1H, NH). 13C NMR (DMSO-d6) δ: 41, 53, 57, 66, 122, 139, 150, 164. IR νmax (cm⁻¹): 3306, 3147, 1735, 1677, 1408. LC/MS (m/z): Calcd. for C33H28N3O5S, Monoisotopic Mass 639.1260 Da, [M+H]^+ 630.1322 Da, Measured Mass 639.1262.

4-(2,4-Dichlorophenyl)-1-(pyridin-4-yl)carbonylthiosemicarbazide (18)
Yield 84% (white powder); m.p. 164–166°C. 1H NMR (DMSO-d6) δ ppm: 7.37–7.45 (m, 3H, CHphenyl), 7.68–8.78 (m, 4H, CHpyridine), 9.76 (s, 1H, NH), 10.05 (s, 1H, NH), 10.95 (s, 1H, NH) [S1].

4-(4,4-Dichlorophenyl)-1-(pyridin-4-yl)carbonylthiosemicarbazide (19)
Yield 85% (yellow powder); m.p. 182–183°C. 1H NMR (DMSO-d6) δ ppm: 7.42–7.86 (m, 3H, CHphenyl), 8.69–8.78 (m, 4H, CHpyridine), 9.76 (s, 1H, NH), 10.04 (s, 1H, NH), 10.95 (s, 1H, NH). 13C NMR (DMSO-d6) δ: 122, 129, 131, 132, 133, 148, 150, 169. IR νmax (cm⁻¹): 3202, 3027, 1735, 1592, 1414 [S7].

Experimental data for 1-(pyridin-4-ylacetyl)-4-substituted thiosemicabazide (20-25)

4-Phenyl-1-(pyridin-4-ylacetyl)thiosemicarbazide (20)
Yield 78% (yellow powder); m.p. 116–118°C. 1H NMR (DMSO-d6) δ ppm: 3.58 (s, 2H, CH2), 7.19–7.45 (m, 5H, CHphenyl), 8.46–8.51 (m, 4H, CHpyridine), 9.73, 10.01, 10.25 (3s, 3H, 3NH) [S8].

4-(2-Fluorophenyl)-1-(pyridin-4-ylacetyl)thiosemicarbazide (21)
Yield 68% (yellow powder); m.p. 148–149°C. 1H NMR (DMSO-d6) δ ppm: 3.58 (s, 2H, CH2), 7.18–7.39 (m, 4H, CHphenyl), 8.50–8.52 (m, 4H, CHpyridine), 9.55, 9.80, 10.33 (3s, 3H, 3NH) [S8].

4-(4-Bromophenyl)-1-(pyridin-4-ylacetyl)thiosemicarbazide (22)
Yield 77% (yellow powder); m.p. 220–222°C. 1H NMR (DMSO-d6) δ ppm: 3.93 (s, 2H, CH2), 7.03–7.74 (m, 4H, CHphenyl), 8.33-8.58 (m, 4H, CHpyridine), 9.75; 10.27; 13.90 (3s, 3H, 3NH). 13C NMR (DMSO-d6) δ ppm: 31; 122; 123; 124; 125; 130; 130; 132; 132; 137; 139; 143; 148; 151; 168. IR νmax (cm⁻¹): 3166, 1688, 1400. LC/MS (m/z): Calcd. for C13H13BrN3O5S, Monoisotopic Mass 363.9993 Da, [M+H]^+ 365.0066 Da, Measured Mass 363.9997.

4-Methoxycarbonylmethyl-1-(pyridin-4-ylacetyl)thiosemicarbazide (23)
Yield 73% (white powder); m.p. 178–180°C. 1H NMR (DMSO-d6) δ ppm: 3.36 (s, 2H, CH2), 3.63 (s, 3H, CH3), 4.20 (s, 2H, CH2), 7.82–8.76 (m, 4H, CHpyridine), 8.54; 9.76; 10.82 (3s, 3H, 3NH). 13C NMR (DMSO-d6) δ ppm: 45, 52, 122, 139, 150, 164, 170, 182. LC/MS (m/z): Calcd. for C13H13N3O3S, Monoisotopic Mass 282.0787 Da, [M+H]^+ 283.0860 Da.
4-(2,4-Dichlorophenyl)-1-(pyridin-4-ylacetyl)thiosemicarbazide (24)
Yield 72% (yellow powder); m.p. 150–151 °C. 1H NMR (DMSO-d6) δ ppm: 3.64 (s, 2H, CH2), 7.33–7.50 (m, 3H, CHphenyl), 7.73–8.56 (m, 4H, CHpyridine), 9.64 (s, 1H, NH), 9.92 (s, 1H, NH), 10.39 (s, 1H, NH). 13C NMR (DMSO-d6) δ: 56, 122, 124, 125, 127, 128, 129, 130, 132, 133, 144, 149, 168, 179, 182. IR νmax (cm⁻¹): 3231, 2929, 1682, 1609, 1421. LC/MS (m/z): Calcd. for C14H12ClN5O5S, Monoisotopic Mass 354.0109 Da, [M+H]+ 355.0182 Da, Measured Mass 354.1012.

4-(3,4-Dichlorophenyl)-1-(pyridin-4-ylacetyl)thiosemicarbazide (25)
Yield 75% (white powder); m.p. 161–163 °C. 1H NMR (DMSO-d6) δ ppm: 3.59 (s, 2H, CH2), 7.35–7.74 (m, 3H, CHphenyl), 7.83–8.51 (m, 4H, CHpyridine), 9.88 (s, 1H, NH), 10.16 (s, 1H, NH), 10.21 (s, 1H, NH). 13C NMR (DMSO-d6) δ: 35, 123, 126, 127, 130, 131, 137, 139, 148, 150, 170, 181. IR νmax (cm⁻¹): 3333, 3074, 1668, 1590, 1427. LC/MS (m/z): Calcd. for C14H12ClN5O5S, Monoisotopic Mass 354.0109 Da, [M+H]+ 355.0182 Da, Measured Mass 354.0113.

X-ray analysis: crystal structures of 4, 7, 11, 13 and 14

In the crystal structure of 4 the inversion related molecules form molecular dimers through the pair bifurcated intermolecular hydrogen bonds N1–H1…O5 and N3–H3…O5. Moreover, the benzene rings belonging to the inversion related molecules partially overlap each other with the π…π distance of 3.4567(7) Å characteristic for the overlapping π-aromatic ring systems.

In the crystal of 7 the molecules related by c glide planes are linked into molecular chains parallel to Z crystallographic axis via the pair of intermolecular hydrogen bonds N1–H1…O5 and N4–H4…S2. Additionally, the molecules related by translation a form molecular chains by N3–H3…N53 hydrogen bond. The combination of these two types of chains gives molecular planes parallel to (010) crystallographic plane.

In the crystal structure of 11 the net of intermolecular hydrogen bonds gives the molecular planes parallel to the (010) crystallographic plane as a combination of two molecular chains. The first one is formed by molecules related by translation a and connected via N1–H1…O5 and N4–H4…O5 bifurcated hydrogen bonds, while the second one is created by molecules related by c glide planes through N3 – H3…N53 hydrogen bond.

In the crystal of 13 the molecules A and B from asymmetric part of the unit cell form the molecular dimer using N1A–H1A…N54B and N1B–H1B…N54A hydrogen bonds. The π…π interaction between pyridine rings within this dimer is observed; the centroid-to-centroid separation and the angle between the overlapping planes of these rings are 3.4559(13) Å and 0.57(11)°, respectively. Moreover, the intermolecular hydrogen bonds N4A–H4A…O5A and N4B–H4B…O5B linking molecules A and B related by 2i axis (independently of each other) into molecular chains along b direction. Similar chains are formed by the A and 2-propanol molecules via O2–H1…S2A hydrogen bond.

In the crystal of 14 the molecules related by centers of inversion along c translation are linked into molecular chains via two pairs of intermolecular hydrogen bonds N1–H1…N54 and N3–H3…S2. The similar molecular chains are created also by molecules related by 2-fold axis in b direction resulting in molecular layers parallel to (100) crystallographic plane. Moreover, the pyridine rings belonging to the inversion related molecules partially overlap each other with centroid-to-centroid separation of 3.4259(16) Å, π…π distance of 3.1808(11) Å and slippage of 1.273 Å.
Theoretical calculations

Fig. 1S. The energy effect upon C2–N3 ($\varphi_1 = N1$–C2–N3–N4) rotation calculated for 4 using AM1 method.

Fig. 2S. The energy effect upon N3–N4 ($\varphi_2 = C2$–N3–N4–C5) rotation calculated for 4 using AM1 method.
Fig. 3S. Overlay of molecules 4, 7, 11, 13 and 14 in the conformations observed in the crystalline state and in the active site of MtGS by least-squares fitting of the atoms of pyridine systems (RMS = 0.0066 Å for 4, 0.0109 Å for 7, 0.0198 Å for 11, 0.0159 Å for 13 and 0.0164 Å for 14).
References

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X-ray structure determination

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**_diffrn_Laue_measured_fraction_max_** 0.959
**_diffrn_Laue_measured_fraction_full_** 0.998
**_reflns_number_total_** 3041
**_reflns_number_gt_** 2964
**_reflns_threshold_expression_** 'I > 2σ(I)'
**_reflns_Friedel_coverage_** 0.000
**_reflns_Friedel_fraction_max_** .
**_reflns_Friedel_fraction_full_** .

Reflections were merged by SHELXL according to the crystal class for the calculation of statistics and refinement.

/reflns_Friedel_fraction is defined as the number of unique Friedel pairs measured divided by the number that would be possible theoretically, ignoring centric projections and systematic absences.

;  
**computing_data_collection_** 'CrysAlisPro 1.171.39.16b (Rigaku OD, 2015)'
**computing_cell_refinement_** 'CrysAlisPro 1.171.39.16b (Rigaku OD, 2015)'
**computing_data_reduction_** 'CrysAlisPro 1.171.39.16b (Rigaku OD, 2015)'
**computing_structure_solution_** 'SHELXS-2013/1 (Sheldrick, 2014)'
**computing_structure_refinement_** 'SHELXL-2014/7 (Sheldrick, 2014)'
**computing_molecular_graphics_** 'ORTEP3 for Windows'
computing_publication_material 'SHELXL-2014/7 and WINGX'

refine_special_details

refine_ls_structure_factor_coef Fsqd

refine_ls_matrix_type full

refine_ls_weighting_scheme calc

refine_ls_weighting_details

\[ w = 1 / \left( \sigma(Fo^2)^2 + (0.0526P)^2 + 0.4585P \right) \]

atom_sites_solution_primary difmap

atom_sites_solution_secondary difmap

atom_sites_solution_hydrogens difmap

refine_ls_hydrogen_treatment mixed

refine_ls_extinction_method 'SHELXL-2014/7 (Sheldrick 2014')

refine_ls_extinction_coef 0.0061(7)

refine_ls_extinction_expression

\[ Fc^* = kFc \left[ 1 + 0.001xFc^2 \right]^{1/4} \]

refine_ls_number_reflns 3041

refine_ls_number_parameters 200

refine_ls_number_restraints 0

refine_ls_R_factor_all 0.0344

refine_ls_R_factor_gt 0.0336

refine_ls_wR_factor_ref 0.0924

refine_ls_wR_factor_gt 0.0919

refine_ls_goodness_of_fit_ref 1.068

refine_ls_shift/su_mean 0.000

loop

atom_site_label
atom_site_type_symbol
atom_site_fract_x
atom_site_fract_y
atom_site_fract_z
atom_site_U_iso_or_equiv
atom_site_adp_type
atom_site_occupancy
atom_site_symmetry_order
atom_site_calc_flag
atom_site_refinement_flags_posn
atom_site_refinement_flags_adp
atom_site_refinement_flags_occupancy
atom_site_disorder_assembly
atom_site_disorder_group

S2 S -0.04981(6) 0.93024(6) 0.79299(3) 0.01851(13) Uani 1 1 d . . . .
C112 C 0.44256(6) 0.63514(6) 0.65676(3) 0.02420(14) Uani 1 1 d . . . .
C114 Cl -0.21596(8) 0.05515(6) 0.43692(3) 0.03278(15) Uani 1 1 d . . . .
O5 O 0.50820(17) 1.21192(17) 1.07306(9) 0.0205(3) Uani 1 1 d . . . .
N1 H 0.1972(9) 0.7249(2) 0.78786(10) 0.0162(3) Uani 1 1 d . . . .
O5 O 0.50820(17) 1.21192(17) 1.07306(9) 0.0205(3) Uani 1 1 d . . . .
H1 H 0.269(3) 0.724(3) 0.8115(16) 0.020 Uiso 1 1 d . U . . . .
N3 N 0.2197(2) 0.95531(19) 0.92821(10) 0.0162(3) Uani 1 1 d . . . .
H3 H 0.318(3) 0.935(3) 0.9452(15) 0.019 Uiso 1 1 d . U . . . .
N4 N 0.2228(2) 1.13209(19) 0.97884(10) 0.0154(3) Uani 1 1 d . . . .
H4 H 0.135(3) 1.162(3) 0.96031(15) 0.018 Uiso 1 1 d . . . .
N52 N 0.1509(2) 1.4666(2) 1.06302(10) 0.0188(3) Uani 1 1 d . . . .
C5 C 0.3727(2) 1.2538(2) 0.84886(11) 0.0146(3) Uani 1 1 d . . . .
C11 C -0.0750(2) 0.769(2) 0.70041(11) 0.0159(3) Uani 1 1 d . . . .
C12 C 0.1866(2) 0.5137(2) 0.63525(12) 0.0171(3) Uani 1 1 d . . . .
C13 C 0.0989(3) 0.3545(2) 0.55345(12) 0.0209(4) Uani 1 1 d . . . .
H13 H 0.1749 0.3128 0.5112 0.025 Uiso 1 1 calc R U . . . .
C14 C 0.1039(3) 0.2599(2) 0.53654(12) 0.0215(4) Uani 1 1 d . . . .
C15 C 0.2195(3) 0.3233(3) 0.59699(13) 0.0221(4) Uani 1 1 d . . . .
H15 H -0.3562 0.2609 0.5828 0.027 Uiso 1 1 calc R U . . . .
C16 C -0.1295(3) 0.4808(2) 0.67890(12) 0.0193(3) Uani 1 1 d . . . .
H16 H 0.2067 0.5230 0.7201 0.023 Uiso 1 1 calc R U . . . .
C51 C 0.3664(2) 1.4455(2) 1.09387(11) 0.0141(3) Uani 1 1 d . . . .
C53 C 0.5146(2) 1.7693(2) 1.19904(12) 0.0184(3) Uani 1 1 d . . . .
H53 H 0.6173 1.8702 1.2443 0.022 Uiso 1 1 calc R U . . . .
C54 C 0.3504(3) 1.7936(2) 1.16894(12) 0.0210(4) Uani 1 1 d . . . .
H54 H 0.3403 1.9110 1.1936 0.025 Uiso 1 1 calc R U . . . .
C55 C 0.1998(3) 1.6388(3) 1.10085(13) 0.0227(4) Uani 1 1 d . . . . .
H55 H 0.0596 1.6562 1.0807 0.027 Uiso 1 1 calc R U . .
C56 C 0.5244(2) 1.5914(2) 1.16058(11) 0.0161(3) Uani 1 1 d . . . . .
H56 H 0.6336 1.5710 1.1791 0.019 Uiso 1 1 calc R U . .

loop_
_atom_site_aniso_label
_atom_site_aniso_U_11
_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
S2 0.0172(2) 0.0197(2) 0.0176(2) -0.00037(15) -0.00255(15) 0.01044(16)
Cl12 0.0181(2) 0.0274(2) 0.0252(2) 0.00222(17) 0.00428(16) 0.0062(2)
Cl14 0.0443(3) 0.0199(2) 0.0190(2) -0.00743(16) -0.00413(18) 0.0062(2)
O5 0.0208(6) 0.0187(6) 0.0202(6) -0.0021(5) -0.0036(5) 0.0116(5)
N1 0.0149(6) 0.0174(7) 0.0159(7) -0.0030(5) -0.0032(5) 0.0088(5)
N3 0.0187(7) 0.0130(6) 0.0156(6) -0.0020(5) -0.0016(5) 0.0092(5)
N4 0.0167(7) 0.0123(6) 0.0157(6) -0.0021(5) -0.0015(5) 0.0083(5)
N52 0.0203(7) 0.0157(7) 0.0182(7) -0.0005(5) -0.0030(5) 0.0090(6)
C2 0.0133(7) 0.0135(7) 0.0158(7) 0.0011(6) 0.0011(6) 0.0036(6)
C5 0.0149(7) 0.0145(7) 0.0130(7) 0.0013(6) 0.0022(6) 0.0063(6)
C11 0.0197(8) 0.0135(7) 0.0127(7) -0.0001(6) -0.0009(6) 0.0076(6)
C12 0.0174(8) 0.0164(8) 0.0170(7) 0.0025(6) 0.0009(6) 0.0080(6)
C13 0.0304(9) 0.0181(8) 0.0161(8) 0.0020(6) 0.0046(7) 0.0136(7)
C14 0.0308(9) 0.0128(8) 0.0140(7) -0.0012(6) -0.0024(6) 0.0054(7)
C15 0.0203(8) 0.0178(8) 0.0201(8) 0.0015(6) -0.0019(6) 0.0022(7)
C16 0.0201(8) 0.0165(8) 0.0169(8) -0.0001(6) 0.0031(6) 0.0060(6)
C51 0.0163(7) 0.0128(7) 0.0126(7) 0.0019(6) 0.0021(6) 0.0064(6)
C53 0.0209(8) 0.0130(7) 0.0153(7) 0.0003(6) 0.0006(6) 0.0034(6)
C54 0.0277(9) 0.0134(8) 0.0208(8) -0.0002(6) 0.0016(7) 0.0106(7)
C55 0.0249(9) 0.0178(8) 0.0247(8) 0.0007(7) -0.0040(7) 0.0121(7)
C56 0.0152(7) 0.0158(7) 0.0147(7) 0.0013(6) 0.0005(6) 0.0056(6)

_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.
;
loop_
_geom_bond_atom_site_label_1
_geom_bond_atom_site_label_2
_geom_bond_distance
_geom_bond_site_symmetry_2
_geom_bond_publ_flag
S2 C2 1.6730(16) . ?
Cl12 C12 1.7422(17) . ?
Cl14 C14 1.7383(17) . ?
O5 C5 1.238(2) . ?
N1 C2 1.355(2) . ?
N1 C11 1.409(2) . ?
N1 H1 0.79(2) . ?
N3 C3 1.359(2) . ?
N3 N4 1.3805(18) . ?
N3 H3 0.85(2) . ?
N4 C5 1.330(2) . ?
N4 H4 0.83(2) . ?
N52 C55 1.337(2) . ?
N52 C51 1.343(2) . ?
C5 C51 1.495(2) . ?
C11 C16 1.393(2) . ?
C11 C12 1.399(2) . ?
C12 C13 1.388(2) . ?
C13 C14 1.380(3) . ?
C13 H13 0.9300 . ?
C14 C15 1.383(3) . ?
C15 C16 1.385(2) . ?
C15 H15 0.9300 . ?
C16 H16 0.9300 . ?
C51 C56 1.387(2) . ?
C53 C54 1.379(2) . ?
C53 C56 1.393(2) . ?
C53 H53 0.9300 . ?
C54 C55 1.394(2) . ?
C54 H54 0.9300 . ?
C55 H55 0.9300 . ?
C56 H56 0.9300 . ?

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_geom_angle_atom_site_label_2
_geom_angle_atom_site_label_3
_geom_angle
_geom_angle_site_symmetry_1
_geom_angle_site_symmetry_3
_geom_angle_publ_flag
C2 N1 C11 127.93(14) . . ?
C2 N1 H1 116.7(16) . . ?
C11 N1 H1 115.3(16) . . ?
C2 N3 N4 119.61(14) . . ?
C2 N3 H3 120.0(14) . . ?
N4 N3 H3 115.0(14) . . ?
C5 N4 N3 119.61(14) . . ?
C5 N4 H4 122.5(15) . . ?
C5 N4 H4 117.6(15) . . ?
C5 N52 C51 116.99(14) . . ?
N1 C2 N3 111.82(14) . . ?
N1 C2 S2 126.49(12) . . ?
N3 C2 S2 121.68(12) . . ?
O5 C5 N4 122.29(15) . . ?
O5 C5 C51 123.01(14) . . ?
N4 C5 C51 114.69(14) . . ?
C16 C11 C12 118.08(14) . . ?
C16 C11 N1 122.55(14) . . ?
C12 C11 N1 119.21(15) . . ?
C13 C12 C11 121.70(15) . . ?
C13 C12 C11 118.58(13) . . ?
C11 C12 C11 119.72(12) . . ?
C14 C13 C12 118.24(15) . . ?
C14 C13 H13 120.9 . . ?
C12 C13 H13 120.9 . . ?
C13 C14 C15 121.69(15) . . ?
C13 C14 C14 118.90(13) . . ?
C15 C14 C14 119.40(14) . . ?
C14 C15 C16 119.25(16) . . ?
C14 C15 H15 120.4 . . ?
C16 C15 H15 120.4 . . ?
C15 C16 C11 120.94(16) . . ?
C15 C16 H16 119.5 . . ?
C11 C16 H16 119.5 . . ?
N52 C51 C56 123.88(15) . . ?
N52 C51 C5 116.16(14) . . ?
C56 C51 C5 116.99(14) . . ?
C54 C53 C56 118.96(15) . . ?
C54 C53 H53 120.5 . . ?
C56 C53 H53 120.5 . . ?
C53 C54 C55 118.74(15) . . ?
C53 C54 H54 120.6 . . ?
C55 C54 H54 120.6 . . ?
N52 C55 C54 123.33(16) . . ?
N52 C55 H55 118.3 . . ?
C54 C55 H55 118.3 . . ?
C51 C56 C53 118.09(15) . . ?
C51 C56 H56 121.0 . . ?
C3 C56 H56 121.0 . . ?

loop_
  _geom_torsion_atom_site_label_1
  _geom_torsion_atom_site_label_2
  _geom_torsion_atom_site_label_3
  _geom_torsion_atom_site_label_4
  _geom_torsion_site_symmetry_1
  _geom_torsion_site_symmetry_2
  _geom_torsion_site_symmetry_3
  _geom_torsion_site_symmetry_4

C2 N3 N4 C5 -154.75(15) . . . . ?
C11 N1 C2 N3 166.21(15) . . . . ?
C11 N1 C2 S2 -15.1(2) . . . . ?
N4 N3 C2 N1 162.99(14) . . . . ?
N4 N3 C2 -15.7(2) . . . . ?
N3 N4 C5 -15.7(2) . . . . ?
N4 N3 C5 S1 176.16(13) . . . . ?
C2 N1 C11 C16 -38.0(3) . . . . ?
C2 N1 C11 C12 146.60(17) . . . . ?
C16 C11 C12 C13 -2.9(2) . . . . ?
N1 C11 C12 C13 172.78(15) . . . . ?
C16 C11 C12 C12 177.64(12) . . . . ?
N1 C11 C12 C12 -6.7(2) . . . . ?
N1 C12 C13 C14 0.9(2) . . . . ?
C12 C12 C13 C14 -179.85(12) . . . . ?
C12 C13 C14 C15 1.9(3) . . . . ?
C12 C13 C14 C14 -177.73(13) . . . . ?
C13 C14 C15 C16 -2.8(3) . . . . ?
C14 C14 C15 C16 176.90(13) . . . . ?
C14 C15 C16 C11 0.7(3) . . . . ?
C12 C11 C16 C15 2.0(2) . . . . ?
N1 C11 C16 C15 -173.47(15) . . . . ?
C5 S2 C51 C56 -1.0(2) . . . . ?
C55 N52 C51 C5 -178.49(14) . . . . ?
O5 C5 C51 N52 -177.25(15) . . . . ?
N4 C5 C51 N52 4.1(2) . . . . ?
O5 C5 C51 C56 5.1(2) . . . . ?
N4 C5 C51 C56 -173.48(14) . . . . ?
C56 C53 C54 C55 -0.1(3) . . . . ?
C51 N52 C55 C54 0.4(3) . . . . ?
C53 C54 C55 N52 0.2(3) . . . . ?
N52 C51 C56 C53 1.1(2) . . . . ?
C5 C51 C56 C53 178.49(14) . . . . ?
C54 C53 C56 C51 -0.5(2) . . . . ?

_refine_diff_density_max  0.391
_refine_diff_density_min  -0.415
_refine_diff_density_rms   0.064

_shelx_res_file

; shelx.res created by SHELXL-2014/7

TITL mp-mm7 in P-1
CELL  1.54178   7.4539   7.8084  14.3223  104.442  92.188  112.898
ZERR  2.00   0.0002   0.0002   0.0002   0.002   0.002   0.002
LATT  1
SFAC C H CL N O S
UNIT 26 20 4 8 2 2
MREG  2
FMAP  2
ACTA
BOND SH
CONF
PLAN  -2
HKLF  4
REM    mp-mm7 in P-1
REM    R1 =  0.0336 for  2964 Fo > 4sig(Fo) and  0.0344 for all  3041 data
REM 200 parameters refined using  0 restraints
END

WGHT  0.0515   0.4827
REM Highest difference peak  0.391, deepest hole -0.415, 1-sigma level  0.064
Q1  1   0.3003  0.5649  0.6430  11.00000  0.05  0.39
Q2  1   0.1562  0.5542  0.6771  11.00000  0.05  0.36
;

CIF for 7
data_shelx

_loop_
_space_group_symop_operation_xyz
'x, y, z'
'x, -y, z+1/2'
'x+1/2, y+1/2, z'
'x+1/2, -y+1/2, z+1/2'
_cell_length_a 7.9236(4)
_cell_length_b 25.2269(2)
_cell_length_c 7.9636(2)

The symmetry employed for this shelxl refinement is uniquely defined by the following loop, which should always be used as a source of symmetry information in preference to the above space-group names. They are only intended as comments.
_cell_angle_alpha 90
_cell_angle_beta 113.018(2)
_cell_angle_gamma 90
_cell_volume 1465.09(9)
_cell_formula_units_Z 4
_cell_measurement_temperature 293(2)
_cell_measurement_reflns_used 194
_cell_measurement_theta_min 9.4
_cell_measurement_theta_max 77.4
_exptl_crystal_description needle
_exptl_crystal_colour colourless
_exptl_crystal_density_meas ?
_exptl_crystal_density_method ?
_exptl_crystal_density_diffn 1.444
_exptl_crystal_F_000 664
_exptl_transmission_factor_min ?
_exptl_transmission_factor_max ?
_exptl_crystal_size_max 0.59
_exptl_crystal_size_mid 0.07
_exptl_crystal_size_min 0.07
_exptl_absorpt_coefficient_mu 3.332
_shelx_estimated_absorpt_T_min ?
_shelx_estimated_absorpt_T_max ?
_exptl_absorpt_correction_type multi
_exptl_absorpt_correction_T_min 0.4190
_exptl_absorpt_correction_T_max 1.0000
_exptl_absorpt_process_details ;
_CrysAlisPro 1.171.39.16b (Rigaku Oxford Diffraction, 2015)
Empirical absorption correction using spherical harmonics,
implemented in SCALE3 ABSPACK scaling algorithm.
;
_exptl_absorpt_special_details ?
_diffrn_ambient_temperature 293(2)
_diffrn_radiation_wavelength 1.54178
_diffrn_radiation_type CuKα
_diffrn_source 'micro-focus sealed X-ray tube'
_diffrn_measurement_device 'four-circle diffractometer'
_diffrn_measurement_device_type 'XtaLAB Synergy, Dualflex, Pilatus 300K'
_diffrn_measurement_method 'w scans'
_diffrn_detector_area_resol_mean ?
_diffrn_reflns_number 7385
_diffrn_reflns_av_unetI/netI 0.0342
_diffrn_reflns_av_R_equivalents 0.0429
_diffrn_reflns_limit_h_min -9
_diffrn_reflns_limit_h_max 9
_diffrn_reflns_limit_k_min -31
_diffrn_reflns_limit_k_max 24
_diffrn_reflns_limit_l_min -9
_diffrn_reflns_limit_l_max 9
_diffrn_reflns_theta_min 6.317
_diffrn_reflns_theta_max 78.939
_diffrn_reflns_theta_full 67.679
_diffrn_measured_fraction_theta_max 0.948
_diffrn_measured_fraction_theta_full 0.999
_diffrn_Laue_measured_fraction_max 0.948
_diffrn_Laue_measured_fraction_full 0.999
_diffrn_point_group_measured_fraction_max 0.830
_diffrn_point_group_measured_fraction_full 0.904
_reflns_number_total 2622
_reflns_number_gt 2597
_reflns_threshold_expression 'I > 2σ(I)'
_reflns_Friedel_coverage 0.741
_reflns_Friedel_fraction_max 0.710
_reflns_Friedel_fraction_full 0.809
_reflns_special_details ;
Reflections were merged by SHELXL according to the crystal
class for the calculation of statistics and refinement.
reflns_Friedel_fraction is defined as the number of unique Friedel pairs measured divided by the number that would be possible theoretically, ignoring centric projections and systematic absences.

Computing data collection: CrysAlisPro 1.171.39.16b (Rigaku OD, 2015)
Computing cell refinement: CrysAlisPro 1.171.39.16b (Rigaku OD, 2015)
Computing data reduction: CrysAlisPro 1.171.39.16b (Rigaku OD, 2015)
Computing structure solution: SHELXS-2013/1 (Sheldrick, 2014)
Computing structure refinement: SHELXL-2014/7 (Sheldrick, 2014)
Computing molecular graphics: ORTEP3 for Windows
Computing publication material: SHELXL-2014/7 and WINGX

Refine special details:

Refine ls structure factor coef Fsqd
Refine ls matrix type full
Refine ls weighting scheme calc
Refine ls weighting details w=1/[\sqrt{2*(F_0^2*(P)+(0.0829P)^2*0.4088P]} where P=(F_0^2+2F_c^2)/3

Atom sites solution primary difmap
Atom sites solution secondary difmap
Atom sites solution hydrogens difmap
Refine ls hydrogen treatment mixed
Refine ls extinction method SHELXL-2014/7 (Sheldrick 2014)
Refine ls extinction coef 0.0047(6)
Refine ls extinction expression \( F_c^* = kF_c \left[ 1 + 0.001xF_c^2 l^3 / \sin(2q) \right]^{1/4} \)
Refine ls abs structure details:

Classical Flack method preferred over Parsons because s.u. lower.

Refine ls abs structure Flack 0.00(2)
Chemical absolute configuration ?
Refine ls number reflns 2622
Refine ls number parameters 201
Refine ls number restraints 2
Refine ls R_factor_all 0.0395
Refine ls R_factor_gt 0.0393
Refine ls W_R_factor_ref 0.1073
Refine ls W_R_factor_gt 0.1071
Refine ls goodness_of_fit_ref 1.076
Refine ls restrained_S_all 1.076
Refine ls shift/su_max 0.001
Refine ls shift/su_mean 0.000

Loop
Atom site label
Atom site type symbol
Atom site fract_x
Atom site fract_y
Atom site fract_z
Atom site U_iso_or_equiv
Atom site adp type
Atom site occupancy
Atom site site symmetry order
Atom site calc flag
Atom site refinement flags_posn
Atom site refinement flags_adp
Atom site refinement flags_occupancy
Atom site disorder assembly
Atom site disorder_group

S2 S 0.67222(10) 0.90743(3) 0.56405(11) 0.0185(3)
S17 S 0.18750(12) 0.72277(3) 0.89163(12) 0.0257(3)
O5 O 0.2214(4) 1.01240(9) 0.3897(3) 0.0172(6)
N1 N 0.4273(4) 0.93221(12) 0.7115(4) 0.0172(6)
N3 N 0.5672(4) 1.00093(12) 0.6312(4) 0.0168(6)
N4 N 0.4452(4) 1.03579(12) 0.6595(4) 0.0153(6)
C56 0.0175(17) 0.0182(17) 0.0227(18)
C55 0.0234(18) 0.0203(18) 0.027(2)
C54 0.0217(19) 0.0260(19) 0.028(2) 0.0007(16) 0.0161(16) 0.0054(14)
C51 0.0
C18 0.032(2) 0.026(2) 0.032(2) 0.0031(17) 0.0128(18)
C16 0.0163(17) 0.0183(17) 0.0209(18)
C14 0.0272(19) 0.0132(16) 0.0160(17) -0.0007(13) 0.0064(14) -0.0038(13)
C12 0.0245(19) 0.0180(17) 0.0233(19) 0.0011(14) 0.0068(15) 0.0048(14)
C10 0.0163(17) 0.0183(17) 0.0209(18) -0.0001(14) 0.0063(14) 0.0025(13)
C8 0.032(2) 0.026(2) 0.032(2) 0.0031(17) 0.0128(18) -0.0082(17)
C6 0.0147(16) 0.0148(15) 0.0163(16) 0.0017(13) 0.0081(12) 0.0006(12)
C4 0.0257(19) 0.0188(17) 0.0159(17) 0.0019(12) 0.0057(13) 0.0009(13)
C2 0.0345(18) 0.0203(18) 0.027(2) -0.0043(14) 0.0087(16) 0.0040(14)
C0 0.0175(17) 0.0182(17) 0.0227(18) -0.0028(14) 0.0075(13) -0.0012(13)

loop_
_atom_site_aniso_label
_atom_site_aniso_U_11
_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
S2 0.0174(4) 0.0196(4) 0.0212(4) -0.0002(3) 0.0099(3) 0.0052(3)
S1 0.0310(5) 0.0148(4) 0.0293(5) 0.0033(4) 0.0098(4) -0.0028(3)
O5 0.0178(6) 0.0186(11) 0.0166(11) -0.0035(10) 0.0073(9) -0.0018(9)
N4 0.0195(14) 0.0139(14) 0.0230(15) -0.0011(12) 0.0123(12) 0.0018(12)
N3 0.0131(13) 0.0165(15) 0.0252(16) -0.0009(12) 0.0123(12) 0.0007(11)
N2 0.0125(14) 0.0168(14) 0.0182(14) -0.0011(11) 0.0076(11) 0.0008(11)
N1 0.0162(14) 0.0228(16) 0.0263(17) -0.0077(15) 0.0095(12) 0.0004(11)
C2 0.0111(15) 0.0191(17) 0.0198(17) -0.0004(12) 0.0026(12) 0.0008(12)
C3 0.0142(15) 0.0127(15) 0.0166(17) 0.0030(12) 0.0071(13) -0.0016(12)
C4 0.0204(17) 0.0140(16) 0.0151(16) -0.0016(12) 0.0073(13) -0.0007(13)
C5 0.0183(17) 0.0162(17) 0.0185(17) 0.0041(13) 0.0058(13) 0.0013(13)
C6 0.0183(17) 0.0181(17) 0.0221(18) -0.0012(14) 0.0076(14) -0.0031(14)
C7 0.0227(19) 0.0132(16) 0.0160(17) -0.0007(13) 0.0064(14) -0.0038(13)
C8 0.0245(19) 0.0180(17) 0.0233(19) 0.0011(14) 0.0068(15) 0.0048(14)
C9 0.0163(17) 0.0183(17) 0.0209(18) -0.0001(14) 0.0063(14) 0.0025(13)
C10 0.032(2) 0.026(2) 0.032(2) 0.0031(17) 0.0128(18) -0.0082(17)
C11 0.0147(16) 0.0148(15) 0.0163(16) 0.0017(13) 0.0081(12) 0.0006(12)
C12 0.0257(19) 0.0188(17) 0.0159(17) 0.0019(12) 0.0057(13) 0.0009(13)
C13 0.0217(19) 0.0260(19) 0.028(2) 0.0007(16) 0.0161(16) 0.0054(14)
C14 0.0234(18) 0.0203(18) 0.027(2) -0.0043(14) 0.0087(16) 0.0040(14)
C15 0.0175(17) 0.0182(17) 0.0227(18) -0.0028(14) 0.0075(13) -0.0012(13)

_all

; 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.
;

loop_
_geom_bond_atom_site_label_1
S2 C2 1.695(4) .?
S17 C14 1.769(4) .?
S17 C18 1.796(5) .?
O5 C5 1.226(4) .?
N1 C2 1.335(5) .?
N1 C11 1.423(5) .?
N1 H1 0.87(5) .?
N3 C2 1.357(5) .?
N3 N4 1.389(4) .?
N3 H3 0.79(5) .?
N4 C5 1.362(4) .?
N4 H4 0.94(5) .?
N53 C52 1.335(5) .?
N53 C54 1.340(5) .?
C5 C51 1.488(5) .?
C11 C12 1.388(5) .?
C11 C16 1.397(5) .?
C12 C13 1.388(5) .?
C12 H12 0.9300 .?
C13 C14 1.388(5) .?
C13 H13 0.9300 .?
C14 C15 1.406(6) .?
C15 C16 1.379(5) .?
C15 H15 0.9300 .?
C16 H16 0.9300 .?
C18 H81 0.9600 .?
C18 H82 0.9600 .?
C18 H83 0.9600 .?
C51 C56 1.388(5) .?
C51 C52 1.389(5) .?
C52 H52 0.9300 .?
C54 C55 1.390(6) .?
C54 H54 0.9300 .?
C55 C56 1.377(6) .?
C55 H55 0.9300 .?
C56 H56 0.9300 .?

C14 S17 C18 103.51(19) .?
C2 N1 C11 130.2(3) .?
C12 C11 H1 118(3) .?
C11 N1 H1 111(3) .?
C2 N3 N4 120.8(3) .?
C13 C12 C11 120.6(3) .?
C13 C12 H12 119.7 . . .
C11 C12 H12 119.7 . . .
C12 C13 C14 120.3(3) . . .
C12 C13 H13 119.9 . . .
C14 C13 H13 119.9 . . .
C13 C14 C15 118.8(3) . . .
C13 C14 S17 124.0(3) . . .
C15 C14 S17 117.2(3) . . .
C16 C15 C14 121.1(3) . . .
C16 C15 H15 119.5 . . .
C14 C15 H15 119.5 . . .
C15 C16 C11 119.6(3) . . .
C15 C16 H16 120.2 . . .
C11 C16 H16 120.2 . . .
S17 C18 H81 109.5 . . .
S17 C18 H82 109.5 . . .
H81 C18 H82 109.5 . . .
S17 C18 H83 109.5 . . .
H81 C18 H83 109.5 . . .
H82 C18 H83 109.5 . . .
C56 C51 C52 118.8(3) . . .
C56 C51 C5 125.0(3) . . .
C52 C51 C5 116.1(3) . . .
N53 C52 C51 123.2(3) . . .
N53 C52 H52 118.4 . . .
C51 C52 H52 118.4 . . .
N53 C54 C55 122.8(3) . . .
N53 C54 H54 118.6 . . .
C55 C54 H54 118.6 . . .
C56 C55 C54 119.3(4) . . .
C56 C55 H55 120.4 . . .
C54 C55 H55 120.4 . . .
C55 C56 C51 118.3(3) . . .
C55 C56 H56 120.8 . . .
C51 C56 H56 120.8 . . .

loop_
    _geom_torsion_atom_site_label_1
    _geom_torsion_atom_site_label_2
    _geom_torsion_atom_site_label_3
    _geom_torsion_atom_site_label_4
    _geom_torsion_site_symmetry_1
    _geom_torsion_site_symmetry_2
    _geom_torsion_site_symmetry_3
    _geom_torsion_site_symmetry_4
    _geom_torsion_publ_flag
C2 N3 N4 C5 -69.1(4) . . .
C11 N1 C2 N3 179.4(3) . . .
C11 N1 C2 S2 1.0(5) . . .
N4 N3 C2 N1 -11.0(5) . . .
N4 N3 C2 S2 167.5(3) . . .
N3 N4 C5 O5 -5.5(5) . . .
N3 N4 C5 C51 169.8(3) . . .
C2 N1 C11 C12 -142.7(4) . . .
C2 N1 C11 C16 44.1(5) . . .
C16 C11 C12 C13 0.2(6) . . .
N1 C11 C12 C13 -173.3(3) . . .
C11 C12 C13 C14 0.4(6) . . .
C12 C13 C14 C15 -0.4(5) . . .
C12 C13 C14 S17 178.3(3) . . .
C18 S17 C14 C13 -15.8(4) . . .
C18 S17 C14 C15 163.0(3) . . .
C13 C14 C15 C16 -0.1(6) . . .
S17 C14 C15 C16 -178.9(3) . . .
C14 C15 C16 C11 0.6(6) . . .
C12 C11 C16 C15 -0.7(5) . . .
N1 C11 C16 C15 172.3(4) . . .
O5 C5 C51 C56 -148.9(4) . . .
N4 C5 C51 C56 35.8(5) . . .
O5 C5 C51 C52 35.0(5) . . . . ?
N4 C5 C52 -140.3(3) . . . . ?
C54 N53 C52 1.9(5) . . . . ?
C56 C51 C52 N53 -2.2(5) . . . . ?
C5 C51 C52 N53 174.1(3) . . . . ?
C52 N53 C54 C55 0.3(6) . . . . ?
C54 C55 C56 C51 -2.2(5) . . . . ?
C52 C51 C56 C55 0.5(5) . . . . ?
C5 C51 C56 C55 -175.5(3) . . . . ?

_refine_diff_density_max 0.277
_refine_diff_density_min -0.330
_refine_diff_density_rms 0.066

_shelx_res_file
;

shelx.res created by SHELXL-2014/7

TITL mm20 in Cc
CELL  1.54178   7.9236  25.2269  7.9636  90.000  113.018  90.000
ZERR     4.00   0.0004   0.0002   0.0002    0.000    0.002    0.000
LATT -7
SYMM    X, - Y, 1/2 + Z
SFAC C H N O S
UNIT 56   56   16   4   8
MERG   2
FMAP   2
PLAN -2
ACTA
BOND SH
CONF
L.S.  40
WGHT  0.082900   0.408800
EXTI  0.004704
FVAR   0.65116
S2  5  0.672223  0.907429  0.564053  11.00000  0.01697  0.01962 =
    0.02121 -0.00022  0.00985  0.00524
S17  5  0.187504  0.722768  0.891634  11.00000  0.03103  0.01482 =
    0.02927  0.00325  0.00976 -0.00276
O5  4  0.221374  1.012404  0.389651  11.00000  0.01758  0.01861 =
    0.01655 -0.00345  0.00728 -0.00184
N1  3  0.427275  0.932214  0.711469  11.00000  0.01948  0.01296 =
    0.02298 -0.00108  0.01228  0.00184
N3  3  0.567240  1.000926  0.631168  11.00000  0.01312  0.01650 =
    0.02522 -0.00086  0.01234  0.00672
N4  3  0.445150  1.035786  0.659492  11.00000  0.01250  0.01681 =
    0.01815 -0.00111  0.00756  0.00078
N53 3   0.164147  1.067318  0.585358  11.00000  0.01616  0.02277 =
    0.02627 -0.00668  0.00955  0.00039
C2  1  0.547894  0.947746  0.642334  11.00000  0.01109  0.01906 =
    0.01194 -0.00074  0.00255  0.00077
C5  1  0.269832  1.037534  0.532616  11.00000  0.01424  0.01268 =
    0.01664  0.00303  0.00710 -0.00158
C11 1  0.373362  0.880327  0.740316  11.00000  0.02044  0.01405 =
    0.01509 -0.00164  0.00734 -0.00066
C12 1  0.189627  0.873373  0.708785  11.00000  0.01830  0.01619 =
    0.01851  0.00141  0.00583  0.00126
AFIX  43
H12 2  0.107055  0.900893  0.658754  11.00000 -1.20000
AFIX  0
C13 1  0.128037  0.825710  0.751266  11.00000  0.01833  0.01814 =
    0.02213 -0.00124  0.00763 -0.00311
AFIX  43
H13 2  0.004527  0.821416  0.728903  11.00000 -1.20000

21
| AFIX | C14 | 0.250108 | 0.784432 | 0.826999 | 11.00000 | 0.02718 | 0.01321 = 0.01603 - 0.00072 | 0.00639 - 0.00377 |
|------|-----|----------|----------|----------|-----------|---------|-----------------------------|---------------------|
| AFIX | C15 | 0.435952 | 0.791771 | 0.858410 | 11.00000 | 0.02452 | 0.01800 = 0.02333 | 0.00113 | 0.00683 | 0.00481 |
| AFIX | C16 | 0.497207 | 0.838922 | 0.815026 | 11.00000 | 0.01628 | 0.01829 = 0.02091 - 0.00010 | 0.00626 | 0.00252 |
| AFIX | C17 | 0.518870 | 0.764383 | 0.909257 | 11.00000 | 1.20000 |
| AFIX | C18 | 0.497207 | 0.838922 | 0.815026 | 11.00000 | 0.01628 | 0.01829 = 0.02091 - 0.00010 | 0.00626 | 0.00252 |
| AFIX | C19 | 0.620278 | 0.843137 | 0.835385 | 11.00000 | 1.20000 |
| AFIX | C20 | 0.620278 | 0.843137 | 0.835385 | 11.00000 | 1.20000 |
| AFIX | C21 | 0.138216 | 1.068384 | 0.585471 | 11.00000 | 0.01470 | 0.01480 = 0.01632 | 0.00173 | 0.00808 | 0.00060 |
| AFIX | C22 | 0.138216 | 1.068384 | 0.585471 | 11.00000 | 0.01470 | 0.01480 = 0.01632 | 0.00173 | 0.00808 | 0.00060 |
| AFIX | C23 | 0.064887 | 1.016658 | 0.461688 | 11.00000 | 1.20000 |
| AFIX | C24 | 0.064887 | 1.016658 | 0.461688 | 11.00000 | 1.20000 |
| AFIX | C25 | 0.047149 | 1.136612 | 0.737773 | 11.00000 | 0.02336 | 0.02031 = 0.02684 - 0.00427 | 0.00875 | 0.00402 |
| AFIX | C26 | 0.047149 | 1.136612 | 0.737773 | 11.00000 | 0.02336 | 0.02031 = 0.02684 - 0.00427 | 0.00875 | 0.00402 |
| AFIX | C27 | 0.209698 | 1.113001 | 0.922917 | 11.00000 | 1.20000 |
| AFIX | C28 | 0.209698 | 1.113001 | 0.922917 | 11.00000 | 1.20000 |
| AFIX | C29 | 0.070203 | 1.167931 | 0.804619 | 11.00000 | 0.03901 |
| AFIX | C30 | 0.070203 | 1.167931 | 0.804619 | 11.00000 | 0.03901 |
| AFIX | C31 | 0.180262 | 1.114464 | 0.688907 | 11.00000 | 0.01820 = 0.02273 - 0.00279 | 0.00751 | 0.00117 |
| AFIX | C32 | 0.180262 | 1.114464 | 0.688907 | 11.00000 | 0.01820 = 0.02273 - 0.00279 | 0.00751 | 0.00117 |

REM mm20 in Cc
REM R1 = 0.0393 for 2597 Fo > 4sig(Fo) and 0.0395 for all 2622 data
REM 201 parameters refined using 2 restraints

END

WGHT   0.0844  0.1856

REM Highest difference peak 0.277, deepest hole -0.330, 1-sigma level 0.066
Q1 1 0.2039 0.7241 1.1592 11.00000 0.05 0.28
Q2 1 0.0972 0.7258 0.9250 11.00000 0.05 0.27

CIF for 11
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atom_type_symbol
atom_type_description
atom_type_scat_dispersion_real
atom_type_scat_dispersion_imag
atom_type_scat_source
'C' 'C' 0.0181 0.0091
'H' 'H' 0.0000 0.0000
'Cl' 'Cl' 0.3639 0.7018
'N' 'N' 0.0311 0.0180
'O' 'O' 0.0492 0.0322
'S' 'S' 0.3331 0.5567
International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

space_group_crystal_system        monoclinic
space_group_IT_number             14
space_group_name_H-M_alt         'P 21/c'
space_group_name_Hall             '-P 2ybc'

shelx_space_group_comment
The symmetry employed for this shelx refinement is uniquely defined
by the following loop, which should always be used as a source of
symmetry information in preference to the above space-group names.
They are only intended as comments.

loop_
space_group_symop_operation_xyz
'x, y, z'
'x, y+1/2, z+1/2'
'x, -y, -z'
'x, -y+1/2, z-1/2'

cell_length_a                    4.51760(10)
cell_length_b                    27.8468(4)
cell_length_c                    11.84240(10)
cell_angle_alpha                 90

cell_measurement_temperature     293(2)
cell_measurement_theta_min       10.1

cell_absolute_coefficient_mu     5.328
shelx_estimated_absorpt_T_min    ?
shelx_estimated_absorpt_T_max    ?

exptl_crystal_description        prism
exptl_crystal_colour             colourless
exptl_crystal_density_meas       ?
exptl_crystal_density_method     ?
exptl_crystal_density_diffm     1.538
exptl_crystal_F_000              696
exptl_transmission_factor_min    ?
exptl_transmission_factor_max    ?
exptl_crystal_size_max          0.59
exptl_crystal_size_mid          0.23
exptl_crystal_size_min          0.14
exptl_absorpt_coefficient_mu     5.328
shelx_estimated_absorpt_T_min    ?
shelx_estimated_absorpt_T_max    ?
exptl_absorpt_correction_type    multi-scan
exptl_absorpt_correction_T_min   0.2818
exptl_absorpt_correction_T_max   1.0000
Reflections were merged by SHELXL according to the crystal class for the calculation of statistics and refinement.

Reflections Friedel fraction is defined as the number of unique Friedel pairs measured divided by the number that would be possible theoretically, ignoring centric projections and systematic absences.
\[ F_c^{**} = k F_c [1 + 0.001 x F_c^2 / \sin(2 \theta)]^{1/4} \]

Refine LS extinction expression

Refine LS number refinements: 3028
Refine LS number parameters: 200
Refine LS number restraints: 0
Refine LS R factor all: 0.0429
Refine LS R factor gt: 0.0402
Refine LS goodness of fit ref: 1.044
Refine LS restrained S all: 1.044
Refine LS shift/su max: 0.000
Refine LS shift/su mean: 0.000

Loop

Atom site label
Atom site type symbol
Atom site fraction x
Atom site fraction y
Atom site fraction z
Atom site Uiso or equiv
Atom site occupancy
Atom site symmetry order
Atom site calc flag
Atom site refinement flags posn
Atom site refinement flags adp
Atom site refinement flags occupancy
Atom site disorder assembly
Atom site disorder group

S2 S 0.34164(12) 0.33702(2) 0.39834(4) 0.03492(18) Uani 1 1 d . . . .
C112 Cl 0.14348(18) 0.43635(2) 0.01969(4) 0.0549(2) Uani 1 1 d . . . .
C114 Cl 0.4779(2) 0.55803(2) 0.33033(6) 0.0636(2) Uani 1 1 d . . . .
O5 O 0.4945(3) 0.32404(5) 0.02058(10) 0.0260(3) Uani 1 1 d . . . .
N1 N 0.0152(4) 0.36895(6) 0.20905(13) 0.0306(4) Uani 1 1 d . . . .
H1 H 0.076(5) 0.3632(9) 0.142(2) 0.037 Uiso 1 1 calc R U . . . .
N3 N 0.2130(3) 0.29664(6) 0.19837(12) 0.0243(3) Uani 1 1 d . . . .
H3 H 0.356(5) 0.2752(8) 0.2195(19) 0.029 Uiso 1 1 d . U . . . .
N4 N 0.0855(3) 0.29399(6) 0.0243(3) Uani 1 1 d . . . .
H4 H 0.103(6) 0.2909(8) 0.0689(19) 0.028 Uiso 1 1 d . U . . . .
N53 N 0.3109(3) 0.27156(6) 0.25529(13) 0.0276(4) Uani 1 1 d . . . .
C2 C 0.1704(4) 0.33549(7) 0.26298(15) 0.0238(4) Uani 1 1 d . . . .
C5 C 0.2472(4) 0.30438(8) 0.33077(15) 0.0316(4) Uani 1 1 d . . . .
H5 H 0.2637 0.4774 0.4588 0.045 Uiso 1 1 calc R U . . . .
C16 C 0.1348(5) 0.42562(8) 0.35706(17) 0.0352(5) Uani 1 1 d . . . .
H16 H 0.0816 0.4032 0.4148 0.042 Uiso 1 1 calc R U . . . .
C51 C 0.0679(4) 0.30708(6) 0.11721(14) 0.0196(3) Uani 1 1 d . . . .
C52 C 0.1529(4) 0.27286(6) 0.15030(14) 0.0216(4) Uani 1 1 d . . . .
H52 H 0.1922 0.2500 0.0973 0.026 Uiso 1 1 calc R U . . . .
C54 C 0.2472(4) 0.30438(8) 0.33077(15) 0.0316(4) Uani 1 1 d . . . .
H54 H 0.3578 0.3040 0.4036 0.038 Uiso 1 1 calc R U . . . .
C55 C 0.0268(5) 0.33871(7) 0.30657(16) 0.0324(5) Uani 1 1 d . . . .
H55 H 0.0126 0.3604 0.3622 0.039 Uiso 1 1 calc R U . . . .
C56 C 0.1349(4) 0.34018(7) 0.19737(16) 0.0266(4) Uani 1 1 d . . . .
H56 H 0.2851 0.3629 0.1783 0.032 Uiso 1 1 calc R U . . . .

Loop

Atom site aniso label
Atom site aniso U_11
Atom site aniso U_12
Atom site aniso U_13
Atom site aniso U_14
Atom site aniso U_15
Atom site aniso U_16

At last, the document consists of numerical data related to a chemical structure, possibly a crystallographic refinement report.
S2 0.0398(3) 0.0417(3) 0.0184(3) 0.00103(18) -0.00114(2) -0.0016(2)
C12 0.1029(6) 0.0403(3) 0.0240(3) 0.0087(2) 0.0175(3) 0.0155(3)
C14 0.1097(6) 0.0272(3) 0.0644(4) -0.0001(3) 0.0478(4) -0.0003(3)
O5 0.0165(6) 0.0378(7) 0.0227(6) -0.0013(5) -0.0007(5) -0.0032(5)
N1 0.0350(9) 0.0395(9) 0.0240(8) -0.0025(9) -0.0020(9) -0.0037(9)
N3 0.0208(7) 0.0351(9) 0.0146(7) -0.0042(6) -0.0052(6) 0.0007(6)
N4 0.0155(7) 0.0358(8) 0.0037(6) -0.0045(5) -0.0033(6) 0.0068(6)
N53 0.0274(8) 0.0318(8) 0.0073(6) -0.0073(6) -0.0063(6) 0.0068(6)
C2 0.0208(9) 0.0329(10) 0.0011(7) -0.0030(7) -0.0022(9)
C11 0.0287(10) 0.0329(10) 0.0011(7) -0.0022(9)
C12 0.0505(13) 0.0366(11) 0.0248(10) 0.0018(8) 0.0099(9) -0.0022(9)
C13 0.0714(17) 0.0302(11) 0.0237(12) 0.0081(9) 0.0179(11) -0.00011(10)
C14 0.0536(13) 0.0263(10) 0.0445(12) -0.0025(9) -0.0220(10) -0.0077(9)
C15 0.0463(12) 0.0391(11) 0.0300(10) -0.0059(9) 0.0132(9) -0.0084(9)
C16 0.0405(11) 0.0416(11) 0.0232(10) 0.0022(8) 0.0040(8) -0.0010(9)
S2 C2 1.6733(17) . ?
C12 C12 1.745(2) . ?
C14 C14 1.747(2) . ?
O5 C5 1.217(2) . ?
N1 C2 1.349(2) . ?
N1 C11 1.403(3) . ?
N3 C2 1.355(2) . ?
N3 N4 1.3911(19) . ?
N4 C5 1.353(2) . ?
N53 C54 1.339(3) . ?
N53 C52 1.339(2) . ?
C5 C51 1.494(2) . ?
C11 C16 1.394(3) . ?
C11 C12 1.398(3) . ?
C12 C13 1.377(3) . ?
C13 C14 1.382(3) . ?
C14 C15 1.376(3) . ?
C15 C16 1.384(3) . ?
C51 C56 1.388(3) . ?
C51 C52 1.393(2) . ?
C54 C55 1.379(3) . ?
C55 C56 1.388(3) . ?
C2 N3 N4 121.22(15) . . ?  
C5 N4 N3 120.62(14) . . ?  
C5 N3 C52 117.63(16) . . ?  
N1 C2 N3 114.50(15) . . ?  
N1 C2 S2 127.01(15) . . ?  
N3 C2 S2 118.48(13) . . ?  
O5 C5 N4 122.71(15) . . ?  
O5 C5 C51 122.36(15) . . ?  
N4 C5 C51 114.92(14) . . ?  
C16 C11 C12 117.68(19) . . ?  
C16 C11 N1 124.67(18) . . ?  
C12 C11 N1 117.50(17) . . ?  
C13 C12 C11 122.03(19) . . ?  
C13 C12 C11 118.67(17) . . ?  
C11 C12 C11 119.30(17) . . ?  
C12 C13 C14 118.5(2) . . ?  
C15 C14 C13 121.3(2) . . ?  
C15 C14 C14 119.74(17) . . ?  
C13 C14 C14 118.92(18) . . ?  
C14 C15 C16 119.53(19) . . ?  
C15 C16 C11 120.93(2) . . ?  
C56 C51 C52 118.80(15) . . ?  
N53 C52 C51 122.69(17) . . ?  
C54 C55 C56 118.53(18) . . ?  
C55 C56 C5 118.64(18) . . ?

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.geom_torsion_site_symmetry_3  
.geom_torsion_site_symmetry_4  
.geom_torsion_publ_flag  
C2 N3 N4 C5 92.1(2) . . . . ?  
C11 N1 C2 N3 -176.89(19) . . . . ?  
C11 N1 C2 S2 4.4(3) . . . . ?  
N4 N3 C2 N1 3.5(2) . . . . ?  
N4 N3 C2 S2 -177.69(12) . . . . ?  
N3 N4 C5 3.0(3) . . . . ?  
N3 N4 C5 C51 -176.47(15) . . . . ?  
C2 N1 C11 C16 -32.6(3) . . . . ?  
C2 N1 C11 C12 151.9(2) . . . . ?  
C16 C11 C12 C13 1.0(3) . . . . ?  
N1 C11 C12 C13 -174.8(2) . . . . ?  
C16 C11 C12 C12 179.45(16) . . . . ?  
N1 C11 C12 C12 -4.8(3) . . . . ?  
C11 C12 C13 C14 0.3(4) . . . . ?  
C12 C12 C13 C14 179.86(19) . . . . ?  
C12 C13 C14 C15 0.9(4) . . . . ?  
C12 C13 C14 C14 -177.97(19) . . . . ?  
C13 C14 C15 C16 -1.3(4) . . . . ?  
C14 C14 C15 C16 177.56(18) . . . . ?  
C14 C15 C16 C11 0.5(3) . . . . ?  
C12 C11 C16 C15 0.6(3) . . . . ?  
N1 C11 C16 C15 -174.9(2) . . . . ?  
O5 C5 C51 C56 -29.1(2) . . . . ?  
N4 C5 C51 C56 150.30(16) . . . . ?  
O5 C5 C51 C52 150.14(17) . . . . ?  
N4 C5 C51 C52 -30.4(2) . . . . ?  
C54 N53 C52 C51 1.0(3) . . . . ?  
C56 C51 C52 N53 -2.4(3) . . . . ?  
C5 C51 C52 N53 178.36(15) . . . . ?  
C52 N53 C4 C55 0.9(3) . . . . ?
N53 C54 C55 C56 -1.4(3) . . . . 
C54 C55 C56 C51 0.0(3) . . . . ?
C52 C51 C56 C55 1.8(3) . . . . ?
C5 C51 C56 C55 -178.92(16) . . . . ?

_refine_diff_density_max 0.400
_refine_diff_density_min -0.407
_refine_diff_density_rms 0.061

_shelx_res_file

; shelx.res created by SHELXL-2014/7

TITL mm28 in P21/c
CELL 1.54178 4.5176 27.8468 11.8424 90.000 98.573 90.000
ZERR  4.00 0.0001 0.0004 0.0001 0.000 0.001 0.000
LATT 1
SYMM - X, 1/2 + Y, 1/2 - Z
SFAC C H CL N O S
UNIT 52   40   8    16   4   4
MERG  2
BOND &H
CONF
FMAP  2
PLAN  -2
ACCTA L.S. 40
WGHT  0.061800  0.764200
EXTI 0.003596
FVAR  0.60458
S2  6  0.341639  0.337020  0.398338  11.00000  0.03984  0.04174 =
   0.01836  0.00103 -0.01143 -0.00164
CL12  3 -0.143476  0.436353  0.019690  11.00000  0.10288  0.04028 =
   0.02399  0.00869  0.01752  0.01551
CL14  3 -0.477875  0.558034  0.330334  11.00000  0.10966  0.02725 =
   0.06442 -0.00005  0.04780  0.00026
O5  5  0.494454  0.324042  0.020582  11.00000  0.01647  0.03780 =
   0.02274 -0.00132 -0.00069 -0.00321
N1  4 -0.015243  0.368954  0.209048  11.00000  0.03499  0.03952 =
   0.01476  0.00033 -0.00427  0.00630
H1  2 -0.075819  0.363186  0.141838  11.00000 -1.20000
N3  4  0.213004  0.296638  0.198371  11.00000  0.02084  0.03505 =
   0.01465  0.00416 -0.00525  0.00071
H3  2  0.355800  0.275182  0.219480  11.00000 -1.20000
N4  4  0.085536  0.293989  0.084129  11.00000  0.01550  0.03581 =
   0.01549  0.00368 -0.00453 -0.00327
H4  2 -0.102579  0.290894  0.068919  11.00000 -1.20000
N53 4 -0.310866  0.271561 -0.255285  11.00000  0.02738  0.03175 =
   0.02060 -0.00727 -0.00626  0.00676
C2  1  0.170444  0.335491  0.262978  11.00000  0.02083  0.03111 =
   0.01845  0.00511 -0.00008 -0.00643
C5  1  0.238334  0.309258  0.009391  11.00000  0.01744  0.01798 =
   0.01893 -0.00031 -0.00015  0.00404
C11  1 -0.102783  0.414103  0.244969  11.00000  0.02870  0.03286 =
   0.02340  0.00107  0.00120 -0.00298
C12  1 -0.182867  0.448970  0.161059  11.00000  0.05053  0.03361 =
   0.02482  0.00178  0.00994 -0.00225
C13  1 -0.294208  0.493098  0.186203  11.00000  0.07137  0.03021 =
   0.03735  0.00812  0.01788 -0.00008
AFIX  43
H13  2 -0.347015  0.515778  0.129106  11.00000 -1.20000
AFIX  0
C14  1 -0.325717  0.502927  0.298161  11.00000  0.05355  0.02635 =
   0.04454 -0.00253  0.02202 -0.00767
C15  1 -0.244736  0.469948  0.383667  11.00000  0.04632  0.03907 =
   0.02998 -0.00586  0.01323 -0.00842
AFIX  43
H15  2 -0.263692  0.477379  0.458817  11.00000 -1.20000
AFIX 0
C16 1 -0.134825 0.425619 0.357062 11.00000 0.04049 0.04159 =
0.02319 0.00216 0.00397 -0.00098
AFIX 43
H16 2 -0.081634 0.403214 0.414759 11.00000 -1.20000
AFIX 0
C51 1 0.067886 0.307085 -0.117206 11.00000 0.01729 0.02372 =
0.01610 -0.00236 -0.00671 0.01435
AFIX 43
H52 2 -0.192239 0.249982 -0.097283 11.00000 -1.20000
AFIX 0
C54 1 -0.247208 0.304383 -0.330767 11.00000 0.03492 0.04065 =
0.01881 0.00736 0.00402 0.01016
AFIX 43
H55 2 0.012588 0.360383 -0.362195 11.00000 -1.20000
AFIX 0
C56 1 -0.134914 0.387060 -0.306570 11.00000 0.01600 0.02927 =
0.02295 0.00270 0.00671 0.01435
AFIX 43
H56 2 0.285095 0.362870 -0.178272 11.00000 -1.20000
AFIX 0
HKLF 4
REM mm28 in P21/c
REM R1 = 0.0402 for 2761 Fo > 4sig(Fo) and 0.0429 for all 3028 data
REM 200 parameters refined using 0 restraints
END
WGHT 0.0610 0.7552
REM Highest difference peak 0.400, deepest hole -0.407, 1-sigma level 0.061
Q1 1 -0.2687 0.5580 0.3405 11.00000 0.05 0.40
Q2 1 0.1096 0.3341 0.4070 11.00000 0.05 0.32

CIF for 13

data_shelx

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_atom_type_symbol
_atom_type_description
_atom_type_scat_dispers
ion_real
_atom_type_scat_dispersion_imag
_atom_type_scat_source
'C' 'C' 0.0181 0.0091
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'
'F' 'F' 0.0727 0.0534
International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'O' 'O' 0.0492 0.0322
International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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The symmetry employed for this shelx refinement is uniquely defined by the following loop, which should always be used as a source of symmetry information in preference to the above space-group names. They are only intended as comments.

| symmetry operation | 
|--------------------|
| x, y, z            |
| -x+1/2, y+1/2, -z+1/2 |
| -x, -y, -z         |
| x-1/2, -y+1/2, z-1/2 |

| cell length a | 13.4658(2) |
| cell length b | 9.80280(10) |
| cell length c | 23.9666(3)  |
| cell angle alpha | 90.0          |
| cell angle beta | 104.4020(10) |
| cell angle gamma | 90.0          |
| cell volume    | 3064.23(7)   |
| cell formula units Z | 4             |
| cell measurement temperature | 293(2)        |
| cell measurement reflections used | 472           |
| cell measurement theta min  | 3.8           |
| cell measurement theta max  | 77.4          |

| exptl crystal description | prism |
| exptl crystal colour | colourless |
| exptl crystal density measurement | 1.389 |
| exptl crystal density measurement type | \( F(000) \) |
| exptl crystal density measurement F(000) | 1336 |
| exptl transmission factor min | ? |
| exptl transmission factor max | ? |
| exptl crystal size max | 0.58 |
| exptl crystal size mid | 0.27 |
| exptl crystal size min | 0.06 |
| exptl absorption coefficient \( \mu \) | 2.070 |
| exptl absorption coefficient type | multi-scan |
| exptl absorption correction T min | 0.349 |
| exptl absorption correction T max | 1.000 |
| exptl absorption correction process details | ? |
| CrysalisPro 1.171.39.16b (Rigaku Oxford Diffraction, 2015) |

Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

| diffn source | 'micro-focus sealed X-ray tube' |
| diffn measurement device | 'four-circle diffractometer' |
| diffn measurement device type | 'XtaLAB Synergy, Dualflex, Pilatus 300K' |
| diffn measurement method | 'w scans' |
| diffn detector area resol mean | ? |
| diffn reflections number | 32030 |
Reflections were merged by SHELXL according to the crystal class for the calculation of statistics and refinement. _reflns_Friedel_fraction is defined as the number of unique Friedel pairs measured divided by the number that would be possible theoretically, ignoring centric projections and systematic absences.

_drawn_reflns_av_unetI/netI 0.0344
_drawn_reflns_av_R_equivalents 0.0511
_drawn_reflns_limit_h_min -17
_drawn_reflns_limit_h_max 13
_drawn_reflns_limit_k_min -11
_drawn_reflns_limit_k_max 12
_drawn_reflns_limit_l_min -28
_drawn_reflns_limit_l_max 30
_drawn_reflns_theta_min 3.808
_drawn_reflns_theta_max 78.856
_drawn_reflns_theta_full 67.684
_drawn_measured_fraction_theta_max 0.955
_drawn_measured_fraction_theta_full 1.000
_drawn_Laue_measured_fraction_max 0.955
_drawn_Laue_measured_fraction_full 1.000
_reflns_number_total 6328
_reflns_number_gt 5725
_reflns_threshold_expression 'I > 2\sigma(I)'
_reflns_Friedel_coverage 0.000
_reflns_Friedel_fraction_max .
_reflns_Friedel_fraction_full .

_reflns_special_details
;

_reflns_Friedel_fraction is defined as the number of unique Friedel pairs measured divided by the number that would be possible theoretically, ignoring centric projections and systematic absences.

_drawn_data_collection 'CrysAlisPro 1.171.39.16b (Rigaku OD, 2015)'
_drawn_cell_refinement 'CrysAlisPro 1.171.39.16b (Rigaku OD, 2015)'
_drawn_data_reduction 'CrysAlisPro 1.171.39.16b (Rigaku OD, 2015)'
_drawn_structure_solution 'SHELXS-2013/1 (Sheldrick, 2014)'
_drawn_structure_refinement 'SHELXL-2014/7 (Sheldrick, 2014)'
_drawn_molecular_graphics 'ORTEP3 for Windows'
_drawn_publication_material 'SHELXL-2014/7 and WINGX'
_refine_special_details

_atom_site_solution_primary difmap
_atom_site_solution_secondary difmap
_atom_site_solution_hydrogens mixed
_refine_hydrogen_treatment mixed
_refine_extinction_method none
_refine_goodness_of_fit_ref 1.062
_refine_goodness_of_fit_gt 1.062

_atom_site_label
_atom_site_type_symbol
_atom_site_fract_x
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.

| loop_ | geom_bond_atom_site_label_1 | geom_bond_atom_site_label_2 | geom_bond_distance | geom_bond_site_symmetry_2 | geom_bond_publ_flag |
|-------|-----------------------------|-----------------------------|--------------------|---------------------------|---------------------|
| S2A   | C2A                         | 1.695(2)                    | .                  |                           | .                   |
| O5A   | C5A                         | 1.226(3)                    | .                  |                           | .                   |
| N1A   | C2A                         | 1.333(3)                    | .                  |                           | .                   |
| N1A   | C11A                        | 1.420(3)                    | .                  |                           | .                   |
| N1A   | H1A                         | 0.89(3)                     | .                  |                           | .                   |
| N3A   | C2A                         | 1.349(3)                    | .                  |                           | .                   |
| N3A   | N4A                         | 1.392(2)                    | .                  |                           | .                   |
| N3A   | H3A                         | 0.83(3)                     | .                  |                           | .                   |
| N4A   | C5A                         | 1.353(3)                    | .                  |                           | .                   |
| N4A   | H4A                         | 0.78(3)                     | .                  |                           | .                   |
| N5A   | C53A                        | 1.338(3)                    | .                  |                           | .                   |
| N5A   | C55A                        | 1.338(3)                    | .                  |                           | .                   |
| F12A  | C12A                        | 1.358(4)                    | .                  |                           | .                   |
| S2B   | C2B                         | 1.506(3)                    | .                  |                           | .                   |
| O5B   | C5B                         | 1.226(3)                    | .                  |                           | .                   |
| N1B   | C2B                         | 1.333(3)                    | .                  |                           | .                   |
| N1B   | C11B                        | 1.420(3)                    | .                  |                           | .                   |
| N1B   | H1B                         | 0.81(3)                     | .                  |                           | .                   |
| N3B   | C2B                         | 1.353(3)                    | .                  |                           | .                   |
| N3B   | N4B                         | 1.390(2)                    | .                  |                           | .                   |
| N3B   | H3B                         | 0.82(3)                     | .                  |                           | .                   |
| N4B   | C5B                         | 1.352(3)                    | .                  |                           | .                   |
C15B H15B 0.9300 . ?
C16B H16B 0.9300 . ?
C51B C52B 1.393(3) . ?
C51B C56B 1.393(3) . ?
C52B C53B 1.385(3) . ?
C52B H52B 0.9300 . ?
C53B H53B 0.9300 . ?
C55B C56B 1.389(3) . ?
C55B H55B 0.9300 . ?
C6B H6B 0.9300 . ?
O2 C2 1.456(5) . ?
O2 H1 0.8200 . ?
C1 C2 1.493(5) . ?
C1 H11 0.9600 . ?
C1 H12 0.9600 . ?
C1 H13 0.9600 . ?
C2 C3 1.494(5) . ?
C2 H21 0.9800 . ?
C3 H31 0.9600 . ?
C3 H32 0.9600 . ?
C3 H33 0.9600 . ?

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C2A N1A H1A 117(2) . . ?
C11A N1A H1A 117(2) . . ?
C2A N3A N4A 121.12(19) . . ?
C2A N3A H3A 121(2) . . ?
N4A N3A H3A 115(2) . . ?
C5A N4A N3A 119.51(19) . . ?
C5A N4A H4A 126(2) . . ?
N3A N4A H4A 114(2) . . ?
C53A N54A C55A 117.19(19) . . ?
N1A C2A N3A 117.67(19) . . ?
N1A C2A S2A 124.03(17) . . ?
N3A C2A S2A 118.24(16) . . ?
O5A C5A N4A 122.1(2) . . ?
O5A C5A C51A 121.82(2) . . ?
N4A C5A C51A 116.11(19) . . ?
C16A C11A C12A 118.3(3) . . ?
C16A C11A N1A 123.4(3) . . ?
C12A C11A N1A 118.2(2) . . ?
F12A C12A C13A 118.5(3) . . ?
F12A C12A C11A 118.2(2) . . ?
C13A C12A C11A 123.3(3) . . ?
C14A C13A C12A 117.7(4) . . ?
C14A C13A C12A 118.1(4) . . ?
C12A C13A H13A 121.1 . . ?
C13A C14A C15A 122.1(3) . . ?
C13A C14A H14A 118.9 . . ?
C15A C14A H14A 118.9 . . ?
C14A C15A C16A 120.5(3) . . ?
C14A C15A H15A 119.8 . . ?
C16A C15A H15A 119.8 . . ?
C11A C16A C15A 118.0(4) . . ?
C11A C16A H16A 121.0 . . ?
C15A C16A H16A 121.0 . . ?
C52A C51A C56A 118.1(2) . . ?
C52A C51A C5A 117.3(2) . . ?
C56A C51A C5A 124.5(2) . . ?
C53A C52A C51A 119.2(2) . . ?
C53A C52A H52A 120.4 . . ?
C51A C52A H52A 120.4 . . ?
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  _geom_torsion_site_symmetry_3
  _geom_torsion_site_symmetry_4
  _geom_torsion_publ_flag
C2A N3A N4A C5A -87.1(3) . . . . ?
C11A N1A C2A N3A 172.9(2) . . . . ?
C11A N1A C2A S2A 9.9(3) . . . . ?
N4A N3A C2A N1A 172.9(2) . . . . ?
N3A N4A C5A 3.1(3) . . . . ?
N3A N4A C5A C51A -179.79(18) . . . . ?
C2A N1A C11A C16A 86.8(3) . . . . ?
C2A N1A C11A C12A 96.6(3) . . . . ?
N1A C11A C12A F12A 2.3(3) . . . . ?
C16A C11A C12A C13A -1.1(4) . . . . ?
C16A C11A C12A C13A 1.4(4) . . . . ?
N1A C11A C12A C13A -77.9(2) . . . . ?
F12A C12A C13A C14A -178.8(2) . . . . ?
C11A C12A C13A C14A 1.4(4) . . . . ?
C12A C13A C14A C15A -1.0(4) . . . . ?
C13A C14A C15A C16A 0.4(5) . . . . ?
C12A C11A C16A C15A 0.4(4) . . . . ?
N1A C11A C16A C15A 177.1(2) . . . . ?
C14A C15A C16A C11A -0.1(4) . . . . ?
O5A C5A C51A C52A 8.7(3) . . . . ?
N4A C5A C51A C52A -168.5(2) . . . . ?
O5A C5A C51A C56A -175.5(2) . . . . ?
N4A C5A C51A C56A 7.3(3) . . . . ?
C56A C51A C52A C53A -3.6(3) . . . . ?
C5A C51A C52A C53A 172.5(2) . . . . ?
C55A N54A C53A C52A 2.2(3) . . . . ?
C51A C52A C53A N54A 0.9(4) . . . . ?
C53A N54A C55A C56A -2.7(3) . . . . ?
N54A C55A C56A C51A 0.0(3) . . . . ?
C52A C51A C56A C55A 3.1(3) . . . . ?
C5A C51A C56A C55A -172.6(2) . . . . ?
C2B N3B N4B C5B 86.2(3) . . . . ?
C11B N1B C2B N3B -177.1(2) . . . . ?
C11B N1B C2B S2B 6.0(3) . . . . ?
N4B N3B C2B N1B 8.6(3) . . . . ?
N4B N3B C2B S2B -174.2(17) . . . . ?
N3B N4B C5B O5B -2.9(3) . . . . ?
N3B N4B C5B C51B 179.15(18) . . . . ?
C2B N1B C11B C16B -77.5(3) . . . . ?
C2B N1B C11B C12B 107.2(3) . . . . ?
C16B C11B C12B F12B -179.9(2) . . . . ?
N1B C11B C12B F12B -4.4(3) . . . . ?
C16B C11B C12B C13B -0.1(3) . . . . ?
N1B C11B C12B C13B 175.5(2) . . . . ?
F12B C12B C13B C14B 179.5(2) . . . . ?
C11B C12B C13B C14B -0.4(4) . . . . ?
C12B C13B C14B C15B 0.3(4) . . . . ?
C13B C14B C15B C16B 0.1(4) . . . . ?
C12B C11B C16B C15B 0.5(3) . . . . ?
N1B C11B C16B C15B -174.8(2) . . . . ?
C2B N3B N4B C5B 86.2(3) . . . . ?
C11B N1B C2B N3B -177.1(2) . . . . ?
C11B N1B C2B S2B 6.0(3) . . . . ?
N4B N3B C2B N1B 8.6(3) . . . . ?
N4B N3B C2B S2B -174.2(17) . . . . ?
N3B N4B C5B O5B -2.9(3) . . . . ?
N3B N4B C5B C51B 179.15(18) . . . . ?
C2B N1B C11B C16B -77.5(3) . . . . ?
C2B N1B C11B C12B 107.2(3) . . . . ?
C16B C11B C12B F12B -179.9(2) . . . . ?
N1B C11B C12B F12B -4.4(3) . . . . ?
C16B C11B C12B C13B -0.1(3) . . . . ?
N1B C11B C12B C13B 175.5(2) . . . . ?
F12B C12B C13B C14B 179.5(2) . . . . ?
C11B C12B C13B C14B -0.4(4) . . . . ?
C12B C13B C14B C15B 0.3(4) . . . . ?
C13B C14B C15B C16B 0.1(4) . . . . ?
C12B C11B C16B C15B 0.5(3) . . . . ?
N1B C11B C16B C15B -174.8(2) . . . . ?

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### Data Table

| Bond Target | X | Y | Z | U11 | U12 | U13 | U22 | U33 |
|-------------|---|---|---|-----|-----|-----|-----|-----|
| C14B - C1B  |  0.099188 |  0.261520 |  0.321691 |  0.01782 |  0.02035 = 0.03152 |
| C15B - C1B  |  0.297350 |  0.208086 |  0.219130 |  0.01635 |  0.05567 = 0.02470 |
| C16B - C1B  |  0.145073 | -0.000989 |  0.475073 |  0.01635 |  0.05567 = 0.02470 |
| C11B - C2B  |  0.624067 |  0.253940 |  0.161290 |  0.01591 |  0.03181 |
| C12B - C2B  |  0.137287 |  0.198723 |  0.412033 |  0.01591 |  0.03181 |
| C13B - C2B  |  0.163990 |  0.063061 |  0.419101 |  0.01591 |  0.03181 |
| C14B - C1B  |  0.099188 |  0.261520 |  0.321691 |  0.01782 |  0.02035 = 0.03152 |
| C15B - C1B  |  0.297350 |  0.208086 |  0.219130 |  0.01635 |  0.05567 = 0.02470 |
| C16B - C1B  |  0.145073 | -0.000989 |  0.475073 |  0.01635 |  0.05567 = 0.02470 |

### Additional Notes

- **_refine_diff_density_max:** 1.094
- **_refine_diff_density_min:** -0.587
- **_refine_diff_density_rms:** 0.075

- _shelx_res_file:

  shelx.res created by SHELXL-2014/7
| AFIX | MOLE | S2B   | O5B   | N1B   | H1B   | N3B   | H3B   | N4B   | H4B   | N54B  | F12B  | C2B   | C5B   | C11B  | C12B  | C13B  | AFIX  | H13B  | MOLE  | C14A  | C15A  | C16A  | C14B  | C15B  | C16B  |
|------|------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|------|-------|------|-------|-------|-------|-------|-------|-------|-------|
| 0    | 1    | 0.100249 | 0.073191 | 0.519197 | 11.00000 | 0.03364 | 0.09730 | 0.03994 | 0.01342 | 0.01435 | 0.01707 | 0.03207 | 0.01512 | 0.00409 | 0.00569 |
| 39   | 2    | -0.087874 | 0.032145 | 0.555256 | 11.00000 | -1.50000 |
| 0    | 1    | -0.072773 | 0.205659 | 0.515990 | 11.00000 | 0.03197 | 0.11503 | 0.02141 | 0.00167 | 0.00337 | -0.00033 | 0.02755 | -0.00283 | 0.00216 | -0.00087 |
| 39   | 2    | -0.041788 | 0.253300 | 0.548933 | 11.00000 | -1.50000 |
| 0    | 1    | -0.091297 | 0.272882 | 0.460471 | 11.00000 | 0.03047 | 0.08062 | 0.03027 | 0.00186 | 0.00198 | -0.00422 |
| 39   | 2    | -0.072790 | 0.363544 | 0.457656 | 11.00000 | -1.50000 |
| 0    | 1    | -0.072773 | 0.205659 | 0.515990 | 11.00000 | 0.03197 | 0.11503 | 0.02141 | 0.00167 | 0.00337 | -0.00033 | 0.02755 | -0.00283 | 0.00216 | -0.00087 |
| 39   | 2    | -0.041788 | 0.253300 | 0.548933 | 11.00000 | -1.50000 |
| 0    | 1    | -0.091297 | 0.272882 | 0.460471 | 11.00000 | 0.03047 | 0.08062 | 0.03027 | 0.00186 | 0.00198 | -0.00422 |
| 39   | 2    | -0.072790 | 0.363544 | 0.457656 | 11.00000 | -1.50000 |
| 0    | 1    | -0.072773 | 0.205659 | 0.515990 | 11.00000 | 0.03197 | 0.11503 | 0.02141 | 0.00167 | 0.00337 | -0.00033 | 0.02755 | -0.00283 | 0.00216 | -0.00087 |
| 39   | 2    | -0.041788 | 0.253300 | 0.548933 | 11.00000 | -1.50000 |
| 0    | 1    | -0.091297 | 0.272882 | 0.460471 | 11.00000 | 0.03047 | 0.08062 | 0.03027 | 0.00186 | 0.00198 | -0.00422 |
| 39   | 2    | -0.072790 | 0.363544 | 0.457656 | 11.00000 | -1.50000 |
| 0    | 1    | -0.072773 | 0.205659 | 0.515990 | 11.00000 | 0.03197 | 0.11503 | 0.02141 | 0.00167 | 0.00337 | -0.00033 | 0.02755 | -0.00283 | 0.00216 | -0.00087 |
| 39   | 2    | -0.041788 | 0.253300 | 0.548933 | 11.00000 | -1.50000 |
| 0    | 1    | -0.091297 | 0.272882 | 0.460471 | 11.00000 | 0.03047 | 0.08062 | 0.03027 | 0.00186 | 0.00198 | -0.00422 |
| 39   | 2    | -0.072790 | 0.363544 | 0.457656 | 11.00000 | -1.50000 |
| 0    | 1    | -0.072773 | 0.205659 | 0.515990 | 11.00000 | 0.03197 | 0.11503 | 0.02141 | 0.00167 | 0.00337 | -0.00033 | 0.02755 | -0.00283 | 0.00216 | -0.00087 |
| 39   | 2    | -0.041788 | 0.253300 | 0.548933 | 11.00000 | -1.50000 |
| 0    | 1    | -0.091297 | 0.272882 | 0.460471 | 11.00000 | 0.03047 | 0.08062 | 0.03027 | 0.00186 | 0.00198 | -0.00422 | 0.02775 | 0.00469 | -0.00013 |
REM Highest difference peak 1.094, deepest hole -0.587, 1-sigma level 0.075
Q1 1 0.1992 0.3737 0.5656 11.00000 0.05 1.09
Q2 1 0.1781 0.3975 0.5044 11.00000 0.05 0.78
Q3 1 0.0503 0.1309 0.0574 11.00000 0.05 0.56
Q4 1 -0.0578 0.4428 0.4350 11.00000 0.05 0.56
Q5 1 -0.0484 0.3066 0.5112 11.00000 0.05 0.56
Q6 1 0.2691 0.5047 0.4920 11.00000 0.05 0.52
;

CIF for 14
data_shelx

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_atom_type_scat_dispersion_imag
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0.0000
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'Cl'    'Cl'   0.3639
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'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'N'    'N'   0.0311
0.0180
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'O'    'O'   0.0492
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'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'S'    'S'   0.3331
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'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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_space_group_name_Hall         '-C 2 yc'

_shelx_space_group_comment
The symmetry employed for this shelxl refinement is uniquely defined by the following loop, which should always be used as a source of symmetry information in preference to the above space-group names. They are only intended as comments.

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'x+1/2, y+1/2, z'
'x+1/2, y+1/2, -z+1/2'
'x, -y, z'
'x, -y, z-1/2'
'x+1/2, -y+1/2, z'
'x+1/2, -y+1/2, z-1/2'

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_cell_angle_beta 103.383(2)
_cell_angle_gamma 90
_cell_volume 3066.26(12)
_cell_formula_units_Z 8
_cell_measurement_temperature 293(2)
_cell_measurement_reflns_used 237
_cell_measurement_theta_min 4.90
_cell_measurement_theta_max 77.50

_exptl_crystal_description prism
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_exptl_crystal_size_mid 0.16
_exptl_crystal_size_min 0.03
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_shelx_estimated_absorpt_T_min 0.3444
_shelx_estimated_absorpt_T_max 1.0000

CrysAlisPro 1.171.39.16b (Rigaku Oxford Diffraction, 2015)
Numerical absorption correction based on gaussian integration over a multifaceted crystal model
Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

_diffrn_measurement_device_type 'Rigaku Oxford Diffraction XtaLAB Synergy, Pilatus 300K diffractometer'
_diffrn_measurement_method 'w scans'
_diffrn_detector_area_resol_mean 8718
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_diffrn_reflns_av_R_equivalents 0.0376
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_diffrn_reflns_limit_k_max 11
_diffrn_reflns_limit_l_min -16
_diffrn_reflns_limit_l_max 6
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_diffrn_reflns_point_group_measured_fraction_full 0.996

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Reflections were merged by SHELXL according to the crystal class for the calculation of statistics and refinement.

_reflns_Friedel_fraction is defined as the number of unique Friedel pairs measured divided by the number that would be possible theoretically, ignoring centric projections and systematic absences.

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_atom_site_disorder_assembly
_atom_site_disorder_group
_atom_site_solution_primary
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difmap
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Cl12 Cl 0.38777(4) -0.05295(8) 0.27896(6) 0.0446(3) Uani 1 1 d . . . . .
S2 S 0.34476(3) 0.20423(7) 0.53257(5) 0.0390(2) Uani 1 1 d . . . . .
O5 O 0.21855(11) 0.0596(2) 0.24156(15) 0.0390(5) Uani 1 1 d . . . . .
N1 N 0.35812(11) 0.2259(3) 0.34078(17) 0.0298(5) Uani 1 1 d . . . . .
H1 H 0.3445(17) 0.239(4) 0.274(3) 0.045 Uiso 1 1 d . . . . .
N3 N 0.26808(11) 0.2635(3) 0.36561(17) 0.0320(5) Uani 1 1 d . . . . .
N3 H 0.2359(17) 0.266(4) 0.405(3) 0.048 Uiso 1 1 d . . . . .
N4 N 0.24621(11) 0.28063(3) 0.26136(17) 0.0287(5) Uani 1 1 d . . . . .
N4 H 0.2513(17) 0.361(5) 0.238(3) 0.043 Uiso 1 1 d . . . . .
N54 N 0.16221(10) 0.2328(3) -0.11929(17) 0.0286(5) Uani 1 1 d . . . . .
C2 C 0.32318(13) 0.2312(3) 0.4047(2) 0.0277(6) Uani 1 1 d . . . . .
C5 C 0.22070(12) 0.1735(3) 0.2050(2) 0.0271(6) Uani 1 1 d . . . . .
C11 C 0.41694(13) 0.1907(4) 0.3717(2) 0.0366(7) Uani 1 1 d . . . . .
C12 C 0.43618(13) 0.0646(4) 0.3455(2) 0.0394(8) Uani 1 1 d . . . . .
C13 C 0.49308(19) 0.0284(7) 0.3708(3) 0.0692(15) Uani 1 1 d D . . .
H13 H 0.5082 -0.0549 0.3559 0.104 Uiso 1 1 calc R U . A 1
C14A C 0.5260(8) 0.146(3) 0.426(2) 0.133(17) Uani 0.5 1 d D . P B 1
H14A H 0.5648 0.1260 0.4482 0.199 Uiso 0.5 1 calc R U P B 1
C15A C 0.5128(10) 0.275(2) 0.451(2) 0.089(8) Uani 0.5 1 d D . P B 1
H15A H 0.5383 0.3429 0.4815 0.134 Uiso 0.5 1 calc R U P B 1
C14B C 0.5365(5) 0.1026(15) 0.4235(11) 0.051(3) Uani 0.5 1 d D . P B 2
H14B H 0.5748 0.0769 0.4389 0.076 Uiso 0.5 1 calc R U P B 2
C15B C 0.5152(9) 0.2229(19) 0.4510(18) 0.075(7) Uani 0.5 1 d D . P B 2
H15B H 0.5542 0.2773 0.4941 0.112 Uiso 0.5 1 calc R U P B 2
C16 C 0.4569(2) 0.2834(6) 0.4249(3) 0.0697(14) Uani 1 1 d D . . . .
H16 H 0.4417 0.3638 0.4448 0.105 Uiso 1 1 calc R U . B 1
C51 C 0.19668(11) 0.2021(3) 0.0930(2) 0.0253(6) Uani 1 1 d . . .
C52 C 0.19583(11) 0.3309(3) 0.04790(19) 0.0251(5) Uani 1 1 d . . .
H52 H 0.2068 0.4087 0.0877 0.038 Uiso 1 1 calc R U . . .
C53 C 0.17829(11) 0.3409(3) 0.0578(2) 0.0275(6) Uani 1 1 d . . .
H53 H 0.1777 0.4272 -0.0875 0.041 Uiso 1 1 calc R U . . .
C55 C 0.16129(12) 0.1103(3) 0.0746(2) 0.0322(6) Uani 1 1 d . . .
H55 H 0.1490 0.0346 -0.1161 0.048 Uiso 1 1 calc R U . . .
C56 C 0.17771(13) 0.0904(3) 0.0298(2) 0.0305(6) Uani 1 1 d . . .
H56 H 0.1761 0.0904 0.0355 0.046 Uiso 1 1 calc R U . . .
O21 O 0.0690(4) 0.0851(8) 0.2142(7) 0.192(4) Uani 1 1 d D U .
N22 N 0.0261(5) 0.0762(9) 0.328(10) Uani 1 1 d D U .
C23 C 0.0508(7) 0.0533(10) 0.3844(10) 0.351(10) Uani 1 1 d D U .
H231 H 0.0917 0.0584 0.3982 0.421 Uiso 1 1 calc R U .
H232 H 0.0367 0.1221 0.4233 0.421 Uiso 1 1 calc R U .
H233 H 0.0397 -0.0359 0.4034 0.421 Uiso 1 1 calc R U .
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_atom_site_aniso_U_22
_atom_site_aniso_U_33
_atom_site_aniso_U_12
Atom Site Aniso

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 C2 1.696(3) . ?
O5 C5 1.220(3) . ?
N1 C2 1.335(4) . ?
N1 C11 1.422(4) . ?
N1 H1 0.89(4) . ?
N3 C2 1.346(4) . ?
N3 N4 1.388(3) . ?
N3 H3 1.03(4) . ?
N4 C5 1.351(4) . ?
N4 H4 0.86(4) . ?
N54 C55 1.339(4) . ?
N54 C53 1.339(4) . ?
C5 C51 1.508(4) . ?
C11 C12 1.388(5) . ?
C11 C16 1.391(5) . ?
C12 C13 1.378(5) . ?
C13 C14B 1.333(14) . ?
C13 C14A 1.49(3) . ?
C13 H13 0.9300 . ?
C14A C15A 1.36(3) . ?
C14A H14A 0.9300 . ?
C15A C16 1.31(2) . ?
C15A H15A 0.9300 . ?
C14B C15B 1.36(3) . ?
C14B H14B 0.9300 . ?
C15B C16 1.49(2) . ?
C15B H15B 0.9300 . ?
C16 H16 0.9300 . ?
C51 C56 1.390(4) . ?
C51 C52 1.392(4) . ?
C52 C53 1.388(4) . ?
C52 H52 0.9300 . ?
C53 H53 0.9300 . ?
C55 C56 1.381(4) . ?
C55 H55 0.9300 . ?
C56 H56 0.9300 . ?
O21 N22 1.430(9) . ?
N22 N22 1.262(2) 2 ?
N22 C23 1.507(9) . ?
C23 H231 0.9600 . ?
C23 H232 0.9600 . ?
C23 H233 0.9600 . ?
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  _geom_angle_atom_site_label_2
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  _geom_angle_site_symmetry_3
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C2 N1 C11 123.7(2) . . ?
C2 N1 H1 120(3) . . ?
C11 N1 H1 116(3) . . ?
C2 N3 N4 122.1(2) . . ?
C2 N3 H3 127(2) . . ?
N4 N3 H3 111(2) . . ?
C5 N4 N3 119.4(2) . . ?
C5 N4 H4 126(3) . . ?
N3 N4 H4 115(3) . . ?
C55 N54 C53 117.2(2) . . ?
N1 C2 N3 117.9(2) . . ?
N1 C2 S2 118.4(2) . . ?
N1 C2 S2 123.7(2) . . ?
N1 C2 N3 118.3(4) . . ?
C12 C11 N1 120.5(3) . . ?
C16 C11 N1 121.1(4) . . ?
C13 C12 C11 122.3(4) . . ?
C13 C12 C11 118.1(4) . . ?
C11 C12 C11 119.6(2) . . ?
C14B C13 C12 127.2(8) . . ?
C12 C13 C14A 108.2(8) . . ?
C12 C13 H13 125.9 . . ?
C14A C13 H13 125.9 . . ?
C15A C14A C13 135.0(16) . . ?
C15A C14A C13 112.5 . . ?
C13 C14A H14A 112.5 . . ?
C16 C15A C14A 106.0(17) . . ?
C16 C15A H15A 127.0 . . ?
C14A C15A H15A 127.0 . . ?
C13 C14B C15B 108.2(12) . . ?
C13 C14B H14B 125.9 . . ?
C15B C14B H14B 125.9 . . ?
C14B C15B C16 132.1(13) . . ?
C14B C15B H15B 114.0 . . ?
C16 C15B H15B 114.0 . . ?
C15A C16 C11 129.9(13) . . ?
C11 C16 C15B 111.5(8) . . ?
C15A C16 H16 115.1 . . ?
C11 C16 H16 115.1 . . ?
C56 C51 C52 118.1(2) . . ?
C56 C51 C52 117.4(2) . . ?
C52 C51 C5 124.4(2) . . ?
C53 C52 C5 118.6(2) . . ?
C53 C52 H52 120.7 . . ?
C51 C52 H52 120.7 . . ?
N54 C53 C52 123.5(3) . . ?
N54 C53 H53 118.2 . . ?
C52 C53 H53 118.2 . . ?
N54 C55 C56 123.4(3) . . ?
N54 C55 H55 118.3 . . ?
C56 C55 H55 118.3 . . ?
C55 C56 C51 119.1(3) . . ?
C55 C56 H56 120.4 . . ?
C51 C56 H56 120.4 . . ?
N22 C22 C23 126.0(17) 2 . . ?
N22 C22 O21 120.9(15) 2 . . ?
C23 C22 O21 112.7(11) . . ?
N22 C23 H231 109.5 . . ?
N22 C23 H232 109.5 . . ?
H231 C23 H232 109.5 . . ?
N22 C23 H233 109.5 . . ?
H231 C23 H233 109.5 . . ?
H232 C23 H233 109.5 . . ?

loop_
_geom_torsion_atom_site_label_1
_geom_torsion_atom_site_label_2
_geom_torsion_atom_site_label_3
_geom_torsion_atom_site_label_4
_geom_torsion_site_symmetry_1
_geom_torsion_site_symmetry_2
_geom_torsion_site_symmetry_3
_geom_torsion_site_symmetry_4
_geom_torsion_publ_flag
C2 N3 N4 C5 94.4(3) . . ?
C11 N1 C2 N3 -178.8(3) . . ?
C11 N1 C2 S2 3.1(4) . . . . ?
N4 N3 C2 N1 5.1(4) . . . . ?
N4 N3 C2 -176.7(2) . . . . ?
N3 N4 C5 O5 -3.6(4) . . . . ?
N3 N4 C5 C11 178.3(2) . . . . ?
C2 N1 C11 C12 107.2(3) . . . . ?
C2 N1 C11 -75.5(4) . . . . ?
C16 C11 C12 C13 0.3(5) . . . . ?
N1 C11 C12 C13 177.7(3) . . . . ?
C16 C11 C12 -179.8(3) . . . . ?
N1 C11 C12 -2.4(4) . . . . ?
C11 C12 C13 C14B 1.69(9) . . . . ?
C11 C12 C13 C14B -178.4(8) . . . . ?
C11 C12 C13 C14A -0.8(11) . . . . ?
C12 C11 C13 C14A 179.3(10) . . . . ?
C12 C13 C14A C15A -3(3) . . . . ?
C13 C14A C15A C16 6(4) . . . . ?
C12 C13 C14B C15B 1.0(17) . . . . ?
C13 C14B C15B C16 -6(3) . . . . ?
C14A C15A C16 C11 -7(3) . . . . ?
C12 C11 C16 C15A 4.2(15) . . . . ?
N1 C11 C16 C15A 173.1(14) . . . . ?
C12 C11 C16 C15B -3.9(11) . . . . ?
N1 C11 C16 C15B 178.7(10) . . . . ?
C14B C15B C16 C11 8(3) . . . . ?
O5 C5 C51 C52 7.3(4) . . . . ?
N4 C5 C51 C56 170.8(3) . . . . ?
O5 C5 C51 C52 177.2(3) . . . . ?
N4 C5 C51 C52 -4.7(4) . . . . ?
C56 C51 C52 C53 -2.5(4) . . . . ?
C5 C51 C52 C53 173.0(2) . . . . ?
C55 N54 C53 C52 2.6(4) . . . . ?
C51 C52 C53 N54 -0.2(4) . . . . ?
C53 N54 C55 C56 -2.2(4) . . . . ?
N54 C55 C56 C51 -0.5(4) . . . . ?
C52 C51 C56 C55 2.8(4) . . . . ?
C5 C51 C56 C55 173.0(3) . . . . ?

_refine_diff_density_max 0.618
_refine_diff_density_min -0.843
_refine_diff_density_rms 0.102

_shelx_res_file
;

shelx.res created by SHELXL-2014/7

TITL mm-39a in C2/c
CELL  1.54184  24.0692  9.7448  13.4379  90.000  103.383  90.000
ZERR  8.00  0.0005  0.0002  0.0003  0.000  0.002  0.000
LATT  7
SYMM -X  Y  1/2 -Z
SFAC C H CL N O S
UNIT 112 112 8 40 16 8
MERG  2
FMAP  2
PLAN  -5
ACTA
BOND SH
CONF
DFIX  1.400 0.01 N22 O21
DFIX  1.500 0.01 N22 C23
DFIX  1.400 0.05 C13 C14A C14A C15A C15A C16
DFIX  1.400 0.05 C13 C14B C14B C15B C15B C16
DELU  0.01 0.01 O21 N22
SIMU  0.01 0.01 N22 C23
L.S.  40
WGHT  0.111200 7.292500
FVAR  0.24752

47
H53   2    0.177665    0.427213    -0.087477    11.00000    -1.50000
AFIX  0
C55   1    0.161293    0.110284    -0.074624    11.00000    0.03910    0.02904 = 0.02410 -0.00486    0.00405 -0.00324
AFIX  43
H55   2    0.148967    0.034650    -0.116067    11.00000    -1.50000
AFIX  0
C56   1    0.177709    0.090445    0.029815    11.00000    0.03910    0.02904 = 0.02410 -0.00061    0.00705 -0.00324
AFIX  43
H56   2    0.176095    0.003513    0.057565    11.00000    -1.50000
AFIX  0
MOLE  2
O21   5    0.069044    0.085148    0.214245    11.00000    0.18953    0.13372 = 0.20302    0.04697 -0.05401 -0.02645
N22   4    0.026143    0.076461    0.271966    11.00000    0.17873    0.57893 = 0.18106 -0.00190 -0.05281 -0.09230
C23   1    0.050784    0.053144    0.384429    11.00000    0.20189    0.59128 = 0.21966 -0.01003 -0.03032 -0.07976
AFIX  33
H231  2    0.091684    0.058372    0.398210    11.00000    -1.20000
H232  2    0.036701    0.122133    0.423295    11.00000    -1.20000
H233  2    0.039660 -0.035922    0.403402    11.00000    -1.20000
AFIX  0
HKL   F
REM mm-39a in C2/c
REM R1 = 0.0661 for 2901 Fo > 4sig(Fo) and 0.0688 for all 3143 data
REM 235 parameters refined using 15 restraints
END
WGHT  0.1381    6.7709
REM Highest difference peak 0.618, deepest hole -0.843, 1-sigma level 0.102
Q1   1    0.5177    0.6229    0.2860    11.00000    0.05    0.62
Q2   1    0.4608    0.5533    0.2206    11.00000    0.05    0.62
Q3   1    0.0000    0.0120    0.2500    10.50000    0.05    0.50
Q4   1    0.4303    0.4588    0.3251    11.00000    0.05    0.46
Q5   1    0.4444    0.4258    0.4317    11.00000    0.05    0.33
;