Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
data_global

_journal_coden_Cambridge         1350
_journal_volume                  ?
_journal_page_first              ?
_journal_year                    ?

loop_
_publ_author_name
'Nather, Christian'
'Boeckmann, Jan'
'Evers, Nils'

_publ_contact_author_name        'Dr Christian Nather'
_publ_contact_author_email       cnaether@ac.uni-kiel.de

_publ_section_title
;
New Nickel(II) Thiocyanato Coordination Compounds:
Synthetic Aspects, Polymorphism, Thermal Reactivity and Magnetic Properties

;

# Attachment '- TA71.CIF'

data_ta71
_database_code_depnum_ccdc_archive 'CCDC 838535'
#TrackingRef '- TA71.CIF'

_audit_creation_method           SHELXL-97
_chemical_name_systematic
;
?
;
_chemical_name_common            ?
_chemical_melting_point          ?
_chemical_formula_moiety         'C16 H34 N10 Ni2 S4'
_chemical_formula_sum            'C16 H34 N10 Ni2 S4'
_chemical_formula_weight         612.19

loop_
_atom_type_symbol
_atom_type_description
_atom_type_scat_dispersion_real
_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'
S S 0.1246 0.1234 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
Ni Ni 0.3393 1.1124 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting           triclinic
_symmetry_space_group_name_H-M   'P -1'
_symmetry_space_group_name_Hall  '-P 1'

loop_
_symmetry_equiv_pos_as_xyz
'x, y, z'
'-x, -y, -z'

_cell_length_a                   8.2563(8)
_cell_length_b                   8.7222(9)
_cell_length_c                   9.3840(9)
_cell_angle_alpha                90.353(8)
_cell_angle_beta                 100.282(8)
_cell_angle_gamma                96.189(8)
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
_cell_volume                      660.82(11)
_cell_formula_units_Z             1
_cell_measurement_temperature     293(2)
_cell_measurement_reflns_used     7965
_cell_measurement_theta_min       2.21
_cell_measurement_theta_max       27.00

_exptl_crystal_description        block
_exptl_crystal_colour             blue
_exptl_crystal_size_max           0.13
_exptl_crystal_size_mid           0.09
_exptl_crystal_size_min           0.07
_exptl_crystal_density_meas       ?
_exptl_crystal_density_diffrn     1.538
_exptl_crystal_density_method     'not measured'
_exptl_crystal_F_000              320
_exptl_absorpt_coefficient_mu     1.766
_exptl_absorpt_correction_type    numerical
_exptl_absorpt_correction_T_min   0.823
_exptl_absorpt_correction_T_max   0.879
_exptl_absorpt_process_details    'X-Shape and X-Red32 (STOE, 2008)'

_exptl_special_details
;
?
;

_diffrn_ambient_temperature       293(2)
_diffrn_radiation_wavelength      0.71073
_diffrn_radiation_type            MoK\a
_diffrn_radiation_source          'fine-focus sealed tube'
_diffrn_radiation_monochromator   graphite
_diffrn_measurement_device_type   ?
_diffrn_measurement_method        ?
_diffrn_detector_area_resol_mean  ?
_diffrn_standards_number          ?
_diffrn_standards_interval_count  ?
_diffrn_standards_interval_time   ?
_diffrn_standards_decay_%         ?
_diffrn_reflns_number             7965
_diffrn_reflns_av_R_equivalents   0.0559
_diffrn_reflns_av_sigmaI/netI     0.0425
_diffrn_reflns_limit_h_min        -10
_diffrn_reflns_limit_h_max        10
_diffrn_reflns_limit_k_min        -11
_diffrn_reflns_limit_k_max        11
_diffrn_reflns_limit_l_min        -11
_diffrn_reflns_limit_l_max        11
_diffrn_reflns_theta_min          2.21
_diffrn_reflns_theta_max          27.00
_reflns_number_total              2878
_reflns_number_gt                 2519
_reflns_threshold_expression      >2sigma(I)

_computing_data_collection        ?
_computing_cell_refinement        ?
_computing_data_reduction         ?
_computing_structure_solution     'SHELXS-97 (Sheldrick, 1990)'
_computing_structure_refinement   'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics     ?
_computing_publication_material   ?

_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
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
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^)+(0.0512P)^2^+2.1941P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary     direct
_atom_sites_solution_secondary   difmap
_atom_sites_solution_hydrogens   geom
_refine_ls_hydrogen_treatment    constr
_refine_ls_extinction_method     none
_refine_ls_extinction_coef       ?
_refine_ls_number_reflns         2878
_refine_ls_number_parameters     145
_refine_ls_number_restraints     0
_refine_ls_R_factor_all          0.0778
_refine_ls_R_factor_gt           0.0686
_refine_ls_wR_factor_ref         0.1838
_refine_ls_wR_factor_gt          0.1792
_refine_ls_goodness_of_fit_ref   1.195
_refine_ls_restrained_S_all      1.195
_refine_ls_shift/su_max          0.000
_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_multiplicity
_atom_site_calc_flag
_atom_site_refinement_flags
_atom_site_disorder_assembly
_atom_site_disorder_group
Ni1 Ni 0.10031(10) 0.26414(8) 0.83401(8) 0.0447(2) Uani 1 1 d . . .
S1 S 0.3929(3) -0.1875(2) 0.8553(3) 0.0760(6) Uani 1 1 d . . .
C1 C 0.2863(8) -0.0414(6) 0.8517(6) 0.0485(13) Uani 1 1 d . . .
N1 N 0.2124(7) 0.0622(6) 0.8485(6) 0.0599(13) Uani 1 1 d . . .
S2 S 0.1828(3) 0.2907(2) 1.11887(19) 0.0675(5) Uani 1 1 d . . .
C2 C 0.0787(8) 0.4339(7) 1.1534(6) 0.0519(14) Uani 1 1 d . . .
N2 N 0.0097(7) 0.5348(6) 1.1780(6) 0.0576(13) Uani 1 1 d . . .
N11 N -0.1209(7) 0.1436(7) 0.8629(7) 0.0676(15) Uani 1 1 d . . .
H17A H -0.1018 0.0445 0.8751 0.081 Uiso 1 1 calc R . .
H17B H -0.1420 0.1779 0.9476 0.081 Uiso 1 1 calc R . .
C11 C -0.2696(11) 0.1426(13) 0.7610(13) 0.102(3) Uani 1 1 d . . .
H11A H -0.3082 0.2438 0.7606 0.123 Uiso 1 1 calc R . .
H11B H -0.3538 0.0689 0.7895 0.123 Uiso 1 1 calc R . .
C12 C -0.2461(13) 0.1014(13) 0.6128(14) 0.110(4) Uani 1 1 d . . .
H12A H -0.3541 0.0903 0.5504 0.132 Uiso 1 1 calc R . .
H12B H -0.2054 0.0011 0.6162 0.132 Uiso 1 1 calc R . .
C13 C -0.1315(12) 0.2102(10) 0.5414(9) 0.083(3) Uani 1 1 d . . .
H13A H -0.1414 0.1766 0.4412 0.099 Uiso 1 1 calc R . .
H13B H -0.1679 0.3122 0.5415 0.099 Uiso 1 1 calc R . .
N12 N 0.0439(8) 0.2214(8) 0.6101(6) 0.0689(16) Uani 1 1 d . . .
H18 H 0.0690 0.1232 0.6013 0.083 Uiso 1 1 calc R . .
C14 C 0.1496(15) 0.3075(11) 0.5237(9) 0.096(3) Uani 1 1 d . . .
H14A H 0.1175 0.4113 0.5144 0.116 Uiso 1 1 calc R . .
H14B H 0.1276 0.2604 0.4273 0.116 Uiso 1 1 calc R . .
C15 C 0.3271(13) 0.3191(12) 0.5768(10) 0.095(3) Uani 1 1 d . . .
H15A H 0.3834 0.3482 0.4971 0.113 Uiso 1 1 calc R . .
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
H15B H 0.3559 0.2178 0.6067 0.113 Uiso 1 1 calc R . .
C16 C 0.3905(13) 0.4284(10) 0.6974(13) 0.099(3) Uani 1 1 d . . .
H16A H 0.3567 0.5288 0.6699 0.119 Uiso 1 1 calc R . .
H16B H 0.5106 0.4378 0.7141 0.119 Uiso 1 1 calc R . .
N13 N 0.3342(7) 0.3859(7) 0.8354(7) 0.0667(15) Uani 1 1 d . . .
H19A H 0.3386 0.4739 0.8872 0.080 Uiso 1 1 calc R . .
H19B H 0.4097 0.3298 0.8850 0.080 Uiso 1 1 calc R . .

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
Ni1 0.0526(4) 0.0353(4) 0.0470(4) 0.0011(3) 0.0081(3) 0.0095(3)
S1 0.0750(12) 0.0512(10) 0.0995(15) -0.0100(9) -0.0008(10) 0.0246(9)
C1 0.053(3) 0.038(3) 0.053(3) 0.000(2) 0.006(3) 0.007(2)
N1 0.070(4) 0.049(3) 0.064(3) -0.002(2) 0.012(3) 0.017(3)
S2 0.0983(14) 0.0559(9) 0.0531(9) 0.0014(7) 0.0086(9) 0.0391(9)
C2 0.069(4) 0.044(3) 0.041(3) 0.004(2) 0.005(3) 0.008(3)
N2 0.072(4) 0.044(3) 0.058(3) 0.002(2) 0.006(3) 0.018(3)
N11 0.064(4) 0.058(3) 0.083(4) 0.011(3) 0.019(3) 0.010(3)
C11 0.059(5) 0.098(7) 0.135(9) 0.036(6) -0.007(5) -0.015(5)
C12 0.076(6) 0.095(7) 0.139(10) -0.029(7) -0.024(6) 0.002(5)
C13 0.107(7) 0.066(5) 0.064(4) -0.008(4) -0.017(4) 0.015(5)
N12 0.082(4) 0.083(4) 0.044(3) -0.001(3) 0.006(3) 0.029(3)
C14 0.152(10) 0.079(6) 0.060(5) 0.008(4) 0.023(5) 0.014(6)
C15 0.113(8) 0.105(7) 0.087(6) 0.019(5) 0.058(6) 0.042(6)
C16 0.099(7) 0.068(5) 0.145(9) -0.005(6) 0.068(7) -0.002(5)
N13 0.062(3) 0.055(3) 0.081(4) -0.002(3) 0.009(3) 0.000(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.
;

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
Ni1 N2 2.055(5) 2_567 ?
Ni1 N11 2.069(6) . ?
Ni1 N1 2.070(5) . ?
Ni1 N12 2.092(5) . ?
Ni1 N13 2.099(6) . ?
Ni1 S2 2.6404(19) . ?
S1 C1 1.622(6) . ?
C1 N1 1.142(8) . ?
S2 C2 1.653(7) . ?
C2 N2 1.142(8) . ?
N2 Ni1 2.055(5) 2_567 ?
N11 C11 1.414(11) . ?
C11 C12 1.485(15) . ?
C12 C13 1.511(14) . ?
C13 N12 1.468(11) . ?
N12 C14 1.450(11) . ?
C14 C15 1.453(14) . ?
C15 C16 1.459(14) . ?
C16 N13 1.488(11) . ?
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
loop_
_geom_angle_atom_site_label_1
_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
N2 Ni1 N11 89.6(2) 2_567 . ?
N2 Ni1 N1 179.4(2) 2_567 . ?
N11 Ni1 N1 90.5(2) . . ?
N2 Ni1 N12 93.6(2) 2_567 . ?
N11 Ni1 N12 91.7(3) . . ?
N1 Ni1 N12 87.0(2) . . ?
N2 Ni1 N13 91.5(2) 2_567 . ?
N11 Ni1 N13 172.2(2) . . ?
N1 Ni1 N13 88.3(2) . . ?
N12 Ni1 N13 95.9(3) . . ?
N2 Ni1 S2 92.14(16) 2_567 . ?
N11 Ni1 S2 87.75(19) . . ?
N1 Ni1 S2 87.24(16) . . ?
N12 Ni1 S2 174.24(18) . . ?
N13 Ni1 S2 84.51(18) . . ?
N1 C1 S1 179.3(6) . . ?
C1 N1 Ni1 173.5(6) . . ?
C2 S2 Ni1 101.2(2) . . ?
N2 C2 S2 178.6(7) . . ?
C2 N2 Ni1 163.7(5) . 2_567 ?
C11 N11 Ni1 123.0(5) . . ?
N11 C11 C12 111.6(9) . . ?
C11 C12 C13 118.0(8) . . ?
N12 C13 C12 114.9(7) . . ?
C14 N12 C13 111.9(7) . . ?
C14 N12 Ni1 116.6(6) . . ?
C13 N12 Ni1 117.2(5) . . ?
N12 C14 C15 117.0(7) . . ?
C14 C15 C16 115.8(8) . . ?
C15 C16 N13 114.6(7) . . ?
C16 N13 Ni1 120.8(6) . . ?

_diffrn_measured_fraction_theta_max 0.996
_diffrn_reflns_theta_full        27.00
_diffrn_measured_fraction_theta_full 0.996
_refine_diff_density_max         0.893
_refine_diff_density_min         -0.653
_refine_diff_density_rms         0.096

# Attachment '- TA153.CIF'

data_ta153
_database_code_depnum_ccdc_archive 'CCDC 838536'
#TrackingRef '- TA153.CIF'

_audit_creation_method           SHELXL-97
_chemical_name_systematic
;
?
;
_chemical_name_common            ?
_chemical_melting_point          ?
_chemical_formula_moiety         'C14 H34 N8 Ni S2'
_chemical_formula_sum            'C14 H34 N8 Ni S2'
_chemical_formula_weight         437.32

loop_
_atom_type_symbol
_atom_type_description
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
_atom_type_scat_dispersion_real
_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'
S S 0.1246 0.1234 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
Ni Ni 0.3393 1.1124 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting           monoclinic
_symmetry_space_group_name_H-M   'P 21/c'
_symmetry_space_group_name_Hall  '-P 2ybc'

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

_cell_length_a                   9.4274(6)
_cell_length_b                   18.1438(10)
_cell_length_c                   12.2373(7)
_cell_angle_alpha                90.00
_cell_angle_beta                 96.345(7)
_cell_angle_gamma                90.00
_cell_volume                     2080.4(2)
_cell_formula_units_Z            4
_cell_measurement_temperature    170(2)
_cell_measurement_reflns_used    12789
_cell_measurement_theta_min      2.83
_cell_measurement_theta_max      28.04

_exptl_crystal_description       block
_exptl_crystal_colour            blue
_exptl_crystal_size_max          0.85
_exptl_crystal_size_mid          0.62
_exptl_crystal_size_min          0.45
_exptl_crystal_density_meas      ?
_exptl_crystal_density_diffrn    1.396
_exptl_crystal_density_method    'not measured'
_exptl_crystal_F_000             936
_exptl_absorpt_coefficient_mu    1.148
_exptl_absorpt_correction_type   numerical
_exptl_absorpt_correction_T_min  0.425
_exptl_absorpt_correction_T_max  0.591
_exptl_absorpt_process_details   'X-Shape and X-Red32 (STOE, 2008)'

_exptl_special_details
;
?
;

_diffrn_ambient_temperature      170(2)
_diffrn_radiation_wavelength     0.71073
_diffrn_radiation_type           MoK\a
_diffrn_radiation_source         'fine-focus sealed tube'
_diffrn_radiation_monochromator  graphite
_diffrn_measurement_device_type  ?
_diffrn_measurement_method       ?
_diffrn_detector_area_resol_mean ?
_diffrn_standards_number         ?
_diffrn_standards_interval_count ?
_diffrn_standards_interval_time  ?
_diffrn_standards_decay_%        ?
_diffrn_reflns_number            12789
_diffrn_reflns_av_R_equivalents  0.0833
_diffrn_reflns_av_sigmaI/netI    0.0639
```

```
_diffrn_reflns_limit_h_min       -12
_diffrn_reflns_limit_h_max       9
_diffrn_reflns_limit_k_min       -23
_diffrn_reflns_limit_k_max       23
_diffrn_reflns_limit_l_min       -16
_diffrn_reflns_limit_l_max       16
_diffrn_reflns_theta_min         2.83
_diffrn_reflns_theta_max         28.04
_reflns_number_total             4928
_reflns_number_gt                3937
_reflns_threshold_expression     >2sigma(I)

_computing_data_collection       ?
_computing_cell_refinement       ?
_computing_data_reduction        ?
_computing_structure_solution    'SHELXS-97 (Sheldrick, 1990)'
_computing_structure_refinement  'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics    ?
_computing_publication_material  ?

_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^)+(0.0789P)^2^+0.0000P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary     direct
_atom_sites_solution_secondary   difmap
_atom_sites_solution_hydrogens   geom
_refine_ls_hydrogen_treatment    constr
_refine_ls_extinction_method     SHELXL
_refine_ls_extinction_coef       0.0147(17)
_refine_ls_extinction_expression Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^
_refine_ls_number_reflns         4928
_refine_ls_number_parameters     227
_refine_ls_number_restraints     0
_refine_ls_R_factor_all          0.0630
_refine_ls_R_factor_gt           0.0473
_refine_ls_wR_factor_ref         0.1285
_refine_ls_wR_factor_gt          0.1197
_refine_ls_goodness_of_fit_ref   1.030
_refine_ls_restrained_S_all      1.030
_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_symmetry_multiplicity
_atom_site_calc_flag
_atom_site_refinement_flags
_atom_site_disorder_assembly
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
_atom_site_disorder_group
Ni1 Ni 0.72387(3) 0.423133(16) 0.27838(2) 0.01148(12) Uani 1 1 d . . .
S1 S 1.13954(9) 0.28241(5) 0.44891(7) 0.0344(2) Uani 1 1 d . . .
C1 C 1.2083(4) 0.36538(17) 0.4473(2) 0.0281(6) Uani 1 1 d . . .
N1 N 1.2569(4) 0.42426(16) 0.4447(2) 0.0470(8) Uani 1 1 d . . .
S2 S 0.80309(11) 0.15317(5) 0.34935(7) 0.0419(2) Uani 1 1 d . . .
C2 C 0.7178(4) 0.0773(2) 0.3566(3) 0.0350(7) Uani 1 1 d . . .
N2 N 0.6698(5) 0.02053(15) 0.3590(3) 0.0680(14) Uani 1 1 d . . .
N11 N 0.6119(2) 0.52349(11) 0.31584(17) 0.0180(4) Uani 1 1 d . . .
H11A H 0.5164 0.5144 0.2967 0.022 Uiso 1 1 calc R . .
H11B H 0.6241 0.5282 0.3911 0.022 Uiso 1 1 calc R . .
N12 N 0.8908(3) 0.48407(12) 0.20783(17) 0.0208(5) Uani 1 1 d . . .
H12 H 0.9624 0.4916 0.2654 0.025 Uiso 1 1 calc R . .
N13 N 0.8350(2) 0.32334(12) 0.26061(17) 0.0182(4) Uani 1 1 d . . .
H13A H 0.8986 0.3178 0.3227 0.022 Uiso 1 1 calc R . .
H13B H 0.7696 0.2858 0.2613 0.022 Uiso 1 1 calc R . .
C11 C 0.6404(4) 0.59748(16) 0.2709(3) 0.0293(6) Uani 1 1 d . . .
H11C H 0.6116 0.6356 0.3219 0.035 Uiso 1 1 calc R . .
H11D H 0.5810 0.6039 0.1996 0.035 Uiso 1 1 calc R . .
C12 C 0.7959(4) 0.60901(16) 0.2542(3) 0.0325(7) Uani 1 1 d . . .
H12A H 0.8553 0.6001 0.3248 0.039 Uiso 1 1 calc R . .
H12B H 0.8096 0.6610 0.2333 0.039 Uiso 1 1 calc R . .
C13 C 0.8478(4) 0.55927(16) 0.1666(2) 0.0303(7) Uani 1 1 d . . .
H13C H 0.7708 0.5543 0.1051 0.036 Uiso 1 1 calc R . .
H13D H 0.9303 0.5830 0.1374 0.036 Uiso 1 1 calc R . .
C14 C 0.9609(3) 0.44562(18) 0.1205(2) 0.0265(6) Uani 1 1 d . . .
H14A H 1.0380 0.4775 0.0983 0.032 Uiso 1 1 calc R . .
H14B H 0.8898 0.4391 0.0553 0.032 Uiso 1 1 calc R . .
C15 C 1.0240(3) 0.37054(18) 0.1542(2) 0.0278(6) Uani 1 1 d . . .
H15A H 1.0877 0.3550 0.0993 0.033 Uiso 1 1 calc R . .
H15B H 1.0834 0.3758 0.2257 0.033 Uiso 1 1 calc R . .
C16 C 0.9156(3) 0.31052(17) 0.1645(2) 0.0257(6) Uani 1 1 d . . .
H16A H 0.8481 0.3088 0.0966 0.031 Uiso 1 1 calc R . .
H16B H 0.9649 0.2623 0.1728 0.031 Uiso 1 1 calc R . .
N21 N 0.8492(2) 0.43630(12) 0.43824(17) 0.0190(4) Uani 1 1 d . . .
H21A H 0.9430 0.4282 0.4275 0.023 Uiso 1 1 calc R . .
H21B H 0.8423 0.4851 0.4574 0.023 Uiso 1 1 calc R . .
N22 N 0.5528(2) 0.36145(11) 0.34657(17) 0.0164(4) Uani 1 1 d . . .
H22 H 0.4831 0.3965 0.3559 0.020 Uiso 1 1 calc R . .
N23 N 0.6019(2) 0.41988(12) 0.12223(17) 0.0191(4) Uani 1 1 d . . .
H23A H 0.5359 0.4574 0.1204 0.023 Uiso 1 1 calc R . .
H23B H 0.6623 0.4309 0.0704 0.023 Uiso 1 1 calc R . .
C21 C 0.8215(3) 0.39252(18) 0.5357(2) 0.0288(6) Uani 1 1 d . . .
H21C H 0.8643 0.4179 0.6030 0.035 Uiso 1 1 calc R . .
H21D H 0.8681 0.3438 0.5324 0.035 Uiso 1 1 calc R . .
C22 C 0.6630(3) 0.38148(17) 0.5426(2) 0.0269(6) Uani 1 1 d . . .
H22A H 0.6151 0.4300 0.5341 0.032 Uiso 1 1 calc R . .
H22B H 0.6501 0.3627 0.6168 0.032 Uiso 1 1 calc R . .
C23 C 0.5893(3) 0.32874(16) 0.4575(2) 0.0247(6) Uani 1 1 d . . .
H23C H 0.6523 0.2857 0.4507 0.030 Uiso 1 1 calc R . .
H23D H 0.5005 0.3105 0.4845 0.030 Uiso 1 1 calc R . .
C24 C 0.4779(3) 0.30363(15) 0.2754(2) 0.0234(6) Uani 1 1 d . . .
H24A H 0.4003 0.2826 0.3140 0.028 Uiso 1 1 calc R . .
H24B H 0.5462 0.2635 0.2651 0.028 Uiso 1 1 calc R . .
C25 C 0.4146(3) 0.33067(16) 0.1631(2) 0.0264(6) Uani 1 1 d . . .
H25A H 0.3539 0.3742 0.1733 0.032 Uiso 1 1 calc R . .
H25B H 0.3521 0.2916 0.1278 0.032 Uiso 1 1 calc R . .
C26 C 0.5240(3) 0.35161(16) 0.0860(2) 0.0249(6) Uani 1 1 d . . .
H26A H 0.5931 0.3108 0.0831 0.030 Uiso 1 1 calc R . .
H26B H 0.4750 0.3589 0.0110 0.030 Uiso 1 1 calc R . .

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
Ni1 0.00968(17) 0.01485(17) 0.01032(17) -0.00047(10) 0.00288(11) -0.00136(11)
S1 0.0264(4) 0.0417(4) 0.0345(4) 0.0076(3) 0.0013(3) -0.0004(3)
C1 0.0315(16) 0.0359(16) 0.0161(11) -0.0051(11) -0.0009(11) 0.0129(13)
N1 0.070(2) 0.0345(16) 0.0360(15) -0.0117(12) 0.0019(16) 0.0033(15)
S2 0.0481(6) 0.0344(4) 0.0412(4) 0.0077(3) -0.0051(4) -0.0039(4)
C2 0.0234(15) 0.055(2) 0.0256(14) -0.0016(13) -0.0036(13) 0.0106(14)
N2 0.079(3) 0.0174(13) 0.092(3) 0.0099(14) -0.060(2) -0.0125(14)
N11 0.0193(11) 0.0155(9) 0.0195(10) -0.0026(8) 0.0031(9) 0.0015(8)
N12 0.0183(11) 0.0297(12) 0.0149(9) -0.0007(8) 0.0036(9) -0.0089(9)
N13 0.0137(10) 0.0210(10) 0.0209(10) -0.0028(8) 0.0066(9) 0.0021(8)
C11 0.0349(17) 0.0195(12) 0.0327(14) 0.0047(11) 0.0003(13) -0.0005(12)
C12 0.0400(19) 0.0231(14) 0.0346(15) 0.0001(12) 0.0051(14) -0.0117(12)
C13 0.0412(18) 0.0258(14) 0.0254(13) 0.0055(11) 0.0106(13) -0.0132(13)
C14 0.0193(13) 0.0456(16) 0.0158(11) 0.0007(11) 0.0079(10) -0.0077(12)
C15 0.0135(12) 0.0524(18) 0.0192(12) -0.0036(12) 0.0096(11) 0.0024(12)
C16 0.0224(14) 0.0360(15) 0.0200(12) -0.0077(11) 0.0075(11) 0.0053(11)
N21 0.0142(10) 0.0288(11) 0.0136(9) -0.0015(8) 0.0000(8) 0.0014(9)
N22 0.0154(10) 0.0164(9) 0.0194(9) 0.0003(8) 0.0106(8) -0.0017(8)
N23 0.0165(11) 0.0265(11) 0.0135(9) -0.0007(8) -0.0014(8) -0.0042(8)
C21 0.0250(15) 0.0428(17) 0.0180(11) 0.0074(11) 0.0008(11) 0.0059(12)
C22 0.0262(15) 0.0404(16) 0.0159(11) 0.0043(11) 0.0100(11) 0.0016(12)
C23 0.0265(14) 0.0268(13) 0.0235(12) 0.0072(11) 0.0150(11) 0.0002(11)
C24 0.0168(13) 0.0210(12) 0.0338(14) -0.0027(11) 0.0089(11) -0.0065(10)
C25 0.0149(13) 0.0289(14) 0.0348(14) -0.0086(12) 0.0004(12) -0.0054(10)
C26 0.0232(14) 0.0295(14) 0.0209(12) -0.0114(10) -0.0025(11) -0.0030(11)

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

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
Ni1 N13 2.115(2) . ?
Ni1 N23 2.119(2) . ?
Ni1 N12 2.178(2) . ?
Ni1 N11 2.179(2) . ?
Ni1 N21 2.184(2) . ?
Ni1 N22 2.202(2) . ?
S1 C1 1.640(3) . ?
C1 N1 1.164(5) . ?
S2 C2 1.602(4) . ?
C2 N2 1.126(5) . ?
N11 C11 1.486(3) . ?
N12 C14 1.491(4) . ?
N12 C13 1.495(4) . ?
N13 C16 1.487(3) . ?
C11 C12 1.517(5) . ?
C12 C13 1.523(5) . ?
C14 C15 1.525(4) . ?
C15 C16 1.508(4) . ?
N21 C21 1.480(3) . ?
N22 C23 1.486(3) . ?
N22 C24 1.491(3) . ?
N23 C26 1.483(3) . ?
C21 C22 1.519(4) . ?
C22 C23 1.525(4) . ?
C24 C25 1.517(4) . ?
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
C25 C26 1.521(4) . ?

loop_
_geom_angle_atom_site_label_1
_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
N13 Ni1 N23 96.26(9) . . ?
N13 Ni1 N12 90.34(9) . . ?
N23 Ni1 N12 90.04(9) . . ?
N13 Ni1 N11 173.76(8) . . ?
N23 Ni1 N11 89.19(9) . . ?
N12 Ni1 N11 92.70(9) . . ?
N13 Ni1 N21 87.75(8) . . ?
N23 Ni1 N21 175.31(9) . . ?
N12 Ni1 N21 87.51(8) . . ?
N11 Ni1 N21 86.94(8) . . ?
N13 Ni1 N22 89.75(8) . . ?
N23 Ni1 N22 88.87(8) . . ?
N12 Ni1 N22 178.91(8) . . ?
N11 Ni1 N22 87.31(8) . . ?
N21 Ni1 N22 93.58(8) . . ?
N1 C1 S1 179.2(3) . . ?
N2 C2 S2 173.1(4) . . ?
C11 N11 Ni1 124.45(19) . . ?
C14 N12 C13 108.1(2) . . ?
C14 N12 Ni1 116.32(17) . . ?
C13 N12 Ni1 114.56(19) . . ?
C16 N13 Ni1 121.15(17) . . ?
N11 C11 C12 113.0(2) . . ?
C11 C12 C13 113.8(3) . . ?
N12 C13 C12 113.6(2) . . ?
N12 C14 C15 114.5(2) . . ?
C16 C15 C14 114.8(2) . . ?
N13 C16 C15 111.3(2) . . ?
C21 N21 Ni1 122.48(18) . . ?
C23 N22 C24 107.4(2) . . ?
C23 N22 Ni1 116.34(17) . . ?
C24 N22 Ni1 117.11(16) . . ?
C26 N23 Ni1 119.62(17) . . ?
N21 C21 C22 112.2(2) . . ?
C21 C22 C23 114.9(2) . . ?
N22 C23 C22 114.5(2) . . ?
N22 C24 C25 114.5(2) . . ?
C24 C25 C26 114.6(2) . . ?
N23 C26 C25 111.8(2) . . ?

_diffrn_measured_fraction_theta_max 0.977
_diffrn_reflns_theta_full        28.04
_diffrn_measured_fraction_theta_full 0.977
_refine_diff_density_max         0.840
_refine_diff_density_min         -1.168
_refine_diff_density_rms         0.100

# Attachment '- TA176TT.CIF'

data_ta176tt
_database_code_depnum_ccdc_archive 'CCDC 838537'
#TrackingRef '- TA176TT.CIF'

_audit_creation_method           SHELXL-97
_chemical_name_systematic
;
?
;
```

This journal is © The Royal Society of Chemistry 2011


```
_chemical_name_common            ?
_chemical_melting_point          ?
_chemical_formula_moiety         'C22 H53 N13 Ni2 O S4'
_chemical_formula_sum            'C22 H53 N13 Ni2 O S4'
_chemical_formula_weight         761.43

loop_
_atom_type_symbol
_atom_type_description
_atom_type_scat_dispersion_real
_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'
Ni Ni 0.3393 1.1124 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting           monoclinic
_symmetry_space_group_name_H-M   'C c'
_symmetry_space_group_name_Hall  'C -2yc'

loop_
_symmetry_equiv_pos_as_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                   9.1530(6)
_cell_length_b                   29.835(2)
_cell_length_c                   12.9934(7)
_cell_angle_alpha                90.00
_cell_angle_beta                 97.272(7)
_cell_angle_gamma                90.00
_cell_volume                     3519.7(4)
_cell_formula_units_Z            4
_cell_measurement_temperature    200(2)
_cell_measurement_reflns_used    8474
_cell_measurement_theta_min      2.34
_cell_measurement_theta_max      25.00

_exptl_crystal_description       block
_exptl_crystal_colour            blue
_exptl_crystal_size_max          0.35
_exptl_crystal_size_mid          0.28
_exptl_crystal_size_min          0.25
_exptl_crystal_density_meas      ?
_exptl_crystal_density_diffrn    1.437
_exptl_crystal_density_method    'not measured'
_exptl_crystal_F_000             1616
_exptl_absorpt_coefficient_mu    1.346
_exptl_absorpt_correction_type   numerical
_exptl_absorpt_correction_T_min  0.638
_exptl_absorpt_correction_T_max  0.709
_exptl_absorpt_process_details   'X-Shape and X-Red32 (STOE, 2008)'

_exptl_special_details
;
?
;

_diffrn_ambient_temperature      200(2)
_diffrn_radiation_wavelength     0.71073
_diffrn_radiation_type           MoK\a
_diffrn_radiation_source         'fine-focus sealed tube'
_diffrn_radiation_monochromator  graphite
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
_diffrn_measurement_device_type  ?
_diffrn_measurement_method       ?
_diffrn_detector_area_resol_mean ?
_diffrn_standards_number         ?
_diffrn_standards_interval_count ?
_diffrn_standards_interval_time  ?
_diffrn_standards_decay_%        ?
_diffrn_reflns_number            8474
_diffrn_reflns_av_R_equivalents  0.1100
_diffrn_reflns_av_sigmaI/netI    0.1335
_diffrn_reflns_limit_h_min       -10
_diffrn_reflns_limit_h_max       10
_diffrn_reflns_limit_k_min       -35
_diffrn_reflns_limit_k_max       35
_diffrn_reflns_limit_l_min       -11
_diffrn_reflns_limit_l_max       15
_diffrn_reflns_theta_min         2.34
_diffrn_reflns_theta_max         25.00
_reflns_number_total             5459
_reflns_number_gt                3631
_reflns_threshold_expression     >2sigma(I)

_computing_data_collection       ?
_computing_cell_refinement       ?
_computing_data_reduction        ?
_computing_structure_solution    'SHELXS-97 (Sheldrick, 1990)'
_computing_structure_refinement  'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics    ?
_computing_publication_material  ?

_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^)+(0.0503P)^2^+0.0000P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary     direct
_atom_sites_solution_secondary   difmap
_atom_sites_solution_hydrogens   geom
_refine_ls_hydrogen_treatment    constr
_refine_ls_extinction_method     none
_refine_ls_extinction_coef       ?
_refine_ls_abs_structure_details 'Flack H D (1983), Acta Cryst. A39, 876-881'
_refine_ls_abs_structure_Flack   0.01(3)
_refine_ls_number_reflns         5459
_refine_ls_number_parameters     379
_refine_ls_number_restraints     2
_refine_ls_R_factor_all          0.1115
_refine_ls_R_factor_gt           0.0657
_refine_ls_wR_factor_ref         0.1366
_refine_ls_wR_factor_gt          0.1200
_refine_ls_goodness_of_fit_ref   0.979
_refine_ls_restrained_S_all      0.979
_refine_ls_shift/su_max          0.001
_refine_ls_shift/su_mean         0.000

loop_
_atom_site_label
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
_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_multiplicity
_atom_site_calc_flag
_atom_site_refinement_flags
_atom_site_disorder_assembly
_atom_site_disorder_group
Ni1 Ni 0.28905(11) 0.83345(4) 0.16584(9) 0.0271(3) Uani 1 1 d . . .
Ni2 Ni 0.75020(13) 0.94177(4) 0.58497(11) 0.0323(3) Uani 1 1 d . . .
N1 N 0.428(2) 0.8402(4) -0.2767(10) 0.091(4) Uani 1 1 d . . .
C1 C 0.3180(13) 0.8350(4) -0.2628(9) 0.040(3) Uani 1 1 d . . .
S1 S 0.1497(5) 0.82838(14) -0.2354(3) 0.0749(11) Uani 1 1 d . . .
N2 N 0.5630(10) 0.9570(3) 0.4797(8) 0.048(2) Uani 1 1 d . . .
C2 C 0.4735(11) 0.9679(3) 0.4159(10) 0.041(3) Uani 1 1 d . . .
S2 S 0.3449(3) 0.98411(10) 0.3229(2) 0.0469(8) Uani 1 1 d . . .
N3 N 0.7375(10) 0.8723(3) 0.5441(8) 0.049(2) Uani 1 1 d . . .
C3 C 0.7306(11) 0.8412(4) 0.4873(9) 0.039(3) Uani 1 1 d . . .
S3 S 0.7229(3) 0.79827(9) 0.4097(2) 0.0395(7) Uani 1 1 d . . .
N4 N 0.7739(9) 1.0097(3) 0.6223(7) 0.040(2) Uani 1 1 d . . .
C4 C 0.7734(11) 1.0451(3) 0.6584(9) 0.034(2) Uani 1 1 d . . .
S4 S 0.7733(3) 1.09600(9) 0.7060(3) 0.0455(7) Uani 1 1 d . . .
N11 N 0.4315(9) 0.7758(3) 0.1776(8) 0.040(2) Uani 1 1 d . . .
H11C H 0.4972 0.7793 0.2368 0.048 Uiso 1 1 calc R . .
H11D H 0.3742 0.7513 0.1883 0.048 Uiso 1 1 calc R . .
N12 N 0.4341(9) 0.8688(3) 0.0694(7) 0.033(2) Uani 1 1 d . . .
H12 H 0.5055 0.8823 0.1167 0.040 Uiso 1 1 calc R . .
N13 N 0.1504(8) 0.8908(3) 0.1705(6) 0.0313(19) Uani 1 1 d . . .
H13A H 0.0591 0.8809 0.1830 0.038 Uiso 1 1 calc R . .
H13B H 0.1879 0.9079 0.2266 0.038 Uiso 1 1 calc R . .
C11 C 0.5196(12) 0.7639(4) 0.0913(9) 0.043(3) Uani 1 1 d . . .
H11A H 0.4526 0.7534 0.0303 0.051 Uiso 1 1 calc R . .
H11B H 0.5889 0.7393 0.1140 0.051 Uiso 1 1 calc R . .
C12 C 0.6054(12) 0.8047(3) 0.0614(10) 0.044(3) Uani 1 1 d . . .
H12A H 0.6589 0.8177 0.1255 0.053 Uiso 1 1 calc R . .
H12B H 0.6800 0.7945 0.0178 0.053 Uiso 1 1 calc R . .
C13 C 0.5171(13) 0.8408(4) 0.0050(9) 0.047(3) Uani 1 1 d . . .
H13C H 0.5841 0.8602 -0.0292 0.057 Uiso 1 1 calc R . .
H13D H 0.4470 0.8269 -0.0502 0.057 Uiso 1 1 calc R . .
C14 C 0.3683(13) 0.9062(4) 0.0036(10) 0.050(3) Uani 1 1 d . . .
H14A H 0.3072 0.8931 -0.0573 0.060 Uiso 1 1 calc R . .
H14B H 0.4493 0.9230 -0.0225 0.060 Uiso 1 1 calc R . .
C15 C 0.2750(12) 0.9391(4) 0.0539(9) 0.046(3) Uani 1 1 d . . .
H15A H 0.2542 0.9651 0.0070 0.055 Uiso 1 1 calc R . .
H15B H 0.3320 0.9501 0.1188 0.055 Uiso 1 1 calc R . .
C16 C 0.1297(11) 0.9201(3) 0.0793(9) 0.040(3) Uani 1 1 d . . .
H16A H 0.0632 0.9450 0.0923 0.049 Uiso 1 1 calc R . .
H16B H 0.0822 0.9029 0.0190 0.049 Uiso 1 1 calc R . .
N21 N 0.4185(8) 0.8603(3) 0.3017(6) 0.0330(19) Uani 1 1 d . . .
H1A H 0.5041 0.8438 0.3122 0.040 Uiso 1 1 calc R . .
H1B H 0.4448 0.8890 0.2858 0.040 Uiso 1 1 calc R . .
N22 N 0.1481(8) 0.7952(2) 0.2579(6) 0.0303(18) Uani 1 1 d . . .
H2 H 0.1922 0.7671 0.2652 0.036 Uiso 1 1 calc R . .
N23 N 0.1705(8) 0.8074(3) 0.0286(7) 0.036(2) Uani 1 1 d . . .
H3A H 0.1859 0.8267 -0.0244 0.044 Uiso 1 1 calc R . .
H3B H 0.2140 0.7807 0.0151 0.044 Uiso 1 1 calc R . .
C21 C 0.3588(11) 0.8625(4) 0.4033(8) 0.040(2) Uani 1 1 d . . .
H1C H 0.2837 0.8865 0.4009 0.048 Uiso 1 1 calc R . .
H1D H 0.4392 0.8699 0.4590 0.048 Uiso 1 1 calc R . .
C22 C 0.2908(12) 0.8185(4) 0.4272(8) 0.044(3) Uani 1 1 d . . .
H2A H 0.2815 0.8175 0.5022 0.053 Uiso 1 1 calc R . .
H2B H 0.3579 0.7940 0.4122 0.053 Uiso 1 1 calc R . .
C23 C 0.1392(12) 0.8101(4) 0.3660(8) 0.043(3) Uani 1 1 d . . .
H3C H 0.0873 0.7869 0.4021 0.052 Uiso 1 1 calc R . .
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
H3D H 0.0805 0.8380 0.3644 0.052 Uiso 1 1 calc R . .
C24 C -0.0068(10) 0.7857(3) 0.2115(9) 0.036(3) Uani 1 1 d . . .
H4A H -0.0637 0.8140 0.2083 0.043 Uiso 1 1 calc R . .
H4B H -0.0521 0.7648 0.2576 0.043 Uiso 1 1 calc R . .
C25 C -0.0185(12) 0.7657(4) 0.1043(10) 0.046(3) Uani 1 1 d . . .
H5A H -0.1188 0.7533 0.0862 0.056 Uiso 1 1 calc R . .
H5B H 0.0522 0.7406 0.1049 0.056 Uiso 1 1 calc R . .
C26 C 0.0121(11) 0.7993(4) 0.0199(9) 0.041(3) Uani 1 1 d . . .
H6A H -0.0236 0.7871 -0.0495 0.050 Uiso 1 1 calc R . .
H6B H -0.0405 0.8278 0.0289 0.050 Uiso 1 1 calc R . .
N31 N 0.8642(10) 0.9505(3) 0.4567(8) 0.048(2) Uani 1 1 d . . .
H21C H 0.8391 0.9265 0.4137 0.058 Uiso 1 1 calc R . .
H21D H 0.8237 0.9754 0.4224 0.058 Uiso 1 1 calc R . .
N32 N 0.9433(8) 0.9272(3) 0.6890(6) 0.0292(19) Uani 1 1 d . . .
H22 H 0.9506 0.8961 0.6884 0.035 Uiso 1 1 calc R . .
N33 N 0.6116(9) 0.9324(3) 0.6972(7) 0.040(2) Uani 1 1 d . . .
H23A H 0.5284 0.9493 0.6785 0.048 Uiso 1 1 calc R . .
H23B H 0.5828 0.9028 0.6945 0.048 Uiso 1 1 calc R . .
C31 C 1.0209(13) 0.9554(5) 0.4611(11) 0.063(4) Uani 1 1 d . . .
H21A H 1.0468 0.9876 0.4688 0.075 Uiso 1 1 calc R . .
H21B H 1.0520 0.9448 0.3950 0.075 Uiso 1 1 calc R . .
C32 C 1.1035(12) 0.9296(4) 0.5499(9) 0.048(3) Uani 1 1 d . . .
H22A H 1.0752 0.8976 0.5414 0.058 Uiso 1 1 calc R . .
H22B H 1.2099 0.9316 0.5435 0.058 Uiso 1 1 calc R . .
C33 C 1.0826(10) 0.9431(4) 0.6549(9) 0.041(2) Uani 1 1 d . . .
H23C H 1.0852 0.9763 0.6588 0.050 Uiso 1 1 calc R . .
H23D H 1.1660 0.9316 0.7036 0.050 Uiso 1 1 calc R . .
C34 C 0.9407(11) 0.9386(4) 0.7970(8) 0.043(3) Uani 1 1 d . . .
H24A H 0.9430 0.9717 0.8038 0.051 Uiso 1 1 calc R . .
H24B H 1.0311 0.9267 0.8377 0.051 Uiso 1 1 calc R . .
C35 C 0.8050(12) 0.9205(4) 0.8448(9) 0.050(3) Uani 1 1 d . . .
H25A H 0.7947 0.8881 0.8297 0.059 Uiso 1 1 calc R . .
H25B H 0.8235 0.9240 0.9211 0.059 Uiso 1 1 calc R . .
C36 C 0.6652(13) 0.9430(5) 0.8067(9) 0.055(3) Uani 1 1 d . . .
H26A H 0.6788 0.9758 0.8139 0.066 Uiso 1 1 calc R . .
H26B H 0.5894 0.9339 0.8506 0.066 Uiso 1 1 calc R . .
O1W O 0.2407(12) 0.6940(3) 0.2439(10) 0.094(4) Uani 1 1 d . . .
H1O H 0.1637 0.6789 0.2282 0.142 Uiso 1 1 d R . .
H2O H 0.3073 0.6766 0.2701 0.142 Uiso 1 1 d R . .

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
Ni1 0.0289(6) 0.0282(6) 0.0245(7) 0.0016(6) 0.0049(5) 0.0002(6)
Ni2 0.0333(6) 0.0307(7) 0.0316(8) -0.0027(6) -0.0007(5) -0.0035(6)
N1 0.166(14) 0.064(8) 0.045(8) -0.013(6) 0.026(9) -0.009(10)
C1 0.052(6) 0.026(5) 0.041(7) -0.008(5) -0.001(5) -0.006(5)
S1 0.093(3) 0.073(2) 0.056(2) 0.005(2) -0.002(2) 0.004(2)
N2 0.045(5) 0.052(6) 0.044(7) 0.005(5) -0.010(5) 0.001(5)
C2 0.029(5) 0.039(6) 0.055(8) -0.002(5) 0.004(5) -0.004(5)
S2 0.0467(16) 0.0478(17) 0.0424(19) -0.0013(14) -0.0091(13) 0.0002(13)
N3 0.053(6) 0.037(5) 0.055(7) -0.009(5) 0.001(5) -0.011(4)
C3 0.036(5) 0.040(6) 0.041(7) 0.003(5) 0.002(5) -0.001(5)
S3 0.0441(15) 0.0353(14) 0.0385(17) -0.0025(13) 0.0027(13) 0.0062(12)
N4 0.040(5) 0.034(5) 0.047(6) 0.002(4) 0.003(4) -0.003(4)
C4 0.028(5) 0.038(6) 0.037(6) 0.011(5) 0.007(4) 0.008(5)
S4 0.0427(14) 0.0341(14) 0.058(2) -0.0025(13) 0.0018(13) 0.0034(12)
N11 0.039(5) 0.028(5) 0.055(7) 0.007(4) 0.010(4) 0.001(4)
N12 0.038(4) 0.037(5) 0.026(5) -0.005(4) 0.010(4) -0.009(4)
N13 0.032(4) 0.036(5) 0.025(5) 0.006(4) -0.004(3) 0.002(3)
C11 0.038(6) 0.050(7) 0.044(7) -0.011(6) 0.021(5) 0.011(5)
C12 0.038(6) 0.035(6) 0.064(9) -0.007(6) 0.023(6) -0.010(5)
C13 0.052(6) 0.061(7) 0.034(6) 0.005(6) 0.023(5) 0.021(6)
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
C14 0.060(7) 0.046(7) 0.045(8) 0.018(6) 0.014(6) 0.006(6)
C15 0.056(7) 0.041(6) 0.041(7) 0.008(6) 0.008(5) 0.006(5)
C16 0.054(6) 0.037(6) 0.031(6) 0.017(5) 0.008(5) 0.013(5)
N21 0.035(4) 0.034(4) 0.031(5) -0.006(4) 0.006(4) -0.005(4)
N22 0.034(4) 0.029(4) 0.030(5) 0.008(4) 0.011(4) -0.002(3)
N23 0.032(4) 0.041(5) 0.036(5) 0.000(4) -0.001(4) 0.004(4)
C21 0.043(6) 0.046(6) 0.031(6) 0.001(5) 0.003(5) -0.007(5)
C22 0.049(6) 0.063(7) 0.018(6) 0.008(5) -0.004(5) 0.002(6)
C23 0.056(7) 0.053(7) 0.023(6) 0.005(5) 0.016(5) -0.007(5)
C24 0.029(5) 0.032(5) 0.051(7) 0.000(5) 0.019(5) -0.006(4)
C25 0.041(6) 0.049(7) 0.049(8) 0.000(6) 0.005(5) -0.008(5)
C26 0.041(6) 0.046(6) 0.036(7) -0.006(5) 0.001(5) -0.015(5)
N31 0.057(6) 0.051(6) 0.033(6) 0.001(5) -0.004(4) -0.001(5)
N32 0.033(4) 0.027(4) 0.028(5) -0.005(4) 0.003(4) -0.003(3)
N33 0.035(5) 0.040(5) 0.049(6) 0.012(4) 0.017(4) -0.003(4)
C31 0.061(8) 0.083(10) 0.051(8) -0.001(7) 0.035(7) -0.012(7)
C32 0.044(6) 0.056(7) 0.047(8) 0.005(6) 0.016(6) -0.003(5)
C33 0.025(5) 0.051(6) 0.049(7) -0.003(6) 0.007(4) -0.007(5)
C34 0.052(6) 0.050(6) 0.024(6) -0.002(5) -0.004(5) -0.003(5)
C35 0.063(7) 0.061(7) 0.028(6) 0.003(6) 0.017(5) 0.001(6)
C36 0.056(7) 0.073(8) 0.041(7) -0.008(7) 0.022(6) -0.014(7)
O1W 0.099(8) 0.057(6) 0.129(11) 0.014(6) 0.020(8) -0.002(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
Ni1 N23 2.114(8) . ?
Ni1 N13 2.135(8) . ?
Ni1 N11 2.152(8) . ?
Ni1 N21 2.152(8) . ?
Ni1 N22 2.189(7) . ?
Ni1 N12 2.207(8) . ?
Ni2 N33 2.070(8) . ?
Ni2 N4 2.089(9) . ?
Ni2 N31 2.092(10) . ?
Ni2 N2 2.103(10) . ?
Ni2 N32 2.130(8) . ?
Ni2 N3 2.140(9) . ?
N1 C1 1.052(16) . ?
C1 S1 1.636(14) . ?
N2 C2 1.137(13) . ?
C2 S2 1.649(12) . ?
N3 C3 1.182(13) . ?
C3 S3 1.626(11) . ?
N4 C4 1.156(13) . ?
C4 S4 1.640(11) . ?
N11 C11 1.504(13) . ?
N12 C13 1.461(13) . ?
N12 C14 1.486(13) . ?
N13 C16 1.466(12) . ?
C11 C12 1.525(14) . ?
C12 C13 1.482(16) . ?
C14 C15 1.503(15) . ?
C15 C16 1.520(15) . ?
N21 C21 1.493(13) . ?
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
N22 C23 1.485(13) . ?
N22 C24 1.496(12) . ?
N23 C26 1.460(12) . ?
C21 C22 1.500(14) . ?
C22 C23 1.531(15) . ?
C24 C25 1.507(16) . ?
C25 C26 1.537(16) . ?
N31 C31 1.436(15) . ?
N32 C34 1.446(12) . ?
N32 C33 1.480(12) . ?
N33 C36 1.478(15) . ?
C31 C32 1.509(17) . ?
C32 C33 1.458(15) . ?
C34 C35 1.554(15) . ?
C35 C36 1.473(17) . ?

loop_
_geom_angle_atom_site_label_1
_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
N23 Ni1 N13 94.3(3) . . ?
N23 Ni1 N11 90.5(3) . . ?
N13 Ni1 N11 174.3(4) . . ?
N23 Ni1 N21 177.3(3) . . ?
N13 Ni1 N21 86.9(3) . . ?
N11 Ni1 N21 88.4(3) . . ?
N23 Ni1 N22 89.7(3) . . ?
N13 Ni1 N22 90.5(3) . . ?
N11 Ni1 N22 86.4(3) . . ?
N21 Ni1 N22 92.7(3) . . ?
N23 Ni1 N12 88.7(3) . . ?
N13 Ni1 N12 92.0(3) . . ?
N11 Ni1 N12 91.1(3) . . ?
N21 Ni1 N12 88.8(3) . . ?
N22 Ni1 N12 177.1(3) . . ?
N33 Ni2 N4 91.3(4) . . ?
N33 Ni2 N31 172.1(4) . . ?
N4 Ni2 N31 90.9(4) . . ?
N33 Ni2 N2 88.1(4) . . ?
N4 Ni2 N2 89.6(4) . . ?
N31 Ni2 N2 84.4(4) . . ?
N33 Ni2 N32 93.4(3) . . ?
N4 Ni2 N32 89.8(3) . . ?
N31 Ni2 N32 94.2(3) . . ?
N2 Ni2 N32 178.5(4) . . ?
N33 Ni2 N3 91.4(4) . . ?
N4 Ni2 N3 176.9(4) . . ?
N31 Ni2 N3 86.6(4) . . ?
N2 Ni2 N3 92.0(4) . . ?
N32 Ni2 N3 88.5(3) . . ?
N1 C1 S1 177.0(14) . . ?
C2 N2 Ni2 171.6(10) . . ?
N2 C2 S2 179.4(12) . . ?
C3 N3 Ni2 155.7(9) . . ?
N3 C3 S3 179.3(11) . . ?
C4 N4 Ni2 167.2(9) . . ?
N4 C4 S4 178.2(11) . . ?
C11 N11 Ni1 120.8(7) . . ?
C13 N12 C14 107.6(8) . . ?
C13 N12 Ni1 116.4(7) . . ?
C14 N12 Ni1 117.1(6) . . ?
C16 N13 Ni1 118.1(6) . . ?
N11 C11 C12 110.1(8) . . ?
C13 C12 C11 116.0(9) . . ?
```

```
N12 C13 C12 115.0(10) . . ?
N12 C14 C15 116.8(10) . . ?
C14 C15 C16 114.3(10) . . ?
N13 C16 C15 111.8(9) . . ?
C21 N21 Ni1 121.1(6) . . ?
C23 N22 C24 105.9(7) . . ?
C23 N22 Ni1 117.8(6) . . ?
C24 N22 Ni1 118.2(6) . . ?
C26 N23 Ni1 121.6(7) . . ?
N21 C21 C22 110.6(9) . . ?
C21 C22 C23 114.2(9) . . ?
N22 C23 C22 112.7(8) . . ?
N22 C24 C25 113.6(8) . . ?
C24 C25 C26 113.7(9) . . ?
N23 C26 C25 108.8(9) . . ?
C31 N31 Ni2 125.3(8) . . ?
C34 N32 C33 109.7(8) . . ?
C34 N32 Ni2 117.0(6) . . ?
C33 N32 Ni2 114.9(6) . . ?
C36 N33 Ni2 119.4(6) . . ?
N31 C31 C32 112.3(10) . . ?
C33 C32 C31 117.5(10) . . ?
C32 C33 N32 114.3(9) . . ?
N32 C34 C35 114.8(8) . . ?
C36 C35 C34 114.3(10) . . ?
C35 C36 N33 113.3(10) . . ?

_diffrn_measured_fraction_theta_max 0.998
_diffrn_reflns_theta_full        25.00
_diffrn_measured_fraction_theta_full 0.998
_refine_diff_density_max         0.417
_refine_diff_density_min         -0.403
_refine_diff_density_rms         0.092

# Attachment '- TA181.CIF'

data_ta181
_database_code_depnum_ccdc_archive 'CCDC 838538'
#TrackingRef '- TA181.CIF'

_audit_creation_method           SHELXL-97
_chemical_name_systematic
;
?
;
_chemical_name_common            ?
_chemical_melting_point          ?
_chemical_formula_moiety         'C13 H22 N6 Ni S2'
_chemical_formula_sum            'C13 H22 N6 Ni S2'
_chemical_formula_weight         385.20

loop_
_atom_type_symbol
_atom_type_description
_atom_type_scat_dispersion_real
_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'
S S 0.1246 0.1234 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
Ni Ni 0.3393 1.1124 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting           monoclinic
_symmetry_space_group_name_H-M   'P 21/c'
_symmetry_space_group_name_Hall  '-P 2ybc'

loop_
```

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

_cell_length_a                    13.0449(8)
_cell_length_b                    8.9722(8)
_cell_length_c                    15.1744(9)
_cell_angle_alpha                 90.00
_cell_angle_beta                  98.385(7)
_cell_angle_gamma                 90.00
_cell_volume                      1757.0(2)
_cell_formula_units_Z             4
_cell_measurement_temperature     170(2)
_cell_measurement_reflns_used     16789
_cell_measurement_theta_min       2.64
_cell_measurement_theta_max       27.98

_exptl_crystal_description        block
_exptl_crystal_colour             blue
_exptl_crystal_size_max           0.17
_exptl_crystal_size_mid           0.14
_exptl_crystal_size_min           0.11
_exptl_crystal_density_meas       ?
_exptl_crystal_density_diffrn     1.456
_exptl_crystal_density_method     'not measured'
_exptl_crystal_F_000              808
_exptl_absorpt_coefficient_mu     1.347
_exptl_absorpt_correction_type    numerical
_exptl_absorpt_correction_T_min   0.789
_exptl_absorpt_correction_T_max   0.854
_exptl_absorpt_process_details    'X-Shape and X-Red32 (STOE, 2008)'

_exptl_special_details
;
?
;

_diffrn_ambient_temperature       170(2)
_diffrn_radiation_wavelength      0.71073
_diffrn_radiation_type            MoK\a
_diffrn_radiation_source          'fine-focus sealed tube'
_diffrn_radiation_monochromator   graphite
_diffrn_measurement_device_type   ?
_diffrn_measurement_method        ?
_diffrn_detector_area_resol_mean  ?
_diffrn_standards_number          ?
_diffrn_standards_interval_count  ?
_diffrn_standards_interval_time   ?
_diffrn_standards_decay_%         ?
_diffrn_reflns_number             16789
_diffrn_reflns_av_R_equivalents   0.0399
_diffrn_reflns_av_sigmaI/netI     0.0286
_diffrn_reflns_limit_h_min        -16
_diffrn_reflns_limit_h_max        16
_diffrn_reflns_limit_k_min        -11
_diffrn_reflns_limit_k_max        11
_diffrn_reflns_limit_l_min        -19
_diffrn_reflns_limit_l_max        20
_diffrn_reflns_theta_min          2.64
_diffrn_reflns_theta_max          27.98
_reflns_number_total              4152
_reflns_number_gt                 3509
_reflns_threshold_expression      >2sigma(I)

_computing_data_collection        ?
_computing_cell_refinement        ?
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
_computing_data_reduction        ?
_computing_structure_solution    'SHELXS-97 (Sheldrick, 1990)'
_computing_structure_refinement  'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics    ?
_computing_publication_material  ?

_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^)+(0.0439P)^2^+0.1732P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary     direct
_atom_sites_solution_secondary   difmap
_atom_sites_solution_hydrogens   geom
_refine_ls_hydrogen_treatment    constr
_refine_ls_extinction_method     SHELXL
_refine_ls_extinction_coef       0.0115(9)
_refine_ls_extinction_expression Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^
_refine_ls_number_reflns         4152
_refine_ls_number_parameters     200
_refine_ls_number_restraints     0
_refine_ls_R_factor_all          0.0371
_refine_ls_R_factor_gt           0.0284
_refine_ls_wR_factor_ref         0.0715
_refine_ls_wR_factor_gt          0.0689
_refine_ls_goodness_of_fit_ref   1.042
_refine_ls_restrained_S_all      1.042
_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_symmetry_multiplicity
_atom_site_calc_flag
_atom_site_refinement_flags
_atom_site_disorder_assembly
_atom_site_disorder_group
Ni1 Ni 0.748193(15) 0.69833(2) 0.624213(12) 0.01433(8) Uani 1 1 d . . .
N1 N 0.89331(12) 0.74414(18) 0.59217(10) 0.0223(3) Uani 1 1 d . . .
C1 C 0.97436(13) 0.74568(19) 0.56931(10) 0.0183(3) Uani 1 1 d . . .
S1 S 1.08902(4) 0.74797(7) 0.53805(4) 0.03661(14) Uani 1 1 d . . .
N2 N 0.60089(12) 0.65218(18) 0.65594(10) 0.0229(3) Uani 1 1 d . . .
C2 C 0.51465(13) 0.62296(18) 0.65918(10) 0.0176(3) Uani 1 1 d . . .
S2 S 0.39285(3) 0.58240(6) 0.66316(3) 0.02590(11) Uani 1 1 d . . .
N11 N 0.74403(12) 0.50005(17) 0.54898(9) 0.0212(3) Uani 1 1 d . . .
H11D H 0.6995 0.5135 0.4967 0.025 Uiso 1 1 calc R . .
H11C H 0.8090 0.4831 0.5343 0.025 Uiso 1 1 calc R . .
N12 N 0.81645(11) 0.57610(16) 0.73973(9) 0.0193(3) Uani 1 1 d . . .
H12C H 0.7624 0.5477 0.7699 0.023 Uiso 1 1 calc R . .
N13 N 0.74674(12) 0.89741(17) 0.69815(9) 0.0210(3) Uani 1 1 d . . .
```

```
H13D H 0.7418 0.9755 0.6584 0.025 Uiso 1 1 calc R . .
H13C H 0.6871 0.8977 0.7238 0.025 Uiso 1 1 calc R . .
C11 C 0.71107(16) 0.3656(2) 0.59449(13) 0.0284(4) Uani 1 1 d . . .
H11A H 0.6993 0.2828 0.5511 0.034 Uiso 1 1 calc R . .
H11B H 0.6446 0.3867 0.6163 0.034 Uiso 1 1 calc R . .
C12 C 0.79160(17) 0.3171(2) 0.67321(14) 0.0321(4) Uani 1 1 d . . .
H12A H 0.7542 0.2770 0.7203 0.039 Uiso 1 1 calc R . .
H12B H 0.8327 0.2344 0.6530 0.039 Uiso 1 1 calc R . .
C13 C 0.86646(15) 0.4365(2) 0.71459(12) 0.0267(4) Uani 1 1 d . . .
H13A H 0.9085 0.3947 0.7685 0.032 Uiso 1 1 calc R . .
H13B H 0.9143 0.4617 0.6718 0.032 Uiso 1 1 calc R . .
C14 C 0.89165(16) 0.6598(2) 0.80451(12) 0.0280(4) Uani 1 1 d . . .
H14A H 0.9536 0.6830 0.7763 0.034 Uiso 1 1 calc R . .
H14B H 0.9142 0.5950 0.8566 0.034 Uiso 1 1 calc R . .
C15 C 0.84880(17) 0.8047(2) 0.83714(12) 0.0309(4) Uani 1 1 d . . .
H15A H 0.7811 0.7834 0.8567 0.037 Uiso 1 1 calc R . .
H15B H 0.8964 0.8398 0.8899 0.037 Uiso 1 1 calc R . .
C16 C 0.83413(15) 0.9295(2) 0.76906(11) 0.0247(4) Uani 1 1 d . . .
H16A H 0.8207 1.0240 0.7991 0.030 Uiso 1 1 calc R . .
H16B H 0.8984 0.9419 0.7423 0.030 Uiso 1 1 calc R . .
N21 N 0.67586(11) 0.82235(15) 0.50925(9) 0.0183(3) Uani 1 1 d . . .
C21 C 0.58133(14) 0.7884(2) 0.46604(11) 0.0238(4) Uani 1 1 d . . .
H21 H 0.5476 0.7021 0.4840 0.029 Uiso 1 1 calc R . .
C22 C 0.53064(15) 0.8729(2) 0.39662(12) 0.0290(4) Uani 1 1 d . . .
H22 H 0.4643 0.8439 0.3673 0.035 Uiso 1 1 calc R . .
C23 C 0.57808(15) 1.0005(2) 0.37058(11) 0.0254(4) Uani 1 1 d . . .
H23 H 0.5446 1.0614 0.3238 0.031 Uiso 1 1 calc R . .
C24 C 0.67553(15) 1.0371(2) 0.41430(11) 0.0228(4) Uani 1 1 d . . .
H24 H 0.7100 1.1241 0.3983 0.027 Uiso 1 1 calc R . .
C25 C 0.72220(14) 0.94459(19) 0.48203(11) 0.0201(3) Uani 1 1 d . . .
H25 H 0.7899 0.9688 0.5104 0.024 Uiso 1 1 calc R . .

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
Ni1 0.00985(12) 0.01849(12) 0.01493(11) 0.00123(7) 0.00277(7) -0.00034(8)
N1 0.0158(8) 0.0284(8) 0.0237(7) 0.0020(6) 0.0061(6) -0.0014(6)
C1 0.0170(9) 0.0204(8) 0.0171(7) -0.0007(6) 0.0011(6) -0.0014(6)
S1 0.0172(3) 0.0483(3) 0.0479(3) -0.0090(2) 0.0168(2) -0.0052(2)
N2 0.0151(8) 0.0289(8) 0.0258(7) 0.0037(6) 0.0062(5) -0.0014(6)
C2 0.0191(9) 0.0188(8) 0.0150(7) 0.0022(6) 0.0032(6) 0.0012(6)
S2 0.0145(2) 0.0371(3) 0.0259(2) 0.00492(18) 0.00231(16) -0.00726(18)
N11 0.0199(8) 0.0230(8) 0.0204(6) -0.0033(6) 0.0025(5) 0.0004(6)
N12 0.0163(7) 0.0237(7) 0.0178(6) 0.0050(5) 0.0019(5) 0.0013(6)
N13 0.0190(8) 0.0221(7) 0.0217(7) -0.0028(5) 0.0022(5) 0.0035(6)
C11 0.0276(11) 0.0187(9) 0.0397(10) -0.0043(7) 0.0071(8) -0.0044(7)
C12 0.0428(13) 0.0202(9) 0.0349(10) 0.0045(7) 0.0111(8) -0.0002(8)
C13 0.0274(10) 0.0252(9) 0.0272(9) 0.0061(7) 0.0028(7) 0.0095(8)
C14 0.0274(11) 0.0304(10) 0.0231(8) 0.0028(7) -0.0069(7) 0.0012(8)
C15 0.0387(12) 0.0336(11) 0.0182(8) -0.0027(7) -0.0028(7) 0.0000(9)
C16 0.0239(10) 0.0248(9) 0.0246(8) -0.0044(7) 0.0007(7) -0.0020(7)
N21 0.0179(7) 0.0196(7) 0.0171(6) 0.0016(5) 0.0014(5) -0.0006(5)
C21 0.0230(10) 0.0264(9) 0.0208(8) 0.0033(7) -0.0006(6) -0.0067(7)
C22 0.0234(10) 0.0335(10) 0.0269(9) 0.0046(8) -0.0066(7) -0.0058(8)
C23 0.0299(11) 0.0242(9) 0.0205(8) 0.0025(7) -0.0021(7) 0.0020(7)
C24 0.0283(10) 0.0179(8) 0.0221(8) 0.0017(6) 0.0038(7) -0.0013(7)
C25 0.0196(9) 0.0208(8) 0.0197(7) -0.0002(6) 0.0019(6) -0.0027(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
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
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
Ni1 N1 2.0634(15) . ?
Ni1 N2 2.0894(15) . ?
Ni1 N11 2.1102(14) . ?
Ni1 N13 2.1108(14) . ?
Ni1 N12 2.1478(14) . ?
Ni1 N21 2.1660(14) . ?
N1 C1 1.160(2) . ?
C1 S1 1.6340(18) . ?
N2 C2 1.163(2) . ?
C2 S2 1.6396(17) . ?
N11 C11 1.484(2) . ?
N12 C14 1.487(2) . ?
N12 C13 1.487(2) . ?
N13 C16 1.477(2) . ?
C11 C12 1.534(3) . ?
C12 C13 1.522(3) . ?
C14 C15 1.526(3) . ?
C15 C16 1.517(3) . ?
N21 C21 1.345(2) . ?
N21 C25 1.346(2) . ?
C21 C22 1.385(3) . ?
C22 C23 1.386(3) . ?
C23 C24 1.385(3) . ?
C24 C25 1.390(2) . ?

loop_
_geom_angle_atom_site_label_1
_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
N1 Ni1 N2 179.69(6) . . ?
N1 Ni1 N11 89.58(6) . . ?
N2 Ni1 N11 90.32(6) . . ?
N1 Ni1 N13 92.08(6) . . ?
N2 Ni1 N13 88.02(6) . . ?
N11 Ni1 N13 178.01(6) . . ?
N1 Ni1 N12 90.49(6) . . ?
N2 Ni1 N12 89.80(6) . . ?
N11 Ni1 N12 89.19(6) . . ?
N13 Ni1 N12 91.90(6) . . ?
N1 Ni1 N21 90.85(6) . . ?
N2 Ni1 N21 88.86(6) . . ?
N11 Ni1 N21 91.35(5) . . ?
N13 Ni1 N21 87.52(5) . . ?
N12 Ni1 N21 178.56(5) . . ?
C1 N1 Ni1 168.66(15) . . ?
N1 C1 S1 179.48(16) . . ?
C2 N2 Ni1 169.14(14) . . ?
N2 C2 S2 179.63(17) . . ?
C11 N11 Ni1 114.82(11) . . ?
C14 N12 C13 108.84(14) . . ?
C14 N12 Ni1 115.90(11) . . ?
C13 N12 Ni1 111.46(10) . . ?
C16 N13 Ni1 119.09(11) . . ?
```

```
N11 C11 C12 112.40(16) . . ?
C13 C12 C11 116.51(15) . . ?
N12 C13 C12 114.81(16) . . ?
N12 C14 C15 114.02(16) . . ?
C16 C15 C14 115.08(15) . . ?
N13 C16 C15 111.08(15) . . ?
C21 N21 C25 117.14(15) . . ?
C21 N21 Ni1 122.49(11) . . ?
C25 N21 Ni1 120.22(12) . . ?
N21 C21 C22 123.28(17) . . ?
C21 C22 C23 119.05(18) . . ?
C24 C23 C22 118.43(16) . . ?
C23 C24 C25 119.02(16) . . ?
N21 C25 C24 123.04(17) . . ?

_diffrn_measured_fraction_theta_max 0.981
_diffrn_reflns_theta_full        27.98
_diffrn_measured_fraction_theta_full 0.981
_refine_diff_density_max         0.323
_refine_diff_density_min         -0.389
_refine_diff_density_rms         0.064

# Attachment '- TA219.CIF'

data_ta219
_database_code_depnum_ccdc_archive 'CCDC 838539'
#TrackingRef '- TA219.CIF'

_audit_creation_method           SHELXL-97
_chemical_name_systematic
;
?
;
_chemical_name_common            ?
_chemical_melting_point          ?
_chemical_formula_moiety         'C32 H80 N20 Ni4 O6 S8'
_chemical_formula_sum            'C32 H80 N20 Ni4 O6 S8'
_chemical_formula_weight         1332.48

loop_
_atom_type_symbol
_atom_type_description
_atom_type_scat_dispersion_real
_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'
Ni Ni 0.3393 1.1124 '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'
O O 0.0106 0.0060 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting           triclinic
_symmetry_space_group_name_H-M   'P -1'
_symmetry_space_group_name_Hall  '-P 1'

loop_
_symmetry_equiv_pos_as_xyz
'x, y, z'
'-x, -y, -z'

_cell_length_a                   7.9392(6)
_cell_length_b                   13.2900(10)
_cell_length_c                   14.4127(11)
_cell_angle_alpha                82.925(9)
_cell_angle_beta                 87.647(10)
_cell_angle_gamma                89.775(9)
_cell_volume                     1507.9(2)
```

```
_cell_formula_units_Z            1
_cell_measurement_temperature    170(2)
_cell_measurement_reflns_used    17362
_cell_measurement_theta_min      2.57
_cell_measurement_theta_max      27.00

_exptl_crystal_description       block
_exptl_crystal_colour            blue
_exptl_crystal_size_max          0.30
_exptl_crystal_size_mid          0.16
_exptl_crystal_size_min          0.12
_exptl_crystal_density_meas      ?
_exptl_crystal_density_diffrn    1.467
_exptl_crystal_density_method    'not measured'
_exptl_crystal_F_000             700
_exptl_absorpt_coefficient_mu    1.561
_exptl_absorpt_correction_type   numerical
_exptl_absorpt_correction_T_min  0.735
_exptl_absorpt_correction_T_max  0.818
_exptl_absorpt_process_details   'X-Shape and X-Red32 (STOE, 2008)'

_exptl_special_details
;
?
;

_diffrn_ambient_temperature      170(2)
_diffrn_radiation_wavelength     0.71073
_diffrn_radiation_type           MoK\a
_diffrn_radiation_source         'fine-focus sealed tube'
_diffrn_radiation_monochromator  graphite
_diffrn_measurement_device_type  ?
_diffrn_measurement_method       ?
_diffrn_detector_area_resol_mean ?
_diffrn_standards_number         ?
_diffrn_standards_interval_count ?
_diffrn_standards_interval_time  ?
_diffrn_standards_decay_%        ?
_diffrn_reflns_number            17362
_diffrn_reflns_av_R_equivalents  0.0677
_diffrn_reflns_av_sigmaI/netI    0.0600
_diffrn_reflns_limit_h_min       -10
_diffrn_reflns_limit_h_max       10
_diffrn_reflns_limit_k_min       -16
_diffrn_reflns_limit_k_max       16
_diffrn_reflns_limit_l_min       -18
_diffrn_reflns_limit_l_max       18
_diffrn_reflns_theta_min         2.57
_diffrn_reflns_theta_max         27.00
_reflns_number_total             6273
_reflns_number_gt                4539
_reflns_threshold_expression     >2sigma(I)

_computing_data_collection       ?
_computing_cell_refinement       ?
_computing_data_reduction        ?
_computing_structure_solution    'SHELXS-97 (Sheldrick, 1990)'
_computing_structure_refinement  'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics    ?
_computing_publication_material  ?

_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
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
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^)+(0.0508P)^2^+0.7492P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary     direct
_atom_sites_solution_secondary   difmap
_atom_sites_solution_hydrogens   geom
_refine_ls_hydrogen_treatment    constr
_refine_ls_extinction_method     SHELXL
_refine_ls_extinction_coef       0.0052(10)
_refine_ls_extinction_expression Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^
_refine_ls_number_reflns         6273
_refine_ls_number_parameters     326
_refine_ls_number_restraints     0
_refine_ls_R_factor_all          0.0710
_refine_ls_R_factor_gt           0.0440
_refine_ls_wR_factor_ref         0.1093
_refine_ls_wR_factor_gt          0.0980
_refine_ls_goodness_of_fit_ref   1.015
_refine_ls_restrained_S_all      1.015
_refine_ls_shift/su_max          0.000
_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_multiplicity
_atom_site_calc_flag
_atom_site_refinement_flags
_atom_site_disorder_assembly
_atom_site_disorder_group
Ni1 Ni 0.68158(5) 0.69451(3) -0.02000(3) 0.02025(12) Uani 1 1 d . . .
Ni2 Ni 0.78131(7) 0.70916(4) 0.41196(3) 0.03697(15) Uani 1 1 d . . .
S1 S 0.66965(11) 0.55673(7) 0.13565(6) 0.0305(2) Uani 1 1 d . . .
C1 C 0.5346(4) 0.4713(2) 0.1094(2) 0.0223(6) Uani 1 1 d . . .
N1 N 0.4420(4) 0.4106(2) 0.0909(2) 0.0264(6) Uani 1 1 d . . .
S2 S 0.98899(13) 0.92786(7) 0.13985(7) 0.0378(2) Uani 1 1 d . . .
C2 C 0.8792(4) 0.8469(2) 0.0909(2) 0.0239(7) Uani 1 1 d . . .
N2 N 0.7990(4) 0.7903(2) 0.0564(2) 0.0314(7) Uani 1 1 d . . .
S3 S 0.42440(15) 0.77484(13) 0.67638(9) 0.0646(4) Uani 1 1 d . A .
C3 C 0.5502(6) 0.7610(3) 0.5860(3) 0.0390(9) Uani 1 1 d . . .
N3 N 0.6378(6) 0.7500(3) 0.5236(3) 0.0597(12) Uani 1 1 d . A .
S4 S 1.14787(11) 0.59295(7) 0.16663(7) 0.0349(2) Uani 1 1 d . A .
C4 C 1.0178(4) 0.6342(3) 0.2451(3) 0.0282(7) Uani 1 1 d . . .
N4 N 0.9289(4) 0.6627(3) 0.3015(2) 0.0376(8) Uani 1 1 d . A .
N11 N 0.9116(3) 0.6273(2) -0.0491(2) 0.0287(6) Uani 1 1 d . . .
H11A H 0.9608 0.6081 0.0070 0.034 Uiso 1 1 calc R . .
H11B H 0.8894 0.5689 -0.0747 0.034 Uiso 1 1 calc R . .
N12 N 0.6853(3) 0.8005(2) -0.1411(2) 0.0240(6) Uani 1 1 d . . .
H12 H 0.6235 0.7706 -0.1839 0.029 Uiso 1 1 calc R . .
N13 N 0.4438(4) 0.7402(2) 0.0247(2) 0.0312(6) Uani 1 1 d . . .
H13A H 0.3654 0.7044 -0.0032 0.037 Uiso 1 1 calc R . .
H13B H 0.4323 0.7203 0.0881 0.037 Uiso 1 1 calc R . .
C11 C 1.0378(4) 0.6856(3) -0.1114(3) 0.0403(9) Uani 1 1 d . . .
H11C H 1.1310 0.6401 -0.1270 0.048 Uiso 1 1 calc R . .
H11D H 1.0853 0.7395 -0.0786 0.048 Uiso 1 1 calc R . .
C12 C 0.9612(5) 0.7329(3) -0.2004(3) 0.0421(10) Uani 1 1 d . . .
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
H12A H 0.8922 0.6811 -0.2251 0.051 Uiso 1 1 calc R . .
H12B H 1.0533 0.7531 -0.2473 0.051 Uiso 1 1 calc R . .
C13 C 0.8524(5) 0.8243(3) -0.1890(3) 0.0377(9) Uani 1 1 d . . .
H13C H 0.9140 0.8701 -0.1531 0.045 Uiso 1 1 calc R . .
H13D H 0.8344 0.8613 -0.2517 0.045 Uiso 1 1 calc R . .
C14 C 0.5996(5) 0.8982(3) -0.1313(3) 0.0338(8) Uani 1 1 d . . .
H14A H 0.6020 0.9398 -0.1933 0.041 Uiso 1 1 calc R . .
H14B H 0.6639 0.9351 -0.0887 0.041 Uiso 1 1 calc R . .
C15 C 0.4185(5) 0.8879(3) -0.0942(3) 0.0364(9) Uani 1 1 d . . .
H15A H 0.3637 0.9551 -0.1048 0.044 Uiso 1 1 calc R . .
H15B H 0.3588 0.8417 -0.1307 0.044 Uiso 1 1 calc R . .
C16 C 0.3975(5) 0.8484(3) 0.0081(3) 0.0370(9) Uani 1 1 d . . .
H16A H 0.4698 0.8880 0.0447 0.044 Uiso 1 1 calc R . .
H16B H 0.2789 0.8570 0.0296 0.044 Uiso 1 1 calc R . .
N21 N 1.0096(6) 0.7282(3) 0.4798(3) 0.0635(12) Uani 1 1 d . A .
H21A H 1.0022 0.6887 0.5369 0.076 Uiso 1 1 calc R . .
H21B H 1.0950 0.7018 0.4452 0.076 Uiso 1 1 calc R . .
N22 N 0.7759(4) 0.8583(2) 0.3455(2) 0.0301(6) Uani 1 1 d . A .
H22 H 0.8305 0.8557 0.2875 0.036 Uiso 1 1 calc R . .
N23 N 0.5700(5) 0.6664(3) 0.3466(3) 0.0531(10) Uani 1 1 d . A .
H23A H 0.6062 0.6441 0.2912 0.064 Uiso 1 1 calc R . .
H23B H 0.5226 0.6116 0.3836 0.064 Uiso 1 1 calc R . .
C21 C 1.0625(7) 0.8294(4) 0.4965(4) 0.0669(16) Uani 1 1 d . . .
H21C H 1.1806 0.8267 0.5162 0.080 Uiso 1 1 calc R A .
H21D H 0.9911 0.8519 0.5480 0.080 Uiso 1 1 calc R . .
C22 C 1.0491(6) 0.9045(4) 0.4104(4) 0.0586(13) Uani 1 1 d . A .
H22A H 1.1013 0.8750 0.3563 0.070 Uiso 1 1 calc R . .
H22B H 1.1139 0.9661 0.4186 0.070 Uiso 1 1 calc R . .
C23 C 0.8696(6) 0.9349(3) 0.3885(3) 0.0447(10) Uani 1 1 d . . .
H23C H 0.8087 0.9478 0.4472 0.054 Uiso 1 1 calc R A .
H23D H 0.8718 0.9991 0.3456 0.054 Uiso 1 1 calc R . .
C24 C 0.6071(5) 0.9011(3) 0.3238(3) 0.0408(9) Uani 1 1 d . . .
H24A H 0.6218 0.9689 0.2873 0.049 Uiso 1 1 calc R A .
H24B H 0.5452 0.9103 0.3832 0.049 Uiso 1 1 calc R . .
C25 C 0.5020(6) 0.8366(4) 0.2694(3) 0.0527(12) Uani 1 1 d . A .
H25A H 0.4066 0.8777 0.2438 0.063 Uiso 1 1 calc R . .
H25B H 0.5717 0.8174 0.2158 0.063 Uiso 1 1 calc R . .
C26 C 0.4332(5) 0.7420(4) 0.3249(4) 0.0559(13) Uani 1 1 d . . .
H26A H 0.3785 0.7589 0.3840 0.067 Uiso 1 1 calc R A .
H26B H 0.3467 0.7121 0.2889 0.067 Uiso 1 1 calc R . .
O1W O 0.8181(8) 0.5598(5) 0.4811(5) 0.0529(15) Uani 0.70 1 d P A 1
H1O1 H 0.7548 0.5358 0.5265 0.079 Uiso 1 1 d R . .
H2O1 H 0.7843 0.5105 0.4557 0.079 Uiso 1 1 d R . .
O1W' O 0.722(2) 0.5503(11) 0.4751(11) 0.061(4) Uani 0.30 1 d P A 2
O2W O 0.8197(7) 0.4190(3) 0.3537(3) 0.0914(15) Uani 1 1 d . . .
H1O2 H 0.7152 0.4236 0.3475 0.137 Uiso 1 1 d R . .
H2O2 H 0.8907 0.4319 0.3090 0.137 Uiso 1 1 d R . .
O3W O 0.6633(6) 0.5288(3) 0.6564(3) 0.0851(14) Uani 1 1 d . . .
H1O3 H 0.5879 0.5655 0.6772 0.128 Uiso 1 1 d R . .
H2O3 H 0.7253 0.5162 0.7023 0.128 Uiso 1 1 d R . .

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
Ni1 0.0186(2) 0.01780(19) 0.0252(2) -0.00542(16) -0.00252(16) -0.00276(14)
Ni2 0.0517(3) 0.0343(3) 0.0254(3) -0.0080(2) 0.0044(2) 0.0103(2)
S1 0.0326(4) 0.0285(4) 0.0315(5) -0.0044(4) -0.0099(4) -0.0115(3)
C1 0.0250(15) 0.0211(14) 0.0212(16) -0.0023(12) -0.0039(13) -0.0010(12)
N1 0.0283(14) 0.0219(13) 0.0292(15) -0.0030(12) -0.0033(12) -0.0048(11)
S2 0.0384(5) 0.0381(5) 0.0400(5) -0.0180(4) 0.0027(4) -0.0174(4)
C2 0.0245(15) 0.0223(15) 0.0260(17) -0.0074(13) 0.0000(13) -0.0021(12)
N2 0.0313(15) 0.0275(15) 0.0372(17) -0.0101(13) -0.0052(13) -0.0029(12)
S3 0.0368(6) 0.1249(12) 0.0393(6) -0.0393(7) -0.0003(5) 0.0075(7)
```

```
C3 0.054(2) 0.038(2) 0.0252(19) -0.0049(16) -0.0015(18) 0.0116(18)
N3 0.092(3) 0.049(2) 0.037(2) -0.0074(18) 0.026(2) 0.009(2)
S4 0.0258(4) 0.0426(5) 0.0393(5) -0.0181(4) 0.0031(4) 0.0008(4)
C4 0.0297(17) 0.0249(16) 0.0318(18) -0.0088(14) -0.0064(15) 0.0013(13)
N4 0.0359(17) 0.0441(18) 0.0354(17) -0.0160(15) 0.0004(14) 0.0094(14)
N11 0.0225(13) 0.0258(14) 0.0394(17) -0.0090(13) -0.0043(13) 0.0033(11)
N12 0.0254(13) 0.0198(12) 0.0273(14) -0.0043(11) -0.0015(12) -0.0007(10)
N13 0.0236(14) 0.0357(16) 0.0345(16) -0.0067(13) 0.0035(12) 0.0015(12)
C11 0.0176(16) 0.042(2) 0.061(3) -0.011(2) 0.0077(17) 0.0018(15)
C12 0.037(2) 0.041(2) 0.046(2) -0.0061(19) 0.0198(19) 0.0019(17)
C13 0.0318(19) 0.0293(18) 0.049(2) 0.0038(17) 0.0116(18) -0.0058(15)
C14 0.039(2) 0.0210(16) 0.040(2) 0.0030(15) -0.0030(17) 0.0063(14)
C15 0.0319(18) 0.0369(19) 0.040(2) -0.0039(17) -0.0032(16) 0.0158(16)
C16 0.0347(19) 0.041(2) 0.037(2) -0.0127(17) 0.0008(17) 0.0142(16)
N21 0.074(3) 0.070(3) 0.053(2) -0.024(2) -0.030(2) 0.034(2)
N22 0.0302(15) 0.0334(15) 0.0278(15) -0.0085(13) -0.0025(13) 0.0044(12)
N23 0.047(2) 0.050(2) 0.066(3) -0.027(2) 0.0136(19) -0.0071(17)
C21 0.066(3) 0.078(4) 0.067(3) -0.037(3) -0.037(3) 0.023(3)
C22 0.046(3) 0.076(3) 0.061(3) -0.035(3) -0.017(2) 0.005(2)
C23 0.050(2) 0.039(2) 0.049(2) -0.0162(19) -0.012(2) 0.0043(18)
C24 0.033(2) 0.041(2) 0.047(2) 0.0006(19) -0.0032(18) 0.0061(16)
C25 0.033(2) 0.081(3) 0.045(3) -0.010(2) -0.0101(19) 0.002(2)
C26 0.032(2) 0.070(3) 0.071(3) -0.032(3) -0.001(2) -0.004(2)
O1W 0.066(4) 0.040(3) 0.052(3) -0.001(2) 0.000(3) -0.002(3)
O1W' 0.097(12) 0.042(7) 0.041(7) 0.003(6) 0.011(9) 0.030(9)
O2W 0.148(4) 0.057(2) 0.069(3) -0.013(2) 0.016(3) -0.009(3)
O3W 0.097(3) 0.081(3) 0.067(3) 0.028(2) 0.012(2) 0.033(2)

_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
Ni1 N2 2.035(3) . ?
Ni1 N11 2.080(3) . ?
Ni1 N13 2.082(3) . ?
Ni1 N1 2.098(3) 2_665 ?
Ni1 N12 2.102(3) . ?
Ni1 S1 2.7142(11) . ?
Ni2 N3 2.060(4) . ?
Ni2 N23 2.072(4) . ?
Ni2 N4 2.091(3) . ?
Ni2 N22 2.094(3) . ?
Ni2 N21 2.125(4) . ?
Ni2 O1W 2.133(7) . ?
Ni2 O1W' 2.238(16) . ?
S1 C1 1.653(3) . ?
C1 N1 1.155(4) . ?
N1 Ni1 2.098(3) 2_665 ?
S2 C2 1.631(3) . ?
C2 N2 1.158(4) . ?
S3 C3 1.636(4) . ?
C3 N3 1.138(5) . ?
S4 C4 1.643(4) . ?
C4 N4 1.153(5) . ?
N11 C11 1.473(5) . ?
N12 C14 1.483(4) . ?
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
N12 C13 1.488(5) . ?
N13 C16 1.476(5) . ?
C11 C12 1.507(6) . ?
C12 C13 1.510(5) . ?
C14 C15 1.514(5) . ?
C15 C16 1.506(6) . ?
N21 C21 1.461(7) . ?
N22 C23 1.474(5) . ?
N22 C24 1.484(5) . ?
N23 C26 1.493(6) . ?
C21 C22 1.501(8) . ?
C22 C23 1.514(6) . ?
C24 C25 1.507(6) . ?
C25 C26 1.497(7) . ?

loop_
_geom_angle_atom_site_label_1
_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
N2 Ni1 N11 90.15(12) . . ?
N2 Ni1 N13 92.19(12) . . ?
N11 Ni1 N13 170.49(12) . . ?
N2 Ni1 N1 176.33(13) . 2_665 ?
N11 Ni1 N1 89.97(11) . 2_665 ?
N13 Ni1 N1 87.11(12) . 2_665 ?
N2 Ni1 N12 93.27(12) . . ?
N11 Ni1 N12 96.22(11) . . ?
N13 Ni1 N12 92.85(12) . . ?
N1 Ni1 N12 90.36(11) 2_665 . ?
N2 Ni1 S1 87.63(9) . . ?
N11 Ni1 S1 84.66(9) . . ?
N13 Ni1 S1 86.22(9) . . ?
N1 Ni1 S1 88.74(8) 2_665 . ?
N12 Ni1 S1 178.74(8) . . ?
N3 Ni2 N23 92.08(18) . . ?
N3 Ni2 N4 177.92(15) . . ?
N23 Ni2 N4 88.25(14) . . ?
N3 Ni2 N22 90.72(14) . . ?
N23 Ni2 N22 93.44(15) . . ?
N4 Ni2 N22 91.31(13) . . ?
N3 Ni2 N21 92.11(19) . . ?
N23 Ni2 N21 170.83(16) . . ?
N4 Ni2 N21 87.27(15) . . ?
N22 Ni2 N21 94.66(15) . . ?
N3 Ni2 O1W 92.0(2) . . ?
N23 Ni2 O1W 92.7(2) . . ?
N4 Ni2 O1W 86.0(2) . . ?
N22 Ni2 O1W 173.19(18) . . ?
N21 Ni2 O1W 79.0(2) . . ?
N3 Ni2 O1W' 84.6(4) . . ?
N23 Ni2 O1W' 74.0(5) . . ?
N4 Ni2 O1W' 93.5(4) . . ?
N22 Ni2 O1W' 166.3(4) . . ?
N21 Ni2 O1W' 98.3(5) . . ?
O1W Ni2 O1W' 20.5(4) . . ?
C1 S1 Ni1 103.06(12) . . ?
N1 C1 S1 179.1(3) . . ?
C1 N1 Ni1 162.8(3) . 2_665 ?
N2 C2 S2 178.9(3) . . ?
C2 N2 Ni1 171.9(3) . . ?
N3 C3 S3 179.0(4) . . ?
C3 N3 Ni2 171.7(4) . . ?
N4 C4 S4 178.6(3) . . ?
C4 N4 Ni2 175.3(3) . . ?
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
C11 N11 Ni1 119.1(2) . . ?
C14 N12 C13 107.4(3) . . ?
C14 N12 Ni1 115.7(2) . . ?
C13 N12 Ni1 116.9(2) . . ?
C16 N13 Ni1 119.3(2) . . ?
N11 C11 C12 111.3(3) . . ?
C11 C12 C13 114.3(3) . . ?
N12 C13 C12 114.6(3) . . ?
N12 C14 C15 114.4(3) . . ?
C16 C15 C14 114.7(3) . . ?
N13 C16 C15 110.8(3) . . ?
C21 N21 Ni2 120.0(3) . . ?
C23 N22 C24 107.5(3) . . ?
C23 N22 Ni2 117.0(2) . . ?
C24 N22 Ni2 116.5(2) . . ?
C26 N23 Ni2 119.3(3) . . ?
N21 C21 C22 111.4(4) . . ?
C21 C22 C23 113.7(5) . . ?
N22 C23 C22 113.9(3) . . ?
N22 C24 C25 114.0(3) . . ?
C26 C25 C24 114.7(4) . . ?
N23 C26 C25 110.9(4) . . ?

_diffrn_measured_fraction_theta_max 0.954
_diffrn_reflns_theta_full        27.00
_diffrn_measured_fraction_theta_full 0.954
_refine_diff_density_max         0.950
_refine_diff_density_min         -1.070
_refine_diff_density_rms         0.085

# Attachment '- TA243.CIF'

data_ta243
_database_code_depnum_ccdc_archive 'CCDC 838540'
#TrackingRef '- TA243.CIF'

_audit_creation_method           SHELXL-97
_chemical_name_systematic
;
?
;
_chemical_name_common            ?
_chemical_melting_point          ?
_chemical_formula_moiety         'C13 H22 N6 Ni S2'
_chemical_formula_sum            'C13 H22 N6 Ni S2'
_chemical_formula_weight         385.20

loop_
_atom_type_symbol
_atom_type_description
_atom_type_scat_dispersion_real
_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'
S S 0.1246 0.1234 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
Ni Ni 0.3393 1.1124 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting           orthorhombic
_symmetry_space_group_name_H-M   'P b c a'
_symmetry_space_group_name_Hall  '-P 2ac 2ab'

loop_
_symmetry_equiv_pos_as_xyz
'x, y, z'
'-x+1/2, -y, z+1/2'
'-x, y+1/2, -z+1/2'
```

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

_cell_length_a                    8.5835(3)
_cell_length_b                    14.7666(4)
_cell_length_c                    28.9345(7)
_cell_angle_alpha                 90.00
_cell_angle_beta                  90.00
_cell_angle_gamma                 90.00
_cell_volume                      3667.42(18)
_cell_formula_units_Z             8
_cell_measurement_temperature     293(2)
_cell_measurement_reflns_used     25954
_cell_measurement_theta_min       2.76
_cell_measurement_theta_max       26.85

_exptl_crystal_description        hexagon
_exptl_crystal_colour             blue
_exptl_crystal_size_max           0.09
_exptl_crystal_size_mid           0.07
_exptl_crystal_size_min           0.05
_exptl_crystal_density_meas       ?
_exptl_crystal_density_diffrn     1.395
_exptl_crystal_density_method     'not measured'
_exptl_crystal_F_000              1616
_exptl_absorpt_coefficient_mu     1.290
_exptl_absorpt_correction_type    numerical
_exptl_absorpt_correction_T_min   0.889
_exptl_absorpt_correction_T_max   0.931
_exptl_absorpt_process_details    'X-Shape and X-Red32 (STOE, 2008)'

_exptl_special_details
;
?
;

_diffrn_ambient_temperature       293(2)
_diffrn_radiation_wavelength      0.71073
_diffrn_radiation_type            MoK\a
_diffrn_radiation_source          'fine-focus sealed tube'
_diffrn_radiation_monochromator   graphite
_diffrn_measurement_device_type   ?
_diffrn_measurement_method        ?
_diffrn_detector_area_resol_mean  ?
_diffrn_standards_number          ?
_diffrn_standards_interval_count  ?
_diffrn_standards_interval_time   ?
_diffrn_standards_decay_%         ?
_diffrn_reflns_number             25954
_diffrn_reflns_av_R_equivalents   0.0292
_diffrn_reflns_av_sigmaI/netI     0.0146
_diffrn_reflns_limit_h_min        -10
_diffrn_reflns_limit_h_max        10
_diffrn_reflns_limit_k_min        -18
_diffrn_reflns_limit_k_max        18
_diffrn_reflns_limit_l_min        -36
_diffrn_reflns_limit_l_max        36
_diffrn_reflns_theta_min          2.76
_diffrn_reflns_theta_max          26.85
_reflns_number_total              3880
_reflns_number_gt                 3461
_reflns_threshold_expression      >2sigma(I)

_computing_data_collection        ?
_computing_cell_refinement        ?
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
_computing_data_reduction        ?
_computing_structure_solution    'SHELXS-97 (Sheldrick, 1990)'
_computing_structure_refinement  'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics    ?
_computing_publication_material  ?

_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^)+(0.0254P)^2^+2.1456P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary     direct
_atom_sites_solution_secondary   difmap
_atom_sites_solution_hydrogens   geom
_refine_ls_hydrogen_treatment    constr
_refine_ls_extinction_method     none
_refine_ls_extinction_coef       ?
_refine_ls_number_reflns         3880
_refine_ls_number_parameters     199
_refine_ls_number_restraints     0
_refine_ls_R_factor_all          0.0469
_refine_ls_R_factor_gt           0.0398
_refine_ls_wR_factor_ref         0.0809
_refine_ls_wR_factor_gt          0.0781
_refine_ls_goodness_of_fit_ref   1.146
_refine_ls_restrained_S_all      1.146
_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_symmetry_multiplicity
_atom_site_calc_flag
_atom_site_refinement_flags
_atom_site_disorder_assembly
_atom_site_disorder_group
Ni1 Ni 0.54953(3) 0.654059(19) 0.376845(9) 0.04257(9) Uani 1 1 d . . .
N1 N 0.7569(2) 0.67144(14) 0.33858(7) 0.0550(5) Uani 1 1 d . . .
C1 C 0.8719(3) 0.69014(15) 0.32030(8) 0.0462(5) Uani 1 1 d . . .
S1 S 1.03505(8) 0.71629(6) 0.29479(2) 0.06577(19) Uani 1 1 d . . .
N2 N 0.3460(2) 0.63942(14) 0.41461(7) 0.0521(5) Uani 1 1 d . . .
C2 C 0.2352(3) 0.65319(15) 0.43588(8) 0.0454(5) Uani 1 1 d . . .
S2 S 0.07959(8) 0.67080(6) 0.46669(3) 0.0708(2) Uani 1 1 d . . .
N11 N 0.4228(2) 0.64216(14) 0.31539(7) 0.0540(5) Uani 1 1 d . . .
H11A H 0.3337 0.6733 0.3195 0.065 Uiso 1 1 calc R . .
H11B H 0.4772 0.6718 0.2935 0.065 Uiso 1 1 calc R . .
N12 N 0.6044(2) 0.51314(13) 0.38195(7) 0.0511(5) Uani 1 1 d . . .
H12 H 0.7060 0.5096 0.3732 0.061 Uiso 1 1 calc R . .
N13 N 0.6802(2) 0.67833(13) 0.43732(6) 0.0504(5) Uani 1 1 d . . .
H13A H 0.7816 0.6796 0.4293 0.060 Uiso 1 1 calc R . .
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
H13B H 0.6555 0.7340 0.4476 0.060 Uiso 1 1 calc R . .
C11 C 0.3793(4) 0.5541(2) 0.29549(10) 0.0696(8) Uani 1 1 d . . .
H11C H 0.3578 0.5616 0.2628 0.084 Uiso 1 1 calc R . .
H11D H 0.2848 0.5327 0.3102 0.084 Uiso 1 1 calc R . .
C12 C 0.5061(4) 0.4844(2) 0.30159(10) 0.0741(8) Uani 1 1 d . . .
H12A H 0.6045 0.5106 0.2919 0.089 Uiso 1 1 calc R . .
H12B H 0.4845 0.4333 0.2815 0.089 Uiso 1 1 calc R . .
C13 C 0.5226(4) 0.45029(19) 0.35057(10) 0.0713(8) Uani 1 1 d . . .
H13C H 0.4195 0.4385 0.3629 0.086 Uiso 1 1 calc R . .
H13D H 0.5786 0.3933 0.3500 0.086 Uiso 1 1 calc R . .
C14 C 0.6020(4) 0.47393(18) 0.42891(9) 0.0642(7) Uani 1 1 d . . .
H14A H 0.6309 0.4106 0.4269 0.077 Uiso 1 1 calc R . .
H14B H 0.4963 0.4768 0.4407 0.077 Uiso 1 1 calc R . .
C16 C 0.6634(3) 0.61478(18) 0.47625(8) 0.0574(6) Uani 1 1 d . . .
H16A H 0.5561 0.6151 0.4868 0.069 Uiso 1 1 calc R . .
H16B H 0.7286 0.6347 0.5017 0.069 Uiso 1 1 calc R . .
N21 N 0.5080(2) 0.79993(13) 0.37198(6) 0.0468(4) Uani 1 1 d . . .
C21 C 0.5511(3) 0.84918(17) 0.33554(8) 0.0556(6) Uani 1 1 d . . .
H21 H 0.5979 0.8199 0.3107 0.067 Uiso 1 1 calc R . .
C22 C 0.5293(3) 0.94154(18) 0.33306(9) 0.0610(7) Uani 1 1 d . . .
H22 H 0.5600 0.9733 0.3068 0.073 Uiso 1 1 calc R . .
C23 C 0.4625(3) 0.98612(17) 0.36930(9) 0.0596(6) Uani 1 1 d . . .
H23 H 0.4480 1.0485 0.3684 0.071 Uiso 1 1 calc R . .
C24 C 0.4172(3) 0.93660(17) 0.40706(10) 0.0592(6) Uani 1 1 d . . .
H24 H 0.3712 0.9649 0.4323 0.071 Uiso 1 1 calc R . .
C25 C 0.4406(3) 0.84440(16) 0.40705(9) 0.0519(5) Uani 1 1 d . . .
H25 H 0.4082 0.8113 0.4327 0.062 Uiso 1 1 calc R . .
C15 C 0.7085(3) 0.51999(19) 0.46276(9) 0.0660(7) Uani 1 1 d . . .
H15A H 0.8124 0.5217 0.4496 0.079 Uiso 1 1 calc R . .
H15B H 0.7135 0.4834 0.4906 0.079 Uiso 1 1 calc R . .

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
Ni1 0.04309(15) 0.04508(16) 0.03952(15) 0.00016(12) 0.00431(12) 0.00061(12)
N1 0.0515(11) 0.0604(12) 0.0529(11) 0.0018(10) 0.0114(10) 0.0016(10)
C1 0.0515(13) 0.0436(11) 0.0434(12) -0.0010(9) 0.0007(10) 0.0054(10)
S1 0.0533(4) 0.0805(5) 0.0635(4) 0.0022(3) 0.0121(3) -0.0088(3)
N2 0.0474(10) 0.0549(12) 0.0540(11) 0.0013(9) 0.0071(9) -0.0021(9)
C2 0.0452(11) 0.0426(11) 0.0483(12) -0.0026(10) -0.0017(10) -0.0042(10)
S2 0.0502(3) 0.0863(5) 0.0758(5) -0.0235(4) 0.0144(3) -0.0014(3)
N11 0.0534(11) 0.0616(12) 0.0469(11) -0.0039(9) -0.0017(9) 0.0016(10)
N12 0.0560(11) 0.0474(10) 0.0501(11) -0.0011(9) 0.0068(9) 0.0030(9)
N13 0.0505(11) 0.0544(11) 0.0462(11) 0.0014(9) -0.0014(9) 0.0003(9)
C11 0.0776(19) 0.0740(19) 0.0574(16) -0.0077(14) -0.0078(14) -0.0138(16)
C12 0.104(2) 0.0616(17) 0.0563(16) -0.0154(13) 0.0042(16) -0.0059(16)
C13 0.102(2) 0.0490(15) 0.0632(17) -0.0063(12) -0.0013(16) -0.0065(15)
C14 0.0840(19) 0.0483(14) 0.0602(16) 0.0108(12) 0.0028(14) 0.0038(13)
C16 0.0611(15) 0.0677(16) 0.0434(13) 0.0040(11) -0.0038(11) 0.0038(13)
N21 0.0515(10) 0.0460(10) 0.0430(10) 0.0006(8) 0.0023(8) 0.0007(8)
C21 0.0678(15) 0.0543(14) 0.0446(12) 0.0028(11) 0.0041(11) 0.0012(13)
C22 0.0753(17) 0.0561(15) 0.0516(14) 0.0126(12) -0.0027(13) -0.0041(13)
C23 0.0672(16) 0.0451(13) 0.0664(16) 0.0045(12) -0.0040(13) 0.0015(12)
C24 0.0656(15) 0.0494(13) 0.0625(15) -0.0047(12) 0.0088(13) 0.0028(12)
C25 0.0586(14) 0.0481(12) 0.0489(12) -0.0002(10) 0.0083(11) -0.0010(11)
C15 0.0758(18) 0.0646(16) 0.0577(15) 0.0095(13) -0.0066(14) 0.0123(14)

_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
```

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011


```
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
Ni1 N2 2.072(2) . ?
Ni1 N11 2.092(2) . ?
Ni1 N13 2.1093(19) . ?
Ni1 N1 2.112(2) . ?
Ni1 N12 2.1386(19) . ?
Ni1 N21 2.1879(19) . ?
N1 C1 1.154(3) . ?
C1 S1 1.629(2) . ?
N2 C2 1.150(3) . ?
C2 S2 1.627(2) . ?
N11 C11 1.470(3) . ?
N12 C13 1.476(3) . ?
N12 C14 1.477(3) . ?
N13 C16 1.473(3) . ?
C11 C12 1.509(4) . ?
C12 C13 1.511(4) . ?
C14 C15 1.503(4) . ?
C16 C15 1.504(4) . ?
N21 C21 1.333(3) . ?
N21 C25 1.340(3) . ?
C21 C22 1.378(4) . ?
C22 C23 1.365(4) . ?
C23 C24 1.371(4) . ?
C24 C25 1.376(3) . ?

loop_
_geom_angle_atom_site_label_1
_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
N2 Ni1 N11 90.06(8) . . ?
N2 Ni1 N13 91.65(8) . . ?
N11 Ni1 N13 174.89(8) . . ?
N2 Ni1 N1 179.00(8) . . ?
N11 Ni1 N1 90.16(8) . . ?
N13 Ni1 N1 88.05(8) . . ?
N2 Ni1 N12 92.73(8) . . ?
N11 Ni1 N12 95.24(8) . . ?
N13 Ni1 N12 89.48(8) . . ?
N1 Ni1 N12 88.22(8) . . ?
N2 Ni1 N21 89.95(8) . . ?
N11 Ni1 N21 86.74(8) . . ?
N13 Ni1 N21 88.45(7) . . ?
N1 Ni1 N21 89.08(8) . . ?
N12 Ni1 N21 176.66(8) . . ?
C1 N1 Ni1 172.2(2) . . ?
N1 C1 S1 179.6(2) . . ?
C2 N2 Ni1 163.81(19) . . ?
N2 C2 S2 178.7(2) . . ?
C11 N11 Ni1 122.64(17) . . ?
C13 N12 C14 108.2(2) . . ?
C13 N12 Ni1 117.69(17) . . ?
C14 N12 Ni1 116.23(15) . . ?
C16 N13 Ni1 118.28(16) . . ?
N11 C11 C12 112.0(2) . . ?
```

This journal is © The Royal Society of Chemistry 2011


```
C11 C12 C13 113.9(3) . . ?
N12 C13 C12 114.3(2) . . ?
N12 C14 C15 114.4(2) . . ?
N13 C16 C15 111.7(2) . . ?
C21 N21 C25 116.8(2) . . ?
C21 N21 Ni1 122.87(16) . . ?
C25 N21 Ni1 120.27(15) . . ?
N21 C21 C22 122.9(2) . . ?
C23 C22 C21 119.6(2) . . ?
C22 C23 C24 118.3(2) . . ?
C23 C24 C25 119.1(2) . . ?
N21 C25 C24 123.2(2) . . ?
C14 C15 C16 115.7(2) . . ?

_diffrn_measured_fraction_theta_max 0.984
_diffrn_reflns_theta_full        26.85
_diffrn_measured_fraction_theta_full 0.984
_refine_diff_density_max         0.313
_refine_diff_density_min         -0.305
_refine_diff_density_rms         0.038
```