Supplementary Materials

Bis(3-butylamino-4-nitro)piperazine

Dr. M. John Plater,* Mr David P. B. Bulloch and Prof. William T. A. Harrison
Department of Chemistry, University of Aberdeen, Meston Walk, Aberdeen, AB24 3UE
m.j.plater@abdn.ac.uk

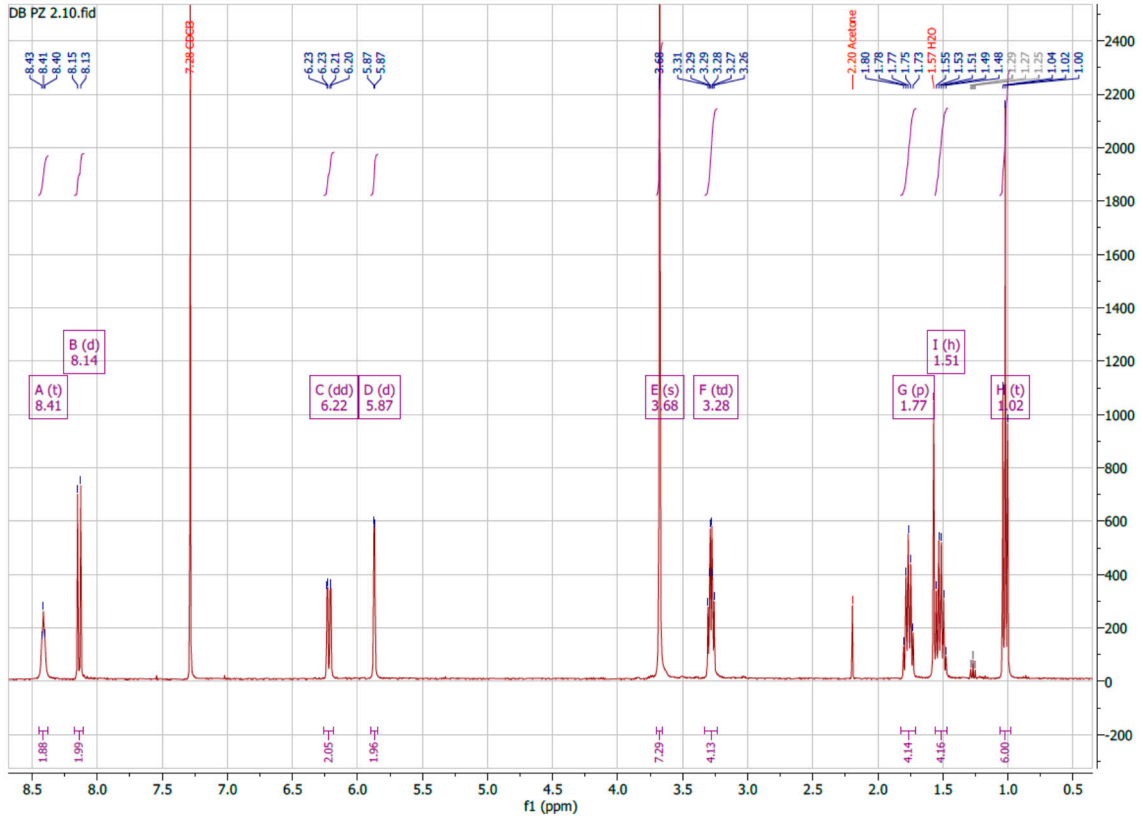


Figure S1 SI 400MHz ^{1}H NMR of compound **5** in $CDCl_3$

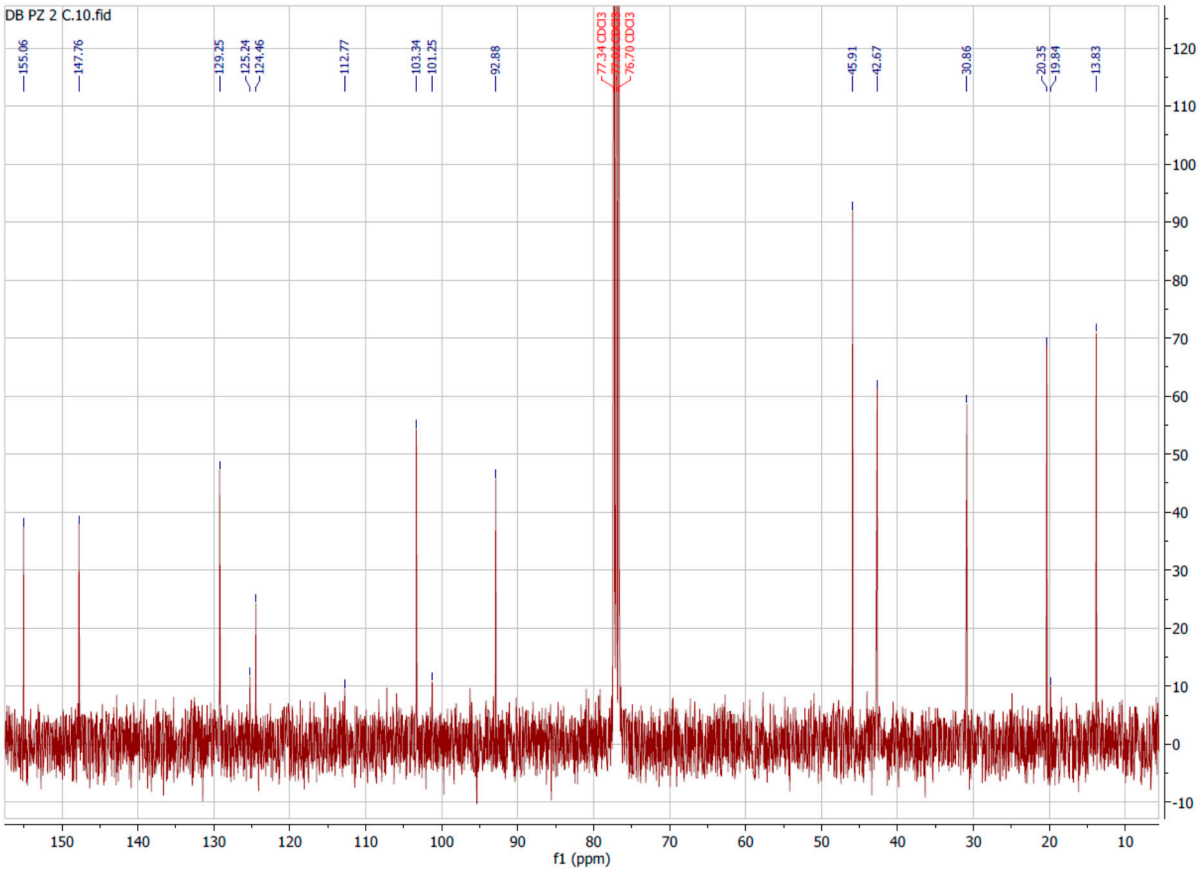


Figure S2 SI 400 MHz ^{13}C NMR of compound **5** in $CDCl_3$

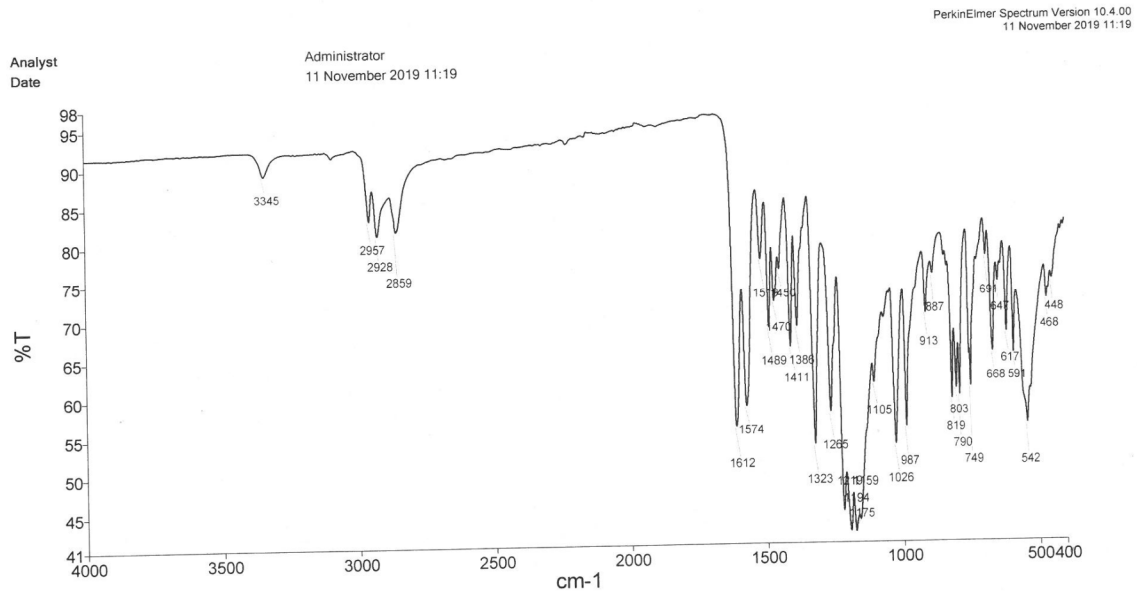


Figure S3 SI FTIR ATR of compound **5**.

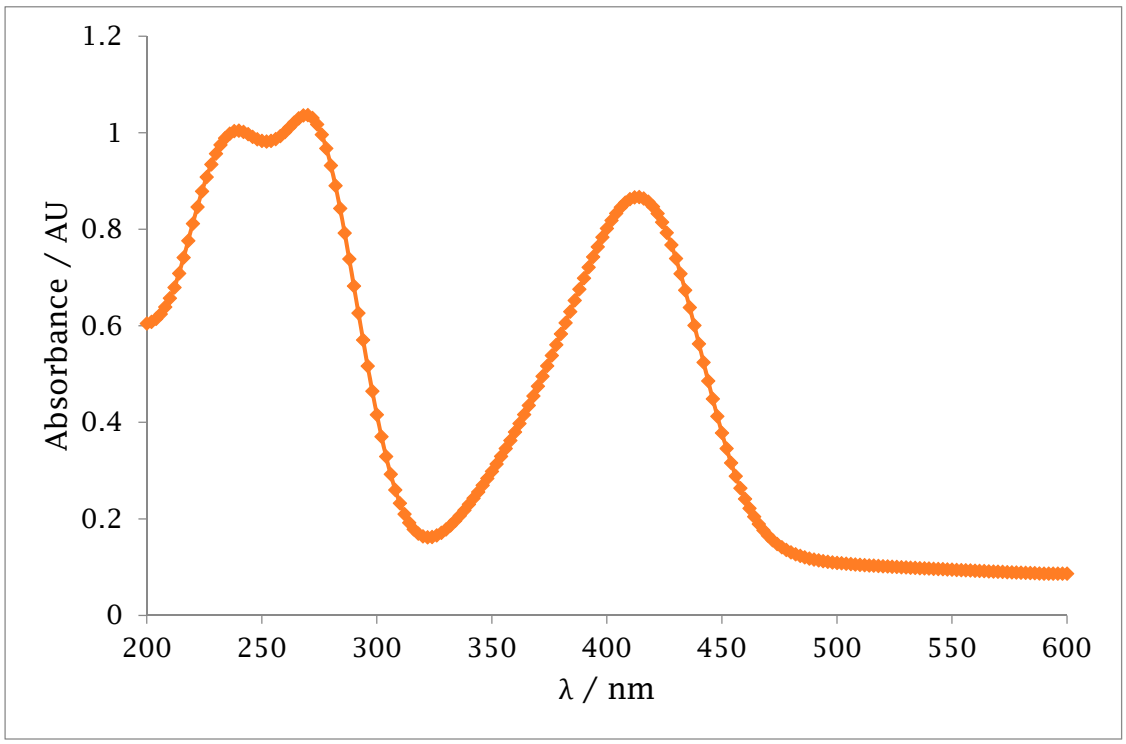


Figure S4 SI UV/Vis of compound **5** in EtOH.

CIF File of compound 5

```
data_db1

_audit_creation_method            'SHELXL-2014/7'
_shelx_SHELXL_version_number      '2014/7'
_chemical_name_systematic         ?
_chemical_name_common             ?
_chemical_melting_point           ?
_chemical_formula_moiety          'C24 H34 N6 O4'
_chemical_formula_sum             'C24 H34 N6 O4'
_chemical_formula_weight          470.57

loop_
 _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
 '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'   0.0311   0.0180
 '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'

_space_group_crystal_system       orthorhombic
_space_group_IT_number            61
_space_group_name_H-M_alt         'P b c a'
_space_group_name_Hall            '-P 2ac 2ab'

_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.
;

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

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

_cell_length_a 22.9241(2)
_cell_length_b 7.32050(10)
_cell_length_c 42.5522(4)
_cell_angle_alpha 90
_cell_angle_beta 90
_cell_angle_gamma 90
_cell_volume 7140.93(13)
_cell_formula_units_Z 12
_cell_measurement_temperature 100(2)
_cell_measurement_reflns_used 14178
_cell_measurement_theta_min 2.8330
_cell_measurement_theta_max 69.7890
_exptl_crystal_description 'cut block'
_exptl_crystal_colour yellow
_exptl_crystal_density_meas ?
_exptl_crystal_density_method ?
_exptl_crystal_density_diffrn 1.313
_exptl_crystal_F_000 3024
_exptl_transmission_factor_min ?
_exptl_transmission_factor_max ?
_exptl_crystal_size_max 0.140
_exptl_crystal_size_mid 0.090
_exptl_crystal_size_min 0.040
_exptl_absorpt_coefficient_mu 0.745
_shelx_estimated_absorpt_T_min 0.903
_shelx_estimated_absorpt_T_max 0.971
loop_
_exptl_crystal_face_index_h
_exptl_crystal_face_index_k
_exptl_crystal_face_index_l
_exptl_crystal_face_perp_dist
1 0 1 0.0200
-1 0 -1 0.0200
1 0 -1 0.0250
-1 0 1 0.0250
0 -1 0 0.0700
0 1 0 0.0700
_exptl_absorpt_correction_T_min 0.892
_exptl_absorpt_correction_T_max 1.000
_exptl_absorpt_correction_type gaussian

```
_exptl_absorpt_process_details
;
CrysAlisPro 1.171.40.67a (Rigaku Oxford Diffraction, 2019)
Numerical absorption correction based on gaussian integration over
                    a multifaceted crystal model
Empirical absorption correction using spherical harmonics,
 implemented in SCALE3 ABSPACK scaling algorithm.
;
#_exptl_absorpt_special_details    ?
_diffrn_ambient_temperature       100(2)
_diffrn_radiation_wavelength      1.54178
_diffrn_radiation_type            CuK\a
_diffrn_source 'Rotating-anode X-ray tube'
_diffrn_source_type 'Rigaku (Cu) X-ray Source'
_diffrn_radiation_monochromator mirror
_diffrn_measurement_device_type 'XtaLAB AFC11 (RCD3): quarter-chi single'
_diffrn_detector_area_resol_mean  ?
_diffrn_reflns_number             31395
_diffrn_reflns_av_unetI/netI      0.0217
_diffrn_reflns_av_R_equivalents   0.0338
_diffrn_reflns_limit_h_min        -24
_diffrn_reflns_limit_h_max        27
_diffrn_reflns_limit_k_min        -8
_diffrn_reflns_limit_k_max        8
_diffrn_reflns_limit_l_min        -51
_diffrn_reflns_limit_l_max        49
_diffrn_reflns_theta_min          2.076
_diffrn_reflns_theta_max          68.243
_diffrn_reflns_theta_full         67.679
_diffrn_measured_fraction_theta_max   0.988
_diffrn_measured_fraction_theta_full  0.989
_diffrn_reflns_Laue_measured_fraction_max    0.988
_diffrn_reflns_Laue_measured_fraction_full   0.989
_diffrn_reflns_point_group_measured_fraction_max   0.988
_diffrn_reflns_point_group_measured_fraction_full  0.989
_reflns_number_total              6453
_reflns_number_gt                 5944
_reflns_threshold_expression      'I > 2\s(I)'

_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 1.171.40.67a (Rigaku OD, 2019)'
_computing_cell_refinement        'CrysAlisPro 1.171.40.67a (Rigaku OD, 2019)'
_computing_data_reduction         'CrysAlisPro 1.171.40.67a (Rigaku OD, 2019)'
_computing_structure_solution     'SHELXT (Sheldrick, 2015'
_computing_structure_refinement   'SHELXL-2014/7 (Sheldrick, 2014)'
_computing_molecular_graphics     'ORTEP-3 (Farrugia, 1997)'
_computing_publication_material   'SHELXL-2014/7 (Sheldrick, 2014)'
_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/[\s^2^(Fo^2^)+(0.0522P)^2^+2.4244P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary      dual
_atom_sites_solution_secondary    ?
_atom_sites_solution_hydrogens    mixed
_refine_ls_hydrogen_treatment     mixed
_refine_ls_extinction_method      'SHELXL-2014/7 (Sheldrick 2014'
_refine_ls_extinction_coef        0.00011(3)
_refine_ls_extinction_expression
 'Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^'
_refine_ls_number_reflns          6453
_refine_ls_number_parameters      473
_refine_ls_number_restraints      0
_refine_ls_R_factor_all           0.0469
_refine_ls_R_factor_gt            0.0425
_refine_ls_wR_factor_ref          0.1125
_refine_ls_wR_factor_gt           0.1095
_refine_ls_goodness_of_fit_ref    1.081
_refine_ls_restrained_S_all       1.081
_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_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
C1 C 0.17035(7) 0.7284(2) 0.33855(3) 0.0357(3) Uani 1 1 d . . . . .
H1A H 0.1923 0.6140 0.3422 0.043 Uiso 1 1 calc R U . . .
H1B H 0.1726 0.8031 0.3579 0.043 Uiso 1 1 calc R U . . .
C2 C 0.19758(7) 0.8319(2) 0.31164(3) 0.0376(3) Uani 1 1 d . . . . .
H2A H 0.1786 0.9528 0.3097 0.045 Uiso 1 1 calc R U . . .
H2B H 0.2394 0.8524 0.3162 0.045 Uiso 1 1 calc R U . . .
C3 C 0.13076(7) 0.7025(3) 0.27487(4) 0.0448(4) Uani 1 1 d . . . . .
H3A H 0.1275 0.6320 0.2551 0.054 Uiso 1 1 calc R U . . .
H3B H 0.1109 0.8212 0.2718 0.054 Uiso 1 1 calc R U . . .
C4 C 0.10117(7) 0.5986(2) 0.30118(4) 0.0407(4) Uani 1 1 d . . . . .
H4A H 0.0589 0.5898 0.2966 0.049 Uiso 1 1 calc R U . . .
H4B H 0.1170 0.4729 0.3019 0.049 Uiso 1 1 calc R U . . .
C5 C 0.07432(7) 0.6274(2) 0.35670(3) 0.0335(3) Uani 1 1 d . . . . .
C6 C 0.09376(7) 0.6343(2) 0.38763(3) 0.0340(3) Uani 1 1 d . . . . .
H6 H 0.1324 0.6748 0.3916 0.041 Uiso 1 1 calc R U . . .
C7 C 0.05847(7) 0.5839(2) 0.41337(3) 0.0341(3) Uani 1 1 d . . . . .
C8 C 0.00136(7) 0.5178(2) 0.40629(4) 0.0350(3) Uani 1 1 d . . . . .
C9 C -0.01853(7) 0.5152(2) 0.37522(4) 0.0374(3) Uani 1 1 d . . . . .
H9 H -0.0571 0.4748 0.3710 0.045 Uiso 1 1 calc R U . . .
C10 C 0.01589(7) 0.5687(2) 0.35090(4) 0.0374(3) Uani 1 1 d . . . . .
H10 H 0.0011 0.5671 0.3301 0.045 Uiso 1 1 calc R U . . .
C11 C 0.13824(7) 0.6510(2) 0.45134(4) 0.0369(3) Uani 1 1 d . . . . .
H11A H 0.1458 0.7756 0.4433 0.044 Uiso 1 1 calc R U . . .
H11B H 0.1670 0.5668 0.4418 0.044 Uiso 1 1 calc R U . . .
C12 C 0.14440(7) 0.6487(2) 0.48681(4) 0.0411(4) Uani 1 1 d . . . . .
H12A H 0.1161 0.7358 0.4960 0.049 Uiso 1 1 calc R U . . .
H12B H 0.1345 0.5252 0.4947 0.049 Uiso 1 1 calc R U . . .
C13 C 0.20500(8) 0.6985(3) 0.49783(4) 0.0479(4) Uani 1 1 d . . . . .
H13A H 0.2154 0.8203 0.4894 0.057 Uiso 1 1 calc R U . . .
H13B H 0.2332 0.6089 0.4893 0.057 Uiso 1 1 calc R U . . .
C14 C 0.21015(10) 0.7019(4) 0.53362(5) 0.0753(7) Uani 1 1 d . . . . .
H14A H 0.2502 0.7336 0.5396 0.113 Uiso 1 1 calc R U . . .
H14B H 0.2004 0.5811 0.5421 0.113 Uiso 1 1 calc R U . . .
H14C H 0.1832 0.7930 0.5422 0.113 Uiso 1 1 calc R U . . .
C15 C 0.23594(6) 0.7055(2) 0.26087(3) 0.0335(3) Uani 1 1 d . . . . .
C16 C 0.22510(7) 0.6514(2) 0.22998(3) 0.0339(3) Uani 1 1 d . . . . .
H16 H 0.1858 0.6477 0.2229 0.041 Uiso 1 1 calc R U . . .
C17 C 0.26945(7) 0.6023(2) 0.20888(3) 0.0340(3) Uani 1 1 d . . . . .
C18 C 0.32825(7) 0.6095(2) 0.22033(4) 0.0368(3) Uani 1 1 d . . . . .

C19 C 0.33887(7) 0.6737(2) 0.25085(4) 0.0381(3) Uani 1 1 d . . . . .
H19 H 0.3781 0.6826 0.2580 0.046 Uiso 1 1 calc R U . . .
C20 C 0.29524(7) 0.7236(2) 0.27060(4) 0.0359(3) Uani 1 1 d . . . . .
H20 H 0.3041 0.7705 0.2909 0.043 Uiso 1 1 calc R U . . .
C21 C 0.19746(7) 0.5275(2) 0.16726(4) 0.0379(3) Uani 1 1 d . . . . .
H21A H 0.1769 0.6465 0.1676 0.045 Uiso 1 1 calc R U . . .
H21B H 0.1757 0.4418 0.1809 0.045 Uiso 1 1 calc R U . . .
C22 C 0.19874(7) 0.4541(2) 0.13400(4) 0.0399(4) Uani 1 1 d . . . . .
H22A H 0.2218 0.3399 0.1336 0.048 Uiso 1 1 calc R U . . .
H22B H 0.2185 0.5439 0.1202 0.048 Uiso 1 1 calc R U . . .
C23 C 0.13807(8) 0.4157(3) 0.12102(4) 0.0467(4) Uani 1 1 d . . . . .
H23A H 0.1174 0.3320 0.1355 0.056 Uiso 1 1 calc R U . . .
H23B H 0.1158 0.5316 0.1202 0.056 Uiso 1 1 calc R U . . .
C24 C 0.13919(9) 0.3314(3) 0.08844(4) 0.0556(5) Uani 1 1 d . . . . .
H24A H 0.0992 0.3081 0.0814 0.083 Uiso 1 1 calc R U . . .
H24B H 0.1608 0.2160 0.0891 0.083 Uiso 1 1 calc R U . . .
H24C H 0.1583 0.4156 0.0738 0.083 Uiso 1 1 calc R U . . .
N1 N 0.10925(5) 0.68476(18) 0.33182(3) 0.0340(3) Uani 1 1 d . . . . .
N2 N 0.19192(6) 0.7339(2) 0.28205(3) 0.0402(3) Uani 1 1 d . . . . .
N3 N 0.07943(6) 0.59451(19) 0.44295(3) 0.0374(3) Uani 1 1 d . . . . .
H1N H 0.0559(8) 0.550(3) 0.4589(4) 0.045 Uiso 1 1 d . U . . .
N4 N 0.25632(6) 0.55064(19) 0.17927(3) 0.0372(3) Uani 1 1 d . . . . .
H2N H 0.2856(9) 0.513(3) 0.1679(4) 0.045 Uiso 1 1 d . U . . .
N5 N -0.03632(6) 0.44496(19) 0.42960(3) 0.0398(3) Uani 1 1 d . . . . .
N6 N 0.37633(6) 0.5471(2) 0.20271(3) 0.0434(3) Uani 1 1 d . . . . .
O1 O -0.02358(5) 0.46248(17) 0.45809(3) 0.0460(3) Uani 1 1 d . . . . .
O2 O -0.08105(5) 0.36218(19) 0.42151(3) 0.0540(3) Uani 1 1 d . . . . .
O3 O 0.36934(5) 0.49534(19) 0.17479(3) 0.0509(3) Uani 1 1 d . . . . .
O4 O 0.42559(5) 0.5422(2) 0.21515(3) 0.0554(3) Uani 1 1 d . . . . .
C25 C 0.54022(7) 0.1430(2) -0.00554(3) 0.0368(3) Uani 1 1 d . . . . .
H25A H 0.5801 0.1835 -0.0112 0.044 Uiso 1 1 calc R U . . .
H25B H 0.5160 0.2533 -0.0023 0.044 Uiso 1 1 calc R U . . .
C26 C 0.48482(7) -0.0323(2) 0.03215(4) 0.0357(3) Uani 1 1 d . . . . .
H26A H 0.4585 0.0712 0.0370 0.043 Uiso 1 1 calc R U . . .
H26B H 0.4880 -0.1095 0.0512 0.043 Uiso 1 1 calc R U . . .
C27 C 0.57950(7) 0.0946(2) 0.04731(3) 0.0337(3) Uani 1 1 d . . . . .
C28 C 0.56323(7) 0.0892(2) 0.07859(3) 0.0344(3) Uani 1 1 d . . . . .
H28 H 0.5249 0.0497 0.0838 0.041 Uiso 1 1 calc R U . . .
C29 C 0.60129(7) 0.1400(2) 0.10305(3) 0.0346(3) Uani 1 1 d . . . . .
C30 C 0.65768(7) 0.2029(2) 0.09440(4) 0.0366(3) Uani 1 1 d . . . . .
C31 C 0.67469(7) 0.2023(2) 0.06270(4) 0.0379(3) Uani 1 1 d . . . . .
H31 H 0.7132 0.2389 0.0573 0.045 Uiso 1 1 calc R U . . .
C32 C 0.63735(7) 0.1505(2) 0.03954(4) 0.0372(3) Uani 1 1 d . . . . .
H32 H 0.6497 0.1515 0.0182 0.045 Uiso 1 1 calc R U . . .
C33 C 0.52527(7) 0.0777(2) 0.14323(4) 0.0387(4) Uani 1 1 d . . . . .
H33A H 0.5170 -0.0475 0.1356 0.046 Uiso 1 1 calc R U . . .

H33B H 0.4954 0.1610 0.1344 0.046 Uiso 1 1 calc R U . . .
C34 C 0.52308(7) 0.0822(2) 0.17875(4) 0.0421(4) Uani 1 1 d . . . . .
H34A H 0.5283 0.2099 0.1859 0.051 Uiso 1 1 calc R U . . .
H34B H 0.5561 0.0097 0.1871 0.051 Uiso 1 1 calc R U . . .
C35 C 0.46688(8) 0.0087(3) 0.19250(4) 0.0483(4) Uani 1 1 d . . . . .
H35A H 0.4347 0.0945 0.1878 0.058 Uiso 1 1 calc R U . . .
H35B H 0.4575 -0.1100 0.1825 0.058 Uiso 1 1 calc R U . . .
C36 C 0.47156(9) -0.0166(3) 0.22756(4) 0.0567(5) Uani 1 1 d . . . . .
H36A H 0.4350 -0.0676 0.2356 0.085 Uiso 1 1 calc R U . . .
H36B H 0.4790 0.1017 0.2376 0.085 Uiso 1 1 calc R U . . .
H36C H 0.5037 -0.1004 0.2323 0.085 Uiso 1 1 calc R U . . .
N7 N 0.54246(5) 0.03728(17) 0.02366(3) 0.0341(3) Uani 1 1 d . . . . .
N8 N 0.58287(6) 0.13418(19) 0.13306(3) 0.0390(3) Uani 1 1 d . . . . .
H3N H 0.6101(8) 0.175(3) 0.1485(4) 0.047 Uiso 1 1 d . U . . .
N9 N 0.69815(6) 0.2716(2) 0.11643(3) 0.0426(3) Uani 1 1 d . . . . .
O5 O 0.68723(5) 0.25859(17) 0.14532(3) 0.0474(3) Uani 1 1 d . . . . .
O6 O 0.74370(5) 0.34621(19) 0.10699(3) 0.0538(3) Uani 1 1 d . . . . .

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
C1 0.0339(8) 0.0368(8) 0.0364(8) 0.0014(6) -0.0039(6) -0.0017(6)
C2 0.0378(8) 0.0410(8) 0.0341(8) -0.0012(6) -0.0007(6) -0.0022(7)
C3 0.0322(8) 0.0670(12) 0.0352(8) -0.0059(8) 0.0001(6) -0.0006(8)
C4 0.0330(8) 0.0500(10) 0.0391(8) -0.0081(7) -0.0011(6) -0.0012(7)
C5 0.0346(8) 0.0281(7) 0.0379(8) 0.0006(6) -0.0012(6) 0.0039(6)
C6 0.0317(7) 0.0321(7) 0.0381(8) -0.0014(6) -0.0013(6) 0.0023(6)
C7 0.0353(8) 0.0289(7) 0.0380(8) -0.0020(6) -0.0001(6) 0.0034(6)
C8 0.0338(8) 0.0319(8) 0.0394(8) 0.0014(6) 0.0018(6) 0.0028(6)
C9 0.0326(8) 0.0345(8) 0.0451(9) -0.0002(7) -0.0021(6) 0.0025(6)
C10 0.0347(8) 0.0370(8) 0.0404(8) -0.0004(6) -0.0037(6) 0.0038(7)
C11 0.0370(8) 0.0357(8) 0.0381(8) -0.0009(6) 0.0008(6) 0.0013(7)
C12 0.0391(9) 0.0458(9) 0.0384(8) -0.0012(7) -0.0004(7) 0.0015(7)
C13 0.0412(9) 0.0551(11) 0.0473(9) 0.0013(8) -0.0042(7) 0.0015(8)
C14 0.0584(13) 0.115(2) 0.0526(11) -0.0108(12) -0.0119(10) -0.0043(13)
C15 0.0343(8) 0.0309(7) 0.0353(7) 0.0053(6) 0.0011(6) -0.0011(6)
C16 0.0313(7) 0.0345(8) 0.0358(8) 0.0039(6) 0.0003(6) -0.0009(6)
C17 0.0374(8) 0.0306(7) 0.0341(7) 0.0064(6) 0.0020(6) -0.0023(6)
C18 0.0326(8) 0.0395(8) 0.0384(8) 0.0068(6) 0.0046(6) -0.0005(6)
C19 0.0327(8) 0.0431(9) 0.0385(8) 0.0082(7) -0.0012(6) -0.0035(7)
C20 0.0359(8) 0.0376(8) 0.0341(7) 0.0044(6) -0.0020(6) -0.0033(7)

C21 0.0371(8) 0.0400(8) 0.0364(8) 0.0024(6) 0.0012(6) -0.0005(7)
C22 0.0401(9) 0.0429(9) 0.0367(8) 0.0012(7) 0.0018(6) -0.0005(7)
C23 0.0423(9) 0.0544(10) 0.0432(9) -0.0020(8) -0.0001(7) -0.0021(8)
C24 0.0527(11) 0.0697(13) 0.0444(10) -0.0039(9) -0.0039(8) -0.0094(10)
N1 0.0326(7) 0.0376(7) 0.0318(6) 0.0003(5) -0.0015(5) 0.0005(5)
N2 0.0325(7) 0.0528(8) 0.0352(7) -0.0052(6) 0.0003(5) -0.0017(6)
N3 0.0363(7) 0.0398(7) 0.0359(7) 0.0004(6) 0.0012(5) -0.0009(6)
N4 0.0344(7) 0.0431(7) 0.0341(6) 0.0014(6) 0.0040(5) 0.0001(6)
N5 0.0372(7) 0.0391(7) 0.0432(7) 0.0018(6) 0.0007(6) 0.0012(6)
N6 0.0355(7) 0.0533(9) 0.0415(8) 0.0041(6) 0.0048(6) -0.0001(6)
O1 0.0456(7) 0.0527(7) 0.0398(6) 0.0005(5) 0.0048(5) -0.0034(6)
O2 0.0417(7) 0.0652(8) 0.0552(7) 0.0064(6) -0.0017(5) -0.0160(6)
O3 0.0407(7) 0.0712(9) 0.0408(6) -0.0042(6) 0.0075(5) 0.0005(6)
O4 0.0325(6) 0.0828(10) 0.0510(7) 0.0009(6) 0.0009(5) 0.0045(6)
C25 0.0381(8) 0.0366(8) 0.0357(8) 0.0028(6) -0.0016(6) -0.0032(7)
C26 0.0355(8) 0.0380(8) 0.0336(7) 0.0024(6) 0.0004(6) -0.0019(7)
C27 0.0352(8) 0.0296(7) 0.0361(8) 0.0003(6) -0.0033(6) 0.0023(6)
C28 0.0331(8) 0.0328(7) 0.0373(8) 0.0004(6) -0.0008(6) 0.0008(6)
C29 0.0381(8) 0.0292(7) 0.0364(8) 0.0011(6) -0.0022(6) 0.0049(6)
C30 0.0338(8) 0.0344(8) 0.0415(8) -0.0002(6) -0.0061(6) 0.0026(7)
C31 0.0321(8) 0.0353(8) 0.0463(9) 0.0010(7) -0.0011(6) 0.0017(6)
C32 0.0369(8) 0.0358(8) 0.0389(8) 0.0013(6) 0.0016(6) 0.0028(7)
C33 0.0391(8) 0.0377(8) 0.0394(8) 0.0008(7) -0.0020(6) 0.0017(7)
C34 0.0415(9) 0.0447(9) 0.0401(8) 0.0004(7) -0.0016(7) 0.0031(7)
C35 0.0437(10) 0.0524(10) 0.0488(10) -0.0047(8) -0.0001(8) 0.0002(8)
C36 0.0505(11) 0.0704(13) 0.0491(10) -0.0010(9) 0.0099(8) 0.0022(10)
N7 0.0349(7) 0.0363(7) 0.0312(6) 0.0021(5) -0.0010(5) -0.0024(6)
N8 0.0385(7) 0.0420(7) 0.0365(7) -0.0003(6) -0.0036(6) -0.0013(6)
N9 0.0395(8) 0.0431(8) 0.0451(8) 0.0016(6) -0.0086(6) 0.0007(6)
O5 0.0467(7) 0.0555(7) 0.0401(6) -0.0016(5) -0.0090(5) -0.0012(6)
O6 0.0401(7) 0.0633(8) 0.0580(7) 0.0066(6) -0.0088(6) -0.0129(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
C1 N1 1.4649(19) . ?
C1 C2 1.508(2) . ?
C1 H1A 0.9900 . ?
C1 H1B 0.9900 . ?
C2 N2 1.4549(19) . ?
C2 H2A 0.9900 . ?
C2 H2B 0.9900 . ?
C3 N2 1.453(2) . ?
C3 C4 1.514(2) . ?
C3 H3A 0.9900 . ?
C3 H3B 0.9900 . ?
C4 N1 1.4604(19) . ?
C4 H4A 0.9900 . ?
C4 H4B 0.9900 . ?
C5 C6 1.391(2) . ?
C5 N1 1.3920(19) . ?
C5 C10 1.428(2) . ?
C6 C7 1.411(2) . ?
C6 H6 0.9500 . ?
C7 N3 1.3495(19) . ?
C7 C8 1.428(2) . ?
C8 C9 1.399(2) . ?
C8 N5 1.419(2) . ?
C9 C10 1.359(2) . ?
C9 H9 0.9500 . ?
C10 H10 0.9500 . ?
C11 N3 1.455(2) . ?
C11 C12 1.516(2) . ?
C11 H11A 0.9900 . ?
C11 H11B 0.9900 . ?
C12 C13 1.511(2) . ?
C12 H12A 0.9900 . ?
C12 H12B 0.9900 . ?
C13 C14 1.528(3) . ?
C13 H13A 0.9900 . ?
C13 H13B 0.9900 . ?
C14 H14A 0.9800 . ?
C14 H14B 0.9800 . ?
C14 H14C 0.9800 . ?
C15 N2 1.3688(19) . ?
C15 C16 1.395(2) . ?
C15 C20 1.427(2) . ?
C16 C17 1.403(2) . ?
C16 H16 0.9500 . ?

C17 N4 1.350(2) . ?
C17 C18 1.434(2) . ?
C18 C19 1.403(2) . ?
C18 N6 1.409(2) . ?
C19 C20 1.356(2) . ?
C19 H19 0.9500 . ?
C20 H20 0.9500 . ?
C21 N4 1.453(2) . ?
C21 C22 1.514(2) . ?
C21 H21A 0.9900 . ?
C21 H21B 0.9900 . ?
C22 C23 1.523(2) . ?
C22 H22A 0.9900 . ?
C22 H22B 0.9900 . ?
C23 C24 1.518(2) . ?
C23 H23A 0.9900 . ?
C23 H23B 0.9900 . ?
C24 H24A 0.9800 . ?
C24 H24B 0.9800 . ?
C24 H24C 0.9800 . ?
N3 H1N 0.925(19) . ?
N4 H2N 0.87(2) . ?
N5 O2 1.2397(18) . ?
N5 O1 1.2537(17) . ?
N6 O4 1.2477(18) . ?
N6 O3 1.2569(18) . ?
C25 N7 1.4650(19) . ?
C25 C26 1.506(2) 5_655 ?
C25 H25A 0.9900 . ?
C25 H25B 0.9900 . ?
C26 N7 1.4615(19) . ?
C26 C25 1.506(2) 5_655 ?
C26 H26A 0.9900 . ?
C26 H26B 0.9900 . ?
C27 N7 1.3816(19) . ?
C27 C28 1.383(2) . ?
C27 C32 1.427(2) . ?
C28 C29 1.408(2) . ?
C28 H28 0.9500 . ?
C29 N8 1.346(2) . ?
C29 C30 1.421(2) . ?
C30 C31 1.404(2) . ?
C30 N9 1.411(2) . ?
C31 C32 1.359(2) . ?
C31 H31 0.9500 . ?
C32 H32 0.9500 . ?

C33 N8 1.450(2) . ?
C33 C34 1.513(2) . ?
C33 H33A 0.9900 . ?
C33 H33B 0.9900 . ?
C34 C35 1.514(2) . ?
C34 H34A 0.9900 . ?
C34 H34B 0.9900 . ?
C35 C36 1.507(3) . ?
C35 H35A 0.9900 . ?
C35 H35B 0.9900 . ?
C36 H36A 0.9800 . ?
C36 H36B 0.9800 . ?
C36 H36C 0.9800 . ?
N8 H3N 0.95(2) . ?
N9 O6 1.2452(19) . ?
N9 O5 1.2582(18) . ?

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 C1 C2 110.91(12) . . ?
N1 C1 H1A 109.5 . . ?
C2 C1 H1A 109.5 . . ?
N1 C1 H1B 109.5 . . ?
C2 C1 H1B 109.5 . . ?
H1A C1 H1B 108.0 . . ?
N2 C2 C1 111.87(13) . . ?
N2 C2 H2A 109.2 . . ?
C1 C2 H2A 109.2 . . ?
N2 C2 H2B 109.2 . . ?
C1 C2 H2B 109.2 . . ?
H2A C2 H2B 107.9 . . ?
N2 C3 C4 110.87(13) . . ?
N2 C3 H3A 109.5 . . ?
C4 C3 H3A 109.5 . . ?
N2 C3 H3B 109.5 . . ?
C4 C3 H3B 109.5 . . ?
H3A C3 H3B 108.1 . . ?
N1 C4 C3 112.72(14) . . ?
N1 C4 H4A 109.0 . . ?
C3 C4 H4A 109.0 . . ?

N1 C4 H4B 109.0 . . ?
C3 C4 H4B 109.0 . . ?
H4A C4 H4B 107.8 . . ?
C6 C5 N1 121.64(14) . . ?
C6 C5 C10 118.35(14) . . ?
N1 C5 C10 119.92(13) . . ?
C5 C6 C7 122.78(14) . . ?
C5 C6 H6 118.6 . . ?
C7 C6 H6 118.6 . . ?
N3 C7 C6 120.31(14) . . ?
N3 C7 C8 122.87(14) . . ?
C6 C7 C8 116.80(13) . . ?
C9 C8 N5 117.20(14) . . ?
C9 C8 C7 120.20(14) . . ?
N5 C8 C7 122.54(14) . . ?
C10 C9 C8 121.76(15) . . ?
C10 C9 H9 119.1 . . ?
C8 C9 H9 119.1 . . ?
C9 C10 C5 119.99(14) . . ?
C9 C10 H10 120.0 . . ?
C5 C10 H10 120.0 . . ?
N3 C11 C12 109.12(13) . . ?
N3 C11 H11A 109.9 . . ?
C12 C11 H11A 109.9 . . ?
N3 C11 H11B 109.9 . . ?
C12 C11 H11B 109.9 . . ?
H11A C11 H11B 108.3 . . ?
C13 C12 C11 113.07(14) . . ?
C13 C12 H12A 109.0 . . ?
C11 C12 H12A 109.0 . . ?
C13 C12 H12B 109.0 . . ?
C11 C12 H12B 109.0 . . ?
H12A C12 H12B 107.8 . . ?
C12 C13 C14 112.60(16) . . ?
C12 C13 H13A 109.1 . . ?
C14 C13 H13A 109.1 . . ?
C12 C13 H13B 109.1 . . ?
C14 C13 H13B 109.1 . . ?
H13A C13 H13B 107.8 . . ?
C13 C14 H14A 109.5 . . ?
C13 C14 H14B 109.5 . . ?
H14A C14 H14B 109.5 . . ?
C13 C14 H14C 109.5 . . ?
H14A C14 H14C 109.5 . . ?
H14B C14 H14C 109.5 . . ?
N2 C15 C16 122.14(14) . . ?

N2 C15 C20 119.82(13) . . ?
C16 C15 C20 117.99(14) . . ?
C15 C16 C17 123.12(14) . . ?
C15 C16 H16 118.4 . . ?
C17 C16 H16 118.4 . . ?
N4 C17 C16 120.50(14) . . ?
N4 C17 C18 122.48(14) . . ?
C16 C17 C18 117.02(13) . . ?
C19 C18 N6 117.75(14) . . ?
C19 C18 C17 119.33(14) . . ?
N6 C18 C17 122.85(14) . . ?
C20 C19 C18 122.40(15) . . ?
C20 C19 H19 118.8 . . ?
C18 C19 H19 118.8 . . ?
C19 C20 C15 119.85(14) . . ?
C19 C20 H20 120.1 . . ?
C15 C20 H20 120.1 . . ?
N4 C21 C22 110.63(13) . . ?
N4 C21 H21A 109.5 . . ?
C22 C21 H21A 109.5 . . ?
N4 C21 H21B 109.5 . . ?
C22 C21 H21B 109.5 . . ?
H21A C21 H21B 108.1 . . ?
C21 C22 C23 112.76(13) . . ?
C21 C22 H22A 109.0 . . ?
C23 C22 H22A 109.0 . . ?
C21 C22 H22B 109.0 . . ?
C23 C22 H22B 109.0 . . ?
H22A C22 H22B 107.8 . . ?
C24 C23 C22 113.01(15) . . ?
C24 C23 H23A 109.0 . . ?
C22 C23 H23A 109.0 . . ?
C24 C23 H23B 109.0 . . ?
C22 C23 H23B 109.0 . . ?
H23A C23 H23B 107.8 . . ?
C23 C24 H24A 109.5 . . ?
C23 C24 H24B 109.5 . . ?
H24A C24 H24B 109.5 . . ?
C23 C24 H24C 109.5 . . ?
H24A C24 H24C 109.5 . . ?
H24B C24 H24C 109.5 . . ?
C5 N1 C4 118.41(13) . . ?
C5 N1 C1 117.84(12) . . ?
C4 N1 C1 112.96(12) . . ?
C15 N2 C3 123.28(13) . . ?
C15 N2 C2 125.36(13) . . ?

C3 N2 C2 110.25(12) . . ?
C7 N3 C11 125.12(13) . . ?
C7 N3 H1N 117.2(12) . . ?
C11 N3 H1N 117.3(11) . . ?
C17 N4 C21 124.63(13) . . ?
C17 N4 H2N 115.8(12) . . ?
C21 N4 H2N 118.9(12) . . ?
O2 N5 O1 120.74(13) . . ?
O2 N5 C8 119.54(13) . . ?
O1 N5 C8 119.71(13) . . ?
O4 N6 O3 120.53(13) . . ?
O4 N6 C18 119.42(14) . . ?
O3 N6 C18 120.05(13) . . ?
N7 C25 C26 111.51(13) . 5_655 ?
N7 C25 H25A 109.3 . . ?
C26 C25 H25A 109.3 5_655 . ?
N7 C25 H25B 109.3 . . ?
C26 C25 H25B 109.3 5_655 . ?
H25A C25 H25B 108.0 . . ?
N7 C26 C25 110.26(12) . 5_655 ?
N7 C26 H26A 109.6 . . ?
C25 C26 H26A 109.6 5_655 . ?
N7 C26 H26B 109.6 . . ?
C25 C26 H26B 109.6 5_655 . ?
H26A C26 H26B 108.1 . . ?
N7 C27 C28 121.77(14) . . ?
N7 C27 C32 119.32(13) . . ?
C28 C27 C32 118.80(14) . . ?
C27 C28 C29 122.47(14) . . ?
C27 C28 H28 118.8 . . ?
C29 C28 H28 118.8 . . ?
N8 C29 C28 119.91(14) . . ?
N8 C29 C30 122.79(14) . . ?
C28 C29 C30 117.25(14) . . ?
C31 C30 N9 117.17(14) . . ?
C31 C30 C29 120.03(14) . . ?
N9 C30 C29 122.79(14) . . ?
C32 C31 C30 121.50(15) . . ?
C32 C31 H31 119.3 . . ?
C30 C31 H31 119.3 . . ?
C31 C32 C27 119.84(14) . . ?
C31 C32 H32 120.1 . . ?
C27 C32 H32 120.1 . . ?
N8 C33 C34 108.80(13) . . ?
N8 C33 H33A 109.9 . . ?
C34 C33 H33A 109.9 . . ?

N8 C33 H33B 109.9 . . ?
C34 C33 H33B 109.9 . . ?
H33A C33 H33B 108.3 . . ?
C33 C34 C35 113.99(14) . . ?
C33 C34 H34A 108.8 . . ?
C35 C34 H34A 108.8 . . ?
C33 C34 H34B 108.8 . . ?
C35 C34 H34B 108.8 . . ?
H34A C34 H34B 107.6 . . ?
C36 C35 C34 111.44(15) . . ?
C36 C35 H35A 109.3 . . ?
C34 C35 H35A 109.3 . . ?
C36 C35 H35B 109.3 . . ?
C34 C35 H35B 109.3 . . ?
H35A C35 H35B 108.0 . . ?
C35 C36 H36A 109.5 . . ?
C35 C36 H36B 109.5 . . ?
H36A C36 H36B 109.5 . . ?
C35 C36 H36C 109.5 . . ?
H36A C36 H36C 109.5 . . ?
H36B C36 H36C 109.5 . . ?
C27 N7 C26 118.78(12) . . ?
C27 N7 C25 118.60(13) . . ?
C26 N7 C25 111.23(12) . . ?
C29 N8 C33 125.31(13) . . ?
C29 N8 H3N 116.1(11) . . ?
C33 N8 H3N 118.6(11) . . ?
O6 N9 O5 121.01(13) . . ?
O6 N9 C30 119.56(14) . . ?
O5 N9 C30 119.42(14) . . ?

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
 _geom_torsion_site_symmetry_1
 _geom_torsion_site_symmetry_2
 _geom_torsion_site_symmetry_3
 _geom_torsion_site_symmetry_4
 _geom_torsion_publ_flag
N1 C1 C2 N2 -55.17(17) . . . . ?
N2 C3 C4 N1 53.23(19) . . . . ?
N1 C5 C6 C7 177.44(14) . . . . ?
C10 C5 C6 C7 0.8(2) . . . . ?

C5 C6 C7 N3 -179.52(14) . . . . ?
C5 C6 C7 C8 2.1(2) . . . . ?
N3 C7 C8 C9 178.08(14) . . . . ?
C6 C7 C8 C9 -3.6(2) . . . . ?
N3 C7 C8 N5 -4.9(2) . . . . ?
C6 C7 C8 N5 173.36(14) . . . . ?
N5 C8 C9 C10 -174.93(14) . . . . ?
C7 C8 C9 C10 2.2(2) . . . . ?
C8 C9 C10 C5 0.9(2) . . . . ?
C6 C5 C10 C9 -2.4(2) . . . . ?
N1 C5 C10 C9 -179.06(14) . . . . ?
N3 C11 C12 C13 177.73(14) . . . . ?
C11 C12 C13 C14 178.10(18) . . . . ?
N2 C15 C16 C17 -172.81(14) . . . . ?
C20 C15 C16 C17 4.5(2) . . . . ?
C15 C16 C17 N4 179.85(14) . . . . ?
C15 C16 C17 C18 0.0(2) . . . . ?
N4 C17 C18 C19 176.63(14) . . . . ?
C16 C17 C18 C19 -3.5(2) . . . . ?
N4 C17 C18 N6 -6.2(2) . . . . ?
C16 C17 C18 N6 173.67(14) . . . . ?
N6 C18 C19 C20 -174.84(15) . . . . ?
C17 C18 C19 C20 2.5(2) . . . . ?
C18 C19 C20 C15 2.2(2) . . . . ?
N2 C15 C20 C19 171.83(15) . . . . ?
C16 C15 C20 C19 -5.6(2) . . . . ?
N4 C21 C22 C23 176.30(14) . . . . ?
C21 C22 C23 C24 -176.52(15) . . . . ?
C6 C5 N1 C4 150.54(14) . . . . ?
C10 C5 N1 C4 -32.9(2) . . . . ?
C6 C5 N1 C1 8.6(2) . . . . ?
C10 C5 N1 C1 -174.79(13) . . . . ?
C3 C4 N1 C5 166.15(13) . . . . ?
C3 C4 N1 C1 -50.16(18) . . . . ?
C2 C1 N1 C5 -165.64(13) . . . . ?
C2 C1 N1 C4 50.45(17) . . . . ?
C16 C15 N2 C3 3.0(2) . . . . ?
C20 C15 N2 C3 -174.32(15) . . . . ?
C16 C15 N2 C2 -163.85(15) . . . . ?
C20 C15 N2 C2 18.9(2) . . . . ?
C4 C3 N2 C15 134.27(16) . . . . ?
C4 C3 N2 C2 -57.17(19) . . . . ?
C1 C2 N2 C15 -132.70(16) . . . . ?
C1 C2 N2 C3 59.04(18) . . . . ?
C6 C7 N3 C11 -1.8(2) . . . . ?
C8 C7 N3 C11 176.39(14) . . . . ?

C12 C11 N3 C7 178.85(14) . . . . ?
C16 C17 N4 C21 -4.8(2) . . . . ?
C18 C17 N4 C21 175.03(14) . . . . ?
C22 C21 N4 C17 -175.44(14) . . . . ?
C9 C8 N5 O2 9.1(2) . . . . ?
C7 C8 N5 O2 -167.96(15) . . . . ?
C9 C8 N5 O1 -172.00(14) . . . . ?
C7 C8 N5 O1 10.9(2) . . . . ?
C19 C18 N6 O4 3.6(2) . . . . ?
C17 C18 N6 O4 -173.61(15) . . . . ?
C19 C18 N6 O3 -177.05(15) . . . . ?
C17 C18 N6 O3 5.7(2) . . . . ?
N7 C27 C28 C29 177.32(14) . . . . ?
C32 C27 C28 C29 1.2(2) . . . . ?
C27 C28 C29 N8 179.17(14) . . . . ?
C27 C28 C29 C30 1.6(2) . . . . ?
N8 C29 C30 C31 178.77(14) . . . . ?
C28 C29 C30 C31 -3.7(2) . . . . ?
N8 C29 C30 N9 -2.7(2) . . . . ?
C28 C29 C30 N9 174.78(14) . . . . ?
N9 C30 C31 C32 -175.47(15) . . . . ?
C29 C30 C31 C32 3.1(2) . . . . ?
C30 C31 C32 C27 -0.2(2) . . . . ?
N7 C27 C32 C31 -178.15(14) . . . . ?
C28 C27 C32 C31 -1.9(2) . . . . ?
N8 C33 C34 C35 -174.50(14) . . . . ?
C33 C34 C35 C36 169.24(15) . . . . ?
C28 C27 N7 C26 -0.5(2) . . . . ?
C32 C27 N7 C26 175.55(13) . . . . ?
C28 C27 N7 C25 139.81(15) . . . . ?
C32 C27 N7 C25 -44.1(2) . . . . ?
C25 C26 N7 C27 -161.18(13) 5_655 . . . ?
C25 C26 N7 C25 55.76(18) 5_655 . . . ?
C26 C25 N7 C27 160.40(13) 5_655 . . . ?
C26 C25 N7 C26 -56.47(18) 5_655 . . . ?
C28 C29 N8 C33 0.3(2) . . . . ?
C30 C29 N8 C33 177.69(14) . . . . ?
C34 C33 N8 C29 177.39(14) . . . . ?
C31 C30 N9 O6 7.8(2) . . . . ?
C29 C30 N9 O6 -170.72(15) . . . . ?
C31 C30 N9 O5 -173.14(14) . . . . ?
C29 C30 N9 O5 8.3(2) . . . . ?

loop_
 _geom_hbond_atom_site_label_D
 _geom_hbond_atom_site_label_H

```
 _geom_hbond_atom_site_label_A
 _geom_hbond_distance_DH
 _geom_hbond_distance_HA
 _geom_hbond_distance_DA
 _geom_hbond_angle_DHA
 _geom_hbond_site_symmetry_A
N3 H1N O1  0.925(19) 1.933(19) 2.6317(18) 130.7(15) .
N4 H2N O3  0.87(2) 1.95(2) 2.6291(18) 134.2(16) .
N8 H3N O5  0.95(2) 1.877(19) 2.6126(18) 131.8(15) .
C1 H1B O6  0.99 2.45 3.1616(19) 128.2 4_655
C2 H2B O5  0.99 2.45 3.2580(19) 138.9 4_655
C9 H9 O3  0.95 2.58 3.3403(19) 137.1 6_556
C10 H10 O4  0.95 2.59 3.496(2) 158.7 6_556
C25 H25B O1  0.99 2.59 3.418(2) 141.4 6_656

_refine_diff_density_max    0.197
_refine_diff_density_min   -0.183
_refine_diff_density_rms    0.033

_shelx_res_file
;

    db1.res created by SHELXL-2014/7


TITL db1_a.res Pbca
CELL  1.54178   22.9241    7.3205   42.5522    90.000    90.000    90.000
ZERR       12    0.0002    0.0001    0.0004     0.000     0.000     0.000
LATT   1
SYMM     0.50000 - X ,           - Y ,    0.50000 + Z
SYMM     0.50000 + X ,    0.50000 - Y ,           - Z
SYMM           - X ,    0.50000 + Y ,    0.50000 - Z
SFAC  C  H  N  O
UNIT  288 408 72 48
L.S. 4
FMAP 2
temp -173
PLAN 20
size 0.14 0.09 0.04
rem cut yellow block
ACTA
BOND $H
CONF
htab
eqiv $4 1-x,-y,-z
mpla 4 c1 c2 c3 c4 n1 n2
```

```
mpla c5 c6 c7 c8 c9 c10
mpla n5 o1 o2
mpla c15 c16 c17 c18 c19 c20
mpla n6 o3 o4
mpla 4 c25 c26 c25_$4 c26_$4 n7
mpla c27 c28 c29 c30 c31 c32
mpla n9 o5 o6
REM Instructions for potential hydrogen bonds
HTAB N3 O1
HTAB N4 O3
HTAB N8 O5
EQIV $1 x-1/2, y, -z+1/2
HTAB C1 O5_$1
EQIV $2 -x+1, y+1/2, -z+1/2
HTAB C1 O6_$2
HTAB C2 O5_$2
HTAB C9 O3_$1
HTAB C10 O4_$1
EQIV $3 x+1/2, y, -z+1/2
HTAB C25 O1_$3
WGHT    0.052200    2.424400
EXTI    0.000110
FVAR       0.17604
mole 1
C1    1    0.170346    0.728441    0.338551    11.00000    0.03393    0.03676 =
      0.03642    0.00138   -0.00393   -0.00166
AFIX  23
H1A   2    0.192348    0.614024    0.342202    11.00000   -1.20000
H1B   2    0.172600    0.803087    0.357914    11.00000   -1.20000
AFIX   0
C2    1    0.197584    0.831916    0.311635    11.00000    0.03783    0.04097 =
      0.03413   -0.00124   -0.00074   -0.00224
AFIX  23
H2A   2    0.178555    0.952795    0.309701    11.00000   -1.20000
H2B   2    0.239436    0.852411    0.316161    11.00000   -1.20000
AFIX   0
C3    1    0.130763    0.702507    0.274866    11.00000    0.03225    0.06700 =
      0.03521   -0.00593    0.00008   -0.00058
AFIX  23
H3A   2    0.127537    0.632032    0.255086    11.00000   -1.20000
H3B   2    0.110883    0.821207    0.271771    11.00000   -1.20000
AFIX   0
C4    1    0.101173    0.598555    0.301176    11.00000    0.03296    0.04996 =
      0.03909   -0.00806   -0.00107   -0.00120
AFIX  23
H4A   2    0.058911    0.589839    0.296631    11.00000   -1.20000
```

```
H4B  2   0.117039   0.472851   0.301925   11.00000  -1.20000
AFIX  0
C5   1   0.074325   0.627404   0.356696   11.00000   0.03460   0.02813 =
     0.03789   0.00063  -0.00120   0.00392
C6   1   0.093761   0.634341   0.387634   11.00000   0.03171   0.03209 =
     0.03812  -0.00145  -0.00129   0.00229
AFIX 43
H6   2   0.132441   0.674785   0.391607   11.00000  -1.20000
AFIX  0
C7   1   0.058469   0.583863   0.413366   11.00000   0.03526   0.02892 =
     0.03800  -0.00196  -0.00011   0.00345
C8   1   0.001362   0.517794   0.406288   11.00000   0.03377   0.03188 =
     0.03939   0.00141   0.00183   0.00278
C9   1  -0.018530   0.515159   0.375216   11.00000   0.03261   0.03451 =
     0.04506  -0.00023  -0.00214   0.00252
AFIX 43
H9   2  -0.057134   0.474847   0.370997   11.00000  -1.20000
AFIX  0
C10  1   0.015889   0.568720   0.350901   11.00000   0.03468   0.03696 =
     0.04042  -0.00039  -0.00372   0.00377
AFIX 43
H10  2   0.001082   0.567128   0.330051   11.00000  -1.20000
AFIX  0
C11  1   0.138243   0.650996   0.451339   11.00000   0.03697   0.03572 =
     0.03809  -0.00093   0.00078   0.00132
AFIX 23
H11A 2   0.145848   0.775551   0.443255   11.00000  -1.20000
H11B 2   0.167014   0.566777   0.441792   11.00000  -1.20000
AFIX  0
C12  1   0.144397   0.648714   0.486814   11.00000   0.03909   0.04585 =
     0.03841  -0.00120  -0.00035   0.00155
AFIX 23
H12A 2   0.116089   0.735777   0.496000   11.00000  -1.20000
H12B 2   0.134549   0.525211   0.494669   11.00000  -1.20000
AFIX  0
C13  1   0.205000   0.698463   0.497834   11.00000   0.04121   0.05511 =
     0.04728   0.00126  -0.00422   0.00155
AFIX 23
H13A 2   0.215415   0.820278   0.489430   11.00000  -1.20000
H13B 2   0.233202   0.608918   0.489282   11.00000  -1.20000
AFIX  0
C14  1   0.210146   0.701862   0.533625   11.00000   0.05839   0.11478 =
     0.05260  -0.01084  -0.01186  -0.00428
AFIX 137
H14A 2   0.250187   0.733591   0.539566   11.00000  -1.50000
H14B 2   0.200368   0.581147   0.542073   11.00000  -1.50000
```

```
H14C  2   0.183185   0.793030   0.542195   11.00000  -1.50000
AFIX   0
C15   1   0.235941   0.705505   0.260871   11.00000   0.03426   0.03090 =
      0.03528   0.00531   0.00106  -0.00107
C16   1   0.225097   0.651425   0.229980   11.00000   0.03132   0.03453 =
      0.03581   0.00392   0.00027  -0.00087
AFIX  43
H16   2   0.185819   0.647694   0.222890   11.00000  -1.20000
AFIX   0
C17   1   0.269450   0.602279   0.208885   11.00000   0.03737   0.03059 =
      0.03413   0.00638   0.00200  -0.00230
C18   1   0.328248   0.609464   0.220330   11.00000   0.03256   0.03954 =
      0.03835   0.00679   0.00456  -0.00053
C19   1   0.338868   0.673710   0.250852   11.00000   0.03272   0.04309 =
      0.03847   0.00816  -0.00122  -0.00346
AFIX  43
H19   2   0.378053   0.682614   0.257955   11.00000  -1.20000
AFIX   0
C20   1   0.295236   0.723592   0.270597   11.00000   0.03588   0.03764 =
      0.03414   0.00444  -0.00201  -0.00333
AFIX  43
H20   2   0.304129   0.770464   0.290857   11.00000  -1.20000
AFIX   0
C21   1   0.197464   0.527537   0.167258   11.00000   0.03714   0.04003 =
      0.03641   0.00240   0.00116  -0.00048
AFIX  23
H21A  2   0.176941   0.646512   0.167576   11.00000  -1.20000
H21B  2   0.175741   0.441839   0.180932   11.00000  -1.20000
AFIX   0
C22   1   0.198736   0.454128   0.133995   11.00000   0.04007   0.04289 =
      0.03673   0.00124   0.00179  -0.00052
AFIX  23
H22A  2   0.221823   0.339862   0.133596   11.00000  -1.20000
H22B  2   0.218511   0.543879   0.120233   11.00000  -1.20000
AFIX   0
C23   1   0.138065   0.415748   0.121018   11.00000   0.04231   0.05443 =
      0.04322  -0.00205  -0.00011  -0.00213
AFIX  23
H23A  2   0.117406   0.332041   0.135510   11.00000  -1.20000
H23B  2   0.115815   0.531558   0.120207   11.00000  -1.20000
AFIX   0
C24   1   0.139186   0.331403   0.088438   11.00000   0.05266   0.06968 =
      0.04444  -0.00388  -0.00387  -0.00940
AFIX 137
H24A  2   0.099156   0.308146   0.081416   11.00000  -1.50000
H24B  2   0.160846   0.216049   0.089088   11.00000  -1.50000
```

```
H24C  2    0.158281    0.415557    0.073776    11.00000   -1.50000
AFIX   0
N1    3    0.109249    0.684758    0.331820    11.00000    0.03255    0.03763 =
      0.03181    0.00035   -0.00149    0.00046
N2    3    0.191919    0.733901    0.282048    11.00000    0.03248    0.05284 =
      0.03524   -0.00516    0.00033   -0.00169
N3    3    0.079426    0.594508    0.442946    11.00000    0.03629    0.03982 =
      0.03594    0.00040    0.00118   -0.00086
H1N   2    0.055916    0.550438    0.458904    11.00000   -1.20000
N4    3    0.256322    0.550640    0.179271    11.00000    0.03437    0.04311 =
      0.03407    0.00142    0.00398    0.00010
H2N   2    0.285571    0.513297    0.167906    11.00000   -1.20000
N5    3   -0.036323    0.444960    0.429601    11.00000    0.03718    0.03910 =
      0.04318    0.00177    0.00072    0.00123
N6    3    0.376332    0.547061    0.202706    11.00000    0.03548    0.05332 =
      0.04154    0.00407    0.00476   -0.00007
O1    4   -0.023582    0.462483    0.458094    11.00000    0.04562    0.05275 =
      0.03975    0.00045    0.00479   -0.00338
O2    4   -0.081049    0.362175    0.421514    11.00000    0.04172    0.06519 =
      0.05523    0.00637   -0.00166   -0.01601
O3    4    0.369336    0.495335    0.174795    11.00000    0.04070    0.07123 =
      0.04083   -0.00421    0.00746    0.00052
O4    4    0.425589    0.542189    0.215153    11.00000    0.03247    0.08284 =
      0.05101    0.00093    0.00085    0.00453
mole 2
C25   1    0.540221    0.143048   -0.005539    11.00000    0.03814    0.03662 =
      0.03568    0.00277   -0.00161   -0.00321
AFIX  23
H25A  2    0.580083    0.183507   -0.011167    11.00000   -1.20000
H25B  2    0.515971    0.253312   -0.002256    11.00000   -1.20000
AFIX   0
C26   1    0.484822   -0.032299    0.032153    11.00000    0.03551    0.03801 =
      0.03362    0.00240    0.00043   -0.00193
AFIX  23
H26A  2    0.458505    0.071194    0.036978    11.00000   -1.20000
H26B  2    0.487953   -0.109464    0.051185    11.00000   -1.20000
AFIX   0
C27   1    0.579497    0.094624    0.047306    11.00000    0.03524    0.02965 =
      0.03611    0.00030   -0.00326    0.00226
C28   1    0.563230    0.089186    0.078592    11.00000    0.03309    0.03276 =
      0.03734    0.00037   -0.00081    0.00085
AFIX  43
H28   2    0.524946    0.049683    0.083774    11.00000   -1.20000
AFIX   0
C29   1    0.601287    0.140005    0.103052    11.00000    0.03810    0.02920 =
      0.03638    0.00106   -0.00224    0.00489
```

```
C30   1    0.657677    0.202888    0.094403    11.00000    0.03380    0.03444 =
      0.04148   -0.00022   -0.00609    0.00264
C31   1    0.674687    0.202285    0.062697    11.00000    0.03209    0.03526 =
      0.04626    0.00098   -0.00112    0.00166
AFIX  43
H31   2    0.713168    0.238898    0.057313    11.00000   -1.20000
AFIX   0
C32   1    0.637348    0.150511    0.039542    11.00000    0.03693    0.03583 =
      0.03894    0.00125    0.00157    0.00283
AFIX  43
H32   2    0.649740    0.151535    0.018238    11.00000   -1.20000
AFIX   0
C33   1    0.525270    0.077654    0.143225    11.00000    0.03909    0.03772 =
      0.03943    0.00076   -0.00202    0.00175
AFIX  23
H33A  2    0.516999   -0.047481    0.135610    11.00000   -1.20000
H33B  2    0.495425    0.161009    0.134442    11.00000   -1.20000
AFIX   0
C34   1    0.523076    0.082153    0.178754    11.00000    0.04149    0.04472 =
      0.04006    0.00039   -0.00156    0.00310
AFIX  23
H34A  2    0.528348    0.209873    0.185856    11.00000   -1.20000
H34B  2    0.556082    0.009692    0.187103    11.00000   -1.20000
AFIX   0
C35   1    0.466880    0.008696    0.192497    11.00000    0.04366    0.05243 =
      0.04882   -0.00471   -0.00014    0.00018
AFIX  23
H35A  2    0.434673    0.094541    0.187794    11.00000   -1.20000
H35B  2    0.457509   -0.109964    0.182543    11.00000   -1.20000
AFIX   0
C36   1    0.471556   -0.016597    0.227557    11.00000    0.05052    0.07044 =
      0.04908   -0.00104    0.00992    0.00219
AFIX 137
H36A  2    0.434983   -0.067560    0.235640    11.00000   -1.50000
H36B  2    0.479008    0.101735    0.237550    11.00000   -1.50000
H36C  2    0.503718   -0.100411    0.232277    11.00000   -1.50000
AFIX   0
N7    3    0.542465    0.037284    0.023665    11.00000    0.03490    0.03629 =
      0.03121    0.00215   -0.00100   -0.00245
N8    3    0.582869    0.134176    0.133060    11.00000    0.03849    0.04203 =
      0.03652   -0.00028   -0.00361   -0.00126
H3N   2    0.610098    0.174622    0.148546    11.00000   -1.20000
N9    3    0.698151    0.271562    0.116430    11.00000    0.03952    0.04306 =
      0.04514    0.00158   -0.00857    0.00068
O5    4    0.687233    0.258586    0.145322    11.00000    0.04671    0.05546 =
      0.04013   -0.00156   -0.00901   -0.00124
```

```
O6    4    0.743704    0.346215    0.106989    11.00000    0.04014    0.06330 =
      0.05803    0.00661   -0.00879   -0.01288
HKLF 4 1 -1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000 -1.0000

REM  db1_a.res Pbca
REM R1 =  0.0425 for    5944 Fo > 4sig(Fo)  and  0.0469 for all    6453 data
REM    473 parameters refined using      0 restraints

END

WGHT      0.0522      2.4291

REM Instructions for potential hydrogen bonds
HTAB C1 O5_$1
HTAB C1 O6_$2
HTAB C2 O5_$2
HTAB C9 O3_$1
HTAB C10 O4_$1
HTAB N3 O1
HTAB N4 O3
HTAB C25 O1_$3
HTAB N8 O5

REM Highest difference peak  0.197,  deepest hole -0.183,  1-sigma level  0.033
Q1    1   0.0275  0.5441  0.4096  11.00000  0.05    0.20
Q2    1   0.6498  0.2317  0.1540  11.00000  0.05    0.19
Q3    1  -0.0108  0.5800  0.3952  11.00000  0.05    0.18
Q4    1   0.2999  0.6242  0.2145  11.00000  0.05    0.17
Q5    1   0.4604  0.3028  0.1333  11.00000  0.05    0.17
Q6    1   0.4689 -0.0709  0.0179  11.00000  0.05    0.16
Q7    1   0.1377  0.7032  0.3356  11.00000  0.05    0.16
Q8    1   0.5430  0.0770  0.0105  11.00000  0.05    0.16
Q9    1   0.1802  0.5348  0.4358  11.00000  0.05    0.15
Q10   1   0.2481  0.6694  0.2197  11.00000  0.05    0.15
Q11   1   0.0994  0.6408  0.3427  11.00000  0.05    0.15
Q12   1   0.6447  0.3515  0.0687  11.00000  0.05    0.15
Q13   1   0.4626  0.1361  0.1994  11.00000  0.05    0.15
Q14   1   0.2313  0.7105  0.2445  11.00000  0.05    0.15
Q15   1   0.3327  0.6630  0.2333  11.00000  0.05    0.14
Q16   1  -0.0030  0.4822  0.3932  11.00000  0.05    0.14
Q17   1   0.2679  0.7133  0.2669  11.00000  0.05    0.14
Q18   1   0.1999  0.7418  0.2960  11.00000  0.05    0.14
Q19   1   0.6106  0.2990  0.0453  11.00000  0.05    0.14
Q20   1   0.4503  0.2048  0.1637  11.00000  0.05    0.14
;
_shelx_res_checksum   77399
```

CHECK CIF FILE of compound 5

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) db1

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR
PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: db1

```
Bond precision: C-C = 0.0021 A Wavelength=1.54178
Cell: a=22.9241(2) b=7.3205(1) c=42.5522(4)
alpha=90 beta=90 gamma=90
Temperature: 100 K
Calculated Reported
Volume 7140.94(13) 7140.93(13)
Space group P b c a P b c a
Hall group -P 2ac 2ab -P 2ac 2ab
Moiety formula C24 H34 N6 O4 C24 H34 N6 O4
Sum formula C24 H34 N6 O4 C24 H34 N6 O4
Mr 470.57 470.57
Dx,g cm-3 1.313 1.313
Z 12 12
Mu (mm-1) 0.745 0.745
F000 3024.0 3024.0
F000' 3033.15
h,k,lmax 27,8,51 27,8,51
Nref 6534 6453
Tmin,Tmax 0.923,0.971 0.892,1.000
Tmin' 0.901
Correction method= # Reported T Limits: Tmin=0.892 Tmax=1.000
AbsCorr = GAUSSIAN
Data completeness= 0.988 Theta(max)= 68.243
R(reflections)= 0.0425( 5944)
wR2(reflections)=
0.1125( 6453)
S = 1.081 Npar= 473
```

The following ALERTS were generated. Each ALERT has the format **test-name_ALERT_alert-type_alert-level.**
Click on the hyperlinks for more details of the test.

Alert level C

```
PLAT230_ALERT_2_C Hirshfeld Test Diff for O6 --N9 . 5.2 s.u.
PLAT410_ALERT_2_C Short Intra H...H Contact H1B ..H6 . 1.95 Ang.
x,y,z = 1_555 Check
PLAT410_ALERT_2_C Short Intra H...H Contact H2B ..H20 . 1.93 Ang.
x,y,z = 1_555 Check
PLAT410_ALERT_2_C Short Intra H...H Contact H3A ..H16 . 1.92 Ang.
x,y,z = 1_555 Check
PLAT410_ALERT_2_C Short Intra H...H Contact H4A ..H10 . 1.95 Ang.
x,y,z = 1_555 Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ..... 4.468 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 68 Report
```

Alert level G

```
PLAT143_ALERT_4_G s.u. on c - Axis Small or Missing ..... 0.00040 Ang.
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 13 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ..... 4.9 Low
PLAT955_ALERT_1_G Reported (CIF) and Actual (FCF) Lmax Differ by . 1 Units
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 8 Info
```

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
7 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
5 **ALERT level G** = General information/check it is not something unexpected
1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
6 ALERT type 2 Indicator that the structure model may be wrong or deficient

```
3 ALERT type 3 Indicator that the structure quality may be low
2 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check
```

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems
it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing
attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

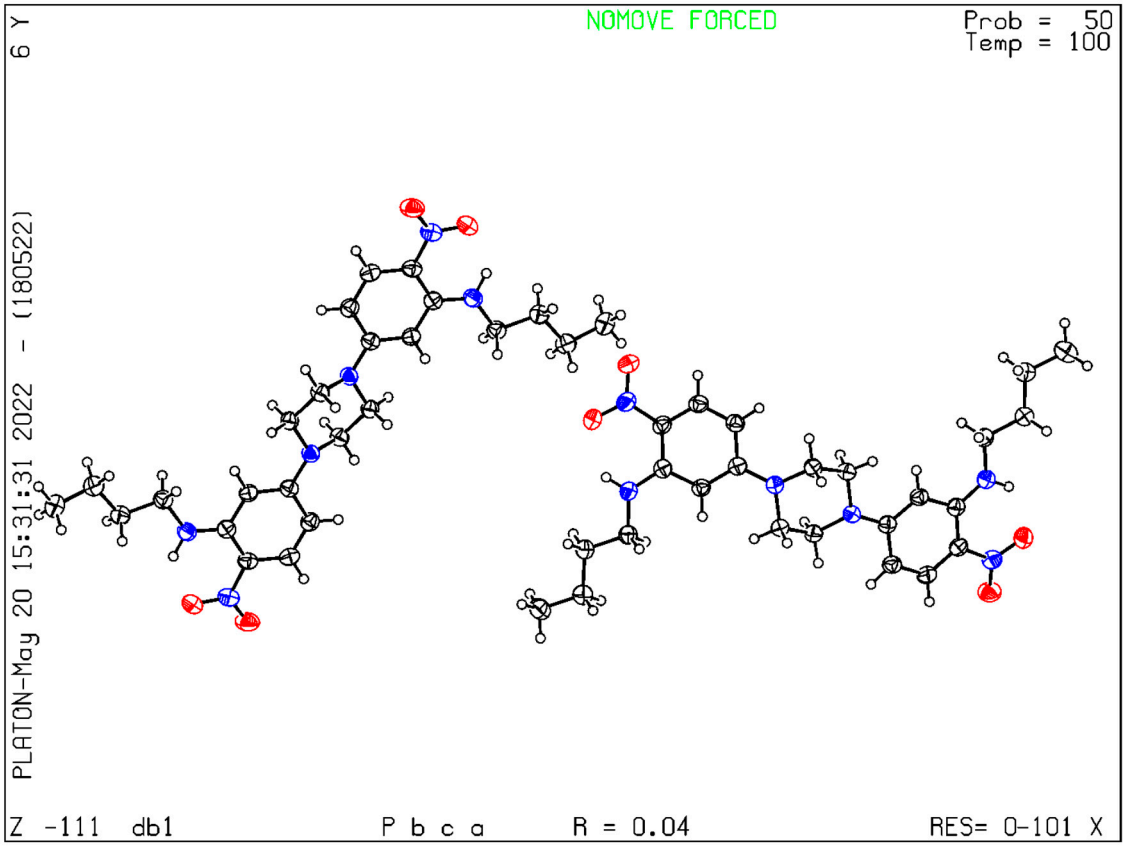
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 18/05/2022; check.def file version of 17/05/2022

Datablock db1 - ellipsoid plot



V200 mol file

```
  ACCLDraw05202209342D


34 36  0  0  0  0  0  0  0  0999 V2000
    9.1959   -6.4719    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0
    9.1959   -7.6531    0.0000
N   0  0  3  0  0  0  0  0  0  0  0  0
   10.2188   -8.2436    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0
   11.2416   -7.6531    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0
   11.2416   -6.4719    0.0000
N   0  0  3  0  0  0  0  0  0  0  0  0
   10.2188   -5.8814    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0
   12.2641   -5.8816    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0
    8.1734   -8.2434    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0
   13.2845   -4.1132    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0
   12.2645   -4.7008    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0
   14.3073   -4.7037    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0
   13.2910   -6.4745    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0
   14.3096   -5.8786    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0
    7.1530  -10.0118    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0
    8.1730   -9.4242    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0
    6.1302   -9.4213    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0
```

```
    7.1465   -7.6505    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0

    6.1279   -8.2464    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0

   15.3321   -6.4689    0.0000
N   0  0  3  0  0  0  0  0  0  0  0  0

   16.3546   -5.8786    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0

   17.3772   -6.4689    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0

   18.3997   -5.8786    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0

   19.4222   -6.4689    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0

   15.3298   -4.1133    0.0000
N   0  3  0  0  0  0  0  0  0  0  0  0

   16.3523   -4.7037    0.0000
O   0  5  0  0  0  0  0  0  0  0  0  0

   15.3298   -2.9326    0.0000
O   0  0  0  0  0  0  0  0  0  0  0  0

    7.1530  -11.1926    0.0000
N   0  0  3  0  0  0  0  0  0  0  0  0

    8.1756  -11.7829    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0

    8.1756  -12.9636    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0

    9.1981  -13.5540    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0

    9.1981  -14.7347    0.0000
C   0  0  0  0  0  0  0  0  0  0  0  0

    5.1077  -10.0117    0.0000
N   0  3  0  0  0  0  0  0  0  0  0  0

    4.0852   -9.4213    0.0000
O   0  5  0  0  0  0  0  0  0  0  0  0

    5.1077  -11.1924    0.0000
O   0  0  0  0  0  0  0  0  0  0  0  0

  5  6  1  0  0  0  0

  4  5  1  0  0  0  0

  3  4  1  0  0  0  0
```

```
  2  3  1  0  0  0  0
  1  2  1  0  0  0  0
  1  6  1  0  0  0  0
  5  7  1  0  0  0  0
  2  8  1  0  0  0  0
13 11  1  0  0  0  0
12 13  2  0  0  0  0
  9 10  1  0  0  0  0
11  9  2  0  0  0  0
10  7  2  0  0  0  0
  7 12  1  0  0  0  0
18 16  1  0  0  0  0
17 18  2  0  0  0  0
14 15  1  0  0  0  0
16 14  2  0  0  0  0
15  8  2  0  0  0  0
  8 17  1  0  0  0  0
13 19  1  0  0  0  0
19 20  1  0  0  0  0
20 21  1  0  0  0  0
21 22  1  0  0  0  0
22 23  1  0  0  0  0
24 26  2  0  0  0  0
24 25  1  0  0  0  0
11 24  1  0  0  0  0
14 27  1  0  0  0  0
27 28  1  0  0  0  0
28 29  1  0  0  0  0
29 30  1  0  0  0  0
30 31  1  0  0  0  0
32 34  2  0  0  0  0
32 33  1  0  0  0  0
```

```
16 32  1  0  0  0  0
M  CHG  4  24   1  25  -1  32   1  33  -1
M  END
```