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data_global
_journal_name_full               Chem.Commun.

_journal_coden_Cambridge         0182
_journal_volume                  ?
_journal_page_first              ?
_journal_year                    ?

_publ_contact_author_name        'Debanjan Biswas'
_publ_contact_author_email       biswasd@mail.nih.gov

_publ_section_title
;
Nitrogen-bound Diazeniumdiolated Amidines
;
loop_
_publ_author_name
'Debanjan Biswas'
'Jeffrey Deschamps'
'Larry Keefer'
'Joseph Hrabie'

# Attachment 'Amidine-Crystal-Data.cif.txt'

#============================================================================
data_4
_database_code_depnum_ccdc_archive 'CCDC 761561'
#TrackingRef 'Amidine-Crystal-Data.cif.txt'

_audit_creation_method           SHELXL-97
_chemical_name_systematic
;
?
;
_chemical_name_common            ?
_chemical_melting_point          ?
_chemical_formula_moiety         'C8 H10 N4 O2'
_chemical_formula_sum            'C8 H10 N4 O2'
_chemical_formula_weight         194.20

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'
O O 0.0106 0.0060 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting           orthorhombic
_symmetry_space_group_name_H-M   Pbca

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                    9.8141(5)
_cell_length_b                    11.4621(7)
_cell_length_c                    17.6029(9)
_cell_angle_alpha                 90.00
_cell_angle_beta                  90.00
_cell_angle_gamma                 90.00
_cell_volume                      1980.15(19)
_cell_formula_units_Z             8
_cell_measurement_temperature     294(2)
_cell_measurement_reflns_used     5886
_cell_measurement_theta_min       2.31
_cell_measurement_theta_max       25.82

_exptl_crystal_description        prisim
_exptl_crystal_colour             colourless
_exptl_crystal_size_min           0.107
_exptl_crystal_size_mid           0.144
_exptl_crystal_size_max           0.551
_exptl_crystal_density_meas       ?
_exptl_crystal_density_diffrn     1.303
_exptl_crystal_density_method     'not measured'
_exptl_crystal_F_000              816
_exptl_absorpt_coefficient_mu     0.098
##########################################################################
# #
# NOTE on absorbtion correction with SADABS: #
# #
# (Semi)empirical correction methods for absorption correction are more of #
# a problem in that they often specify only a relative correction range #
# [ Tmin(emp) & Tmax(emp)]. #
# #
# Acta Cryst. procedures call in such cases for the multiplication of both #
# empirical values with Tmax(expected). #
# #
# THIS RECOMMENDATION IS CURRENTLY UNDER REVIEW! (George #
# and Ton are agruing this point!) #
# #
##########################################################################
_exptl_absorpt_correction_type    multi-scan
_exptl_absorpt_process_details    'SADABS v2.10 (Bruker, 2000c)'
_exptl_absorpt_correction_T_min   0.9482
_exptl_absorpt_correction_T_max   0.9896

_exptl_special_details
;
?
;

_diffrn_ambient_temperature       294(2)
# SMART APEX II parameters
_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   'Bruker SMART APEX II CCD'
_diffrn_measurement_method        '\w scans'
_diffrn_detector_area_resol_mean  ?
_diffrn_reflns_number             19608
```

```
_diffrn_reflns_av_R_equivalents  0.0195
_diffrn_reflns_av_sigmaI/netI    0.0112
_diffrn_reflns_limit_h_min       -13
_diffrn_reflns_limit_h_max       11
_diffrn_reflns_limit_k_min       -15
_diffrn_reflns_limit_k_max       15
_diffrn_reflns_limit_l_min       -23
_diffrn_reflns_limit_l_max       23
_diffrn_reflns_theta_min         2.31
_diffrn_reflns_theta_max         28.37
_reflns_number_total             2462
_reflns_number_gt                1910
_reflns_threshold_expression     I>2\s(I)

_computing_data_collection       'Bruker SMART v5.631 (Bruker, 2001)'
_computing_cell_refinement       'Bruker SMART v5.631 (Bruker, 2001)'
_computing_data_reduction
;
Bruker SAINT v6.45A (Bruker, 2002), Bruker XPREP v6.14 (Bruker, 2001)
;
_computing_structure_solution    'Bruker SHELXTL v6.14 (Bruker, 2000b)'
_computing_structure_refinement  'Bruker SHELXTL v6.14 (Bruker, 2000b)'
_computing_molecular_graphics    'Bruker SHELXTL v6.14 (Bruker, 2000b)'
_computing_publication_material  'Bruker SHELXTL v6.14 (Bruker, 2000b)'

_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^)+(0.0585P)^2^+0.2819P] 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    mixed
_refine_ls_extinction_method     none
_refine_ls_extinction_coef       ?
_refine_ls_number_reflns         2462
_refine_ls_number_parameters     134
_refine_ls_number_restraints     0
_refine_ls_R_factor_all          0.0503
_refine_ls_R_factor_gt           0.0388
_refine_ls_wR_factor_ref         0.1186
_refine_ls_wR_factor_gt          0.1086
_refine_ls_goodness_of_fit_ref   1.029
_refine_ls_restrained_S_all      1.029
_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
N1 N 0.16413(10) 0.24839(9) 0.11856(5) 0.0498(3) Uani 1 1 d . . .
O1 O 0.23757(10) 0.29498(10) 0.06832(6) 0.0714(3) Uani 1 1 d . . .
N2 N 0.20244(12) 0.16960(10) 0.16465(6) 0.0593(3) Uani 1 1 d . . .
N3 N 0.03072(10) 0.28617(9) 0.13396(5) 0.0516(3) Uani 1 1 d . . .
O2 O 0.33715(10) 0.14482(10) 0.14812(6) 0.0712(3) Uani 1 1 d . . .
C3 C 0.3848(2) 0.05895(17) 0.20119(12) 0.1006(7) Uani 1 1 d . . .
H3A H 0.4734 0.0322 0.1860 0.151 Uiso 1 1 calc R . .
H3B H 0.3227 -0.0057 0.2021 0.151 Uiso 1 1 calc R . .
H3C H 0.3902 0.0930 0.2509 0.151 Uiso 1 1 calc R . .
C1' C -0.04200(12) 0.31006(9) 0.07380(6) 0.0468(3) Uani 1 1 d . . .
N1' N -0.00626(14) 0.30174(12) 0.00115(6) 0.0634(3) Uani 1 1 d . . .
H1'A H 0.078(2) 0.2831(17) -0.0098(11) 0.095 Uiso 1 1 d . . .
H2'B H -0.073(2) 0.2994(17) -0.0327(12) 0.095 Uiso 1 1 d . . .
C2' C -0.18176(12) 0.35294(10) 0.09034(6) 0.0489(3) Uani 1 1 d . . .
C3' C -0.25944(14) 0.30218(12) 0.14735(7) 0.0589(3) Uani 1 1 d . . .
H3'A H -0.2242 0.2405 0.1755 0.071 Uiso 1 1 calc R . .
C4' C -0.38923(15) 0.34327(15) 0.16238(8) 0.0710(4) Uani 1 1 d . . .
H4'A H -0.4417 0.3085 0.2001 0.085 Uiso 1 1 calc R . .
C5' C -0.44078(16) 0.43565(15) 0.12151(10) 0.0770(5) Uani 1 1 d . . .
H5'A H -0.5280 0.4632 0.1317 0.092 Uiso 1 1 calc R . .
C6' C -0.36325(17) 0.48747(14) 0.06544(10) 0.0736(4) Uani 1 1 d . . .
H6'A H -0.3979 0.5506 0.0385 0.088 Uiso 1 1 calc R . .
C7' C -0.23470(15) 0.44597(11) 0.04926(8) 0.0613(3) Uani 1 1 d . . .
H7'A H -0.1834 0.4802 0.0109 0.074 Uiso 1 1 calc R . .

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_atom_site_aniso_U_22
_atom_site_aniso_U_33
_atom_site_aniso_U_23
_atom_site_aniso_U_13
_atom_site_aniso_U_12
N1 0.0501(6) 0.0551(5) 0.0443(5) -0.0007(4) -0.0019(4) 0.0018(4)
O1 0.0521(5) 0.0906(7) 0.0715(6) 0.0250(5) 0.0068(4) 0.0017(5)
N2 0.0618(7) 0.0630(6) 0.0530(6) 0.0036(5) -0.0011(5) 0.0108(5)
N3 0.0491(6) 0.0601(6) 0.0455(5) 0.0002(4) -0.0004(4) 0.0052(4)
O2 0.0647(6) 0.0845(7) 0.0644(6) 0.0047(5) -0.0041(4) 0.0248(5)
C3 0.1171(16) 0.0898(12) 0.0949(13) 0.0083(10) -0.0112(11) 0.0518(11)
C1' 0.0497(6) 0.0449(6) 0.0457(6) -0.0012(4) -0.0004(5) -0.0037(5)
N1' 0.0524(6) 0.0925(9) 0.0452(6) -0.0025(5) -0.0018(5) 0.0025(6)
C2' 0.0503(6) 0.0488(6) 0.0476(6) -0.0050(5) -0.0035(5) 0.0010(5)
C3' 0.0586(7) 0.0667(8) 0.0513(6) 0.0001(6) 0.0028(5) 0.0050(6)
C4' 0.0587(8) 0.0919(11) 0.0624(8) -0.0144(7) 0.0090(6) 0.0022(7)
C5' 0.0579(8) 0.0883(11) 0.0848(10) -0.0315(9) -0.0099(8) 0.0208(8)
C6' 0.0737(10) 0.0606(8) 0.0864(10) -0.0091(7) -0.0194(8) 0.0175(7)
C7' 0.0643(8) 0.0522(7) 0.0672(8) 0.0020(6) -0.0083(6) 0.0022(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
N1 O1 1.2595(13) . ?
N1 N2 1.2709(14) . ?
N1 N3 1.4055(14) . ?
N2 O2 1.3831(15) . ?
N3 C1' 1.3060(15) . ?
O2 C3 1.4354(19) . ?
C3 H3A 0.9600 . ?
C3 H3B 0.9600 . ?
C3 H3C 0.9600 . ?
C1' N1' 1.3294(16) . ?
C1' C2' 1.4858(17) . ?
N1' H1'A 0.88(2) . ?
N1' H2'B 0.89(2) . ?
C2' C3' 1.3881(18) . ?
C2' C7' 1.3892(17) . ?
C3' C4' 1.3836(19) . ?
C3' H3'A 0.9300 . ?
C4' C5' 1.377(2) . ?
C4' H4'A 0.9300 . ?
C5' C6' 1.381(2) . ?
C5' H5'A 0.9300 . ?
C6' C7' 1.378(2) . ?
C6' H6'A 0.9300 . ?
C7' H7'A 0.9300 . ?

loop_
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_geom_angle_atom_site_label_2
_geom_angle_atom_site_label_3
_geom_angle
_geom_angle_site_symmetry_1
_geom_angle_site_symmetry_3
_geom_angle_publ_flag
O1 N1 N2 125.46(10) . . ?
O1 N1 N3 122.54(10) . . ?
N2 N1 N3 111.80(10) . . ?
N1 N2 O2 107.12(10) . . ?
C1' N3 N1 114.66(10) . . ?
N2 O2 C3 108.40(12) . . ?
O2 C3 H3A 109.5 . . ?
O2 C3 H3B 109.5 . . ?
H3A C3 H3B 109.5 . . ?
O2 C3 H3C 109.5 . . ?
H3A C3 H3C 109.5 . . ?
H3B C3 H3C 109.5 . . ?
N3 C1' N1' 128.37(12) . . ?
N3 C1' C2' 114.51(10) . . ?
N1' C1' C2' 117.12(11) . . ?
C1' N1' H1'A 118.5(13) . . ?
```

```
C1' N1' H2'B 116.9(12) . . ?
H1'A N1' H2'B 123.0(18) . . ?
C3' C2' C7' 119.52(12) . . ?
C3' C2' C1' 120.66(11) . . ?
C7' C2' C1' 119.81(11) . . ?
C4' C3' C2' 120.07(13) . . ?
C4' C3' H3'A 120.0 . . ?
C2' C3' H3'A 120.0 . . ?
C5' C4' C3' 120.02(15) . . ?
C5' C4' H4'A 120.0 . . ?
C3' C4' H4'A 120.0 . . ?
C4' C5' C6' 120.13(14) . . ?
C4' C5' H5'A 119.9 . . ?
C6' C5' H5'A 119.9 . . ?
C7' C6' C5' 120.25(14) . . ?
C7' C6' H6'A 119.9 . . ?
C5' C6' H6'A 119.9 . . ?
C6' C7' C2' 119.98(14) . . ?
C6' C7' H7'A 120.0 . . ?
C2' C7' H7'A 120.0 . . ?

loop_
_geom_torsion_atom_site_label_1
_geom_torsion_atom_site_label_2
_geom_torsion_atom_site_label_3
_geom_torsion_atom_site_label_4
_geom_torsion
_geom_torsion_site_symmetry_1
_geom_torsion_site_symmetry_2
_geom_torsion_site_symmetry_3
_geom_torsion_site_symmetry_4
_geom_torsion_publ_flag
O1 N1 N2 O2 -0.84(16) . . . . ?
N3 N1 N2 O2 -175.79(9) . . . . ?
O1 N1 N3 C1' 41.94(15) . . . . ?
N2 N1 N3 C1' -142.94(11) . . . . ?
N1 N2 O2 C3 177.92(12) . . . . ?
N1 N3 C1' N1' 0.89(18) . . . . ?
N1 N3 C1' C2' -178.47(9) . . . . ?
N3 C1' C2' C3' -41.56(16) . . . . ?
N1' C1' C2' C3' 139.00(13) . . . . ?
N3 C1' C2' C7' 137.40(12) . . . . ?
N1' C1' C2' C7' -42.04(16) . . . . ?
C7' C2' C3' C4' 0.77(19) . . . . ?
C1' C2' C3' C4' 179.73(12) . . . . ?
C2' C3' C4' C5' -0.9(2) . . . . ?
C3' C4' C5' C6' 0.0(2) . . . . ?
C4' C5' C6' C7' 1.0(2) . . . . ?
C5' C6' C7' C2' -1.1(2) . . . . ?
C3' C2' C7' C6' 0.25(19) . . . . ?
C1' C2' C7' C6' -178.73(12) . . . . ?

loop_
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_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
N1' H1'A O1 0.88(2) 2.09(2) 2.6703(17) 123.2(16) .
```

```
N1' H2'B O1 0.89(2) 2.24(2) 3.0075(17) 144.8(17) 4_455
N1' H2'B O2 0.89(2) 2.30(2) 3.1051(16) 150.0(17) 4_455

_diffrn_measured_fraction_theta_max 0.997
_diffrn_reflns_theta_full        25.00
_diffrn_measured_fraction_theta_full 1.000
_refine_diff_density_max         0.149
_refine_diff_density_min         -0.146
_refine_diff_density_rms         0.028

#=============================================================================
data_9
_database_code_depnum_ccdc_archive 'CCDC 761562'
#TrackingRef 'Amidine-Crystal-Data.cif.txt'

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;
?
;
_chemical_name_common            ?
_chemical_melting_point          ?
_chemical_formula_moiety         'C16 H18 N4 O2'
_chemical_formula_sum            'C16 H18 N4 O2'
_chemical_formula_weight         298.34

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'

_symmetry_cell_setting           orthorhombic
_symmetry_space_group_name_H-M   Pbca

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

_cell_length_a                   9.9137(7)
_cell_length_b                   13.7220(10)
_cell_length_c                   22.4918(16)
_cell_angle_alpha                90.00
_cell_angle_beta                 90.00
_cell_angle_gamma                90.00
_cell_volume                     3059.7(4)
_cell_formula_units_Z            8
_cell_measurement_temperature    100(2)
_cell_measurement_reflns_used    8050
_cell_measurement_theta_min      2.69
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_cell_measurement_theta_max      29.13

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_exptl_crystal_colour            colorless
_exptl_crystal_size_min          0.275
_exptl_crystal_size_mid          0.337
_exptl_crystal_size_max          0.488
_exptl_crystal_density_meas      ?
_exptl_crystal_density_diffrn    1.295
_exptl_crystal_density_method    'not measured'
_exptl_crystal_F_000             1264
_exptl_absorpt_coefficient_mu    0.089
##################################################################################
# #
# NOTE on absorbtion correction with SADABS: #
# #
# (Semi)empirical correction methods for absorption correction are more of #
# a problem in that they often specify only a relative correction range #
# [ Tmin(emp) & Tmax(emp)]. #
# #
# Acta Cryst. procedures call in such cases for the multiplication of both #
# empirical values with Tmax(expected). #
# #
# THIS RECOMMENDATION IS CURRENTLY UNDER REVIEW! (George #
# and Ton are agruing this point!) #
# #
##################################################################################
_exptl_absorpt_correction_type   multi-scan
_exptl_absorpt_process_details   'SADABS v2.10 (Bruker, 2000c)'
_exptl_absorpt_correction_T_min  0.9581
_exptl_absorpt_correction_T_max  0.9761

_exptl_special_details
;
?
;

_diffrn_ambient_temperature      100(2)
# SMART APEX II parameters
_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  'Bruker SMART APEX II CCD'
_diffrn_measurement_method       '\w scans'
_diffrn_detector_area_resol_mean ?
_diffrn_reflns_number            25385
_diffrn_reflns_av_R_equivalents  0.0263
_diffrn_reflns_av_sigmaI/netI    0.0178
_diffrn_reflns_limit_h_min       -12
_diffrn_reflns_limit_h_max       13
_diffrn_reflns_limit_k_min       -18
_diffrn_reflns_limit_k_max       18
_diffrn_reflns_limit_l_min       -27
_diffrn_reflns_limit_l_max       30
_diffrn_reflns_theta_min         1.81
_diffrn_reflns_theta_max         29.17
_reflns_number_total             4131
_reflns_number_gt                3516
_reflns_threshold_expression     I>2\s(I)

_computing_data_collection       'Bruker SMART v5.631 (Bruker, 2001)'
```

```
_computing_cell_refinement       'Bruker SMART v5.631 (Bruker, 2001)'
_computing_data_reduction
;
Bruker SAINT v6.45A (Bruker, 2002), Bruker XPREP v6.14 (Bruker, 2001)
;
_computing_structure_solution    'Bruker SHELXTL v6.14 (Bruker, 2000b)'
_computing_structure_refinement  'Bruker SHELXTL v6.14 (Bruker, 2000b)'
_computing_molecular_graphics    'Bruker SHELXTL v6.14 (Bruker, 2000b)'
_computing_publication_material  'Bruker SHELXTL v6.14 (Bruker, 2000b)'

_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^)+(0.0596P)^2^+0.7787P] 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         4131
_refine_ls_number_parameters     201
_refine_ls_number_restraints     0
_refine_ls_R_factor_all          0.0444
_refine_ls_R_factor_gt           0.0373
_refine_ls_wR_factor_ref         0.1084
_refine_ls_wR_factor_gt          0.1032
_refine_ls_goodness_of_fit_ref   1.043
_refine_ls_restrained_S_all      1.043
_refine_ls_shift/su_max          0.000
_refine_ls_shift/su_mean         0.000

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_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
N1 N 0.53139(8) -0.09689(5) 0.36447(4) 0.01579(17) Uani 1 1 d . . .
O1 O 0.63318(7) -0.09296(5) 0.39774(3) 0.02145(16) Uani 1 1 d . . .
N2 N 0.52967(8) -0.10581(6) 0.30791(4) 0.01739(18) Uani 1 1 d . . .
O2 O 0.66272(7) -0.10869(5) 0.28834(3) 0.01998(16) Uani 1 1 d . . .
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N3 N 0.39993(8) -0.09989(6) 0.38856(4) 0.01741(17) Uani 1 1 d . . .
C3 C 0.65802(11) -0.10615(7) 0.22374(5) 0.0215(2) Uani 1 1 d . . .
H3B H 0.6102 -0.1648 0.2091 0.026 Uiso 1 1 calc R . .
H3A H 0.7512 -0.1079 0.2079 0.026 Uiso 1 1 calc R . .
C4 C 0.58771(10) -0.01639(7) 0.20066(4) 0.0191(2) Uani 1 1 d . . .
C5 C 0.64892(11) 0.07509(7) 0.20554(5) 0.0224(2) Uani 1 1 d . . .
H5 H 0.7360 0.0806 0.2229 0.027 Uiso 1 1 calc R . .
C6 C 0.58278(11) 0.15818(8) 0.18511(5) 0.0262(2) Uani 1 1 d . . .
H6 H 0.6251 0.2201 0.1883 0.031 Uiso 1 1 calc R . .
C7 C 0.45481(12) 0.15079(8) 0.16006(5) 0.0268(2) Uani 1 1 d . . .
H7 H 0.4097 0.2076 0.1463 0.032 Uiso 1 1 calc R . .
C8 C 0.39306(11) 0.06016(8) 0.15524(5) 0.0252(2) Uani 1 1 d . . .
H8 H 0.3058 0.0548 0.1381 0.030 Uiso 1 1 calc R . .
C9 C 0.45972(11) -0.02272(7) 0.17565(5) 0.0218(2) Uani 1 1 d . . .
H9 H 0.4171 -0.0845 0.1724 0.026 Uiso 1 1 calc R . .
C10 C 0.36309(10) -0.01622(7) 0.41256(4) 0.01572(18) Uani 1 1 d . . .
N11 N 0.24625(9) -0.01521(6) 0.44263(4) 0.02009(18) Uani 1 1 d . . .
C12 C 0.16817(11) -0.10441(8) 0.45102(5) 0.0249(2) Uani 1 1 d . . .
H12C H 0.0821 -0.0988 0.4299 0.037 Uiso 1 1 calc R . .
H12B H 0.1511 -0.1143 0.4935 0.037 Uiso 1 1 calc R . .
H12A H 0.2189 -0.1600 0.4353 0.037 Uiso 1 1 calc R . .
C13 C 0.17976(11) 0.07325(8) 0.46367(5) 0.0262(2) Uani 1 1 d . . .
H13C H 0.1833 0.0755 0.5072 0.039 Uiso 1 1 calc R . .
H13B H 0.0854 0.0732 0.4506 0.039 Uiso 1 1 calc R . .
H13A H 0.2259 0.1304 0.4473 0.039 Uiso 1 1 calc R . .
C14 C 0.44555(10) 0.07503(6) 0.40979(4) 0.01575(19) Uani 1 1 d . . .
C15 C 0.48967(10) 0.11261(7) 0.35529(4) 0.01779(19) Uani 1 1 d . . .
H15 H 0.4622 0.0830 0.3191 0.021 Uiso 1 1 calc R . .
C16 C 0.57415(10) 0.19376(7) 0.35446(5) 0.0214(2) Uani 1 1 d . . .
H16 H 0.6048 0.2191 0.3176 0.026 Uiso 1 1 calc R . .
C17 C 0.61386(11) 0.23791(7) 0.40734(5) 0.0239(2) Uani 1 1 d . . .
H17 H 0.6721 0.2929 0.4066 0.029 Uiso 1 1 calc R . .
C18 C 0.56808(11) 0.20134(7) 0.46122(5) 0.0237(2) Uani 1 1 d . . .
H18 H 0.5942 0.2320 0.4973 0.028 Uiso 1 1 calc R . .
C19 C 0.48423(10) 0.12015(7) 0.46280(5) 0.0197(2) Uani 1 1 d . . .
H19 H 0.4533 0.0954 0.4998 0.024 Uiso 1 1 calc R . .

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_atom_site_aniso_U_33
_atom_site_aniso_U_23
_atom_site_aniso_U_13
_atom_site_aniso_U_12
N1 0.0151(4) 0.0130(3) 0.0192(4) -0.0003(3) 0.0010(3) 0.0002(3)
O1 0.0163(3) 0.0256(4) 0.0225(4) -0.0007(3) -0.0028(3) 0.0019(3)
N2 0.0149(4) 0.0175(4) 0.0197(4) -0.0010(3) 0.0034(3) -0.0004(3)
O2 0.0151(3) 0.0229(3) 0.0219(4) -0.0005(3) 0.0047(3) 0.0013(3)
N3 0.0143(4) 0.0182(4) 0.0198(4) -0.0012(3) 0.0032(3) -0.0020(3)
C3 0.0232(5) 0.0203(4) 0.0211(5) -0.0013(3) 0.0072(4) 0.0033(4)
C4 0.0210(5) 0.0181(4) 0.0183(4) -0.0012(3) 0.0070(4) 0.0007(3)
C5 0.0190(5) 0.0228(5) 0.0254(5) -0.0011(4) 0.0026(4) -0.0034(4)
C6 0.0270(6) 0.0187(5) 0.0329(6) -0.0006(4) 0.0009(4) -0.0048(4)
C7 0.0272(6) 0.0195(5) 0.0338(6) 0.0007(4) 0.0010(4) 0.0009(4)
C8 0.0205(5) 0.0231(5) 0.0319(6) -0.0026(4) 0.0004(4) -0.0003(4)
C9 0.0217(5) 0.0179(4) 0.0258(5) -0.0029(4) 0.0055(4) -0.0028(4)
C10 0.0141(4) 0.0186(4) 0.0145(4) 0.0004(3) -0.0016(3) -0.0001(3)
N11 0.0155(4) 0.0223(4) 0.0225(4) -0.0017(3) 0.0037(3) 0.0003(3)
C12 0.0177(5) 0.0287(5) 0.0284(6) 0.0032(4) 0.0046(4) -0.0042(4)
C13 0.0192(5) 0.0308(5) 0.0285(6) -0.0055(4) 0.0045(4) 0.0061(4)
C14 0.0130(4) 0.0142(4) 0.0200(5) -0.0008(3) -0.0009(3) 0.0016(3)
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```
C15 0.0174(4) 0.0165(4) 0.0194(4) -0.0008(3) 0.0000(4) 0.0024(3)
C16 0.0196(5) 0.0167(4) 0.0279(5) 0.0029(4) 0.0040(4) 0.0017(3)
C17 0.0203(5) 0.0149(4) 0.0364(6) -0.0012(4) -0.0015(4) -0.0003(4)
C18 0.0243(5) 0.0176(4) 0.0293(5) -0.0043(4) -0.0071(4) 0.0010(4)
C19 0.0206(5) 0.0179(4) 0.0205(5) -0.0012(3) -0.0026(4) 0.0027(4)

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

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_geom_bond_site_symmetry_2
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N1 O1 1.2574(11) . ?
N1 N2 1.2782(12) . ?
N1 N3 1.4120(11) . ?
N2 O2 1.3911(10) . ?
O2 C3 1.4542(12) . ?
N3 C10 1.3202(12) . ?
C3 C4 1.5073(14) . ?
C3 H3B 0.9900 . ?
C3 H3A 0.9900 . ?
C4 C9 1.3908(15) . ?
C4 C5 1.3986(13) . ?
C5 C6 1.3931(15) . ?
C5 H5 0.9500 . ?
C6 C7 1.3919(16) . ?
C6 H6 0.9500 . ?
C7 C8 1.3903(15) . ?
C7 H7 0.9500 . ?
C8 C9 1.3931(15) . ?
C8 H8 0.9500 . ?
C9 H9 0.9500 . ?
C10 N11 1.3414(12) . ?
C10 C14 1.4967(13) . ?
N11 C12 1.4603(13) . ?
N11 C13 1.4602(13) . ?
C12 H12C 0.9800 . ?
C12 H12B 0.9800 . ?
C12 H12A 0.9800 . ?
C13 H13C 0.9800 . ?
C13 H13B 0.9800 . ?
C13 H13A 0.9800 . ?
C14 C19 1.3970(13) . ?
C14 C15 1.3999(14) . ?
C15 C16 1.3935(13) . ?
C15 H15 0.9500 . ?
C16 C17 1.3916(15) . ?
C16 H16 0.9500 . ?
C17 C18 1.3879(16) . ?
C17 H17 0.9500 . ?
C18 C19 1.3905(14) . ?
C18 H18 0.9500 . ?
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C19 H19 0.9500 . ?

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O1 N1 N2 127.37(8) . . ?
O1 N1 N3 120.91(8) . . ?
N2 N1 N3 111.53(8) . . ?
N1 N2 O2 107.76(7) . . ?
N2 O2 C3 106.56(7) . . ?
C10 N3 N1 112.76(8) . . ?
O2 C3 C4 112.24(8) . . ?
O2 C3 H3B 109.2 . . ?
C4 C3 H3B 109.2 . . ?
O2 C3 H3A 109.2 . . ?
C4 C3 H3A 109.2 . . ?
H3B C3 H3A 107.9 . . ?
C9 C4 C5 118.92(9) . . ?
C9 C4 C3 120.67(9) . . ?
C5 C4 C3 120.39(9) . . ?
C6 C5 C4 120.30(10) . . ?
C6 C5 H5 119.8 . . ?
C4 C5 H5 119.8 . . ?
C7 C6 C5 120.19(9) . . ?
C7 C6 H6 119.9 . . ?
C5 C6 H6 119.9 . . ?
C8 C7 C6 119.87(10) . . ?
C8 C7 H7 120.1 . . ?
C6 C7 H7 120.1 . . ?
C7 C8 C9 119.72(10) . . ?
C7 C8 H8 120.1 . . ?
C9 C8 H8 120.1 . . ?
C4 C9 C8 121.00(9) . . ?
C4 C9 H9 119.5 . . ?
C8 C9 H9 119.5 . . ?
N3 C10 N11 117.00(8) . . ?
N3 C10 C14 124.02(8) . . ?
N11 C10 C14 118.95(8) . . ?
C10 N11 C12 120.94(8) . . ?
C10 N11 C13 124.18(8) . . ?
C12 N11 C13 114.56(8) . . ?
N11 C12 H12C 109.5 . . ?
N11 C12 H12B 109.5 . . ?
H12C C12 H12B 109.5 . . ?
N11 C12 H12A 109.5 . . ?
H12C C12 H12A 109.5 . . ?
H12B C12 H12A 109.5 . . ?
N11 C13 H13C 109.5 . . ?
N11 C13 H13B 109.5 . . ?
H13C C13 H13B 109.5 . . ?
N11 C13 H13A 109.5 . . ?
H13C C13 H13A 109.5 . . ?
H13B C13 H13A 109.5 . . ?
C19 C14 C15 119.88(9) . . ?
C19 C14 C10 119.02(8) . . ?
C15 C14 C10 121.02(8) . . ?
C16 C15 C14 119.60(9) . . ?
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```
C16 C15 H15 120.2 . . ?
C14 C15 H15 120.2 . . ?
C15 C16 C17 120.42(10) . . ?
C15 C16 H16 119.8 . . ?
C17 C16 H16 119.8 . . ?
C18 C17 C16 119.76(9) . . ?
C18 C17 H17 120.1 . . ?
C16 C17 H17 120.1 . . ?
C17 C18 C19 120.50(10) . . ?
C17 C18 H18 119.8 . . ?
C19 C18 H18 119.8 . . ?
C18 C19 C14 119.82(9) . . ?
C18 C19 H19 120.1 . . ?
C14 C19 H19 120.1 . . ?

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_geom_torsion_site_symmetry_1
_geom_torsion_site_symmetry_2
_geom_torsion_site_symmetry_3
_geom_torsion_site_symmetry_4
_geom_torsion_publ_flag
O1 N1 N2 O2 -1.15(12) . . . . ?
N3 N1 N2 O2 -176.07(7) . . . . ?
N1 N2 O2 C3 -172.83(7) . . . . ?
O1 N1 N3 C10 71.02(11) . . . . ?
N2 N1 N3 C10 -113.68(9) . . . . ?
N2 O2 C3 C4 58.72(10) . . . . ?
O2 C3 C4 C9 -107.47(11) . . . . ?
O2 C3 C4 C5 70.84(12) . . . . ?
C9 C4 C5 C6 -0.56(15) . . . . ?
C3 C4 C5 C6 -178.89(10) . . . . ?
C4 C5 C6 C7 0.44(17) . . . . ?
C5 C6 C7 C8 -0.21(17) . . . . ?
C6 C7 C8 C9 0.11(17) . . . . ?
C5 C4 C9 C8 0.46(15) . . . . ?
C3 C4 C9 C8 178.78(9) . . . . ?
C7 C8 C9 C4 -0.23(16) . . . . ?
N1 N3 C10 N11 -172.48(8) . . . . ?
N1 N3 C10 C14 5.39(13) . . . . ?
N3 C10 N11 C12 3.41(14) . . . . ?
C14 C10 N11 C12 -174.58(9) . . . . ?
N3 C10 N11 C13 -169.76(9) . . . . ?
C14 C10 N11 C13 12.25(14) . . . . ?
N3 C10 C14 C19 -122.27(11) . . . . ?
N11 C10 C14 C19 55.57(12) . . . . ?
N3 C10 C14 C15 54.52(13) . . . . ?
N11 C10 C14 C15 -127.64(10) . . . . ?
C19 C14 C15 C16 1.26(14) . . . . ?
C10 C14 C15 C16 -175.50(9) . . . . ?
C14 C15 C16 C17 -0.51(14) . . . . ?
C15 C16 C17 C18 -0.53(15) . . . . ?
C16 C17 C18 C19 0.83(16) . . . . ?
C17 C18 C19 C14 -0.07(15) . . . . ?
C15 C14 C19 C18 -0.98(14) . . . . ?
C10 C14 C19 C18 175.85(9) . . . . ?

_diffrn_measured_fraction_theta_max 1.000
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_diffrn_reflns_theta_full        29.17
_diffrn_measured_fraction_theta_full 1.000
_refine_diff_density_max         0.373
_refine_diff_density_min         -0.229
_refine_diff_density_rms         0.042
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