

Supporting Information

Giant Dielectric Anisotropy and Relaxor Ferroelectricity Induced by Proton Transfers in $\text{NH}^+\cdots\text{N}$ Bonded Supramolecular Aggregates

Marek Szafrński¹ and Andrzej Katrusiak²

¹Faculty of Physics, Adam Mickiewicz University, Umultowska 85, 61-614 Poznań, Poland;

²Faculty of Chemistry, Adam Mickiewicz University, Grunwaldzka 6, 60-780 Poznań, Poland

Table S1. Atomic coordinates and equivalent isotropic displacement parameters ($\text{\AA}^2$) for 1,4-diazabicyclo[2.2.2]octane hydroiodide at 120, 300 and 370 K. The coordinates equal 0 or expressed as fractions 1/3 and 2/3 are fixed due to the special positions, and were not refined.

T=120 K				
	x/a	y/b	z/c	U(eq)
I(1)	0	0	0	0.017(1)
N(1)	2/3	1/3	0.2619(8)	0.018(1)
C(1)	0.8968(5)	0.4484(2)	0.3559(5)	0.036(5)
H(1)	2/3	1/3	0.085(16)	0.010(20)
H(2)	0.961(4)	0.364(3)	0.2770(29)	0.027(6)
T= 300 K				
	x/a	y/b	z/c	U(eq)
I(1)	0	0	0	0.045(1)
N(1)	2/3	1/3	0.2643(7)	0.043(1)
C(1)	0.8937(5)	0.4469(2)	0.3563(5)	0.049(1)
H(1)	2/3	1/3	0.108(17)	0.019(19)
H(2)	0.955(5)	0.361(4)	0.276(3)	0.064(8)
T= 370 K				
	x/a	y/b	z/c	U(eq)
I(1)	0	0	0	0.061(1)
N(1)	2/3	1/3	0.2651(10)	0.058(1)
C(1)	0.8924(8)	0.4462(4)	0.3572(7)	0.063(1)
H(1)	2/3	1/3	0.091(20)	0.030(30)
H(2)	0.953(9)	0.365(7)	0.272(5)	0.110(15)

Table S2. Bond lengths (Å) and angles (°) for 1,4-diazabicyclo[2.2.2]octane hydroiodide at 120, 300 and 370 K.

Temperature [K]	120	300	370
N(1)-C(1)	1.489(3)	1.479(3)	1.474(5)
C(1)-C(1) ^a	1.536(5)	1.536(6)	1.531(8)
C(1)-N(1)-C(1) ^b	109.28(17)	109.51(18)	109.4(2)
N(1)-C(1)-C(1) ^a	109.67(17)	109.43(18)	109.6(2)
^a symmetry code	<i>x</i> , <i>y</i> , 1- <i>z</i>	<i>x</i> , <i>y</i> , 1- <i>z</i>	<i>x</i> , <i>y</i> , 1- <i>z</i>
^b symmetry code	1- <i>z</i> + <i>y</i> , 1- <i>x</i> , <i>z</i>	1- <i>z</i> + <i>y</i> , 1- <i>x</i> , <i>z</i>	1- <i>z</i> + <i>y</i> , 1- <i>x</i> , <i>z</i>

Table S3. Anisotropic displacement parameters (Å² x 10³) for 1,4-diazabicyclo[2.2.2]octane hydroiodide at 120, 300 and 370 K.

T=120 K						
	U ₁₁	U ₂₂	U ₃₃	U ₂₃	U ₁₃	U ₁₂
I(1)	17(1)	17(1)	19(1)	0	0	8(1)
N(1)	20(1)	20(1)	15(2)	0	0	10(1)
C(1)	18(1)	25(1)	21(1)	1(1)	1(1)	9(1)
T=300 K						
	U ₁₁	U ₂₂	U ₃₃	U ₂₃	U ₁₃	U ₁₂
I(1)	45(1)	45(1)	46(1)	0	0	22(1)
N(1)	52(1)	52(1)	24(2)	0	0	26(1)
C(1)	44(1)	54(1)	44(1)	2(1)	4(1)	22(1)
T=370 K						
	U ₁₁	U ₂₂	U ₃₃	U ₂₃	U ₁₃	U ₁₂
I(1)	61(1)	61(1)	61(1)	0	0	30(1)
N(1)	72(2)	72(2)	29(3)	0	0	36(1)
C(1)	62(2)	74(2)	50(2)	4(1)	8(2)	31(1)

CIF for dabcoHI, T=370 K

```
_audit_creation_method          SHELXL-97

_chemical_name_systematic

;
    1,4-diazabicyclo[2.2.2]octane monohydroiodide
;

_chemical_name_common           dabco hydroiodide
_chemical_melting_point         ?
_chemical_formula_moiety        ?
_chemical_formula_sum
    'C6 H13 I N2'
_chemical_formula_weight        240.08

loop_
  _atom_type_symbol
  _atom_type_description
  _atom_type_scatter_dispersion_real
  _atom_type_scatter_dispersion_imag
  _atom_type_scatter_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'
  'I'  'I'   -0.4742    1.8119
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting          hexagonal
_symmetry_space_group_name_H-M  p-6m2
```

loop_

_symmetry_equiv_pos_as_xyz

'x, y, z'

'-y, x-y, z'

'-x+y, -x, z'

'x, y, -z'

'-y, x-y, -z'

'-x+y, -x, -z'

'-y, -x, z'

'-x+y, y, z'

'x, x-y, z'

'-y, -x, -z'

'-x+y, y, -z'

'x, x-y, -z'

_cell_length_a 7.1042(10)

_cell_length_b 7.1042(10)

_cell_length_c 5.3600(11)

_cell_angle_alpha 90.00

_cell_angle_beta 90.00

_cell_angle_gamma 120.00

_cell_volume 234.27(7)

_cell_formula_units_Z 1

_cell_measurement_temperature 370(2)

_cell_measurement_reflns_used ?

_cell_measurement_theta_min ?

_cell_measurement_theta_max ?

_exptl_crystal_description parallelepiped

_exptl_crystal_colour	colourless
_exptl_crystal_size_max	0.15
_exptl_crystal_size_mid	0.09
_exptl_crystal_size_min	0.08
_exptl_crystal_density_meas	?
_exptl_crystal_density_diffn	1.702
_exptl_crystal_density_method	'not measured'
_exptl_crystal_F_000	116
_exptl_absorpt_coefficient_mu	3.349
_exptl_absorpt_correction_type	empirical via \psi scans
_exptl_absorpt_correction_T_min	0.605
_exptl_absorpt_correction_T_max	0.765
_exptl_absorpt_process_details	\psi scans for 5 reflections

_exptl_special_details

;

?

;

_diffn_ambient_temperature	370(2)
_diffn_radiation_wavelength	0.71073
_diffn_radiation_type	MoK\alpha
_diffn_radiation_source	'fine-focus sealed tube'
_diffn_radiation_monochromator	graphite
_diffn_measurement_device_type	KUMA KM-4
_diffn_measurement_method	theta-2theta
_diffn_detector_area_resol_mean	?
_diffn_standards_number	100
_diffn_standards_interval_count	?
_diffn_standards_interval_time	?

_diffrn_standards_decay_%	?
_diffrn_reflns_number	586
_diffrn_reflns_av_R_equivalents	0.0305
_diffrn_reflns_av_sigmaI/netI	0.0279
_diffrn_reflns_limit_h_min	-7
_diffrn_reflns_limit_h_max	9
_diffrn_reflns_limit_k_min	-8
_diffrn_reflns_limit_k_max	0
_diffrn_reflns_limit_l_min	-6
_diffrn_reflns_limit_l_max	0
_diffrn_reflns_theta_min	3.31
_diffrn_reflns_theta_max	26.82
_reflns_number_total	236
_reflns_number_gt	228
_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 $F^2 > 2\sigma(F^2)$ 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^)+(0.0200P)^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     mixed
_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     0.04(8)
_refine_ls_number_reflns           236
_refine_ls_number_parameters       18
_refine_ls_number_restraints       0
_refine_ls_R_factor_all            0.0201
_refine_ls_R_factor_gt             0.0194
_refine_ls_wR_factor_ref           0.0441
_refine_ls_wR_factor_gt            0.0439
_refine_ls_goodness_of_fit_ref     1.127
_refine_ls_restrained_S_all        1.127
_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

I1 I 0.0000 0.0000 0.0000 0.0607(2) Uani 1 12 d S . .
N1 N 0.6667 0.3333 0.2651(10) 0.0577(14) Uani 1 6 d S . .
H1 H 0.6667 0.3333 0.09(2) 0.03(3) Uiso 0.50 6 d SP . .
H0 H 0.6667 0.3333 0.0000 0.111 Uiso 0.00 12 d SP . .
C1 C 0.8924(8) 0.4462(4) 0.3572(7) 0.0633(11) Uani 1 2 d S . .
H2 H 0.953(9) 0.365(7) 0.272(5) 0.110(15) Uiso 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

I1 0.0606(2) 0.0606(2) 0.0609(3) 0.000 0.000 0.03032(12)
N1 0.072(2) 0.072(2) 0.029(3) 0.000 0.000 0.0360(11)
C1 0.062(2) 0.074(2) 0.050(2) 0.0040(10) 0.0080(19) 0.0310(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

N1 C1 1.474(5) . ?

N1 C1 1.474(5) 3_665 ?

N1 C1 1.474(5) 2_655 ?

C1 C1 1.531(8) 4_556 ?

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
C1 N1 C1 109.4(2) . 3_665 ?
C1 N1 C1 109.4(2) . 2_655 ?
C1 N1 C1 109.4(2) 3_665 2_655 ?
N1 C1 C1 109.6(2) . 4_556 ?

_diffrn_measured_fraction_theta_max    1.000
_diffrn_reflns_theta_full              26.82
_diffrn_measured_fraction_theta_full    1.000
_refine_diff_density_max                0.477
_refine_diff_density_min               -0.204
_refine_diff_density_rms               0.062

```

#AND

CIF for dabcoHI, T=300 K

```

_audit_creation_method                SHELXL-97
_chemical_name_systematic

;
    1,4-diazabicyclo[2.2.2]octane monohydroiodide
;
_chemical_name_common                 dabco hydroiodide
_chemical_melting_point                ?
_chemical_formula_moiety               ?
_chemical_formula_sum
    'C6 H13 I N2'
_chemical_formula_weight               240.08

loop_

```

```

_atom_type_symbol
_atom_type_description
_atom_type_scatter_dispersion_real
_atom_type_scatter_dispersion_imag
_atom_type_scatter_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'
'I'  'I'   -0.4742    1.8119
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

```

```

_symmetry_cell_setting      hexagonal
_symmetry_space_group_name_H-M  P-6m2

```

```

loop_

```

```

_symmetry_equiv_pos_as_xyz

```

```

'x, y, z'
'-y, x-y, z'
'-x+y, -x, z'
'x, y, -z'
'-y, x-y, -z'
'-x+y, -x, -z'
'-y, -x, z'
'-x+y, y, z'
'x, x-y, z'
'-y, -x, -z'
'-x+y, y, -z'

```

'x, x-y, -z'

_cell_length_a	7.0930 (10)
_cell_length_b	7.0930 (10)
_cell_length_c	5.3460 (11)
_cell_angle_alpha	90.00
_cell_angle_beta	90.00
_cell_angle_gamma	120.00
_cell_volume	232.93 (7)
_cell_formula_units_Z	1
_cell_measurement_temperature	300 (2)
_cell_measurement_reflns_used	?
_cell_measurement_theta_min	?
_cell_measurement_theta_max	?
_exptl_crystal_description	parallelepiped
_exptl_crystal_colour	colourless
_exptl_crystal_size_max	0.15
_exptl_crystal_size_mid	0.09
_exptl_crystal_size_min	0.08
_exptl_crystal_density_meas	?
_exptl_crystal_density_diffn	1.712
_exptl_crystal_density_method	'not measured'
_exptl_crystal_F_000	116
_exptl_absorpt_coefficient_mu	3.369
_exptl_absorpt_correction_type	empirical via \psi scans
_exptl_absorpt_correction_T_min	0.605
_exptl_absorpt_correction_T_max	0.765
_exptl_absorpt_process_details	5 reflection \psi scans

_exptl_special_details

;

?

;

_diffrn_ambient_temperature	300 (2)
_diffrn_radiation_wavelength	0.71073
_diffrn_radiation_type	MoK\alpha
_diffrn_radiation_source	'fine-focus sealed tube'
_diffrn_radiation_monochromator	graphite
_diffrn_measurement_device_type	KUMA KM-4
_diffrn_measurement_method	theta-2theta
_diffrn_detector_area_resol_mean	?
_diffrn_standards_number	100
_diffrn_standards_interval_count	?
_diffrn_standards_interval_time	?
_diffrn_standards_decay_%	0
_diffrn_reflns_number	1370
_diffrn_reflns_av_R_equivalents	0.0474
_diffrn_reflns_av_sigmaI/netI	0.0294
_diffrn_reflns_limit_h_min	-9
_diffrn_reflns_limit_h_max	9
_diffrn_reflns_limit_k_min	-9
_diffrn_reflns_limit_k_max	9
_diffrn_reflns_limit_l_min	0
_diffrn_reflns_limit_l_max	7
_diffrn_reflns_theta_min	3.32
_diffrn_reflns_theta_max	30.07
_reflns_number_total	309
_reflns_number_gt	284

_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 $F^2 > 2\sigma(F^2)$ 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)+(0.0133P)^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 mixed

_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	-0.06(5)
_refine_ls_number_reflns	309
_refine_ls_number_parameters	18
_refine_ls_number_restraints	0
_refine_ls_R_factor_all	0.0238
_refine_ls_R_factor_gt	0.0153
_refine_ls_wR_factor_ref	0.0333
_refine_ls_wR_factor_gt	0.0301
_refine_ls_goodness_of_fit_ref	1.035
_refine_ls_restrained_S_all	1.035
_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

I1 I 0.0000 0.0000 1.0000 0.04530(13) Uani 1 12 d S . .
N1 N 0.3333 0.6667 0.7357(7) 0.0429(8) Uani 1 6 d S . .
H1 H 0.3333 0.6667 0.892(17) 0.019(19) Uiso 0.50 6 d SP . .
C1 C 0.1063(5) 0.5531(2) 0.6437(5) 0.0487(6) Uani 1 2 d S . .
H2 H 0.045(5) 0.639(4) 0.724(3) 0.064(8) Uiso 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

I1 0.04474(14) 0.04474(14) 0.04640(18) 0.000 0.000 0.02237(7)
N1 0.0522(13) 0.0522(13) 0.0242(16) 0.000 0.000 0.0261(7)
C1 0.0443(13) 0.0544(12) 0.0439(14) 0.0020(6) 0.0039(11) 0.0221(7)

__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 C1 1.479(3) . ?

N1 C1 1.479(3) 3_565 ?

N1 C1 1.479(3) 2_665 ?

C1 C1 1.536(6) 4_556 ?

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

C1 N1 C1 109.51(18) . 3_565 ?

C1 N1 C1 109.51(18) . 2_665 ?

C1 N1 C1 109.51(18) 3_565 2_665 ?

N1 C1 C1 109.43(18) . 4_556 ?

_diffrn_measured_fraction_theta_max 1.000

_diffrn_reflns_theta_full 30.07

_diffrn_measured_fraction_theta_full 1.000

_refine_diff_density_max 0.287

_refine_diff_density_min -0.658

_refine_diff_density_rms 0.069

#AND

CIF for dabcoHI, T=120 K

```
_audit_creation_method          SHELXL-97
_chemical_name_systematic
;
    1,4-diazabicyclo[2.2.2]octane monohydroiodide
;
_chemical_name_common           dabco hydroiodide
_chemical_melting_point         ?
_chemical_formula_moiety        ?
_chemical_formula_sum
    'C6 H13 I N2'
_chemical_formula_weight        240.08

loop_
  _atom_type_symbol
  _atom_type_description
  _atom_type_scatter_dispersion_real
  _atom_type_scatter_dispersion_imag
  _atom_type_scatter_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'
  'I'  'I'   -0.4742    1.8119
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting          hexagonal
_symmetry_space_group_name_H-M  P-6m2
```

loop_

_symmetry_equiv_pos_as_xyz

'x, y, z'

'-y, x-y, z'

'-x+y, -x, z'

'x, y, -z'

'-y, x-y, -z'

'-x+y, -x, -z'

'-y, -x, z'

'-x+y, y, z'

'x, x-y, z'

'-y, -x, -z'

'-x+y, y, -z'

'x, x-y, -z'

_cell_length_a 7.0339 (10)

_cell_length_b 7.0339 (10)

_cell_length_c 5.3292 (11)

_cell_angle_alpha 90.00

_cell_angle_beta 90.00

_cell_angle_gamma 120.00

_cell_volume 228.34 (7)

_cell_formula_units_Z 1

_cell_measurement_temperature 120 (1)

_cell_measurement_reflns_used ?

_cell_measurement_theta_min ?

_cell_measurement_theta_max ?

_exptl_crystal_description parallelepiped

_exptl_crystal_colour	colourless
_exptl_crystal_size_max	0.15
_exptl_crystal_size_mid	0.09
_exptl_crystal_size_min	0.08
_exptl_crystal_density_meas	?
_exptl_crystal_density_diffn	1.746
_exptl_crystal_density_method	'not measured'
_exptl_crystal_F_000	116
_exptl_absorpt_coefficient_mu	3.436
_exptl_absorpt_correction_type	empirical via \psi scans
_exptl_absorpt_correction_T_min	0.605
_exptl_absorpt_correction_T_max	0.765
_exptl_absorpt_process_details	\psi scans for 5 reflections

_exptl_special_details

;

?

;

_diffn_ambient_temperature	120(1)
_diffn_radiation_wavelength	0.71073
_diffn_radiation_type	MoK\alpha
_diffn_radiation_source	'fine-focus sealed tube'
_diffn_radiation_monochromator	graphite
_diffn_measurement_device_type	KUMA KM-4
_diffn_measurement_method	theta-2theta
_diffn_detector_area_resol_mean	?
_diffn_standards_number	100
_diffn_standards_interval_count	?
_diffn_standards_interval_time	?

_diffrn_standards_decay_%	0
_diffrn_reflns_number	590
_diffrn_reflns_av_R_equivalents	0.0244
_diffrn_reflns_av_sigmaI/netI	0.0239
_diffrn_reflns_limit_h_min	-8
_diffrn_reflns_limit_h_max	7
_diffrn_reflns_limit_k_min	-8
_diffrn_reflns_limit_k_max	7
_diffrn_reflns_limit_l_min	0
_diffrn_reflns_limit_l_max	6
_diffrn_reflns_theta_min	3.34
_diffrn_reflns_theta_max	27.05
_reflns_number_total	232
_reflns_number_gt	232
_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 $F^2 > 2\sigma(F^2)$ 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^)+(0.0095P)^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     mixed
_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    -0.08(5)
_refine_ls_number_reflns          232
_refine_ls_number_parameters      18
_refine_ls_number_restraints      0
_refine_ls_R_factor_all            0.0107
_refine_ls_R_factor_gt             0.0107
_refine_ls_wR_factor_ref           0.0271
_refine_ls_wR_factor_gt            0.0271
_refine_ls_goodness_of_fit_ref     1.151
_refine_ls_restrained_S_all        1.151
_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

I1 I 0.0000 0.0000 0.0000 0.01735(10) Uani 1 12 d S . .
N1 N 0.6667 0.3333 0.2619(8) 0.0184(7) Uani 1 6 d S . .
H1 H 0.6667 0.3333 0.085(16) 0.01(2) Uiso 0.50 6 d SP . .
H0 H 0.6667 0.3333 0.0000 0.111 Uiso 0.00 12 d SP . .
C1 C 0.8968(5) 0.4484(2) 0.3559(5) 0.0219(6) Uani 1 2 d S . .
H2 H 0.961(4) 0.364(3) 0.277(3) 0.027(6) Uiso 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

I1 0.01661(12) 0.01661(12) 0.01884(15) 0.000 0.000 0.00830(6)
N1 0.0203(11) 0.0203(11) 0.0146(16) 0.000 0.000 0.0101(5)
C1 0.0184(12) 0.0248(10) 0.0205(14) 0.0006(5) 0.0012(10) 0.0092(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 C1 1.489(3) . ?

N1 C1 1.489(3) 3_665 ?

N1 C1 1.489(3) 2_655 ?

C1 C1 1.536(5) 4_556 ?

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
C1 N1 C1 109.28(17) . 3_665 ?
C1 N1 C1 109.28(17) . 2_655 ?
C1 N1 C1 109.28(17) 3_665 2_655 ?
N1 C1 C1 109.67(17) . 4_556 ?

_diffrn_measured_fraction_theta_max    1.000
_diffrn_reflns_theta_full              27.05
_diffrn_measured_fraction_theta_full   1.000
_refine_diff_density_max                0.241
_refine_diff_density_min               -0.362
_refine_diff_density_rms               0.058

#END

```