crystals

Supplementary Materials: Theoretical Studies on Hydrogen Bonds in Anions Encapsulated by an Azamacrocyclic Receptor

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Supporting Information

X-Ray data for [H6LCl2], [H4LBr2], and [H4LI2].

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 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
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 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
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 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.
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N2 N 0.72442(17) 0.75474(14) 0.65708(15) 0.0217(4) Uani 1 1 d . . .
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H22N H 0.6617 0.7076 0.6619 0.026 Uiso 1 1 calc R . .
N3 N 0.76246(17) 0.24462(14) 0.45330(15) 0.0216(4) Uani 1 1 d . . .
H31N H 0.7538 0.2161 0.5196 0.026 Uiso 1 1 calc R . .
H32N H 0.6973 0.2898 0.4337 0.026 Uiso 1 1 calc R . .
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H1B H 0.5836 0.9207 0.6067 0.029 Uiso 1 1 calc R . .
C2 C 0.7063(2) 0.84860(17) 0.72483(17) 0.0234(5) Uani 1 1 d . . .
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H2D H 0.7053 0.8258 0.8007 0.028 Uiso 1 1 calc R . .
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H3D H 0.9140 0.7474 0.6740 0.032 Uiso 1 1 calc R . .
C4 C 0.8519(2) 0.59794(18) 0.63142(18) 0.0229(5) Uani 1 1 d . . .
C5 C 0.8197(2) 0.59014(18) 0.51978(19) 0.0289(5) Uani 1 1 d . . .
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C6 C 0.8280(2) 0.49546(18) 0.46668(18) 0.0266(5) Uani 1 1 d . . .
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C10 C 0.8816(2) 0.30512(17) 0.46346(18) 0.0229(5) Uani 1 1 d . . .
H10A H 0.9018 0.3210 0.3902 0.028 Uiso 1 1 calc R . .
H10B H 0.9495 0.2620 0.5023 0.028 Uiso 1 1 calc R . .
C11 C 0.7578(2) 0.15858(17) 0.37081(18) 0.0246(5) Uani 1 1 d . . .
H11A H 0.8286 0.1105 0.3915 0.030 Uiso 1 1 calc R . .
H11B H 0.7664 0.1895 0.2993 0.030 Uiso 1 1 calc R . .
C12 C 0.3613(2) 0.90344(18) 0.6391(2) 0.0284(5) Uani 1 1 d . . .
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H12B H 0.3481 0.9720 0.6732 0.034 Uiso 1 1 calc R . .
C13 C 0.4589(2) 0.84450(19) 0.82262(18) 0.0283(5) Uani 1 1 d . . .
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H13B H 0.5302 0.8071 0.8618 0.042 Uiso 1 1 calc R . .
H13C H 0.3828 0.8068 0.8313 0.042 Uiso 1 1 calc R . .
O1 O 0.9414(2) -0.07260(18) 0.5879(2) 0.0577(6) Uani 1 1 d D . .
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H2W H 0.908(3) -0.0099(18) 0.600(3) 0.086 Uiso 1 1 d D . .
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Cl3 0.0817(5) 0.0198(3) 0.0266(3) 0.0011(2) -0.0028(3) 0.0048(3)
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N2 0.0247(10) 0.0184(9) 0.0223(9) -0.0035(7) 0.0041(8) -0.0030(8)
N3 0.0253(10) 0.0194(9) 0.0207(9) 0.0006(7) 0.0048(8) 0.0049(8)
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C2 0.0317(13) 0.0186(11) 0.0201(10) -0.0064(8) 0.0049(9) -0.0042(10)
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C4 0.0203(11) 0.0244(12) 0.0237(11) -0.0036(9) 0.0023(9) -0.0022(9)
C5 0.0367(14) 0.0215(12) 0.0269(12) 0.0013(9) -0.0010(10) 0.0065(10)
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C7 0.0218(11) 0.0218(11) 0.0230(11) -0.0008(9) 0.0057(9) 0.0011(9)
C8 0.0320(13) 0.0226(12) 0.0212(11) 0.0045(9) 0.0038(10) 0.0008(10)
C9 0.0313(13) 0.0280(13) 0.0188(10) 0.0010(9) 0.0049(10) -0.0041(10)
C10 0.0250(12) 0.0200(11) 0.0238(11) 0.0010(9) 0.0036(9) 0.0016(9)
C11 0.0314(13) 0.0182(11) 0.0243(11) -0.0034(9) 0.0046(10) 0.0053(10)
C12 0.0342(14) 0.0175(11) 0.0328(12) 0.0011(9) 0.0028(11) 0.0031(10)
C13 0.0374(14) 0.0309(13) 0.0182(11) -0.0003(9) 0.0097(10) -0.0061(11)
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 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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N2 C3 1.499(3) . ?
N2 H21N 0.9200 . ?
N2 H22N 0.9200 . ?
N3 C11 1.498(3) . ?
N3 C10 1.502(3) . ?
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C1 C2 1.513(3) . ?
C1 H1A 0.9900 . ?
C1 H1B 0.9900 . ?
C2 H2C 0.9900 . ?
C2 H2D 0.9900 . ?
C3 C4 1.511(3) . ?
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C3 H3D 0.9900 . ?
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C4 C5 1.387(3) . ?
C5 C6 1.385(3) . ?
C5 H5 0.9500 . ?
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C6 H6 0.9500 . ?
C7 C8 1.383(3) . ?
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C9 H9 0.9500 . ?
C10 H10A 0.9900 . ?
C10 H10B 0.9900 . ?
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C1 N1 H1N 107.9 . . ?
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C2 N2 H22N 108.9 . . ?
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C2 C1 H1B 108.6 . . ?
H1A C1 H1B 107.6 . . ?
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C1 C2 H2C 109.0 . . ?
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N2 H22N Cl1  0.92 2.25 3.126(2) 159.5 .
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O1 H1W Cl3  0.918(17) 2.39(3) 3.156(3) 141(3) 1_545
O1 H2W Cl2  0.899(17) 2.403(19) 3.276(2) 164(3) .

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_diffrn_reflns_theta_full            25.0
_diffrn_measured_fraction_theta_full   0.998
_refine_diff_density_max    0.61
_refine_diff_density_min   -0.52
_refine_diff_density_rms    0.072
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[H4L(Br)2]
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;
_chemical_name_common           ?
_chemical_melting_point          ?
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_chemical_formula_sum     'C26 H46 Br4 N6'
_chemical_formula_weight         762.33

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 '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'
 'Br'  'Br'  -0.2901   2.4595
 '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'

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_symmetry_space_group_name_Hall  '-P 1'
_symmetry_cell_setting 'Triclinic'

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

_cell_length_a                 5.7991(5)
_cell_length_b                 9.5238(10)
_cell_length_c                 14.7954(14)
_cell_angle_alpha               74.360(5)
_cell_angle_beta                84.001(6)
_cell_angle_gamma               88.462(6)
_cell_volume                   782.58(13)
_cell_formula_units_Z           1
_cell_measurement_temperature    90.0(5)
_cell_measurement_reflns_used    7160
_cell_measurement_theta_min      2.5
_cell_measurement_theta_max      37.0

_exptl_crystal_description      fragment
_exptl_crystal_colour           colorless
_exptl_crystal_size_max         0.25
_exptl_crystal_size_mid         0.23
_exptl_crystal_size_min         0.20
_exptl_crystal_density_meas     ?
_exptl_crystal_density_diffrn    1.618
_exptl_crystal_density_method    'not measured'
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_exptl_absorpt_coefficient_mu    5.168
_exptl_absorpt_correction_type    'multi-scan'
_exptl_absorpt_correction_T_min   0.300
_exptl_absorpt_correction_T_max   0.356
_exptl_absorpt_process_details    'HKL Scalepack (Otwinowski & Minor 1997)'

_exptl_special_details
;
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;

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_diffrn_radiation_wavelength     0.71073
_diffrn_radiation_type          MoK\a
_diffrn_radiation_source         'fine-focus sealed tube'
_diffrn_radiation_monochromator   graphite
_diffrn_measurement_device  'Nonius KappaCCD (with Oxford Cryostream)'
_diffrn_measurement_method      '\w and \f scans '
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_diffrn_standards_number         ?
_diffrn_standards_interval_count  ?
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_diffrn_reflns_number           33284
_diffrn_reflns_av_R_equivalents   0.023
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_diffrn_reflns_limit_k_max       15
_diffrn_reflns_limit_l_min       -24
_diffrn_reflns_limit_l_max       24
_diffrn_reflns_theta_min         2.8
_diffrn_reflns_theta_max         37.0
_reflns_number_total            7513
_reflns_number_gt               6326
_reflns_threshold_expression     I>2\s(I)

_computing_data_collection  'COLLECT (Nonius 1999)'
_computing_data_reduction  'Denzo and Scalepack (Otwinowski & Minor, 1997)'
_computing_cell_refinement    'Denzo and Scalepack (Otwinowski & Minor,
1997)'
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_computing_structure_solution   'Direct_methods (SIR, Altomare, et al.,
1994)'
_computing_structure_refinement   'SHELXL-97 (Sheldrick, 2008)'
_computing_molecular_graphics     'ORTEP-3 for Windows (Farrugia, 1997)'
_computing_publication_material   'SHELXL-97 (Sheldrick, 2008)'

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 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.
;

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_refine_ls_matrix_type           full
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_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0275P)^2^+0.6097P] 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     SHELXL
_refine_ls_extinction_coef       0.0133(8)
_refine_ls_extinction_expression
 'Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^'
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_refine_ls_goodness_of_fit_ref    1.053
_refine_ls_restrained_S_all      1.053
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loop_
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 _atom_site_adp_type
 _atom_site_occupancy
 _atom_site_symmetry_multiplicity
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Br1 Br 0.84343(2) 0.323097(14) 0.711615(9) 0.01060(4) Uani 1 1 d . . .
Br2 Br 0.84492(2) 0.805221(15) 0.807756(10) 0.01354(4) Uani 1 1 d . . .
N1 N 0.36336(19) 0.28291(12) 0.88993(8) 0.00939(18) Uani 1 1 d . . .
N2 N 0.3433(2) 0.14206(13) 0.74313(8) 0.00956(18) Uani 1 1 d . . .
H21N H 0.461(4) 0.201(2) 0.7358(15) 0.011 Uiso 1 1 d . . .
H22N H 0.221(4) 0.199(2) 0.7296(15) 0.011 Uiso 1 1 d . . .
N3 N 0.4844(2) 0.57342(13) 0.77189(8) 0.00998(18) Uani 1 1 d . . .
H31N H 0.567(4) 0.500(2) 0.7655(15) 0.012 Uiso 1 1 d . . .
H32N H 0.581(4) 0.650(2) 0.7618(15) 0.012 Uiso 1 1 d . . .
C1 C 0.2114(2) 0.15690(14) 0.90398(9) 0.0105(2) Uani 1 1 d . . .
H1A H 0.0552 0.1911 0.8866 0.013 Uiso 1 1 calc R . .
H1B H 0.1962 0.1021 0.9715 0.013 Uiso 1 1 calc R . .
C2 C 0.3065(2) 0.05620(14) 0.84449(9) 0.0113(2) Uani 1 1 d . . .
H2A H 0.4552 0.0133 0.8660 0.014 Uiso 1 1 calc R . .
H2B H 0.1957 -0.0243 0.8516 0.014 Uiso 1 1 calc R . .
C3 C 0.3846(3) 0.04757(15) 0.67641(10) 0.0124(2) Uani 1 1 d . . .
H3A H 0.2405 -0.0058 0.6763 0.015 Uiso 1 1 calc R . .
H3B H 0.5062 -0.0251 0.6980 0.015 Uiso 1 1 calc R . .
C4 C 0.4592(2) 0.13819(14) 0.57791(9) 0.0103(2) Uani 1 1 d . . .
C5 C 0.6802(2) 0.12124(15) 0.53534(10) 0.0121(2) Uani 1 1 d . . .
H5 H 0.7840 0.0525 0.5688 0.015 Uiso 1 1 calc R . .
C6 C 0.7500(2) 0.20425(15) 0.44425(10) 0.0122(2) Uani 1 1 d . . .
H6 H 0.9000 0.1904 0.4155 0.015 Uiso 1 1 calc R . .
C7 C 0.6012(2) 0.30781(15) 0.39473(9) 0.0106(2) Uani 1 1 d . . .
C8 C 0.3801(3) 0.32538(17) 0.43742(10) 0.0148(2) Uani 1 1 d . . .
H8 H 0.2776 0.3958 0.4046 0.018 Uiso 1 1 calc R . .
C9 C 0.3092(2) 0.24004(16) 0.52787(10) 0.0142(2) Uani 1 1 d . . .
H9 H 0.1572 0.2513 0.5558 0.017 Uiso 1 1 calc R . .
C10 C 0.3082(2) 0.59895(16) 0.70152(10) 0.0123(2) Uani 1 1 d . . .
H10A H 0.1706 0.6468 0.7255 0.015 Uiso 1 1 calc R . .
```

```
H10B H 0.2581 0.5036 0.6954 0.015 Uiso 1 1 calc R . .
C11 C 0.3796(2) 0.54823(15) 0.87180(10) 0.0118(2) Uani 1 1 d . . .
H11A H 0.2810 0.6327 0.8771 0.014 Uiso 1 1 calc R . .
H11B H 0.5051 0.5417 0.9132 0.014 Uiso 1 1 calc R . .
C12 C 0.2342(2) 0.40995(15) 0.90567(10) 0.0109(2) Uani 1 1 d . . .
H12A H 0.1842 0.3926 0.9738 0.013 Uiso 1 1 calc R . .
H12B H 0.0934 0.4229 0.8718 0.013 Uiso 1 1 calc R . .
C13 C 0.5564(2) 0.24481(16) 0.94838(10) 0.0127(2) Uani 1 1 d . . .
H13A H 0.6659 0.3265 0.9326 0.019 Uiso 1 1 calc R . .
H13B H 0.6356 0.1584 0.9365 0.019 Uiso 1 1 calc R . .
H13C H 0.4969 0.2241 1.0151 0.019 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
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Br2 0.01074(6) 0.01376(6) 0.01684(7) -0.00558(5) -0.00031(5) -0.00162(4)
N1 0.0082(4) 0.0108(4) 0.0093(4) -0.0027(4) -0.0012(3) -0.0008(3)
N2 0.0106(5) 0.0097(4) 0.0082(4) -0.0021(3) -0.0006(3) -0.0003(4)
N3 0.0111(5) 0.0097(4) 0.0084(4) -0.0015(4) -0.0004(4) -0.0003(4)
C1 0.0104(5) 0.0119(5) 0.0090(5) -0.0027(4) 0.0006(4) -0.0018(4)
C2 0.0144(5) 0.0102(5) 0.0082(5) -0.0009(4) -0.0006(4) -0.0011(4)
C3 0.0177(6) 0.0103(5) 0.0091(5) -0.0029(4) -0.0010(4) 0.0001(4)
C4 0.0128(5) 0.0100(5) 0.0083(5) -0.0025(4) -0.0014(4) 0.0002(4)
C5 0.0128(5) 0.0126(5) 0.0105(5) -0.0024(4) -0.0016(4) 0.0024(4)
C6 0.0102(5) 0.0150(6) 0.0112(5) -0.0036(4) -0.0005(4) 0.0012(4)
C7 0.0114(5) 0.0113(5) 0.0089(5) -0.0024(4) -0.0002(4) -0.0005(4)
C8 0.0125(6) 0.0175(6) 0.0115(6) 0.0001(5) 0.0001(4) 0.0043(5)
C9 0.0121(6) 0.0176(6) 0.0106(5) -0.0008(5) 0.0009(4) 0.0033(5)
C10 0.0113(5) 0.0152(6) 0.0095(5) -0.0017(4) -0.0009(4) -0.0017(4)
C11 0.0155(6) 0.0116(5) 0.0085(5) -0.0036(4) 0.0006(4) -0.0009(4)
C12 0.0109(5) 0.0115(5) 0.0094(5) -0.0017(4) 0.0008(4) 0.0002(4)
C13 0.0116(5) 0.0150(6) 0.0115(5) -0.0029(4) -0.0027(4) 0.0006(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 C13 1.4643(18) . ?
N1 C12 1.4650(18) . ?
N2 C2 1.4982(18) . ?
N2 C3 1.5032(18) . ?
N2 H21N 0.87(2) . ?
N2 H22N 0.89(2) . ?
N3 C11 1.4971(18) . ?
N3 C10 1.5032(18) . ?
N3 H31N 0.86(2) . ?
N3 H32N 0.90(2) . ?
C1 C2 1.5245(19) . ?
C1 H1A 0.9900 . ?
C1 H1B 0.9900 . ?
C2 H2A 0.9900 . ?
C2 H2B 0.9900 . ?
C3 C4 1.5053(19) . ?
C3 H3A 0.9900 . ?
C3 H3B 0.9900 . ?
C4 C5 1.393(2) . ?
C4 C9 1.3955(19) . ?
C5 C6 1.391(2) . ?
C5 H5 0.9500 . ?
C6 C7 1.3968(19) . ?
C6 H6 0.9500 . ?
C7 C8 1.395(2) . ?
C7 C10 1.5098(19) 2_666 ?
C8 C9 1.392(2) . ?
C8 H8 0.9500 . ?
C9 H9 0.9500 . ?
C10 C7 1.5097(19) 2_666 ?
```

```
C10 H10A 0.9900 . ?
C10 H10B 0.9900 . ?
C11 C12 1.5206(19) . ?
C11 H11A 0.9900 . ?
C11 H11B 0.9900 . ?
C12 H12A 0.9900 . ?
C12 H12B 0.9900 . ?
C13 H13A 0.9800 . ?
C13 H13B 0.9800 . ?
C13 H13C 0.9800 . ?

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 _geom_angle_atom_site_label_3
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 _geom_angle_site_symmetry_1
 _geom_angle_site_symmetry_3
 _geom_angle_publ_flag
C1 N1 C13 110.60(11) . . ?
C1 N1 C12 111.70(10) . . ?
C13 N1 C12 112.29(11) . . ?
C2 N2 C3 113.07(10) . . ?
C2 N2 H21N 108.9(14) . . ?
C3 N2 H21N 109.7(14) . . ?
C2 N2 H22N 108.6(14) . . ?
C3 N2 H22N 110.6(14) . . ?
H21N N2 H22N 105.7(19) . . ?
C11 N3 C10 113.61(11) . . ?
C11 N3 H31N 110.4(14) . . ?
C10 N3 H31N 107.8(14) . . ?
C11 N3 H32N 105.2(14) . . ?
C10 N3 H32N 112.3(14) . . ?
H31N N3 H32N 107.3(19) . . ?
N1 C1 C2 111.29(11) . . ?
N1 C1 H1A 109.4 . . ?
C2 C1 H1A 109.4 . . ?
N1 C1 H1B 109.4 . . ?
C2 C1 H1B 109.4 . . ?
H1A C1 H1B 108.0 . . ?
N2 C2 C1 109.11(11) . . ?
N2 C2 H2A 109.9 . . ?
C1 C2 H2A 109.9 . . ?
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N2 C2 H2B 109.9 . . ?
C1 C2 H2B 109.9 . . ?
H2A C2 H2B 108.3 . . ?
N2 C3 C4 110.80(11) . . ?
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 . . ?
C5 C4 C9 118.99(13) . . ?
C5 C4 C3 120.18(12) . . ?
C9 C4 C3 120.84(12) . . ?
C6 C5 C4 120.49(13) . . ?
C6 C5 H5 119.8 . . ?
C4 C5 H5 119.8 . . ?
C5 C6 C7 120.51(13) . . ?
C5 C6 H6 119.7 . . ?
C7 C6 H6 119.7 . . ?
C8 C7 C6 119.07(13) . . ?
C8 C7 C10 122.78(12) . 2_666 ?
C6 C7 C10 118.09(12) . 2_666 ?
C9 C8 C7 120.24(13) . . ?
C9 C8 H8 119.9 . . ?
C7 C8 H8 119.9 . . ?
C8 C9 C4 120.68(13) . . ?
C8 C9 H9 119.7 . . ?
C4 C9 H9 119.7 . . ?
N3 C10 C7 113.18(11) . 2_666 ?
N3 C10 H10A 108.9 . . ?
C7 C10 H10A 108.9 2_666 . ?
N3 C10 H10B 108.9 . . ?
C7 C10 H10B 108.9 2_666 . ?
H10A C10 H10B 107.8 . . ?
N3 C11 C12 112.66(11) . . ?
N3 C11 H11A 109.1 . . ?
C12 C11 H11A 109.1 . . ?
N3 C11 H11B 109.1 . . ?
C12 C11 H11B 109.1 . . ?
H11A C11 H11B 107.8 . . ?
N1 C12 C11 111.73(11) . . ?
N1 C12 H12A 109.3 . . ?
C11 C12 H12A 109.3 . . ?
N1 C12 H12B 109.3 . . ?
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C11 C12 H12B 109.3 . . ?
H12A C12 H12B 107.9 . . ?
N1 C13 H13A 109.5 . . ?
N1 C13 H13B 109.5 . . ?
H13A C13 H13B 109.5 . . ?
N1 C13 H13C 109.5 . . ?
H13A C13 H13C 109.5 . . ?
H13B C13 H13C 109.5 . . ?

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 _geom_torsion_site_symmetry_3
 _geom_torsion_site_symmetry_4
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C12 N1 C1 C2 151.97(11) . . . . ?
C3 N2 C2 C1 -166.91(11) . . . . ?
N1 C1 C2 N2 -55.49(14) . . . . ?
C2 N2 C3 C4 -170.62(11) . . . . ?
N2 C3 C4 C5 115.04(14) . . . . ?
N2 C3 C4 C9 -64.96(17) . . . . ?
C9 C4 C5 C6 -0.2(2) . . . . ?
C3 C4 C5 C6 179.79(13) . . . . ?
C4 C5 C6 C7 1.2(2) . . . . ?
C5 C6 C7 C8 -0.9(2) . . . . ?
C5 C6 C7 C10 176.30(13) . . . 2_666 ?
C6 C7 C8 C9 -0.4(2) . . . . ?
C10 C7 C8 C9 -177.46(14) 2_666 . . . ?
C7 C8 C9 C4 1.4(2) . . . . ?
C5 C4 C9 C8 -1.1(2) . . . . ?
C3 C4 C9 C8 178.91(14) . . . . ?
C11 N3 C10 C7 152.03(11) . . . 2_666 ?
C10 N3 C11 C12 64.12(15) . . . . ?
C1 N1 C12 C11 -168.18(11) . . . . ?
C13 N1 C12 C11 66.91(14) . . . . ?
N3 C11 C12 N1 52.51(15) . . . . ?
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 _geom_hbond_atom_site_label_A
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 _geom_hbond_distance_DA
 _geom_hbond_angle_DHA
 _geom_hbond_site_symmetry_A
N2 H21N Br1  0.87(2) 2.48(2) 3.3398(12) 168.0(19) .
N2 H22N Br1  0.89(2) 2.46(2) 3.3428(12) 170.0(18) 1_455
N3 H31N Br1  0.86(2) 2.52(2) 3.3622(12) 167.2(19) .
N3 H32N Br2  0.90(2) 2.42(2) 3.2614(12) 154.7(18) .

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_diffrn_measured_fraction_theta_full   0.999
_refine_diff_density_max    0.80
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;
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loop_
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 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
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 '-x, -y, -z'

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I2 I 0.24252(5) 0.43797(4) 0.27786(3) 0.02174(11) Uani 1 1 d . . .
N1 N 0.6424(5) 0.6304(5) 0.0920(3) 0.0114(8) Uani 1 1 d . . .
C2 C 0.4538(7) 0.7305(6) 0.0556(4) 0.0153(11) Uani 1 1 d . . .
H2A H 0.3587 0.6742 0.0760 0.018 Uiso 1 1 calc R . .
H2B H 0.4606 0.7678 -0.0177 0.018 Uiso 1 1 calc R . .
C3 C 0.3876(7) 0.8622(6) 0.0954(4) 0.0144(11) Uani 1 1 d . . .
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H3B H 0.2666 0.9336 0.0640 0.017 Uiso 1 1 calc R . .
N4 N 0.3550(6) 0.8096(5) 0.2053(3) 0.0131(9) Uani 1 1 d . . .
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H4B H 0.451(8) 0.736(6) 0.231(4) 0.016 Uiso 1 1 d . . .
C5 C 0.3079(7) 0.9347(6) 0.2480(4) 0.0120(10) Uani 1 1 d . . .
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H5B H 0.4170 0.9768 0.2333 0.014 Uiso 1 1 calc R . .
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C7 C 0.1698(7) 0.7852(6) 0.4055(4) 0.0136(10) Uani 1 1 d . . .
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C8 C 0.1257(7) 0.7531(6) 0.5059(4) 0.0158(11) Uani 1 1 d . . .
H8 H 0.0588 0.6818 0.5374 0.019 Uiso 1 1 calc R . .
C9 C 0.3234(7) 0.9575(6) 0.4132(4) 0.0160(11) Uani 1 1 d . . .
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H10 H 0.3163 0.9738 0.5516 0.020 Uiso 1 1 calc R . .
C11 C 0.1780(7) 0.8235(6) 0.5608(4) 0.0131(10) Uani 1 1 d . . .
C12 C 0.1186(7) 0.7971(6) 0.6687(4) 0.0153(11) Uani 1 1 d . . .
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H12B H -0.0160 0.7941 0.6779 0.018 Uiso 1 1 calc R . .
N13 N 0.2442(6) 0.6546(5) 0.7363(3) 0.0127(9) Uani 1 1 d . . .
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H13B H 0.361(8) 0.659(6) 0.728(4) 0.015 Uiso 1 1 d . . .
C14 C 0.1769(7) 0.6383(6) 0.8421(3) 0.0147(11) Uani 1 1 d . . .
H14A H 0.0560 0.6109 0.8544 0.018 Uiso 1 1 calc R . .
H14B H 0.1490 0.7355 0.8540 0.018 Uiso 1 1 calc R . .
C15 C 0.3258(7) 0.5203(6) 0.9132(4) 0.0139(10) Uani 1 1 d . . .
H15A H 0.4490 0.5445 0.8979 0.017 Uiso 1 1 calc R . .
H15B H 0.2833 0.5217 0.9815 0.017 Uiso 1 1 calc R . .
C16 C 0.7979(7) 0.6891(6) 0.0372(4) 0.0176(11) Uani 1 1 d . . .
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H16B H 0.9222 0.6197 0.0636 0.026 Uiso 1 1 d R . .
H16C H 0.7805 0.7870 0.0444 0.026 Uiso 1 1 d R . .
O1S O 0.7073(5) 0.6193(4) 0.3133(3) 0.0169(8) Uani 1 1 d . . .
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H2SB H 0.6271 0.5940 0.4551 0.040 Uiso 1 1 d R . .
H2SC H 0.7031 0.7359 0.4022 0.040 Uiso 1 1 d R . .

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I2 0.01451(17) 0.0204(2) 0.0289(2) -0.00603(17) 0.00217(14) -0.00638(15)
N1 0.0121(19) 0.008(2) 0.013(2) -0.0037(17) 0.0001(15) -0.0019(16)
C2 0.019(2) 0.016(3) 0.010(2) -0.006(2) 0.0006(19) -0.003(2)
C3 0.015(2) 0.015(3) 0.010(2) -0.003(2) -0.0011(18) -0.002(2)
N4 0.013(2) 0.013(2) 0.014(2) -0.0078(19) 0.0001(17) -0.0025(18)
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C6 0.010(2) 0.011(3) 0.013(2) -0.004(2) -0.0008(18) 0.0014(19)
C7 0.017(2) 0.011(3) 0.015(3) -0.007(2) -0.0037(19) -0.004(2)
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C12 0.016(2) 0.014(3) 0.011(2) -0.004(2) -0.0032(19) 0.001(2)
N13 0.011(2) 0.013(2) 0.011(2) -0.0033(18) -0.0015(16) -0.0011(18)
C14 0.015(2) 0.018(3) 0.011(2) -0.009(2) 0.0024(18) -0.002(2)
C15 0.019(2) 0.016(3) 0.009(2) -0.008(2) -0.0013(19) -0.004(2)
C16 0.018(2) 0.016(3) 0.022(3) -0.010(2) 0.007(2) -0.008(2)
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C2 H2A 0.9900 . ?
C2 H2B 0.9900 . ?
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C3 H3B 0.9900 . ?
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N4 H4B 0.85(6) . ?
C5 C6 1.510(7) . ?
C5 H5A 0.9900 . ?
C5 H5B 0.9900 . ?
C6 C7 1.376(7) . ?
C6 C9 1.389(7) . ?
C7 C8 1.381(7) . ?
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C12 H12B 0.9900 . ?
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N1 C2 C3 111.4(4) . . ?
N1 C2 H2A 109.4 . . ?
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N1 C2 H2B 109.4 . . ?
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H2A C2 H2B 108.0 . . ?
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H12A C12 H12B 107.7 . . ?
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C2 C3 N4 C5 174.6(4) . . . . ?
C3 N4 C5 C6 178.2(4) . . . . ?
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N4 H4B O1S  0.85(6) 2.13(6) 2.935(6) 157(5) .
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N13 H13B I2  0.86(5) 2.76(5) 3.573(5) 159(5) 2_666
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