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Cation-reinforced donor-acceptor pseudorotaxanes

Pascu, Sofia I.; Jarrosson, Thibaut; Naumann, Christoph; Otto, Sijbren; Kaiser, Guido; Sanders, Jeremy K.M.

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# Supplementary Material (ESI) for New Journal of Chemistry
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_publ_contact_author_address
;
Department of Chemistry
University of Cambridge
Lensfield Road
Cambridge
CB2 1EW
UK
;
_publ_contact_author_email   JKMS@CAM.AC.UK

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Cation-reinforced donor-acceptor pseudorotaxanes
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loop_
_publ_author_name
J.Sanders
'Thibaut Jarrosson'
'Guido Kaiser'
'Christoph Naumann'
'Sijbren Otto'
'Sofia Ioana Pascu'
#####
## denoted [Li212]Br2 in text ##
#####

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;
?
;

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# _atom_sites_solution_secondary difmap
_atom_sites_solution_hydrogens geom

# choose from 'none, undef, noref, refall, refxyz, refU, constr, mixed'
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_refine_ls_hydrogen_treatment      mixed

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# General computing
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_computing_structure_refinement
;
CRYSTALS (Watkin et al 2003)
;
_computing_publication_material
;
CRYSTALS (Watkin et al 2003)
;
_computing_molecular_graphics
;
CAMERON (Watkin et al 1996)
;
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_cell_angle_alpha                   90
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_cell_angle_beta                    94.5550(7)
_cell_length_c                      26.2432(6)
_cell_angle_gamma                   90
_cell_volume                        4255.49(15)

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_symmetry_space_group_name_H-M     'P 1 21/c 1 '
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_symmetry_equiv_pos_as_xyz
x, y, z
-x, -y, -z
-x, y+1/2, -z+1/2
x, -y+1/2, z+1/2

# choose from: rm (reference molecule of
# known chirality), ad (anomolous
# dispersion - ie. Flack param), rmad
# (both rm and ad), syn (known from
# synthetic pathway), unk (unknown)
# or . (not applicable).

_chemical_absolute_configuration .

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_atom_type_scatter_dispersion_real
_atom_type_scatter_dispersion_imag
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0.2156 International_Tables_Vol_IV_Table_2.2B

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 'O' 0.0106 0.0060 3.0485 13.2771 2.2868 5.7011 1.5463 0.3239 0.8670 32.9089
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 'N' 0.0061 0.0033 12.2126 0.0057 3.1322 9.8933 2.0125 28.9975 1.1663 0.5826
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 168.2610 0.0377 International_Tables_Vol_IV_Table_2.2B
 'Cl' 0.1484 0.1585 11.4604 0.0104 7.1964 1.1662 6.2556 18.5194 1.6455 47.7784
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 'Br' -0.2901 2.4595 17.1789 2.1723 5.2358 16.5796 5.6377 0.2609 3.9851
 41.4328 2.9557 International_Tables_Vol_IV_Table_2.2B

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 C36 H44 O10, C22 H28 N2 O4, 5(C H2 Cl3), 2(H2 O), 2Br
 ;

_chemical_compound_source

;
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_chemical_formula_weight 1827.73

_cell_measurement_reflns_used 24872
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Non-dispersive F(000):

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_diffn_measurement_device_type

;
 Nonius Kappa CCD

;
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 _computing_data_collection

COLLECT (Nonius BV, 1997)

_computing_data_reduction

Denzo/Scalepack (Otwinowski & Minor, 1996)

_computing_cell_refinement

Denzo/Scalepack (Otwinowski & Minor, 1996)

_computing_structure_solution

SIR97 (Altomare et al, 1999)

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_exptl_absorpt_correction_T_max 0.875
_exptl_absorpt_process_details
;
multi-scan from symmetry-related measurements
Sortav (Blessing 1995)
;

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_diffrn_standards_interval_count 0
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_diffrn_reflns_number           24285
_reflns_number_total            7714
_diffrn_reflns_av_R_equivalents 0.03
# Number of reflections with Friedels Law is 7714
# Number of reflections without Friedels Law is 0
# Theoretical number of reflections is about 7775

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_diffrn_reflns_theta_full       24.586
_diffrn_measured_fraction_theta_full 0.990

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_diffrn_reflns_limit_k_min      -12
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_diffrn_reflns_limit_l_min      -22
_diffrn_reflns_limit_l_max      31
_reflns_limit_h_min             -19
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_reflns_limit_l_max             31

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_refine_ls_number_restraints    204
_refine_ls_number_parameters    541

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_refine_ls_wR_factor_ref        0.1184
_refine_ls_goodness_of_fit_ref  1.0981

#_reflns_number_all 7714
_refine_ls_R_factor_all         0.1577
_refine_ls_wR_factor_all        0.1528

```

```

# The I/u(I) cutoff below was used for refinement as
# well as the _gt R-factors:
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_reflns_number_gt                 3794
_refine_ls_R_factor_gt            0.1000
_refine_ls_wR_factor_gt           0.1184

_refine_ls_shift/su_max           0.088587
_refine_ls_structure_factor_coef  F
_refine_ls_weighting_scheme        calc
_refine_ls_weighting_details
;
Method, part 1, Chebychev polynomial, (Watkin, 1994, Prince, 1982)
[weight] = 1.0/[A~0~*T~0~(x)+A~1~*T~1~(x) ... +A~n-1~*T~n-1~(x)]
where A~i~ are the Chebychev coefficients listed below and x= Fcalc/Fmax
Method = Robust Weighting (Prince, 1982)
W = [weight] * [1-(deltaF/6*sigmaF)^2]^2^
A~i~ are:
2.93 1.99 2.06
;
_diffn_radiation_type             'Mo K\alpha'
_diffn_radiation_wavelength       0.71073

## -----REFERENCES -----##
## Insert your own references - in alphabetic order
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;

SIR97 (Altomare et al, 1999)

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Acta Cryst, A50, 411-437

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Crystallography Laboratory, OXFORD, UK.
;

# Uequiv = arithmetic mean of Ui
# i.e. Uequiv = (U1+U2+U3)/3

# Replace trailing . with the number of unfound
# hydrogen atoms attaced to relavent atom

loop_
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_atom_site_type_symbol
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_atom_site_U_iso_or_equiv
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_atom_site_refinement_flags_adp
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_atom_site_disorder_group
_atom_site_attached_hydrogens
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Br1 Br 0.97035(7) 0.21643(10) 0.05204(4) 0.0556 1.0000 Uani . . . . .
N1 N 0.5749(6) -0.2416(10) 0.0772(4) 0.0768 1.0000 Uani . . . . .
O1 O 0.6953(4) 0.1056(7) 0.1232(3) 0.0560 1.0000 Uani . . . . .
O2 O 0.8278(4) -0.0995(7) 0.1330(2) 0.0532 1.0000 Uani . . . . .
O3 O 0.8757(5) -0.3303(7) 0.0972(2) 0.0568 1.0000 Uani . . . . .
O4 O 0.1962(4) 0.3641(7) -0.0028(3) 0.0583 1.0000 Uani . . . . .
O5 O 0.3420(5) 0.2848(8) 0.0673(3) 0.0658 1.0000 Uani . . . . .
O6 O 0.7014(5) -0.1646(8) 0.0503(3) 0.0717 1.0000 Uani . . . . .
O7 O 0.4319(6) -0.2706(11) 0.0869(4) 0.0912 1.0000 Uani . . . . .
O8 O 0.8909(5) -0.0786(7) 0.0201(3) 0.0673 1.0000 Uani . . . . .
C1 C 0.6363(7) 0.1868(9) 0.0984(3) 0.0554 1.0000 Uani . . . . .
C2 C 0.6560(7) 0.2813(11) 0.0631(4) 0.0621 1.0000 Uani . . . . .
C3 C 0.5874(8) 0.3581(11) 0.0410(4) 0.0688 1.0000 Uani . . . . .
C4 C 0.5067(8) 0.3374(11) 0.0510(4) 0.0642 1.0000 Uani . . . . .
C5 C 0.4008(7) 0.2093(12) 0.0936(4) 0.0635 1.0000 Uani . . . . .
C6 C 0.3804(7) 0.1064(13) 0.1251(4) 0.0684 1.0000 Uani . . . . .
C7 C 0.4485(8) 0.0336(13) 0.1500(4) 0.0726 1.0000 Uani . . . . .
C8 C 0.5317(8) 0.0625(12) 0.1434(4) 0.0652 1.0000 Uani . . . . .
C9 C 0.5519(7) 0.1628(11) 0.1103(4) 0.0551 1.0000 Uani . . . . .
C10 C 0.4865(7) 0.2400(10) 0.0841(4) 0.0569 1.0000 Uani . . . . .
C11 C 0.7800(7) 0.1186(11) 0.1103(4) 0.0617 1.0000 Uani . . . . .
C12 C 0.8362(7) 0.0343(11) 0.1454(4) 0.0595 1.0000 Uani . . . . .
C13 C 0.8897(8) -0.1730(12) 0.1625(4) 0.0638 1.0000 Uani . . . . .
C14 C 0.8735(8) -0.3171(12) 0.1509(4) 0.0682 1.0000 Uani . . . . .
C15 C 0.8606(8) -0.4610(10) 0.0798(5) 0.0659 1.0000 Uani . . . . .
C16 C 0.8645(7) -0.4580(10) 0.0228(4) 0.0593 1.0000 Uani . . . . .
C17 C 0.2005(7) 0.3641(12) 0.0527(4) 0.0682 1.0000 Uani . . . . .
C18 C 0.2545(8) 0.2572(12) 0.0743(4) 0.0702 1.0000 Uani . . . . .
C19 C 0.6255(8) -0.1577(13) 0.0500(4) 0.0664 1.0000 Uani . . . . .
C20 C 0.5668(6) -0.0666(13) 0.0212(4) 0.0621 1.0000 Uani . . . . .
C21 C 0.4840(6) -0.1002(12) 0.0325(4) 0.0607 1.0000 Uani . . . . .
C22 C 0.4895(8) -0.2113(15) 0.0688(5) 0.0772 1.0000 Uani . . . . .
C23 C 0.5856(6) 0.0292(13) -0.0118(4) 0.0629 1.0000 Uani . . . . .
C24 C 0.6036(18) -0.349(3) 0.1063(10) 0.1864 1.0000 Uani D U . . . .
C25 C 0.6293(18) -0.331(2) 0.1610(10) 0.1870 1.0000 Uani D U . . . .
C26 C 0.6595(17) -0.461(2) 0.1825(9) 0.1880 1.0000 Uani D U . . . .
C27 C 0.6859(17) -0.453(2) 0.2366(9) 0.1889 1.0000 Uani D U . . . .
C28 C 0.739(3) -0.560(4) 0.2568(13) 0.1892 0.5000 Uani D U . 1 1 .
C29 C 0.792(3) -0.613(5) 0.218(2) 0.1895 0.5000 Uani D U . 1 1 .
C280 C 0.685(3) -0.581(3) 0.2629(14) 0.1892 0.5000 Uani D U . 1 2 .
C290 C 0.751(4) -0.669(4) 0.250(3) 0.1893 0.5000 Uani D U . 1 2 .
C30 C 1.0267(5) 0.2690(5) 0.18292(16) 0.1413 1.0000 Uani D U . . . .
C31 C 0.8663(2) 0.2082(4) -0.07485(13) 0.0878 1.0000 Uani D U . . . .
Cl1 Cl 1.1130(6) 0.3783(8) 0.1890(4) 0.1443 0.5000 Uani D U . 2 1 .
Cl2 Cl 1.0550(6) 0.1231(7) 0.2153(3) 0.1382 0.5000 Uani D U . 2 1 .
Cl3 Cl 0.9421(6) 0.3380(9) 0.2101(3) 0.1418 0.5000 Uani D U . 2 1 .
Cl101 Cl 1.0745(7) 0.4248(7) 0.1837(3) 0.1397 0.5000 Uani D U . 2 2 .
Cl102 Cl 1.1028(7) 0.1524(8) 0.2041(3) 0.1423 0.5000 Uani D U . 2 2 .
Cl103 Cl 0.9456(6) 0.2698(9) 0.2247(3) 0.1437 0.5000 Uani D U . 2 2 .

```

```

C14 C1 0.8922(5) 0.0578(7) -0.1010(3) 0.0900 0.5000 Uani D U . 3 1 .
C15 C1 0.8981(6) 0.3355(7) -0.1151(3) 0.0896 0.5000 Uani D U . 3 1 .
C16 C1 0.7567(5) 0.2163(9) -0.0714(4) 0.0863 0.5000 Uani D U . 3 1 .
C1104 C1 0.7576(5) 0.2401(8) -0.0714(4) 0.0858 0.5000 Uani D U . 3 2 .
C1105 C1 0.8781(6) 0.0723(7) -0.1157(3) 0.0905 0.5000 Uani D U . 3 2 .
C1106 C1 0.9138(5) 0.3441(7) -0.1001(3) 0.0878 0.5000 Uani D U . 3 2 .
C32 C 0.6032(8) -0.0137(14) 0.2756(5) 0.2104 0.5000 Uani D U . . . .
C1107 C1 0.6154(10) 0.0492(18) 0.3374(6) 0.2108 0.5000 Uani D U . . . .
C1108 C1 0.5375(9) -0.1489(18) 0.2750(6) 0.2095 0.5000 Uani D U . . . .
C1109 C1 0.7021(9) -0.061(2) 0.2570(6) 0.2112 0.5000 Uani D U . . . .
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H41 H 0.4610 0.3932 0.0341 0.0757 1.0000 Uiso . . . . .
H61 H 0.3203 0.0844 0.1302 0.0816 1.0000 Uiso . . . . .
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H81 H 0.5778 0.0116 0.1624 0.0779 1.0000 Uiso . . . . .
H111 H 0.7980 0.2119 0.1143 0.0735 1.0000 Uiso . . . . .
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H151 H 0.9050 -0.5209 0.0957 0.0787 1.0000 Uiso . . . . .
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H282 H 0.7015 -0.6320 0.2680 0.2281 0.5000 Uiso . . . 1 1 .
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H292 H 0.8297 -0.5417 0.2061 0.2285 0.5000 Uiso . . . 1 1 .
H293 H 0.7551 -0.6449 0.1875 0.2285 0.5000 Uiso . . . 1 1 .
H2801 H 0.6915 -0.5656 0.3007 0.2281 0.5000 Uiso . . . 1 2 .
H2802 H 0.6291 -0.6237 0.2536 0.2281 0.5000 Uiso . . . 1 2 .
H2901 H 0.7472 -0.7529 0.2683 0.2282 0.5000 Uiso . . . 1 2 .
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_atom_site_aniso_U_13

_atom_site_aniso_U_12

Li1 0.053(9) 0.052(10) 0.069(10) 0.002(8) -0.003(8) 0.009(7)
Br1 0.0672(7) 0.0517(6) 0.0477(5) -0.0042(5) 0.0041(4) 0.0100(6)
N1 0.059(6) 0.076(7) 0.092(7) -0.032(6) -0.014(5) 0.020(5)
O1 0.056(4) 0.057(4) 0.053(4) -0.005(3) -0.006(3) 0.015(3)
O2 0.053(4) 0.062(4) 0.043(4) -0.005(3) -0.006(3) 0.013(3)
O3 0.070(5) 0.050(4) 0.048(4) 0.001(3) -0.008(3) 0.010(3)
O4 0.060(4) 0.053(4) 0.059(4) -0.007(3) -0.011(3) 0.011(3)
O5 0.066(5) 0.069(5) 0.061(4) 0.005(4) -0.009(3) 0.011(4)
O6 0.045(4) 0.085(5) 0.083(5) -0.038(4) -0.010(4) 0.014(4)
O7 0.065(5) 0.101(7) 0.107(7) -0.002(6) 0.001(5) 0.001(5)
O8 0.084(5) 0.061(5) 0.060(4) -0.014(4) 0.023(4) -0.005(4)
C1 0.079(7) 0.051(6) 0.035(5) -0.013(4) -0.008(5) 0.015(5)
C2 0.074(7) 0.047(5) 0.063(6) -0.011(6) -0.008(5) 0.009(6)
C3 0.084(9) 0.051(7) 0.068(7) 0.004(5) -0.013(6) 0.000(6)
C4 0.064(7) 0.056(6) 0.069(7) -0.008(6) -0.015(6) 0.014(6)
C5 0.065(7) 0.072(7) 0.051(6) -0.014(6) -0.011(5) 0.018(6)
C6 0.061(7) 0.086(8) 0.056(6) 0.001(6) -0.002(5) 0.015(6)
C7 0.083(9) 0.091(9) 0.044(6) 0.005(6) 0.004(5) 0.019(7)
C8 0.077(8) 0.075(7) 0.042(6) -0.006(5) 0.000(5) 0.017(6)
C9 0.059(6) 0.055(6) 0.050(6) -0.019(5) -0.006(5) 0.008(5)
C10 0.069(7) 0.051(6) 0.049(5) -0.011(4) -0.010(5) 0.009(5)
C11 0.066(7) 0.052(6) 0.065(7) -0.011(5) -0.004(5) 0.008(5)
C12 0.062(6) 0.064(7) 0.052(6) -0.006(5) -0.002(5) 0.011(5)
C13 0.075(7) 0.069(7) 0.045(5) -0.011(5) -0.012(5) 0.023(6)
C14 0.079(8) 0.072(8) 0.052(6) 0.012(5) -0.004(5) 0.025(6)
C15 0.074(7) 0.042(6) 0.081(8) -0.004(5) 0.000(6) 0.015(5)
C16 0.071(7) 0.041(5) 0.064(6) -0.005(5) -0.013(5) 0.019(5)
C17 0.058(6) 0.079(8) 0.064(7) -0.009(6) -0.012(5) 0.011(6)
C18 0.067(7) 0.082(9) 0.060(6) -0.008(5) -0.005(5) 0.020(6)
C19 0.062(8) 0.075(8) 0.059(7) -0.035(6) -0.010(6) 0.017(6)
C20 0.043(6) 0.087(8) 0.056(6) -0.030(6) -0.002(5) 0.013(6)
C21 0.045(6) 0.077(8) 0.060(6) -0.034(6) 0.000(5) 0.008(5)
C22 0.064(8) 0.087(9) 0.078(8) -0.029(8) -0.011(6) 0.012(7)
C23 0.045(6) 0.085(8) 0.057(6) -0.031(6) -0.002(5) 0.007(6)
C24 0.170(11) 0.156(10) 0.236(15) 0.067(12) 0.033(12) 0.011(9)
C25 0.171(11) 0.156(10) 0.236(15) 0.067(12) 0.033(12) 0.011(9)
C26 0.172(11) 0.157(10) 0.237(15) 0.067(12) 0.033(12) 0.011(9)
C27 0.173(11) 0.159(10) 0.238(15) 0.067(12) 0.033(12) 0.011(9)
C28 0.173(12) 0.159(10) 0.238(15) 0.066(12) 0.033(12) 0.011(9)
C29 0.173(12) 0.160(10) 0.238(15) 0.066(12) 0.033(12) 0.011(9)
C280 0.173(12) 0.159(10) 0.238(15) 0.067(12) 0.033(12) 0.011(9)
C290 0.174(12) 0.159(10) 0.238(15) 0.067(12) 0.033(12) 0.011(9)
C30 0.240(6) 0.084(3) 0.094(3) 0.000(2) -0.028(3) -0.001(3)
C31 0.0843(19) 0.0788(17) 0.102(2) -0.0004(16) 0.0192(17) 0.0148(14)
Cl1 0.242(6) 0.089(3) 0.096(3) 0.002(3) -0.025(3) -0.005(3)
Cl2 0.237(6) 0.080(3) 0.090(3) -0.002(2) -0.030(3) -0.003(3)
Cl3 0.240(6) 0.082(3) 0.097(3) 0.000(3) -0.028(3) 0.002(3)
Cl101 0.238(6) 0.080(3) 0.094(3) 0.005(3) -0.030(3) 0.002(3)
Cl102 0.242(6) 0.085(3) 0.094(3) -0.001(3) -0.026(3) 0.002(3)
Cl103 0.240(6) 0.088(3) 0.097(3) -0.003(3) -0.026(3) -0.003(3)
Cl4 0.090(2) 0.078(2) 0.103(3) 0.001(2) 0.016(2) 0.020(2)
Cl5 0.084(2) 0.082(2) 0.105(3) 0.000(2) 0.020(2) 0.012(2)
Cl6 0.082(2) 0.077(2) 0.101(3) 0.001(2) 0.021(2) 0.013(2)
Cl104 0.082(2) 0.077(2) 0.101(3) 0.001(2) 0.021(2) 0.012(2)
Cl105 0.091(2) 0.079(2) 0.103(3) -0.001(2) 0.016(2) 0.020(2)
Cl106 0.083(2) 0.0801(19) 0.102(3) -0.0016(19) 0.020(2) 0.0133(17)
C32 0.180(8) 0.256(12) 0.198(9) -0.005(8) 0.036(6) 0.000(8)
Cl107 0.181(8) 0.257(12) 0.198(9) -0.005(8) 0.035(7) 0.000(8)

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Cl108 0.180(8) 0.256(12) 0.197(9) -0.004(8) 0.036(7) 0.001(8)
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N1 . C19 . 1.409(18) yes
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O1 . C11 . 1.419(13) yes
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O3 . C15 . 1.429(12) yes
O4 . C16 2_655 1.432(12) yes
O4 . C17 . 1.454(13) yes
O5 . C5 . 1.357(13) yes
O5 . C18 . 1.445(15) yes
O6 . C19 . 1.207(14) yes
O7 . C22 . 1.225(17) yes
O8 . H1 . 0.907 no
O8 . H2 . 0.903 no
C1 . C2 . 1.393(16) yes
C1 . C9 . 1.422(16) yes
C2 . C3 . 1.429(16) yes
C2 . H21 . 1.000 no
C3 . C4 . 1.345(17) yes
C3 . H31 . 1.000 no
C4 . C10 . 1.379(17) yes
C4 . H41 . 1.000 no
C5 . C6 . 1.395(17) yes
C5 . C10 . 1.438(17) yes
C6 . C7 . 1.427(16) yes
C6 . H61 . 1.000 no
C7 . C8 . 1.379(18) yes
C7 . H71 . 1.000 no
C8 . C9 . 1.399(17) yes
C8 . H81 . 1.000 no
C9 . C10 . 1.437(15) yes
C11 . C12 . 1.503(15) yes
C11 . H111 . 1.000 no
C11 . H112 . 1.000 no
C12 . H121 . 1.000 no
C12 . H122 . 1.000 no
C13 . C14 . 1.525(17) yes
C13 . H131 . 1.000 no
C13 . H132 . 1.000 no
C14 . H141 . 1.000 no
C14 . H142 . 1.000 no
C15 . C16 . 1.500(16) yes
C15 . H151 . 1.000 no
C15 . H152 . 1.000 no
C16 . H161 . 1.000 no
C16 . H162 . 1.000 no
C17 . C18 . 1.476(16) yes
C17 . H171 . 1.000 no

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C17 . H172 . 1.000 no
C18 . H181 . 1.000 no
C18 . H182 . 1.000 no
C19 . C20 . 1.483(17) yes
C20 . C21 . 1.414(16) yes
C20 . C23 . 1.357(17) yes
C21 . C23 2_655 1.398(16) yes
C21 . C22 . 1.48(2) yes
C23 . H231 . 1.000 no
C24 . C25 . 1.473(17) yes
C24 . H241 . 1.000 no
C24 . H242 . 1.000 no
C25 . C26 . 1.511(17) yes
C25 . H251 . 1.001 no
C25 . H252 . 1.000 no
C26 . C27 . 1.449(17) yes
C26 . H261 . 1.001 no
C26 . H262 . 1.001 no
C27 . C28 . 1.457(17) yes
C27 . H271 . 1.001 no
C27 . H272 . 1.001 no
C27 . C280 . 1.483(18) yes
C27 . H271 . 1.001 no
C27 . H272 . 1.001 no
C28 . C29 . 1.484(19) yes
C28 . H281 . 1.000 no
C28 . H282 . 1.001 no
C29 . H291 . 0.997 no
C29 . H292 . 1.003 no
C29 . H293 . 1.003 no
C280 . C290 . 1.451(19) yes
C280 . H2801 . 1.000 no
C280 . H2802 . 1.000 no
C290 . H2901 . 0.998 no
C290 . H2902 . 1.001 no
C290 . H2903 . 1.005 no
C30 . C11 . 1.766(9) yes
C30 . C12 . 1.759(8) yes
C30 . C13 . 1.722(8) yes
C30 . H301 . 1.000 no
C30 . C1101 . 1.766(8) yes
C30 . C1102 . 1.758(8) yes
C30 . C1103 . 1.757(9) yes
C30 . H301 . 1.000 no
C31 . C14 . 1.748(7) yes
C31 . C15 . 1.777(8) yes
C31 . C16 . 1.752(8) yes
C31 . H311 . 1.000 no
C31 . C1104 . 1.766(8) yes
C31 . C1105 . 1.775(7) yes
C31 . C1106 . 1.739(8) yes
C31 . H311 . 1.000 no
C32 . C1107 . 1.742(9) yes
C32 . C1108 . 1.733(9) yes
C32 . C1109 . 1.751(9) yes
C32 . H321 . 1.000 no
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_geom_angle_site_symmetry_2

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C1 . O1 . C11 . 117.0(8) yes
C12 . O2 . C13 . 109.9(7) yes
C14 . O3 . C15 . 113.0(8) yes
C16 2_655 O4 . C17 . 110.1(8) yes
C5 . O5 . C18 . 117.0(9) yes
H1 . O8 . H2 . 112.263 no
O1 . C1 . C2 . 123.6(10) yes
O1 . C1 . C9 . 114.2(9) yes
C2 . C1 . C9 . 122.1(9) yes
C1 . C2 . C3 . 116.6(11) yes
C1 . C2 . H21 . 121.683 no
C3 . C2 . H21 . 121.699 no
C2 . C3 . C4 . 122.8(11) yes
C2 . C3 . H31 . 118.583 no
C4 . C3 . H31 . 118.582 no
C3 . C4 . C10 . 120.7(10) yes
C3 . C4 . H41 . 119.653 no
C10 . C4 . H41 . 119.658 no
O5 . C5 . C6 . 123.4(11) yes
O5 . C5 . C10 . 114.2(11) yes
C6 . C5 . C10 . 122.4(10) yes
C5 . C6 . C7 . 117.6(11) yes
C5 . C6 . H61 . 121.216 no
C7 . C6 . H61 . 121.213 no
C6 . C7 . C8 . 122.0(12) yes
C6 . C7 . H71 . 119.001 no
C8 . C7 . H71 . 119.001 no
C7 . C8 . C9 . 120.3(10) yes
C7 . C8 . H81 . 119.832 no
C9 . C8 . H81 . 119.841 no
C1 . C9 . C8 . 122.0(10) yes
C1 . C9 . C10 . 117.3(10) yes
C8 . C9 . C10 . 120.5(10) yes
C5 . C10 . C9 . 117.1(10) yes
C5 . C10 . C4 . 122.6(10) yes
C9 . C10 . C4 . 120.3(11) yes
O1 . C11 . C12 . 109.2(9) yes
O1 . C11 . H111 . 109.527 no
C12 . C11 . H111 . 109.530 no
O1 . C11 . H112 . 109.528 no
C12 . C11 . H112 . 109.532 no
H111 . C11 . H112 . 109.471 no
C11 . C12 . O2 . 112.1(8) yes
C11 . C12 . H121 . 108.804 no
O2 . C12 . H121 . 108.803 no
C11 . C12 . H122 . 108.810 no
O2 . C12 . H122 . 108.805 no
H121 . C12 . H122 . 109.464 no
O2 . C13 . C14 . 107.7(9) yes
O2 . C13 . H131 . 109.908 no
C14 . C13 . H131 . 109.908 no
O2 . C13 . H132 . 109.908 no
C14 . C13 . H132 . 109.910 no
H131 . C13 . H132 . 109.465 no

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C13 . C14 . O3 . 105.9(9) yes
 C13 . C14 . H141 . 110.356 no
 O3 . C14 . H141 . 110.363 no
 C13 . C14 . H142 . 110.354 no
 O3 . C14 . H142 . 110.359 no
 H141 . C14 . H142 . 109.469 no
 O3 . C15 . C16 . 106.3(9) yes
 O3 . C15 . H151 . 110.254 no
 C16 . C15 . H151 . 110.262 no
 O3 . C15 . H152 . 110.251 no
 C16 . C15 . H152 . 110.259 no
 H151 . C15 . H152 . 109.466 no
 C15 . C16 . O4 2_655 107.4(8) yes
 C15 . C16 . H161 . 109.982 no
 O4 2_655 C16 . H161 . 109.980 no
 C15 . C16 . H162 . 109.982 no
 O4 2_655 C16 . H162 . 109.981 no
 H161 . C16 . H162 . 109.466 no
 O4 . C17 . C18 . 111.3(9) yes
 O4 . C17 . H171 . 109.003 no
 C18 . C17 . H171 . 109.004 no
 O4 . C17 . H172 . 109.006 no
 C18 . C17 . H172 . 109.006 no
 H171 . C17 . H172 . 109.463 no
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 C17 . C18 . H181 . 109.334 no
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 C17 . C18 . H182 . 109.334 no
 O5 . C18 . H182 . 109.335 no
 H181 . C18 . H182 . 109.466 no
 N1 . C19 . O6 . 124.8(12) yes
 N1 . C19 . C20 . 106.4(10) yes
 O6 . C19 . C20 . 128.8(14) yes
 C19 . C20 . C21 . 107.3(12) yes
 C19 . C20 . C23 . 128.2(11) yes
 C21 . C20 . C23 . 124.5(11) yes
 C20 . C21 . C23 2_655 120.8(12) yes
 C20 . C21 . C22 . 108.3(10) yes
 C23 2_655 C21 . C22 . 130.9(11) yes
 C21 . C22 . N1 . 106.3(13) yes
 C21 . C22 . O7 . 128.4(11) yes
 N1 . C22 . O7 . 125.2(14) yes
 C21 2_655 C23 . C20 . 114.6(10) yes
 C21 2_655 C23 . H231 . 122.689 no
 C20 . C23 . H231 . 122.685 no
 N1 . C24 . C25 . 119(2) yes
 N1 . C24 . H241 . 106.966 no
 C25 . C24 . H241 . 107.097 no
 N1 . C24 . H242 . 106.964 no
 C25 . C24 . H242 . 107.090 no
 H241 . C24 . H242 . 109.390 no
 C24 . C25 . C26 . 108.0(16) yes
 C24 . C25 . H251 . 109.845 no
 C26 . C25 . H251 . 109.871 no
 C24 . C25 . H252 . 109.858 no
 C26 . C25 . H252 . 109.864 no
 H251 . C25 . H252 . 109.398 no
 C25 . C26 . C27 . 112.0(17) yes
 C25 . C26 . H261 . 108.889 no
 C27 . C26 . H261 . 108.821 no
 C25 . C26 . H262 . 108.908 no

C27 . C26 . H262 . 108.865 no
H261 . C26 . H262 . 109.330 no
C26 . C27 . C28 . 115.1(17) yes
C26 . C27 . H271 . 108.111 no
C28 . C27 . H271 . 108.017 no
C26 . C27 . H272 . 108.119 no
C28 . C27 . H272 . 108.016 no
H271 . C27 . H272 . 109.333 no
C26 . C27 . C280 . 113.1(19) yes
C26 . C27 . H271 . 108.111 no
C280 . C27 . H271 . 134.518 no
C26 . C27 . H272 . 108.119 no
C280 . C27 . H272 . 75.421 no
H271 . C27 . H272 . 109.333 no
C27 . C28 . C29 . 111.5(17) yes
C27 . C28 . H281 . 109.211 no
C29 . C28 . H281 . 108.821 no
C27 . C28 . H282 . 109.124 no
C29 . C28 . H282 . 108.804 no
H281 . C28 . H282 . 109.379 no
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C27 . C280 . C290 . 113.6(19) yes
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H2901 . C290 . H2903 . 109.272 no
H2902 . C290 . H2903 . 109.005 no
C11 . C30 . C12 . 109.16(10) yes
C11 . C30 . C13 . 109.14(10) yes
C12 . C30 . C13 . 109.07(10) yes
C11 . C30 . H301 . 117.137 no
C12 . C30 . H301 . 108.782 no
C13 . C30 . H301 . 103.217 no
C1101 . C30 . C1102 . 109.06(10) yes
C1101 . C30 . C1103 . 108.95(10) yes
C1102 . C30 . C1103 . 109.01(10) yes
C1101 . C30 . H301 . 109.928 no
C1102 . C30 . H301 . 109.877 no
C1103 . C30 . H301 . 109.991 no
C14 . C31 . C15 . 109.04(10) yes
C14 . C31 . C16 . 109.11(10) yes
C15 . C31 . C16 . 109.04(10) yes
C14 . C31 . H311 . 95.035 no
C15 . C31 . H311 . 124.602 no
C16 . C31 . H311 . 108.612 no
C1104 . C31 . C1105 . 109.08(10) yes
C1104 . C31 . C1106 . 109.09(10) yes
C1105 . C31 . C1106 . 109.14(10) yes
C1104 . C31 . H311 . 109.873 no
C1105 . C31 . H311 . 109.825 no

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Cl106 . C31 . H311 . 109.816 no
Cl107 . C32 . Cl108 . 109.04(10) yes
Cl107 . C32 . Cl109 . 109.07(10) yes
Cl108 . C32 . Cl109 . 109.05(10) yes
Cl107 . C32 . H321 . 109.888 no
Cl108 . C32 . H321 . 109.896 no
Cl109 . C32 . H321 . 109.868 no
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#####
## denoted [Li212][B(C6F5)4]2 in text ##
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;
?
;

_chemical_melting_point            ?

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# _atom_sites_solution_secondary   difmap
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;
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;
CRYSTALS (Watkin et al 2001)
;
_computing_molecular_graphics
;
CAMERON (Watkin et al 1996)
;
#=====

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_symmetry_cell_setting             Triclinic

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# choose from: rm (reference molecule of
# known chirality), ad (anomalous
# dispersion - ie. Flack param), rmad
# (both rm and ad), syn (known from
# synthetic pathway), unk (unknown)
# or . (not applicable).

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0.0030 International_Tables_Vol_IV_Table_2.2B
'F   ' 0.0171 0.0103 3.5392 10.2825 2.6412 4.2944 1.5170 0.2615 1.0243 26.1476
0.2776 International_Tables_Vol_IV_Table_2.2B
'O   ' 0.0106 0.0060 3.0485 13.2771 2.2868 5.7011 1.5463 0.3239 0.8670 32.9089
0.2508 International_Tables_Vol_IV_Table_2.2B
'N   ' 0.0061 0.0033 12.2126 0.0057 3.1322 9.8933 2.0125 28.9975 1.1663 0.5826
-11.5290 International_Tables_Vol_IV_Table_2.2B
'B   ' 0.0013 0.0007 2.0545 23.2185 1.3326 1.0210 1.0979 60.3498 0.7068 0.1403
-0.1932 International_Tables_Vol_IV_Table_2.2B
'Li  ' -0.0003 0.0001 1.1282 3.9546 0.7508 1.0524 0.6175 85.3905 0.4653
168.2610 0.0377 International_Tables_Vol_IV_Table_2.2B
'Cl  ' 0.1484 0.1585 11.4604 0.0104 7.1964 1.1662 6.2556 18.5194 1.6455 47.7784
-9.5574 International_Tables_Vol_IV_Table_2.2B

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_chemical_compound_source
;
?
;
_chemical_formula_weight          2595.03

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_cell_measurement_temperature     180

_exptl_crystal_description       ' block '

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_exptl_crystal_density_meas ?
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_exptl_crystal_F_000      1310.000
_exptl_absorpt_coefficient_mu 0.243

_diffn_measurement_device_type
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Enraf Nonius Kappa CCD
;
_diffn_radiation_monochromator graphite
_computing_data_collection
;
COLLECT (Nonius BV, 1997)
;
_computing_data_reduction
;
Denzo/Scalepack (Otwinowski & Minor, 1996)
;
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;
Denzo/Scalepack (Otwinowski & Minor, 1996)
;
_computing_structure_solution
;
SIR97 (Altomare et al, 1999)
;
_diffn_measurement_method      \w
_exptl_absorpt_correction_type multi-scan
_exptl_absorpt_correction_T_min 0.934
_exptl_absorpt_correction_T_max 0.983
_exptl_absorpt_process_details
;
multi-scan from symmetry-related measurements
Sortav (Blessing 1995)
;
_diffn_ambient_temperature      180(2)
_diffn_reflns_number            26977
_reflns_number_total            12240
_diffn_reflns_av_R_equivalents  0.045
# Number of reflections with Friedels Law is 12240
# Number of reflections without Friedels Law is 0
# Theoretical number of reflections is about 12577

_diffn_reflns_theta_min        5.124
_diffn_reflns_theta_max        27.518
_diffn_measured_fraction_theta_max 0.974

_diffn_reflns_theta_full        27.48
_diffn_measured_fraction_theta_full 0.98

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_diffn_reflns_limit_h_max      13
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_diffn_reflns_limit_l_min      -25

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_diffn_reflms_limit_l_max      25
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_reflms_limit_l_max           25

_refine_diff_density_min      -0.72
_refine_diff_density_max      0.54

_refine_ls_number_reflms      8775
_refine_ls_number_parameters   784

#_refine_ls_R_factor_ref       0.0452
_refine_ls_wR_factor_ref      0.0562
_refine_ls_goodness_of_fit_ref 1.0940

#_reflms_number_all 12240
_refine_ls_R_factor_all       0.0682
_refine_ls_wR_factor_all      0.0666

# The I/u(I) cutoff below was used for refinement as
# well as the _gt R-factors:
_reflms_threshold_expression   I>3.00u(I)
_reflms_number_gt              8775
_refine_ls_R_factor_gt         0.0452
_refine_ls_wR_factor_gt       0.0562

_refine_ls_shift/su_max        0.002676
_refine_ls_structure_factor_coef F
_refine_ls_weighting_scheme    calc
_refine_ls_weighting_details
;
Method = Prince modified Chebychev polynomial, (Watkin, 1994)
W = [weight] * [1-(deltaF/6*sigmaF)^2]^2

2.06 0.801 1.63
;
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_diffn_radiation_wavelength    0.71073

## -----REFERENCES -----##
## Insert your own references - in alphabetic order
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;

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;

Uequiv = arithmetic mean of Ui
i.e. Uequiv = (U1+U2+U3)/3

Replace trailing . with the number of unfound
hydrogen atoms attaced to relavent atom

loop_
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_atom_site_type_symbol
_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
_atom_site_U_iso_or_equiv
_atom_site_occupancy
_atom_site_adp_type
_atom_site_attached_hydrogens
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F3 F 0.21707(15) 0.84968(16) 0.4858(1) 0.0802 1.0000 Uani .
F4 F 0.10450(15) 0.98310(13) 0.42285(9) 0.0674 1.0000 Uani .
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F6 F -0.26802(13) 0.77121(11) 0.53400(6) 0.0467 1.0000 Uani .
F7 F -0.44559(15) 0.71905(12) 0.61546(6) 0.0550 1.0000 Uani .
F8 F -0.67251(15) 0.62851(13) 0.56064(7) 0.0583 1.0000 Uani .
F9 F -0.71281(13) 0.58701(13) 0.41827(7) 0.0578 1.0000 Uani .
F10 F -0.53744(11) 0.6397(1) 0.33524(6) 0.0394 1.0000 Uani .
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F13 F -0.22371(13) 0.4060(1) 0.12715(7) 0.0507 1.0000 Uani .
F14 F -0.32591(12) 0.36101(9) 0.24437(8) 0.0450 1.0000 Uani .
F15 F -0.36048(13) 0.50599(9) 0.35618(6) 0.0424 1.0000 Uani .
F16 F -0.42528(12) 0.73889(9) 0.23037(6) 0.0396 1.0000 Uani .
F17 F -0.55012(14) 0.88802(12) 0.20549(7) 0.0555 1.0000 Uani .
F18 F -0.56935(16) 1.06299(12) 0.3067(1) 0.0660 1.0000 Uani .
F19 F -0.45623(15) 1.0837(1) 0.43494(9) 0.0597 1.0000 Uani .
F20 F -0.32739(12) 0.93349(9) 0.46115(6) 0.0417 1.0000 Uani .
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O3 O -0.21492(13) 1.1190(1) 0.20879(7) 0.0333 1.0000 Uani .
O4 O -0.05350(13) 1.2798(1) 0.29929(7) 0.0345 1.0000 Uani .
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C15 C -0.28442(17) 0.85695(13) 0.0475(1) 0.0286 1.0000 Uani .
C16 C -0.33658(15) 0.96924(13) -0.03168(9) 0.0262 1.0000 Uani .
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C22 C -0.1605(2) 1.11042(15) 0.2739(1) 0.0358 1.0000 Uani .
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C27 C 0.36374(19) 1.38376(15) 0.2452(1) 0.0347 1.0000 Uani .
C28 C 0.42866(18) 1.22092(16) 0.1928(1) 0.0347 1.0000 Uani .
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H82 H 0.1602 1.5519 0.1322 0.0438 1.0000 Uiso .

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H102 H 0.4173 1.4955 0.0892 0.0510 1.0000 Uiso .
H111 H 0.5959 1.6151 0.1026 0.0647 1.0000 Uiso .
H112 H 0.5100 1.7013 0.0848 0.0647 1.0000 Uiso .
H113 H 0.5335 1.5991 0.0241 0.0647 1.0000 Uiso .
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H151 H -0.3016 0.7946 0.0085 0.0343 1.0000 Uiso .
H171 H -0.3780 1.0738 -0.0893 0.0384 1.0000 Uiso .
H181 H -0.3432 1.2209 0.0067 0.0403 1.0000 Uiso .
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H282 H 0.4725 1.2169 0.2372 0.0407 1.0000 Uiso .
H291 H 0.4603 1.0700 0.1489 0.0407 1.0000 Uiso .
H292 H 0.3170 1.0843 0.1754 0.0407 1.0000 Uiso .
H541 H 0.2167 0.7763 0.1773 0.0637 1.0000 Uiso .
H542 H 0.0673 0.7496 0.1866 0.0637 1.0000 Uiso .
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B1 0.032(1) 0.0259(9) 0.0251(9) 0.0064(7) -0.0022(8) 0.0034(7)
F1 0.0514(7) 0.0466(7) 0.0440(7) 0.0166(6) -0.0047(6) 0.0132(6)
F2 0.0569(9) 0.0991(12) 0.0461(8) 0.0200(8) -0.0116(7) 0.0346(8)
F3 0.0410(8) 0.1092(14) 0.0779(11) -0.003(1) -0.0282(8) 0.0020(8)
F4 0.0502(8) 0.0606(9) 0.0778(11) -0.0033(8) -0.0045(8) -0.0206(7)
F5 0.0456(7) 0.0373(6) 0.0585(8) 0.0152(6) -0.0019(6) -0.0010(5)
F6 0.0525(7) 0.0589(8) 0.0257(5) 0.0048(5) -0.0093(5) 0.0013(6)
F7 0.078(1) 0.0684(9) 0.0217(6) 0.0143(6) 0.0025(6) 0.0146(7)
F8 0.0649(9) 0.074(1) 0.0436(7) 0.0269(7) 0.0223(7) 0.0124(7)
F9 0.0394(7) 0.0848(11) 0.0450(7) 0.0118(7) 0.0061(6) -0.0107(7)
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F11 0.0455(7) 0.0307(6) 0.0382(6) 0.0141(5) 0.0050(5) 0.0004(5)
F12 0.0570(8) 0.0533(8) 0.0361(6) 0.0110(6) 0.0173(6) 0.0080(6)
F13 0.0500(8) 0.0437(7) 0.0476(7) -0.0122(6) 0.0098(6) 0.0057(6)
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F16 0.0494(7) 0.0415(6) 0.0270(5) 0.0038(5) -0.0077(5) 0.0104(5)
F17 0.0599(9) 0.0660(9) 0.0478(7) 0.0236(7) -0.0144(6) 0.0188(7)
F18 0.071(1) 0.0476(8) 0.0860(11) 0.0217(8) -0.0110(8) 0.0289(7)
F19 0.0673(9) 0.0351(7) 0.071(1) -0.0048(6) -0.0045(8) 0.0188(6)
F20 0.0524(7) 0.0355(6) 0.0330(6) -0.0022(5) -0.0067(5) 0.0075(5)

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O2 0.0359(7) 0.0381(7) 0.0222(6) 0.0036(5) -0.0024(5) -0.0023(5)
O3 0.0433(8) 0.0286(6) 0.0270(6) 0.0051(5) -0.0040(5) 0.0009(5)
O4 0.0389(7) 0.0317(7) 0.0288(6) -0.0001(5) 0.0031(5) -0.0015(5)
O5 0.0355(7) 0.0287(6) 0.0307(6) 0.0058(5) -0.0000(5) 0.0022(5)
O6 0.0296(7) 0.0345(7) 0.0369(7) 0.0015(6) -0.0015(5) -0.0017(5)
O7 0.0420(7) 0.0281(6) 0.0261(6) 0.0048(5) -0.0027(5) 0.0028(5)
O8 0.0511(9) 0.0467(8) 0.0421(8) 0.0158(7) -0.0122(7) 0.0081(7)
C1 0.0242(8) 0.0290(8) 0.0263(8) 0.0081(7) 0.0001(6) -0.0013(6)
C2 0.0212(8) 0.0274(8) 0.0230(8) 0.0081(6) 0.0005(6) -0.0025(6)
C3 0.0204(7) 0.0290(8) 0.0213(7) 0.0066(6) -0.0001(6) -0.0019(6)
C4 0.0220(8) 0.0281(8) 0.0251(8) 0.0047(7) -0.0006(6) -0.0020(6)
C5 0.0237(8) 0.0286(8) 0.0213(7) 0.0083(6) -0.0022(6) -0.0023(6)
C6 0.0329(9) 0.0264(8) 0.0318(9) 0.0034(7) -0.0007(7) 0.0016(7)
C7 0.0338(9) 0.0249(8) 0.0315(9) 0.0046(7) 0.0032(7) -0.0009(7)
C8 0.0427(11) 0.0264(9) 0.041(1) 0.0108(8) 0.0017(8) 0.0012(8)
C9 0.0513(12) 0.0296(9) 0.0399(11) 0.0131(8) -0.0000(9) -0.0069(8)
C10 0.0469(12) 0.0403(11) 0.0404(11) 0.0061(9) -0.0004(9) -0.0071(9)
C11 0.0504(14) 0.0592(15) 0.0522(14) -0.0020(12) 0.0047(11) -0.0193(11)
C12 0.0256(8) 0.0274(8) 0.0283(8) 0.0060(7) 0.0021(7) -0.0027(6)
C13 0.0327(9) 0.0312(9) 0.0301(9) 0.0118(7) 0.0011(7) -0.0031(7)
C14 0.035(1) 0.0267(8) 0.0351(9) 0.0128(7) 0.0032(8) -0.0015(7)
C15 0.0288(9) 0.0239(8) 0.0330(9) 0.0074(7) 0.0019(7) -0.0015(6)
C16 0.0215(8) 0.0299(9) 0.0271(8) 0.0066(7) 0.0024(6) 0.0012(6)
C17 0.0309(9) 0.0345(9) 0.0305(9) 0.0121(7) 0.0007(7) 0.0050(7)
C18 0.037(1) 0.0265(9) 0.038(1) 0.0112(7) 0.0036(8) 0.0067(7)
C19 0.0300(9) 0.0251(8) 0.0336(9) 0.0060(7) 0.0038(7) 0.0030(7)
C20 0.0211(8) 0.0259(8) 0.0290(8) 0.0077(7) 0.0053(6) 0.0012(6)
C21 0.0217(8) 0.0264(8) 0.0312(9) 0.0089(7) 0.0026(6) 0.0001(6)
C22 0.0432(11) 0.037(1) 0.0275(9) 0.0099(8) -0.0044(8) -0.0038(8)
C23 0.0464(11) 0.0404(11) 0.0270(9) 0.0048(8) 0.0013(8) -0.0044(9)
C24 0.0396(11) 0.038(1) 0.034(1) -0.0030(8) 0.0025(8) 0.0034(8)
C25 0.0409(11) 0.0289(9) 0.0433(11) 0.0019(8) 0.0001(9) 0.0071(8)
C26 0.039(1) 0.0299(9) 0.035(1) 0.0088(8) -0.0022(8) -0.0022(7)
C27 0.035(1) 0.034(1) 0.0338(9) 0.0068(8) -0.0034(8) -0.0047(8)
C28 0.0267(9) 0.041(1) 0.0335(9) 0.0035(8) -0.0032(7) 0.0031(8)
C29 0.033(1) 0.039(1) 0.0297(9) 0.0075(8) -0.0022(7) 0.0048(8)
C30 0.0336(9) 0.0337(9) 0.0261(8) 0.0023(7) -0.0037(7) 0.0043(7)
C31 0.0394(11) 0.0427(11) 0.0288(9) 0.0054(8) -0.0032(8) 0.0085(8)
C32 0.0448(12) 0.0673(15) 0.029(1) 0.004(1) -0.0072(9) 0.0227(11)
C33 0.0375(12) 0.0732(17) 0.0396(12) -0.0065(11) -0.0113(9) 0.0037(11)
C34 0.0400(12) 0.0510(13) 0.0435(12) -0.005(1) -0.0020(9) -0.004(1)
C35 0.036(1) 0.038(1) 0.035(1) 0.0008(8) -0.0019(8) 0.0006(8)
C36 0.037(1) 0.0274(8) 0.0245(8) 0.0056(7) 0.0004(7) 0.0056(7)
C37 0.0444(11) 0.0334(9) 0.0258(9) 0.0071(7) -0.0041(8) 0.0069(8)
C38 0.0598(13) 0.0402(11) 0.0210(8) 0.0101(8) 0.0026(8) 0.0163(9)
C39 0.0472(12) 0.0428(11) 0.033(1) 0.0163(8) 0.0133(9) 0.0101(9)
C40 0.0398(11) 0.0449(11) 0.034(1) 0.0102(8) 0.0039(8) 0.0031(9)
C41 0.037(1) 0.037(1) 0.0230(8) 0.0069(7) -0.0001(7) 0.0043(8)
C42 0.0292(9) 0.0260(8) 0.0258(8) 0.0072(7) -0.0032(7) 0.0036(6)
C43 0.0314(9) 0.0296(9) 0.0319(9) 0.0105(7) 0.0009(7) 0.0029(7)
C44 0.0316(9) 0.0228(8) 0.043(1) 0.0049(7) -0.0051(8) 0.0021(7)
C45 0.0297(9) 0.036(1) 0.0349(9) -0.0012(8) 0.0015(7) 0.0074(7)
C46 0.0311(9) 0.040(1) 0.0288(9) 0.0096(8) 0.0055(7) 0.0057(7)
C47 0.0304(9) 0.0278(8) 0.0308(9) 0.0087(7) -0.0022(7) 0.0026(7)
C48 0.0318(9) 0.0262(8) 0.0306(9) 0.0089(7) -0.0005(7) 0.0026(7)
C49 0.0338(9) 0.0330(9) 0.0299(9) 0.0107(7) 0.0006(7) 0.0048(7)
C50 0.0381(11) 0.0449(11) 0.0417(11) 0.0180(9) -0.0066(9) 0.0083(9)
C51 0.0420(11) 0.0380(11) 0.0574(13) 0.020(1) -0.002(1) 0.0146(9)
C52 0.0430(11) 0.0286(9) 0.0497(12) 0.0028(8) 0.0037(9) 0.0084(8)

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C53 0.036(1) 0.0305(9) 0.0308(9) 0.0047(7) -0.0026(7) 0.0034(7)
C54 0.0668(16) 0.0466(13) 0.0457(13) 0.009(1) -0.0083(11) 0.0035(11)
C11 0.1087(6) 0.0441(3) 0.0606(4) 0.0080(3) -0.0078(4) 0.0171(3)
C12 0.2522(15) 0.0780(6) 0.0500(4) 0.0135(4) -0.0119(6) 0.0881(8)
_refine_ls_extinction_method      None
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_geom_bond_atom_site_label_1
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_geom_bond_atom_site_label_2
_geom_bond_site_symmetry_2
_geom_bond_distance
_geom_bond_publ_flag
N1 . C1 . 1.402(2) yes
N1 . C4 . 1.377(2) yes
N1 . C6 . 1.458(2) yes
B1 . C30 . 1.646(3) yes
B1 . C36 . 1.656(3) yes
B1 . C42 . 1.656(3) yes
B1 . C48 . 1.662(3) yes
F1 . C31 . 1.363(3) yes
F2 . C32 . 1.342(3) yes
F3 . C33 . 1.340(3) yes
F4 . C34 . 1.351(3) yes
F5 . C35 . 1.354(3) yes
F6 . C37 . 1.360(2) yes
F7 . C38 . 1.349(2) yes
F8 . C39 . 1.339(2) yes
F9 . C40 . 1.345(3) yes
F10 . C41 . 1.357(2) yes
F11 . C47 . 1.355(2) yes
F12 . C46 . 1.346(2) yes
F13 . C45 . 1.346(2) yes
F14 . C44 . 1.348(2) yes
F15 . C43 . 1.349(2) yes
F16 . C49 . 1.349(2) yes
F17 . C50 . 1.350(2) yes
F18 . C51 . 1.348(2) yes
F19 . C52 . 1.347(2) yes
F20 . C53 . 1.347(2) yes
O1 . C1 . 1.204(2) yes
O2 . C4 . 1.216(2) yes
O3 . C12 . 1.368(2) yes
O3 . C22 . 1.431(2) yes
O4 . C23 . 1.429(3) yes
O4 . C24 . 1.429(2) yes
O5 . C25 . 1.428(2) yes
O5 . C26 . 1.428(2) yes
O6 . C27 . 1.437(2) yes
O6 . C28 . 1.424(2) yes
O7 . C16 2_575 1.370(2) yes
O7 . C29 . 1.437(2) yes
C1 . C2 . 1.490(2) yes
C2 . C3 . 1.395(2) yes
C2 . C5 . 1.386(2) yes
C3 . C4 . 1.483(2) yes
C3 . C5 2_575 1.383(2) yes
C5 . H51 . 1.000 no
C6 . C7 . 1.524(3) yes
C6 . H61 . 1.000 no
C6 . H62 . 1.000 no
C7 . C8 . 1.530(3) yes

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C7 . H71 . 1.000 no
C7 . H72 . 1.000 no
C8 . C9 . 1.527(3) yes
C8 . H81 . 1.000 no
C8 . H82 . 1.000 no
C9 . C10 . 1.515(3) yes
C9 . H91 . 1.000 no
C9 . H92 . 1.000 no
C10 . C11 . 1.524(3) yes
C10 . H101 . 1.000 no
C10 . H102 . 1.000 no
C11 . H111 . 1.000 no
C11 . H112 . 1.000 no
C11 . H113 . 1.000 no
C12 . C13 . 1.375(3) yes
C12 . C20 . 1.431(2) yes
C13 . C14 . 1.415(3) yes
C13 . H131 . 1.000 no
C14 . C15 . 1.365(3) yes
C14 . H141 . 1.000 no
C15 . C21 . 1.417(2) yes
C15 . H151 . 1.000 no
C16 . C17 . 1.372(3) yes
C16 . C21 . 1.431(2) yes
C17 . C18 . 1.408(3) yes
C17 . H171 . 1.000 no
C18 . C19 . 1.369(3) yes
C18 . H181 . 1.000 no
C19 . C20 . 1.419(2) yes
C19 . H191 . 1.000 no
C20 . C21 . 1.422(2) yes
C22 . C23 . 1.506(3) yes
C22 . H221 . 1.000 no
C22 . H222 . 1.000 no
C23 . H231 . 1.000 no
C23 . H232 . 1.000 no
C24 . C25 . 1.502(3) yes
C24 . H241 . 1.000 no
C24 . H242 . 1.000 no
C25 . H251 . 1.000 no
C25 . H252 . 1.000 no
C26 . C27 . 1.500(3) yes
C26 . H261 . 1.000 no
C26 . H262 . 1.000 no
C27 . H271 . 1.000 no
C27 . H272 . 1.000 no
C28 . C29 . 1.504(3) yes
C28 . H281 . 1.000 no
C28 . H282 . 1.000 no
C29 . H291 . 1.000 no
C29 . H292 . 1.000 no
C30 . C31 . 1.391(3) yes
C30 . C35 . 1.392(3) yes
C31 . C32 . 1.379(3) yes
C32 . C33 . 1.378(4) yes
C33 . C34 . 1.366(4) yes
C34 . C35 . 1.393(3) yes
C36 . C37 . 1.393(2) yes
C36 . C41 . 1.392(3) yes
C37 . C38 . 1.384(3) yes
C38 . C39 . 1.369(3) yes


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C39 . C40 . 1.381(3) yes
C40 . C41 . 1.375(3) yes
C42 . C43 . 1.388(2) yes
C42 . C47 . 1.390(3) yes
C43 . C44 . 1.388(3) yes
C44 . C45 . 1.366(3) yes
C45 . C46 . 1.381(3) yes
C46 . C47 . 1.381(3) yes
C48 . C49 . 1.379(3) yes
C48 . C53 . 1.389(3) yes
C49 . C50 . 1.392(3) yes
C50 . C51 . 1.370(3) yes
C51 . C52 . 1.375(3) yes
C52 . C53 . 1.383(3) yes
C54 . C11 . 1.740(3) yes
C54 . C12 . 1.718(3) yes
C54 . H541 . 1.000 no
C54 . H542 . 1.000 no
loop_
_geom_angle_atom_site_label_1
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C1 . N1 . C4 . 111.97(14) yes
C1 . N1 . C6 . 122.61(15) yes
C4 . N1 . C6 . 125.41(14) yes
C30 . B1 . C36 . 114.23(15) yes
C30 . B1 . C42 . 103.69(14) yes
C36 . B1 . C42 . 111.46(14) yes
C30 . B1 . C48 . 113.53(15) yes
C36 . B1 . C48 . 101.13(14) yes
C42 . B1 . C48 . 113.18(14) yes
C12 . O3 . C22 . 116.57(14) yes
C23 . O4 . C24 . 111.59(15) yes
C25 . O5 . C26 . 113.29(15) yes
C27 . O6 . C28 . 110.82(14) yes
C16 2_575 O7 . C29 . 117.73(14) yes
N1 . C1 . O1 . 124.66(16) yes
N1 . C1 . C2 . 105.65(14) yes
O1 . C1 . C2 . 129.69(16) yes
C1 . C2 . C3 . 107.86(14) yes
C1 . C2 . C5 . 129.38(15) yes
C3 . C2 . C5 . 122.76(15) yes
C2 . C3 . C4 . 107.75(14) yes
C2 . C3 . C5 2_575 123.00(16) yes
C4 . C3 . C5 2_575 129.24(15) yes
N1 . C4 . O2 . 125.33(16) yes
N1 . C4 . C3 . 106.70(14) yes
O2 . C4 . C3 . 127.96(16) yes
C2 . C5 . C3 2_575 114.24(15) yes
C2 . C5 . H51 . 122.883 no
C3 2_575 C5 . H51 . 122.877 no
N1 . C6 . C7 . 112.86(15) yes
N1 . C6 . H61 . 108.622 no
C7 . C6 . H61 . 108.620 no
N1 . C6 . H62 . 108.619 no
C7 . C6 . H62 . 108.615 no

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H61 . C6 . H62 . 109.465 no
C6 . C7 . C8 . 110.05(16) yes
C6 . C7 . H71 . 109.324 no
C8 . C7 . H71 . 109.329 no
C6 . C7 . H72 . 109.328 no
C8 . C7 . H72 . 109.330 no
H71 . C7 . H72 . 109.465 no
C7 . C8 . C9 . 113.91(17) yes
C7 . C8 . H81 . 108.356 no
C9 . C8 . H81 . 108.350 no
C7 . C8 . H82 . 108.356 no
C9 . C8 . H82 . 108.350 no
H81 . C8 . H82 . 109.465 no
C8 . C9 . C10 . 114.97(17) yes
C8 . C9 . H91 . 108.079 no
C10 . C9 . H91 . 108.082 no
C8 . C9 . H92 . 108.076 no
C10 . C9 . H92 . 108.080 no
H91 . C9 . H92 . 109.469 no
C9 . C10 . C11 . 112.2(2) yes
C9 . C10 . H101 . 108.789 no
C11 . C10 . H101 . 108.793 no
C9 . C10 . H102 . 108.793 no
C11 . C10 . H102 . 108.794 no
H101 . C10 . H102 . 109.466 no
C10 . C11 . H111 . 109.462 no
C10 . C11 . H112 . 109.465 no
H111 . C11 . H112 . 109.472 no
C10 . C11 . H113 . 109.467 no
H111 . C11 . H113 . 109.479 no
H112 . C11 . H113 . 109.481 no
O3 . C12 . C13 . 124.54(16) yes
O3 . C12 . C20 . 114.54(15) yes
C13 . C12 . C20 . 120.92(16) yes
C12 . C13 . C14 . 119.62(17) yes
C12 . C13 . H131 . 120.190 no
C14 . C13 . H131 . 120.193 no
C13 . C14 . C15 . 121.30(16) yes
C13 . C14 . H141 . 119.347 no
C15 . C14 . H141 . 119.349 no
C14 . C15 . C21 . 120.01(16) yes
C14 . C15 . H151 . 119.994 no
C21 . C15 . H151 . 119.994 no
O7 2_575 C16 . C17 . 125.38(16) yes
O7 2_575 C16 . C21 . 113.67(15) yes
C17 . C16 . C21 . 120.93(16) yes
C16 . C17 . C18 . 119.60(17) yes
C16 . C17 . H171 . 120.201 no
C18 . C17 . H171 . 120.203 no
C17 . C18 . C19 . 121.73(16) yes
C17 . C18 . H181 . 119.138 no
C19 . C18 . H181 . 119.131 no
C18 . C19 . C20 . 119.60(17) yes
C18 . C19 . H191 . 120.201 no
C20 . C19 . H191 . 120.200 no
C12 . C20 . C19 . 122.01(16) yes
C12 . C20 . C21 . 118.13(15) yes
C19 . C20 . C21 . 119.84(16) yes
C15 . C21 . C16 . 121.73(16) yes
C15 . C21 . C20 . 119.98(16) yes
C16 . C21 . C20 . 118.28(15) yes

O3 . C22 . C23 . 108.86(16) yes
O3 . C22 . H221 . 109.630 no
C23 . C22 . H221 . 109.626 no
O3 . C22 . H222 . 109.624 no
C23 . C22 . H222 . 109.619 no
H221 . C22 . H222 . 109.464 no
O4 . C23 . C22 . 110.42(16) yes
O4 . C23 . H231 . 109.231 no
C22 . C23 . H231 . 109.230 no
O4 . C23 . H232 . 109.239 no
C22 . C23 . H232 . 109.240 no
H231 . C23 . H232 . 109.468 no
O4 . C24 . C25 . 107.30(16) yes
O4 . C24 . H241 . 110.017 no
C25 . C24 . H241 . 110.006 no
O4 . C24 . H242 . 110.013 no
C25 . C24 . H242 . 110.010 no
H241 . C24 . H242 . 109.465 no
O5 . C25 . C24 . 106.98(16) yes
O5 . C25 . H251 . 110.084 no
C24 . C25 . H251 . 110.090 no
O5 . C25 . H252 . 110.094 no
C24 . C25 . H252 . 110.097 no
H251 . C25 . H252 . 109.472 no
O5 . C26 . C27 . 108.02(15) yes
O5 . C26 . H261 . 109.838 no
C27 . C26 . H261 . 109.832 no
O5 . C26 . H262 . 109.836 no
C27 . C26 . H262 . 109.835 no
H261 . C26 . H262 . 109.459 no
O6 . C27 . C26 . 107.75(15) yes
O6 . C27 . H271 . 109.892 no
C26 . C27 . H271 . 109.902 no
O6 . C27 . H272 . 109.902 no
C26 . C27 . H272 . 109.905 no
H271 . C27 . H272 . 109.468 no
O6 . C28 . C29 . 111.29(15) yes
O6 . C28 . H281 . 109.013 no
C29 . C28 . H281 . 109.014 no
O6 . C28 . H282 . 109.017 no
C29 . C28 . H282 . 109.017 no
H281 . C28 . H282 . 109.465 no
O7 . C29 . C28 . 108.00(16) yes
O7 . C29 . H291 . 109.836 no
C28 . C29 . H291 . 109.832 no
O7 . C29 . H292 . 109.845 no
C28 . C29 . H292 . 109.838 no
H291 . C29 . H292 . 109.466 no
B1 . C30 . C31 . 119.16(17) yes
B1 . C30 . C35 . 127.73(17) yes
C31 . C30 . C35 . 112.97(18) yes
F1 . C31 . C30 . 118.92(18) yes
F1 . C31 . C32 . 116.27(19) yes
C30 . C31 . C32 . 124.8(2) yes
F2 . C32 . C31 . 120.2(2) yes
F2 . C32 . C33 . 120.4(2) yes
C31 . C32 . C33 . 119.4(2) yes
F3 . C33 . C32 . 119.7(2) yes
F3 . C33 . C34 . 121.3(3) yes
C32 . C33 . C34 . 119.0(2) yes
F4 . C34 . C33 . 120.3(2) yes

F4 . C34 . C35 . 119.9(2) yes
 C33 . C34 . C35 . 119.8(2) yes
 F5 . C35 . C30 . 121.35(17) yes
 F5 . C35 . C34 . 114.65(19) yes
 C30 . C35 . C34 . 124.0(2) yes
 B1 . C36 . C37 . 128.39(17) yes
 B1 . C36 . C41 . 118.93(15) yes
 C37 . C36 . C41 . 112.45(17) yes
 F6 . C37 . C36 . 120.44(18) yes
 F6 . C37 . C38 . 115.47(17) yes
 C36 . C37 . C38 . 124.08(19) yes
 F7 . C38 . C37 . 119.8(2) yes
 F7 . C38 . C39 . 119.80(19) yes
 C37 . C38 . C39 . 120.42(18) yes
 F8 . C39 . C38 . 121.09(19) yes
 F8 . C39 . C40 . 120.6(2) yes
 C38 . C39 . C40 . 118.31(19) yes
 F9 . C40 . C39 . 120.05(19) yes
 F9 . C40 . C41 . 120.64(18) yes
 C39 . C40 . C41 . 119.3(2) yes
 F10 . C41 . C36 . 119.11(16) yes
 F10 . C41 . C40 . 115.50(17) yes
 C36 . C41 . C40 . 125.36(18) yes
 B1 . C42 . C43 . 127.16(16) yes
 B1 . C42 . C47 . 119.93(15) yes
 C43 . C42 . C47 . 112.89(16) yes
 F15 . C43 . C42 . 121.74(16) yes
 F15 . C43 . C44 . 114.27(16) yes
 C42 . C43 . C44 . 123.99(17) yes
 F14 . C44 . C43 . 120.33(18) yes
 F14 . C44 . C45 . 119.78(17) yes
 C43 . C44 . C45 . 119.89(17) yes
 F13 . C45 . C44 . 120.16(18) yes
 F13 . C45 . C46 . 120.72(18) yes
 C44 . C45 . C46 . 119.12(17) yes
 F12 . C46 . C45 . 120.08(18) yes
 F12 . C46 . C47 . 121.24(18) yes
 C45 . C46 . C47 . 118.66(17) yes
 F11 . C47 . C42 . 119.08(16) yes
 F11 . C47 . C46 . 115.74(16) yes
 C42 . C47 . C46 . 125.15(17) yes
 B1 . C48 . C49 . 127.36(16) yes
 B1 . C48 . C53 . 118.21(16) yes
 C49 . C48 . C53 . 114.04(16) yes
 F16 . C49 . C48 . 121.86(16) yes
 F16 . C49 . C50 . 114.30(17) yes
 C48 . C49 . C50 . 123.83(18) yes
 F17 . C50 . C49 . 120.3(2) yes
 F17 . C50 . C51 . 120.20(18) yes
 C49 . C50 . C51 . 119.46(19) yes
 F18 . C51 . C50 . 120.2(2) yes
 F18 . C51 . C52 . 120.5(2) yes
 C50 . C51 . C52 . 119.28(18) yes
 F19 . C52 . C51 . 119.82(19) yes
 F19 . C52 . C53 . 120.8(2) yes
 C51 . C52 . C53 . 119.33(19) yes
 F20 . C53 . C48 . 119.47(16) yes
 F20 . C53 . C52 . 116.51(17) yes
 C48 . C53 . C52 . 124.03(18) yes
 C11 . C54 . C12 . 113.01(14) yes
 C11 . C54 . H541 . 108.582 no

```
C12 . C54 . H541 . 108.585 no
C11 . C54 . H542 . 108.576 no
C12 . C54 . H542 . 108.581 no
H541 . C54 . H542 . 109.468 no
#===END
```

```
#####
## denoted [Li21]I2 in text ##
#####
```

```
data_3
```

```
_database_code_depnum_ccdc_archive 'CCDC 254112'
```

```
_chemical_name_systematic          # IUPAC name, in full
```

```
;
```

```
?
```

```
;
```

```
_chemical_melting_point            ?
```

```
# choose from 'full, fullcycle, atomblock, userblock, diagonal, sparse'
_refine_ls_matrix_type              full
```

```
# choose from 'heavy, direct, difmap, geom'
_atom_sites_solution_primary        direct
```

```
# _atom_sites_solution_secondary difmap
_atom_sites_solution_hydrogens      geom
```

```
# choose from 'none, undef, noref, refall, refxyz, refU, constr, mixed'
_refine_ls_hydrogen_treatment       mixed
```

```
*****
# General computing
```

```
#=====
```

```
_computing_structure_refinement
```

```
;
```

```
CRYSTALS (Watkin et al 2001)
```

```
;
```

```
_computing_publication_material
```

```
;
```

```
CRYSTALS (Watkin et al 2001)
```

```
;
```

```
_computing_molecular_graphics
```

```
;
```

```
CAMERON (Watkin et al 1996)
```

```
;
```

```
#=====
```

```
_cell_length_a                      7.566(1)
_cell_angle_alpha                   72.355(2)
_cell_length_b                     10.3074(14)
_cell_angle_beta                    88.102(2)
_cell_length_c                     15.598(2)
_cell_angle_gamma                   81.429(2)
_cell_volume                        1146.2(3)
```

```
_symmetry_cell_setting              Triclinic
```

```
_symmetry_space_group_name_H-M     'P -1 '
```

```
loop_
```

```
_symmetry_equiv_pos_as_xyz
```

```

x,y,z
-x,-y,-z

# choose from: rm (reference molecule of
# known chirality), ad (anomalous
# dispersion - ie. Flack param), rmad
# (both rm and ad), syn (known from
# synthetic pathway), unk (unknown)
# or . (not applicable).

_chemical_absolute_configuration .

loop_
_atom_type_symbol
_atom_type_scatter_dispersion_real
_atom_type_scatter_dispersion_imag
_atom_type_scatter_Cromer_Mann_a1
_atom_type_scatter_Cromer_Mann_b1
_atom_type_scatter_Cromer_Mann_a2
_atom_type_scatter_Cromer_Mann_b2
_atom_type_scatter_Cromer_Mann_a3
_atom_type_scatter_Cromer_Mann_b3
_atom_type_scatter_Cromer_Mann_a4
_atom_type_scatter_Cromer_Mann_b4
_atom_type_scatter_Cromer_Mann_c
_atom_type_scatter_source
'C   ' 0.0033 0.0016 2.3100 20.8439 1.0200 10.2075 1.5886 0.5687 0.8650 51.6512
0.2156 International_Tables_Vol_IV_Table_2.2B
'H   ' 0.0000 0.0000 0.4930 10.5109 0.3229 26.1257 0.1402 3.1424 0.0408 57.7998
0.0030 International_Tables_Vol_IV_Table_2.2B
'Li  ' -0.0003 0.0001 1.1282 3.9546 0.7508 1.0524 0.6175 85.3905 0.4653
168.2610 0.0377 International_Tables_Vol_IV_Table_2.2B
'O   ' 0.0106 0.0060 3.0485 13.2771 2.2868 5.7011 1.5463 0.3239 0.8670 32.9089
0.2508 International_Tables_Vol_IV_Table_2.2B
'I   ' -0.4742 1.8119 20.1472 4.3470 18.9949 0.3814 7.5138 27.7660 2.2735
66.8776 4.0712 International_Tables_Vol_IV_Table_2.2B

_cell_formula_units_Z          1
_chemical_formula_sum           ' C38 H56 I2 Li2 O14 '
_chemical_formula_moiety       ' C38 H56 I2 Li2 O14 '
_chemical_compound_source
;
?
;
_chemical_formula_weight        1004.55

_cell_measurement_reflns_used    8794
_cell_measurement_theta_min      2.64
_cell_measurement_theta_max      29.15
_cell_measurement_temperature    150(2)

_exptl_crystal_description      ' colourless '
_exptl_crystal_colour           ' needle '
_exptl_crystal_size_min         0.02
_exptl_crystal_size_mid         0.02
_exptl_crystal_size_max         0.28

_exptl_crystal_density_diffn    1.455
_exptl_crystal_density_meas     ?
# Non-dispersive F(000):
_exptl_crystal_F_000           508.000

```

```

_exptl_absorpt_coefficient_mu    1.430
# Sheldrick geometric definitions 1.00 1.00

_diffrn_measurement_device_type
;
Bruker AXS SMART 1K CCD diffractometer
;
_diffrn_radiation_monochromator  'silicon 111'
_computing_data_collection
;
SMART (Siemens, 1993)
;
_computing_data_reduction
;
Bruker SAINT version 6.36a (Bruker, 2002)
;
_computing_cell_refinement
;
Bruker SAINT version 6.36a (Bruker, 2002)
;
_computing_structure_solution
;
SHELXS 86 (Sheldrick, 1986)
;
_diffrn_measurement_method      'thin slice \w-scans'
_exptl_absorpt_correction_type  multi-scan
_exptl_absorpt_correction_T_min 1.00
_exptl_absorpt_correction_T_max 1.00
_exptl_absorpt_process_details
;

```

Correction applied by SADABS - note that the transmission factors are not real since they include corrections for beam decay and possibly crystal decay (the two cannot be distinguished).
The numbers listed in the CIF are those calculated by CRYSTALS.

```

;
_diffrn_standards_interval_time 0
_diffrn_standards_interval_count 0
_diffrn_standards_number        0
_diffrn_standards_decay_%       0.00

_diffrn_ambient_temperature     150(2)
_diffrn_reflns_number           11642
_reflns_number_total            6117
_diffrn_reflns_av_R_equivalents 0.03
# Number of reflections with Friedels Law is 6117
# Number of reflections without Friedels Law is 0
# Theoretical number of reflections is about 6784

_diffrn_reflns_theta_min        2.033
_diffrn_reflns_theta_max        29.252
_diffrn_measured_fraction_theta_max 0.895

_diffrn_reflns_theta_full        23.402
_diffrn_measured_fraction_theta_full 0.995

_diffrn_reflns_limit_h_min      -10
_diffrn_reflns_limit_h_max      10
_diffrn_reflns_limit_k_min      -13
_diffrn_reflns_limit_k_max      14
_diffrn_reflns_limit_l_min      -21

```

```

_diffn_reflms_limit_l_max      21
_reflms_limit_h_min           -10
_reflms_limit_h_max            10
_reflms_limit_k_min           -13
_reflms_limit_k_max            14
_reflms_limit_l_min            0
_reflms_limit_l_max            21

_refine_diff_density_min       -0.54
_refine_diff_density_max       1.40

_refine_ls_number_reflms       5650
_refine_ls_number_restraints    0
_refine_ls_number_parameters    253

#_refine_ls_R_factor_ref 0.0342
_refine_ls_wR_factor_ref       0.0367
_refine_ls_goodness_of_fit_ref 0.9607

#_reflms_number_all 6098
_refine_ls_R_factor_all        0.0360
_refine_ls_wR_factor_all       0.0435

# The I/u(I) cutoff below was used for refinement as
# well as the _gt R-factors:
_reflms_threshold_expression    I>3.00u(I)
_reflms_number_gt               5650
_refine_ls_R_factor_gt          0.0342
_refine_ls_wR_factor_gt         0.0367

_refine_ls_shift/su_max         0.007556
_refine_ls_structure_factor_coef F
_refine_ls_weighting_scheme     calc
_refine_ls_weighting_details
;
Method, part 1, Chebychev polynomial, (Watkin, 1994, Prince, 1982)
[weight] = 1.0/[A~0~*T~0~(x)+A~1~*T~1~(x) ... +A~n-1~*T~n-1~(x)]
where A~i~ are the Chebychev coefficients listed below and x= Fcalc/Fmax
Method = Robust Weighting (Prince, 1982)
W = [weight] * [1-(deltaF/6*sigmaF)^2]^2
A~i~ are:
0.228 0.242 0.862E-01
;
_diffn_radiation_type          synchrotron
_diffn_radiation_wavelength     0.6923
_diffn_radiation_source         'Daresbury SRS, Station 9.8'
## -----REFERENCES -----##
## Insert your own references - in alphabetic order
_publ_section_references
;

Prince, E.
Mathematical Techniques in Crystallography
and Materials Science
Springer-Verlag, New York, 1982.

Sheldrick, G.M. (1986). SHELXS86. Program for the solution of
crystal structures. Univ. of Gottingen, Federal Republic of Germany.

Watkin D.J. (1994),
Acta Cryst, A50, 411-437

```


Watkin, D.J., Prout, C.K., Carruthers, J.R., Betteridge, P.W. & Cooper
R.I. (2001) CRYSTALS
Issue 11. Chemical Crystallography Laboratory, OXFORD, UK.

Watkin, D.J., Prout, C.K. & Pearce, L.J. (1996) CAMERON, Chemical
Crystallography Laboratory, OXFORD, UK.

;

Uequiv = arithmetic mean of Ui

i.e. Uequiv = (U1+U2+U3)/3

Replace trailing . with the number of unfound

hydrogen atoms attaced to relavent atom

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_occupancy

_atom_site_adp_type

_atom_site_refinement_flags

_atom_site_attached_hydrogens

Li1 Li 0.1481(5) 0.0117(4) 0.1982(3) 0.0313 1.0000 Uani . .
I1 I -0.420306(19) -0.292697(14) 0.068244(9) 0.0302 1.0000 Uani . .
O1 O 0.25839(19) 0.35018(16) 0.25472(9) 0.0256 1.0000 Uani . .
O2 O 0.0313(2) 0.26884(15) 0.1455(1) 0.0266 1.0000 Uani . .
O3 O -0.09666(18) 0.03126(14) 0.1505(1) 0.0260 1.0000 Uani . .
O4 O 0.10593(18) -0.19847(14) 0.25279(9) 0.0236 1.0000 Uani . .
O5 O 0.37054(19) -0.31515(16) 0.40412(9) 0.0271 1.0000 Uani . .
O6 O 0.1856(3) 0.06015(18) 0.30541(11) 0.0428 1.0000 Uani . .
O7 O 0.3354(2) 0.00659(18) 0.11370(12) 0.0368 1.0000 Uani . .
C1 C 0.4057(3) 0.34790(19) 0.30459(13) 0.0230 1.0000 Uani . .
C2 C 0.5732(3) 0.3620(2) 0.27006(13) 0.0262 1.0000 Uani . .
C3 C 0.7157(3) 0.3585(2) 0.32670(14) 0.0296 1.0000 Uani . .
C4 C 0.6905(3) 0.3417(2) 0.41693(14) 0.0273 1.0000 Uani . .
C5 C 0.4835(3) 0.3169(2) 0.54613(13) 0.0238 1.0000 Uani . .
C6 C 0.3140(3) 0.3091(2) 0.57975(13) 0.0288 1.0000 Uani . .
C7 C 0.1713(3) 0.3134(3) 0.52263(15) 0.0320 1.0000 Uani . .
C8 C 0.1979(3) 0.3234(2) 0.43392(14) 0.0275 1.0000 Uani . .
C9 C 0.3723(2) 0.33144(19) 0.39768(12) 0.0219 1.0000 Uani . .
C10 C 0.5173(2) 0.32899(19) 0.45372(12) 0.0223 1.0000 Uani . .
C11 C 0.2857(3) 0.3737(2) 0.15979(13) 0.0261 1.0000 Uani . .
C12 C 0.1079(3) 0.3925(2) 0.11480(13) 0.0263 1.0000 Uani . .
C13 C -0.1194(3) 0.2740(2) 0.09155(14) 0.0276 1.0000 Uani . .
C14 C -0.2172(3) 0.1558(2) 0.13943(17) 0.0315 1.0000 Uani . .
C15 C -0.1802(3) -0.0857(2) 0.19608(17) 0.0314 1.0000 Uani . .
C16 C -0.0441(3) -0.2101(2) 0.20371(15) 0.0280 1.0000 Uani . .
C17 C 0.2335(3) -0.3208(2) 0.26964(13) 0.0250 1.0000 Uani . .
C18 C 0.4016(3) -0.3044(2) 0.31139(13) 0.0261 1.0000 Uani . .
C19 C 0.1426(9) 0.0018(4) 0.3948(2) 0.0880 1.0000 Uani . .
H1 H 0.2355 -0.0001 0.1181 0.0461 1.0000 Uiso . .
H2 H 0.3555 0.0583 0.0700 0.0500 1.0000 Uiso . .
H3 H 0.2080 0.1337 0.3022 0.0500 1.0000 Uiso . .
H21 H 0.5940 0.3747 0.2046 0.0313 1.0000 Uiso . .
H31 H 0.8375 0.3685 0.3009 0.0345 1.0000 Uiso . .
H41 H 0.7935 0.3385 0.4564 0.0320 1.0000 Uiso . .
H61 H 0.2919 0.3003 0.6447 0.0337 1.0000 Uiso . .

```

H71 H 0.0481 0.3090 0.5475 0.0383 1.0000 Uiso . .
H81 H 0.0952 0.3251 0.3947 0.0328 1.0000 Uiso . .
H111 H 0.3435 0.4583 0.1345 0.0309 1.0000 Uiso . .
H112 H 0.3643 0.2929 0.1496 0.0309 1.0000 Uiso . .
H121 H 0.0265 0.4685 0.1295 0.0306 1.0000 Uiso . .
H122 H 0.1240 0.4167 0.0482 0.0306 1.0000 Uiso . .
H131 H -0.2003 0.3632 0.0830 0.0313 1.0000 Uiso . .
H132 H -0.0787 0.2656 0.0316 0.0313 1.0000 Uiso . .
H141 H -0.2573 0.1638 0.1996 0.0363 1.0000 Uiso . .
H142 H -0.3235 0.1565 0.1028 0.0363 1.0000 Uiso . .
H151 H -0.2184 -0.0816 0.2573 0.0374 1.0000 Uiso . .
H152 H -0.2867 -0.0888 0.1609 0.0374 1.0000 Uiso . .
H161 H -0.0956 -0.2953 0.2364 0.0342 1.0000 Uiso . .
H162 H -0.0063 -0.2141 0.1424 0.0342 1.0000 Uiso . .
H171 H 0.1806 -0.3993 0.3115 0.0293 1.0000 Uiso . .
H172 H 0.2633 -0.3406 0.2115 0.0293 1.0000 Uiso . .
H181 H 0.5000 -0.3781 0.3066 0.0305 1.0000 Uiso . .
H182 H 0.4363 -0.2121 0.2793 0.0305 1.0000 Uiso . .
H191 H 0.1126 -0.0925 0.4032 0.1037 1.0000 Uiso . .
H192 H 0.2469 -0.0044 0.4343 0.1037 1.0000 Uiso . .
H193 H 0.0373 0.0603 0.4111 0.1037 1.0000 Uiso . .
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
Li1 0.0247(16) 0.042(2) 0.0276(17) -0.0103(15) -0.0020(13) -0.0060(14)
I1 0.03560(8) 0.02762(8) 0.02669(8) -0.00653(5) 0.00329(5) -0.00694(5)
O1 0.0264(7) 0.0322(7) 0.0184(6) -0.0059(5) -0.0028(5) -0.0080(5)
O2 0.0269(7) 0.0237(6) 0.0248(6) -0.0002(5) -0.0086(5) -0.0035(5)
O3 0.0211(6) 0.0216(6) 0.0339(7) -0.0067(5) -0.0042(5) -0.0010(5)
O4 0.0227(6) 0.0223(6) 0.0256(6) -0.0073(5) -0.0048(5) -0.0011(5)
O5 0.0233(6) 0.0373(8) 0.0185(6) -0.0055(5) -0.0039(5) -0.0028(5)
O6 0.0737(13) 0.0284(8) 0.0268(8) -0.0050(6) -0.0060(8) -0.0147(8)
O7 0.0342(8) 0.0323(8) 0.0367(8) -0.0019(7) 0.0105(7) -0.0032(6)
C1 0.0253(9) 0.0217(8) 0.0212(8) -0.0050(6) -0.0036(6) -0.0030(6)
C2 0.0270(9) 0.0290(9) 0.0224(8) -0.0083(7) 0.0013(7) -0.0022(7)
C3 0.0217(9) 0.0393(11) 0.0253(9) -0.0076(8) 0.0017(7) -0.0018(7)
C4 0.0188(8) 0.037(1) 0.0245(9) -0.0073(8) -0.0027(6) -0.0016(7)
C5 0.0216(8) 0.0271(9) 0.0204(8) -0.0036(7) -0.0043(6) -0.0026(7)
C6 0.0253(9) 0.0376(11) 0.0213(8) -0.0048(8) 0.0015(7) -0.0063(8)
C7 0.0243(9) 0.0443(12) 0.027(1) -0.0077(8) 0.0023(7) -0.0105(8)
C8 0.0222(9) 0.034(1) 0.0254(9) -0.0065(8) -0.0020(7) -0.0078(7)
C9 0.0218(8) 0.0232(8) 0.0198(8) -0.0044(6) -0.0010(6) -0.0048(6)
C10 0.0194(8) 0.0240(8) 0.0211(8) -0.0044(6) -0.0018(6) -0.0009(6)
C11 0.0297(9) 0.0289(9) 0.0186(8) -0.0039(7) -0.0027(7) -0.0069(7)
C12 0.0295(9) 0.0252(9) 0.0219(8) -0.0032(7) -0.0052(7) -0.0043(7)
C13 0.0232(9) 0.0257(9) 0.0295(9) -0.0026(7) -0.0113(7) 0.0009(7)
C14 0.0237(9) 0.0228(9) 0.0442(12) -0.0065(8) -0.0055(8) 0.0016(7)
C15 0.0204(9) 0.0247(9) 0.0483(12) -0.0093(8) -0.0039(8) -0.0038(7)
C16 0.0257(9) 0.0256(9) 0.034(1) -0.0102(8) -0.0057(7) -0.0043(7)
C17 0.0250(9) 0.0236(8) 0.0246(9) -0.0066(7) -0.0054(7) 0.0018(7)
C18 0.0274(9) 0.0306(9) 0.0182(8) -0.0052(7) -0.0034(6) -0.0016(7)
C19 0.186(5) 0.0365(16) 0.0368(16) -0.0072(13) 0.014(2) -0.012(2)
_refine_ls_extinction_method None
loop_
_geom_bond_atom_site_label_1
_geom_bond_site_symmetry_1

```

_geom_bond_atom_site_label_2
_geom_bond_site_symmetry_2
_geom_bond_distance
_geom_bond_publ_flag
O1 . C11 . 1.439(2) yes
O1 . C1 . 1.374(2) yes
O2 . C13 . 1.424(2) yes
O2 . C12 . 1.422(2) yes
O3 . C15 . 1.427(2) yes
O3 . C14 . 1.427(2) yes
O4 . C17 . 1.430(2) yes
O4 . C16 . 1.429(2) yes
O5 . C18 . 1.431(2) yes
O5 . C5 2_656 1.366(2) yes
O6 . H3 . 0.788 no
O6 . C19 . 1.389(4) yes
O7 . H2 . 0.755 no
O7 . H1 . 0.768 no
C1 . C9 . 1.428(3) yes
C1 . C2 . 1.371(3) yes
C2 . H21 . 1.000 no
C2 . C3 . 1.406(3) yes
C3 . H31 . 1.000 no
C3 . C4 . 1.375(3) yes
C4 . H41 . 1.000 no
C4 . C10 . 1.421(3) yes
C5 . C10 . 1.426(3) yes
C5 . C6 . 1.374(3) yes
C6 . H61 . 1.000 no
C6 . C7 . 1.410(3) yes
C7 . H71 . 1.000 no
C7 . C8 . 1.366(3) yes
C8 . H81 . 1.000 no
C8 . C9 . 1.422(3) yes
C9 . C10 . 1.417(3) yes
C11 . H112 . 1.000 no
C11 . H111 . 1.000 no
C11 . C12 . 1.498(3) yes
C12 . H122 . 1.000 no
C12 . H121 . 1.000 no
C13 . H132 . 1.000 no
C13 . H131 . 1.000 no
C13 . C14 . 1.507(3) yes
C14 . H142 . 1.000 no
C14 . H141 . 1.000 no
C15 . H152 . 1.000 no
C15 . H151 . 1.000 no
C15 . C16 . 1.497(3) yes
C16 . H162 . 1.000 no
C16 . H161 . 1.000 no
C17 . H172 . 1.000 no
C17 . H171 . 1.000 no
C17 . C18 . 1.502(3) yes
C18 . H182 . 1.000 no
C18 . H181 . 1.000 no
C19 . H193 . 1.000 no
C19 . H192 . 1.000 no
C19 . H191 . 1.000 no
loop_
_geom_angle_atom_site_label_1
_geom_angle_site_symmetry_1

```

_geom_angle_atom_site_label_2
_geom_angle_site_symmetry_2
_geom_angle_atom_site_label_3
_geom_angle_site_symmetry_3
_geom_angle
_geom_angle_publ_flag
C11 . O1 . C1 . 116.08(15) yes
C13 . O2 . C12 . 111.19(15) yes
C15 . O3 . C14 . 111.00(16) yes
C17 . O4 . C16 . 110.06(15) yes
C18 . O5 . C5 2_656 116.13(15) yes
H3 . O6 . C19 . 105.637 no
H2 . O7 . H1 . 112.033 no
C9 . C1 . C2 . 120.97(17) yes
C9 . C1 . O1 . 114.74(16) yes
C2 . C1 . O1 . 124.28(17) yes
H21 . C2 . C3 . 119.990 no
H21 . C2 . C1 . 119.990 no
C3 . C2 . C1 . 120.02(18) yes
H31 . C3 . C4 . 119.429 no
H31 . C3 . C2 . 119.429 no
C4 . C3 . C2 . 121.14(18) yes
H41 . C4 . C10 . 120.169 no
H41 . C4 . C3 . 120.169 no
C10 . C4 . C3 . 119.66(18) yes
C10 . C5 . C6 . 120.68(17) yes
C10 . C5 . O5 2_656 115.16(16) yes
C6 . C5 . O5 2_656 124.15(18) yes
H61 . C6 . C7 . 120.038 no
H61 . C6 . C5 . 120.037 no
C7 . C6 . C5 . 119.92(19) yes
H71 . C7 . C8 . 119.323 no
H71 . C7 . C6 . 119.323 no
C8 . C7 . C6 . 121.35(19) yes
H81 . C8 . C9 . 120.160 no
H81 . C8 . C7 . 120.160 no
C9 . C8 . C7 . 119.68(18) yes
C10 . C9 . C8 . 119.85(17) yes
C10 . C9 . C1 . 118.28(17) yes
C8 . C9 . C1 . 121.82(17) yes
C9 . C10 . C4 . 119.92(17) yes
C9 . C10 . C5 . 118.49(17) yes
C4 . C10 . C5 . 121.57(17) yes
H112 . C11 . H111 . 109.467 no
H112 . C11 . C12 . 109.688 no
H111 . C11 . C12 . 109.688 no
H112 . C11 . O1 . 109.688 no
H111 . C11 . O1 . 109.688 no
C12 . C11 . O1 . 108.61(16) yes
H122 . C12 . H121 . 109.467 no
H122 . C12 . O2 . 109.353 no
H121 . C12 . O2 . 109.354 no
H122 . C12 . C11 . 109.354 no
H121 . C12 . C11 . 109.354 no
O2 . C12 . C11 . 109.95(16) yes
H132 . C13 . H131 . 109.466 no
H132 . C13 . C14 . 109.818 no
H131 . C13 . C14 . 109.818 no
H132 . C13 . O2 . 109.818 no
H131 . C13 . O2 . 109.818 no
C14 . C13 . O2 . 108.08(16) yes

```

```

H142 . C14 . H141 . 109.467 no
H142 . C14 . O3 . 109.882 no
H141 . C14 . O3 . 109.881 no
H142 . C14 . C13 . 109.882 no
H141 . C14 . C13 . 109.881 no
O3 . C14 . C13 . 107.83(17) yes
H152 . C15 . H151 . 109.466 no
H152 . C15 . C16 . 110.137 no
H151 . C15 . C16 . 110.137 no
H152 . C15 . O3 . 110.136 no
H151 . C15 . O3 . 110.136 no
C16 . C15 . O3 . 106.80(17) yes
H162 . C16 . H161 . 109.467 no
H162 . C16 . O4 . 109.947 no
H161 . C16 . O4 . 109.947 no
H162 . C16 . C15 . 109.948 no
H161 . C16 . C15 . 109.948 no
O4 . C16 . C15 . 107.56(16) yes
H172 . C17 . H171 . 109.466 no
H172 . C17 . C18 . 109.113 no
H171 . C17 . C18 . 109.113 no
H172 . C17 . O4 . 109.113 no
H171 . C17 . O4 . 109.113 no
C18 . C17 . O4 . 110.90(16) yes
H182 . C18 . H181 . 109.466 no
H182 . C18 . O5 . 109.550 no
H181 . C18 . O5 . 109.550 no
H182 . C18 . C17 . 109.550 no
H181 . C18 . C17 . 109.550 no
O5 . C18 . C17 . 109.16(16) yes
H193 . C19 . H192 . 109.476 no
H193 . C19 . H191 . 109.479 no
H192 . C19 . H191 . 109.474 no
H193 . C19 . O6 . 109.468 no
H192 . C19 . O6 . 109.463 no
H191 . C19 . O6 . 109.467 no
#===END

```

```

#####
## denoted [Na2I2]I2 in text ##
#####

```

```

data_4
_database_code_depnum_ccdc_archive 'CCDC 254113'

_chemical_name_systematic      # IUPAC name, in full
;
?
;

_chemical_melting_point        ?

# choose from 'full, fullcycle, atomblock, userblock, diagonal, sparse'
_refine_ls_matrix_type         full

# choose from 'heavy, direct, difmap, geom'
_atom_sites_solution_primary    direct

# _atom_sites_solution_secondary difmap
_atom_sites_solution_hydrogens  geom

```

```

# choose from 'none, undef, noref, refall, refxyz, refU, constr, mixed'
_refine_ls_hydrogen_treatment    mixed

#*****
# General computing
#=====
_computing_structure_refinement
;
CRYSTALS (Watkin et al 2003)
;
_computing_publication_material
;
CRYSTALS (Watkin et al 2003)
;
_computing_molecular_graphics
;
CAMERON (Watkin et al 1996)
;
#=====

_cell_length_a                10.8071(2)
_cell_angle_alpha             78.4706(8)
_cell_length_b                11.2503(2)
_cell_angle_beta              81.8524(7)
_cell_length_c                18.3062(3)
_cell_angle_gamma             86.3758(12)
_cell_volume                  2157.41(7)

_symmetry_cell_setting        Triclinic
_symmetry_space_group_name_H-M  'P -1 '
loop_
_symmetry_equiv_pos_as_xyz
x,y,z
-x,-y,-z

# choose from: rm (reference molecule of
# known chirality), ad (anomolous
# dispersion - ie. Flack param), rmad
# (both rm and ad), syn (known from
# synthetic pathway), unk (unknown)
# or . (not applicable).

_chemical_absolute_configuration .

loop_
_atom_type_symbol
_atom_type_scatter_dispersion_real
_atom_type_scatter_dispersion_imag
_atom_type_scatter_Cromer_Mann_a1
_atom_type_scatter_Cromer_Mann_b1
_atom_type_scatter_Cromer_Mann_a2
_atom_type_scatter_Cromer_Mann_b2
_atom_type_scatter_Cromer_Mann_a3
_atom_type_scatter_Cromer_Mann_b3
_atom_type_scatter_Cromer_Mann_a4
_atom_type_scatter_Cromer_Mann_b4
_atom_type_scatter_Cromer_Mann_c
_atom_type_scatter_source
'C   ' 0.0033 0.0016 2.3100 20.8439 1.0200 10.2075 1.5886 0.5687 0.8650 51.6512
0.2156 International_Tables_Vol_IV_Table_2.2B
'H   ' 0.0000 0.0000 0.4930 10.5109 0.3229 26.1257 0.1402 3.1424 0.0408 57.7998

```

0.0030 International_Tables_Vol_IV_Table_2.2B
'O ' 0.0106 0.0060 3.0485 13.2771 2.2868 5.7011 1.5463 0.3239 0.8670 32.9089
0.2508 International_Tables_Vol_IV_Table_2.2B
'N ' 0.0061 0.0033 12.2126 0.0057 3.1322 9.8933 2.0125 28.9975 1.1663 0.5826
-11.5290 International_Tables_Vol_IV_Table_2.2B
'Na ' 0.0362 0.0249 4.7626 3.2850 3.1736 8.8422 1.2674 0.3136 1.1128 129.4240
0.6760 International_Tables_Vol_IV_Table_2.2B
'I ' -0.4742 1.8119 20.1472 4.3470 18.9949 0.3814 7.5138 27.7660 2.2735
66.8776 4.0712 International_Tables_Vol_IV_Table_2.2B
'Cl ' 0.1484 0.1585 11.4604 0.0104 7.1964 1.1662 6.2556 18.5194 1.6455 47.7784
-9.5574 International_Tables_Vol_IV_Table_2.2B

_cell_formula_units_Z 1
_chemical_formula_sum ' C64 H78 Cl18 I2 N2 Na2 O14 '
_chemical_formula_moiety 'C58 H72 I2 N2 Na2 O14, 6(C H Cl3) '
_chemical_compound_source

;
synthesis as described

;
_chemical_formula_weight 2037.16

_cell_measurement_reflns_used 24277
_cell_measurement_theta_min 5
_cell_measurement_theta_max 27
_cell_measurement_temperature 180

_exptl_crystal_description ' block '
_exptl_crystal_colour ' orange '
_exptl_crystal_size_min 0.12
_exptl_crystal_size_mid 0.23
_exptl_crystal_size_max 0.32

_exptl_crystal_density_diffn 1.568
_exptl_crystal_density_meas ?
Non-dispersive F(000):
_exptl_crystal_F_000 1022
_exptl_absorpt_coefficient_mu 1.352

_diffn_measurement_device_type

;
Nonius Kappa CCD

;
_diffn_radiation_monochromator graphite
_computing_data_collection

;
COLLECT (Nonius BV, 1997)

;
_computing_data_reduction

;
Denzo/Scalepack (Otwinowski & Minor, 1996)

;
_computing_cell_refinement

;
Denzo/Scalepack (Otwinowski & Minor, 1996)

;
_computing_structure_solution

;
SIR92 (Altomare et al, 1994)

;
_diffn_measurement_method \w

```

_exptl_absorpt_correction_type    multi-scan
_exptl_absorpt_process_details
;
Denzo/Scalepack (Otwinowski & Minor, 1996)
;
_exptl_absorpt_correction_T_min    0.76
_exptl_absorpt_correction_T_max    0.87
_diffn_standards_interval_time      0
_diffn_standards_interval_count      0
_diffn_standards_number              0
_diffn_standards_decay_%            0.00

_diffn_ambient_temperature           180
_diffn_reflns_number                 21614
_reflns_number_total                 9407
_diffn_reflns_av_R_equivalents       0.03
# Number of reflections with Friedels Law is 9407
# Number of reflections without Friedels Law is 0
# Theoretical number of reflections is about 9528

_diffn_reflns_theta_min              5.150
_diffn_reflns_theta_max              27.111
_diffn_measured_fraction_theta_max   0.986

_diffn_reflns_theta_full             26.026
_diffn_measured_fraction_theta_full  0.989

_diffn_reflns_limit_h_min            -13
_diffn_reflns_limit_h_max            13
_diffn_reflns_limit_k_min            -12
_diffn_reflns_limit_k_max            14
_diffn_reflns_limit_l_min            -23
_diffn_reflns_limit_l_max            20
_reflns_limit_h_min                 -13
_reflns_limit_h_max                 13
_reflns_limit_k_min                 -13
_reflns_limit_k_max                 14
_reflns_limit_l_min                 0
_reflns_limit_l_max                 23

_refine_diff_density_min             -0.86
_refine_diff_density_max             1.18

_refine_ls_number_reflns             8041
_refine_ls_number_restraints         0
_refine_ls_number_parameters         460

#_refine_ls_R_factor_ref 0.0344
_refine_ls_wR_factor_ref             0.0444
_refine_ls_goodness_of_fit_ref       1.0757

#_reflns_number_all 9407
_refine_ls_R_factor_all              0.0421
_refine_ls_wR_factor_all             0.0461

# The I/u(I) cutoff below was used for refinement as
# well as the _gt R-factors:
_reflns_threshold_expression          I>3.00u(I)
_reflns_number_gt                    8041
_refine_ls_R_factor_gt               0.0344

```



```

_refine_ls_wR_factor_gt          0.0444

_refine_ls_shift/su_max          0.003699
_refine_ls_structure_factor_coef F
_refine_ls_weighting_scheme      calc
_refine_ls_weighting_details
;
Method, part 1, Chebychev polynomial, (Watkin, 1994, Prince, 1982)
[weight] = 1.0/[A~0~*T~0~(x)+A~1~*T~1~(x) ... +A~n-1~*T~n-1~(x)]
where A~i~ are the Chebychev coefficients listed below and x= Fcalc/Fmax
Method = Robust Weighting (Prince, 1982)
W = [weight] * [1-(deltaF/6*sigmaF)^2]^2^
A~i~ are:
1.54 0.427 1.11
;
_diffn_radiation_type            'Mo K\alpha'
_diffn_radiation_wavelength      0.71073

## -----REFERENCES -----##
## Insert your own references - in alphabetic order
_publ_section_references
;

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Crystallography Laboratory, OXFORD, UK.
;

# Uequiv = arithmetic mean of Ui
# i.e. Uequiv = (U1+U2+U3)/3

# Replace trailing . with the number of unfound
# hydrogen atoms attaced to relavent atom

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_occupancy
_atom_site_adp_type
_atom_site_refinement_flags_posn
_atom_site_refinement_flags_adp
_atom_site_refinement_flags_occupancy
_atom_site_disorder_assembly
_atom_site_disorder_group
_atom_site_attached_hydrogens
Na1 Na 0.95129(9) 0.24841(9) 0.26250(5) 0.0348 1.0000 Uani . . . . .
I2 I 1.233837(15) 0.239889(15) 0.253379(9) 0.0375 1.0000 Uani . . . . .
C1 C 1.0536(3) 0.5544(2) 0.27363(16) 0.0429 1.0000 Uani . . . . .
C2 C 0.9666(3) 0.5099(3) 0.34391(17) 0.0484 1.0000 Uani . . . . .
C3 C 0.8146(3) 0.3687(3) 0.40215(16) 0.0499 1.0000 Uani . . . . .
C4 C 0.7314(3) 0.2772(3) 0.38828(18) 0.0514 1.0000 Uani . . . . .
C5 C 0.7336(3) 0.0873(3) 0.35102(18) 0.0547 1.0000 Uani . . . . .
C6 C 0.8204(3) -0.0070(3) 0.32296(17) 0.0529 1.0000 Uani . . . . .
C7 C 0.9559(3) -0.0424(2) 0.21582(16) 0.0432 1.0000 Uani . . . . .
C8 C 1.0451(3) 0.0156(2) 0.15002(15) 0.0385 1.0000 Uani . . . . .
C9 C 1.2270(2) 0.1998(2) -0.05645(16) 0.0402 1.0000 Uani . . . . .
C10 C 1.1686(2) 0.1381(2) 0.01323(15) 0.0385 1.0000 Uani . . . . .
C11 C 1.0402(2) 0.1392(2) 0.02815(14) 0.0342 1.0000 Uani . . . . .
C12 C 0.9649(2) 0.2024(2) -0.02647(13) 0.0313 1.0000 Uani . . . . .
C13 C 0.8324(2) 0.2048(2) -0.01264(15) 0.0349 1.0000 Uani . . . . .
C14 C 1.2357(2) 0.7325(2) 0.06624(16) 0.0390 1.0000 Uani . . . . .
C15 C 1.1772(2) 0.6704(2) 0.13587(16) 0.0382 1.0000 Uani . . . . .
C16 C 1.0490(2) 0.6723(2) 0.15075(14) 0.0345 1.0000 Uani . . . . .
C17 C 0.9738(2) 0.7351(2) 0.09604(14) 0.0320 1.0000 Uani . . . . .
C18 C 0.8408(2) 0.7377(2) 0.11020(15) 0.0363 1.0000 Uani . . . . .
C19 C 0.8160(2) 0.3953(2) 0.11695(14) 0.0317 1.0000 Uani . . . . .
C20 C 0.7358(2) 0.5111(2) 0.01423(14) 0.0322 1.0000 Uani . . . . .
C21 C 0.8750(2) 0.5149(2) -0.00098(13) 0.0294 1.0000 Uani . . . . .
C22 C 0.9231(2) 0.4444(2) 0.06085(13) 0.0289 1.0000 Uani . . . . .
C23 C 1.0509(2) 0.4276(2) 0.06421(13) 0.0313 1.0000 Uani . . . . .
C24 C 0.5809(2) 0.4029(2) 0.11790(15) 0.0378 1.0000 Uani . . . . .
C25 C 0.5080(2) 0.4980(2) 0.15627(16) 0.0403 1.0000 Uani . . . . .
C26 C 0.5552(3) 0.5091(3) 0.22886(16) 0.0437 1.0000 Uani . . . . .
C27 C 0.4804(3) 0.6015(3) 0.2687(2) 0.0551 1.0000 Uani . . . . .
C28 C 0.5332(3) 0.6178(3) 0.3388(2) 0.0593 1.0000 Uani . . . . .
C29 C 0.6533(4) 0.6842(4) 0.3208(2) 0.0727 1.0000 Uani . . . . .
C30 C 0.7476(4) -0.2959(4) 0.55045(19) 0.0619 1.0000 Uani . . . . .
C31 C 0.7551(3) 0.0714(3) 0.62462(18) 0.0486 1.0000 Uani . . . . .
C32 C 0.5230(3) -0.0608(3) 0.85346(18) 0.0470 1.0000 Uani . . . . .
N1 N 0.70826(18) 0.43800(18) 0.08563(11) 0.0318 1.0000 Uani . . . . .
O1 O 0.81862(16) 0.32986(16) 0.17842(10) 0.0380 1.0000 Uani . . . . .
O2 O 0.65904(16) 0.55865(18) -0.02487(11) 0.0427 1.0000 Uani . . . . .
O3 O 0.89415(19) 0.41407(17) 0.33404(10) 0.0417 1.0000 Uani . . . . .
O4 O 0.98261(18) 0.61625(17) 0.21656(11) 0.0408 1.0000 Uani . . . . .
O5 O 0.80648(17) 0.17915(18) 0.36607(10) 0.0402 1.0000 Uani . . . . .
O6 O 0.97417(17) 0.08364(17) 0.09430(10) 0.0393 1.0000 Uani . . . . .
O7 O 0.89393(19) 0.04700(16) 0.25477(10) 0.0409 1.0000 Uani . . . . .
Cl1 Cl 0.60535(11) 0.07307(9) 0.83644(8) 0.0821 1.0000 Uani . . . . .
Cl2 Cl 0.37467(9) -0.03844(11) 0.82313(6) 0.0770 1.0000 Uani . . . . .
Cl3 Cl 0.50406(11) -0.12445(14) 0.94876(7) 0.0961 1.0000 Uani . . . . .
Cl4 Cl 0.59864(10) 0.07769(11) 0.60912(9) 0.0912 1.0000 Uani . . . . .
Cl5 Cl 0.85395(12) 0.03377(12) 0.54816(7) 0.0884 1.0000 Uani . . . . .
Cl6 Cl 0.79631(11) 0.21141(9) 0.64174(8) 0.0792 1.0000 Uani . . . . .
Cl7 Cl 0.88412(9) -0.26106(9) 0.48701(5) 0.0661 1.0000 Uani . . . . .
Cl8 Cl 0.62039(11) -0.20813(15) 0.51540(8) 0.0991 1.0000 Uani . . . . .
Cl9 Cl 0.72480(15) -0.45181(12) 0.56355(8) 0.1006 1.0000 Uani . . . . .
H11 H 1.1134 0.6113 0.2841 0.0527 1.0000 Uiso . . . . .
H12 H 1.1014 0.4838 0.2563 0.0527 1.0000 Uiso . . . . .

```

H21	H	1.0166	0.4795	0.3863	0.0591	1.0000	Uiso	.	.	.	.	.	.
H22	H	0.9092	0.5785	0.3561	0.0591	1.0000	Uiso	.	.	.	.	.	.
H31	H	0.8669	0.3299	0.4419	0.0589	1.0000	Uiso	.	.	.	.	.	.
H32	H	0.7625	0.4373	0.4192	0.0589	1.0000	Uiso	.	.	.	.	.	.
H41	H	0.6742	0.2468	0.4354	0.0598	1.0000	Uiso	.	.	.	.	.	.
H42	H	0.6806	0.3152	0.3475	0.0598	1.0000	Uiso	.	.	.	.	.	.
H51	H	0.6804	0.0497	0.3982	0.0659	1.0000	Uiso	.	.	.	.	.	.
H52	H	0.6789	0.1239	0.3120	0.0659	1.0000	Uiso	.	.	.	.	.	.
H61	H	0.8765	-0.0418	0.3615	0.0631	1.0000	Uiso	.	.	.	.	.	.
H62	H	0.7708	-0.0731	0.3133	0.0631	1.0000	Uiso	.	.	.	.	.	.
H71	H	1.0032	-0.1017	0.2510	0.0531	1.0000	Uiso	.	.	.	.	.	.
H72	H	0.8922	-0.0860	0.1975	0.0531	1.0000	Uiso	.	.	.	.	.	.
H81	H	1.0997	0.0710	0.1665	0.0480	1.0000	Uiso	.	.	.	.	.	.
H82	H	1.0982	-0.0485	0.1286	0.0480	1.0000	Uiso	.	.	.	.	.	.
H91	H	1.3204	0.1977	-0.0668	0.0494	1.0000	Uiso	.	.	.	.	.	.
H101	H	1.2203	0.0935	0.0519	0.0475	1.0000	Uiso	.	.	.	.	.	.
H131	H	0.7889	0.1606	0.0362	0.0430	1.0000	Uiso	.	.	.	.	.	.
H141	H	1.3291	0.7307	0.0558	0.0484	1.0000	Uiso	.	.	.	.	.	.
H151	H	1.2289	0.6250	0.1743	0.0473	1.0000	Uiso	.	.	.	.	.	.
H181	H	0.7973	0.6939	0.1592	0.0443	1.0000	Uiso	.	.	.	.	.	.
H231	H	1.0861	0.3779	0.1087	0.0393	1.0000	Uiso	.	.	.	.	.	.
H241	H	0.5857	0.3257	0.1557	0.0462	1.0000	Uiso	.	.	.	.	.	.
H242	H	0.5353	0.3891	0.0766	0.0462	1.0000	Uiso	.	.	.	.	.	.
H251	H	0.5150	0.5783	0.1210	0.0474	1.0000	Uiso	.	.	.	.	.	.
H252	H	0.4183	0.4756	0.1680	0.0474	1.0000	Uiso	.	.	.	.	.	.
H261	H	0.6442	0.5337	0.2169	0.0524	1.0000	Uiso	.	.	.	.	.	.
H262	H	0.5505	0.4281	0.2635	0.0524	1.0000	Uiso	.	.	.	.	.	.
H271	H	0.4804	0.6814	0.2330	0.0659	1.0000	Uiso	.	.	.	.	.	.
H272	H	0.3926	0.5741	0.2837	0.0659	1.0000	Uiso	.	.	.	.	.	.
H281	H	0.4701	0.6649	0.3683	0.0714	1.0000	Uiso	.	.	.	.	.	.
H282	H	0.5490	0.5359	0.3699	0.0714	1.0000	Uiso	.	.	.	.	.	.
H291	H	0.6838	0.6924	0.3686	0.0894	1.0000	Uiso	.	.	.	.	.	.
H292	H	0.6387	0.7667	0.2901	0.0894	1.0000	Uiso	.	.	.	.	.	.
H293	H	0.7175	0.6377	0.2917	0.0894	1.0000	Uiso	.	.	.	.	.	.
H301	H	0.7552	-0.2760	0.6004	0.0757	1.0000	Uiso	.	.	.	.	.	.
H311	H	0.7649	0.0068	0.6699	0.0576	1.0000	Uiso	.	.	.	.	.	.
H321	H	0.5743	-0.1165	0.8239	0.0571	1.0000	Uiso	.	.	.	.	.	.
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													
Na1		0.0338(5)	0.0349(5)	0.0344(5)	-0.0025(4)	-0.0049(4)	-0.0032(4)						
I2		0.03553(9)	0.03604(9)	0.04163(10)	-0.01058(6)	-0.00462(6)	0.00178(6)						
C1		0.0521(15)	0.0296(11)	0.0499(15)	-0.0054(10)	-0.0208(12)	0.0003(10)						
C2		0.0679(19)	0.0367(13)	0.0431(14)	-0.0071(11)	-0.0164(13)	-0.0028(13)						
C3		0.0620(18)	0.0461(15)	0.0392(14)	-0.0121(11)	0.0037(12)	0.0049(13)						
C4		0.0431(15)	0.0581(18)	0.0483(15)	-0.0060(13)	0.0018(12)	0.0031(13)						
C5		0.0544(17)	0.067(2)	0.0437(15)	-0.0104(14)	0.0011(13)	-0.0260(15)						
C6		0.072(2)	0.0456(15)	0.0403(14)	-0.0014(12)	-0.0036(13)	-0.0246(14)						
C7		0.0546(16)	0.0318(12)	0.0464(14)	-0.0084(10)	-0.0156(12)	-0.0025(11)						
C8		0.0451(13)	0.0356(12)	0.0394(13)	-0.0117(10)	-0.0160(10)	0.0018(10)						
C9		0.0310(11)	0.0436(13)	0.0488(14)	-0.0159(11)	-0.0074(10)	0.0036(10)						
C10		0.0338(12)	0.0394(12)	0.0456(13)	-0.0119(10)	-0.0132(10)	0.0045(10)						
C11		0.0361(12)	0.0307(11)	0.0397(12)	-0.0140(9)	-0.0087(9)	0.0008(9)						
C12		0.0321(11)	0.0277(10)	0.0377(11)	-0.0125(9)	-0.0086(9)	-0.0006(8)						
C13		0.0292(11)	0.0350(11)	0.0432(13)	-0.0139(10)	-0.0052(9)	-0.0010(9)						
C14		0.0312(11)	0.0392(12)	0.0506(14)	-0.0155(11)	-0.0098(10)	0.0008(10)						

```

C15 0.0370(12) 0.0336(12) 0.0478(14) -0.0111(10) -0.0166(10) 0.0044(9)
C16 0.0373(12) 0.0273(10) 0.0421(12) -0.0119(9) -0.0097(10) 0.0002(9)
C17 0.0327(11) 0.0289(10) 0.0381(12) -0.0137(9) -0.0083(9) 0.0026(8)
C18 0.0327(12) 0.0343(12) 0.0438(13) -0.0131(10) -0.0037(10) -0.0008(9)
C19 0.0291(11) 0.0302(10) 0.0393(12) -0.0119(9) -0.0115(9) 0.0037(8)
C20 0.0269(10) 0.0345(11) 0.0391(12) -0.0122(9) -0.0115(9) 0.0015(8)
C21 0.0272(10) 0.0294(10) 0.0352(11) -0.0113(9) -0.0115(8) 0.0043(8)
C22 0.0273(10) 0.0290(10) 0.0345(11) -0.0132(8) -0.0101(8) 0.0047(8)
C23 0.0298(11) 0.0330(11) 0.0354(11) -0.0119(9) -0.0140(9) 0.0054(8)
C24 0.0272(11) 0.0398(12) 0.0484(14) -0.0105(11) -0.0083(10) -0.0041(9)
C25 0.0294(11) 0.0429(13) 0.0461(14) -0.0050(11) -0.0041(10) 0.0036(10)
C26 0.0384(13) 0.0443(14) 0.0482(15) -0.0106(11) -0.0059(11) 0.0061(11)
C27 0.0484(16) 0.0547(17) 0.0617(18) -0.0172(14) -0.0032(14) 0.0108(13)
C28 0.0575(18) 0.0608(19) 0.0602(19) -0.0224(15) 0.0014(15) 0.0068(15)
C29 0.081(3) 0.074(2) 0.068(2) -0.0272(19) -0.0057(19) -0.012(2)
C30 0.069(2) 0.075(2) 0.0454(16) -0.0163(15) -0.0078(15) -0.0142(17)
C31 0.0535(16) 0.0388(14) 0.0517(16) -0.0054(12) -0.0074(13) 0.0023(12)
C32 0.0420(14) 0.0458(14) 0.0550(16) -0.0172(12) -0.0050(12) 0.0055(11)
N1 0.0242(9) 0.0347(10) 0.0387(10) -0.0089(8) -0.0095(7) 0.0002(7)
O1 0.0344(9) 0.0398(9) 0.0397(9) -0.0047(7) -0.0103(7) 0.0009(7)
O2 0.0283(8) 0.0517(11) 0.0486(10) -0.0049(8) -0.0163(7) 0.0038(7)
O3 0.0505(11) 0.0364(9) 0.0387(9) -0.0109(7) -0.0023(8) -0.0010(8)
O4 0.0421(10) 0.0377(9) 0.0418(9) -0.0042(7) -0.0095(7) 0.0035(7)
O5 0.0398(9) 0.0446(10) 0.0356(9) -0.0059(7) -0.0034(7) -0.0065(8)
O6 0.0371(9) 0.0443(10) 0.0365(9) -0.0067(7) -0.0080(7) 0.0012(7)
O7 0.0549(11) 0.0330(9) 0.0348(9) -0.0045(7) -0.0056(8) -0.0091(8)
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C12 0.0602(5) 0.1008(8) 0.0730(6) -0.0117(5) -0.0299(4) 0.0056(5)
C13 0.0767(7) 0.1286(10) 0.0773(6) 0.0261(7) -0.0371(5) -0.0462(7)
C14 0.0573(5) 0.0813(7) 0.1444(11) -0.0341(7) -0.0283(6) -0.0001(5)
C15 0.0896(7) 0.0980(8) 0.0785(7) -0.0384(6) 0.0232(6) -0.0199(6)
C16 0.0781(6) 0.0579(5) 0.1126(8) -0.0356(5) -0.0219(6) -0.0029(4)
C17 0.0662(5) 0.0621(5) 0.0650(5) -0.0007(4) -0.0050(4) -0.0092(4)
C18 0.0673(6) 0.1346(11) 0.1031(9) -0.0413(8) -0.0182(6) 0.0132(7)
C19 0.1238(10) 0.0841(8) 0.0927(8) -0.0023(6) -0.0093(7) -0.0533(8)
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C2 . H21 . 1.000 no
C2 . H22 . 1.000 no
C3 . C4 . 1.490(5) yes
C3 . O3 . 1.432(3) yes
C3 . H31 . 1.000 no
C3 . H32 . 1.000 no
C4 . O5 . 1.419(4) yes
C4 . H41 . 1.000 no
C4 . H42 . 1.000 no
C5 . C6 . 1.492(5) yes
C5 . O5 . 1.429(4) yes
C5 . H51 . 1.000 no
C5 . H52 . 1.000 no

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C6 . O7 . 1.427(3) yes
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C6 . H62 . 1.000 no
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C7 . O7 . 1.427(3) yes
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C7 . H72 . 1.000 no
C8 . O6 . 1.431(3) yes
C8 . H81 . 1.000 no
C8 . H82 . 1.000 no
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C9 . C10 . 1.407(4) yes
C9 . H91 . 1.000 no
C10 . C11 . 1.375(4) yes
C10 . H101 . 1.000 no
C11 . C12 . 1.428(3) yes
C11 . O6 . 1.369(3) yes
C12 . C17 2_765 1.420(3) yes
C12 . C13 . 1.419(3) yes
C13 . C14 2_765 1.365(4) yes
C13 . H131 . 1.000 no
C14 . C15 . 1.409(4) yes
C14 . H141 . 1.000 no
C15 . C16 . 1.374(4) yes
C15 . H151 . 1.000 no
C16 . C17 . 1.428(3) yes
C16 . O4 . 1.368(3) yes
C17 . C18 . 1.423(3) yes
C18 . H181 . 1.000 no
C19 . C22 . 1.488(3) yes
C19 . N1 . 1.387(3) yes
C19 . O1 . 1.218(3) yes
C20 . C21 . 1.492(3) yes
C20 . N1 . 1.402(3) yes
C20 . O2 . 1.202(3) yes
C21 . C23 2_765 1.380(3) yes
C21 . C22 . 1.391(3) yes
C22 . C23 . 1.390(3) yes
C23 . H231 . 1.000 no
C24 . C25 . 1.515(4) yes
C24 . N1 . 1.465(3) yes
C24 . H241 . 1.000 no
C24 . H242 . 1.000 no
C25 . C26 . 1.519(4) yes
C25 . H251 . 1.000 no
C25 . H252 . 1.000 no
C26 . C27 . 1.515(4) yes
C26 . H261 . 1.000 no
C26 . H262 . 1.000 no
C27 . C28 . 1.525(5) yes
C27 . H271 . 1.000 no
C27 . H272 . 1.000 no
C28 . C29 . 1.501(6) yes
C28 . H281 . 1.000 no
C28 . H282 . 1.000 no
C29 . H291 . 1.000 no
C29 . H292 . 1.000 no
C29 . H293 . 1.000 no
C30 . C17 . 1.757(4) yes
C30 . C18 . 1.761(4) yes
C30 . C19 . 1.750(4) yes

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C30 . H301 . 1.000 no
C31 . C14 . 1.749(3) yes
C31 . C15 . 1.742(3) yes
C31 . C16 . 1.761(3) yes
C31 . H311 . 1.000 no
C32 . C11 . 1.749(3) yes
C32 . C12 . 1.757(3) yes
C32 . C13 . 1.737(3) yes
C32 . H321 . 1.000 no
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C2 . C1 . H11 . 109.536 no
O4 . C1 . H11 . 109.536 no
C2 . C1 . H12 . 109.536 no
O4 . C1 . H12 . 109.536 no
H11 . C1 . H12 . 109.467 no
C1 . C2 . O3 . 110.7(2) yes
C1 . C2 . H21 . 109.175 no
O3 . C2 . H21 . 109.174 no
C1 . C2 . H22 . 109.175 no
O3 . C2 . H22 . 109.174 no
H21 . C2 . H22 . 109.467 no
C4 . C3 . O3 . 109.4(2) yes
C4 . C3 . H31 . 109.492 no
O3 . C3 . H31 . 109.492 no
C4 . C3 . H32 . 109.492 no
O3 . C3 . H32 . 109.492 no
H31 . C3 . H32 . 109.467 no
C3 . C4 . O5 . 108.8(2) yes
C3 . C4 . H41 . 109.643 no
O5 . C4 . H41 . 109.643 no
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O5 . C4 . H42 . 109.643 no
H41 . C4 . H42 . 109.467 no
C6 . C5 . O5 . 108.5(3) yes
C6 . C5 . H51 . 109.716 no
O5 . C5 . H51 . 109.716 no
C6 . C5 . H52 . 109.716 no
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C5 . C6 . H61 . 109.521 no
O7 . C6 . H61 . 109.521 no
C5 . C6 . H62 . 109.521 no
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H61 . C6 . H62 . 109.467 no
C8 . C7 . O7 . 110.6(2) yes
C8 . C7 . H71 . 109.190 no
O7 . C7 . H71 . 109.190 no
C8 . C7 . H72 . 109.190 no
O7 . C7 . H72 . 109.190 no
H71 . C7 . H72 . 109.467 no
C7 . C8 . O6 . 108.5(2) yes

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C7 . C8 . H81 . 109.720 no
O6 . C8 . H81 . 109.720 no
C7 . C8 . H82 . 109.720 no
O6 . C8 . H82 . 109.720 no
H81 . C8 . H82 . 109.467 no
C18 2_765 C9 . C10 . 121.6(2) yes
C18 2_765 C9 . H91 . 119.218 no
C10 . C9 . H91 . 119.218 no
C9 . C10 . C11 . 119.9(2) yes
C9 . C10 . H101 . 120.026 no
C11 . C10 . H101 . 120.026 no
C10 . C11 . C12 . 120.7(2) yes
C10 . C11 . O6 . 124.6(2) yes
C12 . C11 . O6 . 114.7(2) yes
C11 . C12 . C17 2_765 118.2(2) yes
C11 . C12 . C13 . 121.9(2) yes
C17 2_765 C12 . C13 . 119.9(2) yes
C12 . C13 . C14 2_765 119.8(2) yes
C12 . C13 . H131 . 120.102 no
C14 2_765 C13 . H131 . 120.102 no
C13 2_765 C14 . C15 . 121.4(2) yes
C13 2_765 C14 . H141 . 119.277 no
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C14 . C15 . C16 . 119.8(2) yes
C14 . C15 . H151 . 120.121 no
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C15 . C16 . O4 . 124.7(2) yes
C17 . C16 . O4 . 114.5(2) yes
C16 . C17 . C12 2_765 118.2(2) yes
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C12 2_765 C17 . C18 . 120.0(2) yes
C17 . C18 . C9 2_765 119.5(2) yes
C17 . C18 . H181 . 120.242 no
C9 2_765 C18 . H181 . 120.242 no
C22 . C19 . N1 . 106.5(2) yes
C22 . C19 . O1 . 128.4(2) yes
N1 . C19 . O1 . 125.1(2) yes
C21 . C20 . N1 . 106.03(18) yes
C21 . C20 . O2 . 129.2(2) yes
N1 . C20 . O2 . 124.8(2) yes
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C19 . C22 . C21 . 108.04(19) yes
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C22 . C23 . C21 2_765 114.4(2) yes
C22 . C23 . H231 . 122.804 no
C21 2_765 C23 . H231 . 122.804 no
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C25 . C24 . H241 . 108.560 no
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C25 . C24 . H242 . 108.560 no
N1 . C24 . H242 . 108.560 no
H241 . C24 . H242 . 109.467 no
C24 . C25 . C26 . 113.0(2) yes
C24 . C25 . H251 . 108.585 no
C26 . C25 . H251 . 108.586 no
C24 . C25 . H252 . 108.585 no
C26 . C25 . H252 . 108.586 no

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H251 . C25 . H252 . 109.467 no
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C25 . C26 . H261 . 108.518 no
C27 . C26 . H261 . 108.518 no
C25 . C26 . H262 . 108.518 no
C27 . C26 . H262 . 108.518 no
H261 . C26 . H262 . 109.467 no
C26 . C27 . C28 . 112.9(3) yes
C26 . C27 . H271 . 108.606 no
C28 . C27 . H271 . 108.605 no
C26 . C27 . H272 . 108.606 no
C28 . C27 . H272 . 108.605 no
H271 . C27 . H272 . 109.467 no
C27 . C28 . C29 . 112.7(3) yes
C27 . C28 . H281 . 108.648 no
C29 . C28 . H281 . 108.648 no
C27 . C28 . H282 . 108.648 no
C29 . C28 . H282 . 108.648 no
H281 . C28 . H282 . 109.467 no
C28 . C29 . H291 . 109.467 no
C28 . C29 . H292 . 109.467 no
H291 . C29 . H292 . 109.475 no
C28 . C29 . H293 . 109.467 no
H291 . C29 . H293 . 109.476 no
H292 . C29 . H293 . 109.476 no
C17 . C30 . C18 . 109.4(2) yes
C17 . C30 . C19 . 108.6(2) yes
C18 . C30 . C19 . 112.2(2) yes
C17 . C30 . H301 . 111.065 no
C18 . C30 . H301 . 107.447 no
C19 . C30 . H301 . 108.175 no
C14 . C31 . C15 . 111.01(19) yes
C14 . C31 . C16 . 110.44(17) yes
C15 . C31 . C16 . 109.88(18) yes
C14 . C31 . H311 . 107.900 no
C15 . C31 . H311 . 108.476 no
C16 . C31 . H311 . 109.073 no
C11 . C32 . C12 . 112.90(17) yes
C11 . C32 . C13 . 110.55(18) yes
C12 . C32 . C13 . 108.40(17) yes
C11 . C32 . H321 . 106.007 no
C12 . C32 . H321 . 108.253 no
C13 . C32 . H321 . 110.706 no
C24 . N1 . C20 . 122.58(19) yes
C24 . N1 . C19 . 125.5(2) yes
C20 . N1 . C19 . 111.69(19) yes
C3 . O3 . C2 . 110.5(2) yes
C1 . O4 . C16 . 116.6(2) yes
C5 . O5 . C4 . 112.4(2) yes
C8 . O6 . C11 . 116.9(2) yes
C6 . O7 . C7 . 111.7(2) yes
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CRYSTALS (Watkin et al 2001)
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_computing_molecular_graphics
;
CAMERON (Watkin et al 1996)
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x,-y,z+1/2
-x+1/2,y+1/2,-z+1/2
x+1/2,-y+1/2,z+1/2

# choose from: rm (reference molecule of
# known chirality), ad (anomalous
# dispersion - ie. Flack param), rmad
# (both rm and ad), syn (known from
# synthetic pathway), unk (unknown)
# or . (not applicable).

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'O' ' 0.0080 0.0060 3.0485 13.2771 2.2868 5.7011 1.5463 0.3239 0.8670 32.9089
0.2508 International_Tables_Vol_IV_Table_2.2B

'Cl' ' 0.1320 0.1590 11.4604 0.0104 7.1964 1.1662 6.2556 18.5194 1.6455 47.7784
-9.5574 International_Tables_Vol_IV_Table_2.2B

_cell_formula_units_Z 4

_chemical_formula_sum ' C122 H96 B2 F48 N2 Na2 O14 '

_chemical_formula_moiety

;

C64 H24 B2 F48, (C36 H44 O10), (C22 H28 N2 O4), 2Na

;

_chemical_compound_source

;

?

;

_chemical_formula_weight 2793.36

_cell_measurement_reflns_used 87214

_cell_measurement_theta_min 5

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_cell_measurement_temperature 180

_exptl_crystal_description ' block '

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_exptl_crystal_size_min 0.18

_exptl_crystal_size_mid 0.23

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_exptl_crystal_density_diffn 1.342

_exptl_crystal_density_meas ?

Non-dispersive F(000):

_exptl_crystal_F_000 5672.001

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_exptl_absorpt_coefficient_mu    0.135
# Sheldrick geometric definitions 0.97 0.98

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;
Nonius Kappa CCD
;
_diffraction_radiation_monochromator graphite
_computing_data_collection
;
COLLECT (Nonius BV, 1997)
;
_computing_data_reduction
;
Denzo/Scalepack (Otwinowski & Minor, 1996)
;
_computing_cell_refinement
;
Denzo/Scalepack (Otwinowski & Minor, 1996)
;
_computing_structure_solution
;
SIR92 (Altomare et al, 1994)
;
_diffraction_measurement_method      \w
_exptl_absorpt_correction_type      multi-scan
_exptl_absorpt_correction_T_min     0.803
_exptl_absorpt_correction_T_max     0.980
_exptl_absorpt_process_details
;
multi-scan from symmetry-related measurements
Sortav (Blessing 1995)
;
_diffraction_standards_interval_time 0
_diffraction_standards_interval_count 0
_diffraction_standards_number        0
_diffraction_standards_decay_%       0.00

_diffraction_ambient_temperature     180
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_reflns_number_total                 15601
_diffraction_reflns_av_R_equivalents 0.00
# Number of reflections with Friedels Law is 15601
# Number of reflections without Friedels Law is 0
# Theoretical number of reflections is about 15863

_diffraction_reflns_theta_min        5.152
_diffraction_reflns_theta_max        27.505
_diffraction_measured_fraction_theta_max 0.982

_diffraction_reflns_theta_full       26.405
_diffraction_measured_fraction_theta_full 0.991

_diffraction_reflns_limit_h_min      -49
_diffraction_reflns_limit_h_max      49
_diffraction_reflns_limit_k_min      0
_diffraction_reflns_limit_k_max      20
_diffraction_reflns_limit_l_min      0
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_reflns_limit_h_min                  -49
_reflns_limit_h_max                  49

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_reflns_limit_k_max      20
_reflns_limit_l_min      0
_reflns_limit_l_max      29

_refine_diff_density_min  -0.78
_refine_diff_density_max  2.01

_refine_ls_number_reflns  6111
_refine_ls_number_restraints 292
_refine_ls_number_parameters 690

#_refine_ls_R_factor_ref    0.1580
_refine_ls_wR_factor_ref    0.1728
_refine_ls_goodness_of_fit_ref 1.1708

#_reflns_number_all 15601
_refine_ls_R_factor_all    0.2420
_refine_ls_wR_factor_all    0.2243

# The I/u(I) cutoff below was used for refinement as
# well as the _gt R-factors:
_reflns_threshold_expression I>3.00u(I)
_reflns_number_gt          6111
_refine_ls_R_factor_gt      0.1580
_refine_ls_wR_factor_gt      0.1728

_refine_ls_shift/su_max     0.027353
_refine_ls_structure_factor_coef F
_refine_ls_weighting_scheme  calc
_refine_ls_weighting_details
;
Method, part 1, Chebychev polynomial, (Watkin, 1994, Prince, 1982)
[weight] = 1.0/[A~0~*T~0~(x)+A~1~*T~1~(x) ... +A~n-1~*T~n-1~(x)]
where A~i~ are the Chebychev coefficients listed below and x= Fcalc/Fmax
Method = Robust Weighting (Prince, 1982)
W = [weight] * [1-(deltaF/6*sigmaF)^2]^2
A~i~ are:
3.82 2.87 2.53
;
_diffn_radiation_type      'Mo K\alpha'
_diffn_radiation_wavelength 0.71073
_exptl_special_details
;
Treatment of residual electron density using the SQUEEZE procedure
(P. v.d. Sluis & A. L. Spek, Acta Crystallogr. 1990, A46, 194.),
implemented in PLATON (PLATON, A Multipurpose Crystallographic Tool,
Utrecht University, Utrecht, The Netherlands, A. L. Spek, 1998.).
Therefore structure Contains Solvent Accessible VOIDS of
265.00 Å3 - equiv to ca 7 H2O molecules
disordered per unit pseudorotaxane 3.5 molec /asym unit.
Identification of the crystallising solvent as water is reliant
upon additional chemical evidence; it cannot be deduced from the
X-ray data alone

```

Each of the 8 CF₃ groups has been modelled as disordered over 2 sites with refined occupancies. In view of the severe shortage of data their temperature factors have been refined isotropically.

One hexyl chain has been modelled as disordered over two sites with refined occupancy & isotropic temperature

```

factors.
;

## -----REFERENCES -----##
## Insert your own references - in alphabetic order
##_publ_section_references
;
Sortav (Blessing 1995)

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Watkin, D.J., Prout, C.K. & Pearce, L.J. (1996) CAMERON, Chemical
Crystallography Laboratory, OXFORD, UK.
;

# Uequiv = arithmetic mean of Ui
# i.e. Uequiv = (U1+U2+U3)/3

# Replace trailing . with the number of unfound
# hydrogen atoms attached to relevant atom

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_occupancy
_atom_site_adp_type
_atom_site_refinement_flags
_atom_site_attached_hydrogens
Na1 Na 0.38217(9) 0.5976(2) 0.38601(16) 0.0484 1.0000 Uani . .
N1 N 0.47521(18) 0.7038(4) 0.5017(3) 0.0384 1.0000 Uani D .
C1 C 0.4569(2) 0.6422(6) 0.4715(4) 0.0402 1.0000 Uani . .
O1 O 0.42964(16) 0.6520(4) 0.4425(3) 0.0466 1.0000 Uani . .

```

C2 C 0.4764(2) 0.5607(5) 0.4824(3) 0.0302 1.0000 Uani . .
C3 C 0.5063(2) 0.5801(5) 0.5195(3) 0.0311 1.0000 Uani . .
C4 C 0.5049(2) 0.6738(5) 0.5334(4) 0.0376 1.0000 Uani . .
O2 O 0.52518(17) 0.7148(4) 0.5633(3) 0.0529 1.0000 Uani . .
C5 C 0.5300(2) 0.5197(5) 0.5379(3) 0.0327 1.0000 Uani . .
C6 C 0.4658(3) 0.7946(5) 0.5031(3) 0.0603 1.0000 Uani DU .
C7 C 0.4496(3) 0.8242(5) 0.5566(4) 0.0701 1.0000 Uani DU .
C8 C 0.4439(3) 0.9178(6) 0.5614(4) 0.0803 1.0000 Uani DU .
C9 C 0.4462(7) 0.9537(8) 0.6222(6) 0.081(5) 0.48(2) Uiso DUP .
C10 C 0.4452(7) 1.049(1) 0.6268(7) 0.082(6) 0.48(2) Uiso DUP .
C11 C 0.4407(8) 1.0850(13) 0.687(1) 0.084(6) 0.48(2) Uiso DUP .
C109 C 0.4750(5) 0.971(1) 0.5635(7) 0.111(6) 0.52(2) Uiso DUP .
C110 C 0.5005(6) 0.9548(18) 0.6120(12) 0.114(7) 0.52(2) Uiso DUP .
C111 C 0.5304(7) 1.017(2) 0.6187(14) 0.119(7) 0.52(2) Uiso DUP .
C12 C 0.4248(2) 0.5214(7) 0.5993(4) 0.0461 1.0000 Uani . .
C13 C 0.4346(3) 0.5992(6) 0.6221(4) 0.0499 1.0000 Uani . .
C14 C 0.4662(3) 0.6052(6) 0.6568(4) 0.0488 1.0000 Uani . .
C15 C 0.4869(3) 0.5357(7) 0.6695(4) 0.0494 1.0000 Uani . .
C16 C 0.4762(2) 0.4547(7) 0.6466(4) 0.0473 1.0000 Uani . .
C17 C 0.4966(2) 0.3793(6) 0.6562(4) 0.0438 1.0000 Uani . .
C18 C 0.4861(2) 0.3042(6) 0.6339(4) 0.0468 1.0000 Uani . .
C19 C 0.4552(3) 0.2980(6) 0.5999(5) 0.0488 1.0000 Uani . .
C20 C 0.4346(2) 0.3669(6) 0.5872(4) 0.0450 1.0000 Uani . .
C21 C 0.4447(2) 0.4474(6) 0.6103(4) 0.0367 1.0000 Uani . .
O3 O 0.39465(17) 0.5090(4) 0.5661(3) 0.0558 1.0000 Uani . .
C22 C 0.3727(3) 0.5800(7) 0.5509(4) 0.0528 1.0000 Uani . .
C23 C 0.3425(3) 0.5492(8) 0.5123(6) 0.0685 1.0000 Uani . .
O4 O 0.35379(19) 0.5203(5) 0.4573(4) 0.0653 1.0000 Uani . .
C24 C 0.3612(4) 0.4307(8) 0.4537(7) 0.0910 1.0000 Uani . .
C25 C 0.3608(4) 0.4062(9) 0.3916(8) 0.0954 1.0000 Uani . .
O5 O 0.3862(2) 0.4522(5) 0.3646(4) 0.0796 1.0000 Uani . .
C26 C 0.3846(3) 0.437(1) 0.3034(7) 0.0860 1.0000 Uani . .
C27 C 0.4092(4) 0.4935(11) 0.2772(7) 0.0971 1.0000 Uani . .
O6 O 0.40147(17) 0.5826(5) 0.2911(3) 0.0610 1.0000 Uani . .
C28 C 0.4204(3) 0.6449(9) 0.2599(5) 0.0693 1.0000 Uani . .
C29 C 0.4512(3) 0.6761(7) 0.2969(4) 0.0574 1.0000 Uani . .
O7 O 0.47382(17) 0.6057(5) 0.3067(3) 0.0598 1.0000 Uani . .
B1 B 0.1788(2) 0.5477(6) 0.2209(4) 0.0349 1.0000 Uani . .
C30 C 0.18833(14) 0.4672(4) 0.1785(2) 0.0336 1.0000 Uani . .
C31 C 0.16175(16) 0.4161(4) 0.1537(3) 0.0361 1.0000 Uani . .
C32 C 0.16856(16) 0.3455(4) 0.1179(3) 0.0386 1.0000 Uani DU .
C33 C 0.20162(18) 0.3245(4) 0.1061(3) 0.0490 1.0000 Uani . .
C34 C 0.2289(2) 0.3753(4) 0.1301(3) 0.0557 1.0000 Uani DU .
C35 C 0.22201(16) 0.4437(4) 0.1654(3) 0.0420 1.0000 Uani . .
C36 C 0.14388(14) 0.5965(4) 0.1923(2) 0.0340 1.0000 Uani . .
C37 C 0.11823(16) 0.6307(4) 0.2273(3) 0.0382 1.0000 Uani . .
C38 C 0.08996(16) 0.6742(4) 0.2022(3) 0.0493 1.0000 Uani DU .
C39 C 0.08444(18) 0.6834(4) 0.1423(3) 0.0574 1.0000 Uani . .
C40 C 0.1083(2) 0.6482(4) 0.1080(3) 0.0647 1.0000 Uani DU .
C41 C 0.13805(16) 0.6060(4) 0.1328(3) 0.0484 1.0000 Uani . .
C42 C 0.21113(15) 0.6162(4) 0.2272(3) 0.0404 1.0000 Uani . .
C43 C 0.24301(18) 0.5948(4) 0.2579(3) 0.0590 1.0000 Uani . .
C44 C 0.2710(2) 0.6496(5) 0.2590(4) 0.0856 1.0000 Uani DU .
C45 C 0.2694(2) 0.7284(6) 0.2346(4) 0.0828 1.0000 Uani . .
C46 C 0.23853(19) 0.7522(5) 0.2033(4) 0.0627 1.0000 Uani DU .
C47 C 0.21073(18) 0.6941(5) 0.2004(3) 0.0454 1.0000 Uani . .
C48 C 0.17196(15) 0.5119(4) 0.2863(3) 0.0407 1.0000 Uani . .
C49 C 0.16049(18) 0.4291(4) 0.2965(3) 0.0491 1.0000 Uani . .
C50 C 0.1542(2) 0.4002(5) 0.3541(4) 0.0710 1.0000 Uani DU .
C51 C 0.1572(2) 0.4550(6) 0.4008(4) 0.0723 1.0000 Uani . .
C52 C 0.16810(19) 0.5372(5) 0.3909(4) 0.0855 1.0000 Uani DU .

C53 C 0.17459(18) 0.5655(5) 0.3364(3) 0.0570 1.0000 Uani . .
C54 C 0.13759(18) 0.2947(5) 0.0948(3) 0.0461(16) 1.0000 Uiso DU .
C55 C 0.2652(2) 0.3523(6) 0.1173(4) 0.085(2) 1.0000 Uiso DU .
C56 C 0.0638(2) 0.7114(5) 0.2404(4) 0.073(2) 1.0000 Uiso DU .
C57 C 0.1031(3) 0.6566(6) 0.0432(4) 0.089(2) 1.0000 Uiso DU .
C58 C 0.3053(2) 0.6184(6) 0.2842(4) 0.087(2) 1.0000 Uiso DU .
C59 C 0.2346(2) 0.8357(6) 0.1749(4) 0.086(2) 1.0000 Uiso DU .
C60 C 0.1453(3) 0.3093(6) 0.3641(5) 0.115(3) 1.0000 Uiso DU .
C61 C 0.1721(3) 0.5983(7) 0.4411(4) 0.111(3) 1.0000 Uiso DU .
F1 F 0.1484(3) 0.219(1) 0.0684(7) 0.046(3) 0.52(3) Uiso DUP .
F2 F 0.1196(4) 0.3325(8) 0.0549(7) 0.049(3) 0.52(3) Uiso DUP .
F3 F 0.1171(4) 0.269(1) 0.1382(5) 0.043(3) 0.52(3) Uiso DUP .
F4 F 0.1106(4) 0.3471(8) 0.0705(7) 0.045(3) 0.48(3) Uiso DUP .
F5 F 0.1436(3) 0.2415(11) 0.0529(8) 0.052(3) 0.48(3) Uiso DUP .
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F7 F 0.2898(4) 0.3941(11) 0.1487(8) 0.082(4) 0.456(15) Uiso DUP .
F8 F 0.2708(4) 0.3640(15) 0.0613(9) 0.099(4) 0.456(15) Uiso DUP .
F9 F 0.2726(4) 0.2676(12) 0.1305(9) 0.088(4) 0.456(15) Uiso DUP .
F10 F 0.2676(3) 0.3174(12) 0.0651(7) 0.086(4) 0.544(15) Uiso DUP .
F11 F 0.2866(3) 0.4219(9) 0.1177(7) 0.081(4) 0.544(15) Uiso DUP .
F12 F 0.2800(4) 0.2976(11) 0.1555(8) 0.096(4) 0.544(15) Uiso DUP .
F13 F 0.0399(5) 0.7590(12) 0.2128(6) 0.069(4) 0.420(14) Uiso DUP .
F14 F 0.0822(4) 0.7624(13) 0.2877(7) 0.075(4) 0.420(14) Uiso DUP .
F15 F 0.0484(5) 0.6574(11) 0.2713(9) 0.084(4) 0.420(14) Uiso DUP .
F16 F 0.0725(3) 0.7180(9) 0.2937(5) 0.076(3) 0.580(14) Uiso DUP .
F17 F 0.0519(3) 0.7902(8) 0.2197(5) 0.065(3) 0.580(14) Uiso DUP .
F18 F 0.0345(4) 0.6567(8) 0.2413(7) 0.085(3) 0.580(14) Uiso DUP .
F19 F 0.1169(3) 0.5913(7) 0.0136(5) 0.087(3) 0.678(11) Uiso DUP .
F20 F 0.1037(4) 0.7351(8) 0.0240(5) 0.088(3) 0.678(11) Uiso DUP .
F21 F 0.0664(3) 0.6334(8) 0.0253(5) 0.095(3) 0.678(11) Uiso DUP .
F22 F 0.0850(7) 0.7195(16) 0.0252(8) 0.095(5) 0.322(11) Uiso DUP .
F23 F 0.1396(6) 0.6771(14) 0.0171(8) 0.089(5) 0.322(11) Uiso DUP .
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F25 F 0.3323(3) 0.6795(8) 0.2793(7) 0.076(3) 0.570(13) Uiso DUP .
F26 F 0.3035(3) 0.6067(11) 0.3393(7) 0.097(4) 0.570(13) Uiso DUP .
F27 F 0.3154(3) 0.547(1) 0.2638(7) 0.091(4) 0.570(13) Uiso DUP .
F28 F 0.3040(4) 0.5554(14) 0.3204(9) 0.093(4) 0.430(13) Uiso DUP .
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F32 F 0.2240(7) 0.8904(12) 0.2047(9) 0.096(4) 0.43(2) Uiso DUP .
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F34 F 0.2632(4) 0.8849(9) 0.1836(8) 0.088(4) 0.57(2) Uiso DUP .
F35 F 0.2095(5) 0.8863(9) 0.2044(6) 0.093(4) 0.57(2) Uiso DUP .
F36 F 0.2234(5) 0.8326(8) 0.1190(8) 0.087(4) 0.57(2) Uiso DUP .
F37 F 0.1334(7) 0.2941(11) 0.4115(11) 0.111(5) 0.417(12) Uiso DUP .
F38 F 0.1739(7) 0.2555(14) 0.3627(12) 0.129(5) 0.417(12) Uiso DUP .
F39 F 0.1214(7) 0.2815(12) 0.321(1) 0.115(5) 0.417(12) Uiso DUP .
F40 F 0.1474(4) 0.2594(9) 0.3184(7) 0.104(4) 0.583(12) Uiso DUP .
F41 F 0.1671(5) 0.272(1) 0.4046(9) 0.127(4) 0.583(12) Uiso DUP .
F42 F 0.1152(5) 0.3022(9) 0.3890(8) 0.122(4) 0.583(12) Uiso DUP .
F43 F 0.1957(6) 0.6429(13) 0.4422(7) 0.108(4) 0.492(16) Uiso DUP .
F44 F 0.1743(6) 0.5561(11) 0.4961(8) 0.104(4) 0.492(16) Uiso DUP .
F45 F 0.1430(7) 0.6548(13) 0.4403(8) 0.123(5) 0.492(16) Uiso DUP .
F46 F 0.1560(6) 0.5755(11) 0.4887(8) 0.108(4) 0.508(16) Uiso DUP .
F47 F 0.2104(6) 0.6102(13) 0.4570(8) 0.114(4) 0.508(16) Uiso DUP .
F48 F 0.1632(6) 0.6730(13) 0.4298(7) 0.118(4) 0.508(16) Uiso DUP .
H51 H 0.5508 0.5335 0.5646 0.0391 1.0000 Uiso . .
H61 H 0.4876 0.8284 0.4992 0.0720 1.0000 Uiso . .
H62 H 0.4491 0.8062 0.4688 0.0720 1.0000 Uiso . .
H71 H 0.4649 0.8061 0.5913 0.0840 1.0000 Uiso . .

```

H72 H 0.4264 0.7957 0.5579 0.0840 1.0000 Uiso . .
H81 H 0.4618 0.9470 0.5388 0.0963 1.0000 Uiso . .
H82 H 0.4202 0.9308 0.5433 0.0963 1.0000 Uiso . .
H91 H 0.4685 0.9337 0.6423 0.0971 0.4825 Uiso . .
H92 H 0.4261 0.9307 0.6430 0.0971 0.4825 Uiso . .
H101 H 0.4676 1.0717 0.6131 0.0980 0.4825 Uiso . .
H102 H 0.4254 1.0701 0.6002 0.0980 0.4825 Uiso . .
H111 H 0.4405 1.1485 0.6851 0.1013 0.4825 Uiso . .
H112 H 0.4605 1.0659 0.7144 0.1013 0.4825 Uiso . .
H113 H 0.4183 1.0643 0.7015 0.1013 0.4825 Uiso . .
H1091 H 0.4675 1.0313 0.5659 0.1331 0.5175 Uiso . .
H1092 H 0.4867 0.9614 0.5262 0.1331 0.5175 Uiso . .
H1101 H 0.4881 0.9560 0.6490 0.1373 0.5175 Uiso . .
H1102 H 0.5106 0.8970 0.6065 0.1373 0.5175 Uiso . .
H1111 H 0.5462 1.0004 0.6531 0.1425 0.5175 Uiso . .
H1112 H 0.5211 1.0755 0.6251 0.1425 0.5175 Uiso . .
H1113 H 0.5436 1.0165 0.5825 0.1425 0.5175 Uiso . .
H131 H 0.4197 0.6506 0.6143 0.0603 1.0000 Uiso . .
H141 H 0.4737 0.6620 0.6726 0.0591 1.0000 Uiso . .
H151 H 0.5091 0.5416 0.6944 0.0598 1.0000 Uiso . .
H181 H 0.5006 0.2523 0.6420 0.0566 1.0000 Uiso . .
H191 H 0.4477 0.2412 0.5840 0.0591 1.0000 Uiso . .
H201 H 0.4126 0.3607 0.5618 0.0541 1.0000 Uiso . .
H221 H 0.3641 0.6059 0.5872 0.0637 1.0000 Uiso . .
H222 H 0.3860 0.6234 0.5295 0.0637 1.0000 Uiso . .
H231 H 0.3255 0.5968 0.5053 0.0822 1.0000 Uiso . .
H232 H 0.3309 0.5011 0.5322 0.0822 1.0000 Uiso . .
H241 H 0.3846 0.4186 0.4733 0.1072 1.0000 Uiso . .
H242 H 0.3431 0.3976 0.4735 0.1072 1.0000 Uiso . .
H251 H 0.3658 0.3440 0.3885 0.1124 1.0000 Uiso . .
H252 H 0.3374 0.4189 0.3720 0.1124 1.0000 Uiso . .
H261 H 0.3909 0.3766 0.2958 0.1020 1.0000 Uiso . .
H262 H 0.3606 0.4487 0.2863 0.1020 1.0000 Uiso . .
H271 H 0.4333 0.4794 0.2930 0.1165 1.0000 Uiso . .
H272 H 0.4074 0.4856 0.2338 0.1165 1.0000 Uiso . .
H281 H 0.4047 0.6940 0.2492 0.0833 1.0000 Uiso . .
H282 H 0.4285 0.6186 0.2234 0.0833 1.0000 Uiso . .
H291 H 0.4632 0.7225 0.2762 0.0693 1.0000 Uiso . .
H292 H 0.4436 0.6981 0.3351 0.0693 1.0000 Uiso . .
H311 H 0.1371 0.4297 0.1616 0.0432 1.0000 Uiso . .
H331 H 0.2064 0.2744 0.0809 0.0594 1.0000 Uiso . .
H351 H 0.2420 0.4782 0.1823 0.0508 1.0000 Uiso . .
H371 H 0.1206 0.6232 0.2708 0.0460 1.0000 Uiso . .
H391 H 0.0637 0.7147 0.1247 0.0679 1.0000 Uiso . .
H411 H 0.1553 0.5824 0.1064 0.0575 1.0000 Uiso . .
H431 H 0.2453 0.5392 0.2791 0.0690 1.0000 Uiso . .
H451 H 0.2896 0.7682 0.2389 0.0984 1.0000 Uiso . .
H471 H 0.1890 0.7110 0.1771 0.0540 1.0000 Uiso . .
H491 H 0.1566 0.3892 0.2627 0.0589 1.0000 Uiso . .
H511 H 0.1517 0.4360 0.4409 0.0874 1.0000 Uiso . .
H531 H 0.1815 0.6263 0.3314 0.0684 1.0000 Uiso . .
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Na1 0.051(2) 0.046(2) 0.047(2) -0.0101(17) -0.0059(16) 0.0111(17)
N1 0.043(4) 0.031(4) 0.041(4) -0.001(3) 0.002(3) 0.004(3)

```


C1 0.041(5) 0.045(5) 0.034(5) 0.004(4) -0.001(4) 0.004(4)
O1 0.038(3) 0.053(4) 0.048(4) 0.001(3) -0.008(3) 0.010(3)
C2 0.024(4) 0.040(5) 0.027(4) -0.003(4) 0.001(3) 0.002(4)
C3 0.029(4) 0.035(5) 0.029(4) -0.001(3) -0.001(3) -0.009(4)
C4 0.041(5) 0.031(5) 0.040(5) -0.005(4) 0.006(4) -0.001(4)
O2 0.046(4) 0.040(4) 0.071(5) -0.013(3) -0.013(3) -0.014(3)
C5 0.025(4) 0.040(5) 0.033(4) -0.007(4) -0.001(3) -0.003(4)
C6 0.078(6) 0.036(4) 0.065(5) 0.005(4) -0.003(4) 0.011(4)
C7 0.089(5) 0.046(4) 0.075(5) 0.001(4) 0.004(4) 0.016(4)
C8 0.102(6) 0.053(4) 0.085(5) -0.002(4) 0.005(5) 0.022(4)
C12 0.042(5) 0.066(7) 0.032(5) 0.015(5) 0.013(4) 0.012(5)
C13 0.064(7) 0.050(6) 0.036(5) -0.009(4) 0.010(5) 0.008(5)
C14 0.051(6) 0.044(6) 0.053(6) -0.013(5) 0.013(5) 0.004(5)
C15 0.046(5) 0.072(7) 0.031(5) -0.006(5) 0.014(4) 0.006(5)
C16 0.043(5) 0.057(6) 0.043(5) -0.005(5) 0.009(4) 0.008(5)
C17 0.037(5) 0.055(6) 0.040(5) 0.002(4) 0.009(4) 0.008(4)
C18 0.041(5) 0.050(6) 0.051(6) 0.008(5) 0.011(4) 0.000(5)
C19 0.050(6) 0.036(5) 0.062(6) 0.006(5) 0.014(5) 0.001(4)
C20 0.041(5) 0.050(6) 0.043(5) 0.018(4) 0.005(4) -0.011(4)
C21 0.036(5) 0.043(5) 0.031(4) -0.002(4) 0.003(4) -0.003(4)
O3 0.049(4) 0.050(4) 0.068(5) 0.010(3) -0.006(3) 0.002(3)
C22 0.055(6) 0.056(6) 0.049(6) 0.011(5) 0.011(5) 0.012(5)
C23 0.038(6) 0.085(8) 0.082(9) 0.022(7) 0.004(6) 0.008(6)
O4 0.059(4) 0.054(5) 0.080(5) 0.004(4) -0.016(4) 0.007(4)
C24 0.102(11) 0.056(8) 0.110(12) 0.011(8) -0.032(9) -0.011(7)
C25 0.094(11) 0.055(8) 0.132(14) -0.003(8) -0.03(1) -0.024(8)
O5 0.061(5) 0.050(5) 0.123(8) -0.027(5) -0.034(5) 0.013(4)
C26 0.058(8) 0.09(1) 0.107(11) -0.062(9) -0.019(7) 0.015(7)
C27 0.072(9) 0.112(12) 0.107(11) -0.06(1) 0.005(8) 0.013(9)
O6 0.040(4) 0.080(5) 0.063(5) -0.029(4) -0.002(3) 0.013(4)
C28 0.052(6) 0.11(1) 0.045(6) -0.001(6) 0.005(5) 0.022(7)
C29 0.058(6) 0.077(8) 0.038(5) 0.014(5) 0.011(5) 0.020(6)
O7 0.043(4) 0.078(5) 0.057(4) -0.013(4) -0.007(3) 0.017(4)
B1 0.030(5) 0.034(5) 0.040(5) 0.003(4) -0.004(4) 0.003(4)
C30 0.035(3) 0.035(3) 0.030(3) -0.006(3) 0.002(2) -0.008(3)
C31 0.039(3) 0.028(3) 0.041(3) 0.003(3) -0.001(3) -0.003(3)
C32 0.044(3) 0.040(4) 0.032(4) 0.004(3) 0.005(3) -0.011(3)
C33 0.054(4) 0.031(4) 0.064(5) -0.007(3) 0.017(3) -0.007(3)
C34 0.072(4) 0.045(4) 0.051(4) -0.012(4) 0.010(4) -0.016(4)
C35 0.035(3) 0.048(4) 0.044(4) 0.002(3) 0.011(3) -0.004(3)
C36 0.027(3) 0.043(3) 0.032(3) -0.008(3) -0.002(2) 0.012(3)
C37 0.048(3) 0.033(3) 0.035(3) -0.003(3) 0.007(3) 0.001(3)
C38 0.042(3) 0.046(4) 0.058(4) 0.003(3) -0.010(3) 0.001(3)
C39 0.051(4) 0.053(4) 0.066(5) -0.008(3) -0.016(3) 0.018(3)
C40 0.071(4) 0.054(4) 0.070(4) -0.019(4) 0.009(4) 0.031(4)
C41 0.042(3) 0.057(4) 0.044(4) -0.005(3) -0.007(3) 0.017(3)
C42 0.023(3) 0.045(4) 0.054(4) -0.012(3) 0.001(3) 0.010(3)
C43 0.052(4) 0.032(4) 0.088(4) -0.019(3) -0.033(3) 0.002(3)
C44 0.080(5) 0.035(4) 0.139(6) -0.030(4) -0.022(4) 0.002(4)
C45 0.036(5) 0.045(5) 0.165(5) -0.020(5) -0.014(4) -0.009(4)
C46 0.030(4) 0.038(4) 0.118(5) -0.023(3) -0.008(4) 0.005(4)
C47 0.036(4) 0.044(4) 0.055(4) -0.005(3) -0.006(3) 0.003(3)
C48 0.035(3) 0.052(4) 0.035(4) -0.011(3) -0.003(3) 0.004(3)
C49 0.046(4) 0.058(4) 0.043(4) -0.006(3) 0.002(3) -0.007(3)
C50 0.055(5) 0.109(4) 0.048(6) -0.003(4) 0.001(4) -0.005(4)
C51 0.086(5) 0.095(5) 0.037(5) -0.007(5) 0.015(4) -0.023(4)
C52 0.085(4) 0.130(4) 0.043(5) 0.019(3) 0.013(4) -0.001(4)
C53 0.072(4) 0.066(4) 0.033(4) -0.010(3) 0.004(3) 0.003(3)
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N1 . C1 . 1.360(12) yes
C1 . C2 . 1.500(12) yes
C1 . O1 . 1.22(1) yes
C2 . C3 . 1.418(11) yes
C2 . C5 2_666 1.363(12) yes
C3 . C5 . 1.367(12) yes
C3 . C4 . 1.508(12) yes
C4 . O2 . 1.19(1) yes
C5 . H51 . 1.000 no
C6 . H62 . 1.000 no
C6 . H61 . 1.000 no
C6 . C7 . 1.486(12) yes
C7 . H72 . 1.000 no
C7 . H71 . 1.000 no
C7 . C8 . 1.492(12) yes
C8 . H82 . 1.000 no
C8 . H81 . 1.000 no
C8 . C109 . 1.456(17) yes
C8 . C9 . 1.498(15) yes
C9 . H92 . 1.000 no
C9 . H91 . 1.000 no
C9 . C10 . 1.506(17) yes
C10 . H102 . 1.000 no
C10 . H101 . 1.000 no
C10 . C11 . 1.509(18) yes
C11 . H113 . 1.000 no
C11 . H112 . 1.000 no
C11 . H111 . 1.000 no
C109 . H1092 . 1.000 no
C109 . H1091 . 1.000 no
C109 . H81 . 0.823 no
C109 . C110 . 1.457(18) yes
C110 . H1102 . 1.000 no
C110 . H1101 . 1.000 no
C110 . C111 . 1.509(19) yes
C111 . H1113 . 1.000 no
C111 . H1112 . 1.000 no
C111 . H1111 . 1.000 no
C12 . O3 . 1.359(12) yes
C12 . C21 . 1.408(13) yes
C12 . C13 . 1.374(14) yes
C13 . H131 . 1.000 no
C13 . C14 . 1.412(14) yes
C14 . H141 . 1.000 no
C14 . C15 . 1.373(14) yes
C15 . H151 . 1.000 no
C15 . C16 . 1.427(15) yes
C16 . C21 . 1.430(13) yes
C16 . C17 . 1.431(13) yes
C17 . C18 . 1.339(14) yes
C17 . O7 2_666 1.399(12) yes
C18 . H181 . 1.000 no
C18 . C19 . 1.384(14) yes
C19 . H191 . 1.000 no

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C19 . C20 . 1.362(14) yes
C20 . H201 . 1.000 no
C20 . C21 . 1.417(13) yes
O3 . C22 . 1.431(12) yes
C22 . H222 . 1.000 no
C22 . H221 . 1.000 no
C22 . C23 . 1.495(17) yes
C23 . H232 . 1.000 no
C23 . H231 . 1.000 no
C23 . O4 . 1.432(15) yes
O4 . C24 . 1.441(16) yes
C24 . H242 . 1.000 no
C24 . H241 . 1.000 no
C24 . C25 . 1.47(2) yes
C25 . H252 . 1.000 no
C25 . H251 . 1.000 no
C25 . O5 . 1.391(18) yes
O5 . C26 . 1.416(17) yes
C26 . H262 . 1.000 no
C26 . H261 . 1.000 no
C26 . C27 . 1.45(2) yes
C27 . H272 . 1.000 no
C27 . H271 . 1.000 no
C27 . O6 . 1.472(17) yes
O6 . C28 . 1.437(16) yes
C28 . H282 . 1.000 no
C28 . H281 . 1.000 no
C28 . C29 . 1.496(17) yes
C29 . H292 . 1.000 no
C29 . H291 . 1.000 no
C29 . O7 . 1.419(12) yes
B1 . C48 . 1.636(12) yes
B1 . C42 . 1.645(11) yes
B1 . C36 . 1.649(11) yes
B1 . C30 . 1.650(12) yes
C30 . C35 . 1.399(8) yes
C30 . C31 . 1.393(8) yes
C31 . H311 . 1.000 no
C31 . C32 . 1.415(9) yes
C32 . C54 . 1.503(9) yes
C32 . C33 . 1.359(9) yes
C33 . H331 . 1.000 no
C33 . C34 . 1.40(1) yes
C34 . C55 . 1.491(11) yes
C34 . C35 . 1.380(9) yes
C35 . H351 . 1.000 no
C36 . C41 . 1.373(9) yes
C36 . C37 . 1.417(8) yes
C37 . H371 . 1.000 no
C37 . C38 . 1.380(9) yes
C38 . C56 . 1.492(11) yes
C38 . C39 . 1.38(1) yes
C39 . H391 . 1.000 no
C39 . C40 . 1.36(1) yes
C40 . C57 . 1.489(12) yes
C40 . C41 . 1.41(1) yes
C41 . H411 . 1.000 no
C42 . C47 . 1.37(1) yes
C42 . C43 . 1.417(9) yes
C43 . H431 . 1.000 no
C43 . C44 . 1.378(11) yes

C44 . C58 . 1.492(12) yes
 C44 . C45 . 1.360(12) yes
 C45 . H451 . 1.000 no
 C45 . C46 . 1.397(12) yes
 C46 . C59 . 1.469(12) yes
 C46 . C47 . 1.405(11) yes
 C47 . H471 . 1.000 no
 C48 . C53 . 1.421(9) yes
 C48 . C49 . 1.40(1) yes
 C49 . H491 . 1.000 no
 C49 . C50 . 1.427(12) yes
 C50 . C60 . 1.491(13) yes
 C50 . C51 . 1.373(11) yes
 C51 . H511 . 1.000 no
 C51 . C52 . 1.382(11) yes
 C52 . C61 . 1.500(13) yes
 C52 . C53 . 1.361(11) yes
 C53 . H531 . 1.000 no
 C60 . F42 . 1.33(2) yes
 C60 . F41 . 1.35(2) yes
 C60 . F40 . 1.315(18) yes
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 C6 . N1 . C1 . 125.6(8) yes
 C4 . N1 . C1 . 113.8(7) yes
 C2 . C1 . O1 . 127.3(8) yes
 C2 . C1 . N1 . 106.6(7) yes
 O1 . C1 . N1 . 126.0(8) yes
 C3 . C2 . C5 2_666 121.8(7) yes
 C3 . C2 . C1 . 107.1(7) yes
 C5 2_666 C2 . C1 . 131.1(7) yes
 C5 . C3 . C4 . 130.2(7) yes
 C5 . C3 . C2 . 122.4(7) yes
 C4 . C3 . C2 . 107.4(7) yes
 O2 . C4 . N1 . 126.9(8) yes
 O2 . C4 . C3 . 128.1(8) yes
 N1 . C4 . C3 . 105.0(7) yes
 H51 . C5 . C2 2_666 122.101 no
 H51 . C5 . C3 . 122.103 no
 C2 2_666 C5 . C3 . 115.8(7) yes
 H62 . C6 . H61 . 109.472 no
 H62 . C6 . C7 . 107.830 no
 H61 . C6 . C7 . 107.820 no
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 H61 . C6 . N1 . 107.815 no
 C7 . C6 . N1 . 116.0(2) yes
 H72 . C7 . H71 . 109.458 no
 H72 . C7 . C8 . 107.768 no
 H71 . C7 . C8 . 107.781 no
 H72 . C7 . C6 . 107.765 no

H71 . C7 . C6 . 107.787 no
C8 . C7 . C6 . 116.2(2) yes
H82 . C8 . H81 . 109.470 no
H82 . C8 . C109 . 128.597 no
H81 . C8 . C109 . 32.973 no
H82 . C8 . C9 . 107.772 no
H81 . C8 . C9 . 107.803 no
C109 . C8 . C9 . 75.9(13) yes
H82 . C8 . C7 . 107.772 no
H81 . C8 . C7 . 107.789 no
C109 . C8 . C7 . 116.2(2) yes
C9 . C8 . C7 . 116.1(2) yes
H92 . C9 . H91 . 109.460 no
H92 . C9 . C10 . 107.774 no
H91 . C9 . C10 . 107.767 no
H92 . C9 . C8 . 107.789 no
H91 . C9 . C8 . 107.767 no
C10 . C9 . C8 . 116.2(2) yes
H102 . C10 . H101 . 109.463 no
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H101 . C10 . C9 . 107.792 no
C11 . C10 . C9 . 116.1(2) yes
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H81 . C109 . C110 . 143.290 no
H1092 . C109 . C8 . 107.782 no
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H81 . C109 . C8 . 41.409 no
C110 . C109 . C8 . 116.1(2) yes
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H1102 . C110 . C109 . 107.821 no
H1101 . C110 . C109 . 107.813 no
C111 . C110 . C109 . 116.1(2) yes
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H1112 . C111 . H1111 . 109.439 no
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H1112 . C111 . C110 . 109.477 no
H1111 . C111 . C110 . 109.489 no
O3 . C12 . C21 . 114.7(9) yes
O3 . C12 . C13 . 123.0(9) yes
C21 . C12 . C13 . 122.3(9) yes
H131 . C13 . C14 . 120.692 no
H131 . C13 . C12 . 120.690 no
C14 . C13 . C12 . 118.6(9) yes
H141 . C14 . C15 . 118.850 no
H141 . C14 . C13 . 118.849 no
C15 . C14 . C13 . 122.3(9) yes

H151 . C15 . C16 . 120.611 no
H151 . C15 . C14 . 120.614 no
C16 . C15 . C14 . 118.8(9) yes
C21 . C16 . C17 . 117.3(9) yes
C21 . C16 . C15 . 120.0(9) yes
C17 . C16 . C15 . 122.7(9) yes
C18 . C17 . 07_2_666 126.5(9) yes
C18 . C17 . C16 . 121.6(9) yes
07_2_666 C17 . C16 . 111.8(8) yes
H181 . C18 . C19 . 119.867 no
H181 . C18 . C17 . 119.873 no
C19 . C18 . C17 . 120.3(9) yes
H191 . C19 . C20 . 118.936 no
H191 . C19 . C18 . 118.936 no
C20 . C19 . C18 . 122.1(9) yes
H201 . C20 . C21 . 120.362 no
H201 . C20 . C19 . 120.364 no
C21 . C20 . C19 . 119.3(9) yes
C12 . C21 . C20 . 122.5(8) yes
C12 . C21 . C16 . 118.0(8) yes
C20 . C21 . C16 . 119.4(8) yes
C22 . O3 . C12 . 119.7(8) yes
H222 . C22 . H221 . 109.470 no
H222 . C22 . C23 . 109.714 no
H221 . C22 . C23 . 109.713 no
H222 . C22 . O3 . 109.712 no
H221 . C22 . O3 . 109.711 no
C23 . C22 . O3 . 108.5(9) yes
H232 . C23 . H231 . 109.467 no
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C25 . C24 . O4 . 108.7(11) yes
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H251 . C25 . C24 . 109.522 no
O5 . C25 . C24 . 109.2(11) yes
C26 . O5 . C25 . 111.4(11) yes
H262 . C26 . H261 . 109.471 no
H262 . C26 . C27 . 109.693 no
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H261 . C26 . O5 . 109.706 no
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H271 . C27 . O6 . 109.267 no
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H271 . C27 . C26 . 109.267 no
O6 . C27 . C26 . 110.3(10) yes
C28 . O6 . C27 . 115.2(10) yes

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H282 . C28 . C29 . 109.222 no
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H282 . C28 . O6 . 109.224 no
H281 . C28 . O6 . 109.219 no
C29 . C28 . O6 . 110.5(9) yes
H292 . C29 . H291 . 109.461 no
H292 . C29 . O7 . 110.122 no
H291 . C29 . O7 . 110.116 no
H292 . C29 . C28 . 110.121 no
H291 . C29 . C28 . 110.118 no
O7 . C29 . C28 . 106.9(10) yes
C17 2_666 O7 . C29 . 115.7(8) yes
C48 . B1 . C42 . 108.1(6) yes
C48 . B1 . C36 . 110.4(6) yes
C42 . B1 . C36 . 108.9(6) yes
C48 . B1 . C30 . 109.2(6) yes
C42 . B1 . C30 . 111.0(6) yes
C36 . B1 . C30 . 109.3(6) yes
C35 . C30 . C31 . 115.3(5) yes
C35 . C30 . B1 . 124.9(6) yes
C31 . C30 . B1 . 119.8(5) yes
H311 . C31 . C32 . 119.009 no
H311 . C31 . C30 . 119.008 no
C32 . C31 . C30 . 122.0(6) yes
C54 . C32 . C33 . 122.3(6) yes
C54 . C32 . C31 . 116.6(5) yes
C33 . C32 . C31 . 121.1(6) yes
H331 . C33 . C34 . 120.935 no
H331 . C33 . C32 . 120.935 no
C34 . C33 . C32 . 118.1(6) yes
C55 . C34 . C35 . 121.5(6) yes
C55 . C34 . C33 . 118.2(6) yes
C35 . C34 . C33 . 120.4(7) yes
H351 . C35 . C34 . 118.445 no
H351 . C35 . C30 . 118.444 no
C34 . C35 . C30 . 123.1(6) yes
C41 . C36 . C37 . 116.3(5) yes
C41 . C36 . B1 . 121.5(6) yes
C37 . C36 . B1 . 122.2(6) yes
H371 . C37 . C38 . 119.586 no
H371 . C37 . C36 . 119.587 no
C38 . C37 . C36 . 120.8(6) yes
C56 . C38 . C39 . 118.2(6) yes
C56 . C38 . C37 . 119.6(5) yes
C39 . C38 . C37 . 122.2(6) yes
H391 . C39 . C40 . 121.268 no
H391 . C39 . C38 . 121.267 no
C40 . C39 . C38 . 117.5(6) yes
C57 . C40 . C41 . 119.7(6) yes
C57 . C40 . C39 . 119.0(7) yes
C41 . C40 . C39 . 121.3(7) yes
H411 . C41 . C36 . 119.114 no
H411 . C41 . C40 . 119.117 no
C36 . C41 . C40 . 121.8(6) yes
C47 . C42 . C43 . 114.8(6) yes
C47 . C42 . B1 . 124.1(6) yes
C43 . C42 . B1 . 120.9(6) yes
H431 . C43 . C44 . 119.613 no
H431 . C43 . C42 . 119.613 no
C44 . C43 . C42 . 120.8(7) yes

C58 . C44 . C45 . 118.3(7) yes
C58 . C44 . C43 . 118.3(7) yes
C45 . C44 . C43 . 123.2(8) yes
H451 . C45 . C46 . 120.989 no
H451 . C45 . C44 . 120.989 no
C46 . C45 . C44 . 118.0(7) yes
C59 . C46 . C47 . 120.3(6) yes
C59 . C46 . C45 . 121.7(7) yes
C47 . C46 . C45 . 118.0(7) yes
H471 . C47 . C42 . 117.500 no
H471 . C47 . C46 . 117.500 no
C42 . C47 . C46 . 125.0(6) yes
C53 . C48 . C49 . 115.1(6) yes
C53 . C48 . B1 . 121.7(6) yes
C49 . C48 . B1 . 123.0(6) yes
H491 . C49 . C50 . 119.181 no
H491 . C49 . C48 . 119.182 no
C50 . C49 . C48 . 121.6(6) yes
C60 . C50 . C51 . 119.3(7) yes
C60 . C50 . C49 . 120.2(6) yes
C51 . C50 . C49 . 120.5(8) yes
H511 . C51 . C52 . 121.001 no
H511 . C51 . C50 . 120.999 no
C52 . C51 . C50 . 118.0(8) yes
C61 . C52 . C53 . 118.5(6) yes
C61 . C52 . C51 . 119.4(7) yes
C53 . C52 . C51 . 122.1(8) yes
H531 . C53 . C52 . 118.779 no
H531 . C53 . C48 . 118.776 no
C52 . C53 . C48 . 122.4(7) yes
F42 . C60 . F41 . 101.1(11) yes
F42 . C60 . F40 . 113.0(11) yes
F41 . C60 . F40 . 102.5(11) yes
F42 . C60 . F39 . 73.9(12) yes
F41 . C60 . F39 . 135.4(10) yes
F40 . C60 . F39 . 46.4(10) yes
F42 . C60 . F38 . 132.2(11) yes
F41 . C60 . F38 . 44.6(11) yes
F40 . C60 . F38 . 61.9(12) yes
F39 . C60 . F38 . 106.4(12) yes
F42 . C60 . F37 . 38.7(11) yes
F41 . C60 . F37 . 63.8(12) yes
F40 . C60 . F37 . 129.2(10) yes
F39 . C60 . F37 . 107.8(12) yes
F38 . C60 . F37 . 104.0(13) yes
F42 . C60 . C50 . 111.3(8) yes
F41 . C60 . C50 . 112.9(8) yes
F40 . C60 . C50 . 115.0(8) yes
F39 . C60 . C50 . 109.9(8) yes
F38 . C60 . C50 . 113.0(8) yes
F37 . C60 . C50 . 115.2(8) yes
C109 . H81 . C8 . 105.617 no
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