Cation-reinforced donor-acceptor pseudorotaxanes

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

Published in:
New Journal of Chemistry

DOI:
10.1039/b415418e

```
# Supplementary Material (ESI) for New Journal of Chemistry
# This journal is © The Royal Society of Chemistry and
# The Centre National de la Recherche Scientifique, 2004



data_global
_journal_name_full               'New J.Chem.(Nouv.J.Chim.)'

_journal_coden_Cambridge         0440

_audit_creation_method           CRYSTALS_ver_12-03-99

_publ_contact_author_name        'Jeremy K.M Sanders'
_publ_contact_author_address
;
Department of Chemistry
University of Cambridge
Lensfield Road
Cambridge
CB2 1EW
UK
;
_publ_contact_author_email       JKMS@CAM.AC.UK

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

data_1
_database_code_depnum_ccdc_archive 'CCDC 254110'

_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                   15.8820(3)
_cell_angle_alpha                90
_cell_length_b                   10.2424(2)
_cell_angle_beta                 94.5550(7)
_cell_length_c                   26.2432(6)
_cell_angle_gamma                90
_cell_volume                     4255.49(15)

_symmetry_cell_setting           Monoclinic
_symmetry_space_group_name_H-M   'P 1 21/c 1 '
loop_
_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 .

loop_
_atom_type_symbol
_atom_type_scat_dispersion_real
_atom_type_scat_dispersion_imag
_atom_type_scat_Cromer_Mann_a1
_atom_type_scat_Cromer_Mann_b1
_atom_type_scat_Cromer_Mann_a2
_atom_type_scat_Cromer_Mann_b2
_atom_type_scat_Cromer_Mann_a3
_atom_type_scat_Cromer_Mann_b3
_atom_type_scat_Cromer_Mann_a4
_atom_type_scat_Cromer_Mann_b4
_atom_type_scat_Cromer_Mann_c
_atom_type_scat_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
'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
'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

_cell_formula_units_Z            2
_chemical_formula_sum            ' C63 H81 Br2 Cl15 N2 O16 '
_chemical_formula_moiety
;
C36 H44 O10, C22 H28 N2 O4, 5(C H2 Cl3), 2(H2 O), 2Br
;
_chemical_compound_source
;
?
;
_chemical_formula_weight         1827.73

_cell_measurement_reflns_used    24872
_cell_measurement_theta_min      5
_cell_measurement_theta_max      25
_cell_measurement_temperature    180

_exptl_crystal_description       ' block '
_exptl_crystal_colour            ' orange '
_exptl_crystal_size_min          0.07
_exptl_crystal_size_mid          0.12
_exptl_crystal_size_max          0.18

_exptl_crystal_density_diffrn    1.426
_exptl_crystal_density_meas      ?
# Non-dispersive F(000):
_exptl_crystal_F_000             1864
_exptl_absorpt_coefficient_mu    1.481
_diffrn_measurement_device_type
;
Nonius Kappa CCD
;
_diffrn_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
;
SIR97 (Altomare et al, 1999)
```

```
;

_diffrn_measurement_method       \w

_exptl_absorpt_correction_type   multi-scan
_exptl_absorpt_correction_T_min  0.766
_exptl_absorpt_correction_T_max  0.875
_exptl_absorpt_process_details
;
multi-scan from symmetry-related measurements
Sortav (Blessing 1995)
;

_diffrn_standards_interval_time  0
_diffrn_standards_interval_count 0
_diffrn_standards_number         0
_diffrn_standards_decay_%        0.00

_diffrn_ambient_temperature      180
_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

_diffrn_reflns_theta_min         5.151
_diffrn_reflns_theta_max         25.347
_diffrn_measured_fraction_theta_max 0.988

_diffrn_reflns_theta_full        24.586
_diffrn_measured_fraction_theta_full 0.990

_diffrn_reflns_limit_h_min       -19
_diffrn_reflns_limit_h_max       19
_diffrn_reflns_limit_k_min       -12
_diffrn_reflns_limit_k_max       12
_diffrn_reflns_limit_l_min       -22
_diffrn_reflns_limit_l_max       31
_reflns_limit_h_min              -19
_reflns_limit_h_max              19
_reflns_limit_k_min              0
_reflns_limit_k_max              12
_reflns_limit_l_min              0
_reflns_limit_l_max              31

_refine_diff_density_min         -1.31
_refine_diff_density_max         1.25

_refine_ls_number_reflns         3794
_refine_ls_number_restraints     204
_refine_ls_number_parameters     541

#_refine_ls_R_factor_ref 0.1000
_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:
_reflns_threshold_expression     I>3.00u(I)
_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
;
_diffrn_radiation_type           'Mo K\a'
_diffrn_radiation_wavelength     0.71073

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

SIR97 (Altomare et al, 1999)

Betteridge, P.W., Carruthers, J.R., Cooper, R.I.,
Prout, K., Watkin, D.J. (2003). J. Appl. Cryst. 36, 1487.

Nonius BV, COLLECT Software, 1997-2001

Otwinowski, Z. & Minor, W. (1996), Processing of X-ray
Diffraction Data Collected in Oscillation Mode. Methods Enzymol.
276, 1997, 307-326. Ed Carter, C.W. & Sweet, R.M., Academic Press.

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

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

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

# Uequiv = arithmetic mean of Ui
# i.e. Ueqiv = (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
Li1 Li 0.8192(11) -0.1927(16) 0.0558(7) 0.0585 1.0000 Uani . . . . . .
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 .
```

```
Cl4 Cl 0.8922(5) 0.0578(7) -0.1010(3) 0.0900 0.5000 Uani D U . 3 1 .
Cl5 Cl 0.8981(6) 0.3355(7) -0.1151(3) 0.0896 0.5000 Uani D U . 3 1 .
Cl6 Cl 0.7567(5) 0.2163(9) -0.0714(4) 0.0863 0.5000 Uani D U . 3 1 .
Cl104 Cl 0.7576(5) 0.2401(8) -0.0714(4) 0.0858 0.5000 Uani D U . 3 2 .
Cl105 Cl 0.8781(6) 0.0723(7) -0.1157(3) 0.0905 0.5000 Uani D U . 3 2 .
Cl106 Cl 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 . . . .
Cl107 Cl 0.6154(10) 0.0492(18) 0.3374(6) 0.2108 0.5000 Uani D U . . . .
Cl108 Cl 0.5375(9) -0.1489(18) 0.2750(6) 0.2095 0.5000 Uani D U . . . .
Cl109 Cl 0.7021(9) -0.061(2) 0.2570(6) 0.2112 0.5000 Uani D U . . . .
H1 H 0.9188 -0.0038 0.0289 0.0500 1.0000 Uiso . . . . . .
H2 H 0.8992 -0.1041 -0.0121 0.0819 1.0000 Uiso . . . . . .
H21 H 0.7152 0.2946 0.0537 0.0737 1.0000 Uiso . . . . . .
H31 H 0.6000 0.4305 0.0172 0.0813 1.0000 Uiso . . . . . .
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 . . . . . .
H71 H 0.4354 -0.0406 0.1728 0.0871 1.0000 Uiso . . . . . .
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 . . . . . .
H112 H 0.7842 0.0905 0.0741 0.0735 1.0000 Uiso . . . . . .
H121 H 0.8962 0.0610 0.1426 0.0709 1.0000 Uiso . . . . . .
H122 H 0.8210 0.0474 0.1813 0.0709 1.0000 Uiso . . . . . .
H131 H 0.9475 -0.1482 0.1532 0.0755 1.0000 Uiso . . . . . .
H132 H 0.8852 -0.1559 0.1997 0.0755 1.0000 Uiso . . . . . .
H141 H 0.9182 -0.3722 0.1691 0.0813 1.0000 Uiso . . . . . .
H142 H 0.8169 -0.3435 0.1616 0.0813 1.0000 Uiso . . . . . .
H151 H 0.9050 -0.5209 0.0957 0.0787 1.0000 Uiso . . . . . .
H152 H 0.8037 -0.4911 0.0886 0.0787 1.0000 Uiso . . . . . .
H161 H 0.9223 -0.4320 0.0142 0.0700 1.0000 Uiso . . . . . .
H162 H 0.8504 -0.5461 0.0081 0.0700 1.0000 Uiso . . . . . .
H171 H 0.2242 0.4494 0.0656 0.0807 1.0000 Uiso . . . . . .
H172 H 0.1422 0.3529 0.0640 0.0807 1.0000 Uiso . . . . . .
H181 H 0.2471 0.2486 0.1116 0.0836 1.0000 Uiso . . . . . .
H182 H 0.2377 0.1737 0.0565 0.0836 1.0000 Uiso . . . . . .
H231 H 0.6446 0.0475 -0.0205 0.0751 1.0000 Uiso . . . . . .
H241 H 0.6537 -0.3845 0.0900 0.2247 1.0000 Uiso . . . . . .
H242 H 0.5572 -0.4151 0.1038 0.2247 1.0000 Uiso . . . . . .
H251 H 0.6759 -0.2651 0.1653 0.2255 1.0000 Uiso . . . . . .
H252 H 0.5800 -0.2997 0.1792 0.2255 1.0000 Uiso . . . . . .
H261 H 0.7085 -0.4909 0.1638 0.2266 1.0000 Uiso . . . . . .
H262 H 0.6125 -0.5257 0.1774 0.2266 1.0000 Uiso . . . . . .
H271 H 0.7185 -0.3698 0.2427 0.2277 1.0000 Uiso . . . . . .
H272 H 0.6340 -0.4500 0.2559 0.2277 1.0000 Uiso . . . . . .
H281 H 0.7760 -0.5288 0.2867 0.2281 0.5000 Uiso . . . 1 1 .
H282 H 0.7015 -0.6320 0.2680 0.2281 0.5000 Uiso . . . 1 1 .
H291 H 0.8274 -0.6861 0.2323 0.2285 0.5000 Uiso . . . 1 1 .
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 .
H2902 H 0.8077 -0.6277 0.2589 0.2282 0.5000 Uiso . . . 1 2 .
H2903 H 0.7453 -0.6858 0.2118 0.2282 0.5000 Uiso . . . 1 2 .
H301 H 1.0031 0.2473 0.1475 0.1670 1.0000 Uiso . . . . . .
H311 H 0.8934 0.1889 -0.0399 0.1061 1.0000 Uiso . . . . . .
H321 H 0.5781 0.0542 0.2515 0.2538 0.5000 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 0.053(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)
```

```
Cl108 0.180(8) 0.256(12) 0.197(9) -0.004(8) 0.036(7) 0.001(8)
Cl109 0.180(8) 0.257(12) 0.200(9) -0.005(8) 0.036(7) 0.001(8)
_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
N1 . C19 . 1.409(18) yes
N1 . C22 . 1.391(16) yes
N1 . C24 . 1.39(2) yes
O1 . C1 . 1.375(12) yes
O1 . C11 . 1.419(13) yes
O2 . C12 . 1.412(13) yes
O2 . C13 . 1.419(12) yes
O3 . C14 . 1.419(13) yes
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
```

```
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 . Cl1 . 1.766(9) yes
C30 . Cl2 . 1.759(8) yes
C30 . Cl3 . 1.722(8) yes
C30 . H301 . 1.000 no
C30 . Cl101 . 1.766(8) yes
C30 . Cl102 . 1.758(8) yes
C30 . Cl103 . 1.757(9) yes
C30 . H301 . 1.000 no
C31 . Cl4 . 1.748(7) yes
C31 . Cl5 . 1.777(8) yes
C31 . Cl6 . 1.752(8) yes
C31 . H311 . 1.000 no
C31 . Cl104 . 1.766(8) yes
C31 . Cl105 . 1.775(7) yes
C31 . Cl106 . 1.739(8) yes
C31 . H311 . 1.000 no
C32 . Cl107 . 1.742(9) yes
C32 . Cl108 . 1.733(9) yes
C32 . Cl109 . 1.751(9) yes
C32 . H321 . 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
C19 . N1 . C22 . 111.8(12) yes
C19 . N1 . C24 . 125.7(14) yes
C22 . N1 . C24 . 122.4(17) yes
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
```

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
C17 . C18 . O5 . 110.0(10) yes
C17 . C18 . H181 . 109.334 no
O5 . C18 . H181 . 109.335 no
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
C28 . C29 . H291 . 109.963 no
C28 . C29 . H292 . 109.488 no
H291 . C29 . H292 . 109.461 no
C28 . C29 . H293 . 109.491 no
H291 . C29 . H293 . 109.441 no
H292 . C29 . H293 . 108.978 no
C27 . C280 . C290 . 113.6(19) yes
C27 . C280 . H2801 . 108.657 no
C290 . C280 . H2801 . 108.178 no
C27 . C280 . H2802 . 108.587 no
C290 . C280 . H2802 . 108.342 no
H2801 . C280 . H2802 . 109.419 no
C280 . C290 . H2901 . 109.940 no
C280 . C290 . H2902 . 109.644 no
H2901 . C290 . H2902 . 109.543 no
C280 . C290 . H2903 . 109.419 no
H2901 . C290 . H2903 . 109.272 no
H2902 . C290 . H2903 . 109.005 no
Cl1 . C30 . Cl2 . 109.16(10) yes
Cl1 . C30 . Cl3 . 109.14(10) yes
Cl2 . C30 . Cl3 . 109.07(10) yes
Cl1 . C30 . H301 . 117.137 no
Cl2 . C30 . H301 . 108.782 no
Cl3 . C30 . H301 . 103.217 no
Cl101 . C30 . Cl102 . 109.06(10) yes
Cl101 . C30 . Cl103 . 108.95(10) yes
Cl102 . C30 . Cl103 . 109.01(10) yes
Cl101 . C30 . H301 . 109.928 no
Cl102 . C30 . H301 . 109.877 no
Cl103 . C30 . H301 . 109.991 no
Cl4 . C31 . Cl5 . 109.04(10) yes
Cl4 . C31 . Cl6 . 109.11(10) yes
Cl5 . C31 . Cl6 . 109.04(10) yes
Cl4 . C31 . H311 . 95.035 no
Cl5 . C31 . H311 . 124.602 no
Cl6 . C31 . H311 . 108.612 no
Cl104 . C31 . Cl105 . 109.08(10) yes
Cl104 . C31 . Cl106 . 109.09(10) yes
Cl105 . C31 . Cl106 . 109.14(10) yes
Cl104 . C31 . H311 . 109.873 no
Cl105 . C31 . H311 . 109.825 no

```
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
#===END

########################################
## denoted [Li212][B(C6F5)4]2 in text ##
########################################

data_2
_database_code_depnum_ccdc_archive 'CCDC 254111'

_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                   10.7147(2)
_cell_angle_alpha                103.2331(7)
_cell_length_b                   13.4449(2)
_cell_angle_beta                 90.1735(7)
_cell_length_c                   19.5512(3)
_cell_angle_gamma                94.5532(7)
_cell_volume                     2732.42(8)

_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_scat_dispersion_real
_atom_type_scat_dispersion_imag
_atom_type_scat_Cromer_Mann_a1
_atom_type_scat_Cromer_Mann_b1
_atom_type_scat_Cromer_Mann_a2
_atom_type_scat_Cromer_Mann_b2
_atom_type_scat_Cromer_Mann_a3
_atom_type_scat_Cromer_Mann_b3
_atom_type_scat_Cromer_Mann_a4
_atom_type_scat_Cromer_Mann_b4
_atom_type_scat_Cromer_Mann_c
_atom_type_scat_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
'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

_cell_formula_units_Z            1
_chemical_formula_sum            ' C108 H76 B2 Cl4 F40 Li2 N2 O16 '
_chemical_formula_moiety         ' C108 H76 B2 Cl4 F40 Li2 N2 O16 '
_chemical_compound_source
;
?
;
_chemical_formula_weight         2595.03

_cell_measurement_reflns_used    20750
_cell_measurement_theta_min      5
_cell_measurement_theta_max      27
_cell_measurement_temperature    180

_exptl_crystal_description       ' block '
```

```
_exptl_crystal_colour            ' red '
_exptl_crystal_size_min          0.12
_exptl_crystal_size_mid          0.24
_exptl_crystal_size_max          0.36

_exptl_crystal_density_diffrn    1.577
_exptl_crystal_density_meas      ?
# Non-dispersive F(000):
_exptl_crystal_F_000             1310.000
_exptl_absorpt_coefficient_mu    0.243

_diffrn_measurement_device_type
;
Enraf Nonius Kappa CCD
;
_diffrn_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
;
SIR97 (Altomare et al, 1999)
;
_diffrn_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)
;
_diffrn_ambient_temperature      180(2)
_diffrn_reflns_number            26977
_reflns_number_total             12240
_diffrn_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

_diffrn_reflns_theta_min         5.124
_diffrn_reflns_theta_max         27.518
_diffrn_measured_fraction_theta_max 0.974

_diffrn_reflns_theta_full        27.48
_diffrn_measured_fraction_theta_full 0.98

_diffrn_reflns_limit_h_min       -13
_diffrn_reflns_limit_h_max       13
_diffrn_reflns_limit_k_min       -17
_diffrn_reflns_limit_k_max       17
_diffrn_reflns_limit_l_min       -25
```

```
_diffrn_reflns_limit_l_max       25
_reflns_limit_h_min              -13
_reflns_limit_h_max              13
_reflns_limit_k_min              -17
_reflns_limit_k_max              16
_reflns_limit_l_min              0
_reflns_limit_l_max              25

_refine_diff_density_min         -0.72
_refine_diff_density_max         0.54

_refine_ls_number_reflns         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

#_reflns_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:
_reflns_threshold_expression     I>3.00u(I)
_reflns_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
;
_diffrn_radiation_type           'Mo K\a'
_diffrn_radiation_wavelength     0.71073

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

Altomare, A., Burla, M.C., Camalli, M., Cascarano, G.L., Giacovazzo, C.,
Guagliardi, A., Grazia, A., Moliterni, G., Polidori, G. and Spagna, R. (1999)
SIR97: a new tool for crystal structure determination
and refinement.J. App. Cryst. 32, 115-119

COLLECT Software, Nonius BV 1997-2001)

Otwinowski, Z. & Minor, W. (1996), Processing of X-ray
Diffraction Data Collected in Oscillation Mode. Methods Enzymol.
276, 1997, 307-326. Ed Carter, C.W. & Sweet, R.M., Academic Press.

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

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

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. Ueqiv = (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_attached_hydrogens
N1 N 0.02193(14) 1.25618(11) 0.05543(8) 0.0277 1.0000 Uani .
B1 B -0.2914(2) 0.73868(15) 0.3715(1) 0.0277 1.0000 Uani .
F1 F -0.14202(13) 0.6211(1) 0.43967(7) 0.0459 1.0000 Uani .
F2 F 0.08860(15) 0.66989(14) 0.49368(8) 0.0653 1.0000 Uani .
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 .
F5 F -0.12421(12) 0.9375(1) 0.36923(7) 0.0467 1.0000 Uani .
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 .
F11 F -0.16132(11) 0.75204(8) 0.24442(6) 0.0373 1.0000 Uani .
F12 F -0.13121(13) 0.60330(11) 0.13062(7) 0.0485 1.0000 Uani .
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 .
Li1 Li 0.1299(4) 1.2458(3) 0.25456(17) 0.0387 1.0000 Uani .
O1 O -0.04399(14) 1.2446(1) -0.05893(7) 0.0342 1.0000 Uani .
O2 O 0.07107(13) 1.2151(1) 0.15995(6) 0.0329 1.0000 Uani .
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 .
O5 O 0.16389(12) 1.3934(1) 0.29679(7) 0.0318 1.0000 Uani .
O6 O 0.32387(12) 1.2801(1) 0.21004(7) 0.0350 1.0000 Uani .
O7 O 0.35060(13) 1.1201(1) 0.08244(7) 0.0323 1.0000 Uani .
O8 O 0.18005(15) 0.15988(12) 0.31650(8) 0.0455 1.0000 Uani .
C1 C -0.01590(16) 1.20386(13) -0.01293(9) 0.0264 1.0000 Uani .
C2 C -0.01221(15) 1.09289(13) -0.01432(9) 0.0237 1.0000 Uani .

```
C3 C 0.02331(15) 1.08464(13) 0.05274(8) 0.0236 1.0000 Uani .
C4 C 0.04335(15) 1.18987(13) 0.09765(9) 0.0255 1.0000 Uani .
C5 C -0.03767(16) 1.00832(13) -0.06960(8) 0.0244 1.0000 Uani .
C6 C 0.03555(18) 1.36774(14) 0.0774(1) 0.0310 1.0000 Uani .
C7 C 0.16180(18) 1.41375(14) 0.0585(1) 0.0305 1.0000 Uani .
C8 C 0.1674(2) 1.53084(15) 0.07994(11) 0.0361 1.0000 Uani .
C9 C 0.2877(2) 1.58418(15) 0.05915(11) 0.0400 1.0000 Uani .
C10 C 0.4049(2) 1.57030(17) 0.09833(12) 0.0436 1.0000 Uani .
C11 C 0.5211(3) 1.6263(2) 0.07548(14) 0.0576 1.0000 Uani .
C12 C -0.23612(16) 1.03012(13) 0.15820(9) 0.0274 1.0000 Uani .
C13 C -0.21905(18) 0.93399(14) 0.1680(1) 0.0309 1.0000 Uani .
C14 C -0.24570(18) 0.84715(14) 0.1120(1) 0.0316 1.0000 Uani .
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 .
C17 C -0.35200(17) 1.06513(15) -0.0420(1) 0.0311 1.0000 Uani .
C18 C -0.33096(18) 1.15129(14) 0.0145(1) 0.0328 1.0000 Uani .
C19 C -0.29415(17) 1.14232(14) 0.0798(1) 0.0296 1.0000 Uani .
C20 C -0.27775(15) 1.04384(13) 0.09159(9) 0.0251 1.0000 Uani .
C21 C -0.30023(16) 0.95541(13) 0.03579(9) 0.0261 1.0000 Uani .
C22 C -0.1605(2) 1.11042(15) 0.2739(1) 0.0358 1.0000 Uani .
C23 C -0.1473(2) 1.21486(16) 0.3232(1) 0.0388 1.0000 Uani .
C24 C -0.0338(2) 1.37751(16) 0.34728(11) 0.0388 1.0000 Uani .
C25 C 0.0520(2) 1.44430(15) 0.31285(11) 0.0385 1.0000 Uani .
C26 C 0.24969(19) 1.44293(15) 0.2570(1) 0.0347 1.0000 Uani .
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 .
C29 C 0.38869(18) 1.11412(15) 0.1518(1) 0.0339 1.0000 Uani .
C30 C -0.14929(18) 0.77757(15) 0.4038(1) 0.0318 1.0000 Uani .
C31 C -0.0843(2) 0.71316(16) 0.4348(1) 0.0372 1.0000 Uani .
C32 C 0.0357(2) 0.7359(2) 0.46273(11) 0.0473 1.0000 Uani .
C33 C 0.1001(2) 0.8275(2) 0.45917(12) 0.0533 1.0000 Uani .
C34 C 0.0430(2) 0.89350(19) 0.42797(12) 0.0478 1.0000 Uani .
C35 C -0.0784(2) 0.86763(16) 0.40054(11) 0.0379 1.0000 Uani .
C36 C -0.39073(18) 0.70570(14) 0.42874(9) 0.0296 1.0000 Uani .
C37 C -0.3765(2) 0.72405(15) 0.5015(1) 0.0343 1.0000 Uani .
C38 C -0.4680(2) 0.69775(16) 0.5455(1) 0.0392 1.0000 Uani .
C39 C -0.5823(2) 0.65271(17) 0.51859(11) 0.0396 1.0000 Uani .
C40 C -0.6026(2) 0.63300(17) 0.44678(11) 0.0394 1.0000 Uani .
C41 C -0.50938(19) 0.66111(15) 0.40509(9) 0.0326 1.0000 Uani .
C42 C -0.26927(17) 0.63996(13) 0.30566(9) 0.0267 1.0000 Uani .
C43 C -0.30441(18) 0.53728(14) 0.3022(1) 0.0304 1.0000 Uani .
C44 C -0.28788(18) 0.45883(14) 0.24382(11) 0.0330 1.0000 Uani .
C45 C -0.23286(18) 0.48074(15) 0.1854(1) 0.0348 1.0000 Uani .
C46 C -0.18809(18) 0.58045(16) 0.1870(1) 0.0330 1.0000 Uani .
C47 C -0.20612(17) 0.65566(14) 0.2466(1) 0.0293 1.0000 Uani .
C48 C -0.36463(17) 0.82959(14) 0.3468(1) 0.0292 1.0000 Uani .
C49 C -0.42571(18) 0.82293(15) 0.2835(1) 0.0316 1.0000 Uani .
C50 C -0.4937(2) 0.89994(17) 0.26899(11) 0.0400 1.0000 Uani .
C51 C -0.5038(2) 0.98774(17) 0.31986(13) 0.0437 1.0000 Uani .
C52 C -0.4470(2) 0.99777(15) 0.38451(12) 0.0411 1.0000 Uani .
C53 C -0.37982(19) 0.91952(14) 0.3966(1) 0.0329 1.0000 Uani .
C54 C 0.1468(3) 0.7832(2) 0.21138(13) 0.0534 1.0000 Uani .
Cl1 Cl 0.12860(9) 0.91289(5) 0.24357(4) 0.0712 1.0000 Uani .
Cl2 Cl 0.18045(16) 0.72148(8) 0.27622(4) 0.1229 1.0000 Uani .
H51 H -0.0641 1.0140 -0.1175 0.0295 1.0000 Uiso .
H61 H -0.0322 1.3957 0.0539 0.0365 1.0000 Uiso .
H62 H 0.0266 1.3881 0.1295 0.0365 1.0000 Uiso .
H71 H 0.1732 1.3916 0.0066 0.0360 1.0000 Uiso .
H72 H 0.2301 1.3892 0.0837 0.0360 1.0000 Uiso .
H81 H 0.0949 1.5539 0.0570 0.0438 1.0000 Uiso .
H82 H 0.1602 1.5519 0.1322 0.0438 1.0000 Uiso .
```

```
H91 H 0.2770 1.6593 0.0681 0.0483 1.0000 Uiso .
H92 H 0.2999 1.5565 0.0078 0.0483 1.0000 Uiso .
H101 H 0.3937 1.5976 0.1498 0.0510 1.0000 Uiso .
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 .
H131 H -0.1880 0.9255 0.2145 0.0376 1.0000 Uiso .
H141 H -0.2360 0.7772 0.1199 0.0387 1.0000 Uiso .
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 .
H191 H -0.2789 1.2048 0.1189 0.0355 1.0000 Uiso .
H221 H -0.2157 1.0625 0.2951 0.0430 1.0000 Uiso .
H222 H -0.0761 1.0835 0.2655 0.0430 1.0000 Uiso .
H231 H -0.2292 1.2462 0.3253 0.0455 1.0000 Uiso .
H232 H -0.1230 1.2077 0.3712 0.0455 1.0000 Uiso .
H241 H -0.1155 1.4085 0.3576 0.0444 1.0000 Uiso .
H242 H 0.0055 1.3698 0.3920 0.0444 1.0000 Uiso .
H251 H 0.0717 1.5128 0.3456 0.0452 1.0000 Uiso .
H252 H 0.0118 1.4536 0.2688 0.0452 1.0000 Uiso .
H261 H 0.2736 1.5148 0.2835 0.0416 1.0000 Uiso .
H262 H 0.2101 1.4439 0.2108 0.0416 1.0000 Uiso .
H271 H 0.4240 1.4147 0.2154 0.0410 1.0000 Uiso .
H272 H 0.4051 1.3849 0.2913 0.0410 1.0000 Uiso .
H281 H 0.4873 1.2551 0.1639 0.0407 1.0000 Uiso .
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 .
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
N1 0.0295(8) 0.0254(7) 0.0272(7) 0.0056(6) 0.0007(6) -0.0020(6)
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)
F10 0.0366(6) 0.0555(7) 0.0244(5) 0.0080(5) -0.0029(4) -0.0021(5)
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)
F14 0.0416(7) 0.0245(5) 0.0654(8) 0.0041(5) 0.0031(6) -0.0011(5)
F15 0.0593(8) 0.0327(6) 0.0389(6) 0.0157(5) 0.0105(6) 0.0031(5)
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)
```

Li1 0.056(2) 0.0298(16) 0.0298(16) 0.0058(13) −0.0094(15) 0.0043(14)
O1 0.0447(8) 0.0307(7) 0.0302(7) 0.0132(5) −0.0049(6) 0.0035(6)
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)

```
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)
Cl1 0.1087(6) 0.0441(3) 0.0606(4) 0.0080(3) -0.0078(4) 0.0171(3)
Cl2 0.2522(15) 0.0780(6) 0.0500(4) 0.0135(4) -0.0119(6) 0.0881(8)
_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
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
```

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

```
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 . Cl1 . 1.740(3) yes
C54 . Cl2 . 1.718(3) yes
C54 . H541 . 1.000 no
C54 . H542 . 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
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
```

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
Cl1 . C54 . Cl2 . 113.01(14) yes
Cl1 . C54 . H541 . 108.582 no

```
Cl2 . C54 . H541 . 108.585 no
Cl1 . C54 . H542 . 108.576 no
Cl2 . 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 (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_scat_dispersion_real
_atom_type_scat_dispersion_imag
_atom_type_scat_Cromer_Mann_a1
_atom_type_scat_Cromer_Mann_b1
_atom_type_scat_Cromer_Mann_a2
_atom_type_scat_Cromer_Mann_b2
_atom_type_scat_Cromer_Mann_a3
_atom_type_scat_Cromer_Mann_b3
_atom_type_scat_Cromer_Mann_a4
_atom_type_scat_Cromer_Mann_b4
_atom_type_scat_Cromer_Mann_c
_atom_type_scat_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_diffrn    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
```

```
_diffrn_reflns_limit_l_max       21
_reflns_limit_h_min              -10
_reflns_limit_h_max              10
_reflns_limit_k_min              -13
_reflns_limit_k_max              14
_reflns_limit_l_min              0
_reflns_limit_l_max              21

_refine_diff_density_min         -0.54
_refine_diff_density_max         1.40

_refine_ls_number_reflns         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

#_reflns_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:
_reflns_threshold_expression     I>3.00u(I)
_reflns_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
;
_diffrn_radiation_type           synchrotron
_diffrn_radiation_wavelength     0.6923
_diffrn_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. Ueqiv = (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 [Na212]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_scat_dispersion_real
_atom_type_scat_dispersion_imag
_atom_type_scat_Cromer_Mann_a1
_atom_type_scat_Cromer_Mann_b1
_atom_type_scat_Cromer_Mann_a2
_atom_type_scat_Cromer_Mann_b2
_atom_type_scat_Cromer_Mann_a3
_atom_type_scat_Cromer_Mann_b3
_atom_type_scat_Cromer_Mann_a4
_atom_type_scat_Cromer_Mann_b4
_atom_type_scat_Cromer_Mann_c
_atom_type_scat_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_diffrn    1.568
_exptl_crystal_density_meas      ?
# Non-dispersive F(000):
_exptl_crystal_F_000             1022
_exptl_absorpt_coefficient_mu    1.352

_diffrn_measurement_device_type
;
Nonius Kappa CCD
;
_diffrn_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)
;

_diffrn_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
_diffrn_standards_interval_time  0
_diffrn_standards_interval_count 0
_diffrn_standards_number         0
_diffrn_standards_decay_%        0.00

_diffrn_ambient_temperature      180
_diffrn_reflns_number            21614
_reflns_number_total             9407
_diffrn_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

_diffrn_reflns_theta_min         5.150
_diffrn_reflns_theta_max         27.111
_diffrn_measured_fraction_theta_max 0.986

_diffrn_reflns_theta_full        26.026
_diffrn_measured_fraction_theta_full 0.989

_diffrn_reflns_limit_h_min       -13
_diffrn_reflns_limit_h_max       13
_diffrn_reflns_limit_k_min       -12
_diffrn_reflns_limit_k_max       14
_diffrn_reflns_limit_l_min       -23
_diffrn_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
;
_diffrn_radiation_type           'Mo K\a'
_diffrn_radiation_wavelength     0.71073

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

Altomare, A., Cascarano, G., Giacovazzo G., Guagliardi A., Burla M.C.,
Polidori, G. & Camalli, M. (1994) SIR92 - a program for automatic solution
of crystal structures by direct methods. J. Appl. Cryst. (27), 435-435

Betteridge, P.W., Carruthers, J.R., Cooper, R.I.,
Prout, K., Watkin, D.J. (2003). J. Appl. Cryst. 36, 1487.

Nonius BV, COLLECT Software, 1997-2001)

Otwinowski, Z. & Minor, W. (1996), Processing of X-ray
Diffraction Data Collected in Oscillation Mode. Methods Enzymol.
276, 1997, 307-326. Ed Carter, C.W. & Sweet, R.M., Academic Press.

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

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

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

# Uequiv = arithmetic mean of Ui
# i.e. Ueqiv = (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)
Cl1 0.0783(6) 0.0499(5) 0.1124(8) 0.0036(5) -0.0157(6) -0.0148(4)
Cl2 0.0602(5) 0.1008(8) 0.0730(6) -0.0117(5) -0.0299(4) 0.0056(5)
Cl3 0.0767(7) 0.1286(10) 0.0773(6) 0.0261(7) -0.0371(5) -0.0462(7)
Cl4 0.0573(5) 0.0813(7) 0.1444(11) -0.0341(7) -0.0283(6) -0.0001(5)
Cl5 0.0896(7) 0.0980(8) 0.0785(7) -0.0384(6) 0.0232(6) -0.0199(6)
Cl6 0.0781(6) 0.0579(5) 0.1126(8) -0.0356(5) -0.0219(6) -0.0029(4)
Cl7 0.0662(5) 0.0621(5) 0.0650(5) -0.0007(4) -0.0050(4) -0.0092(4)
Cl8 0.0673(6) 0.1346(11) 0.1031(9) -0.0413(8) -0.0182(6) 0.0132(7)
Cl9 0.1238(10) 0.0841(8) 0.0927(8) -0.0023(6) -0.0093(7) -0.0533(8)
_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
C1 . C2 . 1.500(4) yes
C1 . O4 . 1.426(3) yes
C1 . H11 . 1.000 no
C1 . H12 . 1.000 no
C2 . O3 . 1.426(3) yes
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
```

C6 . O7 . 1.427(3) yes
C6 . H61 . 1.000 no
C6 . H62 . 1.000 no
C7 . C8 . 1.497(4) yes
C7 . O7 . 1.427(3) yes
C7 . H71 . 1.000 no
C7 . H72 . 1.000 no
C8 . O6 . 1.431(3) yes
C8 . H81 . 1.000 no
C8 . H82 . 1.000 no
C9 . C18 2_765 1.365(4) yes
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 . Cl7 . 1.757(4) yes
C30 . Cl8 . 1.761(4) yes
C30 . Cl9 . 1.750(4) yes

```
C30 . H301 . 1.000 no
C31 . Cl4 . 1.749(3) yes
C31 . Cl5 . 1.742(3) yes
C31 . Cl6 . 1.761(3) yes
C31 . H311 . 1.000 no
C32 . Cl1 . 1.749(3) yes
C32 . Cl2 . 1.757(3) yes
C32 . Cl3 . 1.737(3) yes
C32 . H321 . 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
C2 . C1 . O4 . 109.2(2) yes
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
C3 . C4 . H42 . 109.643 no
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
O5 . C5 . H52 . 109.716 no
H51 . C5 . H52 . 109.467 no
C5 . C6 . O7 . 109.3(2) yes
C5 . C6 . H61 . 109.521 no
O7 . C6 . H61 . 109.521 no
C5 . C6 . H62 . 109.521 no
O7 . C6 . H62 . 109.521 no
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
```

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
C15 . C14 . H141 . 119.277 no
C14 . C15 . C16 . 119.8(2) yes
C14 . C15 . H151 . 120.121 no
C16 . C15 . H151 . 120.121 no
C15 . C16 . C17 . 120.8(2) yes
C15 . C16 . O4 . 124.7(2) yes
C17 . C16 . O4 . 114.5(2) yes
C16 . C17 . C12 2_765 118.2(2) yes
C16 . C17 . C18 . 121.7(2) yes
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
C20 . C21 . C23 2_765 129.0(2) yes
C20 . C21 . C22 . 107.8(2) yes
C23 2_765 C21 . C22 . 123.2(2) yes
C19 . C22 . C21 . 108.04(19) yes
C19 . C22 . C23 . 129.6(2) yes
C21 . C22 . C23 . 122.4(2) yes
C22 . C23 . C21 2_765 114.4(2) yes
C22 . C23 . H231 . 122.804 no
C21 2_765 C23 . H231 . 122.804 no
C25 . C24 . N1 . 113.1(2) yes
C25 . C24 . H241 . 108.560 no
N1 . C24 . H241 . 108.560 no
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

```
H251 . C25 . H252 . 109.467 no
C25 . C26 . C27 . 113.3(2) yes
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
Cl7 . C30 . Cl8 . 109.4(2) yes
Cl7 . C30 . Cl9 . 108.6(2) yes
Cl8 . C30 . Cl9 . 112.2(2) yes
Cl7 . C30 . H301 . 111.065 no
Cl8 . C30 . H301 . 107.447 no
Cl9 . C30 . H301 . 108.175 no
Cl4 . C31 . Cl5 . 111.01(19) yes
Cl4 . C31 . Cl6 . 110.44(17) yes
Cl5 . C31 . Cl6 . 109.88(18) yes
Cl4 . C31 . H311 . 107.900 no
Cl5 . C31 . H311 . 108.476 no
Cl6 . C31 . H311 . 109.073 no
Cl1 . C32 . Cl2 . 112.90(17) yes
Cl1 . C32 . Cl3 . 110.55(18) yes
Cl2 . C32 . Cl3 . 108.40(17) yes
Cl1 . C32 . H321 . 106.007 no
Cl2 . C32 . H321 . 108.253 no
Cl3 . 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
#===END

######################################################
## denoted [Na212][B{3,5-(CF3)2(C6H3)}4]2 in text ##
######################################################

data_5
_database_code_depnum_ccdc_archive 'CCDC 254114'

_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                   38.5110(1)
_cell_angle_alpha                90
_cell_length_b                   15.7246(2)
_cell_angle_beta                 93.6400(5)
_cell_length_c                   22.8791(3)
_cell_angle_gamma                90
_cell_volume                     13826.9(3)

_symmetry_cell_setting           Monoclinic
_symmetry_space_group_name_H-M   'C 1 2/c 1 '
loop_
_symmetry_equiv_pos_as_xyz
x,y,z
-x,-y,-z
x+1/2,y+1/2,z
-x+1/2,-y+1/2,-z
-x,y,-z+1/2
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 (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_scat_dispersion_real
_atom_type_scat_dispersion_imag
_atom_type_scat_Cromer_Mann_a1
_atom_type_scat_Cromer_Mann_b1
_atom_type_scat_Cromer_Mann_a2
_atom_type_scat_Cromer_Mann_b2
_atom_type_scat_Cromer_Mann_a3
_atom_type_scat_Cromer_Mann_b3
_atom_type_scat_Cromer_Mann_a4
_atom_type_scat_Cromer_Mann_b4
_atom_type_scat_Cromer_Mann_c
_atom_type_scat_source
'C   ' 0.0020 0.0020 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 57.7997
0.0030 International_Tables_Vol_IV_Table_2.2B
'B   ' 0.0000 0.0010 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
'F   ' 0.0140 0.0100 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
'Na  ' 0.0300 0.0250 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
'N   ' 0.0040 0.0030 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
'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
_cell_measurement_theta_max      27
_cell_measurement_temperature    180

_exptl_crystal_description       ' block '
_exptl_crystal_colour            ' red '
_exptl_crystal_size_min          0.18
_exptl_crystal_size_mid          0.23
_exptl_crystal_size_max          0.30

_exptl_crystal_density_diffrn    1.342
_exptl_crystal_density_meas      ?
# Non-dispersive F(000):
_exptl_crystal_F_000             5672.001
```

```
_exptl_absorpt_coefficient_mu    0.135
# Sheldrick geometric definitions 0.97 0.98

_diffrn_measurement_device_type
;
Nonius Kappa CCD
;
_diffrn_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)
;
_diffrn_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)
;
_diffrn_standards_interval_time  0
_diffrn_standards_interval_count 0
_diffrn_standards_number         0
_diffrn_standards_decay_%        0.00

_diffrn_ambient_temperature      180
_diffrn_reflns_number            15601
_reflns_number_total             15601
_diffrn_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

_diffrn_reflns_theta_min         5.152
_diffrn_reflns_theta_max         27.505
_diffrn_measured_fraction_theta_max 0.982

_diffrn_reflns_theta_full        26.405
_diffrn_measured_fraction_theta_full 0.991

_diffrn_reflns_limit_h_min       -49
_diffrn_reflns_limit_h_max       49
_diffrn_reflns_limit_k_min       0
_diffrn_reflns_limit_k_max       20
_diffrn_reflns_limit_l_min       0
_diffrn_reflns_limit_l_max       29
_reflns_limit_h_min              -49
_reflns_limit_h_max              49
```

```
_reflns_limit_k_min              0
_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
;
_diffrn_radiation_type           'Mo K\a'
_diffrn_radiation_wavelength     0.71073
_exptl_special_details
;
Treatment of rezidual 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 A**3 - equiv to ca 7 H2O molecules
disordered per unit pseudorotaxane 3.5 molec /asym unit.
Identification of the crystalising 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)

P. v.d. Sluis & A. L. Spek, Acta Crystallogr. 1990, A46, 194.

PLATON, A Multipurpose Crystallographic Tool,
Utrecht University, Utrecht, The Netherlands, A. L. Spek, 1998.

Altomare, A., Cascarano, G., Giacovazzo G., Guagliardi A., Burla M.C.,
Polidori, G. & Camalli, M. (1994) SIR92 - a program for automatic solution
of crystal structures by direct methods. J. Appl. Cryst. (27), 435-435

Nonius BV, COLLECT Software, 1997-2001)

Otwinowski, Z. & Minor, W. (1996), Processing of X-ray
Diffraction Data Collected in Oscillation Mode. Methods Enzymol.
276, 1997, 307-326. Ed Carter, C.W. & Sweet, R.M., Academic Press.

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

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. Ueqiv = (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
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 .
F6 F 0.1229(4) 0.2503(11) 0.1359(6) 0.044(3) 0.48(3) Uiso DUP .
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 .
F24 F 0.0941(7) 0.5974(16) 0.0187(8) 0.095(5) 0.322(11) Uiso DUP .
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 .
F29 F 0.3255(4) 0.674(1) 0.3115(9) 0.074(4) 0.430(13) Uiso DUP .
F30 F 0.3244(5) 0.5750(12) 0.2384(9) 0.090(4) 0.430(13) Uiso DUP .
F31 F 0.2133(6) 0.835(1) 0.127(1) 0.086(4) 0.43(2) Uiso DUP .
F32 F 0.2240(7) 0.8904(12) 0.2047(9) 0.096(4) 0.43(2) Uiso DUP .
F33 F 0.2675(5) 0.8657(11) 0.149(1) 0.085(4) 0.43(2) Uiso DUP .
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 . .
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 0.051(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)
_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
N1 . C6 . 1.474(11) yes
N1 . C4 . 1.397(12) yes
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
```

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
C60 . F39 . 1.39(3) yes
C60 . F38 . 1.39(3) yes
C60 . F37 . 1.23(2) yes
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
C6 . N1 . C4 . 120.6(7) yes
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
H62 . C6 . N1 . 107.816 no
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
H102 . C10 . C11 . 107.789 no
H101 . C10 . C11 . 107.786 no
H102 . C10 . C9 . 107.799 no
H101 . C10 . C9 . 107.792 no
C11 . C10 . C9 . 116.1(2) yes
H113 . C11 . H112 . 109.468 no
H113 . C11 . H111 . 109.461 no
H112 . C11 . H111 . 109.429 no
H113 . C11 . C10 . 109.499 no
H112 . C11 . C10 . 109.478 no
H111 . C11 . C10 . 109.474 no
H1092 . C109 . H1091 . 109.456 no
H1092 . C109 . H81 . 69.095 no
H1091 . C109 . H81 . 107.470 no
H1092 . C109 . C110 . 107.792 no
H1091 . C109 . C110 . 107.791 no
H81 . C109 . C110 . 143.290 no
H1092 . C109 . C8 . 107.782 no
H1091 . C109 . C8 . 107.778 no
H81 . C109 . C8 . 41.409 no
C110 . C109 . C8 . 116.1(2) yes
H1102 . C110 . H1101 . 109.475 no
H1102 . C110 . C111 . 107.788 no
H1101 . C110 . C111 . 107.783 no
H1102 . C110 . C109 . 107.821 no
H1101 . C110 . C109 . 107.813 no
C111 . C110 . C109 . 116.1(2) yes
H1113 . C111 . H1112 . 109.446 no
H1113 . C111 . H1111 . 109.457 no
H1112 . C111 . H1111 . 109.439 no
H1113 . C111 . C110 . 109.488 no
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 . O7 2_666 126.5(9) yes
C18 . C17 . C16 . 121.6(9) yes
O7 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
H232 . C23 . O4 . 109.186 no
H231 . C23 . O4 . 109.187 no
H232 . C23 . C22 . 109.187 no
H231 . C23 . C22 . 109.186 no
O4 . C23 . C22 . 110.6(8) yes
C24 . O4 . C23 . 115.6(10) yes
H242 . C24 . H241 . 109.464 no
H242 . C24 . C25 . 109.671 no
H241 . C24 . C25 . 109.657 no
H242 . C24 . O4 . 109.674 no
H241 . C24 . O4 . 109.665 no
C25 . C24 . O4 . 108.7(11) yes
H252 . C25 . H251 . 109.466 no
H252 . C25 . O5 . 109.550 no
H251 . C25 . O5 . 109.544 no
H252 . C25 . C24 . 109.524 no
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
H261 . C26 . C27 . 109.697 no
H262 . C26 . O5 . 109.703 no
H261 . C26 . O5 . 109.706 no
C27 . C26 . O5 . 108.6(10) yes
H272 . C27 . H271 . 109.466 no
H272 . C27 . O6 . 109.268 no
H271 . C27 . O6 . 109.267 no
H272 . C27 . C26 . 109.271 no
H271 . C27 . C26 . 109.267 no
O6 . C27 . C26 . 110.3(10) yes
C28 . O6 . C27 . 115.2(10) yes

H282 . C28 . H281 . 109.460 no
H282 . C28 . C29 . 109.222 no
H281 . C28 . C29 . 109.218 no
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
#===END
```