```
data_global


# start Validation Reply Form
_vrf_PLAT093_x0701
;
PROBLEM: No su's on H-atoms, but refinement reported as .      mixed
RESPONSE: H atoms were positioned geometrically and refined using a
riding model.
;
_vrf_PLAT220_x0701
;
PROBLEM: Large Non-Solvent    F     Ueq(max)/Ueq(min) ...       5.05 Ratio
RESPONSE: disorder in trifluoro groups cannot be completely resolved
;
_vrf_DIFF005_x0614
;
PROBLEM: _diffrn_measurement_method is missing
RESPONSE: problem corrected.
;
_vrf_PLAT027_x0614
;
PROBLEM: _diffrn_reflns_theta_full (too) Low ............      24.71 Deg.
RESPONSE: crystals do not scatter at high angle.
;
_vrf_PLAT093_x0614
;
PROBLEM: No su's on H-atoms, but refinement reported as .      mixed
RESPONSE: see above
;
_vrf_PLAT242_x0614
;
PROBLEM: Check Low       Ueq as Compared to Neighbors for        C13
RESPONSE: disorder in C13 trifluoro group cannot be completely resolved...
;
_vrf_PLAT027_x0612b2
;
PROBLEM: _diffrn_reflns_theta_full (too) Low ............      24.41 Deg.
RESPONSE: crystals do not scatter at high angle..
;
_vrf_PLAT075_x0612b2
;
PROBLEM: Occupancy       2.00 greater than 1.0 for ......        H1W
RESPONSE: Solvent water molecule is disordered and sits on a
 special position. Only one HW atom was detected in difference map..
;
_vrf_PLAT093_x0612b2
;
PROBLEM: No su's on H-atoms, but refinement reported as .      mixed
RESPONSE: see above.
;
_vrf_PLAT220_x0612b2
;
PROBLEM: Large Non-Solvent    C     Ueq(max)/Ueq(min) ...       6.14 Ratio
RESPONSE: disorder in trifluoro groups cannot be completely resolve...
;
```

```
_vrf_PLAT241_x0612b2
;
PROBLEM: Check High      Ueq as Compared to Neighbors for        C21
RESPONSE: disorder in C21 trifluoro group cannot be completely resolved...
;
_vrf_PLAT242_x0612b2
;
PROBLEM: Check Low       Ueq as Compared to Neighbors for         O6
RESPONSE: Attributable to disorder in the trifluoro groups.
;
_vrf_PLAT305_x0612b2
;
PROBLEM: Isolated Hydrogen Atom (Outside Bond Range ??)         >H1W
RESPONSE: Solvent water molecule is disordered and sits on a
 special position. Only one HW atom was detected in difference map...
;
_vrf_PLAT369_x0612b2
;
PROBLEM: Long   C(sp2)-C(sp2) Bond  C20    -   C21    ...       1.72 Ang.
RESPONSE: Attributable to disorder in the trifluoro groups.
;
_vrf_PLAT773_x0612b2
;
PROBLEM: Suspect C-C Bond in CIF:      C21    -C20     ..       1.72 Ang.
RESPONSE: Attributable to disorder in the trifluoro groups.
;
# end Validation Reply Form

#Added by publCIF (CCDC output) (Fri Sep 7 16:28:56 2007)


_audit_update_record
;
2007-08-31 # Formatted by publCIF
;

#============================================================
# 1. SUBMISSION DETAILS


_publ_contact_author_name         'Dr. Shawn Swavey'
_publ_contact_author_address
;     Department of Chemistry
      University of Dayton
      Dayon, OH 45469-2357 USA
;
_publ_contact_author_phone        '937 229 3145'
_publ_contact_author_fax          '937 229 2635'
_publ_contact_author_email        shawn.swavey@notes.udayton.edu

_publ_requested_journal           'Inorg. Chem.'
_publ_requested_category          ?
_publ_requested_coeditor_name     ?

_publ_contact_letter        ?
```

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

# 2. TITLE AND AUTHOR LIST

_publ_section_title
;
Neodymium, gadolinium and terbium complexes containing hexafluoro- acetylonate
and 2,2'bipyrimidine: structural and spectroscopic characterization
;


# The loop structure below should contain the names and addresses
# of all authors, in the required order of publication. Repeat as
# necessary.

loop_
 _publ_author_name
 _publ_author_address
     'Fratini, Albert'
;     Department of Chemistry
      University of Dayton
      Dayton, OH 45469-2357 USA
;

     'Richards, Gregory'
;     Department of Chemistry
      University of Dayton
      Dayton, OH 45469-2357 USA
;

     'Larder, Emily'
;     Department of Chemistry
      University of Dayton
      Dayton, OH 45469-2357 USA
;

     'Swavey, Shawn'
;     Department of Chemistry
      University of Dayton
      Dayton, OH 45469-2357 USA
;

#=============================================================


data_x0701


_audit_creation_method            SHELXL-97
_chemical_name_systematic         ?
_chemical_name_common             ?
_chemical_melting_point           ?
_chemical_formula_moiety
 'Nd (C5 H1 F6 O2)3 (C8 H6 N4)'
_chemical_formula_sum
```

```
 'C23 H9 F18 N4 Nd O6'
_chemical_formula_weight          923.58

loop_
 _atom_type_symbol
 _atom_type_description
 _atom_type_scat_dispersion_real
 _atom_type_scat_dispersion_imag
 _atom_type_scat_source
 'C'  'C'   0.0033   0.0016
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'H'  'H'   0.0000   0.0000
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'N'  'N'   0.0061   0.0033
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'O'  'O'   0.0106   0.0060
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'F'  'F'   0.0171   0.0103
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'Nd'  'Nd'  -0.1943   3.0179
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting            triclinic
_symmetry_space_group_name_H-M    P-1
_symmetry_space_group_name_Hall   '-P 1'

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

_cell_length_a                    10.8373(8)
_cell_length_b                    12.3695(11)
_cell_length_c                    14.2200(8)
_cell_angle_alpha                 109.612(8)
_cell_angle_beta                  93.083(5)
_cell_angle_gamma                 114.953(7)
_cell_volume                      1584.8(2)
_cell_formula_units_Z             2
_cell_measurement_temperature     140(2)
_cell_measurement_reflns_used     ?
_cell_measurement_theta_min       ?
_cell_measurement_theta_max       25.03

_exptl_crystal_description        plate
_exptl_crystal_colour             'colourless'
_exptl_crystal_size_max           0.517
_exptl_crystal_size_mid           0.0615
_exptl_crystal_size_min           0.0549
_exptl_crystal_density_meas       ?
_exptl_crystal_density_diffrn     1.935
_exptl_crystal_density_method     'not measured'
_exptl_crystal_F_000              890
_exptl_absorpt_coefficient_mu     1.788
_exptl_absorpt_correction_type    analytical
_exptl_absorpt_correction_T_min   0.60922
_exptl_absorpt_correction_T_max   0.91381
```

```
_exptl_absorpt_process_details
;
Multifaceted crystal model (Clark & Reid 1995)
;
_exptl_special_details
;
 Note that the absorption correction parameters Tmin and Tmax also
reflect beam corrections, etc. As a result, the numerical values
for Tmin and Tmax may differ from expected values based solely on
absorption effects and crystal size
;

_diffrn_ambient_temperature       140(2)
_diffrn_radiation_wavelength      0.71073
_diffrn_radiation_type            MoK\a
_diffrn_radiation_source          'fine-focus sealed tube'
_diffrn_radiation_monochromator   graphite
_diffrn_measurement_device_type   'CCD detector Xcalibur3'
_diffrn_measurement_method        'phi and omega scans'
_diffrn_detector_area_resol_mean  ?
_diffrn_standards_number          0
_diffrn_standards_interval_count  0
_diffrn_standards_interval_time   0
_diffrn_standards_decay_%         0.00
_diffrn_reflns_number             31856
_diffrn_reflns_av_R_equivalents   0.0603
_diffrn_reflns_av_sigmaI/netI     0.0315
_diffrn_reflns_limit_h_min        -12
_diffrn_reflns_limit_h_max        12
_diffrn_reflns_limit_k_min        -14
_diffrn_reflns_limit_k_max        14
_diffrn_reflns_limit_l_min        -16
_diffrn_reflns_limit_l_max        16
_diffrn_reflns_theta_min          3.77
_diffrn_reflns_theta_max          25.03
_reflns_number_total              5420
_reflns_number_gt                 5409
_reflns_threshold_expression      >2sigma(I)

_computing_data_collection        'CrysAlis CCD (Oxford Diffraction, 2005)'
_computing_data_reduction         'CrysAlis RED (Oxford Diffraction, 2005)'
_computing_cell_refinement        'CrysAlis RED (Oxford Diffraction, 2005)'
_computing_structure_solution     'Bruker SHELXTL v6.14'
_computing_structure_refinement   'Bruker SHELXTL v6.14'
_computing_molecular_graphics     'Bruker SHELXTL v6.14'
_computing_publication_material   'Bruker SHELXTL v6.14'

_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
```

```
 All F-atoms of the CF~3~ groups show disorder as indicated by the enlarged
 displacement parameters.  Multi-component conformations are modeled
 where possible.
;

_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0000P)^2^+7.0207P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     mixed
_refine_ls_extinction_method      SHELXL
_refine_ls_extinction_coef        0.0007(2)
_refine_ls_extinction_expression
 'Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^'
_refine_ls_number_reflns          5420
_refine_ls_number_parameters      470
_refine_ls_number_restraints      9
_refine_ls_R_factor_all           0.0610
_refine_ls_R_factor_gt            0.0609
_refine_ls_wR_factor_ref          0.1136
_refine_ls_wR_factor_gt           0.1136
_refine_ls_goodness_of_fit_ref    1.746
_refine_ls_restrained_S_all       1.761
_refine_ls_shift/su_max           0.004
_refine_ls_shift/su_mean          0.000


## ===============REFERENCES====================##
_publ_section_
;User-defined structure solution references


Sheldrick, G.M. (1997). SHELXS97 and SHELXL97. University of Gottingen,
Germany.

Clark, R.C. and Reid, J.S. (1995), Acta Cryst. A51, 887-897.

Oxford Diffraction (2005). CrysAlis CCD and CrysAlis RED. Versions
171.27p5, Oxford Diffraction Ltd., Abingdon, Oxfordshire, UK.

Sheldrick, G.M. (2000). SHELXTL. Version 6.10. Bruker AXS Inc., Madison,
Wisconsin, USA.
;


loop_
 _atom_site_label
 _atom_site_type_symbol
 _atom_site_fract_x
 _atom_site_fract_y
 _atom_site_fract_z
 _atom_site_U_iso_or_equiv
 _atom_site_adp_type
```

```
 _atom_site_occupancy
 _atom_site_symmetry_multiplicity
 _atom_site_calc_flag
 _atom_site_refinement_flags
 _atom_site_disorder_assembly
 _atom_site_disorder_group
Nd Nd 0.97719(4) 0.97617(4) 0.74177(3) 0.01651(13) Uani 1 1 d . . .
O1 O 1.0400(5) 1.2056(5) 0.8097(4) 0.0262(11) Uani 1 1 d . . .
N1 N 1.1001(6) 0.9629(6) 0.5771(4) 0.0225(13) Uani 1 1 d . . .
C1 C 1.1903(8) 0.9137(8) 0.5653(6) 0.0292(17) Uani 1 1 d . . .
H1A H 1.2319 0.9091 0.6218 0.035 Uiso 1 1 calc R . .
O2 O 1.2313(5) 1.1084(5) 0.7933(4) 0.0250(11) Uani 1 1 d . . .
N2 N 1.0988(6) 0.9607(6) 0.9091(4) 0.0240(13) Uani 1 1 d . . .
C2 C 1.2233(8) 0.8700(8) 0.4731(6) 0.0308(18) Uani 1 1 d . . .
H2A H 1.2835 0.8335 0.4655 0.037 Uiso 1 1 calc R . .
N3 N 1.0781(6) 0.9354(6) 0.4015(4) 0.0208(13) Uani 1 1 d . . .
C3 C 1.1634(8) 0.8827(7) 0.3921(6) 0.0274(17) Uani 1 1 d . . .
H3B H 1.1834 0.8531 0.3285 0.033 Uiso 1 1 calc R . .
O3 O 0.7420(5) 0.9604(5) 0.7277(4) 0.0288(12) Uani 1 1 d . . .
O4 O 0.8213(5) 0.8029(5) 0.7923(4) 0.0277(12) Uani 1 1 d . . .
N4 N 1.0733(6) 0.9316(6) 1.0654(4) 0.0239(13) Uani 1 1 d . . .
C4 C 1.0490(7) 0.9720(6) 0.4944(5) 0.0183(15) Uani 1 1 d . . .
O5 O 1.0258(5) 0.7910(5) 0.6908(4) 0.0276(12) Uani 1 1 d . . .
C5 C 1.1882(8) 0.9101(8) 0.8991(6) 0.0292(17) Uani 1 1 d . . .
H5A H 1.2309 0.9063 0.8440 0.035 Uiso 1 1 calc R . .
O6 O 0.8155(5) 0.8032(5) 0.5790(4) 0.0301(12) Uani 1 1 d . . .
C6 C 1.2178(8) 0.8641(8) 0.9677(6) 0.0340(19) Uani 1 1 d . . .
H6A H 1.2776 0.8271 0.9590 0.041 Uiso 1 1 calc R . .
C7 C 1.1556(8) 0.8747(8) 1.0502(6) 0.0309(18) Uani 1 1 d . . .
H7A H 1.1714 0.8413 1.0966 0.037 Uiso 1 1 calc R . .
C8 C 1.0478(7) 0.9704(6) 0.9932(5) 0.0184(15) Uani 1 1 d . . .
C10 C 1.2859(8) 1.3304(8) 0.8597(6) 0.0340(19) Uani 1 1 d . . .
H10A H 1.3592 1.4147 0.8877 0.041 Uiso 1 1 calc R . .
C11 C 1.3157(7) 1.2270(7) 0.8339(5) 0.0275(17) Uani 1 1 d . . .
C12 C 1.4688(9) 1.2568(9) 0.8548(8) 0.045(2) Uani 1 1 d . . .
F1 F 1.5569(5) 1.3818(5) 0.9090(5) 0.0667(17) Uani 1 1 d . . .
F2 F 1.4886(6) 1.1918(6) 0.9051(6) 0.075(2) Uani 1 1 d . . .
F3 F 1.5074(6) 1.2230(7) 0.7666(5) 0.083(2) Uani 1 1 d . . .
C9 C 1.1496(9) 1.3112(7) 0.8447(6) 0.0300(17) Uani 1 1 d . A .
C13 C 1.1257(9) 1.4286(8) 0.8754(8) 0.064(3) Uani 1 1 d D . .
F4A F 1.216(2) 1.5293(19) 0.8549(18) 0.068(8) Uiso 0.33 1 d PD A 1
F4B F 1.2495(16) 1.5387(14) 0.8869(17) 0.032(4) Uiso 0.34 1 d PD A 1
F4C F 1.2251(19) 1.5455(14) 0.9198(14) 0.052(5) Uiso 0.33 1 d PD A 1
F6A F 0.9968(11) 1.4111(12) 0.8490(13) 0.022(3) Uiso 0.34 1 d PD A 2
F6B F 1.0204(17) 1.4118(15) 0.8093(14) 0.040(4) Uiso 0.33 1 d PD A 2
F6C F 1.084(3) 1.421(2) 0.7742(14) 0.089(7) Uiso 0.33 1 d PD A 2
F5A F 1.143(3) 1.485(2) 0.9804(13) 0.070(7) Uiso 0.33 1 d PD A 3
F5B F 1.093(2) 1.4445(19) 0.9757(12) 0.048(5) Uiso 0.34 1 d PD A 3
F5C F 1.021(3) 1.425(3) 0.916(3) 0.106(8) Uiso 0.33 1 d PD A 3
C16 C 0.6950(8) 0.7529(8) 0.7943(6) 0.0323(18) Uani 1 1 d . B .
C17 C 0.6562(9) 0.6455(9) 0.8363(7) 0.048(2) Uani 1 1 d . . .
F7A F 0.6275(15) 0.5351(13) 0.7655(10) 0.065(4) Uiso 0.55 1 d P B 4
F8A F 0.7577(11) 0.6690(10) 0.9107(8) 0.050(3) Uiso 0.55 1 d P B 4
F9A F 0.5433(13) 0.6294(12) 0.8780(10) 0.064(3) Uiso 0.55 1 d P B 4
F7B F 0.6828(18) 0.5496(16) 0.7773(13) 0.066(5) Uiso 0.45 1 d P B 5
F8B F 0.7223(13) 0.6997(12) 0.9309(10) 0.047(3) Uiso 0.45 1 d P B 5
F9B F 0.5159(14) 0.5880(13) 0.8317(11) 0.051(3) Uiso 0.45 1 d P B 5
```

```
C14 C 0.6250(8) 0.8795(8) 0.7283(6) 0.0334(18) Uani 1 1 d . B .
C18 C 0.5012(9) 0.8925(10) 0.6863(8) 0.053(3) Uani 1 1 d . . .
F10A F 0.5323(10) 0.9750(10) 0.6409(8) 0.058(2) Uiso 0.65 1 d P C 6
F11A F 0.4407(12) 0.9331(12) 0.7609(9) 0.078(3) Uiso 0.65 1 d P C 6
F12A F 0.3998(12) 0.7787(11) 0.6177(9) 0.074(3) Uiso 0.65 1 d P C 6
F10B F 0.440(2) 0.8065(19) 0.5892(15) 0.063(5) Uiso 0.35 1 d P C 7
F11B F 0.5423(19) 1.0155(18) 0.6937(15) 0.063(5) Uiso 0.35 1 d P C 7
F12B F 0.4006(18) 0.8679(18) 0.7375(13) 0.052(4) Uiso 0.35 1 d P C 7
C21 C 0.7797(9) 0.6865(8) 0.5329(6) 0.038(2) Uani 1 1 d . D .
C22 C 0.6608(13) 0.6193(10) 0.4354(8) 0.061(3) Uani 1 1 d . . .
F13A F 0.5444(19) 0.6051(18) 0.4584(13) 0.087(5) Uiso 0.50 1 d P D 8
F14A F 0.6833(14) 0.6903(13) 0.3781(10) 0.064(4) Uiso 0.50 1 d P D 8
F15A F 0.6543(17) 0.5079(15) 0.3743(12) 0.088(4) Uiso 0.50 1 d P D 8
F13B F 0.5598(17) 0.6582(16) 0.4548(12) 0.075(5) Uiso 0.50 1 d P D 9
F14B F 0.7191(17) 0.6516(16) 0.3647(12) 0.084(5) Uiso 0.50 1 d P D 9
F15B F 0.5887(14) 0.4920(13) 0.4054(10) 0.067(3) Uiso 0.50 1 d P D 9
C19 C 0.9516(9) 0.6734(8) 0.6372(7) 0.038(2) Uani 1 1 d . D .
C23 C 1.0026(12) 0.5864(9) 0.6621(8) 0.056(3) Uani 1 1 d . . .
F16 F 0.9348(8) 0.4625(6) 0.6016(6) 0.096(3) Uani 1 1 d . . .
F17 F 0.9918(11) 0.5952(8) 0.7576(6) 0.110(3) Uani 1 1 d . . .
F18 F 1.1338(8) 0.6232(7) 0.6610(7) 0.104(3) Uani 1 1 d . . .
C15 C 0.5935(9) 0.7820(8) 0.7633(7) 0.040(2) Uani 1 1 d . . .
H15A H 0.5017 0.7344 0.7662 0.048 Uiso 1 1 calc R B .
C20 C 0.8326(11) 0.6145(9) 0.5592(7) 0.049(2) Uani 1 1 d . . .
H20A H 0.7879 0.5247 0.5243 0.059 Uiso 1 1 calc R D .

loop_
 _atom_site_aniso_label
 _atom_site_aniso_U_11
 _atom_site_aniso_U_22
 _atom_site_aniso_U_33
 _atom_site_aniso_U_23
 _atom_site_aniso_U_13
 _atom_site_aniso_U_12
Nd 0.0176(2) 0.0199(2) 0.01371(19) 0.00916(14) 0.00482(13) 0.00816(14)
O1 0.031(3) 0.024(3) 0.025(3) 0.010(2) 0.006(2) 0.013(2)
N1 0.024(3) 0.033(3) 0.019(3) 0.014(3) 0.005(2) 0.018(3)
C1 0.030(4) 0.049(5) 0.027(4) 0.027(4) 0.011(3) 0.026(4)
O2 0.020(2) 0.025(3) 0.025(3) 0.011(2) 0.006(2) 0.005(2)
N2 0.022(3) 0.032(3) 0.020(3) 0.012(3) 0.007(2) 0.013(3)
C2 0.034(4) 0.042(5) 0.034(4) 0.022(4) 0.014(4) 0.027(4)
N3 0.026(3) 0.027(3) 0.016(3) 0.012(3) 0.006(2) 0.015(3)
C3 0.038(4) 0.033(4) 0.021(4) 0.017(3) 0.013(3) 0.021(4)
O3 0.022(3) 0.038(3) 0.028(3) 0.015(2) 0.007(2) 0.014(2)
O4 0.022(3) 0.028(3) 0.029(3) 0.015(2) 0.007(2) 0.005(2)
N4 0.027(3) 0.026(3) 0.022(3) 0.013(3) 0.007(3) 0.013(3)
C4 0.017(3) 0.015(3) 0.019(4) 0.008(3) 0.002(3) 0.002(3)
O5 0.035(3) 0.022(3) 0.027(3) 0.011(2) 0.009(2) 0.014(2)
C5 0.025(4) 0.041(5) 0.029(4) 0.015(4) 0.012(3) 0.019(4)
O6 0.035(3) 0.029(3) 0.023(3) 0.009(2) 0.003(2) 0.013(2)
C6 0.040(5) 0.052(5) 0.028(4) 0.018(4) 0.013(4) 0.035(4)
C7 0.039(4) 0.037(5) 0.028(4) 0.019(4) 0.005(3) 0.022(4)
C8 0.014(3) 0.016(3) 0.020(4) 0.007(3) 0.003(3) 0.003(3)
C10 0.035(4) 0.025(4) 0.031(4) 0.013(4) 0.003(4) 0.005(4)
C11 0.021(4) 0.029(4) 0.022(4) 0.011(3) 0.006(3) 0.003(3)
C12 0.027(4) 0.033(5) 0.058(6) 0.014(5) 0.001(4) 0.004(4)
F1 0.028(3) 0.042(3) 0.094(5) 0.013(3) -0.007(3) -0.002(2)
```

```
F2 0.039(3) 0.064(4) 0.115(6) 0.042(4) -0.012(3) 0.016(3)
F3 0.042(3) 0.093(5) 0.087(5) 0.013(4) 0.032(3) 0.024(3)
C9 0.045(5) 0.027(4) 0.024(4) 0.014(3) 0.006(4) 0.019(4)
C13 0.054(6) 0.021(5) 0.094(9) 0.019(5) -0.015(6) 0.004(5)
C16 0.033(4) 0.030(4) 0.023(4) 0.009(3) 0.004(3) 0.007(4)
C17 0.037(5) 0.044(5) 0.045(6) 0.020(5) 0.011(4) 0.001(4)
C14 0.026(4) 0.038(5) 0.033(4) 0.011(4) 0.006(3) 0.015(4)
C18 0.029(5) 0.053(6) 0.072(7) 0.018(5) 0.023(5) 0.019(5)
C21 0.047(5) 0.034(5) 0.024(4) 0.009(4) 0.006(4) 0.013(4)
C22 0.082(8) 0.035(6) 0.043(6) 0.000(5) 0.012(5) 0.018(6)
C19 0.049(5) 0.033(5) 0.037(5) 0.019(4) 0.014(4) 0.020(4)
C23 0.072(7) 0.033(5) 0.056(7) 0.012(5) -0.005(6) 0.025(5)
F16 0.122(6) 0.031(3) 0.121(6) 0.016(4) -0.013(5) 0.038(4)
F17 0.206(10) 0.101(6) 0.084(5) 0.060(5) 0.033(6) 0.105(7)
F18 0.079(5) 0.065(5) 0.182(9) 0.042(5) 0.017(5) 0.053(4)
C15 0.028(4) 0.043(5) 0.042(5) 0.017(4) 0.011(4) 0.010(4)
C20 0.064(6) 0.026(5) 0.046(6) 0.005(4) 0.001(5) 0.019(5)

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

loop_
 _geom_bond_atom_site_label_1
 _geom_bond_atom_site_label_2
 _geom_bond_distance
 _geom_bond_site_symmetry_2
 _geom_bond_publ_flag
Nd O1 2.431(5) . ?
Nd O2 2.444(5) . ?
Nd O5 2.448(5) . ?
Nd O4 2.464(5) . ?
Nd O3 2.465(5) . ?
Nd O6 2.466(5) . ?
Nd N1 2.747(5) . ?
Nd N2 2.759(6) . ?
Nd N3 2.760(6) 2_776 ?
Nd N4 2.776(6) 2_777 ?
O1 C9 1.248(9) . ?
N1 C4 1.332(9) . ?
N1 C1 1.344(9) . ?
C1 C2 1.370(10) . ?
C1 H1A 0.9300 . ?
O2 C11 1.251(8) . ?
N2 C8 1.334(8) . ?
N2 C5 1.348(9) . ?
C2 C3 1.378(11) . ?
C2 H2A 0.9300 . ?
N3 C3 1.327(9) . ?
N3 C4 1.347(8) . ?
N3 Nd 2.761(6) 2_776 ?
```

```
C3 H3B 0.9300 . ?
O3 C14 1.243(9) . ?
O4 C16 1.249(9) . ?
N4 C8 1.332(9) . ?
N4 C7 1.336(9) . ?
N4 Nd 2.776(6) 2_777 ?
C4 C4 1.482(13) 2_776 ?
O5 C19 1.244(10) . ?
C5 C6 1.368(11) . ?
C5 H5A 0.9300 . ?
O6 C21 1.236(9) . ?
C6 C7 1.381(10) . ?
C6 H6A 0.9300 . ?
C7 H7A 0.9300 . ?
C8 C8 1.488(13) 2_777 ?
C10 C9 1.386(11) . ?
C10 C11 1.386(11) . ?
C10 H10A 0.9300 . ?
C11 C12 1.528(11) . ?
C12 F2 1.311(11) . ?
C12 F1 1.330(10) . ?
C12 F3 1.332(11) . ?
C9 C13 1.507(12) . ?
C13 F4C 1.284(15) . ?
C13 F5C 1.292(17) . ?
C13 F6A 1.332(13) . ?
C13 F6B 1.335(14) . ?
C13 F4A 1.355(16) . ?
C13 F5A 1.384(16) . ?
C13 F4B 1.403(14) . ?
C13 F6C 1.443(16) . ?
C13 F5B 1.457(15) . ?
C16 C15 1.387(12) . ?
C16 C17 1.545(12) . ?
C17 F8B 1.288(15) . ?
C17 F7A 1.289(16) . ?
C17 F8A 1.346(14) . ?
C17 F9A 1.356(14) . ?
C17 F7B 1.357(19) . ?
C17 F9B 1.367(15) . ?
C14 C15 1.375(12) . ?
C14 C18 1.532(12) . ?
C18 F10A 1.322(13) . ?
C18 F12B 1.328(18) . ?
C18 F10B 1.34(2) . ?
C18 F12A 1.341(15) . ?
C18 F11A 1.343(14) . ?
C18 F11B 1.36(2) . ?
C21 C20 1.377(13) . ?
C21 C22 1.550(14) . ?
C22 F13A 1.27(2) . ?
C22 F14B 1.302(18) . ?
C22 F15B 1.321(16) . ?
C22 F15A 1.329(18) . ?
C22 F14A 1.347(17) . ?
C22 F13B 1.376(19) . ?
C19 C20 1.391(13) . ?
```

```
C19 C23 1.527(13) . ?
C23 F18 1.300(13) . ?
C23 F16 1.313(11) . ?
C23 F17 1.341(12) . ?
C15 H15A 0.9300 . ?
C20 H20A 0.9300 . ?

loop_
 _geom_angle_atom_site_label_1
 _geom_angle_atom_site_label_2
 _geom_angle_atom_site_label_3
 _geom_angle
 _geom_angle_site_symmetry_1
 _geom_angle_site_symmetry_3
 _geom_angle_publ_flag
O1 Nd O2 69.72(17) . . ?
O1 Nd O5 154.74(17) . . ?
O2 Nd O5 85.02(17) . . ?
O1 Nd O4 129.72(17) . . ?
O2 Nd O4 130.13(16) . . ?
O5 Nd O4 67.73(17) . . ?
O1 Nd O3 80.21(17) . . ?
O2 Nd O3 149.91(17) . . ?
O5 Nd O3 125.06(17) . . ?
O4 Nd O3 69.57(17) . . ?
O1 Nd O6 128.29(17) . . ?
O2 Nd O6 133.10(16) . . ?
O5 Nd O6 69.29(17) . . ?
O4 Nd O6 75.87(17) . . ?
O3 Nd O6 68.06(17) . . ?
O1 Nd N1 99.21(17) . . ?
O2 Nd N1 67.46(17) . . ?
O5 Nd N1 69.30(17) . . ?
O4 Nd N1 130.56(17) . . ?
O3 Nd N1 120.45(16) . . ?
O6 Nd N1 66.94(17) . . ?
O1 Nd N2 100.56(17) . . ?
O2 Nd N2 66.31(16) . . ?
O5 Nd N2 68.38(17) . . ?
O4 Nd N2 65.03(17) . . ?
O3 Nd N2 120.11(16) . . ?
O6 Nd N2 130.39(18) . . ?
N1 Nd N2 118.44(17) . . ?
O1 Nd N3 65.12(17) . 2_776 ?
O2 Nd N3 98.85(17) . 2_776 ?
O5 Nd N3 120.52(16) . 2_776 ?
O4 Nd N3 130.84(17) . 2_776 ?
O3 Nd N3 68.41(16) . 2_776 ?
O6 Nd N3 65.61(17) . 2_776 ?
N1 Nd N3 58.58(16) . 2_776 ?
N2 Nd N3 162.98(18) . 2_776 ?
O1 Nd N4 65.60(17) . 2_777 ?
O2 Nd N4 96.33(17) . 2_777 ?
O5 Nd N4 119.87(17) . 2_777 ?
O4 Nd N4 66.44(17) . 2_777 ?
O3 Nd N4 69.19(17) . 2_777 ?
O6 Nd N4 130.35(18) . 2_777 ?
```

```
N1 Nd N4 161.52(18) . 2_777 ?
N2 Nd N4 58.11(17) . 2_777 ?
N3 Nd N4 118.58(17) 2_776 2_777 ?
C9 O1 Nd 137.1(5) . . ?
C4 N1 C1 115.8(6) . . ?
C4 N1 Nd 121.9(4) . . ?
C1 N1 Nd 120.1(4) . . ?
N1 C1 C2 122.4(7) . . ?
N1 C1 H1A 118.8 . . ?
C2 C1 H1A 118.8 . . ?
C11 O2 Nd 136.2(5) . . ?
C8 N2 C5 115.8(6) . . ?
C8 N2 Nd 122.0(4) . . ?
C5 N2 Nd 120.0(5) . . ?
C1 C2 C3 117.0(7) . . ?
C1 C2 H2A 121.5 . . ?
C3 C2 H2A 121.5 . . ?
C3 N3 C4 116.0(6) . . ?
C3 N3 Nd 121.0(4) . 2_776 ?
C4 N3 Nd 121.0(4) . 2_776 ?
N3 C3 C2 122.5(7) . . ?
N3 C3 H3B 118.8 . . ?
C2 C3 H3B 118.8 . . ?
C14 O3 Nd 135.0(5) . . ?
C16 O4 Nd 135.8(5) . . ?
C8 N4 C7 116.2(6) . . ?
C8 N4 Nd 121.1(4) . 2_777 ?
C7 N4 Nd 120.5(5) . 2_777 ?
N1 C4 N3 126.2(6) . . ?
N1 C4 C4 117.0(7) . 2_776 ?
N3 C4 C4 116.8(7) . 2_776 ?
C19 O5 Nd 132.5(5) . . ?
N2 C5 C6 122.0(7) . . ?
N2 C5 H5A 119.0 . . ?
C6 C5 H5A 119.0 . . ?
C21 O6 Nd 135.3(5) . . ?
C5 C6 C7 117.4(7) . . ?
C5 C6 H6A 121.3 . . ?
C7 C6 H6A 121.3 . . ?
N4 C7 C6 121.9(7) . . ?
N4 C7 H7A 119.1 . . ?
C6 C7 H7A 119.1 . . ?
N4 C8 N2 126.5(6) . . ?
N4 C8 C8 117.0(7) . 2_777 ?
N2 C8 C8 116.5(7) . 2_777 ?
C9 C10 C11 121.6(7) . . ?
C9 C10 H10A 119.2 . . ?
C11 C10 H10A 119.2 . . ?
O2 C11 C10 127.8(7) . . ?
O2 C11 C12 114.0(7) . . ?
C10 C11 C12 118.2(7) . . ?
F2 C12 F1 107.0(8) . . ?
F2 C12 F3 107.7(8) . . ?
F1 C12 F3 106.8(7) . . ?
F2 C12 C11 111.3(7) . . ?
F1 C12 C11 113.8(8) . . ?
F3 C12 C11 110.0(7) . . ?
```

```
O1 C9 C10 127.5(7) . . ?
O1 C9 C13 114.0(7) . . ?
C10 C9 C13 118.5(7) . . ?
F4C C13 F5C 106.6(18) . . ?
F4C C13 F6A 118.2(13) . . ?
F5C C13 F6A 40.5(14) . . ?
F4C C13 F6B 115.5(13) . . ?
F5C C13 F6B 67.9(16) . . ?
F6A C13 F6B 27.5(8) . . ?
F4C C13 F4A 38.2(12) . . ?
F5C C13 F4A 125.1(18) . . ?
F6A C13 F4A 108.2(14) . . ?
F6B C13 F4A 90.2(15) . . ?
F4C C13 F5A 64.7(14) . . ?
F5C C13 F5A 58.5(18) . . ?
F6A C13 F5A 97.0(14) . . ?
F6B C13 F5A 121.9(15) . . ?
F4A C13 F5A 102.2(16) . . ?
F4C C13 F4B 23.1(10) . . ?
F5C C13 F4B 127.5(17) . . ?
F6A C13 F4B 125.4(12) . . ?
F6B C13 F4B 111.1(13) . . ?
F4A C13 F4B 21.5(12) . . ?
F5A C13 F4B 86.7(15) . . ?
F4C C13 F6C 102.4(14) . . ?
F5C C13 F6C 103.8(19) . . ?
F6A C13 F6C 63.6(12) . . ?
F6B C13 F6C 36.1(10) . . ?
F4A C13 F6C 65.9(14) . . ?
F5A C13 F6C 149.5(16) . . ?
F4B C13 F6C 86.8(14) . . ?
F4C C13 F5B 86.2(13) . . ?
F5C C13 F5B 42.6(15) . . ?
F6A C13 F5B 83.2(11) . . ?
F6B C13 F5B 110.5(14) . . ?
F4A C13 F5B 122.8(15) . . ?
F5A C13 F5B 21.8(12) . . ?
F4B C13 F5B 108.4(14) . . ?
F6C C13 F5B 145.9(15) . . ?
F4C C13 C9 123.1(12) . . ?
F5C C13 C9 118.7(14) . . ?
F6A C13 C9 118.5(8) . . ?
F6B C13 C9 112.3(10) . . ?
F4A C13 C9 116.2(13) . . ?
F5A C13 C9 111.9(12) . . ?
F4B C13 C9 109.9(10) . . ?
F6C C13 C9 98.4(12) . . ?
F5B C13 C9 104.3(10) . . ?
O4 C16 C15 128.0(7) . . ?
O4 C16 C17 111.9(7) . . ?
C15 C16 C17 120.1(7) . . ?
F8B C17 F7A 130.9(12) . . ?
F8B C17 F8A 28.9(6) . . ?
F7A C17 F8A 105.8(11) . . ?
F8B C17 F9A 82.9(10) . . ?
F7A C17 F9A 107.6(11) . . ?
F8A C17 F9A 105.5(10) . . ?
```

```
F8B C17 F7B 113.8(13) . . ?
F7A C17 F7B 23.6(9) . . ?
F8A C17 F7B 85.8(11) . . ?
F9A C17 F7B 126.2(12) . . ?
F8B C17 F9B 108.6(11) . . ?
F7A C17 F9B 83.9(10) . . ?
F8A C17 F9B 126.1(10) . . ?
F9A C17 F9B 27.1(7) . . ?
F7B C17 F9B 105.9(12) . . ?
F8B C17 C16 107.7(9) . . ?
F7A C17 C16 111.8(9) . . ?
F8A C17 C16 114.2(8) . . ?
F9A C17 C16 111.5(9) . . ?
F7B C17 C16 110.6(10) . . ?
F9B C17 C16 110.2(9) . . ?
O3 C14 C15 128.4(8) . . ?
O3 C14 C18 115.3(7) . . ?
C15 C14 C18 116.4(7) . . ?
F10A C18 F12B 124.8(11) . . ?
F10A C18 F10B 81.5(12) . . ?
F12B C18 F10B 105.2(14) . . ?
F10A C18 F12A 106.2(11) . . ?
F12B C18 F12A 78.4(11) . . ?
F10B C18 F12A 29.2(9) . . ?
F10A C18 F11A 105.6(10) . . ?
F12B C18 F11A 29.4(8) . . ?
F10B C18 F11A 128.3(12) . . ?
F12A C18 F11A 105.6(10) . . ?
F10A C18 F11B 31.2(8) . . ?
F12B C18 F11B 105.7(13) . . ?
F10B C18 F11B 111.1(15) . . ?
F12A C18 F11B 130.7(13) . . ?
F11A C18 F11B 79.6(11) . . ?
F10A C18 C14 115.0(8) . . ?
F12B C18 C14 113.1(11) . . ?
F10B C18 C14 110.8(11) . . ?
F12A C18 C14 112.2(9) . . ?
F11A C18 C14 111.5(10) . . ?
F11B C18 C14 110.8(11) . . ?
O6 C21 C20 127.4(8) . . ?
O6 C21 C22 113.4(8) . . ?
C20 C21 C22 119.2(8) . . ?
F13A C22 F14B 132.3(16) . . ?
F13A C22 F15B 77.7(12) . . ?
F14B C22 F15B 112.0(12) . . ?
F13A C22 F15A 112.6(14) . . ?
F14B C22 F15A 78.9(12) . . ?
F15B C22 F15A 37.2(8) . . ?
F13A C22 F14A 105.1(14) . . ?
F14B C22 F14A 31.4(8) . . ?
F15B C22 F14A 128.5(11) . . ?
F15A C22 F14A 105.0(12) . . ?
F13A C22 F13B 27.0(10) . . ?
F14B C22 F13B 111.7(14) . . ?
F15B C22 F13B 102.3(13) . . ?
F15A C22 F13B 132.3(14) . . ?
F14A C22 F13B 81.2(12) . . ?
```

```
F13A C22 C21 111.4(11) . . ?
F14B C22 C21 106.3(11) . . ?
F15B C22 C21 114.1(10) . . ?
F15A C22 C21 110.4(11) . . ?
F14A C22 C21 112.2(10) . . ?
F13B C22 C21 110.4(10) . . ?
O5 C19 C20 128.5(8) . . ?
O5 C19 C23 113.6(8) . . ?
C20 C19 C23 117.9(8) . . ?
F18 C23 F16 106.3(9) . . ?
F18 C23 F17 106.4(10) . . ?
F16 C23 F17 107.0(9) . . ?
F18 C23 C19 112.0(9) . . ?
F16 C23 C19 115.8(9) . . ?
F17 C23 C19 108.8(9) . . ?
C14 C15 C16 121.7(7) . . ?
C14 C15 H15A 119.2 . . ?
C16 C15 H15A 119.2 . . ?
C21 C20 C19 121.2(8) . . ?
C21 C20 H20A 119.4 . . ?
C19 C20 H20A 119.4 . . ?

loop_
 _geom_torsion_atom_site_label_1
 _geom_torsion_atom_site_label_2
 _geom_torsion_atom_site_label_3
 _geom_torsion_atom_site_label_4
 _geom_torsion
 _geom_torsion_site_symmetry_1
 _geom_torsion_site_symmetry_2
 _geom_torsion_site_symmetry_3
 _geom_torsion_site_symmetry_4
 _geom_torsion_publ_flag
O2 Nd O1 C9 0.8(6) . . . . ?
O5 Nd O1 C9 -0.6(9) . . . . ?
O4 Nd O1 C9 126.7(6) . . . . ?
O3 Nd O1 C9 179.6(7) . . . . ?
O6 Nd O1 C9 -128.7(6) . . . . ?
N1 Nd O1 C9 -60.9(7) . . . . ?
N2 Nd O1 C9 60.5(7) . . . . ?
N3 Nd O1 C9 -109.8(7) 2_776 . . . ?
N4 Nd O1 C9 108.1(7) 2_777 . . . ?
O1 Nd N1 C4 -71.4(5) . . . . ?
O2 Nd N1 C4 -134.8(6) . . . . ?
O5 Nd N1 C4 132.0(5) . . . . ?
O4 Nd N1 C4 100.9(5) . . . . ?
O3 Nd N1 C4 12.6(6) . . . . ?
O6 Nd N1 C4 56.5(5) . . . . ?
N2 Nd N1 C4 -178.8(5) . . . . ?
N3 Nd N1 C4 -18.1(5) 2_776 . . . ?
N4 Nd N1 C4 -104.7(7) 2_777 . . . ?
O1 Nd N1 C1 126.1(6) . . . . ?
O2 Nd N1 C1 62.7(6) . . . . ?
O5 Nd N1 C1 -30.5(5) . . . . ?
O4 Nd N1 C1 -61.6(6) . . . . ?
O3 Nd N1 C1 -149.9(5) . . . . ?
O6 Nd N1 C1 -106.0(6) . . . . ?
```

```
N2 Nd N1 C1 18.7(6) . . . . ?
N3 Nd N1 C1 179.4(6) 2_776 . . . ?
N4 Nd N1 C1 92.8(8) 2_777 . . . ?
C4 N1 C1 C2 -2.9(11) . . . . ?
Nd N1 C1 C2 160.7(6) . . . . ?
O1 Nd O2 C11 2.3(6) . . . . ?
O5 Nd O2 C11 -178.3(6) . . . . ?
O4 Nd O2 C11 -123.1(6) . . . . ?
O3 Nd O2 C11 -0.1(8) . . . . ?
O6 Nd O2 C11 126.3(6) . . . . ?
N1 Nd O2 C11 112.1(7) . . . . ?
N2 Nd O2 C11 -109.7(6) . . . . ?
N3 Nd O2 C11 61.6(6) 2_776 . . . ?
N4 Nd O2 C11 -58.7(6) 2_777 . . . ?
O1 Nd N2 C8 71.3(5) . . . . ?
O2 Nd N2 C8 133.5(6) . . . . ?
O5 Nd N2 C8 -132.4(6) . . . . ?
O4 Nd N2 C8 -57.8(5) . . . . ?
O3 Nd N2 C8 -13.4(6) . . . . ?
O6 Nd N2 C8 -99.2(5) . . . . ?
N1 Nd N2 C8 177.9(5) . . . . ?
N3 Nd N2 C8 102.7(7) 2_776 . . . ?
N4 Nd N2 C8 18.9(5) 2_777 . . . ?
O1 Nd N2 C5 -126.0(5) . . . . ?
O2 Nd N2 C5 -63.9(5) . . . . ?
O5 Nd N2 C5 30.3(5) . . . . ?
O4 Nd N2 C5 104.9(6) . . . . ?
O3 Nd N2 C5 149.2(5) . . . . ?
O6 Nd N2 C5 63.5(6) . . . . ?
N1 Nd N2 C5 -19.4(6) . . . . ?
N3 Nd N2 C5 -94.6(8) 2_776 . . . ?
N4 Nd N2 C5 -178.4(6) 2_777 . . . ?
N1 C1 C2 C3 2.2(12) . . . . ?
C4 N3 C3 C2 -2.4(11) . . . . ?
Nd N3 C3 C2 161.9(6) 2_776 . . . ?
C1 C2 C3 N3 0.5(12) . . . . ?
O1 Nd O3 C14 -152.6(7) . . . . ?
O2 Nd O3 C14 -150.2(6) . . . . ?
O5 Nd O3 C14 27.5(7) . . . . ?
O4 Nd O3 C14 -13.4(7) . . . . ?
O6 Nd O3 C14 69.1(7) . . . . ?
N1 Nd O3 C14 112.4(7) . . . . ?
N2 Nd O3 C14 -56.0(7) . . . . ?
N3 Nd O3 C14 140.4(7) 2_776 . . . ?
N4 Nd O3 C14 -85.1(7) 2_777 . . . ?
O1 Nd O4 C16 60.9(7) . . . . ?
O2 Nd O4 C16 157.3(6) . . . . ?
O5 Nd O4 C16 -140.6(7) . . . . ?
O3 Nd O4 C16 4.0(7) . . . . ?
O6 Nd O4 C16 -67.5(7) . . . . ?
N1 Nd O4 C16 -109.2(7) . . . . ?
N2 Nd O4 C16 143.8(7) . . . . ?
N3 Nd O4 C16 -28.8(8) 2_776 . . . ?
N4 Nd O4 C16 79.4(7) 2_777 . . . ?
C1 N1 C4 N3 0.8(10) . . . . ?
Nd N1 C4 N3 -162.4(5) . . . . ?
C1 N1 C4 C4 -179.0(7) . . . 2_776 ?
```

```
Nd N1 C4 C4 17.8(10) . . . 2_776 ?
C3 N3 C4 N1 1.7(10) . . . . ?
Nd N3 C4 N1 -162.5(5) 2_776 . . . ?
C3 N3 C4 C4 -178.5(7) . . . 2_776 ?
Nd N3 C4 C4 17.3(9) 2_776 . . 2_776 ?
O1 Nd O5 C19 -164.6(6) . . . . ?
O2 Nd O5 C19 -165.9(7) . . . . ?
O4 Nd O5 C19 56.8(6) . . . . ?
O3 Nd O5 C19 15.3(7) . . . . ?
O6 Nd O5 C19 -25.9(6) . . . . ?
N1 Nd O5 C19 -98.1(7) . . . . ?
N2 Nd O5 C19 127.7(7) . . . . ?
N3 Nd O5 C19 -68.5(7) 2_776 . . . ?
N4 Nd O5 C19 99.7(7) 2_777 . . . ?
C8 N2 C5 C6 3.9(11) . . . . ?
Nd N2 C5 C6 -159.8(6) . . . . ?
O1 Nd O6 C21 175.4(7) . . . . ?
O2 Nd O6 C21 77.7(8) . . . . ?
O5 Nd O6 C21 16.5(7) . . . . ?
O4 Nd O6 C21 -54.7(7) . . . . ?
O3 Nd O6 C21 -128.1(8) . . . . ?
N1 Nd O6 C21 92.0(7) . . . . ?
N2 Nd O6 C21 -16.5(8) . . . . ?
N3 Nd O6 C21 156.6(8) 2_776 . . . ?
N4 Nd O6 C21 -95.7(8) 2_777 . . . ?
N2 C5 C6 C7 -1.9(12) . . . . ?
C8 N4 C7 C6 3.9(11) . . . . ?
Nd N4 C7 C6 -159.6(6) 2_777 . . . ?
C5 C6 C7 N4 -2.3(12) . . . . ?
C7 N4 C8 N2 -1.7(11) . . . . ?
Nd N4 C8 N2 161.8(5) 2_777 . . . ?
C7 N4 C8 C8 177.8(7) . . . 2_777 ?
Nd N4 C8 C8 -18.8(10) 2_777 . . 2_777 ?
C5 N2 C8 N4 -2.2(11) . . . . ?
Nd N2 C8 N4 161.1(5) . . . . ?
C5 N2 C8 C8 178.3(7) . . . 2_777 ?
Nd N2 C8 C8 -18.3(10) . . . 2_777 ?
Nd O2 C11 C10 -4.1(12) . . . . ?
Nd O2 C11 C12 176.8(5) . . . . ?
C9 C10 C11 O2 2.0(12) . . . . ?
C9 C10 C11 C12 -178.8(7) . . . . ?
O2 C11 C12 F2 -51.2(10) . . . . ?
C10 C11 C12 F2 129.5(8) . . . . ?
O2 C11 C12 F1 -172.3(7) . . . . ?
C10 C11 C12 F1 8.5(11) . . . . ?
O2 C11 C12 F3 68.0(9) . . . . ?
C10 C11 C12 F3 -111.2(8) . . . . ?
Nd O1 C9 C10 -2.4(12) . . . . ?
Nd O1 C9 C13 179.1(5) . . . . ?
C11 C10 C9 O1 1.1(13) . . . . ?
C11 C10 C9 C13 179.5(8) . . . . ?
O1 C9 C13 F4C 174.8(13) . . . . ?
C10 C9 C13 F4C -3.8(17) . . . . ?
O1 C9 C13 F5C 36(2) . . . . ?
C10 C9 C13 F5C -142(2) . . . . ?
O1 C9 C13 F6A -10.0(14) . . . . ?
C10 C9 C13 F6A 171.4(11) . . . . ?
```

```
O1 C9 C13 F6B -39.8(14) . . . . ?
C10 C9 C13 F6B 141.6(13) . . . . ?
O1 C9 C13 F4A -141.6(14) . . . . ?
C10 C9 C13 F4A 39.7(16) . . . . ?
O1 C9 C13 F5A 101.5(14) . . . . ?
C10 C9 C13 F5A -77.1(15) . . . . ?
O1 C9 C13 F4B -164.0(12) . . . . ?
C10 C9 C13 F4B 17.4(15) . . . . ?
O1 C9 C13 F6C -74.4(13) . . . . ?
C10 C9 C13 F6C 107.0(13) . . . . ?
O1 C9 C13 F5B 79.9(12) . . . . ?
C10 C9 C13 F5B -98.7(12) . . . . ?
Nd O4 C16 C15 1.6(13) . . . . ?
Nd O4 C16 C17 -179.3(5) . . . . ?
O4 C16 C17 F8B 65.3(11) . . . . ?
C15 C16 C17 F8B -115.5(11) . . . . ?
O4 C16 C17 F7A -85.0(11) . . . . ?
C15 C16 C17 F7A 94.3(12) . . . . ?
O4 C16 C17 F8A 35.1(12) . . . . ?
C15 C16 C17 F8A -145.7(9) . . . . ?
O4 C16 C17 F9A 154.5(9) . . . . ?
C15 C16 C17 F9A -26.2(13) . . . . ?
O4 C16 C17 F7B -59.7(12) . . . . ?
C15 C16 C17 F7B 119.6(12) . . . . ?
O4 C16 C17 F9B -176.4(9) . . . . ?
C15 C16 C17 F9B 2.8(13) . . . . ?
Nd O3 C14 C15 18.2(13) . . . . ?
Nd O3 C14 C18 -162.9(5) . . . . ?
O3 C14 C18 F10A 9.6(13) . . . . ?
C15 C14 C18 F10A -171.4(9) . . . . ?
O3 C14 C18 F12B -142.5(12) . . . . ?
C15 C14 C18 F12B 36.6(14) . . . . ?
O3 C14 C18 F10B 99.7(13) . . . . ?
C15 C14 C18 F10B -81.3(14) . . . . ?
O3 C14 C18 F12A 131.1(10) . . . . ?
C15 C14 C18 F12A -49.9(12) . . . . ?
O3 C14 C18 F11A -110.7(10) . . . . ?
C15 C14 C18 F11A 68.3(12) . . . . ?
O3 C14 C18 F11B -24.1(14) . . . . ?
C15 C14 C18 F11B 155.0(12) . . . . ?
Nd O6 C21 C20 -5.0(14) . . . . ?
Nd O6 C21 C22 174.5(6) . . . . ?
O6 C21 C22 F13A -72.1(14) . . . . ?
C20 C21 C22 F13A 107.5(14) . . . . ?
O6 C21 C22 F14B 78.2(13) . . . . ?
C20 C21 C22 F14B -102.2(13) . . . . ?
O6 C21 C22 F15B -157.8(10) . . . . ?
C20 C21 C22 F15B 21.8(15) . . . . ?
O6 C21 C22 F15A 162.1(11) . . . . ?
C20 C21 C22 F15A -18.3(15) . . . . ?
O6 C21 C22 F14A 45.4(13) . . . . ?
C20 C21 C22 F14A -135.0(11) . . . . ?
O6 C21 C22 F13B -43.1(14) . . . . ?
C20 C21 C22 F13B 136.4(12) . . . . ?
Nd O5 C19 C20 25.8(13) . . . . ?
Nd O5 C19 C23 -154.9(6) . . . . ?
O5 C19 C23 F18 -52.1(12) . . . . ?
```

```
C20 C19 C23 F18 127.3(10) . . . . ?
O5 C19 C23 F16 -174.3(9) . . . . ?
C20 C19 C23 F16 5.1(15) . . . . ?
O5 C19 C23 F17 65.3(11) . . . . ?
C20 C19 C23 F17 -115.4(10) . . . . ?
O3 C14 C15 C16 -7.1(14) . . . . ?
C18 C14 C15 C16 174.0(8) . . . . ?
O4 C16 C15 C14 -2.8(14) . . . . ?
C17 C16 C15 C14 178.1(8) . . . . ?
O6 C21 C20 C19 -9.6(15) . . . . ?
C22 C21 C20 C19 170.9(9) . . . . ?
O5 C19 C20 C21 -1.2(15) . . . . ?
C23 C19 C20 C21 179.5(9) . . . . ?

_diffrn_measured_fraction_theta_max    0.969
_diffrn_reflns_theta_full              25.03
_diffrn_measured_fraction_theta_full   0.969
_refine_diff_density_max    1.121
_refine_diff_density_min   -2.456
_refine_diff_density_rms    0.135

#===END


data_x0614

_audit_creation_method            SHELXL-97
_chemical_name_systematic         ?

_chemical_name_common             ?
_chemical_melting_point           ?
_chemical_formula_moiety
'Gd (C5 H1 F6 O2)3(C8 H6 N4)'
_chemical_formula_sum
 'C23 H9 F18 Gd N4 O6'
_chemical_formula_weight          936.59

loop_
 _atom_type_symbol
 _atom_type_description
 _atom_type_scat_dispersion_real
 _atom_type_scat_dispersion_imag
 _atom_type_scat_source
 'C'  'C'   0.0033   0.0016
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'H'  'H'   0.0000   0.0000
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'N'  'N'   0.0061   0.0033
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'O'  'O'   0.0106   0.0060
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'F'  'F'   0.0171   0.0103
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'Gd'  'Gd'  -0.1653   3.9035
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting            triclinic
```

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

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

_cell_length_a                    10.8679(8)
_cell_length_b                    12.3310(11)
_cell_length_c                    14.1256(6)
_cell_angle_alpha                 109.200(7)
_cell_angle_beta                  93.222(5)
_cell_angle_gamma                 115.428(7)
_cell_volume                      1570.68(19)
_cell_formula_units_Z             2
_cell_measurement_temperature     140(2)
_cell_measurement_reflns_used     ?
_cell_measurement_theta_min       ?
_cell_measurement_theta_max       24.71

_exptl_crystal_description        block
_exptl_crystal_colour             colourless
_exptl_crystal_size_max           0.322
_exptl_crystal_size_mid           0.0481
_exptl_crystal_size_min           0.0381
_exptl_crystal_density_meas       ?
_exptl_crystal_density_diffrn     1.980
_exptl_crystal_density_method     'not measured'
_exptl_crystal_F_000              898
_exptl_absorpt_coefficient_mu     2.263
_exptl_absorpt_correction_type    analytical
_exptl_absorpt_correction_T_min   0.69619
_exptl_absorpt_correction_T_max   0.92137
_exptl_absorpt_process_details
;
Multifaceted crystal model (Clark & Reid 1995)
;

_exptl_special_details
;
Note that the absorption correction parameters Tmin and Tmax also
reflect beam corrections, etc. As a result, the numerical values
for Tmin and Tmax may differ from expected values based solely on
absorption effects and crystal size
;


_diffrn_ambient_temperature       140(2)
_diffrn_radiation_wavelength      0.71073
_diffrn_radiation_type            MoK\a
_diffrn_radiation_source          'fine-focus sealed tube'
_diffrn_radiation_monochromator   graphite
_diffrn_measurement_device_type   'CCD detector Xcalibur3'
_diffrn_measurement_method        'phi and omega scans'
_diffrn_detector_area_resol_mean  ?
_diffrn_standards_number          0
```

```
_diffrn_standards_interval_count  0
_diffrn_standards_interval_time   0
_diffrn_standards_decay_%         0.00
_diffrn_reflns_number             30703
_diffrn_reflns_av_R_equivalents   0.0865
_diffrn_reflns_av_sigmaI/netI     0.0448
_diffrn_reflns_limit_h_min        -12
_diffrn_reflns_limit_h_max        12
_diffrn_reflns_limit_k_min        -14
_diffrn_reflns_limit_k_max        14
_diffrn_reflns_limit_l_min        -16
_diffrn_reflns_limit_l_max        16
_diffrn_reflns_theta_min          3.80
_diffrn_reflns_theta_max          24.71
_reflns_number_total              5209
_reflns_number_gt                 5187
_reflns_threshold_expression      >2sigma(I)

_computing_data_collection        'CrysAlis CCD (Oxford Diffraction, 2005)'
_computing_cell_refinement        'CrysAlis RED (Oxford Diffraction, 2005)'
_computing_data_reduction         'CrysAlis RED (Oxford Diffraction, 2005)'
_computing_structure_solution     'Bruker SHELXTL v6.14'
_computing_structure_refinement   'Bruker SHELXTL v6.14'
_computing_molecular_graphics     'Bruker SHELXTL v6.14'
_computing_publication_material   'Bruker SHELXTL v6.14'

_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.

 F-atoms of the CF~3~ groups show disorder as indicated by the
 enlarged displacement parameters.  Multi-component conformations are modeled
 where possible.
;


_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0000P)^2^+21.7287P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     mixed
_refine_ls_extinction_method      SHELXL
_refine_ls_extinction_coef        0.0014(3)
_refine_ls_extinction_expression
 'Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^'
_refine_ls_number_reflns          5209
_refine_ls_number_parameters      450
```

```
_refine_ls_number_restraints      0
_refine_ls_R_factor_all           0.0739
_refine_ls_R_factor_gt            0.0735
_refine_ls_wR_factor_ref          0.1470
_refine_ls_wR_factor_gt           0.1469
_refine_ls_goodness_of_fit_ref    1.463
_refine_ls_restrained_S_all       1.463
_refine_ls_shift/su_max           0.005
_refine_ls_shift/su_mean          0.000

loop_
 _atom_site_label
 _atom_site_type_symbol
 _atom_site_fract_x
 _atom_site_fract_y
 _atom_site_fract_z
 _atom_site_U_iso_or_equiv
 _atom_site_adp_type
 _atom_site_occupancy
 _atom_site_symmetry_multiplicity
 _atom_site_calc_flag
 _atom_site_refinement_flags
 _atom_site_disorder_assembly
 _atom_site_disorder_group
Gd1 Gd 0.97606(5) 0.97510(5) 0.74126(4) 0.01533(18) Uani 1 1 d . . .
O1 O 1.0351(7) 1.2000(7) 0.8077(5) 0.0227(15) Uani 1 1 d . . .
N1 N 1.0981(9) 0.9619(8) 0.5783(7) 0.0229(19) Uani 1 1 d . . .
C1 C 1.1900(12) 0.9146(12) 0.5676(9) 0.029(3) Uani 1 1 d . . .
H1A H 1.2318 0.9105 0.6247 0.035 Uiso 1 1 calc R . .
O2 O 1.2259(7) 1.1042(7) 0.7906(5) 0.0251(16) Uani 1 1 d . . .
N2 N 1.0996(9) 0.9627(8) 0.9088(6) 0.0232(19) Uani 1 1 d . . .
C2 C 1.2239(11) 0.8722(10) 0.4747(9) 0.028(3) Uani 1 1 d . . .
H2A H 1.2862 0.8383 0.4678 0.034 Uiso 1 1 calc R . .
N3 N 1.0775(9) 0.9362(8) 0.4018(6) 0.0218(19) Uani 1 1 d . . .
C3 C 1.1613(12) 0.8823(11) 0.3920(9) 0.030(3) Uani 1 1 d . . .
H3B H 1.1785 0.8503 0.3276 0.036 Uiso 1 1 calc R . .
O3 O 0.7471(7) 0.9608(7) 0.7269(6) 0.0282(17) Uani 1 1 d . . .
O4 O 0.8241(7) 0.8034(7) 0.7913(6) 0.0263(16) Uani 1 1 d . . .
N4 N 1.0755(9) 0.9347(8) 1.0669(6) 0.0226(19) Uani 1 1 d . . .
C4 C 1.0480(10) 0.9722(10) 0.4942(8) 0.020(2) Uiso 1 1 d . . .
O5 O 1.0270(7) 0.7953(7) 0.6930(6) 0.0251(16) Uani 1 1 d . . .
C5 C 1.1890(11) 0.9138(11) 0.9002(9) 0.030(3) Uani 1 1 d . . .
H5A H 1.2304 0.9093 0.8441 0.036 Uiso 1 1 calc R . .
O6 O 0.8173(8) 0.8053(7) 0.5808(5) 0.0282(17) Uani 1 1 d . . .
C6 C 1.2221(12) 0.8704(11) 0.9700(9) 0.031(3) Uani 1 1 d . . .
H6A H 1.2846 0.8368 0.9626 0.037 Uiso 1 1 calc R . .
C7 C 1.1586(12) 0.8785(11) 1.0524(9) 0.031(3) Uani 1 1 d . . .
H7A H 1.1737 0.8443 1.0988 0.037 Uiso 1 1 calc R . .
C8 C 1.0484(10) 0.9709(9) 0.9946(8) 0.019(2) Uiso 1 1 d . . .
C10 C 1.2807(12) 1.3274(10) 0.8596(9) 0.031(3) Uani 1 1 d . . .
H10A H 1.3536 1.4126 0.8887 0.037 Uiso 1 1 calc R . .
C11 C 1.3132(10) 1.2235(10) 0.8328(8) 0.023(2) Uani 1 1 d . . .
C12 C 1.4643(12) 1.2544(12) 0.8538(11) 0.044(3) Uani 1 1 d . . .
F1 F 1.5536(8) 1.3792(7) 0.9084(7) 0.062(2) Uani 1 1 d . . .
F2 F 1.4830(8) 1.1867(9) 0.9029(8) 0.073(3) Uani 1 1 d . . .
F3 F 1.5027(9) 1.2207(10) 0.7646(8) 0.081(3) Uani 1 1 d . . .
C9 C 1.1447(12) 1.3073(10) 0.8445(8) 0.025(2) Uani 1 1 d . . .
```

```
C16 C 0.6981(12) 0.7522(10) 0.7932(8) 0.029(3) Uani 1 1 d . . .
C17 C 0.6561(14) 0.6432(13) 0.8341(10) 0.044(3) Uani 1 1 d . . .
C14 C 0.6297(12) 0.8805(11) 0.7277(9) 0.032(3) Uani 1 1 d . . .
C18 C 0.5060(14) 0.8936(14) 0.6870(12) 0.048(3) Uani 1 1 d . . .
C21 C 0.7820(13) 0.6893(11) 0.5350(9) 0.036(3) Uani 1 1 d . . .
C19 C 0.9531(14) 0.6777(12) 0.6386(10) 0.039(3) Uani 1 1 d . . .
C23 C 1.0101(18) 0.5923(13) 0.6644(12) 0.056(4) Uani 1 1 d . . .
F16 F 0.9414(11) 0.4681(8) 0.6043(8) 0.085(3) Uani 1 1 d . . .
F17 F 0.9991(15) 0.6005(11) 0.7604(8) 0.106(4) Uani 1 1 d . . .
F18 F 1.1401(12) 0.6306(10) 0.6606(11) 0.107(4) Uani 1 1 d . . .
C15 C 0.5965(13) 0.7814(12) 0.7620(10) 0.039(3) Uani 1 1 d . . .
H15A H 0.5043 0.7329 0.7643 0.047 Uiso 1 1 calc R . .
C20 C 0.8371(15) 0.6167(12) 0.5603(11) 0.048(3) Uani 1 1 d . . .
H20A H 0.7950 0.5268 0.5239 0.058 Uiso 1 1 calc R . .
F7 F 0.6536(13) 0.5391(9) 0.7681(8) 0.093(4) Uani 1 1 d . . .
F8 F 0.7452(11) 0.6785(10) 0.9172(7) 0.093(4) Uani 1 1 d . . .
F9 F 0.5335(10) 0.6077(11) 0.8562(10) 0.101(4) Uani 1 1 d . . .
F4 F 1.2290(11) 1.5350(8) 0.8895(10) 0.102(4) Uani 1 1 d . . .
F10 F 0.5412(10) 0.9897(13) 0.6577(12) 0.112(5) Uani 1 1 d . . .
F11 F 0.4295(11) 0.9091(14) 0.7546(9) 0.105(4) Uani 1 1 d . . .
F12 F 0.4172(12) 0.7910(12) 0.6071(9) 0.114(5) Uani 1 1 d . . .
F13 F 0.7017(13) 0.6770(13) 0.3735(8) 0.123(5) Uani 1 1 d . . .
F14 F 0.6225(18) 0.4998(10) 0.3898(11) 0.164(8) Uani 1 1 d . . .
F5 F 1.0983(19) 1.4495(14) 0.9698(13) 0.154(6) Uani 1 1 d . . .
C13 C 1.1214(17) 1.4257(15) 0.8743(13) 0.055(4) Uiso 1 1 d . . .
F6 F 1.0148(15) 1.4082(9) 0.8246(14) 0.172(9) Uani 1 1 d . . .
C22 C 0.6669(16) 0.6219(15) 0.4370(12) 0.054(4) Uiso 1 1 d . . .
F15 F 0.5552(11) 0.6306(14) 0.4560(9) 0.117(5) Uani 1 1 d . . .

loop_
 _atom_site_aniso_label
 _atom_site_aniso_U_11
 _atom_site_aniso_U_22
 _atom_site_aniso_U_33
 _atom_site_aniso_U_23
 _atom_site_aniso_U_13
 _atom_site_aniso_U_12
Gd1 0.0158(3) 0.0182(3) 0.0141(3) 0.00872(19) 0.00559(18) 0.00790(19)
O1 0.028(4) 0.024(4) 0.023(4) 0.011(3) 0.007(3) 0.017(3)
N1 0.021(4) 0.027(5) 0.025(5) 0.015(4) 0.005(4) 0.010(4)
C1 0.033(6) 0.050(7) 0.030(6) 0.029(6) 0.014(5) 0.031(6)
O2 0.017(4) 0.024(4) 0.026(4) 0.009(3) 0.006(3) 0.004(3)
N2 0.019(4) 0.028(5) 0.020(5) 0.009(4) 0.005(4) 0.010(4)
C2 0.030(6) 0.028(6) 0.034(7) 0.016(5) 0.010(5) 0.018(5)
N3 0.033(5) 0.025(5) 0.014(4) 0.011(4) 0.005(4) 0.017(4)
C3 0.037(6) 0.031(6) 0.030(6) 0.021(5) 0.011(5) 0.018(6)
O3 0.019(4) 0.037(4) 0.027(4) 0.014(3) 0.007(3) 0.011(3)
O4 0.022(4) 0.026(4) 0.027(4) 0.014(3) 0.003(3) 0.005(3)
N4 0.024(5) 0.026(5) 0.019(5) 0.010(4) 0.004(4) 0.012(4)
O5 0.032(4) 0.023(4) 0.028(4) 0.012(3) 0.010(3) 0.017(4)
C5 0.030(6) 0.044(7) 0.028(6) 0.018(5) 0.016(5) 0.024(6)
O6 0.033(4) 0.024(4) 0.022(4) 0.009(3) 0.004(3) 0.008(3)
C6 0.031(6) 0.036(7) 0.033(7) 0.013(5) 0.010(5) 0.023(6)
C7 0.038(7) 0.032(6) 0.028(6) 0.014(5) 0.003(5) 0.018(6)
C10 0.030(6) 0.017(5) 0.036(7) 0.011(5) 0.002(5) 0.003(5)
C11 0.015(5) 0.023(6) 0.016(5) 0.008(4) 0.001(4) -0.002(4)
C12 0.019(6) 0.030(7) 0.060(9) 0.014(6) -0.002(6) -0.005(5)
```

```
F1 0.031(4) 0.034(4) 0.086(6) 0.009(4) -0.006(4) -0.002(3)
F2 0.032(4) 0.061(6) 0.119(8) 0.043(6) -0.009(5) 0.013(4)
F3 0.043(5) 0.093(7) 0.081(7) 0.013(6) 0.031(5) 0.025(5)
C9 0.041(7) 0.027(6) 0.021(6) 0.014(5) 0.011(5) 0.024(6)
C16 0.030(6) 0.022(6) 0.022(6) 0.010(5) 0.003(5) 0.000(5)
C17 0.044(8) 0.036(7) 0.042(8) 0.026(6) 0.013(6) 0.002(6)
C14 0.029(6) 0.034(6) 0.033(7) 0.013(5) 0.007(5) 0.014(5)
C18 0.035(7) 0.055(9) 0.065(10) 0.027(8) 0.020(7) 0.026(7)
C21 0.041(7) 0.028(7) 0.027(6) 0.013(5) 0.006(5) 0.007(5)
C19 0.050(8) 0.031(7) 0.033(7) 0.017(6) 0.009(6) 0.012(6)
C23 0.074(11) 0.035(8) 0.051(9) 0.006(7) -0.007(8) 0.030(8)
F16 0.110(8) 0.032(5) 0.099(8) 0.012(5) -0.007(6) 0.037(5)
F17 0.200(13) 0.089(8) 0.073(8) 0.049(6) 0.028(8) 0.093(9)
F18 0.080(8) 0.071(7) 0.186(13) 0.041(8) 0.012(8) 0.059(7)
C15 0.028(6) 0.038(7) 0.047(8) 0.018(6) 0.013(6) 0.011(6)
C20 0.057(9) 0.023(6) 0.048(8) 0.003(6) -0.001(7) 0.015(6)
F7 0.158(11) 0.049(6) 0.087(8) 0.040(5) 0.040(7) 0.050(7)
F8 0.093(7) 0.084(7) 0.063(6) 0.062(6) -0.018(5) -0.012(6)
F9 0.069(7) 0.105(8) 0.185(12) 0.120(9) 0.072(8) 0.037(6)
F4 0.083(7) 0.026(5) 0.182(12) 0.033(6) 0.019(7) 0.019(5)
F10 0.058(6) 0.146(11) 0.217(14) 0.137(11) 0.050(8) 0.069(7)
F11 0.094(8) 0.195(13) 0.109(9) 0.089(9) 0.065(7) 0.114(9)
F12 0.097(8) 0.115(10) 0.105(9) -0.002(8) -0.044(7) 0.071(8)
F13 0.127(10) 0.137(11) 0.042(6) 0.051(7) -0.019(6) 0.001(8)
F14 0.224(16) 0.050(7) 0.119(11) -0.037(7) -0.114(11) 0.051(9)
F5 0.219(17) 0.128(12) 0.188(16) 0.058(11) 0.107(14) 0.140(13)
F6 0.142(11) 0.034(6) 0.264(19) -0.003(8) -0.122(12) 0.041(7)
F15 0.065(7) 0.166(12) 0.074(8) 0.018(8) -0.022(6) 0.042(8)

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

loop_
 _geom_bond_atom_site_label_1
 _geom_bond_atom_site_label_2
 _geom_bond_distance
 _geom_bond_site_symmetry_2
 _geom_bond_publ_flag
Gd1 O1 2.383(7) . ?
Gd1 O2 2.396(7) . ?
Gd1 O5 2.406(7) . ?
Gd1 O3 2.410(7) . ?
Gd1 O4 2.413(7) . ?
Gd1 O6 2.415(7) . ?
Gd1 N1 2.714(8) . ?
Gd1 N3 2.737(8) 2_776 ?
Gd1 N2 2.737(9) . ?
Gd1 N4 2.760(8) 2_777 ?
O1 C9 1.251(13) . ?
N1 C4 1.345(13) . ?
```

```
N1 C1 1.349(13) . ?
C1 C2 1.378(15) . ?
C1 H1A 0.9300 . ?
O2 C11 1.257(12) . ?
N2 C5 1.338(13) . ?
N2 C8 1.354(12) . ?
C2 C3 1.387(16) . ?
C2 H2A 0.9300 . ?
N3 C3 1.327(14) . ?
N3 C4 1.339(13) . ?
N3 Gd1 2.737(8) 2_776 ?
C3 H3B 0.9300 . ?
O3 C14 1.238(13) . ?
O4 C16 1.244(13) . ?
N4 C8 1.306(13) . ?
N4 C7 1.342(14) . ?
N4 Gd1 2.760(8) 2_777 ?
C4 C4 1.465(19) 2_776 ?
O5 C19 1.240(14) . ?
C5 C6 1.365(16) . ?
C5 H5A 0.9300 . ?
O6 C21 1.227(13) . ?
C6 C7 1.384(16) . ?
C6 H6A 0.9300 . ?
C7 H7A 0.9300 . ?
C8 C8 1.496(18) 2_777 ?
C10 C9 1.380(15) . ?
C10 C11 1.413(15) . ?
C10 H10A 0.9300 . ?
C11 C12 1.504(15) . ?
C12 F2 1.315(16) . ?
C12 F1 1.326(14) . ?
C12 F3 1.341(16) . ?
C9 C13 1.516(18) . ?
C16 C15 1.390(17) . ?
C16 C17 1.537(15) . ?
C17 F8 1.296(16) . ?
C17 F7 1.303(16) . ?
C17 F9 1.305(15) . ?
C14 C15 1.374(16) . ?
C14 C18 1.524(17) . ?
C18 F10 1.294(16) . ?
C18 F12 1.302(17) . ?
C18 F11 1.322(15) . ?
C21 C20 1.390(18) . ?
C21 C22 1.526(19) . ?
C19 C20 1.365(18) . ?
C19 C23 1.553(19) . ?
C23 F18 1.294(19) . ?
C23 F16 1.310(15) . ?
C23 F17 1.341(18) . ?
C15 H15A 0.9300 . ?
C20 H20A 0.9300 . ?
F4 C13 1.287(17) . ?
F13 C22 1.275(17) . ?
F14 C22 1.274(17) . ?
F5 C13 1.35(2) . ?
```

```
C13 F6 1.215(18) . ?
C22 F15 1.299(18) . ?

loop_
 _geom_angle_atom_site_label_1
 _geom_angle_atom_site_label_2
 _geom_angle_atom_site_label_3
 _geom_angle
 _geom_angle_site_symmetry_1
 _geom_angle_site_symmetry_3
 _geom_angle_publ_flag
O1 Gd1 O2 71.2(2) . . ?
O1 Gd1 O5 154.7(2) . . ?
O2 Gd1 O5 83.5(2) . . ?
O1 Gd1 O3 78.8(2) . . ?
O2 Gd1 O3 150.0(3) . . ?
O5 Gd1 O3 126.5(3) . . ?
O1 Gd1 O4 129.4(2) . . ?
O2 Gd1 O4 129.7(2) . . ?
O5 Gd1 O4 67.9(2) . . ?
O3 Gd1 O4 70.6(2) . . ?
O1 Gd1 O6 127.8(2) . . ?
O2 Gd1 O6 132.3(2) . . ?
O5 Gd1 O6 70.2(3) . . ?
O3 Gd1 O6 68.4(3) . . ?
O4 Gd1 O6 76.1(2) . . ?
O1 Gd1 N1 100.1(2) . . ?
O2 Gd1 N1 66.8(2) . . ?
O5 Gd1 N1 68.7(2) . . ?
O3 Gd1 N1 120.6(2) . . ?
O4 Gd1 N1 130.0(3) . . ?
O6 Gd1 N1 66.8(2) . . ?
O1 Gd1 N3 64.9(2) . 2_776 ?
O2 Gd1 N3 98.7(3) . 2_776 ?
O5 Gd1 N3 120.8(2) . 2_776 ?
O3 Gd1 N3 68.1(3) . 2_776 ?
O4 Gd1 N3 131.3(3) . 2_776 ?
O6 Gd1 N3 65.6(2) . 2_776 ?
N1 Gd1 N3 58.9(2) . 2_776 ?
O1 Gd1 N2 100.3(2) . . ?
O2 Gd1 N2 65.7(2) . . ?
O5 Gd1 N2 68.1(3) . . ?
O3 Gd1 N2 120.9(2) . . ?
O4 Gd1 N2 65.5(2) . . ?
O6 Gd1 N2 131.2(3) . . ?
N1 Gd1 N2 117.7(2) . . ?
N3 Gd1 N2 162.1(3) 2_776 . ?
O1 Gd1 N4 65.3(2) . 2_777 ?
O2 Gd1 N4 97.3(2) . 2_777 ?
O5 Gd1 N4 119.8(2) . 2_777 ?
O3 Gd1 N4 68.9(2) . 2_777 ?
O4 Gd1 N4 66.4(2) . 2_777 ?
O6 Gd1 N4 130.1(3) . 2_777 ?
N1 Gd1 N4 162.0(3) . 2_777 ?
N3 Gd1 N4 118.5(2) 2_776 2_777 ?
N2 Gd1 N4 58.4(2) . 2_777 ?
C9 O1 Gd1 136.8(6) . . ?
```

```
C4 N1 C1 115.7(9) . . ?
C4 N1 Gd1 122.0(6) . . ?
C1 N1 Gd1 120.6(7) . . ?
N1 C1 C2 122.1(10) . . ?
N1 C1 H1A 118.9 . . ?
C2 C1 H1A 118.9 . . ?
C11 O2 Gd1 136.7(7) . . ?
C5 N2 C8 114.9(9) . . ?
C5 N2 Gd1 120.6(7) . . ?
C8 N2 Gd1 122.2(6) . . ?
C1 C2 C3 117.1(10) . . ?
C1 C2 H2A 121.5 . . ?
C3 C2 H2A 121.5 . . ?
C3 N3 C4 116.6(9) . . ?
C3 N3 Gd1 120.9(7) . 2_776 ?
C4 N3 Gd1 120.7(6) . 2_776 ?
N3 C3 C2 122.2(10) . . ?
N3 C3 H3B 118.9 . . ?
C2 C3 H3B 118.9 . . ?
C14 O3 Gd1 135.1(7) . . ?
C16 O4 Gd1 135.6(7) . . ?
C8 N4 C7 116.4(9) . . ?
C8 N4 Gd1 121.2(6) . 2_777 ?
C7 N4 Gd1 120.5(7) . 2_777 ?
N3 C4 N1 126.1(9) . . ?
N3 C4 C4 117.6(11) . 2_776 ?
N1 C4 C4 116.4(11) . 2_776 ?
C19 O5 Gd1 131.7(8) . . ?
N2 C5 C6 122.9(10) . . ?
N2 C5 H5A 118.6 . . ?
C6 C5 H5A 118.6 . . ?
C21 O6 Gd1 135.0(8) . . ?
C5 C6 C7 117.0(10) . . ?
C5 C6 H6A 121.5 . . ?
C7 C6 H6A 121.5 . . ?
N4 C7 C6 121.5(10) . . ?
N4 C7 H7A 119.2 . . ?
C6 C7 H7A 119.2 . . ?
N4 C8 N2 127.0(9) . . ?
N4 C8 C8 118.3(11) . 2_777 ?
N2 C8 C8 114.7(11) . 2_777 ?
C9 C10 C11 122.5(10) . . ?
C9 C10 H10A 118.8 . . ?
C11 C10 H10A 118.8 . . ?
O2 C11 C10 125.6(10) . . ?
O2 C11 C12 115.5(10) . . ?
C10 C11 C12 118.9(9) . . ?
F2 C12 F1 107.5(11) . . ?
F2 C12 F3 106.8(12) . . ?
F1 C12 F3 106.9(10) . . ?
F2 C12 C11 110.8(10) . . ?
F1 C12 C11 114.6(11) . . ?
F3 C12 C11 109.9(11) . . ?
O1 C9 C10 127.1(9) . . ?
O1 C9 C13 114.7(10) . . ?
C10 C9 C13 118.2(11) . . ?
O4 C16 C15 127.8(10) . . ?
```

```
O4 C16 C17 113.1(11) . . ?
C15 C16 C17 119.1(10) . . ?
F8 C17 F7 105.8(13) . . ?
F8 C17 F9 106.7(12) . . ?
F7 C17 F9 107.3(12) . . ?
F8 C17 C16 111.6(10) . . ?
F7 C17 C16 111.7(10) . . ?
F9 C17 C16 113.4(12) . . ?
O3 C14 C15 128.2(11) . . ?
O3 C14 C18 116.3(11) . . ?
C15 C14 C18 115.6(11) . . ?
F10 C18 F12 104.9(14) . . ?
F10 C18 F11 106.3(12) . . ?
F12 C18 F11 104.2(13) . . ?
F10 C18 C14 114.0(11) . . ?
F12 C18 C14 113.2(11) . . ?
F11 C18 C14 113.3(12) . . ?
O6 C21 C20 127.9(12) . . ?
O6 C21 C22 113.8(11) . . ?
C20 C21 C22 118.3(11) . . ?
O5 C19 C20 129.9(12) . . ?
O5 C19 C23 112.1(11) . . ?
C20 C19 C23 117.9(11) . . ?
F18 C23 F16 106.5(13) . . ?
F18 C23 F17 108.4(14) . . ?
F16 C23 F17 106.5(13) . . ?
F18 C23 C19 112.5(13) . . ?
F16 C23 C19 114.6(12) . . ?
F17 C23 C19 108.1(12) . . ?
C14 C15 C16 121.3(11) . . ?
C14 C15 H15A 119.4 . . ?
C16 C15 H15A 119.4 . . ?
C19 C20 C21 119.8(12) . . ?
C19 C20 H20A 120.1 . . ?
C21 C20 H20A 120.1 . . ?
F6 C13 F4 113.8(15) . . ?
F6 C13 F5 102.5(17) . . ?
F4 C13 F5 100.4(15) . . ?
F6 C13 C9 114.7(13) . . ?
F4 C13 C9 116.2(13) . . ?
F5 C13 C9 106.7(12) . . ?
F14 C22 F13 109.0(15) . . ?
F14 C22 F15 104.0(15) . . ?
F13 C22 F15 103.5(15) . . ?
F14 C22 C21 115.7(13) . . ?
F13 C22 C21 112.2(13) . . ?
F15 C22 C21 111.6(12) . . ?

loop_
 _geom_torsion_atom_site_label_1
 _geom_torsion_atom_site_label_2
 _geom_torsion_atom_site_label_3
 _geom_torsion_atom_site_label_4
 _geom_torsion
 _geom_torsion_site_symmetry_1
 _geom_torsion_site_symmetry_2
 _geom_torsion_site_symmetry_3
```

```
 _geom_torsion_site_symmetry_4
 _geom_torsion_publ_flag
O2 Gd1 O1 C9 -0.8(9) . . . . ?
O5 Gd1 O1 C9 -1.0(12) . . . . ?
O3 Gd1 O1 C9 178.7(9) . . . . ?
O4 Gd1 O1 C9 125.6(9) . . . . ?
O6 Gd1 O1 C9 -130.3(9) . . . . ?
N1 Gd1 O1 C9 -61.9(9) . . . . ?
N3 Gd1 O1 C9 -110.4(9) 2_776 . . . ?
N2 Gd1 O1 C9 58.9(9) . . . . ?
N4 Gd1 O1 C9 106.9(10) 2_777 . . . ?
O1 Gd1 N1 C4 -70.0(8) . . . . ?
O2 Gd1 N1 C4 -134.3(8) . . . . ?
O5 Gd1 N1 C4 133.7(8) . . . . ?
O3 Gd1 N1 C4 12.9(9) . . . . ?
O4 Gd1 N1 C4 102.5(8) . . . . ?
O6 Gd1 N1 C4 56.9(7) . . . . ?
N3 Gd1 N1 C4 -17.6(7) 2_776 . . . ?
N2 Gd1 N1 C4 -177.4(7) . . . . ?
N4 Gd1 N1 C4 -104.7(10) 2_777 . . . ?
O1 Gd1 N1 C1 125.7(8) . . . . ?
O2 Gd1 N1 C1 61.4(8) . . . . ?
O5 Gd1 N1 C1 -30.6(8) . . . . ?
O3 Gd1 N1 C1 -151.4(8) . . . . ?
O4 Gd1 N1 C1 -61.8(9) . . . . ?
O6 Gd1 N1 C1 -107.4(9) . . . . ?
N3 Gd1 N1 C1 178.1(9) 2_776 . . . ?
N2 Gd1 N1 C1 18.3(9) . . . . ?
N4 Gd1 N1 C1 91.0(12) 2_777 . . . ?
C4 N1 C1 C2 -3.8(16) . . . . ?
Gd1 N1 C1 C2 161.5(8) . . . . ?
O1 Gd1 O2 C11 3.8(9) . . . . ?
O5 Gd1 O2 C11 -176.3(9) . . . . ?
O3 Gd1 O2 C11 2.7(12) . . . . ?
O4 Gd1 O2 C11 -122.3(9) . . . . ?
O6 Gd1 O2 C11 128.1(9) . . . . ?
N1 Gd1 O2 C11 114.1(9) . . . . ?
N3 Gd1 O2 C11 63.4(9) 2_776 . . . ?
N2 Gd1 O2 C11 -107.4(9) . . . . ?
N4 Gd1 O2 C11 -57.0(9) 2_777 . . . ?
O1 Gd1 N2 C5 -126.7(8) . . . . ?
O2 Gd1 N2 C5 -62.9(8) . . . . ?
O5 Gd1 N2 C5 29.8(8) . . . . ?
O3 Gd1 N2 C5 150.3(8) . . . . ?
O4 Gd1 N2 C5 104.6(8) . . . . ?
O6 Gd1 N2 C5 63.1(9) . . . . ?
N1 Gd1 N2 C5 -19.4(9) . . . . ?
N3 Gd1 N2 C5 -93.7(11) 2_776 . . . ?
N4 Gd1 N2 C5 -179.1(9) 2_777 . . . ?
O1 Gd1 N2 C8 71.5(8) . . . . ?
O2 Gd1 N2 C8 135.2(8) . . . . ?
O5 Gd1 N2 C8 -132.1(8) . . . . ?
O3 Gd1 N2 C8 -11.6(9) . . . . ?
O4 Gd1 N2 C8 -57.3(7) . . . . ?
O6 Gd1 N2 C8 -98.8(8) . . . . ?
N1 Gd1 N2 C8 178.7(7) . . . . ?
N3 Gd1 N2 C8 104.4(10) 2_776 . . . ?
```

```
N4 Gd1 N2 C8 19.1(7) 2_777 . . . ?
N1 C1 C2 C3 1.2(17) . . . . ?
C4 N3 C3 C2 -4.7(16) . . . . ?
Gd1 N3 C3 C2 160.3(8) 2_776 . . . ?
C1 C2 C3 N3 3.3(17) . . . . ?
O1 Gd1 O3 C14 -152.1(10) . . . . ?
O2 Gd1 O3 C14 -151.1(9) . . . . ?
O5 Gd1 O3 C14 27.7(11) . . . . ?
O4 Gd1 O3 C14 -13.0(10) . . . . ?
O6 Gd1 O3 C14 69.3(10) . . . . ?
N1 Gd1 O3 C14 112.7(10) . . . . ?
N3 Gd1 O3 C14 140.6(11) 2_776 . . . ?
N2 Gd1 O3 C14 -56.7(11) . . . . ?
N4 Gd1 O3 C14 -84.4(10) 2_777 . . . ?
O1 Gd1 O4 C16 60.5(11) . . . . ?
O2 Gd1 O4 C16 158.5(9) . . . . ?
O5 Gd1 O4 C16 -141.3(10) . . . . ?
O3 Gd1 O4 C16 4.3(10) . . . . ?
O6 Gd1 O4 C16 -67.3(10) . . . . ?
N1 Gd1 O4 C16 -109.9(10) . . . . ?
N3 Gd1 O4 C16 -29.0(11) 2_776 . . . ?
N2 Gd1 O4 C16 143.6(10) . . . . ?
N4 Gd1 O4 C16 79.0(10) 2_777 . . . ?
C3 N3 C4 N1 1.8(16) . . . . ?
Gd1 N3 C4 N1 -163.2(8) 2_776 . . . ?
C3 N3 C4 C4 -178.0(11) . . . 2_776 ?
Gd1 N3 C4 C4 17.0(15) 2_776 . . 2_776 ?
C1 N1 C4 N3 2.3(15) . . . . ?
Gd1 N1 C4 N3 -162.7(8) . . . . ?
C1 N1 C4 C4 -177.9(11) . . . 2_776 ?
Gd1 N1 C4 C4 17.1(15) . . . 2_776 ?
O1 Gd1 O5 C19 -164.5(9) . . . . ?
O2 Gd1 O5 C19 -164.7(10) . . . . ?
O3 Gd1 O5 C19 15.9(10) . . . . ?
O4 Gd1 O5 C19 57.6(9) . . . . ?
O6 Gd1 O5 C19 -25.1(9) . . . . ?
N1 Gd1 O5 C19 -97.1(10) . . . . ?
N3 Gd1 O5 C19 -68.4(10) 2_776 . . . ?
N2 Gd1 O5 C19 128.9(10) . . . . ?
N4 Gd1 O5 C19 100.6(10) 2_777 . . . ?
C8 N2 C5 C6 2.6(16) . . . . ?
Gd1 N2 C5 C6 -160.5(9) . . . . ?
O1 Gd1 O6 C21 175.9(10) . . . . ?
O2 Gd1 O6 C21 77.1(11) . . . . ?
O5 Gd1 O6 C21 16.5(10) . . . . ?
O3 Gd1 O6 C21 -128.9(11) . . . . ?
O4 Gd1 O6 C21 -54.6(10) . . . . ?
N1 Gd1 O6 C21 91.1(11) . . . . ?
N3 Gd1 O6 C21 156.2(11) 2_776 . . . ?
N2 Gd1 O6 C21 -16.2(12) . . . . ?
N4 Gd1 O6 C21 -96.2(11) 2_777 . . . ?
N2 C5 C6 C7 0.0(18) . . . . ?
C8 N4 C7 C6 5.7(16) . . . . ?
Gd1 N4 C7 C6 -159.1(9) 2_777 . . . ?
C5 C6 C7 N4 -4.4(17) . . . . ?
C7 N4 C8 N2 -3.0(16) . . . . ?
Gd1 N4 C8 N2 161.7(8) 2_777 . . . ?
```

```
C7 N4 C8 C8 177.6(11) . . . 2_777 ?
Gd1 N4 C8 C8 -17.7(15) 2_777 . . 2_777 ?
C5 N2 C8 N4 -1.1(16) . . . . ?
Gd1 N2 C8 N4 161.7(8) . . . . ?
C5 N2 C8 C8 178.3(11) . . . 2_777 ?
Gd1 N2 C8 C8 -18.9(14) . . . 2_777 ?
Gd1 O2 C11 C10 -5.1(16) . . . . ?
Gd1 O2 C11 C12 175.8(8) . . . . ?
C9 C10 C11 O2 2.0(17) . . . . ?
C9 C10 C11 C12 -178.9(11) . . . . ?
O2 C11 C12 F2 -50.9(14) . . . . ?
C10 C11 C12 F2 129.9(12) . . . . ?
O2 C11 C12 F1 -172.8(10) . . . . ?
C10 C11 C12 F1 8.1(17) . . . . ?
O2 C11 C12 F3 66.9(13) . . . . ?
C10 C11 C12 F3 -112.3(12) . . . . ?
Gd1 O1 C9 C10 -0.8(17) . . . . ?
Gd1 O1 C9 C13 179.0(8) . . . . ?
C11 C10 C9 O1 0.8(18) . . . . ?
C11 C10 C9 C13 -178.9(11) . . . . ?
Gd1 O4 C16 C15 1.2(18) . . . . ?
Gd1 O4 C16 C17 -179.8(7) . . . . ?
O4 C16 C17 F8 46.2(16) . . . . ?
C15 C16 C17 F8 -134.7(13) . . . . ?
O4 C16 C17 F7 -71.9(15) . . . . ?
C15 C16 C17 F7 107.1(14) . . . . ?
O4 C16 C17 F9 166.7(12) . . . . ?
C15 C16 C17 F9 -14.2(18) . . . . ?
Gd1 O3 C14 C15 17.0(19) . . . . ?
Gd1 O3 C14 C18 -163.8(8) . . . . ?
O3 C14 C18 F10 -0.9(19) . . . . ?
C15 C14 C18 F10 178.4(13) . . . . ?
O3 C14 C18 F12 119.0(14) . . . . ?
C15 C14 C18 F12 -61.7(17) . . . . ?
O3 C14 C18 F11 -122.6(13) . . . . ?
C15 C14 C18 F11 56.7(17) . . . . ?
Gd1 O6 C21 C20 -6(2) . . . . ?
Gd1 O6 C21 C22 175.8(8) . . . . ?
Gd1 O5 C19 C20 26(2) . . . . ?
Gd1 O5 C19 C23 -156.8(9) . . . . ?
O5 C19 C23 F18 -53.7(17) . . . . ?
C20 C19 C23 F18 124.0(15) . . . . ?
O5 C19 C23 F16 -175.5(13) . . . . ?
C20 C19 C23 F16 2(2) . . . . ?
O5 C19 C23 F17 65.9(16) . . . . ?
C20 C19 C23 F17 -116.4(14) . . . . ?
O3 C14 C15 C16 -6(2) . . . . ?
C18 C14 C15 C16 174.7(11) . . . . ?
O4 C16 C15 C14 -3(2) . . . . ?
C17 C16 C15 C14 178.0(11) . . . . ?
O5 C19 C20 C21 -3(2) . . . . ?
C23 C19 C20 C21 179.6(13) . . . . ?
O6 C21 C20 C19 -7(2) . . . . ?
C22 C21 C20 C19 170.6(13) . . . . ?
O1 C9 C13 F6 -28(2) . . . . ?
C10 C9 C13 F6 151.5(16) . . . . ?
O1 C9 C13 F4 -164.5(12) . . . . ?
```

```
C10 C9 C13 F4 15.3(19) . . . . ?
O1 C9 C13 F5 84.5(15) . . . . ?
C10 C9 C13 F5 -95.7(15) . . . . ?
O6 C21 C22 F14 -176.7(15) . . . . ?
C20 C21 C22 F14 5(2) . . . . ?
O6 C21 C22 F13 57.4(18) . . . . ?
C20 C21 C22 F13 -120.6(15) . . . . ?
O6 C21 C22 F15 -58.1(17) . . . . ?
C20 C21 C22 F15 123.8(15) . . . . ?

_diffrn_measured_fraction_theta_max    0.971
_diffrn_reflns_theta_full              24.71
_diffrn_measured_fraction_theta_full   0.971
_refine_diff_density_max    1.635
_refine_diff_density_min   -4.075
_refine_diff_density_rms    0.154

#===END


data_x0612b2

_audit_creation_method            SHELXL-97
_chemical_name_systematic         ?

_chemical_name_common             ?
_chemical_melting_point           ?
_chemical_formula_moiety
 'Tb (C5 H1 F6 O2)3 (C8 H6 N4), (C4 H3 N2), O0.25 H0.5'
_chemical_formula_sum
 'C27 H13.50 F18 N6 O7.25 Tb'
_chemical_formula_weight          1038.86

loop_
 _atom_type_symbol
 _atom_type_description
 _atom_type_scat_dispersion_real
 _atom_type_scat_dispersion_imag
 _atom_type_scat_source
 'C'  'C'   0.0033   0.0016
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'H'  'H'   0.0000   0.0000
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'N'  'N'   0.0061   0.0033
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'O'  'O'   0.0106   0.0060
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'F'  'F'   0.0171   0.0103
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'Tb'  'Tb'  -0.1723   4.1537
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting            monoclinic
_symmetry_space_group_name_H-M    C2/c
_symmetry_space_group_name_Hall   'C 1 2/c 1'

loop_
```

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

_cell_length_a                    30.002(2)
_cell_length_b                    17.3996(9)
_cell_length_c                    21.2343(13)
_cell_angle_alpha                 90.00
_cell_angle_beta                  133.588(10)
_cell_angle_gamma                 90.00
_cell_volume                      8028.9(8)
_cell_formula_units_Z             8
_cell_measurement_temperature     140(2)
_cell_measurement_reflns_used     ?
_cell_measurement_theta_min       ?
_cell_measurement_theta_max       24.41

_exptl_crystal_description        block
_exptl_crystal_colour             colourless
_exptl_crystal_size_max           0.382
_exptl_crystal_size_mid           0.341
_exptl_crystal_size_min           0.176
_exptl_crystal_density_meas       ?
_exptl_crystal_density_diffrn     1.719
_exptl_crystal_density_method     'not measured'
_exptl_crystal_F_000              4020
_exptl_absorpt_coefficient_mu     1.893
_exptl_absorpt_correction_type    analytical
_exptl_absorpt_correction_T_min   0.54243
_exptl_absorpt_correction_T_max   0.74073
_exptl_absorpt_process_details
;
Multifaceted crystal model (Clark & Reid 1995)
;

_exptl_special_details
;
Note that the absorption correction parameters Tmin and Tmax also
reflect beam corrections, etc. As a result, the numerical values
for Tmin and Tmax may differ from expected values based solely on
absorption effects and crystal size
;

_diffrn_ambient_temperature       140(2)
_diffrn_radiation_wavelength      0.71073
_diffrn_radiation_type            MoK\a
_diffrn_radiation_source          'fine-focus sealed tube'
_diffrn_radiation_monochromator   graphite
_diffrn_measurement_device_type   'CCD detector Xcalibur3'
_diffrn_measurement_method        'phi and omega scans'
_diffrn_detector_area_resol_mean  ?
```

```
_diffrn_standards_number          0
_diffrn_standards_interval_count  0
_diffrn_standards_interval_time   0
_diffrn_standards_decay_%         0.00
_diffrn_reflns_number             74587
_diffrn_reflns_av_R_equivalents   0.0426
_diffrn_reflns_av_sigmaI/netI     0.0145
_diffrn_reflns_limit_h_min        -34
_diffrn_reflns_limit_h_max        34
_diffrn_reflns_limit_k_min        -20
_diffrn_reflns_limit_k_max        20
_diffrn_reflns_limit_l_min        -24
_diffrn_reflns_limit_l_max        24
_diffrn_reflns_theta_min          4.02
_diffrn_reflns_theta_max          24.41
_reflns_number_total              6593
_reflns_number_gt                 6593
_reflns_threshold_expression      >2sigma(I)

_computing_data_collection        'CrysAlis CCD (Oxford Diffraction, 2005)'
_computing_cell_refinement        'CrysAlis RED (Oxford Diffraction, 2005)'
_computing_data_reduction         'CrysAlis RED (Oxford Diffraction, 2005)'
_computing_structure_solution     'Bruker SHELXTL v6.14'
_computing_structure_refinement   'Bruker SHELXTL v6.14'
_computing_molecular_graphics     'Bruker SHELXTL v6.14'
_computing_publication_material   'Bruker SHELXTL v6.14'

_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.

 F-atoms of the CF~3~ groups show disorder as indicated by the
 enlarged displacement parameters.  Multi-component conformations are modeled
 where possible.
;

_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.1125P)^2^+291.3799P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     mixed
_refine_ls_extinction_method      none
_refine_ls_extinction_coef        ?
_refine_ls_number_reflns          6593
_refine_ls_number_parameters      499
_refine_ls_number_restraints      0
_refine_ls_R_factor_all           0.0901
```

```
_refine_ls_R_factor_gt            0.0901
_refine_ls_wR_factor_ref          0.2338
_refine_ls_wR_factor_gt           0.2338
_refine_ls_goodness_of_fit_ref    1.127
_refine_ls_restrained_S_all       1.127
_refine_ls_shift/su_max           0.036
_refine_ls_shift/su_mean          0.002

loop_
 _atom_site_label
 _atom_site_type_symbol
 _atom_site_fract_x
 _atom_site_fract_y
 _atom_site_fract_z
 _atom_site_U_iso_or_equiv
 _atom_site_adp_type
 _atom_site_occupancy
 _atom_site_symmetry_multiplicity
 _atom_site_calc_flag
 _atom_site_refinement_flags
 _atom_site_disorder_assembly
 _atom_site_disorder_group
Tb Tb 0.24011(2) 0.85379(3) 0.76134(3) 0.0288(2) Uani 1 1 d . . .
O5 O 0.1952(3) 0.8631(4) 0.8182(5) 0.0338(17) Uani 1 1 d . . .
O1 O 0.2213(4) 0.8489(5) 0.6325(5) 0.043(2) Uani 1 1 d . . .
O2 O 0.1408(4) 0.7969(4) 0.6486(5) 0.0374(18) Uani 1 1 d . . .
O3 O 0.3238(4) 0.8273(5) 0.9106(5) 0.0379(17) Uani 1 1 d . . .
C5 C 0.2659(5) 0.6551(6) 0.8097(7) 0.034(2) Uani 1 1 d . . .
N1 N 0.3033(4) 0.7447(5) 0.7687(6) 0.0322(19) Uani 1 1 d . . .
O4 O 0.3267(4) 0.9099(4) 0.7952(5) 0.0383(18) Uani 1 1 d . . .
N3 N 0.2296(4) 0.7128(5) 0.7964(6) 0.033(2) Uani 1 1 d . . .
C4 C 0.3063(5) 0.6732(6) 0.7955(7) 0.034(2) Uani 1 1 d . . .
N4 N 0.2666(5) 0.5828(5) 0.8310(6) 0.040(2) Uani 1 1 d . . .
C1 C 0.3353(6) 0.7562(7) 0.7469(8) 0.044(3) Uani 1 1 d . . .
H1A H 0.3326 0.8035 0.7241 0.053 Uiso 1 1 calc R . .
O6 O 0.2729(4) 0.9740(5) 0.8426(5) 0.046(2) Uani 1 1 d . . .
N2 N 0.3412(5) 0.6164(6) 0.8081(8) 0.049(3) Uani 1 1 d . . .
C6 C 0.2264(6) 0.5663(7) 0.8382(8) 0.049(3) Uani 1 1 d . . .
H6A H 0.2254 0.5167 0.8534 0.059 Uiso 1 1 calc R . .
C11 C 0.1067(6) 0.7881(7) 0.5689(8) 0.044(3) Uani 1 1 d . . .
C14 C 0.3807(8) 0.8510(9) 0.9675(11) 0.066(4) Uani 1 1 d . . .
C10 C 0.1186(7) 0.8060(8) 0.5180(8) 0.054(3) Uani 1 1 d . . .
H10A H 0.0879 0.7993 0.4581 0.065 Uiso 1 1 calc R . .
C9 C 0.1757(6) 0.8340(8) 0.5556(8) 0.048(3) Uani 1 1 d . . .
C8 C 0.1890(6) 0.6932(7) 0.8026(9) 0.046(3) Uani 1 1 d . . .
H8A H 0.1622 0.7301 0.7923 0.055 Uiso 1 1 calc R . .
C2 C 0.3721(6) 0.7000(8) 0.7573(10) 0.055(3) Uani 1 1 d . . .
H2B H 0.3944 0.7087 0.7422 0.066 Uiso 1 1 calc R . .
C7 C 0.1872(6) 0.6201(8) 0.8239(10) 0.052(3) Uani 1 1 d . . .
H7A H 0.1595 0.6071 0.8286 0.062 Uiso 1 1 calc R . .
C3 C 0.3749(7) 0.6319(9) 0.7898(11) 0.063(4) Uani 1 1 d . . .
H3A H 0.4012 0.5943 0.8000 0.075 Uiso 1 1 calc R . .
C22 C 0.3083(7) 1.0796(7) 0.9361(9) 0.052(3) Uani 1 1 d . . .
C16 C 0.3820(9) 0.9188(10) 0.8675(13) 0.076(5) Uani 1 1 d . . .
C21 C 0.2791(17) 1.0073(12) 0.8879(12) 0.21(2) Uani 1 1 d . . .
C12 C 0.0456(7) 0.7495(9) 0.5235(9) 0.061(4) Uani 1 1 d . . .
C15 C 0.4130(8) 0.8935(10) 0.9538(11) 0.073(4) Uani 1 1 d . . .
```

```
H15A H 0.4543 0.9053 1.0003 0.088 Uiso 1 1 calc R . .
C17 C 0.4202(7) 0.9672(10) 0.8575(11) 0.065(4) Uani 1 1 d . . .
C19 C 0.2054(8) 0.8992(16) 0.8769(12) 0.101(8) Uani 1 1 d . . .
F6 F 0.4122(6) 0.7583(8) 1.0677(7) 0.113(4) Uani 1 1 d . . .
C20 C 0.2368(10) 0.9754(12) 0.9098(16) 0.091(6) Uani 1 1 d . . .
H20A H 0.2334 1.0059 0.9423 0.109 Uiso 1 1 calc R . .
O7 O 0.1706(4) 0.9521(4) 0.6711(5) 0.0412(19) Uani 1 1 d . . .
N1S N 1.0457(5) 0.9416(7) 0.5961(7) 0.051(3) Uani 1 1 d . . .
C2S C 0.9974(6) 0.9788(8) 0.5273(8) 0.045(3) Uani 1 1 d . . .
N3S N 0.9427(5) 0.9816(9) 0.5021(8) 0.065(4) Uani 1 1 d . . .
C4S C 1.0391(7) 0.9027(10) 0.6428(10) 0.065(4) Uani 1 1 d . . .
H4SA H 1.0727 0.8766 0.6923 0.078 Uiso 1 1 calc R . .
C5S C 0.9838(8) 0.9003(11) 0.6196(10) 0.073(5) Uani 1 1 d . . .
H5SA H 0.9791 0.8726 0.6521 0.088 Uiso 1 1 calc R . .
C6S C 0.9378(7) 0.9382(14) 0.5501(11) 0.091(7) Uani 1 1 d . . .
H6SA H 0.8997 0.9353 0.5327 0.109 Uiso 1 1 calc R . .
O1W O 0.5000 0.6481(14) 0.7500 0.051(6) Uiso 0.50 2 d SP . .
H1W H 0.4917 0.6962 0.7744 0.032 Uiso 1.50 1 d P . .
F7 F 0.4026(5) 0.9585(8) 0.7814(8) 0.104(4) Uani 1 1 d . . .
F8 F 0.4808(5) 0.9539(8) 0.9232(9) 0.116(4) Uani 1 1 d . . .
F10 F 0.0415(6) 0.7224(10) 0.5743(8) 0.143(7) Uani 1 1 d . . .
F9 F 0.4211(7) 1.0402(6) 0.8641(9) 0.128(5) Uani 1 1 d . . .
F11 F 0.0304(7) 0.6987(9) 0.4662(11) 0.143(6) Uani 1 1 d . . .
F4 F 0.4715(7) 0.8523(11) 1.1173(9) 0.145(6) Uani 1 1 d . . .
F1 F 0.3547(8) 1.0764(8) 1.0128(8) 0.175(9) Uani 1 1 d . . .
F5 F 0.3878(7) 0.8487(13) 1.0893(14) 0.192(10) Uani 1 1 d . . .
F2 F 0.2689(9) 1.1334(9) 0.9154(13) 0.156(7) Uani 1 1 d . . .
F12 F 0.0005(5) 0.7989(9) 0.4725(10) 0.140(6) Uani 1 1 d . . .
F3 F 0.3200(10) 1.1163(11) 0.8959(13) 0.178(8) Uani 1 1 d . . .
C18 C 0.4151(8) 0.8394(17) 1.0601(10) 0.101(8) Uani 1 1 d . . .
F13 F 0.1196(6) 0.8479(13) 0.8437(8) 0.167(9) Uani 1 1 d . . .
F14 F 0.1856(7) 0.9117(9) 0.9663(10) 0.133(5) Uiso 1 1 d . . .
C13 C 0.1838(9) 0.8513(11) 0.4928(12) 0.073(5) Uiso 1 1 d . . .
F16 F 0.1535(10) 0.8072(13) 0.4269(15) 0.183(8) Uiso 1 1 d . . .
C23 C 0.1679(9) 0.8842(11) 0.8951(12) 0.073(4) Uiso 1 1 d . . .
F17 F 0.2400(9) 0.8317(11) 0.5280(12) 0.150(6) Uiso 1 1 d . . .
F15 F 0.1929(14) 0.8201(18) 0.943(2) 0.237(11) Uiso 1 1 d . . .
F18 F 0.1920(11) 0.9185(15) 0.4885(15) 0.197(8) Uiso 1 1 d . . .

loop_
 _atom_site_aniso_label
 _atom_site_aniso_U_11
 _atom_site_aniso_U_22
 _atom_site_aniso_U_33
 _atom_site_aniso_U_23
 _atom_site_aniso_U_13
 _atom_site_aniso_U_12
Tb 0.0355(3) 0.0215(3) 0.0364(3) -0.00357(19) 0.0275(3) -0.00353(19)
O5 0.039(4) 0.028(4) 0.038(4) -0.006(3) 0.028(4) -0.003(3)
O1 0.045(5) 0.047(5) 0.042(5) 0.009(4) 0.032(4) 0.006(4)
O2 0.044(4) 0.028(4) 0.038(4) -0.008(3) 0.028(4) -0.013(3)
O3 0.041(4) 0.034(4) 0.034(4) -0.001(3) 0.025(4) -0.005(3)
C5 0.037(6) 0.029(6) 0.032(6) -0.007(4) 0.021(5) -0.006(4)
N1 0.034(5) 0.032(5) 0.037(5) 0.000(4) 0.027(4) 0.002(4)
O4 0.039(4) 0.036(4) 0.043(4) -0.013(3) 0.029(4) -0.018(3)
N3 0.043(5) 0.022(4) 0.043(5) -0.004(4) 0.032(5) -0.003(4)
C4 0.047(6) 0.025(5) 0.032(5) 0.001(4) 0.027(5) 0.000(5)
```

```
N4 0.048(6) 0.027(5) 0.046(6) 0.001(4) 0.032(5) -0.003(4)
C1 0.053(7) 0.037(6) 0.056(7) 0.006(6) 0.042(7) 0.004(5)
O6 0.054(5) 0.031(4) 0.037(4) -0.013(4) 0.025(4) -0.006(4)
N2 0.067(7) 0.031(5) 0.072(7) 0.010(5) 0.057(7) 0.015(5)
C6 0.064(8) 0.025(6) 0.054(7) 0.005(5) 0.039(7) -0.008(6)
C11 0.052(7) 0.031(6) 0.041(7) -0.004(5) 0.030(6) -0.006(5)
C14 0.068(10) 0.056(9) 0.071(10) 0.006(7) 0.048(9) 0.006(7)
C10 0.062(8) 0.060(9) 0.032(6) -0.003(6) 0.029(6) -0.015(7)
C9 0.054(8) 0.048(7) 0.040(7) 0.006(6) 0.033(7) 0.007(6)
C8 0.056(7) 0.037(7) 0.065(8) 0.003(6) 0.049(7) 0.001(6)
C2 0.051(8) 0.055(8) 0.075(9) 0.006(7) 0.050(8) 0.008(6)
C7 0.057(8) 0.046(7) 0.069(9) 0.001(7) 0.050(8) -0.011(6)
C3 0.068(9) 0.049(8) 0.086(11) 0.008(8) 0.059(9) 0.021(7)
C22 0.079(10) 0.024(6) 0.058(8) -0.018(6) 0.050(8) -0.013(6)
C16 0.088(13) 0.064(10) 0.098(14) -0.001(9) 0.072(12) 0.001(9)
C21 0.27(4) 0.046(11) 0.029(9) -0.006(8) -0.011(14) 0.057(17)
C12 0.066(9) 0.061(9) 0.042(7) -0.016(7) 0.032(7) -0.028(8)
C15 0.059(9) 0.073(11) 0.065(10) 0.008(8) 0.035(9) -0.001(8)
C17 0.052(9) 0.077(11) 0.076(10) -0.008(8) 0.048(9) -0.021(8)
C19 0.058(10) 0.19(2) 0.073(11) -0.073(14) 0.053(10) -0.054(13)
F6 0.120(10) 0.108(10) 0.065(7) 0.033(7) 0.047(7) 0.019(8)
C20 0.108(16) 0.078(13) 0.124(18) -0.011(12) 0.094(16) -0.007(11)
O7 0.037(4) 0.023(4) 0.052(5) 0.000(3) 0.026(4) -0.002(3)
N1S 0.040(6) 0.069(8) 0.043(6) 0.009(5) 0.028(5) 0.006(5)
C2S 0.038(6) 0.055(8) 0.035(6) -0.007(6) 0.022(6) -0.001(6)
N3S 0.036(6) 0.110(11) 0.050(7) 0.018(7) 0.031(6) 0.012(6)
C4S 0.050(8) 0.085(11) 0.055(8) 0.010(8) 0.035(7) 0.003(8)
C5S 0.065(10) 0.106(14) 0.055(9) 0.018(9) 0.044(9) 0.005(9)
C6S 0.048(9) 0.17(2) 0.068(10) 0.029(12) 0.044(9) 0.009(11)
F7 0.106(8) 0.133(10) 0.126(9) -0.025(8) 0.101(8) -0.045(7)
F8 0.084(8) 0.129(11) 0.129(10) 0.055(9) 0.071(8) 0.025(7)
F10 0.096(8) 0.222(16) 0.073(7) -0.021(9) 0.044(7) -0.106(10)
F9 0.154(12) 0.045(6) 0.112(9) 0.016(6) 0.064(9) -0.018(7)
F11 0.150(12) 0.143(12) 0.172(13) -0.121(11) 0.125(12) -0.106(11)
F4 0.111(11) 0.224(19) 0.081(9) 0.024(10) 0.060(9) -0.011(11)
F1 0.152(13) 0.101(10) 0.057(7) -0.005(7) -0.010(8) -0.009(9)
F5 0.072(9) 0.26(3) 0.171(17) -0.048(15) 0.058(11) 0.025(11)
F2 0.153(14) 0.091(10) 0.190(17) -0.030(10) 0.105(14) -0.003(9)
F12 0.049(6) 0.126(11) 0.140(12) -0.005(9) 0.025(7) -0.019(7)
F3 0.198(18) 0.162(15) 0.172(16) -0.047(13) 0.127(16) -0.113(15)
C18 0.042(9) 0.19(3) 0.023(7) 0.026(10) 0.003(7) 0.047(12)
F13 0.073(7) 0.39(3) 0.070(7) -0.037(11) 0.060(7) -0.061(11)

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

loop_
 _geom_bond_atom_site_label_1
 _geom_bond_atom_site_label_2
 _geom_bond_distance
```

```
 _geom_bond_site_symmetry_2
 _geom_bond_publ_flag
Tb O7 2.332(8) . ?
Tb O5 2.354(7) . ?
Tb O3 2.364(8) . ?
Tb O4 2.375(7) . ?
Tb O1 2.383(8) . ?
Tb O2 2.399(7) . ?
Tb O6 2.446(8) . ?
Tb N1 2.610(9) . ?
Tb N3 2.645(9) . ?
O5 C19 1.232(18) . ?
O1 C9 1.235(16) . ?
O2 C11 1.243(14) . ?
O3 C14 1.304(19) . ?
C5 N4 1.333(14) . ?
C5 N3 1.361(14) . ?
C5 C4 1.470(16) . ?
N1 C1 1.337(15) . ?
N1 C4 1.346(14) . ?
O4 C16 1.27(2) . ?
N3 C8 1.355(15) . ?
C4 N2 1.327(15) . ?
N4 C6 1.346(17) . ?
C1 C2 1.375(18) . ?
C1 H1A 0.9300 . ?
O6 C21 1.02(3) . ?
N2 C3 1.335(19) . ?
C6 C7 1.365(19) . ?
C6 H6A 0.9300 . ?
C11 C10 1.386(18) . ?
C11 C12 1.518(19) . ?
C14 C15 1.40(2) . ?
C14 C18 1.47(2) . ?
C10 C9 1.39(2) . ?
C10 H10A 0.9300 . ?
C9 C13 1.54(2) . ?
C8 C7 1.363(18) . ?
C8 H8A 0.9300 . ?
C2 C3 1.34(2) . ?
C2 H2B 0.9300 . ?
C7 H7A 0.9300 . ?
C3 H3A 0.9300 . ?
C22 F1 1.211(18) . ?
C22 F3 1.29(2) . ?
C22 F2 1.32(2) . ?
C22 C21 1.47(2) . ?
C16 C15 1.44(2) . ?
C16 C17 1.55(2) . ?
C21 C20 1.72(5) . ?
C12 F10 1.257(18) . ?
C12 F11 1.304(18) . ?
C12 F12 1.31(2) . ?
C15 H15A 0.9300 . ?
C17 F9 1.28(2) . ?
C17 F7 1.316(19) . ?
C17 F8 1.344(19) . ?
```

```
C19 C23 1.44(2) . ?
C19 C20 1.49(3) . ?
F6 C18 1.43(3) . ?
C20 H20A 0.9300 . ?
N1S C2S 1.316(16) . ?
N1S C4S 1.325(18) . ?
C2S N3S 1.330(16) . ?
C2S C2S 1.47(3) 5_776 ?
N3S C6S 1.36(2) . ?
C4S C5S 1.37(2) . ?
C4S H4SA 0.9300 . ?
C5S C6S 1.31(2) . ?
C5S H5SA 0.9300 . ?
C6S H6SA 0.9300 . ?
O1W H1W 1.0998 . ?
F4 C18 1.25(2) . ?
F5 C18 1.33(3) . ?
F13 C23 1.23(2) . ?
F13 F15 1.76(3) . ?
F14 C23 1.30(2) . ?
F14 F15 1.73(3) . ?
C13 F18 1.21(3) . ?
C13 F16 1.27(3) . ?
C13 F17 1.33(2) . ?
C23 F15 1.33(3) . ?

loop_
 _geom_angle_atom_site_label_1
 _geom_angle_atom_site_label_2
 _geom_angle_atom_site_label_3
 _geom_angle
 _geom_angle_site_symmetry_1
 _geom_angle_site_symmetry_3
 _geom_angle_publ_flag
O7 Tb O5 82.4(3) . . ?
O7 Tb O3 140.0(3) . . ?
O5 Tb O3 77.3(3) . . ?
O7 Tb O4 96.8(3) . . ?
O5 Tb O4 135.2(3) . . ?
O3 Tb O4 75.2(3) . . ?
O7 Tb O1 73.3(3) . . ?
O5 Tb O1 145.5(3) . . ?
O3 Tb O1 136.4(3) . . ?
O4 Tb O1 72.9(3) . . ?
O7 Tb O2 72.0(3) . . ?
O5 Tb O2 77.6(3) . . ?
O3 Tb O2 134.3(3) . . ?
O4 Tb O2 144.9(3) . . ?
O1 Tb O2 72.0(3) . . ?
O7 Tb O6 69.5(3) . . ?
O5 Tb O6 70.9(3) . . ?
O3 Tb O6 71.4(3) . . ?
O4 Tb O6 67.1(3) . . ?
O1 Tb O6 120.2(3) . . ?
O2 Tb O6 132.5(3) . . ?
O7 Tb N1 141.6(3) . . ?
O5 Tb N1 132.0(3) . . ?
```

```
O3 Tb N1 73.8(3) . . ?
O4 Tb N1 71.5(3) . . ?
O1 Tb N1 68.3(3) . . ?
O2 Tb N1 96.3(3) . . ?
O6 Tb N1 131.1(3) . . ?
O7 Tb N3 134.3(3) . . ?
O5 Tb N3 72.4(3) . . ?
O3 Tb N3 70.4(3) . . ?
O4 Tb N3 127.9(3) . . ?
O1 Tb N3 108.1(3) . . ?
O2 Tb N3 65.8(3) . . ?
O6 Tb N3 131.5(3) . . ?
N1 Tb N3 62.1(3) . . ?
C19 O5 Tb 136.3(9) . . ?
C9 O1 Tb 134.2(8) . . ?
C11 O2 Tb 134.4(8) . . ?
C14 O3 Tb 133.1(9) . . ?
N4 C5 N3 126.1(11) . . ?
N4 C5 C4 116.7(10) . . ?
N3 C5 C4 117.1(9) . . ?
C1 N1 C4 115.5(10) . . ?
C1 N1 Tb 122.4(7) . . ?
C4 N1 Tb 122.1(7) . . ?
C16 O4 Tb 131.3(10) . . ?
C8 N3 C5 116.0(10) . . ?
C8 N3 Tb 123.4(7) . . ?
C5 N3 Tb 120.6(7) . . ?
N2 C4 N1 126.0(11) . . ?
N2 C4 C5 116.6(10) . . ?
N1 C4 C5 117.4(10) . . ?
C5 N4 C6 115.7(10) . . ?
N1 C1 C2 121.9(12) . . ?
N1 C1 H1A 119.1 . . ?
C2 C1 H1A 119.1 . . ?
C21 O6 Tb 151(3) . . ?
C4 N2 C3 116.1(11) . . ?
N4 C6 C7 122.2(11) . . ?
N4 C6 H6A 118.9 . . ?
C7 C6 H6A 118.9 . . ?
O2 C11 C10 128.4(12) . . ?
O2 C11 C12 114.7(11) . . ?
C10 C11 C12 116.9(11) . . ?
O3 C14 C15 128.7(15) . . ?
O3 C14 C18 117.3(16) . . ?
C15 C14 C18 113.7(17) . . ?
C11 C10 C9 120.6(12) . . ?
C11 C10 H10A 119.7 . . ?
C9 C10 H10A 119.7 . . ?
O1 C9 C10 129.5(12) . . ?
O1 C9 C13 114.4(13) . . ?
C10 C9 C13 116.1(12) . . ?
N3 C8 C7 120.8(12) . . ?
N3 C8 H8A 119.6 . . ?
C7 C8 H8A 119.6 . . ?
C3 C2 C1 117.8(13) . . ?
C3 C2 H2B 121.1 . . ?
C1 C2 H2B 121.1 . . ?
```

```
C8 C7 C6 119.1(12) . . ?
C8 C7 H7A 120.4 . . ?
C6 C7 H7A 120.4 . . ?
N2 C3 C2 122.5(12) . . ?
N2 C3 H3A 118.7 . . ?
C2 C3 H3A 118.7 . . ?
F1 C22 F3 109.2(18) . . ?
F1 C22 F2 114.4(16) . . ?
F3 C22 F2 91.2(16) . . ?
F1 C22 C21 118.5(14) . . ?
F3 C22 C21 105(2) . . ?
F2 C22 C21 114(2) . . ?
O4 C16 C15 131.2(16) . . ?
O4 C16 C17 111.9(15) . . ?
C15 C16 C17 116.9(16) . . ?
O6 C21 C22 141(5) . . ?
O6 C21 C20 112(3) . . ?
C22 C21 C20 107(3) . . ?
F10 C12 F11 111.5(15) . . ?
F10 C12 F12 107.4(18) . . ?
F11 C12 F12 100.1(15) . . ?
F10 C12 C11 114.3(12) . . ?
F11 C12 C11 112.7(14) . . ?
F12 C12 C11 109.9(12) . . ?
C14 C15 C16 119.6(16) . . ?
C14 C15 H15A 120.2 . . ?
C16 C15 H15A 120.2 . . ?
F9 C17 F7 102.1(15) . . ?
F9 C17 F8 98.5(14) . . ?
F7 C17 F8 111.5(14) . . ?
F9 C17 C16 119.1(16) . . ?
F7 C17 C16 113.3(14) . . ?
F8 C17 C16 111.2(15) . . ?
O5 C19 C23 118.9(16) . . ?
O5 C19 C20 124.3(18) . . ?
C23 C19 C20 113.1(17) . . ?
C19 C20 C21 120.4(16) . . ?
C19 C20 H20A 119.8 . . ?
C21 C20 H20A 119.8 . . ?
C2S N1S C4S 117.6(12) . . ?
N1S C2S N3S 125.1(12) . . ?
N1S C2S C2S 119.1(14) . 5_776 ?
N3S C2S C2S 115.8(14) . 5_776 ?
C2S N3S C6S 114.8(13) . . ?
N1S C4S C5S 121.1(14) . . ?
N1S C4S H4SA 119.5 . . ?
C5S C4S H4SA 119.5 . . ?
C6S C5S C4S 117.8(15) . . ?
C6S C5S H5SA 121.1 . . ?
C4S C5S H5SA 121.1 . . ?
C5S C6S N3S 123.5(14) . . ?
C5S C6S H6SA 118.2 . . ?
N3S C6S H6SA 118.2 . . ?
F4 C18 F5 112.4(17) . . ?
F4 C18 F6 102.4(16) . . ?
F5 C18 F6 88(2) . . ?
F4 C18 C14 120(2) . . ?
```

```
F5 C18 C14 120.9(16) . . ?
F6 C18 C14 104.2(17) . . ?
C23 F13 F15 49.1(13) . . ?
C23 F14 F15 49.8(13) . . ?
F18 C13 F16 122(2) . . ?
F18 C13 F17 92.6(19) . . ?
F16 C13 F17 97.6(18) . . ?
F18 C13 C9 114.5(19) . . ?
F16 C13 C9 114.5(18) . . ?
F17 C13 C9 110.9(16) . . ?
F13 C23 F14 120.4(17) . . ?
F13 C23 F15 87(2) . . ?
F14 C23 F15 82.1(18) . . ?
F13 C23 C19 121.1(16) . . ?
F14 C23 C19 118.5(17) . . ?
F15 C23 C19 103(2) . . ?
C23 F15 F14 48.1(14) . . ?
C23 F15 F13 44.1(14) . . ?
F14 F15 F13 77.9(16) . . ?

loop_
 _geom_torsion_atom_site_label_1
 _geom_torsion_atom_site_label_2
 _geom_torsion_atom_site_label_3
 _geom_torsion_atom_site_label_4
 _geom_torsion
 _geom_torsion_site_symmetry_1
 _geom_torsion_site_symmetry_2
 _geom_torsion_site_symmetry_3
 _geom_torsion_site_symmetry_4
 _geom_torsion_publ_flag
O7 Tb O5 C19 -91(2) . . . . ?
O3 Tb O5 C19 54(2) . . . . ?
O4 Tb O5 C19 1(2) . . . . ?
O1 Tb O5 C19 -136(2) . . . . ?
O2 Tb O5 C19 -164(2) . . . . ?
O6 Tb O5 C19 -20(2) . . . . ?
N1 Tb O5 C19 108(2) . . . . ?
N3 Tb O5 C19 128(2) . . . . ?
O7 Tb O1 C9 -65.9(11) . . . . ?
O5 Tb O1 C9 -18.9(14) . . . . ?
O3 Tb O1 C9 146.4(11) . . . . ?
O4 Tb O1 C9 -168.8(12) . . . . ?
O2 Tb O1 C9 10.1(11) . . . . ?
O6 Tb O1 C9 -119.2(11) . . . . ?
N1 Tb O1 C9 114.8(12) . . . . ?
N3 Tb O1 C9 66.1(12) . . . . ?
O7 Tb O2 C11 69.1(11) . . . . ?
O5 Tb O2 C11 155.0(11) . . . . ?
O3 Tb O2 C11 -147.0(10) . . . . ?
O4 Tb O2 C11 -6.8(13) . . . . ?
O1 Tb O2 C11 -8.7(10) . . . . ?
O6 Tb O2 C11 106.1(11) . . . . ?
N1 Tb O2 C11 -73.4(11) . . . . ?
N3 Tb O2 C11 -128.9(11) . . . . ?
O7 Tb O3 C14 -73.2(13) . . . . ?
O5 Tb O3 C14 -134.5(12) . . . . ?
```

```
O4 Tb O3 C14 9.8(12) . . . . ?
O1 Tb O3 C14 54.0(13) . . . . ?
O2 Tb O3 C14 167.4(11) . . . . ?
O6 Tb O3 C14 -60.6(12) . . . . ?
N1 Tb O3 C14 84.4(12) . . . . ?
N3 Tb O3 C14 149.9(13) . . . . ?
O7 Tb N1 C1 45.8(11) . . . . ?
O5 Tb N1 C1 -166.4(8) . . . . ?
O3 Tb N1 C1 -111.0(9) . . . . ?
O4 Tb N1 C1 -31.5(9) . . . . ?
O1 Tb N1 C1 47.0(9) . . . . ?
O2 Tb N1 C1 114.7(9) . . . . ?
O6 Tb N1 C1 -64.8(10) . . . . ?
N3 Tb N1 C1 173.0(10) . . . . ?
O7 Tb N1 C4 -134.8(8) . . . . ?
O5 Tb N1 C4 13.1(10) . . . . ?
O3 Tb N1 C4 68.5(8) . . . . ?
O4 Tb N1 C4 147.9(9) . . . . ?
O1 Tb N1 C4 -133.6(9) . . . . ?
O2 Tb N1 C4 -65.9(8) . . . . ?
O6 Tb N1 C4 114.6(8) . . . . ?
N3 Tb N1 C4 -7.6(8) . . . . ?
O7 Tb O4 C16 132.7(12) . . . . ?
O5 Tb O4 C16 46.7(14) . . . . ?
O3 Tb O4 C16 -7.3(12) . . . . ?
O1 Tb O4 C16 -157.1(13) . . . . ?
O2 Tb O4 C16 -159.0(12) . . . . ?
O6 Tb O4 C16 68.4(12) . . . . ?
N1 Tb O4 C16 -84.9(13) . . . . ?
N3 Tb O4 C16 -57.2(13) . . . . ?
N4 C5 N3 C8 -2.4(17) . . . . ?
C4 C5 N3 C8 174.6(10) . . . . ?
N4 C5 N3 Tb 177.2(8) . . . . ?
C4 C5 N3 Tb -5.7(13) . . . . ?
O7 Tb N3 C8 -37.5(11) . . . . ?
O5 Tb N3 C8 22.2(9) . . . . ?
O3 Tb N3 C8 104.7(9) . . . . ?
O4 Tb N3 C8 156.4(9) . . . . ?
O1 Tb N3 C8 -121.5(9) . . . . ?
O2 Tb N3 C8 -61.7(9) . . . . ?
O6 Tb N3 C8 64.6(10) . . . . ?
N1 Tb N3 C8 -173.7(10) . . . . ?
O7 Tb N3 C5 142.9(7) . . . . ?
O5 Tb N3 C5 -157.4(8) . . . . ?
O3 Tb N3 C5 -75.0(8) . . . . ?
O4 Tb N3 C5 -23.2(9) . . . . ?
O1 Tb N3 C5 58.9(8) . . . . ?
O2 Tb N3 C5 118.7(8) . . . . ?
O6 Tb N3 C5 -115.0(8) . . . . ?
N1 Tb N3 C5 6.7(7) . . . . ?
C1 N1 C4 N2 5.4(17) . . . . ?
Tb N1 C4 N2 -174.0(9) . . . . ?
C1 N1 C4 C5 -172.5(10) . . . . ?
Tb N1 C4 C5 8.0(13) . . . . ?
N4 C5 C4 N2 -2.2(15) . . . . ?
N3 C5 C4 N2 -179.5(10) . . . . ?
N4 C5 C4 N1 176.0(10) . . . . ?
```

```
N3 C5 C4 N1 -1.4(15) . . . . ?
N3 C5 N4 C6 1.2(17) . . . . ?
C4 C5 N4 C6 -175.9(10) . . . . ?
C4 N1 C1 C2 -4.5(18) . . . . ?
Tb N1 C1 C2 175.0(10) . . . . ?
O7 Tb O6 C21 108(4) . . . . ?
O5 Tb O6 C21 19(4) . . . . ?
O3 Tb O6 C21 -63(4) . . . . ?
O4 Tb O6 C21 -145(4) . . . . ?
O1 Tb O6 C21 163(4) . . . . ?
O2 Tb O6 C21 70(4) . . . . ?
N1 Tb O6 C21 -110(4) . . . . ?
N3 Tb O6 C21 -24(4) . . . . ?
N1 C4 N2 C3 -2(2) . . . . ?
C5 C4 N2 C3 175.9(12) . . . . ?
C5 N4 C6 C7 0.5(18) . . . . ?
Tb O2 C11 C10 5(2) . . . . ?
Tb O2 C11 C12 -178.2(9) . . . . ?
Tb O3 C14 C15 -10(2) . . . . ?
Tb O3 C14 C18 163.2(13) . . . . ?
O2 C11 C10 C9 4(2) . . . . ?
C12 C11 C10 C9 -173.2(13) . . . . ?
Tb O1 C9 C10 -8(2) . . . . ?
Tb O1 C9 C13 170.6(9) . . . . ?
C11 C10 C9 O1 -2(2) . . . . ?
C11 C10 C9 C13 179.5(14) . . . . ?
C5 N3 C8 C7 2.0(18) . . . . ?
Tb N3 C8 C7 -177.7(10) . . . . ?
N1 C1 C2 C3 0(2) . . . . ?
N3 C8 C7 C6 -1(2) . . . . ?
N4 C6 C7 C8 -1(2) . . . . ?
C4 N2 C3 C2 -3(2) . . . . ?
C1 C2 C3 N2 3(2) . . . . ?
Tb O4 C16 C15 5(3) . . . . ?
Tb O4 C16 C17 -173.5(9) . . . . ?
Tb O6 C21 C22 169.5(14) . . . . ?
Tb O6 C21 C20 -18(5) . . . . ?
F1 C22 C21 O6 -107(3) . . . . ?
F3 C22 C21 O6 15(4) . . . . ?
F2 C22 C21 O6 114(4) . . . . ?
F1 C22 C21 C20 81(3) . . . . ?
F3 C22 C21 C20 -156.9(18) . . . . ?
F2 C22 C21 C20 -59(2) . . . . ?
O2 C11 C12 F10 -9(2) . . . . ?
C10 C11 C12 F10 168.2(16) . . . . ?
O2 C11 C12 F11 -137.9(15) . . . . ?
C10 C11 C12 F11 40(2) . . . . ?
O2 C11 C12 F12 111.5(15) . . . . ?
C10 C11 C12 F12 -71.0(18) . . . . ?
O3 C14 C15 C16 2(3) . . . . ?
C18 C14 C15 C16 -171.0(18) . . . . ?
O4 C16 C15 C14 0(3) . . . . ?
C17 C16 C15 C14 178.6(15) . . . . ?
O4 C16 C17 F9 88.8(19) . . . . ?
C15 C16 C17 F9 -90(2) . . . . ?
O4 C16 C17 F7 -31(2) . . . . ?
C15 C16 C17 F7 150.0(16) . . . . ?
```

```
O4 C16 C17 F8 -157.8(15) . . . . ?
C15 C16 C17 F8 23(2) . . . . ?
Tb O5 C19 C23 -175.8(12) . . . . ?
Tb O5 C19 C20 28(3) . . . . ?
O5 C19 C20 C21 -17(3) . . . . ?
C23 C19 C20 C21 -175.2(18) . . . . ?
O6 C21 C20 C19 11(3) . . . . ?
C22 C21 C20 C19 -174.6(19) . . . . ?
C4S N1S C2S N3S 1(2) . . . . ?
C4S N1S C2S C2S -178.7(15) . . . 5_776 ?
N1S C2S N3S C6S -4(2) . . . . ?
C2S C2S N3S C6S 176.1(17) 5_776 . . . ?
C2S N1S C4S C5S 1(2) . . . . ?
N1S C4S C5S C6S -1(3) . . . . ?
C4S C5S C6S N3S -2(3) . . . . ?
C2S N3S C6S C5S 4(3) . . . . ?
O3 C14 C18 F4 171(2) . . . . ?
C15 C14 C18 F4 -14(3) . . . . ?
O3 C14 C18 F5 -38(3) . . . . ?
C15 C14 C18 F5 136(3) . . . . ?
O3 C14 C18 F6 58(2) . . . . ?
C15 C14 C18 F6 -128.3(17) . . . . ?
O1 C9 C13 F18 -63(2) . . . . ?
C10 C9 C13 F18 115(2) . . . . ?
O1 C9 C13 F16 149.0(17) . . . . ?
C10 C9 C13 F16 -33(2) . . . . ?
O1 C9 C13 F17 40(2) . . . . ?
C10 C9 C13 F17 -141.7(16) . . . . ?
F15 F13 C23 F14 -79(2) . . . . ?
F15 F13 C23 C19 103(3) . . . . ?
F15 F14 C23 F13 82(2) . . . . ?
F15 F14 C23 C19 -100(2) . . . . ?
O5 C19 C23 F13 -14(4) . . . . ?
C20 C19 C23 F13 145(2) . . . . ?
O5 C19 C23 F14 168(2) . . . . ?
C20 C19 C23 F14 -33(3) . . . . ?
O5 C19 C23 F15 80(3) . . . . ?
C20 C19 C23 F15 -121(2) . . . . ?
F13 C23 F15 F14 -121.3(16) . . . . ?
C19 C23 F15 F14 117.5(18) . . . . ?
F14 C23 F15 F13 121.3(16) . . . . ?
C19 C23 F15 F13 -121.2(18) . . . . ?
C23 F14 F15 F13 -37.5(13) . . . . ?
C23 F13 F15 F14 40.6(14) . . . . ?

_diffrn_measured_fraction_theta_max    0.995
_diffrn_reflns_theta_full              24.41
_diffrn_measured_fraction_theta_full   0.995
_refine_diff_density_max    3.116
_refine_diff_density_min   -1.409
_refine_diff_density_rms    0.222
```