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Nanometer-size metal complexes: magnetic materials and DNA probes

Volume II

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University of Dublin
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Dublin 2
Ireland

A thesis submitted to the University of Dublin, in partial fulfilment of the requirements for the degree of Ph.D.

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Appendix A
Crystal structure data of [Cr(2,2'-bipyridine)$_2$Cl$_2$]BF$_4$

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goodness of fit S are based on F^2^, conventional R-factors R are based
on F, with F set to zero for negative F^2^. The threshold expression of
F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
not relevant to the choice of reflections for refinement.  R-factors based
on F^2^ are statistically about twice as large as those based on F, and R-
factors based on ALL data will be even larger.
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C2A C -1.7165(4) -0.8477(3) -0.1301(3) 0.0252(8) Uani 1 1 d . . .
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C3A C -1.7552(4) -0.9234(4) -0.1954(3) 0.0307(10) Uani 1 1 d . . .
H3A H -1.7025 -0.9566 -0.2190 0.037 Uiso 1 1 calc R . .
C4A C -1.8715(4) -0.9505(4) -0.2264(3) 0.0349(10) Uani 1 1 d . . .
H4A H -1.8990 -1.0043 -0.2694 0.042 Uiso 1 1 calc R . .
C5A C -1.9472(4) -0.8982(4) -0.1937(3) 0.0296(9) Uani 1 1 d . . .
H5A H -2.0275 -0.9152 -0.2147 0.036 Uiso 1 1 calc R . .
C6A C -1.9052(4) -0.8209(3) -0.1302(3) 0.0216(8) Uani 1 1 d . . .
C7A C -1.9783(3) -0.7591(3) -0.0927(3) 0.0217(8) Uani 1 1 d . . .
C8A C -2.0994(4) -0.7652(4) -0.1235(3) 0.0294(9) Uani 1 1 d . . .
H8A H -2.1401 -0.8119 -0.1709 0.035 Uiso 1 1 calc R . .
C9A C -2.1597(4) -0.7021(4) -0.0841(3) 0.0318(10) Uani 1 1 d . . .
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C10A C -2.0987(4) -0.6362(4) -0.0135(3) 0.0298(9) Uani 1 1 d . . .
H10A H -2.1386 -0.5939 0.0154 0.036 Uiso 1 1 calc R . .
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H11A H -1.9365 -0.5875 0.0620 0.031 Uiso 1 1 calc R . .
N12A N -1.9190(3) -0.6930(3) -0.0254(2) 0.0205(7) Uani 1 1 d . . .
N1B N -1.7061(3) -0.7951(2) 0.1053(2) 0.0196(7) Uani 1 1 d . . .
C2B C -1.7843(4) -0.8392(3) 0.1355(3) 0.0247(8) Uani 1 1 d . . .
H2B H -1.8625 -0.8161 0.1126 0.030 Uiso 1 1 calc R . .
C3B C -1.7546(4) -0.9181(4) 0.1998(3) 0.0330(10) Uani 1 1 d . . .
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C4B C -1.6414(5) -0.9513(4) 0.2327(3) 0.0345(10) Uani 1 1 d . . .
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F12' F -1.5307(7) -0.7130(6) -0.3345(6) 0.051(2) Uiso 0.50 1 d P A 2
F13' F -1.3720(8) -0.8073(7) -0.2564(7) 0.036(2) Uiso 0.50 1 d P A 2
F14' F -1.5532(10) -0.8554(9) -0.2581(8) 0.076(3) Uiso 0.50 1 d P A 2
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F12 F -1.5243(9) -0.7006(8) -0.3010(8) 0.075(3) Uiso 0.50 1 d P B 1
F13 F -1.3722(10) -0.7991(9) -0.2395(8) 0.052(4) Uiso 0.50 1 d P B 1
F14 F -1.5515(6) -0.8655(5) -0.2852(5) 0.0337(15) Uiso 0.50 1 d P B 1
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C2S C -0.9030(12) -1.0376(8) -0.0057(7) 0.041(3) Uani 0.50 1 d P . 2
C3S C -0.7870(13) -1.0703(10) -0.0046(9) 0.052(3) Uani 0.50 1 d P C 2
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H3S3 H -0.7967 -1.1238 -0.0509 0.078 Uiso 0.50 1 calc PR C 2
N1S' N -0.4895(15) -0.9779(10) -0.0104(11) 0.053(3) Uani 0.50 1 d P . 1
C2S' C -0.5991(11) -1.0449(8) 0.0136(8) 0.043(3) Uani 0.50 1 d P . 1
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C9A 0.0180(19) 0.034(2) 0.039(3) 0.003(2) 0.0051(18) -0.0001(17)
C10A 0.023(2) 0.030(2) 0.036(2) 0.0042(19) 0.0105(18) 0.0057(17)
C11A 0.023(2) 0.027(2) 0.028(2) -0.0004(17) 0.0081(17) 0.0017(16)
N12A 0.0190(15) 0.0205(16) 0.0199(16) -0.0002(13) 0.0043(13) 0.0001(12)
N1B 0.0197(15) 0.0190(16) 0.0171(15) -0.0030(12) 0.0026(12) 0.0016(12)
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C3S' 0.057(7) 0.036(6) 0.045(7) -0.010(5) 0.016(6) -0.002(6)

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 All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix.  The cell esds are taken
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 used when they are defined by crystal symmetry.  An approximate (isotropic)
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Cr1 Cl2 2.2966(11) . ?
Cr1 Cl1 2.2967(11) . ?
N1A C2A 1.341(5) . ?
N1A C6A 1.367(5) . ?
C2A C3A 1.379(6) . ?
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C7B C8B 1.391(6) . ?
C8B C9B 1.373(7) . ?
C9B C10B 1.390(8) . ?
C10B C11B 1.383(6) . ?
C11B N12B 1.341(5) . ?
B1' F14' 1.333(13) . ?
B1' F11' 1.345(15) . ?
B1' F13' 1.421(12) . ?
B1' F12' 1.520(11) . ?
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N1S C2S 1.10(3) 3_335 ?
N1S C2S 1.557(18) . ?
C2S N1S 1.10(3) 3_335 ?
C2S C3S 1.48(2) . ?
N1S' N1S' 0.76(2) 3_435 ?
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N12A Cr1 N1B 93.04(13) . . ?
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N1B Cr1 Cl2 91.04(10) . . ?
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N12B Cr1 Cl1 96.40(11) . . ?
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C3A C4A C5A 119.0(4) . . ?
C6A C5A C4A 119.7(4) . . ?
N1A C6A C5A 120.7(4) . . ?
N1A C6A C7A 115.2(3) . . ?
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C7A N12A C11A 119.2(4) . . ?
C7A N12A Cr1 115.9(3) . . ?
C11A N12A Cr1 124.7(3) . . ?
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C3B C4B C5B 119.2(4) . . ?
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C9B C8B C7B 119.7(4) . . ?
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C11B C10B C9B 118.7(4) . . ?
N12B C11B C10B 122.3(4) . . ?
C11B N12B C7B 119.0(4) . . ?
C11B N12B Cr1 125.0(3) . . ?

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F11' B1' F13' 117.5(9) . . ?
F14' B1' F12' 107.1(7) . . ?
F11' B1' F12' 104.2(9) . . ?
F13' B1' F12' 101.1(6) . . ?
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Appendix B
Crystal structure data of [Cr(1,10-phenanthroline)$_2$Cl$_2$](PF$_6$)

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;
;
_chemical_name_common            ?
_chemical_melting_point          ?
_chemical_formula_moiety         ?
_chemical_formula_sum
 'C26 H18 Cl2 Cr F6 N5 P'
_chemical_formula_weight         668.32

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 _atom_type_scat_source
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 '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'
 'F'  'F'   0.0171   0.0103
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'P'  'P'   0.1023   0.0942
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'Cl'  'Cl'   0.1484   0.1585
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'Cr'  'Cr'   0.3209   0.6236
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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_symmetry_space_group_name_H-M    C2/c

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 '-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'

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_cell_length_b                  12.642(3)
_cell_length_c                  13.010(3)
_cell_angle_alpha               90.00
_cell_angle_beta                103.521(7)
_cell_angle_gamma               90.00
_cell_volume                    2603.0(10)
_cell_formula_units_Z           4
_cell_measurement_temperature   153(2)
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_exptl_crystal_density_diffrn   1.705
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_diffrn_radiation_monochromator graphite
_diffrn_measurement_device_type 'CCD area detector'
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Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
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on F, with F set to zero for negative F^2^. The threshold expression of
F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
not relevant to the choice of reflections for refinement.  R-factors based
on F^2^ are statistically about twice as large as those based on F, and R-
factors based on ALL data will be even larger.
;

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N12 N 0.40870(13) 0.29235(16) 0.22013(17) 0.0205(4) Uani 1 1 d . . .
N1 N 0.48089(14) 0.18805(16) 0.08716(17) 0.0207(5) Uani 1 1 d . . .
C14 C 0.41539(16) 0.25315(19) 0.0435(2) 0.0217(5) Uani 1 1 d . . .
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C5 C 0.38486(17) 0.2655(2) -0.0659(2) 0.0263(6) Uani 1 1 d . . .
C13 C 0.37722(16) 0.31070(19) 0.1150(2) 0.0211(5) Uani 1 1 d . . .
C8 C 0.31033(17) 0.3799(2) 0.0759(2) 0.0258(6) Uani 1 1 d . . .
C9 C 0.27472(18) 0.4332(2) 0.1508(2) 0.0304(6) Uani 1 1 d . . .
H9 H 0.2296 0.4818 0.1282 0.036 Uiso 1 1 calc R . .
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C4 C 0.42507(19) 0.2064(2) -0.1318(2) 0.0307(6) Uani 1 1 d . . .
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C7 C 0.27987(18) 0.3906(2) -0.0359(2) 0.0302(6) Uani 1 1 d . . .
H7 H 0.2345 0.4377 -0.0630 0.036 Uiso 1 1 calc R . .
C2 C 0.51903(17) 0.1358(2) 0.0224(2) 0.0249(5) Uani 1 1 d . . .
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F11 F 0.2324(2) 0.7014(2) 0.1041(2) 0.0877(10) Uani 1 1 d . . .
F12 F 0.33140(15) 0.67708(18) 0.01045(19) 0.0614(7) Uani 1 1 d . . .
F13 F 0.30373(17) 0.8403(3) 0.0658(3) 0.1026(12) Uani 1 1 d . . .
C1S C 0.5000 0.5000 0.0000 0.229(8) Uiso 1 2 d SD . .
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Cl1 0.0228(3) 0.0266(3) 0.0256(4) -0.0016(2) 0.0045(3) 0.0029(2)
N12 0.0237(11) 0.0205(10) 0.0194(11) 0.0012(8) 0.0092(9) -0.0011(8)
N1 0.0230(11) 0.0216(10) 0.0182(11) 0.0011(8) 0.0066(9) -0.0001(8)
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C10 0.0346(15) 0.0279(14) 0.0362(16) -0.0022(12) 0.0190(13) 0.0046(11)
C5 0.0308(14) 0.0257(13) 0.0218(13) 0.0013(10) 0.0049(11) -0.0046(11)
C13 0.0211(12) 0.0206(12) 0.0225(13) 0.0014(10) 0.0069(10) -0.0022(9)
C8 0.0244(13) 0.0231(13) 0.0299(15) 0.0026(11) 0.0067(11) 0.0014(10)
C9 0.0274(14) 0.0255(13) 0.0399(17) 0.0043(12) 0.0114(12) 0.0070(11)
C6 0.0357(16) 0.0324(14) 0.0239(14) 0.0061(12) 0.0000(12) -0.0004(12)
C4 0.0410(16) 0.0331(14) 0.0185(13) -0.0016(11) 0.0081(12) -0.0060(12)
C7 0.0282(14) 0.0296(14) 0.0295(15) 0.0070(12) 0.0001(12) 0.0045(11)
C2 0.0255(13) 0.0254(12) 0.0248(14) -0.0021(10) 0.0076(11) -0.0004(10)
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C3 0.0362(15) 0.0301(13) 0.0239(14) -0.0069(11) 0.0133(12) -0.0031(11)
P1 0.0345(6) 0.0342(6) 0.0329(6) -0.0039(4) 0.0179(5) -0.0015(4)
F11 0.129(2) 0.0819(17) 0.0805(19) 0.0378(15) 0.0812(19) 0.0501(17)
F12 0.0600(14) 0.0777(16) 0.0553(14) 0.0075(11) 0.0312(11) 0.0310(12)
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 used when they are defined by crystal symmetry. An approximate (isotropic)
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C10 C9 1.369(4) . ?
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C4 C3 1.369(4) . ?
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P1 F13 1.566(2) . ?
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P1 F12 1.594(2) 7_565 ?
P1 F12 1.594(2) . ?
C1S C2S 1.447(9) . ?
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C2S N3S 1.268(11) . ?
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N12 Cr1 N1 93.85(8) 2_655 . ?
N1 Cr1 N1 171.26(11) 2_655 . ?
N12 Cr1 Cl1 89.24(6) . 2_655 ?
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N1 Cr1 Cl1 92.00(6) . 2_655 ?
N12 Cr1 Cl1 173.39(6) . . ?
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C2 N1 Cr1 128.84(18) . . ?
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N1 C14 C13 116.9(2) . . ?
C5 C14 C13 119.9(2) . . ?
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C14 C5 C4 117.0(2) . . ?
C14 C5 C6 118.7(3) . . ?
C4 C5 C6 124.3(3) . . ?
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C7 C6 C5 121.1(3) . . ?
C3 C4 C5 119.8(3) . . ?
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N12 C11 C10 122.2(3) . . ?
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F13 P1 F11 89.7(2) 7_565 . ?
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F11 P1 F12 90.91(12) . . ?
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_diffrn_measured_fraction_theta_full  0.873
_refine_diff_density_max    0.728
_refine_diff_density_min   -0.552
_refine_diff_density_rms    0.105
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Appendix C
Crystal structure data of [Cr(1,10-phenanthroline)$_3$](ClO$_4$)$_3$

```
data_crphenm

_audit_creation_method          SHELXL-97
_chemical_name_systematic
;
;
_chemical_name_common            ?
_chemical_melting_point          ?
_chemical_formula_moiety         ?
_chemical_formula_sum
 'C43.50 H36.25 Cl3 Cr N9.75 O12.50'
_chemical_formula_weight         1053.92

loop_
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 _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'
 'Cl'  'Cl'   0.1484   0.1585
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'Cr'  'Cr'   0.3209   0.6236
 '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

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

_cell_length_a                   12.0910(16)
_cell_length_b                   12.2613(16)
```

```
_cell_length_c                    18.198(2)
_cell_angle_alpha                 84.554(3)
_cell_angle_beta                  89.470(2)
_cell_angle_gamma                 62.841(2)
_cell_volume                      2387.8(5)
_cell_formula_units_Z             2
_cell_measurement_temperature     153(2)
_cell_measurement_reflns_used     ?
_cell_measurement_theta_min       ?
_cell_measurement_theta_max       ?

_exptl_crystal_description        block
_exptl_crystal_colour             yellow
_exptl_crystal_size_max           0.30
_exptl_crystal_size_mid           0.20
_exptl_crystal_size_min           0.20
_exptl_crystal_density_meas       Not_measured
_exptl_crystal_density_diffrn     1.466
_exptl_crystal_density_method     'not measured'
_exptl_crystal_F_000              1081
_exptl_absorpt_coefficient_mu     0.480
_exptl_absorpt_correction_type    empirical
_exptl_absorpt_correction_T_min   0.705
_exptl_absorpt_correction_T_max   0.928
_exptl_absorpt_process_details    SADABS

_exptl_special_details
;
 ?
;

_diffrn_ambient_temperature       153(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 area detector'
_diffrn_measurement_method        'phi and omega scans'
_diffrn_detector_area_resol_mean  ?
_diffrn_standards_number          ?
_diffrn_standards_interval_count  ?
_diffrn_standards_interval_time   ?
_diffrn_standards_decay_%         ?
_diffrn_reflns_number             27695
_diffrn_reflns_av_R_equivalents   0.0809
_diffrn_reflns_av_sigmaI/netI     0.0843
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_diffrn_reflns_limit_k_min        -16
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_diffrn_reflns_limit_l_min       -23
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_diffrn_reflns_theta_min         1.12
_diffrn_reflns_theta_max         28.70
_reflns_number_total             10686
_reflns_number_gt                6519
_reflns_threshold_expression     >2sigma(I)

_computing_data_collection       'Bruker SMART'
_computing_cell_refinement       'Bruker SMART'
_computing_data_reduction        'Bruker SHELXTL'
_computing_structure_solution    'SHELXS-97 (Sheldrick, 1990)'
_computing_structure_refinement  'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics    'Bruker SHELXTL'
_computing_publication_material  'Bruker SHELXTL'

_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.
;

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_refine_ls_matrix_type           full
_refine_ls_weighting_scheme      calc
_refine_ls_weighting_details
'calc w=1/[\s^2^(Fo^2^)+(0.1621P)^2^+0.0000P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary     direct
_atom_sites_solution_secondary   difmap
_atom_sites_solution_hydrogens   geom
_refine_ls_hydrogen_treatment    mixed
_refine_ls_extinction_method     none
_refine_ls_extinction_coef       ?
_refine_ls_number_reflns         10686
_refine_ls_number_parameters     616
_refine_ls_number_restraints     3
_refine_ls_R_factor_all          0.1210
_refine_ls_R_factor_gt           0.0843
_refine_ls_wR_factor_ref         0.2637
_refine_ls_wR_factor_gt          0.2335
_refine_ls_goodness_of_fit_ref   1.027
_refine_ls_restrained_S_all      1.027
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 _atom_site_fract_z
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 _atom_site_adp_type
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N1A N 0.5880(3) 0.6495(3) 0.83314(18) 0.0343(7) Uani 1 1 d . . .
C2A C 0.4869(4) 0.6473(4) 0.8601(3) 0.0434(10) Uani 1 1 d . . .
H2A H 0.4264 0.6484 0.8267 0.052 Uiso 1 1 calc R . .
C3A C 0.4657(5) 0.6435(5) 0.9353(3) 0.0578(13) Uani 1 1 d . . .
H3A H 0.3911 0.6440 0.9524 0.069 Uiso 1 1 calc R . .
C4A C 0.5522(5) 0.6391(5) 0.9840(3) 0.0556(13) Uani 1 1 d . . .
H4A H 0.5393 0.6340 1.0356 0.067 Uiso 1 1 calc R . .
C5A C 0.6614(5) 0.6420(4) 0.9581(2) 0.0493(11) Uani 1 1 d . . .
C6A C 0.7603(5) 0.6368(5) 1.0036(3) 0.0581(13) Uani 1 1 d . . .
H6A H 0.7528 0.6326 1.0557 0.070 Uiso 1 1 calc R . .
C7A C 0.8621(5) 0.6376(5) 0.9755(3) 0.0556(13) Uani 1 1 d . . .
H7A H 0.9237 0.6374 1.0076 0.067 Uiso 1 1 calc R . .
C8A C 0.8801(4) 0.6388(4) 0.8974(2) 0.0461(10) Uani 1 1 d . . .
C9A C 0.9869(4) 0.6347(4) 0.8638(3) 0.0523(12) Uani 1 1 d . . .
H9A H 1.0543 0.6288 0.8932 0.063 Uiso 1 1 calc R . .
C10A C 0.9932(4) 0.6393(4) 0.7887(3) 0.0485(11) Uani 1 1 d . . .
H10A H 1.0649 0.6371 0.7658 0.058 Uiso 1 1 calc R . .
C11A C 0.8947(4) 0.6470(4) 0.7456(2) 0.0414(10) Uani 1 1 d . . .
H11A H 0.9003 0.6507 0.6934 0.050 Uiso 1 1 calc R . .
N12A N 0.7919(3) 0.6494(3) 0.77618(18) 0.0326(7) Uani 1 1 d . . .
C13A C 0.7853(4) 0.6447(4) 0.8509(2) 0.0355(9) Uani 1 1 d . . .
C14A C 0.6750(4) 0.6457(4) 0.8817(2) 0.0372(9) Uani 1 1 d . . .
N1B N 0.7151(3) 0.4739(3) 0.71742(18) 0.0343(7) Uani 1 1 d . . .
C2B C 0.7167(4) 0.3867(4) 0.7669(2) 0.0403(9) Uani 1 1 d . . .
H2B H 0.6785 0.4083 0.8127 0.048 Uiso 1 1 calc R . .
C3B C 0.7747(5) 0.2617(4) 0.7522(3) 0.0558(13) Uani 1 1 d . . .
H3B H 0.7762 0.1998 0.7884 0.067 Uiso 1 1 calc R . .
C4B C 0.8285(5) 0.2300(4) 0.6859(3) 0.0574(13) Uani 1 1 d . . .
H4B H 0.8663 0.1462 0.6760 0.069 Uiso 1 1 calc R . .
C5B C 0.8283(4) 0.3205(4) 0.6326(3) 0.0462(11) Uani 1 1 d . . .
C6B C 0.8838(4) 0.2989(5) 0.5621(3) 0.0575(14) Uani 1 1 d . . .
H6B H 0.9241 0.2168 0.5489 0.069 Uiso 1 1 calc R . .
C7B C 0.8811(4) 0.3888(5) 0.5146(3) 0.0564(13) Uani 1 1 d . . .
H7B H 0.9210 0.3696 0.4690 0.068 Uiso 1 1 calc R . .
C8B C 0.8190(4) 0.5148(5) 0.5306(2) 0.0466(11) Uani 1 1 d . . .
```

C9B C 0.8119(5) 0.6146(6) 0.4834(3) 0.0585(14) Uani 1 1 d . . .
H9B H 0.8487 0.6022 0.4366 0.070 Uiso 1 1 calc R . .
C10B C 0.7514(5) 0.7306(5) 0.5054(3) 0.0569(13) Uani 1 1 d . . .
H10B H 0.7448 0.7994 0.4734 0.068 Uiso 1 1 calc R . .
C11B C 0.6996(4) 0.7478(4) 0.5746(2) 0.0447(10) Uani 1 1 d . . .
H11B H 0.6591 0.8290 0.5891 0.054 Uiso 1 1 calc R . .
N12B N 0.7043(3) 0.6561(3) 0.62086(18) 0.0348(7) Uani 1 1 d . . .
C13B C 0.7651(3) 0.5391(4) 0.5997(2) 0.0382(9) Uani 1 1 d . . .
C14B C 0.7688(4) 0.4423(4) 0.6509(2) 0.0367(9) Uani 1 1 d . . .
N1C N 0.4670(3) 0.6915(3) 0.67928(18) 0.0325(7) Uani 1 1 d . . .
C2C C 0.4316(4) 0.6092(4) 0.6601(2) 0.0390(9) Uani 1 1 d . . .
H2C H 0.4874 0.5238 0.6694 0.047 Uiso 1 1 calc R . .
C3C C 0.3157(4) 0.6449(4) 0.6271(3) 0.0500(11) Uani 1 1 d . . .
H3C H 0.2928 0.5841 0.6151 0.060 Uiso 1 1 calc R . .
C4C C 0.2342(4) 0.7670(4) 0.6117(3) 0.0476(11) Uani 1 1 d . . .
H4C H 0.1554 0.7912 0.5883 0.057 Uiso 1 1 calc R . .
C5C C 0.2680(4) 0.8559(4) 0.6309(2) 0.0374(9) Uani 1 1 d . . .
C6C C 0.1897(4) 0.9856(4) 0.6208(2) 0.0430(10) Uani 1 1 d . . .
H6C H 0.1096 1.0160 0.5978 0.052 Uiso 1 1 calc R . .
C7C C 0.2259(4) 1.0663(4) 0.6430(2) 0.0409(10) Uani 1 1 d . . .
H7C H 0.1704 1.1520 0.6362 0.049 Uiso 1 1 calc R . .
C8C C 0.3483(4) 1.0245(4) 0.6769(2) 0.0372(9) Uani 1 1 d . . .
C9C C 0.3913(4) 1.1034(4) 0.7012(2) 0.0413(10) Uani 1 1 d . . .
H9C H 0.3404 1.1901 0.6950 0.050 Uiso 1 1 calc R . .
C10C C 0.5089(4) 1.0540(4) 0.7343(3) 0.0467(11) Uani 1 1 d . . .
H10C H 0.5389 1.1063 0.7521 0.056 Uiso 1 1 calc R . .
C11C C 0.5827(4) 0.9270(4) 0.7413(2) 0.0386(9) Uani 1 1 d . . .
H11C H 0.6640 0.8942 0.7632 0.046 Uiso 1 1 calc R . .
N12C N 0.5442(3) 0.8496(3) 0.71860(17) 0.0329(7) Uani 1 1 d . . .
C13C C 0.4265(3) 0.8997(3) 0.6868(2) 0.0316(8) Uani 1 1 d . . .
C14C C 0.3872(3) 0.8131(3) 0.6650(2) 0.0310(8) Uani 1 1 d . . .
Cl1 Cl 0.84830(11) 0.28611(12) 0.95701(7) 0.0526(3) Uani 0.386(6) 1 d P A 2
O11 O 0.7713(9) 0.2409(10) 0.9304(5) 0.052(2) Uiso 0.386(6) 1 d P A 2
O12 O 0.9779(10) 0.1810(9) 0.9713(6) 0.063(3) Uiso 0.386(6) 1 d P A 2
O13 O 0.8115(9) 0.3469(10) 1.0266(5) 0.046(3) Uiso 0.386(6) 1 d P A 2
O14 O 0.8606(10) 0.3727(10) 0.9022(6) 0.040(3) Uiso 0.386(6) 1 d P A 2
Cl1' Cl 0.84830(11) 0.28611(12) 0.95701(7) 0.0526(3) Uani 0.614(6) 1 d P A 1
O11' O 0.7254(7) 0.3014(7) 0.9305(4) 0.0659(18) Uiso 0.614(6) 1 d P A 1
O12' O 0.9354(9) 0.1672(9) 0.9480(6) 0.106(3) Uiso 0.614(6) 1 d P A 1
O13' O 0.8333(8) 0.3038(8) 1.0320(5) 0.071(2) Uiso 0.614(6) 1 d P A 1
O14' O 0.8784(8) 0.3740(8) 0.9175(5) 0.067(3) Uiso 0.614(6) 1 d P A 1
Cl2 Cl 0.88117(12) 0.92018(12) 0.61488(7) 0.0563(3) Uani 0.386(6) 1 d P A 2
O21 O 0.9101(13) 0.9899(12) 0.5636(7) 0.064(4) Uiso 0.386(6) 1 d P A 2
O22 O 0.7338(14) 0.9705(14) 0.5951(10) 0.115(5) Uiso 0.386(6) 1 d P A 2
O23 O 0.9506(9) 0.7805(9) 0.6106(6) 0.050(2) Uiso 0.386(6) 1 d P A 2
O24 O 0.861(2) 0.945(2) 0.6786(13) 0.171(8) Uiso 0.386(6) 1 d P A 2
Cl2' Cl 0.88117(12) 0.92018(12) 0.61488(7) 0.0563(3) Uani 0.614(6) 1 d P A 1
O21' O 0.8729(8) 1.0199(8) 0.5615(5) 0.070(3) Uiso 0.614(6) 1 d P A 1
O22' O 0.7675(5) 0.9484(5) 0.6442(3) 0.0503(14) Uiso 0.614(6) 1 d P A 1
O23' O 0.9307(6) 0.8092(6) 0.5843(4) 0.0533(16) Uiso 0.614(6) 1 d P A 1

```
O24' O 0.9619(5) 0.9188(5) 0.6771(3) 0.0502(14) Uiso 0.614(6) 1 d P A 1
Cl3 Cl 0.38115(12) 0.38913(12) 0.78376(8) 0.0598(4) Uani 0.386(6) 1 d P A 2
O31 O 0.3120(10) 0.3410(9) 0.8140(5) 0.063(3) Uiso 0.386(6) 1 d P A 2
O32 O 0.4251(15) 0.4633(15) 0.8203(8) 0.112(5) Uiso 0.386(6) 1 d P A 2
O33 O 0.3102(15) 0.4036(14) 0.7110(9) 0.115(5) Uiso 0.386(6) 1 d P A 2
O34 O 0.4884(11) 0.3428(11) 0.7404(7) 0.082(3) Uiso 0.386(6) 1 d P A 2
Cl3' Cl 0.38115(12) 0.38913(12) 0.78376(8) 0.0598(4) Uani 0.614(6) 1 d P A 1
O31' O 0.3885(9) 0.2874(8) 0.8347(5) 0.096(3) Uiso 0.614(6) 1 d P A 1
O32' O 0.3122(7) 0.5191(7) 0.7923(4) 0.090(2) Uiso 0.614(6) 1 d P A 1
O33' O 0.4198(7) 0.3600(7) 0.7137(4) 0.079(2) Uiso 0.614(6) 1 d P A 1
O34' O 0.4961(13) 0.3614(14) 0.8185(8) 0.166(5) Uiso 0.614(6) 1 d P A 1
C1SA C 0.429(3) 0.780(2) 0.4383(14) 0.082(8) Uiso 0.532(8) 1 d PD B 2
H1S1 H 0.3408 0.8004 0.4373 0.123 Uiso 0.532(8) 1 calc PR B 2
H1S2 H 0.4769 0.7013 0.4681 0.123 Uiso 0.532(8) 1 calc PR B 2
H1S3 H 0.4588 0.7745 0.3878 0.123 Uiso 0.532(8) 1 calc PR B 2
C2SA C 0.4446(10) 0.8735(11) 0.4697(6) 0.068(3) Uiso 0.532(8) 1 d PD B 2
N3SA N 0.4570(10) 0.9510(10) 0.4926(6) 0.085(3) Uiso 0.532(8) 1 d P B 2
C4SA C 0.430(3) 0.762(2) 0.4340(18) 0.089(10) Uiso 0.468(8) 1 d PD B 1
H4S1 H 0.4691 0.8035 0.4587 0.134 Uiso 0.468(8) 1 calc PR B 1
H4S2 H 0.3396 0.8057 0.4383 0.134 Uiso 0.468(8) 1 calc PR B 1
H4S3 H 0.4501 0.7600 0.3817 0.134 Uiso 0.468(8) 1 calc PR B 1
C5SA C 0.4759(13) 0.6371(13) 0.4683(8) 0.075(4) Uiso 0.468(8) 1 d PD B 1
N6SA N 0.5252(16) 0.5332(16) 0.4994(10) 0.124(6) Uiso 0.468(8) 1 d P . 1
C1SB C 0.7330(11) 0.0131(11) 1.0060(6) 0.108(3) Uiso 0.75 1 d P C 2
H1SA H 0.6734 -0.0047 1.0349 0.162 Uiso 0.75 1 calc PR C 2
H1SB H 0.7116 0.1004 1.0062 0.162 Uiso 0.75 1 calc PR C 2
H1SC H 0.8170 -0.0378 1.0277 0.162 Uiso 0.75 1 calc PR C 2
C2SB C 0.7285(11) -0.0144(11) 0.9303(7) 0.104(3) Uiso 0.75 1 d P C 2
N3SB N 0.7453(11) -0.0362(11) 0.8666(8) 0.144(4) Uiso 0.75 1 d P C 2
C1SC C 0.9734(9) 0.0629(8) 1.1508(5) 0.110(3) Uani 1 1 d D . .
H3SD H 1.0022 0.0991 1.1873 0.166 Uiso 1 1 calc R . .
H3SE H 0.9723 0.1027 1.1013 0.166 Uiso 1 1 calc R . .
H3SF H 1.0297 -0.0255 1.1526 0.166 Uiso 1 1 calc R . .
C2SC C 0.8477(10) 0.0815(7) 1.1677(4) 0.098(3) Uani 1 1 d D . .
N3SC N 0.7510(9) 0.0877(8) 1.1777(5) 0.156(4) Uani 1 1 d . . .
C1SD C 1.1161(6) 0.2543(7) 0.8134(4) 0.086(2) Uani 1 1 d . . .
H3S1 H 1.0945 0.1864 0.8138 0.129 Uiso 1 1 calc R . .
H3S2 H 1.0740 0.3051 0.8532 0.129 Uiso 1 1 calc R . .
H3S3 H 1.2063 0.2209 0.8209 0.129 Uiso 1 1 calc R . .
C2SD C 1.0771(5) 0.3289(5) 0.7432(3) 0.0566(13) Uani 1 1 d . . .
N3SD N 1.0504(4) 0.3834(5) 0.6870(3) 0.0701(13) Uani 1 1 d . . .
O1W O 0.628(7) -0.039(7) 0.964(5) 0.35(4) Uiso 0.25 1 d P D 1
O1W' O 0.660(3) -0.059(3) 0.901(2) 0.168(12) Uiso 0.25 1 d P D 1

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 _atom_site_aniso_U_22
 _atom_site_aniso_U_33
 _atom_site_aniso_U_23
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_atom_site_aniso_U_13
_atom_site_aniso_U_12
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N1A 0.0359(16) 0.0314(17) 0.0354(18) -0.0028(13) 0.0029(13) -0.0154(14)
C2A 0.041(2) 0.042(2) 0.047(3) -0.0015(19) 0.0081(18) -0.0194(19)
C3A 0.058(3) 0.066(3) 0.052(3) -0.002(2) 0.018(2) -0.031(3)
C4A 0.065(3) 0.062(3) 0.038(3) -0.005(2) 0.016(2) -0.028(3)
C5A 0.064(3) 0.047(3) 0.032(2) -0.0055(19) 0.0093(19) -0.021(2)
C6A 0.077(3) 0.064(3) 0.033(2) -0.008(2) -0.002(2) -0.031(3)
C7A 0.068(3) 0.063(3) 0.039(3) -0.008(2) -0.010(2) -0.032(3)
C8A 0.053(2) 0.044(3) 0.043(3) -0.007(2) -0.0060(19) -0.023(2)
C9A 0.048(3) 0.055(3) 0.059(3) -0.005(2) -0.008(2) -0.028(2)
C10A 0.041(2) 0.055(3) 0.056(3) -0.006(2) 0.0006(19) -0.028(2)
C11A 0.040(2) 0.042(2) 0.043(2) -0.0080(19) 0.0075(17) -0.0189(18)
N12A 0.0353(16) 0.0303(17) 0.0336(17) -0.0057(13) 0.0032(13) -0.0157(13)
C13A 0.041(2) 0.032(2) 0.036(2) -0.0070(16) -0.0001(16) -0.0178(17)
C14A 0.046(2) 0.033(2) 0.031(2) -0.0047(16) 0.0054(16) -0.0165(18)
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C2B 0.050(2) 0.030(2) 0.039(2) -0.0004(17) -0.0078(18) -0.0172(18)
C3B 0.069(3) 0.035(2) 0.060(3) 0.000(2) -0.014(2) -0.022(2)
C4B 0.059(3) 0.030(2) 0.070(3) -0.014(2) -0.011(2) -0.008(2)
C5B 0.039(2) 0.041(2) 0.050(3) -0.017(2) -0.0072(18) -0.0082(19)
C6B 0.045(2) 0.058(3) 0.060(3) -0.032(3) 0.000(2) -0.010(2)
C7B 0.038(2) 0.071(3) 0.052(3) -0.027(3) 0.003(2) -0.014(2)
C8B 0.037(2) 0.066(3) 0.037(2) -0.020(2) 0.0046(17) -0.021(2)
C9B 0.055(3) 0.092(4) 0.035(2) -0.010(3) 0.012(2) -0.038(3)
C10B 0.075(3) 0.075(4) 0.037(3) -0.006(2) 0.011(2) -0.048(3)
C11B 0.055(3) 0.054(3) 0.035(2) -0.0056(19) 0.0030(18) -0.032(2)
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C13B 0.0300(18) 0.048(2) 0.035(2) -0.0128(18) -0.0004(15) -0.0151(17)
C14B 0.0341(19) 0.037(2) 0.038(2) -0.0117(17) -0.0015(15) -0.0142(16)
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C3C 0.045(2) 0.047(3) 0.066(3) -0.015(2) -0.005(2) -0.027(2)
C4C 0.037(2) 0.049(3) 0.054(3) -0.011(2) -0.0055(19) -0.016(2)
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C14C 0.0358(19) 0.031(2) 0.0263(18) -0.0064(15) 0.0035(14) -0.0146(16)
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Cl1' 0.0573(7) 0.0563(7) 0.0477(7) -0.0076(5) 0.0054(5) -0.0288(6)
Cl2 0.0664(7) 0.0548(7) 0.0615(8) -0.0220(6) 0.0267(6) -0.0371(6)
Cl2' 0.0664(7) 0.0548(7) 0.0615(8) -0.0220(6) 0.0267(6) -0.0371(6)
Cl3 0.0672(8) 0.0642(8) 0.0684(9) -0.0285(6) 0.0255(6) -0.0441(7)

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Cl3' 0.0672(8) 0.0642(8) 0.0684(9) -0.0285(6) 0.0255(6) -0.0441(7)
C1SC 0.145(8) 0.086(5) 0.085(5) 0.003(4) -0.024(5) -0.041(5)
C2SC 0.145(8) 0.065(4) 0.049(4) 0.001(3) 0.012(4) -0.020(5)
N3SC 0.163(8) 0.119(6) 0.101(6) 0.019(4) 0.070(6) 0.002(6)
C1SD 0.091(5) 0.098(5) 0.072(4) -0.012(4) 0.028(3) -0.045(4)
C2SD 0.044(2) 0.066(3) 0.063(3) -0.032(3) 0.016(2) -0.023(2)
N3SD 0.065(3) 0.063(3) 0.074(3) -0.024(3) -0.001(2) -0.018(2)

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 All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix.  The cell esds are taken
 into account individually in the estimation of esds in distances, angles
 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry.  An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
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Cr1 N1C 2.067(3) . ?
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C5A C6A 1.435(7) . ?
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C9A C10A 1.365(7) . ?
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Cl1 O13 1.491(10) . ?
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C6A C7A C8A 121.0(4) . . ?
C13A C8A C9A 117.2(4) . . ?
C13A C8A C7A 118.4(4) . . ?
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N1A C14A C5A 123.2(4) . . ?
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C5C C14C C13C 119.8(3) . . ?
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O21 Cl2 O22 101.2(8) . . ?
O23 Cl2 O22 109.7(7) . . ?
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C5SA N6SA N6SA 121(2) . 2_666 ?
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Appendix D
Crystal structure data of [Cr(4,7-dimethyl-1,10-phenanthroline)$_3$] (PF_6)$_3$

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'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
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'O'  'O'   0.0106   0.0060
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_symmetry_space_group_name_H-M    P2(1)/c

loop_
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 'x, -y-1/2, z-1/2'

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 _cell_length_c                 13.8832(13)
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 _cell_angle_beta                103.411(2)
 _cell_angle_gamma               90.00
 _cell_volume                   9888.9(16)
 _cell_formula_units_Z           8
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goodness of fit S are based on F^2^, conventional R-factors R are based
on F, with F set to zero for negative F^2^. The threshold expression of
F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
not relevant to the choice of reflections for refinement.  R-factors based
on F^2^ are statistically about twice as large as those based on F, and R-
factors based on ALL data will be even larger.
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Cr2 Cr -0.40937(9) 0.37517(4) -0.14015(13) 0.0065(4) Uiso 1 1 d . . .
N1A N 0.0918(5) 0.1030(2) -0.1059(7) 0.008(2) Uiso 1 1 d . . .
N2A N 0.0410(5) 0.1668(2) -0.1061(7) 0.009(2) Uiso 1 1 d . . .
C1A C 0.1183(6) 0.0713(3) -0.1051(9) 0.018(3) Uiso 1 1 d . . .
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C2A C 0.1649(7) 0.0619(3) -0.1624(9) 0.019(3) Uiso 1 1 d . . .
H2A H 0.1813 0.0389 -0.1597 0.023 Uiso 1 1 calc R . .
C3A C 0.1890(7) 0.0839(3) -0.2229(10) 0.021(3) Uiso 1 1 d . . .
C4A C 0.1626(6) 0.1173(3) -0.2266(9) 0.014(3) Uiso 1 1 d . . .
C5A C 0.1128(6) 0.1265(3) -0.1694(9) 0.016(3) Uiso 1 1 d . . .
C6A C 0.0833(6) 0.1601(3) -0.1702(9) 0.014(3) Uiso 1 1 d . . .
C7A C 0.1023(6) 0.1847(3) -0.2343(9) 0.014(3) Uiso 1 1 d . . .
C8A C 0.0711(8) 0.2176(3) -0.2324(11) 0.031(3) Uiso 1 1 d . . .
C9A C 0.0298(7) 0.2234(3) -0.1653(9) 0.019(3) Uiso 1 1 d . . .
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C10A C 0.0151(7) 0.1980(3) -0.1026(9) 0.019(3) Uiso 1 1 d . . .
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C11A C 0.1786(8) 0.1430(3) -0.2941(11) 0.033(4) Uiso 1 1 d . . .
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C12A C 0.1491(8) 0.1755(3) -0.2950(11) 0.034(4) Uiso 1 1 d . . .
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H13B H 0.2249 0.0550 -0.3233 0.049 Uiso 1 1 calc R . .
H13C H 0.2897 0.0673 -0.2333 0.049 Uiso 1 1 calc R . .
C14A C 0.0873(9) 0.2448(4) -0.3015(12) 0.048(4) Uiso 1 1 d . . .
H14A H 0.1179 0.2352 -0.3433 0.072 Uiso 1 1 calc R . .
H14B H 0.1134 0.2638 -0.2627 0.072 Uiso 1 1 calc R . .
H14C H 0.0410 0.2531 -0.3435 0.072 Uiso 1 1 calc R . .
```

N1B N -0.0539(5) 0.1474(2) 0.0399(7) 0.012(2) Uiso 1 1 d . . .
N2B N -0.0721(5) 0.1129(2) -0.1280(7) 0.017(2) Uiso 1 1 d . . .
C1B C -0.0443(7) 0.1657(3) 0.1231(9) 0.020(3) Uiso 1 1 d . . .
H1B H 0.0047 0.1710 0.1567 0.024 Uiso 1 1 calc R . .
C2B C -0.0993(6) 0.1771(3) 0.1631(9) 0.017(3) Uiso 1 1 d . . .
H2B H -0.0888 0.1890 0.2245 0.020 Uiso 1 1 calc R . .
C3B C -0.1693(7) 0.1714(3) 0.1140(10) 0.021(3) Uiso 1 1 d . . .
C4B C -0.1853(7) 0.1511(3) 0.0264(9) 0.018(3) Uiso 1 1 d . . .
C5B C -0.1239(6) 0.1405(3) -0.0063(8) 0.007(2) Uiso 1 1 d . . .
C6B C -0.1332(6) 0.1216(3) -0.0991(9) 0.012(3) Uiso 1 1 d . . .
C7B C -0.2052(6) 0.1140(3) -0.1547(9) 0.014(3) Uiso 1 1 d . . .
C8B C -0.2106(7) 0.0961(3) -0.2449(9) 0.018(3) Uiso 1 1 d . . .
C9B C -0.1488(6) 0.0888(3) -0.2732(10) 0.018(3) Uiso 1 1 d . . .
H9B H -0.1518 0.0778 -0.3351 0.021 Uiso 1 1 calc R . .
C10B C -0.0805(6) 0.0967(3) -0.2143(9) 0.014(3) Uiso 1 1 d . . .
H10B H -0.0378 0.0904 -0.2362 0.017 Uiso 1 1 calc R . .
C11B C -0.2553(7) 0.1412(3) -0.0306(10) 0.022(3) Uiso 1 1 d . . .
H11B H -0.2974 0.1480 -0.0078 0.026 Uiso 1 1 calc R . .
C12B C -0.2663(7) 0.1229(3) -0.1149(9) 0.017(3) Uiso 1 1 d . . .
H12B H -0.3146 0.1160 -0.1475 0.021 Uiso 1 1 calc R . .
C13B C -0.2334(7) 0.1853(3) 0.1544(10) 0.028(3) Uiso 1 1 d . . .
H13D H -0.2801 0.1788 0.1096 0.042 Uiso 1 1 calc R . .
H13E H -0.2300 0.2102 0.1590 0.042 Uiso 1 1 calc R . .
H13F H -0.2310 0.1757 0.2202 0.042 Uiso 1 1 calc R . .
C14B C -0.2857(7) 0.0859(3) -0.3067(11) 0.032(4) Uiso 1 1 d . . .
H14D H -0.3239 0.0935 -0.2735 0.048 Uiso 1 1 calc R . .
H14E H -0.2880 0.0611 -0.3143 0.048 Uiso 1 1 calc R . .
H14F H -0.2938 0.0967 -0.3721 0.048 Uiso 1 1 calc R . .
N1C N 0.0195(5) 0.0809(2) 0.0554(7) 0.015(2) Uiso 1 1 d . . .
N2C N 0.1115(5) 0.1327(2) 0.0892(7) 0.014(2) Uiso 1 1 d . . .
C1C C -0.0275(6) 0.0555(2) 0.0358(8) 0.008(2) Uiso 1 1 d . . .
H1C H -0.0651 0.0561 -0.0234 0.010 Uiso 1 1 calc R . .
C2C C -0.0237(7) 0.0275(3) 0.0999(9) 0.018(3) Uiso 1 1 d . . .
H2C H -0.0590 0.0097 0.0842 0.021 Uiso 1 1 calc R . .
C3C C 0.0310(7) 0.0258(3) 0.1853(10) 0.019(3) Uiso 1 1 d . . .
C4C C 0.0829(7) 0.0524(3) 0.2068(9) 0.017(3) Uiso 1 1 d . . .
C5C C 0.0750(6) 0.0791(3) 0.1406(9) 0.015(3) Uiso 1 1 d . . .
C6C C 0.1257(6) 0.1073(3) 0.1564(9) 0.014(3) Uiso 1 1 d . . .
C7C C 0.1864(7) 0.1063(3) 0.2391(10) 0.027(3) Uiso 1 1 d . . .
C8C C 0.2361(7) 0.1350(3) 0.2529(10) 0.027(3) Uiso 1 1 d . . .
C9C C 0.2209(8) 0.1605(3) 0.1814(11) 0.030(3) Uiso 1 1 d . . .
H9C H 0.2535 0.1795 0.1870 0.036 Uiso 1 1 calc R . .
C10C C 0.1602(7) 0.1594(3) 0.1026(10) 0.026(3) Uiso 1 1 d . . .
H10C H 0.1520 0.1776 0.0563 0.032 Uiso 1 1 calc R . .
C11C C 0.1423(7) 0.0530(3) 0.2900(10) 0.020(3) Uiso 1 1 d . . .
H11C H 0.1475 0.0349 0.3367 0.024 Uiso 1 1 calc R . .
C12C C 0.1927(7) 0.0787(3) 0.3062(10) 0.028(3) Uiso 1 1 d . . .
H12C H 0.2325 0.0781 0.3630 0.034 Uiso 1 1 calc R . .
C13C C 0.0362(9) -0.0044(4) 0.2543(12) 0.046(4) Uiso 1 1 d . . .
H13G H 0.0784 -0.0014 0.3106 0.070 Uiso 1 1 calc R . .

C11E C -0.5010(6) 0.4445(3) 0.1343(9) 0.015(3) Uiso 1 1 d . . .
H11E H -0.5049 0.4628 0.1777 0.018 Uiso 1 1 calc R . .
C12E C -0.5390(7) 0.4151(3) 0.1376(10) 0.026(3) Uiso 1 1 d . . .
H12E H -0.5696 0.4135 0.1833 0.031 Uiso 1 1 calc R . .
C13E C -0.4118(7) 0.5072(3) 0.1251(10) 0.028(3) Uiso 1 1 d . . .
H13M H -0.4439 0.5026 0.1703 0.042 Uiso 1 1 calc R . .
H13N H -0.4298 0.5272 0.0846 0.042 Uiso 1 1 calc R . .
H13O H -0.3616 0.5114 0.1635 0.042 Uiso 1 1 calc R . .
C14E C -0.6221(8) 0.3499(4) 0.1497(12) 0.042(4) Uiso 1 1 d . . .
H14M H -0.6229 0.3708 0.1880 0.062 Uiso 1 1 calc R . .
H14N H -0.6031 0.3311 0.1949 0.062 Uiso 1 1 calc R . .
H14O H -0.6721 0.3445 0.1125 0.062 Uiso 1 1 calc R . .
N1F N -0.3153(5) 0.3577(2) -0.0440(7) 0.013(2) Uiso 1 1 d . . .
N2F N -0.3320(5) 0.3910(2) -0.2120(7) 0.009(2) Uiso 1 1 d . . .
C1F C -0.3105(7) 0.3406(3) 0.0399(9) 0.017(3) Uiso 1 1 d . . .
H1F H -0.3537 0.3367 0.0633 0.020 Uiso 1 1 calc R . .
C2F C -0.2445(7) 0.3286(3) 0.0931(10) 0.021(3) Uiso 1 1 d . . .
H2F H -0.2436 0.3160 0.1518 0.025 Uiso 1 1 calc R . .
C3F C -0.1801(7) 0.3339(3) 0.0664(10) 0.024(3) Uiso 1 1 d . . .
C4F C -0.1856(7) 0.3533(3) -0.0250(9) 0.017(3) Uiso 1 1 d . . .
C5F C -0.2529(6) 0.3642(3) -0.0719(9) 0.012(3) Uiso 1 1 d . . .
C6F C -0.2604(6) 0.3823(2) -0.1663(8) 0.008(2) Uiso 1 1 d . . .
C7F C -0.1989(6) 0.3902(3) -0.2016(8) 0.008(2) Uiso 1 1 d . . .
C8F C -0.2125(7) 0.4098(3) -0.2915(9) 0.016(3) Uiso 1 1 d . . .
C9F C -0.2834(6) 0.4174(3) -0.3386(9) 0.018(3) Uiso 1 1 d . . .
H9F H -0.2926 0.4290 -0.4003 0.022 Uiso 1 1 calc R . .
C10F C -0.3411(7) 0.4087(3) -0.2993(9) 0.021(3) Uiso 1 1 d . . .
H10F H -0.3894 0.4150 -0.3336 0.025 Uiso 1 1 calc R . .
C11F C -0.1222(7) 0.3607(3) -0.0652(10) 0.023(3) Uiso 1 1 d . . .
H11F H -0.0751 0.3525 -0.0320 0.028 Uiso 1 1 calc R . .
C12F C -0.1290(7) 0.3787(3) -0.1470(10) 0.025(3) Uiso 1 1 d . . .
H12F H -0.0862 0.3842 -0.1700 0.030 Uiso 1 1 calc R . .
C13F C -0.1072(8) 0.3193(4) 0.1253(12) 0.040(4) Uiso 1 1 d . . .
H13P H -0.0675 0.3258 0.0935 0.060 Uiso 1 1 calc R . .
H13Q H -0.1106 0.2943 0.1271 0.060 Uiso 1 1 calc R . .
H13R H -0.0966 0.3282 0.1930 0.060 Uiso 1 1 calc R . .
C14F C -0.1482(8) 0.4224(3) -0.3335(11) 0.037(4) Uiso 1 1 d . . .
H14P H -0.1017 0.4145 -0.2910 0.056 Uiso 1 1 calc R . .
H14Q H -0.1486 0.4474 -0.3359 0.056 Uiso 1 1 calc R . .
H14R H -0.1532 0.4133 -0.4005 0.056 Uiso 1 1 calc R . .
P1 P -0.1842(2) 0.23861(9) -0.1236(3) 0.0313(10) Uani 1 1 d . . .
P2 P -0.1692(3) 0.48384(10) -0.0708(3) 0.0384(11) Uani 1 1 d . . .
P3 P 0.2286(2) -0.00436(8) 0.0964(3) 0.0240(9) Uani 1 1 d . . .
P4 P -0.6167(2) 0.25504(10) -0.2285(3) 0.0354(10) Uani 1 1 d . . .
P5 P 0.0871(2) 0.16746(12) 0.4052(3) 0.0448(12) Uani 1 1 d . . .
P6 P -0.4740(2) 0.40739(10) -0.5713(3) 0.0334(10) Uani 1 1 d . . .
F1 F -0.1274(7) 0.2415(3) -0.0239(9) 0.107(5) Uani 1 1 d . . .
F2 F -0.2434(7) 0.2229(3) -0.0716(10) 0.096(4) Uani 1 1 d . . .
F3 F -0.2398(6) 0.2351(3) -0.2270(8) 0.087(4) Uani 1 1 d . . .
F4 F -0.1260(6) 0.2543(2) -0.1765(10) 0.090(4) Uani 1 1 d . . .

F5 F -0.2144(5) 0.2757(2) -0.1108(7) 0.049(2) Uani 1 1 d . . .
F6 F -0.1535(5) 0.20093(19) -0.1383(7) 0.054(3) Uani 1 1 d . . .
F7 F -0.2094(6) 0.5123(3) -0.0205(8) 0.080(4) Uani 1 1 d . . .
F8 F -0.1269(6) 0.4565(2) -0.1251(9) 0.079(3) Uani 1 1 d . . .
F9 F -0.0980(6) 0.4858(2) 0.0169(8) 0.073(3) Uani 1 1 d . . .
F10 F -0.2398(5) 0.4812(2) -0.1590(7) 0.066(3) Uani 1 1 d . . .
F11 F -0.2016(6) 0.4533(2) -0.0189(8) 0.076(3) Uani 1 1 d . . .
F12 F -0.1363(5) 0.5131(2) -0.1301(8) 0.069(3) Uani 1 1 d . . .
F13 F 0.1455(6) -0.0039(4) 0.0823(10) 0.140(6) Uani 1 1 d . . .
F14 F 0.2270(9) -0.0432(2) 0.0805(8) 0.123(6) Uani 1 1 d . . .
F15 F 0.3110(6) -0.0031(5) 0.1117(13) 0.185(9) Uani 1 1 d . . .
F16 F 0.2363(8) -0.0073(3) 0.2103(8) 0.101(4) Uani 1 1 d . . .
F17 F 0.2198(7) -0.0012(3) -0.0174(8) 0.095(4) Uani 1 1 d . . .
F18 F 0.2270(8) 0.0353(2) 0.1113(9) 0.106(5) Uani 1 1 d . . .
F19 F -0.7019(5) 0.2506(3) -0.2674(8) 0.078(3) Uani 1 1 d . . .
F20 F -0.6275(6) 0.2667(3) -0.1228(8) 0.084(4) Uani 1 1 d . . .
F21 F -0.6081(6) 0.2171(2) -0.1900(7) 0.070(3) Uani 1 1 d . . .
F22 F -0.6014(5) 0.2435(2) -0.3310(7) 0.063(3) Uani 1 1 d . . .
F23 F -0.5300(5) 0.2617(2) -0.1890(7) 0.058(3) Uani 1 1 d . . .
F24 F -0.6242(5) 0.2933(2) -0.2648(8) 0.059(3) Uani 1 1 d . . .
F25 F 0.1145(7) 0.1998(3) 0.4756(9) 0.108(5) Uani 1 1 d . . .
F26 F 0.0122(6) 0.1871(3) 0.3626(8) 0.082(3) Uani 1 1 d . . .
F27 F 0.1621(5) 0.1485(3) 0.4509(8) 0.081(4) Uani 1 1 d . . .
F28 F 0.0524(5) 0.1517(2) 0.4899(7) 0.061(3) Uani 1 1 d . . .
F29 F 0.1199(6) 0.1837(3) 0.3210(8) 0.079(3) Uani 1 1 d . . .
F30 F 0.0567(6) 0.1365(2) 0.3350(7) 0.062(3) Uani 1 1 d . . .
F31 F -0.5532(5) 0.3915(3) -0.6031(7) 0.067(3) Uani 1 1 d . . .
F32 F -0.5034(6) 0.4399(2) -0.6389(6) 0.062(3) Uani 1 1 d . . .
F33 F -0.4510(5) 0.3895(2) -0.6626(6) 0.053(3) Uani 1 1 d . . .
F34 F -0.3962(6) 0.4244(2) -0.5426(8) 0.069(3) Uani 1 1 d . . .
F35 F -0.4447(6) 0.3758(2) -0.5026(7) 0.059(3) Uani 1 1 d . . .
F36 F -0.4961(5) 0.42557(19) -0.4788(6) 0.048(2) Uani 1 1 d . . .
O1 O 0.0190(7) 0.0827(3) -0.3980(16) 0.141(9) Uani 1 1 d . . .
O2 O -0.4216(10) 0.1950(4) -0.1311(13) 0.127(7) Uani 1 1 d . . .
O3 O -0.5712(10) 0.5106(4) -0.6845(14) 0.126(6) Uiso 1 1 d . . .
O4 O 0.0382(10) 0.0658(4) 0.4674(13) 0.122(6) Uani 1 1 d . . .
O5 O -0.4110(8) 0.3203(4) -0.7316(14) 0.092(5) Uani 1 1 d . . .
O6 O -0.4007(10) 0.3320(4) 0.1975(11) 0.104(6) Uani 1 1 d . . .
O7 O -0.7521(12) 0.2570(9) -0.0204(16) 0.207(12) Uani 1 1 d . . .
O8 O 0.2346(6) 0.2056(7) -0.0463(10) 0.147(9) Uani 1 1 d . . .

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P1 0.027(2) 0.029(2) 0.036(3) 0.0170(19) 0.0051(19) 0.0062(18)

P2 0.052(3) 0.030(2) 0.028(3) -0.0070(19) 0.000(2) -0.011(2)
P3 0.026(2) 0.0178(18) 0.027(2) -0.0001(16) 0.0025(17) -0.0106(16)
P4 0.044(3) 0.036(2) 0.026(3) 0.0004(19) 0.007(2) -0.001(2)
P5 0.037(3) 0.064(3) 0.032(3) 0.010(2) 0.005(2) -0.003(2)
P6 0.042(3) 0.035(2) 0.023(2) -0.0029(18) 0.008(2) 0.005(2)
F1 0.113(10) 0.094(9) 0.082(10) -0.016(7) -0.041(8) 0.035(8)
F2 0.108(10) 0.076(7) 0.137(12) 0.041(7) 0.095(9) 0.017(7)
F3 0.088(8) 0.093(8) 0.063(8) -0.032(6) -0.018(6) 0.046(7)
F4 0.075(8) 0.054(6) 0.163(13) 0.049(7) 0.076(8) 0.015(6)
F5 0.049(6) 0.046(5) 0.049(7) -0.001(5) 0.003(5) 0.008(5)
F6 0.045(6) 0.035(5) 0.084(8) 0.019(5) 0.019(5) 0.005(4)
F7 0.076(8) 0.089(8) 0.084(9) -0.041(7) 0.034(7) -0.010(7)
F8 0.084(8) 0.055(6) 0.095(10) -0.034(6) 0.015(7) -0.006(6)
F9 0.082(8) 0.068(7) 0.052(7) -0.013(6) -0.020(6) -0.004(6)
F10 0.071(7) 0.068(6) 0.045(7) 0.002(5) -0.017(5) -0.023(6)
F11 0.076(8) 0.072(7) 0.076(9) 0.033(6) 0.010(6) -0.026(6)
F12 0.066(7) 0.067(6) 0.068(8) 0.010(6) 0.000(6) -0.020(6)
F13 0.053(8) 0.246(17) 0.122(12) -0.124(12) 0.021(8) -0.035(10)
F14 0.31(2) 0.020(5) 0.049(8) 0.000(5) 0.058(10) 0.014(8)
F15 0.040(7) 0.30(2) 0.206(18) -0.189(16) 0.020(9) 0.004(11)
F16 0.193(14) 0.073(7) 0.027(7) 0.007(5) 0.009(8) 0.044(8)
F17 0.160(12) 0.081(8) 0.056(8) 0.016(6) 0.050(8) 0.037(8)
F18 0.203(15) 0.038(6) 0.110(11) -0.008(6) 0.103(11) -0.012(8)
F19 0.036(6) 0.112(9) 0.087(9) 0.007(7) 0.016(6) -0.009(6)
F20 0.104(9) 0.093(8) 0.067(9) -0.029(6) 0.046(7) -0.044(7)
F21 0.125(10) 0.030(5) 0.055(7) 0.010(5) 0.022(7) 0.002(6)
F22 0.075(7) 0.077(7) 0.041(7) -0.016(5) 0.024(6) -0.018(6)
F23 0.045(6) 0.055(6) 0.068(8) 0.001(5) 0.002(5) 0.006(5)
F24 0.058(7) 0.038(5) 0.084(8) 0.013(5) 0.024(6) 0.013(5)
F25 0.139(12) 0.127(10) 0.056(9) -0.033(8) 0.021(8) -0.065(9)
F26 0.095(9) 0.073(7) 0.074(9) -0.007(6) 0.011(7) 0.015(7)
F27 0.044(6) 0.138(10) 0.060(8) 0.031(7) 0.010(6) 0.004(7)
F28 0.046(6) 0.091(7) 0.050(7) 0.013(6) 0.019(5) 0.000(6)
F29 0.098(9) 0.098(8) 0.048(7) 0.025(6) 0.032(7) -0.004(7)
F30 0.101(8) 0.036(5) 0.046(7) -0.008(5) 0.010(6) 0.005(5)
F31 0.052(6) 0.097(8) 0.050(7) -0.003(6) 0.011(5) -0.010(6)
F32 0.108(9) 0.056(6) 0.024(6) 0.006(5) 0.020(6) 0.032(6)
F33 0.057(6) 0.065(6) 0.034(6) -0.007(5) 0.005(5) 0.020(5)
F34 0.076(8) 0.076(7) 0.060(8) -0.012(6) 0.022(6) -0.024(6)
F35 0.089(8) 0.050(5) 0.034(6) 0.012(5) 0.006(5) 0.021(6)
F36 0.070(7) 0.040(5) 0.038(6) -0.005(4) 0.022(5) 0.010(5)
O1 0.031(8) 0.083(10) 0.27(2) 0.094(13) -0.055(11) -0.036(8)
O2 0.171(17) 0.131(14) 0.086(14) -0.003(11) 0.048(13) 0.071(13)
O4 0.144(16) 0.117(13) 0.109(16) -0.004(11) 0.040(13) 0.026(12)
O5 0.074(11) 0.080(10) 0.122(16) -0.014(11) 0.017(11) -0.016(9)
O6 0.156(16) 0.127(13) 0.046(11) 0.026(9) 0.059(11) -0.002(11)
O7 0.139(19) 0.38(4) 0.104(19) 0.06(2) 0.025(15) 0.05(2)
O8 0.019(7) 0.38(3) 0.042(10) 0.095(14) 0.004(7) 0.010(12)

_geom_special_details

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

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Cr1 N2B 2.054(10) . ?
Cr1 N1C 2.060(9) . ?
Cr1 N1A 2.065(9) . ?
Cr1 N2A 2.065(9) . ?
Cr1 N1B 2.076(9) . ?
Cr2 N1D 2.036(10) . ?
Cr2 N2E 2.040(8) . ?
Cr2 N2F 2.034(9) . ?
Cr2 N2D 2.059(9) . ?
Cr2 N1F 2.062(10) . ?
Cr2 N1E 2.073(9) . ?
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N2A C10A 1.322(13) . ?
N2A C6A 1.346(13) . ?
C1A C2A 1.360(15) . ?
C2A C3A 1.351(16) . ?
C3A C4A 1.399(15) . ?
C3A C13A 1.477(16) . ?
C4A C5A 1.403(15) . ?
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Appendix E
Crystal structure data of [Cr(4,7-dimethyl-1,10-phenanthroline)$_2$]$_2$(μ-OH)(Cl$_3$)(ClO$_4$)$_2$

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'C28 H34.50 Cl2.50 Cr N4 O8'
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'-x, y, -z+1/2'
'x+1/2, y+1/2, z'
'-x+1/2, y+1/2, -z+1/2'
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'-x, -y, -z'
'x, -y, z-1/2'
'-x+1/2, -y+1/2, -z'
'x+1/2, -y+1/2, z-1/2'

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_refine_special_details
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Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
goodness of fit S are based on F^2^, conventional R-factors R are based
on F, with F set to zero for negative F^2^. The threshold expression of
F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
not relevant to the choice of reflections for refinement.  R-factors based
on F^2^ are statistically about twice as large as those based on F, and R-
factors based on ALL data will be even larger.
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'calc w=1/[\s^2^(Fo^2^)+(0.1000P)^2^+0.0000P] where P=(Fo^2^+2Fc^2^)/3'
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O1 O 0.0000 0.3996(3) 0.2500 0.0392(17) Uiso 1 2 d S . .
Cl1 Cl 0.1709(2) 0.44882(12) 0.19870(12) 0.0918(9) Uani 1 1 d . . .
Cl2 Cl 0.0000 0.53531(18) 0.2500 0.227(5) Uani 1 2 d S . .
Cl3 Cl -0.01685(17) 0.23062(11) -0.03095(9) 0.0583(7) Uani 1 1 d . . .
O2 O -0.0700(5) 0.2830(3) -0.0300(3) 0.0696(18) Uani 1 1 d . . .
O3 O 0.0568(5) 0.2208(3) 0.0311(3) 0.086(2) Uani 1 1 d . . .
O4 O -0.0952(5) 0.1868(3) -0.0439(3) 0.0706(18) Uani 1 1 d . . .
O5 O 0.0405(4) 0.2314(3) -0.0827(2) 0.0691(18) Uani 1 1 d . . .
N1 N -0.0275(4) 0.3043(2) 0.1547(2) 0.0316(14) Uani 1 1 d . . .
N2 N 0.1696(4) 0.3195(3) 0.2254(3) 0.0392(15) Uani 1 1 d . . .
N3 N 0.1133(4) 0.3645(2) 0.0892(3) 0.0356(15) Uani 1 1 d . . .
N4 N -0.0527(4) 0.4224(2) 0.1091(2) 0.0313(13) Uani 1 1 d . . .
C1 C -0.1261(6) 0.2974(3) 0.1189(3) 0.0380(18) Uani 1 1 d . . .
H1 H -0.1680 0.3299 0.1039 0.046 Uiso 1 1 calc R . .
C2 C -0.1702(6) 0.2445(4) 0.1026(3) 0.043(2) Uani 1 1 d . . .
H2 H -0.2414 0.2418 0.0771 0.052 Uiso 1 1 calc R . .
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C4 C -0.0114(6) 0.2014(3) 0.1605(3) 0.0412(19) Uani 1 1 d . . .
C5 C 0.0297(6) 0.2554(3) 0.1750(3) 0.0365(18) Uani 1 1 d . . .
C6 C 0.1375(6) 0.2644(3) 0.2133(3) 0.0384(18) Uani 1 1 d . . .
C7 C 0.2032(6) 0.2179(4) 0.2353(3) 0.044(2) Uani 1 1 d . . .
C8 C 0.3079(7) 0.2293(4) 0.2722(4) 0.050(2) Uani 1 1 d . . .
C9 C 0.3377(6) 0.2854(4) 0.2838(4) 0.056(2) Uani 1 1 d . . .
H9 H 0.4078 0.2940 0.3083 0.068 Uiso 1 1 calc R . .
C10 C 0.2679(6) 0.3292(4) 0.2607(3) 0.046(2) Uani 1 1 d . . .
H10 H 0.2907 0.3671 0.2702 0.055 Uiso 1 1 calc R . .
C11 C 0.0590(7) 0.1550(3) 0.1848(4) 0.050(2) Uani 1 1 d . . .
H11 H 0.0336 0.1174 0.1755 0.060 Uiso 1 1 calc R . .
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C12 C 0.1590(7) 0.1628(4) 0.2199(4) 0.050(2) Uani 1 1 d . . .
H12 H 0.2019 0.1307 0.2350 0.060 Uiso 1 1 calc R . .
C13 C -0.1643(8) 0.1398(4) 0.1018(4) 0.067(3) Uani 1 1 d . . .
H13A H -0.1617 0.1167 0.1413 0.101 Uiso 1 1 calc R . .
H13B H -0.1250 0.1206 0.0730 0.101 Uiso 1 1 calc R . .
H13C H -0.2387 0.1449 0.0779 0.101 Uiso 1 1 calc R . .
C14 C 0.3855(7) 0.1821(4) 0.2985(4) 0.075(3) Uani 1 1 d . . .
H14A H 0.4565 0.1980 0.3162 0.112 Uiso 1 1 calc R . .
H14B H 0.3884 0.1557 0.2625 0.112 Uiso 1 1 calc R . .
H14C H 0.3618 0.1619 0.3337 0.112 Uiso 1 1 calc R . .
C15 C 0.1961(6) 0.3358(3) 0.0807(3) 0.044(2) Uani 1 1 d . . .
H15 H 0.2388 0.3166 0.1177 0.053 Uiso 1 1 calc R . .
C16 C 0.2249(7) 0.3321(3) 0.0198(4) 0.053(2) Uani 1 1 d . . .
H16 H 0.2861 0.3111 0.0162 0.063 Uiso 1 1 calc R . .
C17 C 0.1643(6) 0.3588(3) -0.0347(3) 0.045(2) Uani 1 1 d . . .
C18 C 0.0749(6) 0.3893(3) -0.0274(3) 0.0374(19) Uani 1 1 d . . .
C19 C 0.0518(5) 0.3907(3) 0.0356(3) 0.0296(16) Uani 1 1 d . . .
C20 C -0.0385(5) 0.4220(3) 0.0464(3) 0.0318(17) Uani 1 1 d . . .
C21 C -0.1068(5) 0.4488(3) -0.0058(3) 0.0335(17) Uani 1 1 d . . .
C22 C -0.1961(6) 0.4785(3) 0.0073(3) 0.0396(18) Uani 1 1 d . . .
C23 C -0.2098(6) 0.4770(3) 0.0714(3) 0.0430(19) Uani 1 1 d . . .
H23 H -0.2699 0.4950 0.0816 0.052 Uiso 1 1 calc R . .
C24 C -0.1379(6) 0.4499(3) 0.1201(3) 0.0383(18) Uani 1 1 d . . .
H24 H -0.1486 0.4505 0.1639 0.046 Uiso 1 1 calc R . .
C25 C 0.0033(6) 0.4189(3) -0.0794(3) 0.042(2) Uani 1 1 d . . .
H25 H 0.0167 0.4186 -0.1225 0.050 Uiso 1 1 calc R . .
C26 C -0.0827(6) 0.4471(3) -0.0695(3) 0.0379(18) Uani 1 1 d . . .
H26 H -0.1279 0.4661 -0.1056 0.045 Uiso 1 1 calc R . .
C27 C 0.1962(7) 0.3545(4) -0.1004(4) 0.064(3) Uani 1 1 d . . .
H27A H 0.2663 0.3363 -0.0936 0.096 Uiso 1 1 calc R . .
H27B H 0.1996 0.3924 -0.1188 0.096 Uiso 1 1 calc R . .
H27C H 0.1434 0.3318 -0.1313 0.096 Uiso 1 1 calc R . .
C28 C -0.2756(6) 0.5083(3) -0.0470(4) 0.054(2) Uani 1 1 d . . .
H28A H -0.2376 0.5323 -0.0723 0.080 Uiso 1 1 calc R . .
H28B H -0.3228 0.5318 -0.0273 0.080 Uiso 1 1 calc R . .
H28C H -0.3181 0.4803 -0.0764 0.080 Uiso 1 1 calc R . .
O6 O 0.0052(17) 0.0737(7) 0.0057(11) 0.162(9) Uani 0.50 1 d P . .
O7 O 0.0581(10) 0.1126(5) -0.1840(6) 0.205(6) Uani 1 1 d . . .
O8 O -0.073(2) 0.0956(8) -0.1224(11) 0.185(10) Uani 0.50 1 d P . .
O9 O -0.098(2) -0.0187(8) -0.2726(9) 0.213(12) Uani 0.50 1 d P . .
O10 O -0.0591(5) -0.0019(3) -0.1399(4) 0.108(3) Uani 1 1 d . . .

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Cl1 0.116(2) 0.095(2) 0.0600(16) 0.0069(14) 0.0118(15) -0.0213(18)
Cl2 0.569(14) 0.050(3) 0.032(2) 0.000 0.007(4) 0.000
Cl3 0.0550(13) 0.0847(19) 0.0372(11) 0.0012(11) 0.0152(10) -0.0160(13)
O2 0.079(4) 0.056(4) 0.078(4) -0.013(3) 0.027(4) -0.024(4)
O3 0.062(4) 0.164(7) 0.036(3) 0.012(4) 0.017(3) -0.009(4)
O4 0.067(4) 0.058(4) 0.089(5) 0.003(3) 0.024(3) -0.016(3)
O5 0.065(4) 0.113(5) 0.032(3) 0.008(3) 0.018(3) -0.014(4)
N1 0.037(3) 0.043(4) 0.018(3) 0.000(3) 0.013(2) 0.002(3)
N2 0.036(4) 0.055(5) 0.027(3) 0.003(3) 0.008(3) -0.002(3)
N3 0.037(3) 0.049(4) 0.025(3) 0.001(3) 0.015(3) -0.004(3)
N4 0.037(3) 0.038(4) 0.022(3) 0.000(3) 0.014(2) -0.004(3)
C1 0.040(4) 0.055(5) 0.022(3) -0.004(3) 0.014(3) -0.003(4)
C2 0.050(5) 0.056(6) 0.028(4) -0.008(4) 0.018(4) -0.007(5)
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C4 0.053(5) 0.045(5) 0.035(4) -0.010(4) 0.031(4) -0.011(4)
C5 0.043(4) 0.052(5) 0.021(3) -0.002(3) 0.020(3) -0.001(4)
C6 0.047(5) 0.053(5) 0.021(3) 0.003(4) 0.020(3) 0.000(4)
C7 0.048(5) 0.063(6) 0.025(4) 0.011(4) 0.020(4) 0.018(4)
C8 0.054(5) 0.073(7) 0.031(4) 0.013(4) 0.022(4) 0.018(5)
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C10 0.041(5) 0.072(6) 0.025(4) 0.000(4) 0.006(3) -0.006(4)
C11 0.078(6) 0.039(5) 0.044(5) -0.002(4) 0.037(5) 0.001(5)
C12 0.068(6) 0.052(6) 0.041(5) 0.010(4) 0.032(4) 0.025(5)
C13 0.085(7) 0.064(7) 0.062(6) -0.014(5) 0.036(5) -0.019(5)
C14 0.076(6) 0.102(8) 0.048(5) 0.005(5) 0.019(5) 0.040(6)
C15 0.049(5) 0.053(5) 0.035(4) 0.004(4) 0.021(4) 0.001(4)
C16 0.061(5) 0.057(6) 0.051(5) -0.003(4) 0.035(4) -0.003(4)
C17 0.057(5) 0.053(5) 0.030(4) -0.007(4) 0.018(4) -0.017(4)
C18 0.050(5) 0.040(5) 0.026(4) -0.006(3) 0.015(3) -0.024(4)
C19 0.039(4) 0.029(4) 0.022(3) -0.002(3) 0.010(3) -0.014(3)
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C21 0.034(4) 0.036(4) 0.029(4) -0.003(3) 0.005(3) -0.012(3)
C22 0.041(4) 0.035(5) 0.038(4) 0.005(3) -0.001(3) -0.018(4)
C23 0.039(4) 0.048(5) 0.043(4) 0.008(4) 0.013(4) 0.001(4)
C24 0.051(5) 0.041(5) 0.027(4) 0.005(3) 0.017(3) -0.001(4)
C25 0.062(5) 0.046(5) 0.020(3) -0.006(3) 0.015(3) -0.026(4)
C26 0.051(5) 0.039(5) 0.021(3) 0.002(3) 0.004(3) -0.019(4)
C27 0.076(6) 0.091(7) 0.035(4) -0.012(5) 0.035(4) -0.013(5)
C28 0.054(5) 0.048(5) 0.052(5) 0.007(4) -0.002(4) -0.005(4)
O6 0.21(2) 0.099(14) 0.24(2) 0.028(13) 0.186(19) 0.061(13)
O7 0.191(11) 0.185(12) 0.222(13) 0.047(9) 0.016(10) -0.042(10)
O8 0.31(3) 0.096(14) 0.18(2) -0.010(13) 0.10(2) -0.036(17)
O9 0.40(3) 0.149(16) 0.104(15) -0.079(13) 0.090(19) -0.18(2)
O10 0.076(5) 0.097(6) 0.137(7) -0.060(5) -0.003(4) 0.040(4)

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All esds (except the esd in the dihedral angle between two l.s. planes)
are estimated using the full covariance matrix. The cell esds are taken

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 into account individually in the estimation of esds in distances, angles
 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry.  An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
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Cr1 N1 2.053(6) . ?
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Cr1 N2 2.060(6) . ?
Cr1 N3 2.079(5) . ?
Cr1 Cl1 2.193(3) . ?
O1 Cr1 1.964(2) 2 ?
Cl3 O2 1.419(6) . ?
Cl3 O4 1.428(6) . ?
Cl3 O3 1.431(6) . ?
Cl3 O5 1.440(5) . ?
N1 C1 1.326(8) . ?
N1 C5 1.384(9) . ?
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N3 C15 1.312(9) . ?
N3 C19 1.357(8) . ?
N4 C24 1.341(8) . ?
N4 C20 1.358(7) . ?
C1 C2 1.382(10) . ?
C2 C3 1.353(10) . ?
C3 C4 1.389(10) . ?
C3 C13 1.506(10) . ?
C4 C5 1.389(10) . ?
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C5 C6 1.447(10) . ?
C6 C7 1.400(10) . ?
C7 C8 1.415(11) . ?
C7 C12 1.427(11) . ?
C8 C9 1.388(12) . ?
C8 C14 1.514(11) . ?
C9 C10 1.383(11) . ?
C11 C12 1.338(11) . ?
C15 C16 1.403(9) . ?
C16 C17 1.370(11) . ?
C17 C18 1.398(11) . ?
C17 C27 1.519(9) . ?
C18 C19 1.409(8) . ?
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C18 C25 1.429(10) . ?
C19 C20 1.441(9) . ?
C20 C21 1.382(9) . ?
C21 C22 1.427(10) . ?
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N1 Cr1 N2 80.1(2) . . ?
N4 Cr1 N2 167.7(2) . . ?
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N2 Cr1 N3 91.0(2) . . ?
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Cr1 O1 Cr1 148.7(4) 2 . ?
O2 Cl3 O4 108.3(4) . . ?
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O4 Cl3 O3 110.1(4) . . ?
O2 Cl3 O5 109.5(4) . . ?
O4 Cl3 O5 109.4(4) . . ?
O3 Cl3 O5 109.0(3) . . ?
C1 N1 C5 116.1(6) . . ?
C1 N1 Cr1 129.3(5) . . ?
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C10 N2 C6 118.3(7) . . ?
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C6 N2 Cr1 113.2(5) . . ?
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N1 C1 C2 122.4(7) . . ?
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C2 C3 C4 118.2(7) . . ?
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N1 C5 C4 123.6(7) . . ?
N1 C5 C6 114.9(7) . . ?
C4 C5 C6 121.6(7) . . ?
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N2 C6 C5 116.8(7) . . ?
C7 C6 C5 119.7(7) . . ?
C6 C7 C8 117.1(8) . . ?
C6 C7 C12 117.7(7) . . ?
C8 C7 C12 125.3(8) . . ?
C9 C8 C7 117.9(8) . . ?
C9 C8 C14 120.6(8) . . ?
C7 C8 C14 121.4(9) . . ?
C10 C9 C8 121.6(8) . . ?
N2 C10 C9 121.5(8) . . ?
C12 C11 C4 122.3(8) . . ?
C11 C12 C7 122.2(8) . . ?
N3 C15 C16 123.3(7) . . ?
C17 C16 C15 119.7(8) . . ?
C16 C17 C18 118.4(7) . . ?
C16 C17 C27 119.3(8) . . ?
C18 C17 C27 122.4(7) . . ?
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C24 C23 C22 120.5(7) . . ?
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C25 C26 C21 121.2(7) . . ?

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Appendix F
Crystal structure data of [Cr(1,10-phenanthroline-5,6-dione)$_2$(OH)(H$_2$O)(ClO$_4$)$_2$

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'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
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'-x, -y, -z'
'x, -y-1/2, z-1/2'
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_cell_volume                    2663.5(3)
_cell_formula_units_Z            4
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F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
not relevant to the choice of reflections for refinement.  R-factors based
on F^2^ are statistically about twice as large as those based on F, and R-
factors based on ALL data will be even larger.
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H1W1 H 0.9175 0.8673 0.0262 0.047 Uiso 1 1 d R . .
H1W2 H 0.9495 0.8211 0.1231 0.047 Uiso 1 1 d R . .
O2W O 0.7007(3) 0.8712(3) -0.0132(3) 0.0468(10) Uani 1 1 d . . .
H2W1 H 0.7409 0.8844 -0.0680 0.056 Uiso 1 1 d R . .
H2W2 H 0.6299 0.8412 -0.0180 0.056 Uiso 1 1 d R . .
N1A N 0.8003(3) 0.8942(3) 0.2536(3) 0.0331(9) Uani 1 1 d . . .
C2A C 0.8051(4) 0.8274(4) 0.3187(4) 0.0378(12) Uani 1 1 d . . .
H2A H 0.7798 0.7633 0.3071 0.045 Uiso 1 1 calc R . .
C3A C 0.8457(5) 0.8482(4) 0.4024(4) 0.0420(13) Uani 1 1 d . . .
H3A H 0.8488 0.7992 0.4474 0.050 Uiso 1 1 calc R . .
C4A C 0.8818(4) 0.9417(4) 0.4193(4) 0.0380(12) Uani 1 1 d . . .
H4A H 0.9103 0.9578 0.4762 0.046 Uiso 1 1 calc R . .
C5A C 0.8759(4) 1.0113(4) 0.3524(3) 0.0306(10) Uani 1 1 d . . .
C6A C 0.9184(4) 1.1109(4) 0.3675(4) 0.0341(11) Uani 1 1 d . . .
O6A O 0.9535(3) 1.1385(3) 0.4385(3) 0.0460(10) Uani 1 1 d . . .
C7A C 0.9218(4) 1.1806(4) 0.2862(4) 0.0331(11) Uani 1 1 d . . .
O7A O 0.9717(3) 1.2555(3) 0.2914(3) 0.0452(10) Uani 1 1 d . . .
C8A C 0.8658(4) 1.1509(4) 0.2039(3) 0.0316(10) Uani 1 1 d . . .
C9A C 0.8590(4) 1.2138(4) 0.1318(4) 0.0358(11) Uani 1 1 d . . .
H9A H 0.8869 1.2781 0.1350 0.043 Uiso 1 1 calc R . .
C10A C 0.8105(4) 1.1807(4) 0.0546(4) 0.0399(12) Uani 1 1 d . . .
H10A H 0.8037 1.2222 0.0039 0.048 Uiso 1 1 calc R . .
C11A C 0.7725(4) 1.0869(4) 0.0525(4) 0.0359(11) Uani 1 1 d . . .
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N12A N 0.7802(3) 1.0251(3) 0.1207(3) 0.0301(9) Uani 1 1 d . . .
C13A C 0.8255(4) 1.0574(4) 0.1962(3) 0.0282(10) Uani 1 1 d . . .
C14A C 0.8339(4) 0.9853(4) 0.2701(3) 0.0283(10) Uani 1 1 d . . .
N1B N 0.5868(3) 0.8813(3) 0.1516(3) 0.0294(9) Uani 1 1 d . . .
C2B C 0.5366(4) 0.9646(4) 0.1665(4) 0.0346(11) Uani 1 1 d . . .
H2B H 0.5747 1.0238 0.1707 0.042 Uiso 1 1 calc R . .
C3B C 0.4301(4) 0.9687(4) 0.1763(4) 0.0368(12) Uani 1 1 d . . .
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C4B C 0.3742(4) 0.8842(4) 0.1671(4) 0.0365(12) Uani 1 1 d . . .
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C5B C 0.4251(4) 0.7969(4) 0.1510(3) 0.0315(10) Uani 1 1 d . . .
C6B C 0.3672(4) 0.7048(4) 0.1391(3) 0.0369(12) Uani 1 1 d . . .
O6B O 0.2744(3) 0.6998(3) 0.1377(3) 0.0443(10) Uani 1 1 d . . .
C7B C 0.4298(5) 0.6101(4) 0.1275(4) 0.0430(14) Uani 1 1 d . . .
O7B O 0.3847(4) 0.5313(4) 0.1229(4) 0.0676(15) Uani 1 1 d . . .
C8B C 0.5435(5) 0.6169(4) 0.1228(4) 0.0372(12) Uani 1 1 d . . .
C9B C 0.6041(5) 0.5348(4) 0.1098(4) 0.0469(14) Uani 1 1 d . . .
H9B H 0.5735 0.4722 0.1016 0.056 Uiso 1 1 calc R . .
C10B C 0.7108(5) 0.5451(4) 0.1091(4) 0.0461(14) Uani 1 1 d . . .
H10B H 0.7543 0.4898 0.1028 0.055 Uiso 1 1 calc R . .
C11B C 0.7504(5) 0.6368(4) 0.1178(4) 0.0430(14) Uani 1 1 d . . .
H11B H 0.8229 0.6436 0.1155 0.052 Uiso 1 1 calc R . .
N12B N 0.6953(3) 0.7175(3) 0.1293(3) 0.0333(9) Uani 1 1 d . . .
C13B C 0.5910(4) 0.7071(4) 0.1317(3) 0.0298(10) Uani 1 1 d . . .
C14B C 0.5317(4) 0.7982(4) 0.1452(3) 0.0290(10) Uani 1 1 d . . .
Cl1 Cl 0.11191(10) 0.03098(10) 0.16209(9) 0.0391(3) Uani 1 1 d . . .
O11 O 0.1249(4) -0.0690(4) 0.1361(4) 0.0732(15) Uani 1 1 d . . .
O12 O 0.0522(4) 0.0832(5) 0.0966(3) 0.0740(17) Uani 1 1 d . . .
O13 O 0.2125(3) 0.0746(4) 0.1687(3) 0.0542(11) Uani 1 1 d . . .
O14 O 0.0645(3) 0.0369(4) 0.2475(3) 0.0529(11) Uani 1 1 d . . .
Cl2 Cl 0.53438(10) 0.23267(10) 0.10791(8) 0.0366(3) Uani 1 1 d . . .
O21 O 0.4459(4) 0.2743(5) 0.1475(4) 0.0784(18) Uani 1 1 d . . .
O22 O 0.5872(4) 0.3038(3) 0.0564(3) 0.0581(12) Uani 1 1 d . . .
O23 O 0.6009(3) 0.1947(3) 0.1764(3) 0.0439(10) Uani 1 1 d . . .
O24 O 0.5042(3) 0.1530(3) 0.0494(3) 0.0417(9) Uani 1 1 d . . .

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O1W 0.032(2) 0.045(2) 0.041(2) 0.0062(17) 0.0015(16) 0.0047(16)
O2W 0.038(2) 0.070(3) 0.032(2) 0.0040(18) -0.0030(17) -0.0141(19)
N1A 0.034(2) 0.032(2) 0.033(2) 0.0031(17) -0.0012(18) -0.0009(17)
C2A 0.042(3) 0.034(3) 0.037(3) 0.006(2) 0.000(2) -0.005(2)
C3A 0.050(3) 0.042(3) 0.034(3) 0.011(2) -0.001(2) -0.005(2)
C4A 0.040(3) 0.044(3) 0.031(3) 0.002(2) -0.004(2) 0.000(2)
C5A 0.027(3) 0.034(3) 0.030(2) 0.000(2) -0.0012(19) 0.0012(19)
C6A 0.030(3) 0.038(3) 0.034(3) -0.005(2) -0.003(2) 0.003(2)
O6A 0.052(3) 0.043(2) 0.042(2) -0.0035(17) -0.0171(19) 0.0004(18)
C7A 0.031(3) 0.028(2) 0.040(3) -0.005(2) 0.004(2) 0.0019(19)
O7A 0.052(3) 0.036(2) 0.048(2) -0.0065(17) -0.0039(19) -0.0085(18)
C8A 0.026(2) 0.031(2) 0.037(3) 0.001(2) 0.001(2) 0.0055(18)

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C9A 0.035(3) 0.030(3) 0.042(3) 0.004(2) 0.002(2) 0.004(2)
C10A 0.036(3) 0.042(3) 0.042(3) 0.010(2) -0.002(2) -0.001(2)
C11A 0.035(3) 0.041(3) 0.032(3) 0.005(2) -0.001(2) 0.001(2)
N12A 0.026(2) 0.035(2) 0.030(2) 0.0009(17) -0.0020(16) 0.0013(16)
C13A 0.023(2) 0.032(2) 0.029(2) 0.0004(19) 0.0025(18) -0.0008(18)
C14A 0.023(2) 0.029(2) 0.033(2) -0.0020(19) 0.0015(18) 0.0002(17)
N1B 0.030(2) 0.028(2) 0.031(2) 0.0028(16) 0.0008(16) -0.0005(16)
C2B 0.035(3) 0.031(3) 0.039(3) 0.001(2) 0.000(2) 0.001(2)
C3B 0.036(3) 0.036(3) 0.038(3) 0.005(2) 0.003(2) 0.005(2)
C4B 0.030(3) 0.045(3) 0.035(3) 0.006(2) 0.000(2) 0.003(2)
C5B 0.030(3) 0.039(3) 0.025(2) 0.0047(19) -0.0011(19) -0.001(2)
C6B 0.036(3) 0.045(3) 0.030(2) 0.008(2) -0.002(2) -0.008(2)
O6B 0.0275(19) 0.061(3) 0.045(2) 0.0132(19) -0.0013(16) -0.0097(18)
C7B 0.052(4) 0.036(3) 0.041(3) 0.003(2) -0.004(3) -0.017(2)
O7B 0.059(3) 0.050(3) 0.094(4) 0.009(3) -0.007(3) -0.023(2)
C8B 0.048(3) 0.032(3) 0.032(3) 0.003(2) 0.001(2) -0.003(2)
C9B 0.066(4) 0.029(3) 0.045(3) 0.000(2) 0.005(3) -0.003(3)
C10B 0.064(4) 0.030(3) 0.045(3) 0.003(2) 0.012(3) 0.012(3)
C11B 0.046(4) 0.043(3) 0.040(3) 0.003(2) 0.004(3) 0.011(2)
N12B 0.037(2) 0.033(2) 0.030(2) 0.0015(17) 0.0014(17) 0.0024(18)
C13B 0.036(3) 0.031(2) 0.023(2) 0.0023(18) -0.0010(19) -0.004(2)
C14B 0.030(2) 0.032(2) 0.025(2) 0.0034(18) -0.0016(18) -0.0035(19)
Cl1 0.0316(7) 0.0466(8) 0.0393(7) 0.0030(5) 0.0069(5) 0.0042(5)
O11 0.075(4) 0.057(3) 0.088(4) -0.015(3) 0.015(3) -0.005(3)
O12 0.052(3) 0.122(5) 0.048(3) 0.023(3) 0.008(2) 0.033(3)
O13 0.043(2) 0.066(3) 0.053(3) 0.009(2) 0.009(2) -0.006(2)
O14 0.041(2) 0.075(3) 0.044(2) 0.011(2) 0.0110(19) 0.012(2)
Cl2 0.0403(7) 0.0361(7) 0.0332(6) -0.0042(5) -0.0034(5) 0.0058(5)
O21 0.066(3) 0.111(5) 0.058(3) -0.020(3) -0.006(3) 0.042(3)
O22 0.082(3) 0.034(2) 0.059(3) 0.006(2) -0.009(2) -0.010(2)
O23 0.053(3) 0.044(2) 0.034(2) -0.0044(17) -0.0086(18) 0.0051(18)
O24 0.046(2) 0.043(2) 0.036(2) -0.0021(17) -0.0053(17) -0.0065(17)

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 All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix.  The cell esds are taken
 into account individually in the estimation of esds in distances, angles
 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry.  An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
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Cr1 N12A 2.164(4) . ?
Cr1 N12B 2.186(4) . ?
N1A C2A 1.337(7) . ?
N1A C14A 1.340(6) . ?
C2A C3A 1.383(8) . ?
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Cl1 O12 1.434(5) . ?
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N1B Cr1 O1W 167.30(16) . . ?
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C4B C3B C2B 118.8(5) . . ?
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C14B C5B C6B 120.8(5) . . ?
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O6B C6B C7B 118.7(5) . . ?
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O7B C7B C6B 119.2(6) . . ?
C8B C7B C6B 118.8(5) . . ?
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C8B C9B C10B 119.2(5) . . ?
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N1B C14B C13B 116.1(4) . . ?
C5B C14B C13B 121.4(5) . . ?
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O11 Cl1 O13 107.6(3) . . ?
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Appendix G
Crystal structure data of [Ru(1,10-phenanthroline)$_3$](PF$_6$)$_2$

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;
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 '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'
 'F'  'F'   0.0171   0.0103
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'P'  'P'   0.1023   0.0942
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'Ru'  'Ru'  -1.2594   0.8363
 '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'

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_symmetry_space_group_name_H-M    Cc

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'

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_cell_length_b                  15.878(2)
_cell_length_c                  12.0933(17)
_cell_angle_alpha               90.00
_cell_angle_beta                101.426(2)
_cell_angle_gamma               90.00
_cell_volume                    6956.9(17)
_cell_formula_units_Z           8
_cell_measurement_temperature   153(2)
_cell_measurement_reflns_used   ?
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_cell_measurement_theta_max     ?

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_exptl_special_details
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_computing_data_reduction        'Bruker SHELXTL'
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Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
goodness of fit S are based on F^2^, conventional R-factors R are based
on F, with F set to zero for negative F^2^. The threshold expression of
F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
not relevant to the choice of reflections for refinement.  R-factors based
on F^2^ are statistically about twice as large as those based on F, and R-
factors based on ALL data will be even larger.
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_refine_ls_weighting_details
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_atom_sites_solution_secondary   difmap
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'Flack H D (1983), Acta Cryst. A39, 876-881'
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N1A N 0.5472(2) 0.8788(6) 0.6679(7) 0.041(2) Uani 1 1 d . . .
C2A C 0.5274(3) 0.8638(10) 0.7455(8) 0.059(4) Uani 1 1 d . . .
H2A H 0.5232 0.8080 0.7680 0.070 Uiso 1 1 calc R . .
C3A C 0.5125(3) 0.9353(9) 0.7947(9) 0.051(3) Uani 1 1 d . . .
H3A H 0.4984 0.9265 0.8512 0.061 Uiso 1 1 calc R . .
C4A C 0.5184(3) 1.0160(13) 0.7614(12) 0.086(6) Uani 1 1 d . . .
H4A H 0.5072 1.0620 0.7919 0.103 Uiso 1 1 calc R . .
C5A C 0.5407(3) 1.0308(8) 0.6833(10) 0.049(3) Uani 1 1 d . . .
C6A C 0.5499(4) 1.1085(8) 0.6437(13) 0.072(5) Uani 1 1 d . . .
H6A H 0.5408 1.1571 0.6751 0.087 Uiso 1 1 calc R . .
C7A C 0.5705(4) 1.1214(7) 0.5648(12) 0.062(3) Uani 1 1 d . . .
H7A H 0.5755 1.1767 0.5416 0.075 Uiso 1 1 calc R . .
C8A C 0.5847(3) 1.0486(6) 0.5169(11) 0.050(3) Uani 1 1 d . . .
C9A C 0.6056(3) 1.0552(8) 0.4323(11) 0.057(3) Uani 1 1 d . . .
H9A H 0.6115 1.1085 0.4050 0.068 Uiso 1 1 calc R . .
C10A C 0.6168(3) 0.9849(9) 0.3922(12) 0.061(4) Uani 1 1 d . . .
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C11A C 0.6060(3) 0.9055(7) 0.4260(8) 0.040(2) Uani 1 1 d . . .
H11A H 0.6131 0.8567 0.3902 0.049 Uiso 1 1 calc R . .
N12A N 0.5863(2) 0.8959(4) 0.5053(7) 0.0275(17) Uani 1 1 d . . .
C13A C 0.5754(3) 0.9671(6) 0.5512(8) 0.039(2) Uani 1 1 d . . .
C14A C 0.5540(3) 0.9580(7) 0.6348(9) 0.042(3) Uani 1 1 d . . .
N1B N 0.5567(2) 0.6873(6) 0.6654(7) 0.040(2) Uani 1 1 d . . .
C2B C 0.5288(3) 0.6342(7) 0.6419(10) 0.054(3) Uani 1 1 d . . .
H2B H 0.5127 0.6400 0.5706 0.064 Uiso 1 1 calc R . .
C3B C 0.5212(4) 0.5727(10) 0.7100(13) 0.086(6) Uani 1 1 d . . .
H3B H 0.4997 0.5387 0.6896 0.104 Uiso 1 1 calc R . .
C4B C 0.5472(3) 0.5598(9) 0.8170(11) 0.073(4) Uani 1 1 d . . .
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C5B C 0.5775(3) 0.6107(8) 0.8395(10) 0.056(3) Uani 1 1 d . . .
C6B C 0.6052(3) 0.6043(8) 0.9452(10) 0.057(3) Uani 1 1 d . . .
H6B H 0.6028 0.5617 0.9984 0.068 Uiso 1 1 calc R . .
C7B C 0.6333(3) 0.6565(7) 0.9668(9) 0.047(3) Uani 1 1 d . . .
H7B H 0.6503 0.6521 1.0366 0.057 Uiso 1 1 calc R . .
C8B C 0.6388(3) 0.7199(6) 0.8876(8) 0.035(2) Uani 1 1 d . . .
C9B C 0.6689(3) 0.7757(7) 0.9018(10) 0.042(3) Uani 1 1 d . . .
H9B H 0.6869 0.7740 0.9698 0.051 Uiso 1 1 calc R . .
C10B C 0.6729(3) 0.8329(6) 0.8187(9) 0.042(2) Uani 1 1 d . . .
H10B H 0.6935 0.8698 0.8274 0.051 Uiso 1 1 calc R . .
C11B C 0.6443(3) 0.8341(6) 0.7180(8) 0.035(2) Uani 1 1 d . . .
H11B H 0.6466 0.8727 0.6597 0.042 Uiso 1 1 calc R . .
N12B N 0.6160(2) 0.7852(4) 0.7038(7) 0.027(2) Uani 1 1 d . . .
C13B C 0.6126(3) 0.7272(6) 0.7831(9) 0.033(2) Uani 1 1 d . . .
C14B C 0.5809(3) 0.6725(7) 0.7623(9) 0.042(2) Uani 1 1 d . . .
N1C N 0.5886(2) 0.7094(4) 0.4626(7) 0.0304(18) Uani 1 1 d . . .
C2C C 0.6202(3) 0.6731(5) 0.4683(8) 0.032(2) Uani 1 1 d . . .
H2C H 0.6386 0.6796 0.5348 0.039 Uiso 1 1 calc R . .
C3C C 0.6284(3) 0.6234(7) 0.3772(10) 0.046(3) Uani 1 1 d . . .
H3C H 0.6520 0.5978 0.3842 0.056 Uiso 1 1 calc R . .
C4C C 0.6039(3) 0.6131(7) 0.2858(9) 0.057(3) Uani 1 1 d . . .
H4C H 0.6100 0.5808 0.2258 0.068 Uiso 1 1 calc R . .
C5C C 0.5681(3) 0.6492(7) 0.2734(8) 0.045(3) Uani 1 1 d . . .
C6C C 0.5375(3) 0.6391(8) 0.1793(9) 0.054(3) Uani 1 1 d . . .
H6C H 0.5408 0.6048 0.1177 0.065 Uiso 1 1 calc R . .
C7C C 0.5046(3) 0.6756(7) 0.1749(10) 0.053(3) Uani 1 1 d . . .
H7C H 0.4860 0.6693 0.1089 0.063 Uiso 1 1 calc R . .
C8C C 0.4967(3) 0.7246(6) 0.2685(8) 0.035(2) Uani 1 1 d . . .
C9C C 0.4640(3) 0.7652(7) 0.2666(10) 0.041(3) Uani 1 1 d . . .
H9C H 0.4443 0.7605 0.2032 0.049 Uiso 1 1 calc R . .
C10C C 0.4605(3) 0.8119(7) 0.3562(9) 0.044(3) Uani 1 1 d . . .
H10C H 0.4378 0.8398 0.3565 0.053 Uiso 1 1 calc R . .
C11C C 0.4895(3) 0.8204(7) 0.4494(9) 0.040(2) Uani 1 1 d . . .
H11C H 0.4862 0.8538 0.5118 0.048 Uiso 1 1 calc R . .
N12C N 0.5219(2) 0.7817(5) 0.4513(7) 0.0260(19) Uani 1 1 d . . .
C13C C 0.5258(3) 0.7350(6) 0.3617(7) 0.0255(19) Uani 1 1 d . . .
C14C C 0.5611(3) 0.6958(6) 0.3660(7) 0.031(2) Uani 1 1 d . . .
Ru1B Ru 0.812606(19) 0.76637(4) 0.83090(4) 0.02803(18) Uani 1 1 d . . .
N1F N 0.7999(2) 0.8852(5) 0.8811(6) 0.0272(16) Uani 1 1 d . . .
C2F C 0.7792(3) 0.9086(6) 0.9558(8) 0.034(2) Uani 1 1 d . . .
H2F H 0.7688 0.8660 0.9951 0.041 Uiso 1 1 calc R . .
C3F C 0.7724(3) 0.9922(7) 0.9777(11) 0.053(3) Uani 1 1 d . . .
H3F H 0.7580 1.0065 1.0318 0.064 Uiso 1 1 calc R . .
C4F C 0.7873(3) 1.0557(7) 0.9182(10) 0.048(3) Uani 1 1 d . . .
H4F H 0.7823 1.1131 0.9312 0.058 Uiso 1 1 calc R . .
C5F C 0.8088(3) 1.0354(6) 0.8418(9) 0.039(2) Uani 1 1 d . . .
C6F C 0.8248(3) 1.0943(7) 0.7766(11) 0.055(3) Uani 1 1 d . . .
H6F H 0.8202 1.1527 0.7846 0.065 Uiso 1 1 calc R . .
C7F C 0.8464(3) 1.0702(8) 0.7034(12) 0.062(4) Uani 1 1 d . . .

H7F H 0.8577 1.1120 0.6656 0.074 Uiso 1 1 calc R . .
C8F C 0.8523(3) 0.9833(7) 0.6822(9) 0.039(2) Uani 1 1 d . . .
C9F C 0.8734(3) 0.9522(8) 0.6083(10) 0.053(3) Uani 1 1 d . . .
H9F H 0.8850 0.9904 0.5658 0.064 Uiso 1 1 calc R . .
C10F C 0.8778(3) 0.8666(7) 0.5953(9) 0.046(3) Uani 1 1 d . . .
H10F H 0.8923 0.8460 0.5445 0.055 Uiso 1 1 calc R . .
C11F C 0.8607(3) 0.8108(7) 0.6578(8) 0.042(2) Uani 1 1 d . . .
H11F H 0.8634 0.7520 0.6478 0.050 Uiso 1 1 calc R . .
N12F N 0.8407(2) 0.8369(5) 0.7304(7) 0.0353(19) Uani 1 1 d . . .
C13F C 0.8357(3) 0.9215(7) 0.7459(8) 0.037(2) Uani 1 1 d . . .
C14F C 0.8137(3) 0.9486(5) 0.8253(9) 0.035(2) Uani 1 1 d . . .
N1D N 0.7689(3) 0.7637(5) 0.6950(8) 0.031(2) Uani 1 1 d . . .
C2D C 0.7425(3) 0.8213(7) 0.6602(8) 0.041(2) Uani 1 1 d . . .
H2D H 0.7405 0.8683 0.7074 0.049 Uiso 1 1 calc R . .
C3D C 0.7180(3) 0.8142(7) 0.5566(9) 0.046(3) Uani 1 1 d . . .
H3D H 0.6995 0.8558 0.5349 0.055 Uiso 1 1 calc R . .
C4D C 0.7204(3) 0.7487(8) 0.4869(9) 0.050(3) Uani 1 1 d . . .
H4D H 0.7039 0.7452 0.4161 0.060 Uiso 1 1 calc R . .
C5D C 0.7470(3) 0.6863(7) 0.5185(8) 0.040(2) Uani 1 1 d . . .
C6D C 0.7519(4) 0.6148(8) 0.4490(9) 0.057(4) Uani 1 1 d . . .
H6D H 0.7350 0.6056 0.3799 0.068 Uiso 1 1 calc R . .
C7D C 0.7802(4) 0.5613(9) 0.4817(10) 0.056(3) Uani 1 1 d . . .
H7D H 0.7831 0.5155 0.4337 0.067 Uiso 1 1 calc R . .
C8D C 0.8062(3) 0.5706(7) 0.5859(9) 0.042(2) Uani 1 1 d . . .
C9D C 0.8372(3) 0.5201(7) 0.6251(11) 0.052(3) Uani 1 1 d . . .
H9D H 0.8424 0.4745 0.5799 0.062 Uiso 1 1 calc R . .
C10D C 0.8591(3) 0.5340(7) 0.7219(11) 0.051(3) Uani 1 1 d . . .
H10D H 0.8799 0.4988 0.7459 0.062 Uiso 1 1 calc R . .
C11D C 0.8519(3) 0.6028(6) 0.7932(9) 0.041(2) Uani 1 1 d . . .
H11D H 0.8671 0.6104 0.8656 0.049 Uiso 1 1 calc R . .
N12D N 0.8244(2) 0.6545(5) 0.7576(6) 0.0342(18) Uani 1 1 d . . .
C13D C 0.8001(3) 0.6398(6) 0.6564(8) 0.036(2) Uani 1 1 d . . .
C14D C 0.7716(3) 0.6960(7) 0.6225(7) 0.034(2) Uani 1 1 d . . .
N1E N 0.8556(2) 0.7615(4) 0.9697(7) 0.0250(18) Uani 1 1 d . . .
C2E C 0.8903(3) 0.7908(7) 0.9801(9) 0.040(2) Uani 1 1 d . . .
H2E H 0.8972 0.8166 0.9164 0.048 Uiso 1 1 calc R . .
C3E C 0.9167(3) 0.7852(9) 1.0807(12) 0.062(4) Uani 1 1 d . . .
H3E H 0.9412 0.8042 1.0833 0.075 Uiso 1 1 calc R . .
C4E C 0.9069(3) 0.7522(7) 1.1747(9) 0.040(3) Uani 1 1 d . . .
H4E H 0.9243 0.7490 1.2439 0.048 Uiso 1 1 calc R . .
C5E C 0.8714(3) 0.7236(6) 1.1673(8) 0.039(2) Uani 1 1 d . . .
C6E C 0.8570(4) 0.6891(7) 1.2610(9) 0.053(3) Uani 1 1 d . . .
H6E H 0.8733 0.6837 1.3321 0.064 Uiso 1 1 calc R . .
C7E C 0.8218(3) 0.6645(7) 1.2517(8) 0.044(2) Uani 1 1 d . . .
H7E H 0.8133 0.6455 1.3166 0.053 Uiso 1 1 calc R . .
C8E C 0.7970(3) 0.6667(6) 1.1443(8) 0.042(2) Uani 1 1 d . . .
C9E C 0.7607(3) 0.6339(6) 1.1257(9) 0.051(3) Uani 1 1 d . . .
H9E H 0.7509 0.6115 1.1864 0.061 Uiso 1 1 calc R . .
C10E C 0.7401(3) 0.6349(6) 1.0186(8) 0.039(2) Uani 1 1 d . . .
H10E H 0.7162 0.6104 1.0040 0.047 Uiso 1 1 calc R . .

C11E C 0.7539(3) 0.6717(6) 0.9299(9) 0.037(2) Uani 1 1 d . . .
H11E H 0.7386 0.6737 0.8569 0.044 Uiso 1 1 calc R . .
N12E N 0.7882(2) 0.7044(4) 0.9452(6) 0.0276(17) Uani 1 1 d . . .
C13E C 0.8099(3) 0.6998(5) 1.0521(8) 0.036(2) Uani 1 1 d . . .
C14E C 0.8462(3) 0.7294(5) 1.0636(8) 0.031(2) Uani 1 1 d . . .
P1 P 0.94908(10) 0.9962(3) 0.4127(3) 0.0802(13) Uani 0.653(14) 1 d PD A 2
F11 F 0.9370(4) 0.9011(8) 0.4223(12) 0.102(6) Uiso 0.653(14) 1 d PD A 2
F12 F 0.9497(4) 0.9973(12) 0.5497(9) 0.096(5) Uiso 0.653(14) 1 d PD A 2
F13 F 0.9090(2) 1.0281(7) 0.3940(8) 0.053(3) Uiso 0.653(14) 1 d PD A 2
F14 F 0.9495(4) 0.9882(12) 0.2862(10) 0.105(6) Uiso 0.653(14) 1 d PD A 2
F15 F 0.9913(4) 0.9666(12) 0.4447(15) 0.133(7) Uiso 0.653(14) 1 d PD A 2
F16 F 0.9629(6) 1.0928(10) 0.4345(18) 0.162(8) Uiso 0.653(14) 1 d PD A 2
P1' P 0.94908(10) 0.9962(3) 0.4127(3) 0.0802(13) Uani 0.347(14) 1 d PD A 1
F11' F 0.9766(8) 0.9230(16) 0.446(3) 0.141(15) Uiso 0.347(14) 1 d PD A 1
F12' F 0.9498(7) 1.0375(18) 0.5320(16) 0.095(10) Uiso 0.347(14) 1 d PD A 1
F13' F 0.9087(4) 0.9722(17) 0.401(2) 0.091(9) Uiso 0.347(14) 1 d PD A 1
F14' F 0.9455(5) 0.9548(13) 0.2845(11) 0.044(5) Uiso 0.347(14) 1 d PD A 1
F15' F 0.9834(5) 1.0494(12) 0.3890(17) 0.070(7) Uiso 0.347(14) 1 d PD A 1
F16' F 0.9295(9) 1.0854(15) 0.360(3) 0.177(17) Uiso 0.347(14) 1 d PD A 1
P2 P 0.68618(7) 0.46270(14) 0.1992(2) 0.0307(5) Uani 1 1 d D . .
F21 F 0.68516(18) 0.5644(3) 0.2041(6) 0.0453(14) Uani 1 1 d D . .
F22 F 0.72973(15) 0.4658(3) 0.2397(5) 0.0424(14) Uani 1 1 d D . .
F23 F 0.68795(18) 0.3627(3) 0.1966(6) 0.0431(12) Uani 1 1 d D . .
F24 F 0.68907(16) 0.4671(4) 0.0706(4) 0.0444(14) Uani 1 1 d D . .
F25 F 0.64228(14) 0.4609(4) 0.1627(5) 0.0419(13) Uani 1 1 d D . .
F26 F 0.68239(16) 0.4585(4) 0.3284(4) 0.0435(14) Uani 1 1 d D . .
P3 P 0.69737(8) 1.08658(18) 0.6857(3) 0.0517(8) Uani 1 1 d . . .
F31 F 0.6966(2) 1.0945(6) 0.8147(6) 0.090(3) Uani 1 1 d . . .
F32 F 0.6977(3) 1.0759(6) 0.5544(7) 0.098(3) Uani 1 1 d . . .
F33 F 0.6634(2) 1.0236(6) 0.6708(7) 0.083(3) Uani 1 1 d . . .
F34 F 0.6701(2) 1.1640(5) 0.6614(8) 0.098(3) Uani 1 1 d . . .
F35 F 0.7246(2) 1.0101(5) 0.7098(7) 0.087(3) Uani 1 1 d . . .
F36 F 0.7306(3) 1.1492(6) 0.7020(14) 0.156(6) Uani 1 1 d . . .
P4 P 0.93258(8) 0.5717(2) 0.5122(3) 0.0503(7) Uani 1 1 d . . .
F41 F 0.9023(2) 0.6399(7) 0.5212(7) 0.109(4) Uani 1 1 d . . .
F42 F 0.9018(3) 0.5008(8) 0.4846(8) 0.127(5) Uani 1 1 d . . .
F43 F 0.96403(19) 0.6428(5) 0.5376(6) 0.070(2) Uani 1 1 d . . .
F44 F 0.92825(18) 0.5894(6) 0.3810(5) 0.074(2) Uani 1 1 d . . .
F45 F 0.9639(2) 0.5024(5) 0.5036(7) 0.077(2) Uani 1 1 d . . .
F46 F 0.93727(18) 0.5540(5) 0.6440(5) 0.0581(17) Uani 1 1 d . . .
O1W O 0.9896(4) 0.7657(10) 0.3805(12) 0.136(5) Uiso 1 1 d . . .

loop_
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Ru1A 0.0286(4) 0.0305(4) 0.0229(3) -0.0009(3) 0.0045(3) 0.0065(3)
N1A 0.030(4) 0.061(6) 0.030(4) -0.006(4) 0.004(4) -0.002(4)
C2A 0.039(6) 0.109(11) 0.031(5) -0.009(6) 0.014(5) 0.012(7)
C3A 0.042(6) 0.073(9) 0.037(6) -0.013(6) 0.006(5) 0.002(6)
C4A 0.033(6) 0.151(17) 0.065(9) -0.067(10) -0.014(6) 0.036(8)
C5A 0.040(6) 0.052(7) 0.047(6) -0.017(5) -0.009(5) 0.009(5)
C6A 0.063(9) 0.042(7) 0.090(11) -0.025(7) -0.036(8) 0.013(6)
C7A 0.069(8) 0.038(6) 0.067(9) -0.012(6) -0.016(7) 0.000(6)
C8A 0.041(6) 0.017(5) 0.077(9) -0.001(5) -0.024(6) 0.005(4)
C9A 0.047(7) 0.047(7) 0.071(8) 0.017(6) 0.002(6) -0.016(6)
C10A 0.037(6) 0.065(8) 0.073(9) 0.011(7) -0.010(6) -0.018(6)
C11A 0.037(5) 0.051(6) 0.033(5) 0.019(5) 0.005(4) 0.000(5)
N12A 0.023(4) 0.017(4) 0.040(4) -0.001(3) 0.001(3) 0.007(3)
C13A 0.033(5) 0.039(6) 0.038(6) -0.011(4) -0.007(4) 0.005(4)
C14A 0.025(5) 0.040(6) 0.052(6) -0.018(5) -0.015(4) 0.015(4)
N1B 0.028(4) 0.054(6) 0.036(5) 0.008(4) 0.002(3) -0.006(4)
C2B 0.062(8) 0.048(7) 0.048(6) 0.018(5) 0.006(6) -0.014(6)
C3B 0.068(9) 0.104(13) 0.077(10) 0.054(9) -0.010(7) -0.046(9)
C4B 0.060(8) 0.087(10) 0.066(8) 0.048(8) 0.001(6) -0.006(7)
C5B 0.047(6) 0.067(8) 0.052(7) 0.027(6) 0.005(5) -0.006(6)
C6B 0.048(7) 0.076(9) 0.042(6) 0.023(6) -0.001(5) 0.006(6)
C7B 0.053(7) 0.060(7) 0.032(5) 0.011(5) 0.016(5) 0.021(6)
C8B 0.036(6) 0.038(6) 0.027(5) -0.007(4) 0.000(4) 0.018(4)
C9B 0.037(6) 0.052(7) 0.033(6) -0.015(5) -0.007(4) 0.017(5)
C10B 0.037(5) 0.037(6) 0.048(6) -0.002(5) -0.002(5) 0.002(4)
C11B 0.039(5) 0.028(5) 0.034(5) -0.006(4) 0.002(4) 0.001(4)
N12B 0.031(5) 0.026(5) 0.025(4) -0.003(3) 0.007(4) 0.011(3)
C13B 0.034(5) 0.030(5) 0.033(5) -0.010(4) 0.002(4) 0.007(4)
C14B 0.039(6) 0.051(6) 0.044(6) 0.013(5) 0.027(5) 0.003(5)
N1C 0.044(5) 0.015(4) 0.031(4) 0.001(3) 0.005(4) 0.009(3)
C2C 0.033(5) 0.028(5) 0.033(5) -0.009(4) 0.000(4) 0.010(4)
C3C 0.038(6) 0.049(6) 0.052(6) -0.005(5) 0.008(5) 0.023(5)
C4C 0.070(8) 0.048(7) 0.049(7) -0.022(5) 0.005(6) 0.019(6)
C5C 0.045(6) 0.046(6) 0.042(5) -0.016(5) 0.006(5) 0.014(5)
C6C 0.069(8) 0.049(7) 0.038(6) -0.019(5) -0.002(5) 0.002(6)
C7C 0.058(7) 0.043(6) 0.048(6) -0.010(5) -0.010(5) -0.001(5)
C8C 0.045(6) 0.025(5) 0.035(5) 0.000(4) 0.010(5) -0.006(4)
C9C 0.029(5) 0.034(6) 0.057(7) 0.012(5) 0.003(5) -0.009(4)
C10C 0.028(5) 0.052(7) 0.048(6) 0.022(5) -0.005(4) 0.001(5)
C11C 0.036(5) 0.040(6) 0.044(6) 0.008(5) 0.007(4) 0.000(5)
N12C 0.026(4) 0.032(4) 0.020(4) 0.003(3) 0.005(3) 0.002(3)
C13C 0.034(5) 0.018(4) 0.024(4) 0.002(4) 0.004(4) -0.002(4)
C14C 0.040(5) 0.030(5) 0.020(4) 0.000(3) -0.005(4) 0.003(4)
Ru1B 0.0267(4) 0.0337(4) 0.0237(3) 0.0027(3) 0.0051(3) -0.0071(3)
N1F 0.025(4) 0.024(4) 0.033(4) -0.004(3) 0.007(3) -0.003(3)
C2F 0.037(5) 0.027(5) 0.035(5) -0.006(4) 0.001(4) -0.005(4)
C3F 0.045(7) 0.039(7) 0.073(8) -0.011(6) 0.003(6) 0.012(5)
C4F 0.045(6) 0.025(5) 0.064(7) -0.010(5) -0.018(5) 0.008(4)
C5F 0.029(5) 0.037(5) 0.045(6) 0.011(5) -0.007(4) -0.003(4)
C6F 0.048(7) 0.030(6) 0.075(8) 0.014(5) -0.012(6) -0.008(5)

C7F 0.043(7) 0.052(8) 0.080(9) 0.040(7) -0.012(6) -0.017(6)
C8F 0.032(5) 0.038(6) 0.044(6) 0.014(5) -0.002(4) -0.011(4)
C9F 0.032(6) 0.080(9) 0.049(7) 0.029(6) 0.010(5) -0.016(5)
C10F 0.035(5) 0.053(7) 0.055(6) 0.010(5) 0.024(5) -0.007(5)
C11F 0.046(6) 0.049(6) 0.031(5) 0.004(4) 0.012(4) -0.006(5)
N12F 0.036(4) 0.036(5) 0.033(4) 0.007(4) 0.007(3) -0.008(4)
C13F 0.040(6) 0.043(6) 0.022(4) 0.010(4) -0.007(4) -0.015(5)
C14F 0.026(4) 0.033(4) 0.038(5) 0.011(5) -0.011(4) -0.007(4)
N1D 0.033(5) 0.035(5) 0.025(5) 0.002(3) 0.004(4) -0.009(3)
C2D 0.047(6) 0.039(6) 0.035(5) 0.005(4) 0.003(4) -0.010(5)
C3D 0.040(6) 0.057(7) 0.035(6) 0.023(5) -0.005(5) 0.001(5)
C4D 0.053(7) 0.066(8) 0.024(5) 0.012(5) -0.009(5) -0.029(6)
C5D 0.044(6) 0.051(6) 0.022(5) 0.003(4) 0.000(4) -0.018(5)
C6D 0.077(9) 0.073(8) 0.024(5) -0.011(5) 0.018(5) -0.051(7)
C7D 0.060(8) 0.074(9) 0.038(6) -0.018(6) 0.021(6) -0.019(7)
C8D 0.045(6) 0.039(6) 0.046(6) -0.016(4) 0.020(5) -0.017(5)
C9D 0.051(7) 0.042(6) 0.075(8) -0.015(6) 0.043(6) -0.018(5)
C10D 0.042(6) 0.028(5) 0.092(10) -0.002(6) 0.033(6) -0.002(5)
C11D 0.040(6) 0.036(6) 0.046(6) -0.001(4) 0.007(5) -0.005(4)
N12D 0.041(5) 0.035(4) 0.029(4) -0.008(3) 0.012(3) 0.002(4)
C13D 0.038(5) 0.036(5) 0.034(5) -0.008(4) 0.009(4) -0.019(4)
C14D 0.033(5) 0.047(6) 0.021(5) -0.005(4) 0.008(4) -0.018(5)
N1E 0.022(4) 0.022(4) 0.032(5) -0.002(3) 0.007(3) -0.003(3)
C2E 0.031(5) 0.044(6) 0.044(6) -0.002(5) 0.003(4) -0.005(4)
C3E 0.036(6) 0.073(9) 0.077(10) -0.038(7) 0.009(6) -0.002(6)
C4E 0.031(5) 0.052(7) 0.033(6) -0.006(5) -0.006(4) 0.000(5)
C5E 0.054(7) 0.024(5) 0.035(5) -0.002(4) -0.001(5) 0.008(4)
C6E 0.083(9) 0.034(6) 0.035(5) -0.003(4) -0.009(5) 0.010(6)
C7E 0.064(7) 0.039(6) 0.030(5) 0.005(4) 0.013(5) -0.010(5)
C8E 0.065(7) 0.028(5) 0.032(5) 0.004(4) 0.010(5) 0.004(5)
C9E 0.083(8) 0.028(5) 0.049(6) -0.001(4) 0.032(6) -0.018(5)
C10E 0.043(6) 0.035(5) 0.037(5) 0.000(4) 0.005(4) -0.009(4)
C11E 0.042(6) 0.022(5) 0.050(6) 0.002(4) 0.016(5) -0.010(4)
N12E 0.033(4) 0.019(4) 0.032(4) 0.006(3) 0.007(3) 0.010(3)
C13E 0.050(6) 0.018(4) 0.041(5) 0.004(4) 0.012(4) 0.003(4)
C14E 0.036(5) 0.024(5) 0.033(5) -0.003(4) 0.006(4) 0.005(4)
P1 0.0459(19) 0.129(4) 0.066(2) 0.029(2) 0.0103(17) 0.008(2)
P1' 0.0459(19) 0.129(4) 0.066(2) 0.029(2) 0.0103(17) 0.008(2)
P2 0.0374(14) 0.0291(13) 0.0265(10) 0.0029(10) 0.0082(9) -0.0008(10)
F21 0.057(4) 0.019(3) 0.060(3) 0.001(3) 0.011(3) 0.000(3)
F22 0.037(3) 0.032(3) 0.054(4) 0.008(3) 0.001(3) 0.001(2)
F23 0.053(3) 0.025(3) 0.053(3) -0.007(3) 0.016(3) -0.003(3)
F24 0.052(4) 0.054(4) 0.029(3) 0.007(3) 0.013(3) 0.003(3)
F25 0.033(3) 0.046(3) 0.048(3) -0.002(3) 0.012(2) 0.000(2)
F26 0.060(4) 0.046(3) 0.028(3) 0.000(2) 0.017(3) -0.006(3)
P3 0.0480(19) 0.0359(16) 0.064(2) 0.0112(14) -0.0052(14) 0.0023(13)
F31 0.112(7) 0.090(6) 0.056(5) -0.036(4) -0.014(4) 0.010(5)
F32 0.111(7) 0.118(7) 0.070(5) 0.042(5) 0.030(5) 0.061(6)
F33 0.085(6) 0.086(6) 0.064(5) -0.009(4) -0.017(4) -0.039(5)
F34 0.096(6) 0.085(6) 0.118(7) 0.035(5) 0.035(5) 0.057(5)

F35 0.096(6) 0.056(5) 0.088(6) 0.003(4) -0.030(5) 0.034(4)
F36 0.080(6) 0.067(6) 0.309(17) 0.009(8) 0.010(8) -0.036(5)
P4 0.0324(14) 0.073(2) 0.0454(16) -0.0068(15) 0.0085(12) 0.0050(14)
F41 0.082(6) 0.180(10) 0.075(5) 0.034(6) 0.038(4) 0.088(6)
F42 0.088(6) 0.218(13) 0.087(6) -0.066(7) 0.046(5) -0.100(7)
F43 0.061(4) 0.079(5) 0.075(5) -0.016(4) 0.022(4) -0.020(4)
F44 0.047(4) 0.135(7) 0.039(3) 0.006(4) 0.008(3) 0.010(4)
F45 0.073(5) 0.084(6) 0.077(5) -0.014(4) 0.025(4) 0.026(4)
F46 0.054(4) 0.077(5) 0.045(4) -0.002(3) 0.013(3) 0.001(3)

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

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Appendix H
Crystal structure data of [Fe(1,10-phenanthroline-5,6-dione)$_3$](PF$_6$)$_2$

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'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'
'P'  'P'   0.1023   0.0942
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'Fe'  'Fe'   0.3463   0.8444
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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_symmetry_space_group_name_H-M    P2(1)

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

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```

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_cell_angle_beta                93.257(4)
_cell_angle_gamma               90.00
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_cell_formula_units_Z           2
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_diffrn_standards_number        ?
_diffrn_standards_interval_count ?
_diffrn_standards_interval_time ?
_diffrn_standards_decay_%       ?
_diffrn_reflns_number           15533
_diffrn_reflns_av_R_equivalents 0.0904
_diffrn_reflns_av_sigmaI/netI   0.1608
_diffrn_reflns_limit_h_min      -14
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_diffrn_reflns_limit_l_min       -16
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_diffrn_reflns_theta_min         1.56
_diffrn_reflns_theta_max         26.44
_reflns_number_total            8696
_reflns_number_gt               4444
_reflns_threshold_expression     >2sigma(I)

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_computing_cell_refinement       'Bruker SMART'
_computing_data_reduction        'Bruker SHELXTL'
_computing_structure_solution    'SHELXS-97 (Sheldrick, 1990)'
_computing_structure_refinement  'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics    'Bruker SHELXTL'
_computing_publication_material  'Bruker SHELXTL'

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

_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^)+(0.1587P)^2^+0.0000P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary     direct
_atom_sites_solution_secondary   difmap
_atom_sites_solution_hydrogens   geom
_refine_ls_hydrogen_treatment    mixed
_refine_ls_extinction_method     none
_refine_ls_extinction_coef       ?
_refine_ls_abs_structure_details
'Flack H D (1983), Acta Cryst. A39, 876-881'
_refine_ls_abs_structure_Flack   -0.05(4)
_refine_ls_number_reflns         8696
_refine_ls_number_parameters     547
_refine_ls_number_restraints     1
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_refine_ls_R_factor_gt           0.0883
_refine_ls_wR_factor_ref         0.2779
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_refine_ls_goodness_of_fit_ref   0.947
_refine_ls_restrained_S_all      0.947
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Fe1 Fe 0.75555(11) 0.59640(9) 0.19697(9) 0.0287(3) Uani 1 1 d . . .
N1A N 0.7889(7) 0.7279(6) 0.2177(6) 0.0312(19) Uani 1 1 d . . .
C2A C 0.7976(9) 0.5708(8) -0.0274(7) 0.043(3) Uani 1 1 d . . .
H2A H 0.7664 0.5108 -0.0234 0.051 Uiso 1 1 calc R . .
C3A C 0.8380(8) 0.5975(11) -0.1184(7) 0.047(3) Uani 1 1 d . . .
H3A H 0.8328 0.5574 -0.1759 0.056 Uiso 1 1 calc R . .
C4A C 0.8868(10) 0.6836(10) -0.1265(9) 0.055(3) Uani 1 1 d . . .
H4A H 0.9195 0.7015 -0.1881 0.065 Uiso 1 1 calc R . .
C5A C 0.8876(9) 0.7427(9) -0.0450(8) 0.043(3) Uani 1 1 d . . .
C6A C 0.9309(10) 0.8367(10) -0.0455(10) 0.057(3) Uani 1 1 d . . .
O6A O 0.9766(8) 0.8693(7) -0.1193(7) 0.073(3) Uani 1 1 d . . .
C7A C 0.9255(10) 0.9008(9) 0.0523(9) 0.051(3) Uani 1 1 d . . .
O7A O 0.9662(8) 0.9768(7) 0.0493(7) 0.070(3) Uani 1 1 d . . .
C8A C 0.8712(9) 0.8621(8) 0.1386(9) 0.044(3) Uani 1 1 d . . .
C9A C 0.8557(9) 0.9148(8) 0.2268(9) 0.047(3) Uani 1 1 d . . .
H9A H 0.8773 0.9777 0.2309 0.056 Uiso 1 1 calc R . .
C10A C 0.8065(9) 0.8699(8) 0.3088(9) 0.047(3) Uani 1 1 d . . .
H10A H 0.7958 0.9033 0.3700 0.057 Uiso 1 1 calc R . .
C11A C 0.7745(9) 0.7816(7) 0.3030(8) 0.037(2) Uani 1 1 d . . .
H11A H 0.7404 0.7549 0.3599 0.045 Uiso 1 1 calc R . .
N12A N 0.7990(6) 0.6238(6) 0.0573(6) 0.036(2) Uani 1 1 d . . .
C13A C 0.8354(8) 0.7711(7) 0.1375(7) 0.031(2) Uani 1 1 d . . .
C14A C 0.8414(8) 0.7116(7) 0.0469(7) 0.034(2) Uani 1 1 d . . .
N1B N 0.5969(7) 0.6174(6) 0.1556(6) 0.035(2) Uani 1 1 d . . .
C2B C 0.5385(9) 0.6998(8) 0.1497(7) 0.036(2) Uani 1 1 d . . .
H2B H 0.5767 0.7549 0.1697 0.043 Uiso 1 1 calc R . .
C3B C 0.4284(9) 0.7040(8) 0.1159(8) 0.040(3) Uani 1 1 d . . .
H3B H 0.3927 0.7625 0.1102 0.048 Uiso 1 1 calc R . .
C4B C 0.3653(9) 0.6254(7) 0.0891(7) 0.034(2) Uani 1 1 d . . .
H4B H 0.2877 0.6295 0.0671 0.040 Uiso 1 1 calc R . .
C5B C 0.4191(8) 0.5432(8) 0.0957(7) 0.034(2) Uani 1 1 d . . .
C6B C 0.3653(9) 0.4553(8) 0.0730(8) 0.040(3) Uani 1 1 d . . .
O6B O 0.2647(6) 0.4505(6) 0.0499(6) 0.055(2) Uani 1 1 d . . .
```

C7B C 0.4346(10) 0.3634(8) 0.0811(8) 0.040(2) Uani 1 1 d . . .
O7B O 0.3867(7) 0.2915(6) 0.0574(6) 0.055(2) Uani 1 1 d . . .
C8B C 0.5560(9) 0.3705(7) 0.1140(7) 0.036(2) Uani 1 1 d . . .
C9B C 0.6239(10) 0.2916(8) 0.1263(8) 0.039(2) Uani 1 1 d . . .
H9B H 0.5930 0.2322 0.1132 0.047 Uiso 1 1 calc R . .
C10B C 0.7356(10) 0.3020(8) 0.1574(9) 0.046(3) Uani 1 1 d . . .
H10B H 0.7829 0.2499 0.1691 0.055 Uiso 1 1 calc R . .
C11B C 0.7792(9) 0.3930(7) 0.1718(7) 0.036(2) Uani 1 1 d . . .
H11B H 0.8579 0.4004 0.1875 0.044 Uiso 1 1 calc R . .
N12B N 0.7139(7) 0.4685(6) 0.1642(6) 0.0299(18) Uani 1 1 d . . .
C13B C 0.5998(8) 0.4568(7) 0.1362(7) 0.032(2) Uani 1 1 d . . .
C14B C 0.5396(8) 0.5404(7) 0.1277(7) 0.033(2) Uani 1 1 d . . .
N1C N 0.7261(7) 0.5733(5) 0.3423(6) 0.034(2) Uani 1 1 d . . .
C2C C 0.6292(8) 0.5651(7) 0.3858(7) 0.034(2) Uani 1 1 d . . .
H2C H 0.5609 0.5711 0.3445 0.041 Uiso 1 1 calc R . .
C3C C 0.6231(9) 0.5478(9) 0.4902(8) 0.046(3) Uani 1 1 d . . .
H3C H 0.5511 0.5414 0.5180 0.055 Uiso 1 1 calc R . .
C4C C 0.7189(9) 0.5399(8) 0.5539(8) 0.043(3) Uani 1 1 d . . .
H4C H 0.7151 0.5288 0.6253 0.052 Uiso 1 1 calc R . .
C5C C 0.8223(9) 0.5491(7) 0.5083(7) 0.037(2) Uani 1 1 d . . .
C6C C 0.9321(9) 0.5432(8) 0.5723(8) 0.042(3) Uani 1 1 d . . .
O6C O 0.9376(7) 0.5343(6) 0.6632(5) 0.054(2) Uani 1 1 d . . .
C7C C 1.0432(9) 0.5508(8) 0.5155(8) 0.040(2) Uani 1 1 d . . .
O7C O 1.1340(6) 0.5464(5) 0.5659(5) 0.0440(18) Uani 1 1 d . . .
C8C C 1.0357(8) 0.5589(7) 0.4024(8) 0.034(2) Uani 1 1 d . . .
C9C C 1.1292(9) 0.5560(7) 0.3458(8) 0.036(2) Uani 1 1 d . . .
H9C H 1.2025 0.5536 0.3792 0.043 Uiso 1 1 calc R . .
C10C C 1.1168(9) 0.5565(8) 0.2388(8) 0.041(3) Uani 1 1 d . . .
H10C H 1.1806 0.5510 0.1984 0.050 Uiso 1 1 calc R . .
C11C C 1.0079(9) 0.5653(7) 0.1927(7) 0.036(2) Uani 1 1 d . . .
H11C H 0.9990 0.5671 0.1200 0.043 Uiso 1 1 calc R . .
N12C N 0.9158(7) 0.5711(5) 0.2467(6) 0.0283(18) Uani 1 1 d . . .
C13C C 0.9323(8) 0.5667(7) 0.3504(7) 0.035(2) Uani 1 1 d . . .
C14C C 0.8234(7) 0.5633(7) 0.4047(7) 0.030(2) Uani 1 1 d . . .
P1 P 0.4969(3) -0.0023(2) 0.1988(2) 0.0496(8) Uani 1 1 d . . .
F11 F 0.5383(14) -0.0553(12) 0.2942(13) 0.178(6) Uiso 1 1 d . . .
F12 F 0.5486(10) -0.0791(8) 0.1296(9) 0.117(4) Uiso 1 1 d . . .
F13 F 0.4425(9) 0.0743(8) 0.2647(8) 0.117(3) Uiso 1 1 d . . .
F14 F 0.3769(12) -0.0455(10) 0.1928(10) 0.143(4) Uiso 1 1 d . . .
F15 F 0.6097(9) 0.0517(8) 0.1934(8) 0.109(3) Uiso 1 1 d . . .
F16 F 0.4499(9) 0.0564(8) 0.1030(9) 0.113(3) Uiso 1 1 d . . .
P2 P 0.0286(3) 0.7980(2) 0.5697(2) 0.0531(9) Uani 1 1 d . . .
F21 F -0.0538(8) 0.8539(6) 0.6375(7) 0.089(3) Uiso 1 1 d . . .
F22 F 0.1314(7) 0.8623(6) 0.6071(6) 0.077(2) Uiso 1 1 d . . .
F23 F -0.0670(7) 0.7335(6) 0.5238(6) 0.074(2) Uiso 1 1 d . . .
F24 F 0.0037(7) 0.8676(6) 0.4757(6) 0.073(2) Uiso 1 1 d . . .
F25 F 0.1150(7) 0.7441(5) 0.5012(6) 0.069(2) Uiso 1 1 d . . .
F26 F 0.0601(7) 0.7311(6) 0.6606(6) 0.072(2) Uiso 1 1 d . . .
N1SA N 0.1347(10) 0.7739(8) 0.0915(10) 0.069(3) Uiso 1 1 d . . .
C2SA C 0.1557(11) 0.7959(9) 0.1720(10) 0.055(3) Uiso 1 1 d . . .

C3SA C 0.1851(14) 0.8187(12) 0.2812(12) 0.081(4) Uiso 1 1 d . . .
H3S1 H 0.1526 0.7723 0.3254 0.122 Uiso 1 1 calc R . .
H3S2 H 0.1546 0.8794 0.2970 0.122 Uiso 1 1 calc R . .
H3S3 H 0.2675 0.8194 0.2933 0.122 Uiso 1 1 calc R . .
N1SB N 0.3970(12) 0.4230(10) 0.3032(11) 0.091(4) Uiso 1 1 d . . .
C2SB C 0.3599(17) 0.3635(15) 0.3395(15) 0.100(6) Uiso 1 1 d . . .
C3SB C 0.330(4) 0.258(3) 0.358(3) 0.26(2) Uiso 1 1 d . . .
H3SA H 0.3018 0.2300 0.2937 0.391 Uiso 1 1 calc R . .
H3SB H 0.2717 0.2539 0.4085 0.391 Uiso 1 1 calc R . .
H3SC H 0.3979 0.2254 0.3846 0.391 Uiso 1 1 calc R . .
N1SC N 0.4001(16) 0.6827(14) 0.4144(15) 0.129(6) Uiso 1 1 d . . .
C2SC C 0.3729(16) 0.6928(13) 0.4998(16) 0.097(6) Uiso 1 1 d . . .
C3SC C 0.3514(15) 0.7100(12) 0.6027(13) 0.087(5) Uiso 1 1 d . . .
H3SD H 0.3078 0.6589 0.6293 0.131 Uiso 1 1 calc R . .
H3SE H 0.3083 0.7672 0.6073 0.131 Uiso 1 1 calc R . .
H3SF H 0.4233 0.7158 0.6432 0.131 Uiso 1 1 calc R . .

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_atom_site_aniso_U_23
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_atom_site_aniso_U_12
Fe1 0.0291(7) 0.0361(7) 0.0211(6) 0.0000(6) 0.0025(5) -0.0007(7)
N1A 0.030(4) 0.041(5) 0.022(4) -0.002(4) -0.005(3) -0.005(4)
C2A 0.038(6) 0.067(8) 0.024(5) -0.005(5) -0.001(4) -0.002(5)
C3A 0.039(5) 0.083(8) 0.019(5) -0.005(6) 0.001(4) 0.002(7)
C4A 0.039(7) 0.095(11) 0.030(6) 0.011(6) 0.012(5) 0.007(7)
C5A 0.030(6) 0.068(8) 0.033(6) 0.014(5) 0.003(4) -0.003(5)
C6A 0.037(7) 0.078(9) 0.056(8) 0.038(7) 0.006(6) 0.007(6)
O6A 0.070(7) 0.075(6) 0.077(7) 0.034(5) 0.040(5) 0.011(5)
C7A 0.041(7) 0.066(8) 0.045(7) 0.016(6) -0.015(5) -0.009(6)
O7A 0.065(6) 0.071(6) 0.070(6) 0.027(5) -0.023(5) -0.038(5)
C8A 0.037(6) 0.047(6) 0.047(7) 0.010(5) -0.008(5) -0.009(5)
C9A 0.039(7) 0.038(6) 0.062(8) -0.005(6) -0.014(6) -0.001(5)
C10A 0.042(7) 0.045(7) 0.052(7) -0.005(6) -0.014(5) 0.011(5)
C11A 0.042(6) 0.041(6) 0.027(5) -0.007(4) -0.006(5) -0.006(5)
N12A 0.026(4) 0.051(6) 0.031(5) -0.005(4) -0.002(3) -0.008(4)
C13A 0.028(5) 0.037(5) 0.028(5) 0.005(4) -0.001(4) 0.004(4)
C14A 0.032(6) 0.047(6) 0.023(5) 0.009(4) 0.003(4) 0.002(5)
N1B 0.033(4) 0.048(6) 0.024(4) 0.005(4) 0.005(3) -0.003(4)
C2B 0.035(6) 0.047(6) 0.026(5) -0.003(5) 0.014(4) 0.001(5)
C3B 0.036(6) 0.050(6) 0.033(6) 0.000(5) 0.002(5) 0.003(5)
C4B 0.030(5) 0.048(6) 0.024(5) 0.001(4) 0.008(4) -0.004(5)
C5B 0.033(6) 0.057(7) 0.012(5) -0.006(4) 0.002(4) -0.006(5)
C6B 0.027(6) 0.060(7) 0.035(6) 0.000(5) 0.007(4) -0.011(5)
O6B 0.032(4) 0.078(6) 0.055(5) -0.012(4) 0.004(4) -0.012(4)
C7B 0.046(7) 0.046(6) 0.028(5) 0.001(5) 0.002(5) -0.008(5)

O7B 0.053(5) 0.051(5) 0.061(5) -0.017(4) 0.002(4) -0.017(4)
C8B 0.043(6) 0.038(6) 0.026(5) -0.006(4) 0.002(4) -0.006(5)
C9B 0.046(7) 0.037(6) 0.035(6) -0.001(5) 0.001(5) -0.007(5)
C10B 0.045(7) 0.043(6) 0.049(6) -0.005(5) -0.001(5) 0.008(5)
C11B 0.033(6) 0.048(6) 0.028(5) -0.005(5) 0.004(4) -0.008(5)
N12B 0.029(4) 0.037(5) 0.023(4) 0.006(3) 0.002(3) -0.002(4)
C13B 0.027(5) 0.048(6) 0.023(5) 0.001(4) 0.006(4) -0.011(5)
C14B 0.037(6) 0.040(6) 0.021(5) -0.005(4) 0.003(4) -0.012(5)
N1C 0.038(5) 0.043(5) 0.021(4) -0.006(3) 0.001(3) 0.002(4)
C2C 0.027(5) 0.045(6) 0.031(5) 0.005(4) 0.006(4) 0.007(4)
C3C 0.024(5) 0.078(8) 0.036(6) 0.002(5) 0.010(4) 0.010(5)
C4C 0.047(7) 0.060(7) 0.024(5) 0.003(5) 0.015(5) -0.001(6)
C5C 0.045(6) 0.042(6) 0.025(5) -0.008(4) -0.003(4) 0.004(5)
C6C 0.046(7) 0.045(6) 0.034(6) -0.002(5) -0.002(5) 0.024(5)
O6C 0.068(6) 0.072(5) 0.021(4) 0.007(4) -0.003(4) 0.016(4)
C7C 0.045(6) 0.047(6) 0.025(5) -0.001(5) -0.001(5) 0.007(5)
O7C 0.055(5) 0.050(5) 0.027(4) -0.003(3) -0.006(3) 0.006(4)
C8C 0.023(5) 0.038(5) 0.042(6) -0.004(4) -0.001(4) 0.003(4)
C9C 0.041(6) 0.036(5) 0.032(5) 0.003(4) 0.000(4) 0.006(5)
C10C 0.029(6) 0.050(6) 0.047(7) -0.003(5) 0.018(5) -0.005(5)
C11C 0.040(6) 0.043(6) 0.025(5) 0.000(4) 0.002(4) -0.001(5)
N12C 0.033(4) 0.023(4) 0.030(4) -0.006(3) 0.010(3) -0.003(3)
C13C 0.038(6) 0.041(6) 0.025(5) -0.002(4) 0.002(4) -0.001(4)
C14C 0.022(5) 0.045(6) 0.024(5) -0.003(4) 0.002(4) 0.007(4)
P1 0.051(2) 0.0622(19) 0.0363(16) -0.0018(14) 0.0110(14) -0.0017(16)
P2 0.077(2) 0.0416(17) 0.0403(17) -0.0038(14) -0.0037(16) 0.0013(16)

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

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Fe1 N1B 1.951(8) . ?
Fe1 N12B 1.965(8) . ?
Fe1 N12A 1.966(8) . ?
Fe1 N1A 1.968(8) . ?
Fe1 N1C 1.979(8) . ?
Fe1 N12C 2.006(8) . ?
N1A C13A 1.365(12) . ?

N1A C11A 1.379(12) . ?
C2A N12A 1.348(12) . ?
C2A C3A 1.363(14) . ?
C3A C4A 1.39(2) . ?
C4A C5A 1.368(17) . ?
C5A C14A 1.421(14) . ?
C5A C6A 1.461(18) . ?
C6A O6A 1.227(13) . ?
C6A C7A 1.585(19) . ?
C7A O7A 1.207(14) . ?
C7A C8A 1.445(16) . ?
C8A C13A 1.388(14) . ?
C8A C9A 1.404(16) . ?
C9A C10A 1.409(17) . ?
C10A C11A 1.340(15) . ?
N12A C14A 1.381(13) . ?
C13A C14A 1.472(14) . ?
N1B C14B 1.348(13) . ?
N1B C2B 1.384(13) . ?
C2B C3B 1.355(14) . ?
C3B C4B 1.400(15) . ?
C4B C5B 1.353(15) . ?
C5B C6B 1.451(16) . ?
C5B C14B 1.466(13) . ?
C6B O6B 1.216(12) . ?
C6B C7B 1.569(17) . ?
C7B O7B 1.220(13) . ?
C7B C8B 1.481(15) . ?
C8B C13B 1.383(14) . ?
C8B C9B 1.406(15) . ?
C9B C10B 1.371(15) . ?
C10B C11B 1.428(15) . ?
C11B N12B 1.344(13) . ?
N12B C13B 1.391(12) . ?
C13B C14B 1.411(15) . ?
N1C C2C 1.316(12) . ?
N1C C14C 1.381(11) . ?
C2C C3C 1.394(14) . ?
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N1C C14C C13C 115.3(8) . . ?
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F14 P1 F13 84.9(7) . . ?
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F14 P1 F12 93.9(7) . . ?
F13 P1 F12 178.2(6) . . ?
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Appendix I
Crystal structure data of [Cu(1,10-phenanthroline-5,6-dione)$_3$](PF$_6$)$_2$

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 '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'
 'F'  'F'   0.0171   0.0103
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'P'  'P'   0.1023   0.0942
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'Cu'  'Cu'   0.3201   1.2651
 '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'

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_symmetry_space_group_name_H-M    P2(1)

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 'x, y, z'
 '-x, y+1/2, -z'
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_cell_angle_beta                 93.108(5)
_cell_angle_gamma                90.00
_cell_volume                    2301.5(9)
_cell_formula_units_Z            2
_cell_measurement_temperature    153(2)
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F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
not relevant to the choice of reflections for refinement.  R-factors based
on F^2^ are statistically about twice as large as those based on F, and R-
factors based on ALL data will be even larger.
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_atom_sites_solution_secondary   difmap
_atom_sites_solution_hydrogens   geom
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'Flack H D (1983), Acta Cryst. A39, 876-881'
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C2A C 1.0357(9) 0.1704(8) 1.1493(7) 0.061(2) Uani 1 1 d . . .
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C3A C 0.9258(10) 0.1641(6) 1.1167(7) 0.062(3) Uani 1 1 d . . .
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C4A C 0.8649(9) 0.2415(6) 1.0904(6) 0.057(2) Uani 1 1 d . . .
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C7A C 0.9216(8) 0.5052(6) 1.0806(6) 0.053(2) Uani 1 1 d . . .
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C8A C 1.0400(7) 0.5016(6) 1.1163(6) 0.0492(18) Uani 1 1 d . . .
C9A C 1.1045(9) 0.5822(7) 1.1259(7) 0.064(2) Uani 1 1 d . . .
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C7B C 1.4378(8) 0.3161(7) 1.5730(6) 0.060(2) Uani 1 1 d . . .
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N12B N 1.2341(6) 0.2913(4) 1.3532(5) 0.0505(17) Uani 1 1 d . . .
C13B C 1.3262(8) 0.2983(5) 1.4066(6) 0.0513(19) Uani 1 1 d . . .
C14B C 1.4320(8) 0.2976(5) 1.3530(6) 0.051(2) Uani 1 1 d . . .
N1C N 1.2953(7) 0.1222(5) 1.2086(5) 0.0607(18) Uani 1 1 d . . .
C2C C 1.2806(12) 0.0660(8) 1.2918(9) 0.086(4) Uani 1 1 d . . .
H2C H 1.2456 0.0895 1.3491 0.103 Uiso 1 1 calc R . .
C3C C 1.3171(16) -0.0248(10) 1.2908(13) 0.122(7) Uani 1 1 d . . .
H3C H 1.3099 -0.0612 1.3497 0.146 Uiso 1 1 calc R . .
C4C C 1.3581(15) -0.0598(9) 1.2175(13) 0.107(6) Uani 1 1 d . . .
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C5C C 1.3748(9) -0.0050(7) 1.1318(8) 0.075(3) Uani 1 1 d . . .
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O6C O 1.4724(12) -0.1166(9) 1.0385(10) 0.181(8) Uani 1 1 d . . .
O7C O 1.4918(7) -0.0048(9) 0.8797(10) 0.147(6) Uani 1 1 d . . .
C7C C 1.4433(8) 0.0205(11) 0.9524(12) 0.102(6) Uani 1 1 d . . .
C8C C 1.3920(8) 0.1219(10) 0.9483(8) 0.079(4) Uani 1 1 d . . .
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C11C C 1.3017(7) 0.2895(7) 0.9676(7) 0.060(2) Uani 1 1 d . . .
H11C H 1.2702 0.3492 0.9723 0.072 Uiso 1 1 calc R . .
N12C N 1.3051(6) 0.2324(6) 1.0488(5) 0.0530(17) Uani 1 1 d . . .
C13C C 1.3471(6) 0.1478(6) 1.0409(6) 0.0452(18) Uani 1 1 d . . .
C14C C 1.3386(7) 0.0868(6) 1.1281(6) 0.054(2) Uani 1 1 d . . .
P1 P 1.0062(2) 0.3736(2) 0.8084(2) 0.0722(7) Uani 1 1 d . . .
F11 F 0.8985(11) 0.3244(11) 0.790(2) 0.314(15) Uani 1 1 d . . .
F12 F 1.1192(11) 0.4176(11) 0.8328(19) 0.263(12) Uani 1 1 d . . .
F13 F 0.9488(11) 0.4401(6) 0.8822(8) 0.142(4) Uani 1 1 d . . .
F14 F 0.9736(15) 0.4408(16) 0.7302(10) 0.241(10) Uani 1 1 d . . .
F15 F 1.0644(12) 0.3027(12) 0.7426(8) 0.203(8) Uani 1 1 d . . .
F16 F 1.0420(9) 0.3040(6) 0.9004(6) 0.126(3) Uani 1 1 d . . .
P2 P 0.4812(4) 0.5629(2) 1.4317(3) 0.0890(10) Uani 1 1 d . . .
F21 F 0.5598(12) 0.5115(7) 1.3657(11) 0.182(6) Uani 1 1 d . . .
F22 F 0.5009(10) 0.4922(6) 1.5212(7) 0.125(3) Uani 1 1 d . . .
F23 F 0.3933(10) 0.6167(7) 1.4983(7) 0.132(4) Uani 1 1 d . . .
F24 F 0.4437(12) 0.6306(6) 1.3452(7) 0.146(4) Uani 1 1 d . . .
F25 F 0.3751(10) 0.4972(7) 1.3913(8) 0.143(4) Uani 1 1 d . . .
F26 F 0.5626(10) 0.6224(7) 1.4831(10) 0.139(4) Uani 1 1 d . . .

C1S C 0.842(2) 0.492(2) 1.491(2) 0.205(12) Uiso 1 1 d . . .
H1SA H 0.8913 0.5357 1.5271 0.308 Uiso 1 1 calc R . .
H1SB H 0.8579 0.4295 1.5155 0.308 Uiso 1 1 calc R . .
H1SC H 0.7637 0.5074 1.5024 0.308 Uiso 1 1 calc R . .
C2S C 0.8614(10) 0.4972(9) 1.3695(9) 0.083(3) Uiso 1 1 d . . .
N3S N 0.8919(12) 0.4471(10) 1.3000(10) 0.112(4) Uiso 1 1 d . . .
C4S C 0.1088(18) 0.6915(16) 1.5949(15) 0.138(6) Uiso 1 1 d . . .
H4SA H 0.0359 0.6693 1.6146 0.207 Uiso 1 1 calc R . .
H4SB H 0.1127 0.7584 1.6036 0.207 Uiso 1 1 calc R . .
H4SC H 0.1682 0.6623 1.6375 0.207 Uiso 1 1 calc R . .
C5S C 0.1214(16) 0.6712(14) 1.5018(15) 0.122(5) Uiso 1 1 d . . .
N6S N 0.1529(13) 0.6592(11) 1.3914(12) 0.126(5) Uiso 1 1 d . . .
C7S C 1.7015(16) 0.0400(14) 1.2834(14) 0.128(6) Uiso 1 1 d . . .
H7SA H 1.6492 -0.0029 1.3129 0.193 Uiso 1 1 calc R . .
H7SB H 1.7110 0.0942 1.3271 0.193 Uiso 1 1 calc R . .
H7SC H 1.7740 0.0097 1.2776 0.193 Uiso 1 1 calc R . .
C8S C 1.6562(11) 0.0691(9) 1.1802(10) 0.083(3) Uiso 1 1 d . . .
N9S N 1.6381(11) 0.0894(10) 1.1042(11) 0.111(4) Uiso 1 1 d . . .

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C2A 0.066(6) 0.065(6) 0.051(5) 0.000(4) 0.012(4) 0.008(5)
C3A 0.101(8) 0.040(4) 0.046(5) -0.007(4) 0.024(5) 0.004(5)
C4A 0.075(6) 0.063(6) 0.035(4) 0.002(3) 0.021(4) 0.006(4)
C5A 0.061(5) 0.044(4) 0.037(4) 0.010(3) 0.017(3) 0.008(4)
C6A 0.053(5) 0.062(5) 0.045(4) 0.004(4) 0.012(3) 0.004(4)
O6A 0.056(4) 0.073(4) 0.076(4) 0.021(3) 0.001(3) 0.003(3)
C7A 0.065(5) 0.050(5) 0.047(4) 0.005(3) 0.019(4) 0.011(4)
O7A 0.069(4) 0.067(4) 0.083(5) 0.016(4) 0.006(3) 0.017(3)
C8A 0.053(5) 0.054(5) 0.042(4) 0.010(3) 0.013(3) 0.009(4)
C9A 0.076(7) 0.065(6) 0.053(5) 0.014(4) 0.016(4) -0.004(5)
C10A 0.052(6) 0.095(8) 0.077(6) 0.009(6) 0.010(5) -0.007(5)
C11A 0.038(5) 0.105(9) 0.078(6) 0.029(6) 0.019(4) 0.007(5)
N12A 0.051(4) 0.083(5) 0.043(3) 0.013(3) 0.016(3) 0.023(4)
C13A 0.068(5) 0.045(4) 0.033(3) 0.008(3) 0.019(3) 0.006(4)
C14A 0.063(6) 0.078(6) 0.036(4) 0.005(4) 0.023(4) 0.034(5)
N1B 0.081(5) 0.031(3) 0.040(3) 0.004(2) 0.012(3) -0.003(3)
C2B 0.078(6) 0.041(4) 0.039(4) 0.001(3) 0.017(4) 0.000(4)
C3B 0.082(6) 0.048(4) 0.059(5) 0.005(4) 0.019(5) -0.015(4)
C4B 0.066(5) 0.075(6) 0.047(5) -0.004(4) 0.004(4) -0.023(5)
C5B 0.058(5) 0.051(4) 0.050(5) 0.008(3) -0.001(4) -0.011(4)
C6B 0.056(5) 0.084(6) 0.037(4) -0.006(4) -0.003(4) 0.000(4)

O6B 0.077(5) 0.096(5) 0.065(4) -0.001(4) -0.012(4) 0.010(4)
C7B 0.063(5) 0.082(6) 0.036(4) 0.008(4) -0.001(3) -0.010(5)
O7B 0.092(5) 0.118(6) 0.042(4) -0.014(4) 0.008(3) -0.031(5)
C8B 0.081(6) 0.068(5) 0.040(4) 0.003(4) 0.021(4) -0.008(5)
C9B 0.082(7) 0.126(10) 0.043(5) -0.005(5) 0.016(5) -0.030(7)
C10B 0.073(7) 0.101(9) 0.090(8) -0.002(6) 0.042(6) -0.006(6)
C11B 0.046(4) 0.083(7) 0.055(5) 0.003(4) 0.005(4) -0.005(4)
N12B 0.071(4) 0.037(4) 0.045(3) 0.010(3) 0.017(3) 0.000(3)
C13B 0.066(5) 0.045(4) 0.044(4) -0.001(3) 0.005(4) -0.011(3)
C14B 0.074(6) 0.037(4) 0.045(4) 0.004(3) 0.017(4) -0.010(3)
N1C 0.078(5) 0.060(5) 0.044(4) 0.000(3) 0.000(3) -0.001(4)
C2C 0.123(10) 0.067(7) 0.066(6) 0.016(5) -0.013(6) -0.030(7)
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C4C 0.145(13) 0.063(7) 0.103(10) -0.020(7) -0.071(10) 0.016(7)
C5C 0.088(7) 0.060(6) 0.071(6) -0.031(5) -0.050(6) 0.028(5)
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O7C 0.074(5) 0.181(11) 0.191(11) -0.143(10) 0.060(6) -0.027(6)
C7C 0.038(5) 0.130(11) 0.136(11) -0.106(11) -0.009(6) 0.012(6)
C8C 0.039(5) 0.137(10) 0.063(6) -0.055(7) 0.012(4) -0.013(5)
C9C 0.054(6) 0.210(17) 0.045(6) -0.050(8) 0.023(5) -0.071(9)
C10C 0.077(6) 0.142(11) 0.043(4) -0.002(8) 0.017(4) -0.045(9)
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N12C 0.049(4) 0.076(5) 0.035(3) 0.005(3) 0.007(3) 0.002(3)
C13C 0.034(4) 0.061(5) 0.040(4) -0.019(3) 0.004(3) 0.014(3)
C14C 0.049(5) 0.061(5) 0.048(4) -0.018(4) -0.020(3) 0.011(4)
P1 0.0761(17) 0.0852(18) 0.0573(14) -0.0124(13) 0.0229(12) -0.0009(14)
F11 0.115(8) 0.143(10) 0.67(4) -0.199(19) -0.091(16) 0.005(8)
F12 0.118(9) 0.169(12) 0.49(3) 0.168(17) -0.094(13) -0.036(8)
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F15 0.220(12) 0.29(2) 0.103(7) -0.025(8) 0.067(7) 0.132(14)
F16 0.170(8) 0.115(6) 0.100(6) 0.019(5) 0.062(6) 0.004(6)
P2 0.126(3) 0.0681(18) 0.0742(19) 0.0041(15) 0.0187(19) 0.0161(19)
F21 0.229(13) 0.083(6) 0.251(14) 0.000(7) 0.160(12) 0.034(7)
F22 0.187(9) 0.090(6) 0.093(6) 0.004(4) -0.024(6) 0.030(6)
F23 0.178(9) 0.126(7) 0.098(6) 0.009(5) 0.056(6) 0.041(7)
F24 0.268(14) 0.089(5) 0.087(5) 0.013(4) 0.053(7) 0.049(7)
F25 0.177(10) 0.108(7) 0.141(8) 0.015(6) -0.025(7) 0.003(7)
F26 0.147(8) 0.096(6) 0.174(10) 0.006(6) -0.004(7) 0.024(6)

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All esds (except the esd in the dihedral angle between two l.s. planes)
are estimated using the full covariance matrix. The cell esds are taken
into account individually in the estimation of esds in distances, angles
and torsion angles; correlations between esds in cell parameters are only
used when they are defined by crystal symmetry. An approximate (isotropic)
treatment of cell esds is used for estimating esds involving l.s. planes.
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Cu1 N12B 2.162(6) . ?
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C6A C7A 1.548(13) . ?
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C13B C14B 1.485(12) . ?
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P2 F23 1.612(9) . ?
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N1B Cu1 N1C 86.7(3) . . ?
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N1B Cu1 N12A 99.3(3) . . ?
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N1C Cu1 N12A 172.7(3) . . ?
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F22 P2 F25 87.0(6) . . ?
F23 P2 F25 86.8(7) . . ?
N3S C2S C1S 139.6(17) . . ?
C4S C5S N6S 170.1(19) . . ?
N9S C8S C7S 170.6(16) . . ?

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_refine_diff_density_rms 0.127

_

Appendix L
Crystal structure data of [Co(1,10-phenanthroline-5,6-dione)$_3$](PF$_6$)$_2$

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_chemical_formula_weight         1102.60

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 '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'
 'F'  'F'   0.0171   0.0103
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'P'  'P'   0.1023   0.0942
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'Co'  'Co'   0.3494   0.9721
 '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'

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_symmetry_space_group_name_H-M    P2(1)

loop_
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 'x, y, z'
 '-x, y+1/2, -z'
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_cell_length_c                  13.2646(9)
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_cell_angle_beta                92.6560(10)
_cell_angle_gamma               90.00
_cell_volume                    2300.2(3)
_cell_formula_units_Z           2
_cell_measurement_temperature   153(2)
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on F, with F set to zero for negative F^2^. The threshold expression of
F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
not relevant to the choice of reflections for refinement.  R-factors based
on F^2^ are statistically about twice as large as those based on F, and R-
factors based on ALL data will be even larger.
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'Flack H D (1983), Acta Cryst. A39, 876-881'
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C2A C 0.7806(6) 0.1539(4) 0.7909(4) 0.0571(14) Uani 1 1 d . . .
H2A H 0.7445 0.1793 0.8469 0.069 Uiso 1 1 calc R . .
C3A C 0.8142(7) 0.0653(5) 0.7959(5) 0.071(2) Uani 1 1 d . . .
H3A H 0.8037 0.0307 0.8555 0.085 Uiso 1 1 calc R . .
C4A C 0.8625(6) 0.0253(4) 0.7168(6) 0.071(2) Uani 1 1 d . . .
H4A H 0.8854 -0.0373 0.7195 0.086 Uiso 1 1 calc R . .
C5A C 0.8776(5) 0.0789(4) 0.6310(5) 0.0568(16) Uani 1 1 d . . .
C6A C 0.9334(5) 0.0405(5) 0.5433(6) 0.075(2) Uani 1 1 d . . .
O6A O 0.9737(6) -0.0340(5) 0.5400(5) 0.126(3) Uani 1 1 d . . .
C7A C 0.9428(4) 0.1021(6) 0.4509(6) 0.074(2) Uani 1 1 d . . .
O7A O 0.9899(5) 0.0745(5) 0.3767(6) 0.106(3) Uani 1 1 d . . .
C8A C 0.8937(4) 0.1965(5) 0.4509(4) 0.0550(15) Uani 1 1 d . . .
C9A C 0.8918(5) 0.2537(7) 0.3678(4) 0.068(2) Uani 1 1 d . . .
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C10A C 0.8445(5) 0.3394(6) 0.3745(4) 0.0625(19) Uani 1 1 d . . .
H10A H 0.8401 0.3784 0.3169 0.075 Uiso 1 1 calc R . .
C11A C 0.8024(4) 0.3698(4) 0.4656(3) 0.0450(12) Uani 1 1 d . . .
H11A H 0.7712 0.4298 0.4696 0.054 Uiso 1 1 calc R . .
N12A N 0.8058(3) 0.3153(3) 0.5469(3) 0.0349(7) Uani 1 1 d . . .
C13A C 0.8479(4) 0.2292(4) 0.5399(3) 0.0403(10) Uani 1 1 d . . .
C14A C 0.8410(4) 0.1704(3) 0.6294(4) 0.0423(11) Uani 1 1 d . . .
N1B N 0.9237(3) 0.3774(2) 0.7538(3) 0.0312(7) Uani 1 1 d . . .
C2B C 1.0169(4) 0.3800(3) 0.7015(3) 0.0354(9) Uani 1 1 d . . .
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C4B C 1.1343(4) 0.3890(4) 0.8524(4) 0.0412(10) Uani 1 1 d . . .
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C5B C 1.0384(4) 0.3866(3) 0.9073(3) 0.0360(8) Uani 1 1 d . . .
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C6B C 1.0480(4) 0.3938(3) 1.0193(3) 0.0409(10) Uani 1 1 d . . .
O6B O 1.1355(3) 0.3954(3) 1.0666(3) 0.0501(8) Uani 1 1 d . . .
C7B C 0.9366(4) 0.4025(4) 1.0748(3) 0.0435(10) Uani 1 1 d . . .
O7B O 0.9397(4) 0.4150(4) 1.1638(3) 0.0589(10) Uani 1 1 d . . .
C8B C 0.8290(4) 0.3964(4) 1.0129(3) 0.0415(10) Uani 1 1 d . . .
C9B C 0.7266(5) 0.4040(5) 1.0581(4) 0.0565(14) Uani 1 1 d . . .
H9B H 0.7243 0.4147 1.1286 0.068 Uiso 1 1 calc R . .
C10B C 0.6301(5) 0.3960(5) 1.0007(4) 0.0555(13) Uani 1 1 d . . .
H10B H 0.5596 0.3999 1.0308 0.067 Uiso 1 1 calc R . .
C11B C 0.6358(4) 0.3817(4) 0.8965(4) 0.0460(11) Uani 1 1 d . . .
H11B H 0.5682 0.3763 0.8563 0.055 Uiso 1 1 calc R . .
N12B N 0.7343(3) 0.3753(2) 0.8516(3) 0.0354(8) Uani 1 1 d . . .
C13B C 0.8275(4) 0.3832(3) 0.9092(3) 0.0338(8) Uani 1 1 d . . .
C14B C 0.9348(4) 0.3814(3) 0.8551(3) 0.0333(8) Uani 1 1 d . . .
N1C N 0.7015(3) 0.4879(3) 0.6624(3) 0.0343(7) Uani 1 1 d . . .
C2C C 0.7624(4) 0.5653(4) 0.6732(4) 0.0426(10) Uani 1 1 d . . .
H2C H 0.8397 0.5607 0.6931 0.051 Uiso 1 1 calc R . .
C3C C 0.7155(5) 0.6520(3) 0.6561(4) 0.0469(11) Uani 1 1 d . . .
H3C H 0.7601 0.7057 0.6656 0.056 Uiso 1 1 calc R . .
C4C C 0.6056(5) 0.6594(3) 0.6257(4) 0.0450(11) Uani 1 1 d . . .
H4C H 0.5730 0.7181 0.6131 0.054 Uiso 1 1 calc R . .
C5C C 0.5414(4) 0.5797(3) 0.6134(3) 0.0378(9) Uani 1 1 d . . .
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O6C O 0.3747(4) 0.6560(3) 0.5543(4) 0.0597(10) Uani 1 1 d . . .
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O7C O 0.2566(3) 0.4967(3) 0.5561(3) 0.0584(10) Uani 1 1 d . . .
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C9C C 0.3614(4) 0.3215(4) 0.5877(4) 0.0414(10) Uani 1 1 d . . .
H9C H 0.2848 0.3180 0.5656 0.050 Uiso 1 1 calc R . .
C10C C 0.4204(4) 0.2424(3) 0.6130(4) 0.0417(10) Uani 1 1 d . . .
H10C H 0.3845 0.1841 0.6089 0.050 Uiso 1 1 calc R . .
C11C C 0.5320(4) 0.2488(3) 0.6443(4) 0.0395(9) Uani 1 1 d . . .
H11C H 0.5720 0.1942 0.6619 0.047 Uiso 1 1 calc R . .
N12C N 0.5863(3) 0.3307(2) 0.6507(3) 0.0338(7) Uani 1 1 d . . .
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P1 P 0.49573(13) 0.95000(12) 0.69390(11) 0.0547(4) Uani 1 1 d . . .
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F12 F 0.4166(17) 0.9084(17) 0.7572(15) 0.115(6) Uiso 0.373(10) 1 d P A 2
F13 F 0.5790(14) 1.0278(10) 0.6417(11) 0.080(4) Uiso 0.373(10) 1 d P A 2
F14 F 0.6072(16) 0.9147(15) 0.7355(17) 0.119(6) Uiso 0.373(10) 1 d P A 2
F15 F 0.4808(16) 0.8778(12) 0.6130(13) 0.088(5) Uiso 0.373(10) 1 d P A 2
F16 F 0.5166(12) 1.0415(11) 0.7670(11) 0.088(4) Uiso 0.373(10) 1 d P A 2
F11' F 0.3751(7) 0.9920(6) 0.7009(7) 0.085(2) Uiso 0.627(10) 1 d P A 1
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F14' F 0.6089(6) 0.8928(6) 0.6770(7) 0.077(2) Uiso 0.627(10) 1 d P A 1
F15' F 0.4457(8) 0.8870(6) 0.5959(7) 0.074(2) Uiso 0.627(10) 1 d P A 1
F16' F 0.5456(10) 0.9903(9) 0.7913(9) 0.117(3) Uiso 0.627(10) 1 d P A 1
P2 P 0.97595(17) 0.64695(11) 0.92976(12) 0.0613(4) Uani 1 1 d . . .

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F22 F 0.8863(10) 0.5805(8) 0.8807(9) 0.049(3) Uiso 0.373(10) 1 d P A 2
F23 F 1.0697(10) 0.7156(8) 0.9609(10) 0.056(3) Uiso 0.373(10) 1 d P A 2
F24 F 1.0355(17) 0.5792(12) 1.0063(14) 0.094(5) Uiso 0.373(10) 1 d P A 2
F25 F 0.9207(9) 0.7133(6) 0.8381(6) 0.041(2) Uiso 0.373(10) 1 d P A 2
F26 F 0.917(2) 0.7107(19) 1.0078(19) 0.132(9) Uiso 0.373(10) 1 d P A 2
F21' F 1.0741(8) 0.5971(6) 0.8725(7) 0.077(2) Uiso 0.627(10) 1 d P A 1
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F23' F 1.0654(10) 0.7080(9) 0.9912(10) 0.102(4) Uiso 0.627(10) 1 d P A 1
F24' F 0.9938(6) 0.5748(4) 1.0218(5) 0.0512(14) Uiso 0.627(10) 1 d P A 1
F25' F 0.9608(9) 0.7183(6) 0.8442(7) 0.079(2) Uiso 0.627(10) 1 d P A 1
F26' F 0.8836(5) 0.6974(4) 0.9977(4) 0.0465(14) Uiso 0.627(10) 1 d P A 1
N1S N 0.1360(6) 0.1705(6) 0.5952(6) 0.085(2) Uani 1 1 d D . .
C2S C 0.1565(6) 0.1513(5) 0.6795(5) 0.0640(16) Uani 1 1 d D . .
C3S C 0.1858(9) 0.1280(6) 0.7835(6) 0.088(2) Uani 1 1 d D . .
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H3S3 H 0.2659 0.1393 0.7975 0.131 Uiso 1 1 calc R . .
N3S N 0.3980(9) 0.2686(10) 0.9133(11) 0.136(4) Uani 1 1 d . . .
C5S C 0.3775(7) 0.2536(8) 0.9975(9) 0.091(3) Uani 1 1 d . . .
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H6S1 H 0.4136 0.2563 1.1493 0.133 Uiso 1 1 calc R . .
H6S2 H 0.3268 0.1769 1.1160 0.133 Uiso 1 1 calc R . .
H6S3 H 0.2860 0.2818 1.1199 0.133 Uiso 1 1 calc R . .
N7S N 0.4130(15) 0.5260(10) 0.8104(12) 0.071(4) Uiso 0.481(19) 1 d PD B 2
C8S C 0.3558(14) 0.5897(11) 0.8303(16) 0.072(4) Uiso 0.481(19) 1 d PD B 2
C9S C 0.334(3) 0.6855(16) 0.851(3) 0.158(13) Uiso 0.481(19) 1 d PD B 2
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H9S2 H 0.3604 0.7001 0.9200 0.236 Uiso 0.481(19) 1 calc PR B 2
H9S3 H 0.2537 0.6975 0.8427 0.236 Uiso 0.481(19) 1 calc PR B 2
N7S' N 0.3753(15) 0.5244(10) 0.7950(10) 0.073(4) Uiso 0.519(19) 1 d PD B 1
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C9S' C 0.340(2) 0.5774(19) 0.9796(13) 0.116(8) Uiso 0.519(19) 1 d PD B 1
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C2A 0.083(4) 0.045(3) 0.042(3) 0.011(2) -0.011(3) 0.000(3)
C3A 0.100(5) 0.045(3) 0.064(4) 0.014(3) -0.033(4) -0.002(3)
C4A 0.081(4) 0.034(2) 0.094(5) -0.002(3) -0.054(4) 0.010(3)

C5A 0.049(3) 0.053(3) 0.064(3) -0.026(3) -0.036(3) 0.021(2)
C6A 0.051(3) 0.080(4) 0.088(5) -0.055(4) -0.041(3) 0.033(3)
O6A 0.131(5) 0.120(5) 0.120(5) -0.072(4) -0.070(4) 0.098(5)
C7A 0.028(2) 0.102(5) 0.092(5) -0.066(5) 0.004(3) -0.002(3)
O7A 0.066(3) 0.125(5) 0.132(5) -0.092(5) 0.050(3) -0.026(3)
C8A 0.028(2) 0.091(4) 0.046(3) -0.034(3) 0.0025(19) -0.007(2)
C9A 0.043(3) 0.127(6) 0.037(3) -0.032(3) 0.015(2) -0.031(3)
C10A 0.052(3) 0.113(6) 0.0229(19) 0.002(3) 0.0065(18) -0.032(4)
C11A 0.037(2) 0.073(4) 0.0250(19) 0.0077(19) 0.0029(15) -0.010(2)
N12A 0.0309(16) 0.051(2) 0.0226(16) 0.0000(14) 0.0027(13) 0.0065(15)
C13A 0.0253(18) 0.060(3) 0.035(2) -0.0141(19) -0.0046(16) 0.0076(18)
C14A 0.039(2) 0.043(2) 0.043(2) -0.0149(19) -0.0155(19) 0.0111(19)
N1B 0.0410(17) 0.0257(15) 0.0271(15) 0.0017(11) 0.0030(13) -0.0017(12)
C2B 0.050(2) 0.0319(19) 0.0253(18) 0.0004(14) 0.0067(17) -0.0023(17)
C3B 0.049(2) 0.046(2) 0.040(2) 0.0025(19) 0.014(2) -0.005(2)
C4B 0.036(2) 0.045(2) 0.042(2) -0.0035(19) -0.0027(18) -0.0064(19)
C5B 0.041(2) 0.0346(19) 0.033(2) 0.0015(16) 0.0015(17) -0.0033(17)
C6B 0.051(3) 0.042(2) 0.029(2) -0.0052(17) -0.0030(18) 0.002(2)
O6B 0.0514(19) 0.055(2) 0.0429(18) -0.0011(16) -0.0100(16) 0.0004(17)
C7B 0.048(2) 0.052(3) 0.030(2) -0.0011(19) 0.0056(18) -0.006(2)
O7B 0.065(2) 0.079(3) 0.0322(18) -0.0038(17) -0.0001(16) -0.012(2)
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C5S 0.063(4) 0.104(7) 0.107(7) 0.009(6) 0.006(5) -0.024(5)
C6S 0.074(5) 0.097(6) 0.092(6) 0.003(5) -0.018(4) -0.014(5)

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All esds (except the esd in the dihedral angle between two l.s. planes)
are estimated using the full covariance matrix. The cell esds are taken
into account individually in the estimation of esds in distances, angles
and torsion angles; correlations between esds in cell parameters are only
used when they are defined by crystal symmetry. An approximate (isotropic)
treatment of cell esds is used for estimating esds involving l.s. planes.
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Appendix M
Crystal structure data of 3,9-$Me_2phenHClO_4$

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 'C14 H13 Cl N2 O4'
_chemical_formula_weight         308.71

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'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'H'  'H'   0.0000   0.0000
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'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'
'Cl'  'Cl'   0.1484   0.1585
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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_symmetry_space_group_name_H-M    P2(1)/n

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

_cell_length_a                   6.726(4)
_cell_length_b                   19.272(14)
_cell_length_c                   10.360(7)
```

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_cell_angle_alpha                 90.00
_cell_angle_beta                  96.836(14)
_cell_angle_gamma                 90.00
_cell_volume                      1333.3(16)
_cell_formula_units_Z             4
_cell_measurement_temperature     173(2)
_cell_measurement_reflns_used     ?
_cell_measurement_theta_min       ?
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_exptl_special_details
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 ?
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_reflns_number_gt                1788
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_computing_cell_refinement       ?
_computing_data_reduction        ?
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_computing_publication_material  ?

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

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_refine_ls_weighting_scheme      calc
_refine_ls_weighting_details
'calc w=1/[\s^2^(Fo^2^)+(0.1000P)^2^+0.0000P] where P=(Fo^2^+2Fc^2^)/3'
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_atom_sites_solution_secondary   difmap
_atom_sites_solution_hydrogens   geom
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_refine_ls_extinction_method     none
_refine_ls_extinction_coef       ?
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_refine_ls_wR_factor_gt          0.1638
_refine_ls_goodness_of_fit_ref   1.187
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_refine_ls_shift/su_mean         0.000

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 _atom_site_refinement_flags
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Cl1 Cl 0.74649(16) 0.16361(5) 0.87758(10) 0.0285(4) Uani 1 1 d . . .
O1 O 0.6756(5) 0.10251(15) 0.9373(3) 0.0412(9) Uani 1 1 d . . .
O2 O 0.9065(5) 0.14598(17) 0.8035(3) 0.0425(9) Uani 1 1 d . . .
O3 O 0.5866(5) 0.19489(18) 0.7924(3) 0.0457(9) Uani 1 1 d . . .
O4 O 0.8171(5) 0.21300(16) 0.9764(3) 0.0440(9) Uani 1 1 d . . .
N1 N 0.7828(5) 0.00142(18) 0.6397(3) 0.0259(9) Uani 1 1 d . . .
N2 N 0.7707(5) 0.12616(17) 0.5147(4) 0.0208(8) Uani 1 1 d . . .
C1 C 0.7672(6) 0.1903(2) 0.4641(4) 0.0251(10) Uani 1 1 d . . .
C2 C 0.7323(6) 0.1963(2) 0.3281(4) 0.0307(10) Uani 1 1 d . . .
H2 H 0.7268 0.2409 0.2887 0.037 Uiso 1 1 calc R . .
C3 C 0.7061(6) 0.1379(2) 0.2518(4) 0.0286(10) Uani 1 1 d . . .
H3 H 0.6826 0.1426 0.1600 0.034 Uiso 1 1 calc R . .
C4 C 0.7134(6) 0.0721(2) 0.3070(4) 0.0245(10) Uani 1 1 d . . .
C5 C 0.7456(6) 0.0672(2) 0.4435(4) 0.0222(9) Uani 1 1 d . . .
C6 C 0.7533(5) 0.0016(2) 0.5079(4) 0.0225(9) Uani 1 1 d . . .
C7 C 0.7309(6) -0.0586(2) 0.4298(5) 0.0287(10) Uani 1 1 d . . .
C8 C 0.7397(6) -0.1229(2) 0.4977(5) 0.0308(11) Uani 1 1 d . . .
H8 H 0.7256 -0.1654 0.4510 0.037 Uiso 1 1 calc R . .
C9 C 0.7685(6) -0.1231(2) 0.6295(5) 0.0327(11) Uani 1 1 d . . .
H9 H 0.7743 -0.1660 0.6752 0.039 Uiso 1 1 calc R . .
C10 C 0.7901(6) -0.0596(2) 0.7000(4) 0.0287(10) Uani 1 1 d . . .
C11 C 0.7020(6) -0.0528(2) 0.2917(5) 0.0316(11) Uani 1 1 d . . .
H11 H 0.6886 -0.0937 0.2403 0.038 Uiso 1 1 calc R . .
C12 C 0.6933(6) 0.0094(2) 0.2326(4) 0.0297(11) Uani 1 1 d . . .
H12 H 0.6737 0.0118 0.1404 0.036 Uiso 1 1 calc R . .
C13 C 0.8228(7) -0.0590(3) 0.8454(4) 0.0383(12) Uani 1 1 d . . .
H13A H 0.8474 -0.0113 0.8761 0.057 Uiso 1 1 calc R . .
H13B H 0.9388 -0.0880 0.8756 0.057 Uiso 1 1 calc R . .
H13C H 0.7037 -0.0773 0.8797 0.057 Uiso 1 1 calc R . .
C14 C 0.8023(7) 0.2511(2) 0.5492(4) 0.0325(11) Uani 1 1 d . . .
H14A H 0.6842 0.2594 0.5941 0.049 Uiso 1 1 calc R . .
H14B H 0.8272 0.2918 0.4969 0.049 Uiso 1 1 calc R . .
H14C H 0.9189 0.2426 0.6135 0.049 Uiso 1 1 calc R . .
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O1 0.067(2) 0.0276(17) 0.0289(19) 0.0029(15) 0.0070(17) -0.0094(16)
O2 0.046(2) 0.043(2) 0.041(2) -0.0055(16) 0.0140(16) 0.0091(16)
O3 0.0375(18) 0.053(2) 0.045(2) 0.0171(18) -0.0009(16) 0.0013(16)
O4 0.065(2) 0.0301(18) 0.038(2) -0.0150(15) 0.0086(17) -0.0092(16)
N1 0.0207(17) 0.031(2) 0.027(2) 0.0039(16) 0.0048(15) 0.0019(14)
N2 0.0254(18) 0.0207(19) 0.017(2) 0.0040(15) 0.0049(15) 0.0008(14)
C1 0.024(2) 0.029(2) 0.024(3) 0.0017(19) 0.0079(18) -0.0010(17)
C2 0.031(2) 0.034(3) 0.027(3) 0.005(2) 0.0016(19) 0.0009(19)
C3 0.028(2) 0.039(3) 0.018(2) 0.0035(19) 0.0028(19) 0.0024(19)
C4 0.022(2) 0.032(2) 0.020(3) -0.0020(18) 0.0025(18) 0.0021(17)
C5 0.022(2) 0.023(2) 0.022(3) -0.0033(17) 0.0037(18) 0.0007(16)
C6 0.0128(18) 0.031(2) 0.024(3) -0.0025(18) 0.0015(16) -0.0002(16)
C7 0.015(2) 0.037(3) 0.035(3) -0.005(2) 0.0054(18) -0.0006(17)
C8 0.020(2) 0.032(3) 0.041(3) -0.006(2) 0.0084(19) -0.0024(18)
C9 0.020(2) 0.032(2) 0.048(3) 0.006(2) 0.008(2) 0.0039(18)
C10 0.020(2) 0.037(3) 0.029(3) 0.003(2) 0.0060(19) 0.0042(18)
C11 0.022(2) 0.042(3) 0.030(3) -0.011(2) 0.0020(19) 0.0006(19)
C12 0.025(2) 0.041(3) 0.023(3) -0.005(2) 0.0022(19) -0.0015(19)
C13 0.040(3) 0.041(3) 0.034(3) 0.013(2) 0.007(2) 0.007(2)
C14 0.039(2) 0.027(2) 0.032(3) 0.000(2) 0.006(2) -0.001(2)

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

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 _geom_bond_atom_site_label_2
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Cl1 O2 1.435(3) . ?
Cl1 O4 1.437(3) . ?
Cl1 O1 1.437(3) . ?
Cl1 O3 1.439(3) . ?
N1 C10 1.330(5) . ?
N1 C6 1.356(5) . ?
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N2 C1 1.341(5) . ?
N2 C5 1.353(5) . ?
C1 C2 1.405(6) . ?
C1 C14 1.469(6) . ?
C2 C3 1.374(6) . ?
C3 C4 1.391(6) . ?
C4 C5 1.409(6) . ?
C4 C12 1.429(6) . ?
C5 C6 1.429(6) . ?
C6 C7 1.411(6) . ?
C7 C8 1.423(6) . ?
C7 C11 1.426(7) . ?
C8 C9 1.357(6) . ?
C9 C10 1.424(6) . ?
C10 C13 1.496(7) . ?
C11 C12 1.343(6) . ?

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O2 Cl1 O4 109.4(2) . . ?
O2 Cl1 O1 110.1(2) . . ?
O4 Cl1 O1 109.5(2) . . ?
O2 Cl1 O3 108.9(2) . . ?
O4 Cl1 O3 108.6(2) . . ?
O1 Cl1 O3 110.2(2) . . ?
C10 N1 C6 117.9(4) . . ?
C1 N2 C5 124.4(4) . . ?
N2 C1 C2 117.5(4) . . ?
N2 C1 C14 120.4(4) . . ?
C2 C1 C14 122.1(4) . . ?
C3 C2 C1 120.2(4) . . ?
C2 C3 C4 121.1(4) . . ?
C3 C4 C5 117.8(4) . . ?
C3 C4 C12 123.6(4) . . ?
C5 C4 C12 118.6(4) . . ?
N2 C5 C4 119.1(4) . . ?
N2 C5 C6 119.6(4) . . ?
C4 C5 C6 121.3(4) . . ?
N1 C6 C7 124.6(4) . . ?
N1 C6 C5 117.7(4) . . ?
C7 C6 C5 117.7(4) . . ?
C6 C7 C8 115.9(4) . . ?
C6 C7 C11 120.2(4) . . ?
C8 C7 C11 123.9(4) . . ?
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C9 C8 C7 119.5(4) . . ?
C8 C9 C10 120.5(4) . . ?
N1 C10 C9 121.6(4) . . ?
N1 C10 C13 117.3(4) . . ?
C9 C10 C13 121.1(4) . . ?
C12 C11 C7 121.4(4) . . ?
C11 C12 C4 120.8(4) . . ?

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_diffrn_measured_fraction_theta_full 0.807
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_refine_diff_density_rms 0.125

Appendix N
Crystal structure data of $[Cr(TPA)_2Cl_2]Cl$