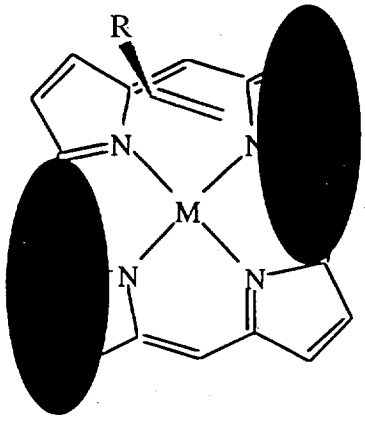
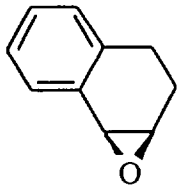
Side-on approach

Re face



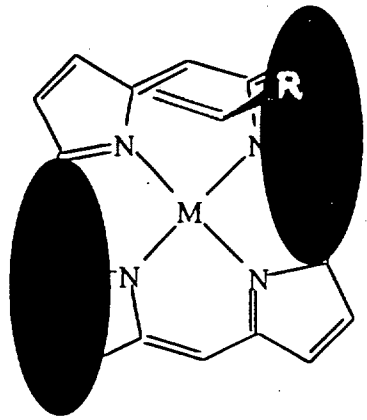


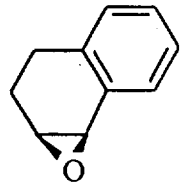
(1*S*, 2*R*) epoxide

(favored)

Side-on approach

Si face





(1*R*, 2*S*) epoxide

(disfavored)

Scheme

Table 1. NMR chemical shifts (δ) of the central protons of the chiroporphyrin free bases **1a-l** in $CDCl_3$, and enantiomeric excesses (*ee*) for the epoxidation [a] of 1,2-dihydronaphthalene with the corresponding manganese chiroporphyrin catalysts **2a-l**.

Free base/catalyst	δ	*ee* [b] (%) ; absolute configuration [c]
1a/2a	-1.66	60 ; (1*S*,2*R*)
1b/2b	-1.60	70 ; (1*S*,2*R*)
1c/2c	-1.51	65 ; (1*S*,2*R*)
1d/2d	-1.36	73 ; (1*S*,2*R*)
1e/2e	-1.41	68 ; (1*S*,2*R*)
1f/2f	-1.50	75 ; (1*S*,2*R*)
1g/2g	-1.27	75 ; (1*S*,2*R*)
1h/2h	-1-58	75 ; (1*S*,2*R*)
1i/2i	-1.29	80 ; (1*S*,2*R*)
1j/2j	-1.21	86 ; (1*S*,2*R*)
1k/2k	-1.39	83 ; (1*S*,2*R*)
1l/2l	-1.15	79 ; (1*S*,2*R*)

[a] Reactions were generally run for 1 h with 1.0 μmol of catalyst, 100 μmol of iodosylbenzene, 250 μmol of pyridine, 20 μL of *n*-tridecane as internal standard, and 1.00 mmol of olefin in 2 mL of CH_2Cl_2. Epoxide yield, determined by GC relative to *n*-tridecane and based on PhIO consumption, was 85-95 %; β-tetralone (2-7 %) was obtained as side-products. [b] Determined by GC using a chiral capillary column (Cyclodex-B, 30 m x 0.25 mm). [c] The absolute configuration was assigned on the basis of the sign of the optical rotation, see ref. 7 and references therein.

Table 1. Crystal data and structure refinement for 3g.

Identification code	Ni(TBCP)•2C_8H_{18}
Empirical formula	C100 H144 N4 Ni O8
Formula weight	1588.90
Temperature	193(2) K
Wavelength	0.71073 A
Crystal system	Orthorhombic
Space group	C222(1)
Unit cell dimensions	a = 11.9940(2) A alpha = 90 deg. b = 35.0895(8) A beta = 90 deg. c = 22.2423(4) A gamma = 90 deg.
Volume, Z	9361.0(3) A^3, 4
Density (calculated)	1.127 Mg/m^3
Absorption coefficient	0.263 mm^-1
F(000)	3456
Crystal size	0.2 x 0.2 x 0.2 mm
Theta range for data collection	1.16 to 25.95 deg.
Limiting indices	-14<=h<=14, -40<=k<=42, -12<=l<=27
Reflections collected	21894
Independent reflections	8296 [R(int) = 0.0739]
Absorption correction	SADABS
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8296 / 0 / 511
Goodness-of-fit on F^2	1.130
Final R indices [a] [I>2sigma(I)]	R1 = 0.0790, wR2 = 0.1840
R indices (all data)	R1 = 0.1215, wR2 = 0.2045
Absolute structure parameter	0.00(2)
Extinction coefficient	0.00259(7)
Largest diff. peak and hole	2.132 and -0.501 e.A^-3

[a] $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ and $wR_2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[wF_o^4]\}^{1/2}$ The conventional R-factors R_1 are based on F, with F set to zero for negative F^2. The criterion of $F^2 < 2\sigma(F^2)$ was used only for calculating R_1. R-factors based on F^2 (wR_2) are statistically about twice as large as those based on F, and R-factors based on all data will be even larger.

Table 2. Atomic coordinates and equivalent isotropic displacement parameters (A^2) for 3g. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Ni	0.5000	0.311260(7)	0.7500	0.02821(6)
O(1)	0.56938(9)	0.21293(3)	1.00916(5)	0.0521(4)
O(2)	0.54316(8)	0.20990(3)	0.90963(5)	0.0486(4)
O(3)	0.65615(11)	0.41040(4)	0.58106(6)	0.0678(4)
O(4)	0.72799(10)	0.40136(4)	0.48871(6)	0.0610(4)
N(1)	0.65670(8)	0.31029(3)	0.75777(5)	0.0286(3)
N(2)	0.51351(9)	0.31140(3)	0.66532(4)	0.0273(3)
C(1)	0.66985(12)	0.28665(4)	0.86091(6)	0.0318(4)
C(2)	0.71580(11)	0.29584(4)	0.80541(7)	0.0325(4)
C(3)	0.83306(13)	0.29687(4)	0.79141(7)	0.0410(5)
C(4)	0.84505(11)	0.31336(4)	0.73782(6)	0.0352(4)
C(5)	0.73481(11)	0.32038(4)	0.71469(7)	0.0327(4)
C(6)	0.71083(12)	0.33058(4)	0.65549(7)	0.0331(4)
C(7)	0.60537(12)	0.32298(4)	0.63154(7)	0.0329(4)
C(8)	0.58194(12)	0.31821(4)	0.56808(6)	0.0366(4)
C(9)	0.48095(13)	0.30212(4)	0.56397(6)	0.0403(5)
C(10)	0.43651(12)	0.29841(4)	0.62428(6)	0.0325(4)
C(11)	0.74651(12)	0.26848(5)	0.90557(7)	0.0380(5)
C(12)	0.79194(12)	0.22864(4)	0.89490(7)	0.0382(5)
C(13)	0.75336(13)	0.20682(5)	0.84115(8)	0.0505(6)
C(14)	0.91398(13)	0.22193(6)	0.91057(8)	0.0578(6)
C(15)	0.71448(13)	0.23555(5)	0.94825(7)	0.0421(5)
C(16)	0.60204(13)	0.21870(4)	0.95862(7)	0.0410(5)
C(17)	0.43398(12)	0.19269(5)	0.92072(7)	0.0449(5)
C(18)	0.4430(2)	0.15068(5)	0.93867(10)	0.0761(7)
C(19)	0.3721(2)	0.13082(6)	0.88972(11)	0.0993(9)
C(20)	0.4400(2)	0.13504(9)	0.82922(13)	0.1457(12)
C(21)	0.4290(2)	0.17533(8)	0.81489(10)	0.1138(11)
C(22)	0.36404(14)	0.19132(5)	0.86408(7)	0.0515(5)
C(23)	0.3114(2)	0.23046(6)	0.84974(13)	0.1422(11)
C(24)	0.27923(14)	0.16033(5)	0.87911(8)	0.0522(6)
C(25)	0.2049(2)	0.16898(9)	0.93278(11)	0.1240(12)
C(26)	0.2011(2)	0.14957(6)	0.82729(9)	0.0722(7)
C(31)	0.80910(12)	0.33908(4)	0.61590(7)	0.0375(5)
C(32)	0.88022(13)	0.37404(5)	0.62317(7)	0.0454(5)
C(33)	0.8453(2)	0.40422(5)	0.66821(9)	0.0641(7)
C(34)	1.0080(2)	0.36955(6)	0.61781(8)	0.0661(6)
C(35)	0.81379(13)	0.36909(5)	0.56601(7)	0.0430(5)
C(36)	0.72314(14)	0.39551(5)	0.54915(8)	0.0525(6)
C(37)	0.6537(2)	0.42952(5)	0.46312(9)	0.0621(6)
C(38)	0.5398(2)	0.41335(6)	0.44337(14)	0.1032(10)
C(39)	0.5351(2)	0.42428(6)	0.37702(12)	0.0993(9)
C(40)	0.6087(2)	0.39719(7)	0.34414(12)	0.1180(11)
C(41)	0.7264(2)	0.41120(8)	0.36383(11)	0.0969(10)
C(42)	0.7029(2)	0.44470(6)	0.40552(9)	0.0698(7)
C(43)	0.7981(2)	0.47207(8)	0.41497(13)	0.1214(11)
C(44)	0.5946(2)	0.46209(6)	0.37584(10)	0.0836(8)
C(45)	0.6182(3)	0.47828(9)	0.31174(11)	0.1454(13)
C(46)	0.5380(2)	0.49362(6)	0.41094(12)	0.1138(10)
C(51)	0.1957(3)	0.10070(11)	1.0782(2)	0.179(2)
C(52)	0.1877(3)	0.09534(10)	1.1469(2)	0.203(2)
C(53)	0.0786(2)	0.08957(7)	1.1703(2)	0.183(2)
C(54)	0.0547(2)	0.08668(8)	1.2453(2)	0.259(2)
C(63)	0.3411(6)	-0.0174(2)	1.1649(3)	0.352(4)
C(64)	0.4043(6)	0.0061(2)	1.1987(4)	0.517(9)
C(61)	0.3132(6)	-0.0071(3)	1.0403(3)	0.448(6)
C(62)	0.3773(5)	-0.0035(2)	1.0940(3)	0.371(5)

Table 3. Bond lengths [A] and angles [deg] for 3g.

Ni-N(1)	1.8878(10)
Ni-N(1)#1	1.8878(10)
Ni-N(2)#1	1.8904(9)
Ni-N(2)	1.8904(10)
O(1)-C(16)	1.208(2)
O(2)-C(16)	1.335(2)
O(2)-C(17)	1.463(2)
O(3)-C(36)	1.193(2)
O(4)-C(36)	1.361(2)
O(4)-C(37)	1.447(2)
N(1)-C(2)	1.372(2)
N(1)-C(5)	1.386(2)
N(2)-C(10)	1.376(2)
N(2)-C(7)	1.394(2)
C(1)-C(10)#1	1.381(2)
C(1)-C(2)	1.390(2)
C(1)-C(11)	1.496(2)
C(2)-C(3)	1.441(2)
C(3)-C(4)	1.333(2)
C(4)-C(5)	1.440(2)
C(5)-C(6)	1.395(2)
C(6)-C(7)	1.398(2)
C(6)-C(31)	1.501(2)
C(7)-C(8)	1.449(2)
C(8)-C(9)	1.340(2)
C(9)-C(10)	1.449(2)
C(10)-C(1)#1	1.381(2)
C(11)-C(12)	1.519(2)
C(11)-C(15)	1.544(2)
C(12)-C(13)	1.493(2)
C(12)-C(14)	1.523(2)
C(12)-C(15)	1.526(2)
C(15)-C(16)	1.490(2)
C(17)-C(22)	1.514(2)
C(17)-C(18)	1.531(3)
C(18)-C(19)	1.548(3)
C(19)-C(24)	1.539(3)
C(19)-C(20)	1.580(4)
C(20)-C(21)	1.455(4)
C(21)-C(22)	1.456(3)
C(22)-C(24)	1.526(2)
C(22)-C(23)	1.545(3)
C(24)-C(25)	1.520(3)
C(24)-C(26)	1.533(3)
C(31)-C(32)	1.503(2)
C(31)-C(35)	1.531(2)
C(32)-C(35)	1.511(2)
C(32)-C(33)	1.517(2)
C(32)-C(34)	1.545(2)
C(35)-C(36)	1.477(2)
C(37)-C(42)	1.508(3)
C(37)-C(38)	1.543(3)
C(38)-C(39)	1.526(4)
C(39)-C(40)	1.489(3)
C(39)-C(44)	1.507(3)
C(40)-C(41)	1.558(4)
C(41)-C(42)	1.523(3)
C(42)-C(43)	1.507(3)
C(42)-C(44)	1.581(3)
C(44)-C(46)	1.515(3)
C(44)-C(45)	1.561(3)
C(51)-C(52)	1.542(6)
C(52)-C(53)	1.423(5)
C(53)-C(54)	1.695(6)
C(54)-C(54)#2	1.329(6)
C(63)-C(64)	1.349(10)
C(63)-C(62)	1.705(9)
C(61)-C(62)	1.426(9)
C(61)-C(61)#3	1.861(14)
N(1)-Ni-N(1)#1	177.93(7)
N(1)-Ni-N(2)#1	89.67(4)
N(1)#1-Ni-N(2)#1	90.34(4)

N(1)-Ni-N(2)	90.34(4)
N(1)#1-Ni-N(2)	89.67(4)
N(2)#1-Ni-N(2)	179.71(6)
C(16)-O(2)-C(17)	115.56(12)
C(36)-O(4)-C(37)	117.70(14)
C(2)-N(1)-C(5)	106.21(11)
C(2)-N(1)-Ni	126.28(9)
C(5)-N(1)-Ni	127.23(9)
C(10)-N(2)-C(7)	105.63(10)
C(10)-N(2)-Ni	127.05(9)
C(7)-N(2)-Ni	127.26(9)
C(10)#1-C(1)-C(2)	120.60(13)
C(10)#1-C(1)-C(11)	122.46(13)
C(2)-C(1)-C(11)	116.41(13)
N(1)-C(2)-C(1)	124.53(12)
N(1)-C(2)-C(3)	109.15(12)
C(1)-C(2)-C(3)	125.80(13)
C(4)-C(3)-C(2)	108.04(13)
C(3)-C(4)-C(5)	107.13(12)
N(1)-C(5)-C(6)	125.38(12)
N(1)-C(5)-C(4)	109.27(12)
C(6)-C(5)-C(4)	124.79(13)
C(5)-C(6)-C(7)	119.83(13)
C(5)-C(6)-C(31)	116.29(13)
C(7)-C(6)-C(31)	121.65(13)
N(2)-C(7)-C(6)	124.42(13)
N(2)-C(7)-C(8)	109.76(12)
C(6)-C(7)-C(8)	124.66(13)
C(9)-C(8)-C(7)	106.89(13)
C(8)-C(9)-C(10)	107.90(13)
N(2)-C(10)-C(1)#1	124.11(13)
N(2)-C(10)-C(9)	109.71(12)
C(1)#1-C(10)-C(9)	125.98(13)
C(1)-C(11)-C(12)	120.59(13)
C(1)-C(11)-C(15)	125.04(13)
C(12)-C(11)-C(15)	59.76(10)
C(13)-C(12)-C(11)	119.07(13)
C(13)-C(12)-C(14)	113.68(14)
C(11)-C(12)-C(14)	116.83(14)
C(13)-C(12)-C(15)	121.01(13)
C(11)-C(12)-C(15)	60.94(10)
C(14)-C(12)-C(15)	115.57(13)
C(16)-C(15)-C(12)	127.45(14)
C(16)-C(15)-C(11)	128.10(13)
C(12)-C(15)-C(11)	59.30(10)
O(1)-C(16)-O(2)	123.33(14)
O(1)-C(16)-C(15)	120.27(14)
O(2)-C(16)-C(15)	116.37(13)
O(2)-C(17)-C(22)	111.62(13)
O(2)-C(17)-C(18)	112.23(13)
C(22)-C(17)-C(18)	103.04(14)
C(17)-C(18)-C(19)	102.2(2)
C(24)-C(19)-C(18)	101.7(2)
C(24)-C(19)-C(20)	100.3(2)
C(18)-C(19)-C(20)	105.9(2)
C(21)-C(20)-C(19)	103.3(2)
C(20)-C(21)-C(22)	105.0(2)
C(21)-C(22)-C(17)	110.0(2)
C(21)-C(22)-C(24)	104.3(2)
C(17)-C(22)-C(24)	102.09(13)
C(21)-C(22)-C(23)	114.0(2)
C(17)-C(22)-C(23)	111.7(2)
C(24)-C(22)-C(23)	114.0(2)
C(25)-C(24)-C(22)	114.9(2)
C(25)-C(24)-C(26)	106.3(2)
C(22)-C(24)-C(26)	114.7(2)
C(25)-C(24)-C(19)	116.0(2)
C(22)-C(24)-C(19)	91.77(14)
C(26)-C(24)-C(19)	113.1(2)
C(6)-C(31)-C(32)	123.02(13)
C(6)-C(31)-C(35)	126.22(13)
C(32)-C(31)-C(35)	59.72(10)
C(31)-C(32)-C(35)	61.06(10)
C(31)-C(32)-C(33)	118.94(14)
C(35)-C(32)-C(33)	119.37(14)
C(31)-C(32)-C(34)	118.14(14)

C(35)-C(32)-C(34)	116.51(14)
C(33)-C(32)-C(34)	113.3(2)
C(36)-C(35)-C(32)	121.98(14)
C(36)-C(35)-C(31)	126.06(14)
C(32)-C(35)-C(31)	59.22(10)
O(3)-C(36)-O(4)	123.4(2)
O(3)-C(36)-C(35)	128.3(2)
O(4)-C(36)-C(35)	108.29(14)
O(4)-C(37)-C(42)	109.5(2)
O(4)-C(37)-C(38)	114.0(2)
C(42)-C(37)-C(38)	103.6(2)
C(39)-C(38)-C(37)	102.5(2)
C(40)-C(39)-C(44)	105.8(2)
C(40)-C(39)-C(38)	107.0(2)
C(44)-C(39)-C(38)	102.8(2)
C(39)-C(40)-C(41)	101.4(2)
C(42)-C(41)-C(40)	104.3(2)
C(43)-C(42)-C(37)	113.8(2)
C(43)-C(42)-C(41)	115.9(2)
C(37)-C(42)-C(41)	108.5(2)
C(43)-C(42)-C(44)	115.8(2)
C(37)-C(42)-C(44)	99.7(2)
C(41)-C(42)-C(44)	101.3(2)
C(39)-C(44)-C(46)	115.0(2)
C(39)-C(44)-C(45)	115.0(2)
C(46)-C(44)-C(45)	106.6(2)
C(39)-C(44)-C(42)	92.4(2)
C(46)-C(44)-C(42)	115.8(2)
C(45)-C(44)-C(42)	111.9(2)
C(53)-C(52)-C(51)	115.9(3)
C(52)-C(53)-C(54)	121.6(3)
C(54)#2-C(54)-C(53)	108.8(4)
C(64)-C(63)-C(62)	101.4(6)
C(62)-C(61)-C(61)#3	141.6(6)
C(61)-C(62)-C(63)	127.7(6)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,y,-z+3/2 #2 -x,y,-z+5/2 #3 x,-y,-z+2

Table 4. Anisotropic displacement parameters (A^2) for 3g.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Ni	0.02627(10)	0.03304(11)	0.02534(11)	0.000	-0.00072(13)	0.000
O(1)	0.0496(6)	0.0706(7)	0.0362(6)	0.0060(6)	-0.0011(6)	0.0000(6)
O(2)	0.0396(6)	0.0735(7)	0.0328(6)	0.0077(6)	-0.0048(5)	-0.0188(5)
O(3)	0.0635(7)	0.0705(8)	0.0695(8)	0.0172(7)	0.0256(7)	0.0268(6)
O(4)	0.0544(7)	0.0678(8)	0.0606(8)	0.0221(7)	0.0027(6)	0.0191(6)
N(1)	0.0315(5)	0.0340(5)	0.0202(6)	0.0053(6)	0.0026(5)	-0.0013(5)
N(2)	0.0284(6)	0.0326(5)	0.0208(5)	-0.0008(5)	-0.0003(5)	0.0028(6)
C(1)	0.0284(7)	0.0380(8)	0.0289(7)	-0.0008(7)	-0.0027(7)	-0.0032(7)
C(2)	0.0263(7)	0.0350(8)	0.0364(8)	-0.0012(7)	-0.0080(7)	0.0009(6)
C(3)	0.0358(8)	0.0533(9)	0.0340(8)	0.0054(8)	-0.0070(8)	0.0002(7)
C(4)	0.0185(6)	0.0515(8)	0.0357(9)	-0.0029(8)	0.0021(6)	0.0000(7)
C(5)	0.0263(7)	0.0348(8)	0.0370(8)	-0.0025(7)	0.0043(7)	0.0002(6)
C(6)	0.0325(7)	0.0386(8)	0.0282(7)	0.0006(7)	0.0082(7)	0.0034(7)
C(7)	0.0349(8)	0.0349(8)	0.0288(7)	-0.0030(7)	0.0000(7)	-0.0005(6)
C(8)	0.0322(7)	0.0521(9)	0.0253(7)	0.0017(7)	0.0045(7)	0.0072(7)
C(9)	0.0499(10)	0.0480(9)	0.0231(7)	-0.0024(7)	-0.0001(7)	0.0095(8)
C(10)	0.0381(8)	0.0348(8)	0.0246(7)	-0.0012(6)	-0.0031(7)	0.0010(7)
C(11)	0.0358(8)	0.0442(9)	0.0342(8)	0.0083(8)	-0.0076(7)	-0.0061(7)
C(12)	0.0301(8)	0.0438(8)	0.0408(9)	0.0062(8)	-0.0076(7)	0.0005(7)
C(13)	0.0431(9)	0.0545(10)	0.0538(11)	-0.0028(9)	-0.0034(9)	0.0095(8)
C(14)	0.0359(9)	0.0796(12)	0.0578(11)	0.0111(10)	-0.0036(9)	0.0068(9)
C(15)	0.0409(8)	0.0515(10)	0.0337(9)	0.0059(8)	-0.0103(8)	-0.0047(8)
C(16)	0.0466(9)	0.0408(8)	0.0358(8)	0.0039(8)	-0.0076(8)	-0.0003(8)
C(17)	0.0372(8)	0.0611(10)	0.0365(8)	-0.0017(9)	0.0022(7)	-0.0063(9)
C(18)	0.0731(12)	0.0613(11)	0.0938(14)	0.0324(11)	-0.0296(11)	-0.0174(10)
C(19)	0.1063(15)	0.0498(11)	0.142(2)	0.0209(13)	-0.0672(14)	-0.0286(11)
C(20)	0.094(2)	0.212(3)	0.131(2)	-0.101(2)	-0.028(2)	0.067(2)
C(21)	0.0567(11)	0.225(3)	0.0597(13)	-0.030(2)	0.0125(11)	-0.049(2)
C(22)	0.0603(10)	0.0511(9)	0.0431(9)	0.0124(9)	-0.0106(8)	-0.0122(9)
C(23)	0.133(2)	0.0684(13)	0.225(3)	0.051(2)	-0.126(2)	-0.0317(13)
C(24)	0.0392(9)	0.0617(11)	0.0556(11)	0.0043(10)	-0.0011(9)	-0.0094(9)
C(25)	0.0552(12)	0.238(3)	0.079(2)	-0.046(2)	0.0121(13)	-0.041(2)
C(26)	0.0665(12)	0.0708(12)	0.0792(14)	0.0040(11)	-0.0121(11)	-0.0260(10)
C(31)	0.0330(8)	0.0453(8)	0.0341(8)	0.0084(7)	0.0075(7)	0.0044(7)
C(32)	0.0406(9)	0.0529(10)	0.0427(9)	0.0045(9)	0.0032(8)	-0.0029(8)
C(33)	0.0787(13)	0.0509(11)	0.0625(12)	-0.0046(10)	0.0097(11)	-0.0154(10)
C(34)	0.0363(8)	0.0987(13)	0.0634(11)	0.0228(10)	0.0076(10)	-0.0133(11)
C(35)	0.0362(8)	0.0554(9)	0.0373(8)	0.0129(8)	0.0154(7)	0.0071(8)
C(36)	0.0475(9)	0.0539(10)	0.0559(11)	0.0174(9)	0.0170(9)	0.0003(9)
C(37)	0.0625(12)	0.0511(10)	0.0727(12)	0.0216(10)	-0.0112(11)	0.0052(9)
C(38)	0.0400(11)	0.0618(12)	0.208(3)	0.043(2)	-0.0147(14)	0.0028(9)
C(39)	0.0844(15)	0.0616(12)	0.152(2)	0.0088(14)	-0.0680(13)	0.0022(11)
C(40)	0.129(2)	0.100(2)	0.125(2)	-0.037(2)	-0.065(2)	0.032(2)
C(41)	0.095(2)	0.127(2)	0.0682(15)	0.017(2)	-0.0084(14)	0.0293(15)
C(42)	0.0683(12)	0.0716(12)	0.0695(13)	0.0268(11)	-0.0194(11)	-0.0154(11)
C(43)	0.105(2)	0.122(2)	0.138(2)	0.051(2)	-0.033(2)	-0.0534(15)
C(44)	0.1053(15)	0.0566(11)	0.0890(14)	0.0137(11)	-0.0545(12)	-0.0010(12)
C(45)	0.184(3)	0.149(2)	0.103(2)	0.072(2)	-0.054(2)	0.007(2)
C(46)	0.125(2)	0.0618(13)	0.154(2)	0.0119(15)	-0.068(2)	0.0206(13)
C(51)	0.156(3)	0.194(4)	0.188(3)	-0.029(3)	-0.106(2)	0.032(3)
C(52)	0.127(2)	0.120(2)	0.364(5)	-0.030(3)	-0.145(2)	0.028(2)
C(53)	0.120(2)	0.0687(14)	0.361(4)	-0.073(2)	-0.112(2)	0.0387(15)
C(54)	0.251(3)	0.076(2)	0.449(4)	0.023(2)	-0.278(3)	-0.019(2)
C(63)	0.418(8)	0.308(8)	0.328(8)	-0.078(7)	0.093(8)	-0.056(7)
C(64)	0.595(20)	0.401(13)	0.556(16)	0.016(11)	0.043(15)	-0.068(13)
C(61)	0.434(8)	0.333(8)	0.576(14)	-0.153(8)	-0.239(8)	0.063(8)
C(62)	0.299(6)	0.449(9)	0.365(9)	-0.084(7)	0.054(7)	0.144(6)

Table 5. Hydrogen coordinates and isotropic displacement parameters (A^2) for 3g.

	x	y	z	U(eq)
H(3)	0.89035(13)	0.28765(4)	0.81554(7)	0.049
H(4)	0.91204(11)	0.31923(4)	0.71891(6)	0.042
H(8)	0.62818(12)	0.32502(4)	0.53628(6)	0.044
H(9)	0.44561(13)	0.29466(4)	0.52867(6)	0.048
H(11)	0.80135(12)	0.28612(5)	0.92267(7)	0.046
H(13A)	0.78907(13)	0.18234(5)	0.84046(8)	0.076
H(13B)	0.77220(13)	0.22062(5)	0.80530(8)	0.076
H(13C)	0.67401(13)	0.20346(5)	0.84313(8)	0.076
H(14A)	0.93334(13)	0.23671(6)	0.94536(8)	0.087
H(14B)	0.95997(13)	0.22946(6)	0.87731(8)	0.087
H(14C)	0.92555(13)	0.19538(6)	0.91892(8)	0.087
H(15)	0.75678(13)	0.23701(5)	0.98588(7)	0.050
H(17)	0.39455(12)	0.20696(5)	0.95207(7)	0.054
H(18A)	0.4125(2)	0.14631(5)	0.97847(10)	0.091
H(18B)	0.5198(2)	0.14199(5)	0.93765(10)	0.091
H(19)	0.3468(2)	0.10497(6)	0.89936(11)	0.119
H(20A)	0.5176(2)	0.12810(9)	0.83491(13)	0.175
H(20B)	0.4084(2)	0.11929(9)	0.79771(13)	0.175
H(21A)	0.3907(2)	0.17876(8)	0.77686(10)	0.137
H(21B)	0.5016(2)	0.18739(8)	0.81246(10)	0.137
H(23A)	0.2700(2)	0.23925(6)	0.88397(13)	0.213
H(23B)	0.2623(2)	0.22803(6)	0.81588(13)	0.213
H(23C)	0.3693(2)	0.24844(6)	0.84046(13)	0.213
H(25A)	0.1543(2)	0.14815(9)	0.93909(11)	0.186
H(25B)	0.1633(2)	0.19184(9)	0.92517(11)	0.186
H(25C)	0.2502(2)	0.17240(9)	0.96796(11)	0.186
H(26A)	0.2444(2)	0.14379(6)	0.79216(9)	0.108
H(26B)	0.1521(2)	0.17053(6)	0.81881(9)	0.108
H(26C)	0.1579(2)	0.12767(6)	0.83843(9)	0.108
H(31)	0.85289(12)	0.31629(4)	0.60639(7)	0.045
H(33A)	0.8984(2)	0.42468(5)	0.66785(9)	0.096
H(33B)	0.8423(2)	0.39321(5)	0.70769(9)	0.096
H(33C)	0.7730(2)	0.41386(5)	0.65759(9)	0.096
H(34A)	1.0430(2)	0.39390(6)	0.62334(8)	0.099
H(34B)	1.0263(2)	0.35980(6)	0.57871(8)	0.099
H(34C)	1.0340(2)	0.35217(6)	0.64803(8)	0.099
H(35)	0.85964(13)	0.36123(5)	0.53170(7)	0.052
H(37)	0.6426(2)	0.45043(5)	0.49171(9)	0.075
H(38A)	0.5369(2)	0.38592(6)	0.44855(14)	0.124
H(38B)	0.4792(2)	0.42494(6)	0.46574(14)	0.124
H(39)	0.4591(2)	0.42542(6)	0.36093(12)	0.119
H(40A)	0.5988(2)	0.39923(7)	0.30098(12)	0.142
H(40B)	0.5955(2)	0.37108(7)	0.35654(12)	0.142
H(41A)	0.7665(2)	0.39121(8)	0.38481(11)	0.116
H(41B)	0.7699(2)	0.41924(8)	0.32932(11)	0.116
H(43A)	0.8254(2)	0.48058(8)	0.37669(13)	0.182
H(43B)	0.8570(2)	0.45947(8)	0.43643(13)	0.182
H(43C)	0.7726(2)	0.49358(8)	0.43781(13)	0.182
H(45A)	0.6561(3)	0.50229(9)	0.31514(11)	0.218
H(45B)	0.5490(3)	0.48183(9)	0.29079(11)	0.218
H(45C)	0.6641(3)	0.46068(9)	0.28985(11)	0.218
H(46A)	0.5812(2)	0.51657(6)	0.40780(12)	0.171
H(46B)	0.5321(2)	0.48632(6)	0.45242(12)	0.171
H(46C)	0.4648(2)	0.49796(6)	0.39485(12)	0.171
H(51A)	0.2722(3)	0.10434(11)	1.0670(2)	0.269
H(51B)	0.1529(3)	0.12261(11)	1.0665(2)	0.269
H(51C)	0.1668(3)	0.07849(11)	1.0584(2)	0.269
H(52A)	0.2333(3)	0.07365(10)	1.1580(2)	0.244
H(52B)	0.2194(3)	0.11766(10)	1.1661(2)	0.244
H(53A)	0.0504(2)	0.06623(7)	1.1525(2)	0.220
H(53B)	0.0325(2)	0.11013(7)	1.1551(2)	0.220
H(54A)	0.0874(2)	0.06348(8)	1.2612(2)	0.310
H(54B)	0.0887(2)	0.10821(8)	1.2656(2)	0.310
H(63A)	0.2623(6)	-0.0135(2)	1.1723(3)	0.422
H(63B)	0.3598(6)	-0.0439(2)	1.1720(3)	0.422
H(64A)	0.3909(6)	0.0010(2)	1.2405(4)	0.776
H(64B)	0.3851(6)	0.0321(2)	1.1900(4)	0.776

H(64C)	0.4816(6)	0.0019(2)	1.1898(4)	0.776
H(61A)	0.2409(6)	0.0021(3)	1.0534(3)	0.537
H(61B)	0.3043(6)	-0.0345(3)	1.0374(3)	0.537
H(62A)	0.3950(5)	0.0234(2)	1.0973(3)	0.445
H(62B)	0.4473(5)	-0.0163(2)	1.0857(3)	0.445

Table 1. Crystal data and structure refinement for 3i.

Identification code	$Ni(TMPCP)\cdot 6HCON(CH_3)_2$
Empirical formula	C90 H114 N14 Ni O10
Formula weight	1610.66
Temperature	193(2) K
Wavelength	0.71073 A
Crystal system	Orthorhombic
Space group	P2(1)2(1)2
Unit cell dimensions	a = 16.1891(8) A alpha = 90 deg. b = 21.6791(11) A beta = 90 deg. c = 11.9781(6) A gamma = 90 deg.
Volume, Z	4203.9(4) A^3, 2
Density (calculated)	1.272 Mg/m^3
Absorption coefficient	0.299 mm^-1
F(000)	1720
Crystal size	0.1 x 0.25 x 0.5 mm
Theta range for data collection	1.57 to 26.86 deg.
Limiting indices	-19<=h<=17, -20<=k<=27, -15<=l<=15
Reflections collected	23071
Independent reflections	8127 [R(int) = 0.0419]
Absorption correction	SADABS
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8127 / 0 / 512
Goodness-of-fit on F^2	1.112
Final R indices [a] [I>2sigma(I)]	R1 = 0.0558, wR2 = 0.1417
R indices (all data)	R1 = 0.0863, wR2 = 0.1578
Absolute structure parameter	0.006(18)
Largest diff. peak and hole	1.164 and -0.537 e.A^-3

[a] $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ and $wR_2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[wF_o^4]\}^{1/2}$ The conventional R-factors R_1 are based on F, with F set to zero for negative F^2. The criterion of $F^2 < 2\sigma(F^2)$ was used only for calculating R_1. R-factors based on F^2 (wR_2) are statistically about twice as large as those based on F, and R-factors based on all data will be even larger.

Table 2. Atomic coordinates and equivalent isotropic displacement parameters (A^2) for 3i. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Ni	0.0000	0.5000	0.92588(5)	0.02324(17)
O(11)	0.2093(2)	0.63663(14)	1.0561(3)	0.0587(9)
O(31)	0.1929(2)	0.32727(15)	0.8133(3)	0.0551(9)
N(1)	0.0000	0.5000	1.0858(3)	0.0256(8)
N(2)	0.11687(17)	0.48891(13)	0.9253(2)	0.0249(7)
N(3)	0.0000	0.5000	0.7675(3)	0.0281(9)
N(11)	0.3461(3)	0.64604(19)	1.0603(4)	0.0649(12)
N(31)	0.3294(2)	0.31648(18)	0.7875(3)	0.0480(10)
C(1)	0.0406(2)	0.5075(2)	1.2694(3)	0.0316(9)
C(2)	0.0668(2)	0.51014(18)	1.1552(3)	0.0294(9)
C(3)	0.1484(2)	0.51638(16)	1.1200(3)	0.0292(9)
C(4)	0.1712(2)	0.5025(2)	1.0121(3)	0.0267(7)
C(5)	0.2540(2)	0.4906(2)	0.9765(3)	0.0353(10)
C(6)	0.2504(2)	0.4681(2)	0.8721(3)	0.0358(10)
C(7)	0.1650(2)	0.46787(18)	0.8386(3)	0.0287(9)
C(8)	0.1378(2)	0.45608(18)	0.7313(3)	0.0293(9)
C(9)	0.0608(2)	0.47603(18)	0.6972(3)	0.0296(9)
C(10)	0.0359(2)	0.48369(19)	0.5823(3)	0.0395(11)
C(11)	0.2153(2)	0.52663(19)	1.2053(3)	0.0327(9)
C(12)	0.2207(3)	0.5782(2)	1.2877(3)	0.0394(10)
C(13)	0.1573(3)	0.6296(2)	1.2923(4)	0.0563(13)
C(14)	0.2584(3)	0.5639(3)	1.3988(4)	0.0610(14)
C(15)	0.2809(3)	0.5775(2)	1.1899(3)	0.0374(10)
C(16)	0.2749(3)	0.6210(2)	1.0979(4)	0.0443(11)
C(17)	0.3413(4)	0.6904(3)	0.9658(7)	0.118(3)
C(18)	0.4256(3)	0.6347(3)	1.1095(5)	0.0593(15)
C(19)	0.4800(5)	0.5965(3)	1.0591(7)	0.114(3)
C(20)	0.5600(8)	0.5895(7)	1.1101(13)	0.189(9)
C(21)	0.5737(8)	0.6186(10)	1.1975(15)	0.210(10)
C(22)	0.5213(7)	0.6571(8)	1.2488(12)	0.204(7)
C(23)	0.4479(4)	0.6640(4)	1.2007(8)	0.122(3)
C(31)	0.1957(2)	0.4338(2)	0.6435(3)	0.0337(9)
C(32)	0.1818(3)	0.3778(2)	0.5718(4)	0.0431(10)
C(33)	0.1091(3)	0.3369(2)	0.5899(5)	0.0659(15)
C(34)	0.2071(4)	0.3818(3)	0.4498(4)	0.0678(16)
C(35)	0.2534(3)	0.3781(2)	0.6559(3)	0.0399(10)
C(36)	0.2540(3)	0.3380(2)	0.7570(4)	0.0417(11)
C(37)	0.3361(4)	0.2871(3)	0.8977(4)	0.0666(15)
C(38)	0.4000(3)	0.3150(2)	0.7171(4)	0.0496(12)
C(39)	0.4727(4)	0.3448(3)	0.7476(6)	0.0755(18)
C(40)	0.5426(4)	0.3408(3)	0.6765(8)	0.093(2)
C(41)	0.5374(5)	0.3086(4)	0.5797(7)	0.094(2)
C(42)	0.4674(4)	0.2805(3)	0.5488(6)	0.083(2)
C(43)	0.3983(3)	0.2833(2)	0.6163(4)	0.0574(14)
O(51)	0.2787(2)	0.85836(18)	0.8297(3)	0.0629(10)
N(51)	0.4090(2)	0.84004(17)	0.8892(3)	0.0398(9)
C(51)	0.3299(3)	0.8564(2)	0.9025(4)	0.0454(12)
C(52)	0.4664(3)	0.8393(2)	0.9818(4)	0.0558(13)
C(53)	0.4400(3)	0.8243(3)	0.7807(4)	0.0672(15)
O(71)	0.5000	0.5000	0.8275(8)	0.253(10)
N(71)	0.5000	0.5000	0.6413(6)	0.0766(17)
C(71)	0.5371(12)	0.4968(11)	0.7442(17)	0.139(9)
C(72)	0.562(2)	0.520(3)	0.525(3)	0.38(3)
C(73)	0.4220(6)	0.5018(8)	0.5987(15)	0.094(5)
N(61)	0.6693(3)	0.8219(3)	0.2716(4)	0.097(2)
C(62)	0.6782(14)	0.8811(5)	0.2836(12)	0.400(17)
C(63)	0.6229(5)	0.8040(6)	0.1775(6)	0.173(5)
O(61)	0.7464(4)	0.7915(4)	0.4184(5)	0.162(2)
C(61)	0.7113(5)	0.7817(4)	0.3308(6)	0.118(3)

Table 3. Bond lengths [A] and angles [deg] for 3i.

Ni-N(3)	1.897(4)
Ni-N(2)#1	1.907(3)
Ni-N(2)	1.907(3)
Ni-N(1)	1.915(4)
O(11)-C(16)	1.221(5)
O(31)-C(36)	1.219(5)
N(1)-C(2)	1.382(4)
N(1)-C(2)#1	1.382(4)
N(2)-C(7)	1.375(5)
N(2)-C(4)	1.393(4)
N(3)-C(9)	1.395(4)
N(3)-C(9)#1	1.395(4)
N(11)-C(16)	1.352(6)
N(11)-C(18)	1.436(7)
N(11)-C(17)	1.488(8)
N(31)-C(36)	1.358(6)
N(31)-C(38)	1.420(6)
N(31)-C(37)	1.469(6)
C(1)-C(1)#1	1.355(7)
C(1)-C(2)	1.433(5)
C(2)-C(3)	1.392(5)
C(3)-C(4)	1.378(5)
C(3)-C(11)	1.507(5)
C(4)-C(5)	1.429(5)
C(5)-C(6)	1.343(5)
C(6)-C(7)	1.440(5)
C(7)-C(8)	1.383(5)
C(8)-C(9)	1.381(5)
C(8)-C(31)	1.490(5)
C(9)-C(10)	1.443(5)
C(10)-C(10)#1	1.361(8)
C(11)-C(12)	1.493(6)
C(11)-C(15)	1.541(6)
C(12)-C(14)	1.496(6)
C(12)-C(13)	1.518(6)
C(12)-C(15)	1.524(6)
C(15)-C(16)	1.454(6)
C(18)-C(23)	1.314(9)
C(18)-C(19)	1.350(8)
C(19)-C(20)	1.441(15)
C(20)-C(21)	1.24(2)
C(21)-C(22)	1.34(2)
C(22)-C(23)	1.329(12)
C(31)-C(32)	1.503(6)
C(31)-C(35)	1.535(6)
C(32)-C(33)	1.490(6)
C(32)-C(34)	1.521(6)
C(32)-C(35)	1.536(6)
C(35)-C(36)	1.490(6)
C(38)-C(43)	1.390(7)
C(38)-C(39)	1.391(7)
C(39)-C(40)	1.419(9)
C(40)-C(41)	1.357(11)
C(41)-C(42)	1.338(9)
C(42)-C(43)	1.381(7)
O(51)-C(51)	1.204(5)
N(51)-C(51)	1.338(6)
N(51)-C(53)	1.434(6)
N(51)-C(52)	1.447(6)
O(71)-C(71)	1.17(2)
O(71)-C(71)#2	1.17(2)
N(71)-C(73)	1.363(10)
N(71)-C(73)#2	1.363(10)
N(71)-C(71)#2	1.372(17)
N(71)-C(71)	1.372(17)
N(71)-C(72)	1.77(3)
N(71)-C(72)#2	1.77(3)
C(71)-C(71)#2	1.21(4)

C(71)-C(73)#2	1.87(3)
C(72)-C(73)#2	1.04(5)
C(73)-C(72)#2	1.04(5)
C(73)-C(71)#2	1.87(3)
N(61)-C(62)	1.299(14)
N(61)-C(61)	1.313(9)
N(61)-C(63)	1.410(8)
O(61)-C(61)	1.211(8)
N(3)-Ni-N(2)#1	89.78(8)
N(3)-Ni-N(2)	89.78(8)
N(2)#1-Ni-N(2)	179.56(17)
N(3)-Ni-N(1)	180.000(2)
N(2)#1-Ni-N(1)	90.22(8)
N(2)-Ni-N(1)	90.22(8)
C(2)-N(1)-C(2)#1	106.0(4)
C(2)-N(1)-Ni	126.99(18)
C(2)#1-N(1)-Ni	126.99(18)
C(7)-N(2)-C(4)	106.0(3)
C(7)-N(2)-Ni	127.3(2)
C(4)-N(2)-Ni	126.7(2)
C(9)-N(3)-C(9)#1	105.7(4)
C(9)-N(3)-Ni	127.13(19)
C(9)#1-N(3)-Ni	127.13(19)
C(16)-N(11)-C(18)	124.0(4)
C(16)-N(11)-C(17)	117.9(5)
C(18)-N(11)-C(17)	118.1(5)
C(36)-N(31)-C(38)	124.9(4)
C(36)-N(31)-C(37)	117.2(4)
C(38)-N(31)-C(37)	117.7(4)
C(1)#1-C(1)-C(2)	107.3(2)
N(1)-C(2)-C(3)	125.1(3)
N(1)-C(2)-C(1)	109.7(3)
C(3)-C(2)-C(1)	125.0(3)
C(4)-C(3)-C(2)	121.2(3)
C(4)-C(3)-C(11)	118.4(3)
C(2)-C(3)-C(11)	119.4(3)
C(3)-C(4)-N(2)	125.2(3)
C(3)-C(4)-C(5)	124.8(3)
N(2)-C(4)-C(5)	109.4(3)
C(6)-C(5)-C(4)	107.6(3)
C(5)-C(6)-C(7)	107.6(4)
N(2)-C(7)-C(8)	125.6(3)
N(2)-C(7)-C(6)	109.4(3)
C(8)-C(7)-C(6)	124.5(4)
C(9)-C(8)-C(7)	120.3(3)
C(9)-C(8)-C(31)	117.4(3)
C(7)-C(8)-C(31)	121.0(3)
C(8)-C(9)-N(3)	125.0(3)
C(8)-C(9)-C(10)	124.7(3)
N(3)-C(9)-C(10)	109.7(3)
C(10)#1-C(10)-C(9)	107.3(2)
C(12)-C(11)-C(3)	126.9(4)
C(12)-C(11)-C(15)	60.3(3)
C(3)-C(11)-C(15)	121.3(3)
C(11)-C(12)-C(14)	117.2(4)
C(11)-C(12)-C(13)	122.3(4)
C(14)-C(12)-C(13)	113.3(4)
C(11)-C(12)-C(15)	61.5(3)
C(14)-C(12)-C(15)	114.9(4)
C(13)-C(12)-C(15)	117.9(4)
C(16)-C(15)-C(12)	122.3(4)
C(16)-C(15)-C(11)	120.6(4)
C(12)-C(15)-C(11)	58.3(3)
O(11)-C(16)-N(11)	119.6(4)
O(11)-C(16)-C(15)	123.2(4)
N(11)-C(16)-C(15)	117.1(4)
C(23)-C(18)-C(19)	119.2(7)
C(23)-C(18)-N(11)	120.3(5)
C(19)-C(18)-N(11)	120.4(6)
C(18)-C(19)-C(20)	117.6(9)
C(21)-C(20)-C(19)	117.6(14)
C(20)-C(21)-C(22)	126.2(17)

C(23)-C(22)-C(21)	115.9(13)
C(18)-C(23)-C(22)	123.5(9)
C(8)-C(31)-C(32)	124.8(4)
C(8)-C(31)-C(35)	124.7(3)
C(32)-C(31)-C(35)	60.8(3)
C(33)-C(32)-C(31)	121.1(4)
C(33)-C(32)-C(34)	112.7(4)
C(31)-C(32)-C(34)	117.6(4)
C(33)-C(32)-C(35)	120.2(4)
C(31)-C(32)-C(35)	60.6(3)
C(34)-C(32)-C(35)	115.3(4)
C(36)-C(35)-C(31)	122.8(3)
C(36)-C(35)-C(32)	122.4(4)
C(31)-C(35)-C(32)	58.6(3)
O(31)-C(36)-N(31)	121.0(4)
O(31)-C(36)-C(35)	123.8(4)
N(31)-C(36)-C(35)	115.1(4)
C(43)-C(38)-C(39)	118.3(5)
C(43)-C(38)-N(31)	120.7(5)
C(39)-C(38)-N(31)	121.0(5)
C(38)-C(39)-C(40)	119.3(6)
C(41)-C(40)-C(39)	119.7(7)
C(42)-C(41)-C(40)	121.5(7)
C(41)-C(42)-C(43)	120.3(7)
C(42)-C(43)-C(38)	121.0(6)
C(51)-N(51)-C(53)	120.4(4)
C(51)-N(51)-C(52)	121.8(4)
C(53)-N(51)-C(52)	117.8(4)
O(51)-C(51)-N(51)	125.6(4)
C(71)-O(71)-C(71)#2	62.4(16)
C(73)-N(71)-C(73)#2	135.9(16)
C(73)-N(71)-C(71)#2	86.0(12)
C(73)#2-N(71)-C(71)#2	138.0(14)
C(73)-N(71)-C(71)	138.0(14)
C(73)#2-N(71)-C(71)	86.0(12)
C(71)#2-N(71)-C(71)	52.2(19)
C(73)-N(71)-C(72)	102.6(17)
C(73)#2-N(71)-C(72)	35.9(16)
C(71)#2-N(71)-C(72)	160.3(19)
C(71)-N(71)-C(72)	118.3(16)
C(73)-N(71)-C(72)#2	35.9(16)
C(73)#2-N(71)-C(72)#2	102.6(17)
C(71)#2-N(71)-C(72)#2	118.3(16)
C(71)-N(71)-C(72)#2	160.3(19)
C(72)-N(71)-C(72)#2	76(3)
O(71)-C(71)-C(71)#2	58.8(8)
O(71)-C(71)-N(71)	122.7(16)
C(71)#2-C(71)-N(71)	63.9(10)
O(71)-C(71)-C(73)#2	168.9(14)
C(71)#2-C(71)-C(73)#2	110.6(7)
N(71)-C(71)-C(73)#2	46.8(8)
C(73)#2-C(72)-N(71)	50.1(13)
C(72)#2-C(73)-N(71)	94(2)
C(72)#2-C(73)-C(71)#2	135(2)
N(71)-C(73)-C(71)#2	47.2(7)
C(62)-N(61)-C(61)	122.5(8)
C(62)-N(61)-C(63)	114.9(8)
C(61)-N(61)-C(63)	121.7(8)
O(61)-C(61)-N(61)	126.4(9)

Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,z #2 -x+1,-y+1,z

Table 4. Anisotropic displacement parameters (A^2) for 3i.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Ni	0.0250(3)	0.0261(3)	0.0186(3)	0.000	0.000	-0.0010(4)
O(11)	0.070(2)	0.0392(19)	0.067(2)	0.0124(18)	-0.008(2)	-0.0089(17)
O(31)	0.062(2)	0.052(2)	0.0514(19)	0.0029(16)	0.0210(18)	-0.0022(17)
N(1)	0.026(2)	0.027(2)	0.0239(19)	0.000	0.000	-0.008(2)
N(2)	0.0240(15)	0.0291(19)	0.0216(13)	0.0002(16)	-0.0001(13)	-0.0013(13)
N(3)	0.030(2)	0.031(2)	0.0225(19)	0.000	0.000	0.000(3)
N(11)	0.070(3)	0.043(2)	0.082(3)	0.006(2)	0.018(3)	-0.013(2)
N(31)	0.054(2)	0.048(2)	0.042(2)	0.0109(19)	0.0091(19)	0.016(2)
C(1)	0.0355(19)	0.039(3)	0.0206(15)	-0.0020(19)	-0.0059(15)	-0.007(2)
C(2)	0.033(2)	0.031(3)	0.0243(16)	-0.0004(18)	-0.0043(16)	-0.0023(18)
C(3)	0.032(2)	0.025(2)	0.0306(19)	0.0025(15)	-0.0085(18)	0.0006(16)
C(4)	0.0273(18)	0.0281(19)	0.0246(16)	-0.005(2)	-0.0029(14)	0.001(2)
C(5)	0.0279(19)	0.043(3)	0.0351(19)	-0.004(2)	-0.0053(16)	-0.005(2)
C(6)	0.023(2)	0.044(2)	0.041(2)	-0.001(2)	0.0075(19)	0.0004(18)
C(7)	0.026(2)	0.029(2)	0.031(2)	-0.0002(18)	0.0028(17)	-0.0004(17)
C(8)	0.029(2)	0.032(2)	0.027(2)	0.0000(17)	0.0042(17)	0.0015(17)
C(9)	0.033(2)	0.034(2)	0.0214(17)	-0.0012(16)	0.0049(17)	0.0034(17)
C(10)	0.040(2)	0.057(3)	0.0223(17)	-0.0003(19)	0.0009(17)	0.0079(18)
C(11)	0.035(2)	0.033(2)	0.030(2)	0.0020(18)	-0.0096(18)	-0.0036(18)
C(12)	0.042(3)	0.045(3)	0.032(2)	-0.008(2)	-0.005(2)	-0.007(2)
C(13)	0.057(3)	0.049(3)	0.063(3)	-0.021(3)	-0.008(3)	0.001(2)
C(14)	0.069(3)	0.075(4)	0.040(3)	-0.006(2)	-0.017(2)	0.001(3)
C(15)	0.030(2)	0.041(3)	0.040(2)	-0.002(2)	-0.0083(19)	-0.0072(19)
C(16)	0.047(3)	0.035(2)	0.051(3)	-0.002(2)	0.001(2)	-0.012(2)
C(17)	0.107(5)	0.099(6)	0.149(7)	0.073(5)	0.050(5)	0.004(4)
C(18)	0.039(3)	0.053(3)	0.086(4)	-0.015(3)	0.024(3)	-0.010(2)
C(19)	0.133(7)	0.095(5)	0.115(5)	0.024(4)	0.072(5)	0.048(5)
C(20)	0.112(10)	0.181(14)	0.27(2)	0.104(13)	0.118(12)	0.092(10)
C(21)	0.064(7)	0.28(2)	0.29(2)	0.082(19)	0.013(12)	-0.060(11)
C(22)	0.085(8)	0.276(17)	0.250(16)	-0.086(12)	-0.049(9)	-0.046(9)
C(23)	0.057(4)	0.134(7)	0.174(8)	-0.083(7)	-0.003(5)	-0.019(5)
C(31)	0.035(2)	0.040(2)	0.0259(19)	0.0011(18)	0.0024(18)	0.0083(19)
C(32)	0.043(3)	0.051(3)	0.035(2)	-0.014(2)	-0.002(2)	0.008(2)
C(33)	0.057(3)	0.055(3)	0.086(4)	-0.022(3)	-0.006(3)	-0.003(3)
C(34)	0.084(4)	0.082(4)	0.037(3)	-0.026(3)	-0.006(3)	0.027(3)
C(35)	0.041(2)	0.046(3)	0.033(2)	0.006(2)	0.016(2)	0.012(2)
C(36)	0.051(3)	0.034(2)	0.040(2)	-0.0039(19)	0.006(2)	0.003(2)
C(37)	0.083(4)	0.066(4)	0.051(3)	0.015(3)	0.001(3)	0.020(3)
C(38)	0.050(3)	0.032(3)	0.067(3)	0.009(2)	0.000(3)	0.016(2)
C(39)	0.069(4)	0.059(4)	0.099(5)	0.011(3)	-0.018(3)	0.009(3)
C(40)	0.043(4)	0.076(5)	0.162(8)	0.037(5)	-0.005(4)	-0.002(3)
C(41)	0.091(6)	0.083(5)	0.108(6)	0.018(5)	0.036(5)	0.021(4)
C(42)	0.086(5)	0.069(4)	0.094(5)	-0.007(4)	0.033(4)	0.021(4)
C(43)	0.074(4)	0.040(3)	0.059(3)	-0.003(2)	0.021(3)	0.010(3)
O(51)	0.042(2)	0.088(3)	0.058(2)	-0.003(2)	-0.0111(18)	0.0088(18)
N(51)	0.037(2)	0.038(2)	0.044(2)	-0.0002(17)	-0.0034(17)	0.0068(16)
C(51)	0.047(3)	0.045(3)	0.044(3)	0.003(2)	0.006(2)	0.002(2)
C(52)	0.057(3)	0.044(3)	0.066(3)	-0.004(2)	-0.015(3)	0.003(2)
C(53)	0.064(3)	0.078(4)	0.060(3)	-0.006(3)	0.003(3)	0.023(3)
O(71)	0.53(3)	0.156(10)	0.076(5)	0.000	0.000	0.131(15)
N(71)	0.077(4)	0.076(4)	0.077(4)	0.000	0.000	-0.009(5)
C(71)	0.21(2)	0.049(8)	0.159(15)	-0.008(15)	-0.087(15)	-0.002(19)
C(72)	0.25(4)	0.63(9)	0.25(4)	-0.16(5)	0.22(3)	-0.02(5)
C(73)	0.031(5)	0.081(8)	0.171(17)	-0.018(13)	-0.053(7)	-0.016(7)
N(61)	0.076(4)	0.156(6)	0.059(3)	-0.007(4)	-0.023(3)	0.042(4)
C(62)	0.80(4)	0.128(10)	0.271(15)	-0.100(10)	-0.30(2)	0.296(19)
C(63)	0.127(7)	0.332(16)	0.059(4)	0.049(7)	-0.045(5)	-0.090(9)
O(61)	0.145(5)	0.185(7)	0.156(6)	0.019(6)	-0.021(5)	0.018(5)
C(61)	0.130(7)	0.136(7)	0.087(5)	0.030(5)	-0.061(5)	-0.041(6)

Table 5. Hydrogen coordinates and isotropic displacement parameters (A^2) for 3i.

	x	y	z	U(eq)
H(1)	0.0734	0.5146	1.3319	0.038
H(5)	0.3017	0.4973	1.0180	0.042
H(6)	0.2950	0.4551	0.8293	0.043
H(10)	0.0638	0.4690	0.5198	0.047
H(11)	0.2387	0.4880	1.2338	0.039
H(13A)	0.1357	0.6369	1.2188	0.084
H(13B)	0.1829	0.6666	1.3197	0.084
H(13C)	0.1130	0.6180	1.3414	0.084
H(14A)	0.2166	0.5660	1.4555	0.092
H(14B)	0.3011	0.5933	1.4147	0.092
H(14C)	0.2816	0.5231	1.3973	0.092
H(15)	0.3373	0.5651	1.2091	0.045
H(17A)	0.2853	0.6930	0.9399	0.177
H(17B)	0.3763	0.6766	0.9061	0.177
H(17C)	0.3593	0.7304	0.9905	0.177
H(19)	0.4663	0.5756	0.9939	0.137
H(20)	0.6000	0.5640	1.0787	0.227
H(21)	0.6252	0.6132	1.2305	0.253
H(22)	0.5354	0.6777	1.3142	0.244
H(23)	0.4103	0.6910	1.2332	0.146
H(31)	0.2215	0.4677	0.6021	0.040
H(33A)	0.0959	0.3357	0.6680	0.099
H(33B)	0.1219	0.2960	0.5644	0.099
H(33C)	0.0627	0.3525	0.5489	0.099
H(34A)	0.1587	0.3814	0.4036	0.102
H(34B)	0.2416	0.3473	0.4313	0.102
H(34C)	0.2371	0.4194	0.4374	0.102
H(35)	0.3080	0.3838	0.6219	0.048
H(37A)	0.2846	0.2911	0.9367	0.100
H(37B)	0.3791	0.3068	0.9399	0.100
H(37C)	0.3491	0.2442	0.8886	0.100
H(39)	0.4754	0.3672	0.8137	0.091
H(40)	0.5918	0.3601	0.6963	0.112
H(41)	0.5835	0.3060	0.5336	0.113
H(42)	0.4653	0.2590	0.4817	0.099
H(43)	0.3500	0.2637	0.5940	0.069
H(51)	0.3130	0.8672	0.9741	0.054
H(52A)	0.4378	0.8502	1.0493	0.084
H(52B)	0.5099	0.8684	0.9682	0.084
H(52C)	0.4894	0.7987	0.9895	0.084
H(53A)	0.3959	0.8262	0.7273	0.101
H(53B)	0.4624	0.7834	0.7823	0.101
H(53C)	0.4825	0.8530	0.7598	0.101
H(62A)	0.7107	0.8892	0.3490	0.601
H(62B)	0.6249	0.9000	0.2915	0.601
H(62C)	0.7056	0.8977	0.2193	0.601
H(63A)	0.5966	0.8395	0.1456	0.259
H(63B)	0.5816	0.7746	0.1996	0.259
H(63C)	0.6588	0.7856	0.1232	0.259
H(61A)	0.7145	0.7418	0.3028	0.141