Inorganic Chemistry
including bioinorganic chemistry
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Table S0. Crystal Data, Experimental Details of the X-ray Diffraction Study and Structure Solution.

(a) Crystal Parameters at 20°C

from a least-squares fit of the setting angles of 25 reflections with $13.1° < \theta < 20.7°$

Chem. Formula $C_{21}H_{28}GdN_5NiO_{15}$

Fw = 806.44	λ = 0.71073 Å
Crystal Habit: light-purple nearly cube	Crystal Dimensions: 0.45 x 0.40 x 0.40 mm
Crystal System: Monoclinic	Absences: 0k0: k = 2n + 1
Space Group: $P2_1$ (No. 4)	V = 1449.2(3) $Å^3$
a = 10.1351(6) Å	Z = 2
b = 16.079(2) Å	F(000) = 802
c = 8.8948(9) Å	Dcalc = 1.848 $g.cm^{-3}$
β = 91.138(7)°	μ = 2.997 mm^{-1}

(b) Measurement of Intensity Data

Instrument: Enraf-Nonius CAD4	Scan speed: 1.27-16.48°/min
Radiation: MoKα (λ = 0.71073 Å) graphite monochromated	Max 2θ: 54° (h -12,12 k -20,20 l 0,11)
Detector window: Height: 4.0 mm Width: (2.70 + 0.40 tanθ) mm	Standards: Intensity: 3 refl. every 2h Orientation: 3 refl. every 400 data refl.
Scan mode: ω-2θ	Take-off angle: 2.95°
Scan width: (0.80 + 0.35tanθ)°	No. of refl. collected: 6709

Table S0. (Continued)

(c) Treatment of Intensity Data

Reduction to Fo^2 and $\sigma(Fo^2)$, corrections for backgrounds, attenuator and LP in the usual manner [1]. No significative standard intensities variations (±0.6%). Absorption corrections by psi scanns [2] (Tmin = 0.8163, Tmax = 0.9994). 6315 unique reflections (Rav = 0.0136 on I). No. of observed data [$I > 2\sigma(I)$] : 6122

(d) Structure Solution and Refinement

Patterson method using SHELXS-86 [3]. Full-matrix least-squares refinement using SHELXL-93 [4]. All non-H atoms anisotropic. H atoms found on a difference Fourier map, introduced in calculations with the riding model with Uiso equal to 1.1 times that of atom of attachment, except H bonded to water molecules O(14) and O(15) allowed to vary. Scattering factors were taken from "International Tables for Crystallography" [5]. Refinement on Fo^2 for all reflections (6315). Weighted R-factors wR and goodnesses of fit S are based on Fo^2, conventional R-factors R are based on Fo, with Fo set to zero for negative Fo^2. Last refinement cycle: wR(all) = 0.0427, R(all) = 0.0177, wR(obs) = 0.0416, R(obs) = 0.0163 with 6122 obs. reflections > $4\sigma(Fo)$, 403 variable parameters, $w = [\sigma^2(Fo^2) + (0.0224\ P)^2 + 0.2976\ P]^{-1}$ where $P = (Fo^2 + 2Fc^2)/3$, S(all) = 1.053, S(obs) = 1.043, max. shift/esd = 0.002, mean shift/esd = 0.000, max. and min. residual peaks of 0.637 and -0.307 e/Å^3. Absolute configuration was determined: Flack coefficient [6] = -0.010(6).

Table S0. (Continued)

[1] Fair, C.K. MolEN. Structure Solution Procedures, Enraf-Nonius, Delft, Holland, 1990.

[2] North, A.C.T., Phillips, D.C. and Mathews, F.S. Acta Crystallogr., Sect. A, 1968, A21, 351-359.

[3] Sheldrick, G.M. SHELXS-86. Program for Crystal Structure Solution. Univ. of Göttingen: Göttingen, Germany, 1986.

[4] Sheldrick, G.M. SHELXL-93. Program for the refinement of crystal structures from diffraction data. Univ. of Göttingen: Göttingen, Germany, 1993.

[5] "International Tables for Crystallography", Vol. C, Kluwer Academic Publishers, Dordrecht, The Netherlands, 1992, Tables 4.2.6.8 and 6.1.1.4.

[6] Flack, H.D. Acta Crystallogr., Sect. A, 1983, A39, 876-881.

Table S2. Hydrogen atomic positional and thermal parameters ($Å^2$ x 100).

Atom	x/a	y/b	z/c	U
H(14A)	0.2259(33)	0.4388(21)	0.2385(31)	4.9(9)
H(14B)	0.1557(27)	0.4158(24)	0.3559(38)	5.6(11)
H(15A)	0.5472(32)	0.4738(21)	0.6834(29)	5.0(11)
H(15B)	0.6441(30)	0.4480(26)	0.5837(43)	6.9(13)
H(3)	0.0769(3)	0.1928(2)	0.8443(4)	5.4
H(4)	0.1631(3)	0.0895(2)	0.6920(5)	5.7
H(5)	0.3245(3)	0.1211(2)	0.5247(4)	5.0
H(7)	0.4764(3)	0.2104(2)	0.4203(3)	3.8
H(8A)	0.6160(3)	0.2648(2)	0.2907(3)	4.1
H(8B)	0.6773(3)	0.3410(2)	0.3763(3)	4.1
H(10A)	0.6755(3)	0.4793(2)	0.2644(4)	4.5
H(10B)	0.6017(3)	0.4970(2)	0.1118(4)	4.5
H(13)	0.0911(3)	0.7707(2)	0.4620(4)	5.0
H(14)	0.2193(4)	0.8095(2)	0.2606(4)	5.9
H(15)	0.3748(3)	0.7202(2)	0.1715(3)	5.1
H(17)	0.4974(3)	0.6008(2)	0.1793(3)	3.9
H(18A)	0.0187(4)	0.3838(3)	1.0056(4)	7.0
H(18B)	0.0957(4)	0.3002(3)	1.0299(4)	7.0
H(18C)	-0.0279(4)	0.3031(3)	0.9212(4)	7.0
H(19A)	0.4763(3)	0.2905(2)	0.0579(3)	5.2
H(19B)	0.4758(3)	0.3786(2)	-0.0177(3)	5.2
H(19C)	0.3946(3)	0.3632(2)	0.1279(3)	5.2
H(20A)	0.7949(4)	0.3716(2)	0.1502(4)	6.2
H(20B)	0.7206(4)	0.3868(2)	-0.0036(4)	6.2
H(20C)	0.7250(4)	0.2974(2)	0.0667(4)	6.2
H(21A)	-0.0360(4)	0.6592(2)	0.7584(4)	6.4
H(21B)	-0.0571(4)	0.6961(2)	0.5967(4)	6.4
H(21C)	0.0458(4)	0.7368(2)	0.7083(4)	6.4

Table S3. Final anisotropic displacement parameters ($Å^2$ x 100).

Atom	U11	U22	U33	U12	U13	U23
Gd	3.39(1)	3.21(1)	2.74(1)	0.59(1)	0.57(0)	-0.05(1)
Ni	2.74(1)	2.56(1)	2.82(1)	0.24(1)	0.55(1)	0.13(1)
O(1)	3.29(9)	2.51(8)	3.27(9)	0.04(7)	0.88(7)	0.15(7)
O(2)	3.8(1)	2.78(8)	2.87(9)	0.78(7)	0.59(7)	0.44(7)
O(3)	5.2(1)	4.0(1)	4.5(1)	-0.39(9)	2.4(1)	0.08(9)
O(4)	4.8(1)	4.1(1)	3.8(1)	2.00(9)	0.75(9)	0.19(8)
O(5)	4.30(9)	4.7(1)	3.57(8)	1.3(1)	0.63(7)	0.2(1)
O(6)	7.3(2)	10.3(2)	3.8(1)	4.7(2)	0.4(1)	0.5(1)
O(7)	7.2(2)	9.5(2)	3.4(1)	3.1(2)	1.0(1)	-0.8(1)
O(8)	5.0(1)	3.9(1)	3.64(9)	-0.2(1)	0.20(7)	-0.13(9)
O(9)	7.2(2)	3.8(1)	5.5(1)	0.3(1)	-0.3(1)	-0.2(1)
O(10)	7.8(2)	7.0(2)	8.4(2)	-3.2(2)	-1.6(2)	0.2(2)
O(11)	3.2(1)	8.2(2)	4.7(1)	0.4(1)	0.33(9)	-0.7(1)
O(12)	5.5(1)	7.4(2)	6.4(1)	0.6(1)	2.2(1)	-0.6(2)
O(13)	3.4(1)	7.3(2)	9.4(2)	-0.9(1)	-0.7(1)	-0.3(2)
O(14)	3.5(1)	4.6(1)	3.4(1)	0.37(8)	0.14(8)	-0.08(8)
O(15)	3.3(1)	8.0(2)	4.2(1)	-0.1(1)	0.45(9)	-1.1(1)
N(1)	3.1(1)	3.2(1)	3.0(1)	0.54(8)	0.38(8)	0.08(9)
N(2)	3.55(9)	3.21(9)	3.14(9)	0.0(1)	0.71(7)	0.0(1)
N(3)	4.1(1)	5.7(2)	3.4(1)	1.0(1)	0.2(1)	-0.4(1)
N(4)	6.0(2)	4.4(1)	3.0(1)	-1.0(1)	-0.2(1)	0.2(1)
N(5)	3.6(1)	4.0(1)	6.0(2)	-0.1(1)	0.6(1)	0.2(1)
C(1)	2.8(1)	2.7(1)	3.4(1)	0.11(9)	0.1(1)	0.6(1)
C(2)	3.5(1)	3.3(1)	4.0(2)	0.1(1)	0.8(1)	0.3(1)
C(3)	4.5(2)	4.2(2)	6.0(2)	-0.5(1)	1.7(1)	1.2(1)
C(4)	4.7(2)	3.1(1)	7.9(2)	-0.3(1)	1.2(2)	0.8(2)
C(5)	4.6(2)	2.8(1)	6.3(2)	0.5(1)	0.4(1)	0.2(1)
C(6)	3.3(1)	2.7(1)	4.0(1)	0.4(1)	0.3(1)	0.4(1)
C(7)	3.6(1)	3.0(1)	3.8(1)	0.7(1)	0.2(1)	-0.2(1)
C(8)	3.5(1)	3.9(1)	3.8(1)	1.0(1)	0.8(1)	-0.1(1)
C(9)	3.9(1)	4.1(1)	3.4(1)	0.6(1)	1.3(1)	0.0(1)
C(10)	4.0(2)	4.1(1)	4.3(2)	-0.1(1)	1.6(1)	0.3(1)
C(11)	3.4(1)	2.6(1)	3.0(1)	0.27(9)	-0.3(1)	0.07(9)
C(12)	4.0(1)	3.2(1)	3.3(1)	0.4(1)	-0.4(1)	-0.3(1)
C(13)	5.2(2)	3.7(2)	4.6(2)	1.5(1)	-0.3(1)	-0.1(1)
C(14)	7.5(2)	3.4(2)	5.2(2)	1.2(2)	-0.2(2)	1.2(1)
C(15)	6.1(2)	3.6(2)	4.4(2)	0.4(1)	0.6(1)	1.2(1)
C(16)	3.9(1)	2.9(1)	3.3(1)	0.2(1)	-0.2(1)	0.3(1)
C(17)	4.1(1)	3.5(1)	3.0(1)	-0.4(1)	0.5(1)	0.5(1)
C(18)	7.0(2)	6.6(2)	5.7(2)	-2.2(2)	3.7(2)	-0.9(2)
C(19)	5.6(2)	4.7(2)	3.7(2)	0.8(1)	0.1(1)	-0.3(1)
C(20)	5.5(2)	6.0(2)	5.3(2)	1.3(2)	2.5(2)	0.0(2)
C(21)	5.9(2)	6.1(2)	5.5(2)	3.4(2)	1.0(2)	0.1(2)

Table S4. Bond lengths (Å) and angles (deg) with e.s.d.'s in parentheses.

Gd-O(1)	2.354(2)	Gd-O(6)	2.556(2)
Gd-O(2)	2.319(2)	Gd-O(8)	2.492(2)
Gd-O(3)	2.550(2)	Gd-O(9)	2.512(2)
Gd-O(4)	2.541(2)	Gd-O(11)	2.492(2)
Gd-O(5)	2.528(2)	Gd-O(12)	2.614(2)
Ni-O(1)	2.035(2)	Ni-N(1)	2.006(2)
Ni-O(2)	2.032(2)	Ni-N(2)	2.027(2)
Ni-O(14)	2.163(2)	Ni-O(15)	2.138(2)
O(1)-C(1)	1.320(3)	O(2)-C(11)	1.325(3)
C(1)-C(2)	1.412(3)	C(11)-C(12)	1.413(4)
C(2)-C(3)	1.372(4)	C(12)-C(13)	1.368(4)
C(3)-C(4)	1.392(5)	C(13)-C(14)	1.389(5)
C(4)-C(5)	1.368(5)	C(14)-C(15)	1.363(5)
C(5)-C(6)	1.411(4)	C(15)-C(16)	1.417(4)
C(6)-C(1)	1.410(4)	C(16)-C(11)	1.401(4)
C(2)-O(3)	1.390(3)	C(12)-O(4)	1.394(4)
O(3)-C(18)	1.435(3)	O(4)-C(21)	1.437(3)
C(6)-C(7)	1.458(4)	C(16)-C(17)	1.449(4)
C(7)-N(1)	1.275(3)	C(17)-N(2)	1.275(4)
N(1)-C(8)	1.466(3)	N(2)-C(10)	1.476(3)
C(8)-C(9)	1.534(4)	C(9)-C(10)	1.531(4)
C(9)-C(19)	1.517(4)	C(9)-C(20)	1.532(4)
O(14)-H(14A)	0.92(2)	O(15)-H(15A)	0.81(2)
O(14)-H(14B)	0.84(2)	O(15)-H(15B)	0.86(2)
N(3)-O(5)	1.265(3)	N(4)-O(8)	1.272(4)
N(3)-O(6)	1.265(3)	N(4)-O(9)	1.255(4)
N(3)-O(7)	1.209(3)	N(4)-O(10)	1.214(4)
N(5)-O(11)	1.251(3)	N(5)-O(12)	1.256(4)
N(5)-O(13)	1.220(4)		

Shortest intra- and intermolecular Metal-Metal distances

Gd...Ni	3.5213(3)	Gd...Ni'''	6.7115(3)
Gd...Gd''	8.8948(1)	Ni...Ni(i)	8.3065(1)
Gd...Gd'''		Ni...Ni(ii)	

Symmetry operations:

'	1 + X	Y	Z
''	X	Y	-1 + Z
'''	X	Y	1 + Z
i	1 - X	1/2 + Y	1 - Z
ii	1 - X	-1/2 + Y	1 - Z

Table S4. (Continued)

O(1)-Gd-O(2)	66.90(6)	O(3)-Gd-O(11)	75.59(8)
O(1)-Gd-O(3)	63.40(6)	O(3)-Gd-O(12)	72.88(9)
O(1)-Gd-O(4)	127.60(6)	O(4)-Gd-O(5)	120.48(7)
O(1)-Gd-O(5)	110.57(7)	O(4)-Gd-O(6)	74.59(8)
O(1)-Gd-O(6)	157.19(8)	O(4)-Gd-O(8)	113.06(8)
O(1)-Gd-O(8)	72.82(7)	O(4)-Gd-O(9)	68.72(8)
O(1)-Gd-O(9)	119.09(8)	O(4)-Gd-O(11)	77.33(8)
O(1)-Gd-O(11)	73.20(7)	O(4)-Gd-O(12)	72.79(9)
O(1)-Gd-O(12)	113.98(8)	O(5)-Gd-O(6)	49.95(7)
O(2)-Gd-O(3)	127.81(6)	O(5)-Gd-O(8)	70.25(6)
O(2)-Gd-O(4)	64.44(6)	O(5)-Gd-O(9)	73.56(8)
O(2)-Gd-O(5)	146.24(7)	O(5)-Gd-O(11)	137.16(8)
O(2)-Gd-O(6)	135.62(9)	O(5)-Gd-O(12)	96.29(7)
O(2)-Gd-O(8)	77.32(7)	O(6)-Gd-O(8)	105.29(8)
O(2)-Gd-O(9)	78.90(8)	O(6)-Gd-O(9)	70.73(11)
O(2)-Gd-O(11)	75.95(7)	O(6)-Gd-O(11)	111.83(8)
O(2)-Gd-O(12)	115.84(8)	O(6)-Gd-O(12)	63.70(9)
O(3)-Gd-O(4)	145.10(8)	O(8)-Gd-O(9)	50.80(8)
O(3)-Gd-O(5)	69.61(8)	O(8)-Gd-O(11)	142.87(7)
O(3)-Gd-O(6)	95.59(9)	O(8)-Gd-O(12)	166.53(7)
O(3)-Gd-O(8)	101.82(8)	O(9)-Gd-O(11)	144.02(9)
O(3)-Gd-O(9)	140.33(8)	O(9)-Gd-O(12)	126.18(9)
O(11)-Gd-O(12)	48.90(8)		
O(1)-Ni-O(2)	78.59(7)	O(2)-Ni-O(14)	87.85(8)
O(1)-Ni-N(1)	92.25(8)	O(2)-Ni-O(15)	90.54(9)
O(1)-Ni-N(2)	171.26(10)	N(1)-Ni-N(2)	96.49(10)
O(1)-Ni-O(14)	88.66(8)	N(1)-Ni-O(14)	90.07(8)
O(1)-Ni-O(15)	91.73(8)	N(1)-Ni-O(15)	91.64(9)
O(2)-Ni-N(1)	170.65(8)	N(2)-Ni-O(14)	91.29(8)
O(2)-Ni-N(2)	92.68(9)	N(2)-Ni-O(15)	88.06(9)
O(14)-Ni-O(15)	178.23(9)		
Gd-O(1)-Ni	106.49(7)	Gd-O(2)-Ni	107.90(7)
Gd-O(1)-C(1)	125.2(2)	Gd-O(2)-C(11)	126.1(2)
Ni-O(1)-C(1)	122.4(2)	Ni-O(2)-C(11)	124.5(2)
O(1)-C(1)-C(6)	124.7(2)	O(2)-C(11)-C(16)	123.9(2)
O(1)-C(1)-C(2)	118.3(2)	O(2)-C(11)-C(12)	118.0(2)
C(6)-C(1)-C(2)	117.1(2)	C(16)-C(11)-C(12)	118.1(2)
C(1)-C(2)-C(3)	122.6(3)	C(11)-C(12)-C(13)	121.6(3)
C(1)-C(2)-O(3)	113.0(2)	C(11)-C(12)-O(4)	113.6(2)
C(3)-C(2)-O(3)	124.4(3)	C(13)-C(12)-O(4)	124.8(3)
C(2)-O(3)-C(18)	115.8(2)	C(12)-O(4)-C(21)	116.2(2)
Gd-O(3)-C(2)	118.2(2)	Gd-O(4)-C(12)	117.3(2)
Gd-O(3)-C(18)	126.0(2)	Gd-O(4)-C(21)	125.6(2)
C(2)-C(3)-C(4)	119.4(3)	C(12)-C(13)-C(14)	120.1(3)
C(3)-C(4)-C(5)	120.2(3)	C(13)-C(14)-C(15)	119.8(3)
C(4)-C(5)-C(6)	121.1(3)	C(14)-C(15)-C(16)	121.4(3)
C(5)-C(6)-C(1)	119.7(3)	C(15)-C(16)-C(11)	118.9(3)

Table S4. (Continued)

C(5)-C(6)-C(7)	116.2(2)	C(15)-C(16)-C(17)	115.8(2)
C(1)-C(6)-C(7)	124.0(2)	C(11)-C(16)-C(17)	125.3(2)
C(6)-C(7)-N(1)	127.6(2)	C(16)-C(17)-N(2)	128.1(2)
C(7)-N(1)-C(8)	116.7(2)	C(17)-N(2)-C(10)	115.7(2)
Ni-N(1)-C(7)	122.5(2)	Ni-N(2)-C(17)	123.1(2)
Ni-N(1)-C(8)	120.7(2)	Ni-N(2)-C(10)	120.7(2)
N(1)-C(8)-C(9)	114.9(2)	N(2)-C(10)-C(9)	116.7(2)
C(8)-C(9)-C(10)	111.3(2)	C(10)-C(9)-C(19)	112.4(3)
C(8)-C(9)-C(19)	111.2(2)	C(10)-C(9)-C(20)	105.8(3)
C(8)-C(9)-C(20)	105.8(2)	C(19)-C(9)-C(20)	110.0(3)
Ni-O(14)-H(14A)	111(2)	Ni-O(15)-H(15A)	109(3)
Ni-O(14)-H(14B)	119(3)	Ni-O(15)-H(15B)	120(3)
H(14A)-O(14)-H(14B)	98(3)	H(15A)-O(15)-H(15B)	114(3)
Gd-O(5)-N(3)	97.6(2)	Gd-O(8)-N(4)	96.5(2)
Gd-O(6)-N(3)	96.2(2)	Gd-O(9)-N(4)	96.0(2)
Gd-O(11)-N(5)	100.9(2)	Gd-O(12)-N(5)	94.7(2)
O(5)-N(3)-O(6)	116.0(2)	O(8)-N(4)-O(9)	116.2(3)
O(5)-N(3)-O(7)	121.8(3)	O(8)-N(4)-O(10)	121.3(3)
O(6)-N(3)-O(7)	122.1(3)	O(9)-N(4)-O(10)	122.5(3)
O(11)-N(5)-O(12)	115.1(3)	O(11)-N(5)-O(13)	121.0(3)
O(12)-N(5)-O(13)	123.9(3)		

Hydrogen bonds

D	H	D-H	A	H...A	D...A	D-H...O
O(14)	H(14A)	0.92(2)	O(5'')	2.02(2)	2.927(3)	169(3)
	H(14B)	0.84(2)	O(11)	2.04(3)	2.775(3)	146(3)
O(15)	H(15A)	0.81(2)	O(8)	1.97(2)	2.703(3)	151(3)
	H(15B)	0.86(2)	O(13')	2.08(3)	2.868(3)	153(4)

Table S5. Least-squares planes equations and deviations (Å) therefrom.

Plane 1: - 0.7716 X - 0.2397 Y - 0.5893 Z + 7.1644 = 0
Chi Squared = 353.9

O(1)	-0.019(2)
O(2)	0.019(2)
N(1)	0.016(2)
N(2)	-0.016(2)
Ni	-0.0155(3)*
O(14)	2.146(2)*
O(15)	-2.152(2)*

Plane 2: - 0.7840 X - 0.2143 Y - 0.5826 Z + 7.0237 = 0

Gd	0.0000(1)
O(1)	0.000(2)
Ni	0.0000(3)

Plane 3: - 0.7827 X - 0.2818 Y - 0.5550 Z + 7.3797 = 0

Gd	0.0000(1)
O(2)	0.000(2)
Ni	0.0000(3)

Plane 4: - 0.7986 X - 0.2410 Y - 0.5515 Z + 7.0576 = 0

Gd	0.0000(1)
O(1)	0.000(2)
O(2)	0.000(2)

Plane 5: - 0.7650 X - 0.2560 Y - 0.5909 Z + 7.2752 = 0

Ni	0.0000(3)
O(1)	0.000(2)
O(2)	0.000(2)

Plane 6: - 0.7790 X - 0.2264 Y - 0.5847 Z + 7.0976 = 0

Ni	0.0000(3)
N(1)	0.000(2)
N(2)	0.000(2)
C(8)	-0.430(3)*
C(9)	0.182(3)*
C(10)	-0.352(3)*
C(19)	1.698(3)*
C(20)	-0.333(4)*

Table S5. (Continued)

Plane 7: - 0.7036 X + 0.1462 Y - 0.6954 Z + 5.1875 = 0
Chi Squared = 50.4

C(1)	0.011(3)
C(2)	-0.005(3)
C(3)	-0.008(3)
C(4)	0.014(4)
C(5)	-0.007(3)
C(6)	-0.006(3)

Plane 8: - 0.6630 X - 0.4390 Y - 0.6064 Z + 8.5127 = 0
Chi Squared = 245.8

C(11)	-0.029(3)
C(12)	0.014(3)
C(13)	0.009(3)
C(14)	-0.018(4)
C(15)	-0.003(3)
C(16)	0.020(3)

* denote atoms not included in planes calculations.

Dihedral angle (deg) between:

Plane	2	and	Plane	3	=	4.2(5)
Plane	4	and	Plane	5	=	3.1(8)