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Crystal and Molecular Structure of CCDC 932753

C8H18N2NiO6

Abstract. "IUPAC-name", $C_8H_{18}N_2NiO_6$, M_r = 296.93, monoclinic, $P2_1$, a = 7.5532(5), b = 9.1933(6), c = 8.2722(5) Å, β = 100.225(1)°, V = 565.29(6) Å^3, Z = 2, D_x = 1.773 gcm^{-3}, F(000) = 312, μ = 17.35 cm^{-1}, λ(MoK$\overline{\alpha}$) = 0.71073 Å, T = 100(1) K, 5182 reflections measured, *GooF* = 1.055, *wR*(F^2) = 0.0689 for 2671 unique reflections and 226 parameters, 1 restraints and *R*(*F*) = 0.0270 for 2596 reflections obeying $F_o \geq 4.0\ \sigma(F_o)$ criterion of observability.

The asymmetric unit consists of one molecule of the title compound.

Experimental

X-ray diffraction: Crystal and Molecular Structure.

Beautiful small green-colored block-shaped crystals were obtained by recrystallisation from …..

A crystal with the dimensions of 0.14 x 0.12 x 0.09 mm was mounted on top of a glass fiber and aligned on a *Bruker*[1] *SMART APEX CCD* diffractometer (Platform with full three-circle goniometer). The diffractometer was equipped with a 4K *CCD* detector set 60.0 mm from the crystal. The crystal was cooled to 100(1) K using the *Bruker KRYOFLEX* low-temperature device. Intensity measurements were performed using graphite monochromated Mo-K$\overline{\alpha}$ radiation from a sealed ceramic diffraction tube (*SIEMENS*). Generator settings were 50 KV/ 40 mA. *SMART* was used for preliminary determination of the unit cell constants and data collection control. The intensities of reflections of a hemisphere were collected by a combination of 3 sets of exposures (frames). Each set had a different ϕ angle for the crystal and each exposure covered a range of 0.3° in ω. A total of 1800 frames were collected with an exposure time of 20.0 seconds per frame. The overall data collection time was 13.0 h. Data integration and global cell refinement was performed with the program *SAINT*. The final unit cell was obtained from the *xyz* centroids of 3992 reflections after integration. Intensity data were corrected for Lorentz and polarization effects, scale variation, for decay and absorption: a multi-scan absorption correction was applied, based on the intensities of symmetry-related reflections measured at different angular settings (*SADABS*)[2], and reduced to F_o^2. The program suite *SAINTPLUS* was used for space group determination (*XPREP*).[1]

The unit cell[3] was identified as monoclinic. reduced cell calculations did not indicate any higher metric lattice symmetry.[4] Space group $P2_1$, was derived from the systematic extinctions. The $|E|$ distribution statistics were indicative of a non-centrosymmetric space group.[5] and examination of the final atomic coordinates of the structure did not yield obvious extra crystallographic or metric symmetry elements.[6,7]

The structure was solved by direct methods using the program *SIR2004.*[8] The positional and anisotropic displacement parameters for the non-hydrogen atoms were refined. A subsequent difference Fourier synthesis resulted in the location of all the hydrogen atoms, which coordinates and isotropic displacement parameters were refined.

Final refinement on F^2 carried out by full-matrix least-squares techniques converged at $wR(F^2)$ = 0.0689 for 2671 reflections and $R(F)$ = 0.0270 for 2596 reflections with $F_o \geq 4.0\ \sigma(F_o)$ and 226 parameters and 1 restraints. The final difference Fourier map was essentially featureless: no significant peaks (0.47(6) e/Å^3) having chemical meaning above the general background were observed.

The absolute structure of the molecule actually chosen was determined by Flack's[9,10,11,12] refinement (**x** = -0.009(11)).

The positional and anisotropic displacement parameters for the non-hydrogen atoms and isotropic displacement parameters for hydrogen atoms were refined on F^2 with full-matrix least-squares procedures minimizing the function $Q = \sum_h[w(|(F_o^2) - k(F_c^2)|)^2]$, where $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$, $P = [\max(F_o^2,0) + 2F_c^2] / 3$, F_0 and F_c are the observed and calculated structure factor amplitudes, respectively; ultimately the suggested *a* (=0.0483) and *b* (= 0.0955) were used in the final refinement.

Crystal data and numerical details on data collection and refinement are given in Table 1. Final fractional atomic coordinates, equivalent displacement parameters and anisotropic displacement parameters for the non-hydrogen atoms are given in Table 2. Molecular geometry data are collected in Table 3. Tables of atom positions, displacement parameters, comprehensive distances and angles and tables of (F_o^2), (F_c^2) and $\sigma(F_o^2)$ are given as supplementary material[*1] for this paper. Neutral atom scattering factors and anomalous dispersion corrections were taken from *International Tables for Crystallography.*[14]

All refinement calculations and graphics were performed on a HP XW6200 (Intel XEON 3.2 Ghz) / Debian-Linux computer at the University of Groningen with the program packages *SHELXL*[14] (least-square refinements), a locally modified version of the program *PLUTO*[16] (preparation of illustrations) and *PLATON*[17] package (checking the final results for missed symmetry with the *MISSYM* option, solvent accessible voids with the *SOLV* option, calculation of geometric data and the *ORTEP*[17] illustrations).

[*1] Supplementary crystallographic data for this paper are available from the IUCr electronic archives (Reference: CCDC:932753, as a CIF[13] file). These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk).

Results and discussion.

The asymmetric unit contains one formula unit molecule with no atom setting at special position. The monoclinic unit cell contains two formula units of the title compound. The chiral centers of N1 and N2 showed both the *R*-configuration and the chiral centers of C2 and C7 showed both the *S*-configuration.[17]

A search of the distances yielded intermolecular- and intramolecular-contacts shorter than the sum of the van der Waals radii[18] for the atoms: the moieties are linked by hydrogen bonds[18,19,20,21] (Table **4**.), forming an infinite three-dimensional network along the base vectors.

No missed symmetry (*MISSYM*) or solvent-accessible voids were detected by procedures implemented in *PLATON.*[22,23]

References.

1. Bruker (2000). *SMART*, *SAINTPLUS* and *XPREP. Area Detector Control and Integration Software.* Smart Apex *Software Reference Manuals*. Bruker Analytical X-ray Instruments. Inc., Madison, Wisconsin, USA.
2. Sheldrick, G.M. (2001). *SADABS*. Version 2. *Multi-Scan Absorption Correction Program*. University of Göttingen, Germany.
3. Duisenberg, A. J. M. (1992). J. Appl. Cryst. **25**, 92-96.
4. Spek, A.L. (1988). J. Appl. Cryst. **21**, 578-579.
5. Snow, M.R. & Tiekink, E.R.T. (1988). Acta Cryst. B**44**, 676-677
6. Le Page, Y. (1987). J. Appl. Cryst. **20**, 264-269.
7. Le Page, Y. (1988). J. Appl. Cryst. **21**, 983-984.
8. Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G.L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. *SIR2004*: an improved tool for crystal structure determination and refinement. J. Appl. Cryst. (2005). **38**, 381-388.
9. Flack, H.D. (1983). Acta. Cryst. A**39**, 876-881.
10. Flack, H.D. & Bernardinelli, G. (1999). Acta Cryst. A**55**, 908-915.
11. Flack, H.D. & Bernardinelli, G. (2000). J. Appl. Cryst. **33**, 1143-1148.
12. Herbst-Irmer, R. & Sheldrick, G.M. (1998). Acta Cryst. B**54**, 443-449.
13. Hall, S.R, Allen, F.H. & Brown, I.D. (1991). Acta Cryst. A**47**, 655-685.
14. Wilson, A.J.C. (1992) Ed. *International Tables for Crystallography*, Volume C, Kluwer Academic Publishers, Dordrecht, The Netherlands.
15. Sheldrick, G.M. (1997). *SHELXL-97*. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
16. Meetsma, A. (2005*). PLUTO. Molecular Graphics Program*. Version of January 2006. University of Groningen, The Netherlands.

17. Spek, A.L. (2005). *PLATON*. *Program for the Automated Analysis of Molecular Geometry* (*A Multipurpose Crystallographic Tool*). Version of August 2005. University of Utrecht, The Netherlands. Spek, A.L. (2003). J.Appl. Cryst. **36**, 7-13.
18. Bondi, A. (1964). J. Phys. Chem. **68**, 441-451.
19. Berkovitch-Yellin, Z. & Leiserowitz, L. (1984). Acta Cryst. B**40**, 159-165.
20. Steiner, Th. (1996). Cryst. Rev., **6**,1-57.
21. Jeffrey, G.A., Maluszynska, M. & Mitra, J. (1985). Int. J. Biol. Macromol. **7**, 336-348.
22. Spek, A.L. (1990). Acta Cryst. A**46**, C-34.
23. Spek, A.L. (1994). Am. Crystallogr. Assoc. Abstr. **22**, 66.
24. Hahn, T. (1983) Ed. *International Tables for Crystallography*, Volume A, Space-group symmetry, Kluwer Academic Publishers, Dordrecht, The Netherlands.
25. Fisher, R.X. & Tillmanns, E. (1988). Acta Cryst. C**44**, 775-776.

Table 1.

a. Crystal data and details of the structure determination.

Moiety_Formula	$C_8H_{18}N_2NiO_6$
Formula_Weight, g.mol^{-1}	296.93
Crystal system	monoclinic
Space group, no.[24]	$P2_1$, 4
a, Å	7.5532(5)
b, Å	9.1933(6)
c, Å	8.2722(5)
β, deg	100.225(1)
V, Å^3	565.29(6)
Θ range unit cell: min.-max., deg; reflections	2.50 - 29.75 ; 3992
Formula_*Z*	2
SpaceGroup_*Z*	2
Z' (= Formula_Z / SpaceGroup_*Z)*	1
ρ_{calc}, g.cm^{-3}	1.774
F(000), electrons	312
μ(Mo K$\overline{\alpha}$), cm^{-1}	17.35
Color, habit	green, block
Approx. crystal dimension, mm	0.14 x 0.12 x 0.09

b. Data collection.

Radiation type; λ, Å	Mo K$\overline{\alpha}$, 0.71073
Monochromator	Graphite
Measurement device type	Bruker SMART APEX
	CCD area-detector diffractometer
Detector Area resolution (pixels / mm)	4096 x 4096 / 62 x 62 (binned 512)
Temperature, K	100(1)
Measurement method	φ- and ω-scans
θ range; min. max., deg	2.74, 28.27
Index ranges	h: -10→10; k: -11→12; l: -11→11
Min.- Max. absorption transmission factor	0.7795 – 0.8595
X-ray exposure time, h	13.0
Total data	5182
Unique data	2671
Data with criterion: ($F_o \geq 4.0\ \sigma\ (F_o)$)	2596
$R_{int} = \sum [\lvert F_o^2 - F_o^2(\text{mean})\rvert] / \sum [F_o^2]$	0.0155
$R_{sig} = \sum \sigma(F_o^2) / \sum [F_o^2]$	0.0274

c. Refinement.

Number of reflections	2671
Number of refined parameters	226
Number of restraints	1
Final agreement factors:	
$wR(F^2) = [\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]]^{1/2}$	0.0689
Weighting scheme: *a, b*	0.0483, 0.0955
$w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$	
And $P = [\max(F_o^2,0) + 2F_c^2] / 3$	
$R(F) = \sum (\|\|F_o\| - \|F_c\|\|) / \sum \|F_o\|$	0.0270
For $F_o > 4.0\ \sigma(F_o)$	
Absolute-Structure parameter Flack's **x**	-0.009(11)
GooF = $S = [\sum [w(F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$	1.055
n = number of reflections	
p = number of parameters refined	
Residual electron density in final	
Difference Fourier map, e/Å^3	-0.33, 0.47(6)
Max. (shift/σ) final cycle	<0.001
Mean. (shift/σ) final cycle	<0.001

Table 2. Final fractional atomic coordinates and equivalent isotropic displacement parameters with s.u.'s in parentheses.

Atoms of the Asymmetric Unit.

Non-Hydrogen parameters

Atom	*x*	*y*	*z*	U_{eq} (Å^2)*
Ni	0.22589(3)	0.26991(3)	0.36142(3)	0.00845(7)
O1	0.4035(2)	0.10655(19)	0.3335(2)	0.0123(5)
O2	0.6792(2)	0.06366(19)	0.2817(2)	0.0126(4)
O3	0.0484(2)	0.4429(2)	0.3559(2)	0.0113(5)
O4	-0.1203(2)	0.59987(19)	0.1877(2)	0.0130(4)
O5	0.0335(3)	0.1089(2)	0.3707(3)	0.0129(5)
O6	0.28236(19)	0.2710(4)	0.61566(17)	0.0126(4)
N1	0.4586(3)	0.3995(2)	0.3544(3)	0.0102(6)
N2	0.1701(3)	0.3048(2)	0.1043(2)	0.0093(5)
C1	0.5616(3)	0.1461(3)	0.3235(3)	0.0103(6)
C2	0.6172(3)	0.3013(2)	0.3758(3)	0.0093(6)
C3	0.7073(3)	0.2928(3)	0.5574(3)	0.0124(6)
C4	0.4257(3)	0.4713(3)	0.1917(3)	0.0109(6)
C5	0.3397(3)	0.3631(3)	0.0611(3)	0.0113(6)
C6	-0.0197(3)	0.4908(3)	0.2136(3)	0.0105(6)
C7	0.0162(3)	0.4066(3)	0.0619(3)	0.0108(6)
C8	-0.1550(3)	0.3249(3)	-0.0136(3)	0.0154(6)

Hydrogen parameters:

H2	0.703(4)	0.339(3)	0.313(4)	0.006(6)
H3	0.614(4)	0.257(5)	0.621(3)	0.018(7)
H3'	0.801(5)	0.227(4)	0.566(5)	0.036(11)
H3"	0.752(5)	0.373(5)	0.593(5)	0.024(9)
H4	0.346(4)	0.548(3)	0.203(3)	0.006(6)
H4'	0.530(4)	0.510(4)	0.162(4)	0.020(8)
H5	0.423(3)	0.284(4)	0.057(3)	0.016(7)
H5'	0.318(4)	0.420(3)	-0.051(4)	0.011(7)
H7	0.044(4)	0.472(3)	-0.019(4)	0.012(7)
H8	-0.184(3)	0.253(4)	0.057(3)	0.010(6)
H8'	-0.141(3)	0.273(5)	-0.110(3)	0.019(6)
H8"	-0.245(4)	0.391(4)	-0.050(4)	0.021(8)
H11	0.468(4)	0.458(4)	0.429(4)	0.024(9)
H12	0.154(4)	0.230(4)	0.046(4)	0.018(9)
H13	0.027(4)	0.065(4)	0.456(4)	0.017(8)
H13'	-0.067(5)	0.109(4)	0.328(5)	0.032(10)
H14	0.223(5)	0.219(4)	0.677(4)	0.022(9)
H14'	0.294(4)	0.338(4)	0.647(3)	0.008(8)

*) $U_{eq} = 1/3 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a_i}.\mathbf{a_j}$ [25]

Anisotropic (displacement) parameters (Å^2)

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Ni	0.00888(12)	0.00866(13)	0.00823(12)	0.00078(12)	0.00266(8)	0.00051(11)
O1	0.0114(8)	0.0093(9)	0.0165(8)	0.0011(7)	0.0036(7)	0.0003(6)
O2	0.0116(7)	0.0126(8)	0.0137(7)	-0.0023(6)	0.0028(6)	0.0010(6)
O3	0.0121(8)	0.0122(9)	0.0097(8)	0.0000(7)	0.0020(6)	0.0008(7)
O4	0.0144(7)	0.0117(8)	0.0134(7)	-0.0003(6)	0.0035(6)	0.0039(6)
O5	0.0092(8)	0.0145(10)	0.0150(9)	0.0035(7)	0.0023(7)	-0.0016(7)
O6	0.0169(7)	0.0117(8)	0.0098(6)	-0.0008(10)	0.0040(5)	-0.0023(9)
N1	0.0132(10)	0.010(1)	0.0073(9)	-0.0001(7)	0.0018(7)	0.0014(7)
N2	0.0103(8)	0.0087(11)	0.0090(8)	-0.0011(6)	0.0017(7)	0.0005(6)
C1	0.0116(9)	0.0113(11)	0.0075(9)	0.0022(8)	0.0001(8)	-0.0001(8)
C2	0.0089(8)	0.0079(12)	0.0116(9)	-0.0002(7)	0.0030(7)	0.0001(7)
C3	0.0127(9)	0.0111(14)	0.0123(9)	-0.0021(8)	-0.0004(7)	0.0004(8)
C4	0.012(1)	0.0102(11)	0.0105(9)	0.0010(8)	0.0021(8)	0.0000(8)
C5	0.0109(9)	0.0128(11)	0.0106(9)	0.0001(8)	0.0031(8)	-0.0001(8)
C6	0.0104(9)	0.0092(10)	0.0125(10)	-0.0012(8)	0.0041(8)	-0.0020(8)
C7	0.0113(9)	0.0117(11)	0.0093(9)	0.0001(8)	0.0019(7)	0.0020(7)
C8	0.0138(10)	0.0181(12)	0.0134(10)	-0.0043(9)	-0.0004(9)	0.0021(8)

Thermal vibration amplitudes (Å^2)

$$F(\mathbf{h}) = F_o(\mathbf{h}) \exp\left(-2\pi^2 \sum_{i=1}^{3}\sum_{j=1}^{3} h_i h_j a_i^* a_j^* U_{ij}\right)$$

or

$$F(\mathbf{h}) = F_o(\mathbf{h}) \exp\left(-8\pi^2 U_{iso} (\sin(\theta)/\lambda)^2\right)$$

Table 3. Selected data on the geometry.

Standard deviations in the last decimal place are given in parentheses.

Interatomic Distances (Å)

Ni	-O1	2.0539(17)	N1	-C2	1.485(3)
Ni	-O3	2.0753(17)	N1	-C4	1.480(3)
Ni	-O5	2.085(2)	N2	-C5	1.490(3)
Ni	-O6	2.0704(14)	N2	-C7	1.485(3)
Ni	-N1	2.133(2)	C1	-C2	1.528(3)
Ni	-N2	2.1181(17)	C2	-C3	1.537(3)
O1	-C1	1.265(3)	C4	-C5	1.526(4)
O2	-C1	1.262(3)	C6	-C7	1.539(4)
O3	-C6	1.276(3)	C7	-C8	1.530(3)
O4	-C6	1.253(3)			

Hydrogen parameters:

O5	-H13	0.82(3)	C3	-H3"	0.84(5)
O5	-H13'	0.78(4)	C4	-H4	0.94(3)
O6	-H14	0.88(4)	C4	-H4'	0.94(3)
O6	-H14'	0.67(4)	C5	-H5	0.97(3)
N1	-H11	0.81(4)	C5	-H5'	1.05(3)
N2	-H12	0.84(4)	C7	-H7	0.95(3)
C2	-H2	0.96(3)	C8	-H8	0.93(3)
C3	-H3	1.01(3)	C8	-H8'	0.95(3)
C3	-H3'	0.92(4)	C8	-H8"	0.92(3)

Bond Angles (deg.)

O1	-Ni	-O3	171.95(7)	C2	-N1	-C4	112.4(2)
O1	-Ni	-O5	87.62(7)	Ni	-N2	-C5	105.61(14)
O1	-Ni	-O6	95.56(9)	Ni	-N2	-C7	109.91(14)
O1	-Ni	-N1	81.23(7)	C5	-N2	-C7	112.77(18)
O1	-Ni	-N2	90.88(7)	O1	-C1	-O2	124.3(2)
O3	-Ni	-O5	95.34(7)	O1	-C1	-C2	117.7(2)
O3	-Ni	-O6	91.97(9)	O2	-C1	-C2	117.8(2)
O3	-Ni	-N1	95.95(7)	N1	-C2	-C1	111.01(19)
O3	-Ni	-N2	81.33(7)	N1	-C2	-C3	110.80(19)
O5	-Ni	-O6	89.16(10)	C1	-C2	-C3	106.32(18)
O5	-Ni	-N1	168.71(8)	N1	-C4	-C5	109.6(2)
O5	-Ni	-N2	97.40(9)	N2	-C5	-C4	109.93(19)
O6	-Ni	-N1	90.16(9)	O3	-C6	-O4	124.5(2)
O6	-Ni	-N2	171.01(11)	O3	-C6	-C7	118.6(2)
N1	-Ni	-N2	84.61(9)	O4	-C6	-C7	116.9(2)
Ni	-O1	-C1	116.12(17)	N2	-C7	-C6	111.61(19)
Ni	-O3	-C6	115.96(15)	N2	-C7	-C8	110.9(2)

Ni	-N1	-C2	107.96(13)	C6	-C7	-C8	108.75(19)
Ni	-N1	-C4	105.53(16)				

Hydrogen parameters:

Ni	-O5	-H13	122(2)	H3'	-C3	-H3''	107(4)
Ni	-O5	-H13'	128(3)	N1	-C4	-H4	104.5(15)
H13	-O5	-H13'	101(4)	N1	-C4	-H4'	113(2)
Ni	-O6	-H14	124(2)	C5	-C4	-H4	111.0(17)
Ni	-O6	-H14'	113(2)	C5	-C4	-H4'	110(2)
H14	-O6	-H14'	109(3)	H4	-C4	-H4'	109(3)
Ni	-N1	-H11	108(2)	N2	-C5	-H5	109.3(18)
C2	-N1	-H11	111(2)	N2	-C5	-H5'	112.6(17)
C4	-N1	-H11	112(2)	C4	-C5	-H5	108.5(16)
Ni	-N2	-H12	116(2)	C4	-C5	-H5'	106.3(16)
C5	-N2	-H12	102(2)	H5	-C5	-H5'	110(2)
C7	-N2	-H12	111(2)	N2	-C7	-H7	107.7(18)
N1	-C2	-H2	108.8(17)	C6	-C7	-H7	110.4(18)
C1	-C2	-H2	111.1(17)	C8	-C7	-H7	107.4(19)
C3	-C2	-H2	108.8(19)	C7	-C8	-H8	111.6(15)
C2	-C3	-H3	107.3(15)	C7	-C8	-H8'	112.1(16)
C2	-C3	-H3'	108(3)	C7	-C8	-H8''	109(2)
C2	-C3	-H3''	112(3)	H8	-C8	-H8'	104(3)
H3	-C3	-H3'	110(3)	H8	-C8	-H8''	116(3)
H3	-C3	-H3''	112(4)	H8'	-C8	-H8''	104(3)

Torsion Angles (deg.)

O5	-Ni	-O1	-C1	-178.65(18)
O6	-Ni	-O1	-C1	-89.73(18)
N1	-Ni	-O1	-C1	-0.43(17)
N2	-Ni	-O1	-C1	83.98(17)
O5	-Ni	-O3	-C6	-94.40(18)
O6	-Ni	-O3	-C6	176.26(18)
N1	-Ni	-O3	-C6	85.89(18)
N2	-Ni	-O3	-C6	2.29(17)
O1	-Ni	-N1	-C2	-13.78(16)
O1	-Ni	-N1	-C4	106.62(15)
O3	-Ni	-N1	-C2	173.82(16)
O3	-Ni	-N1	-C4	-65.78(15)
O6	-Ni	-N1	-C2	81.82(18)
O6	-Ni	-N1	-C4	-157.78(17)
N2	-Ni	-N1	-C2	-105.51(17)
N2	-Ni	-N1	-C4	14.89(15)
O1	-Ni	-N2	-C5	-67.10(15)
O1	-Ni	-N2	-C7	170.99(15)
O3	-Ni	-N2	-C5	110.88(15)
O3	-Ni	-N2	-C7	-11.04(15)

O5	-Ni	-N2	-C5	-154.81(15)
O5	-Ni	-N2	-C7	83.28(16)
N1	-Ni	-N2	-C5	14.00(15)
N1	-Ni	-N2	-C7	-107.91(16)
Ni	-O1	-C1	-O2	-168.82(19)
Ni	-O1	-C1	-C2	15.2(3)
Ni	-O3	-C6	-O4	-175.40(19)
Ni	-O3	-C6	-C7	7.3(3)
Ni	-N1	-C2	-C1	24.1(2)
Ni	-N1	-C2	-C3	-93.84(18)
C4	-N1	-C2	-C1	-91.9(2)
C4	-N1	-C2	-C3	150.2(2)
Ni	-N1	-C4	-C5	-41.2(2)
C2	-N1	-C4	-C5	76.3(2)
Ni	-N2	-C5	-C4	-40.6(2)
C7	-N2	-C5	-C4	79.5(2)
Ni	-N2	-C7	-C6	17.1(2)
Ni	-N2	-C7	-C8	-104.33(18)
C5	-N2	-C7	-C6	-100.5(2)
C5	-N2	-C7	-C8	138.1(2)
O1	-C1	-C2	-N1	-27.2(3)
O1	-C1	-C2	-C3	93.4(2)
O2	-C1	-C2	-N1	156.5(2)
O2	-C1	-C2	-C3	-82.9(3)
N1	-C4	-C5	-N2	57.2(3)
O3	-C6	-C7	-N2	-16.8(3)
O3	-C6	-C7	-C8	105.8(2)
O4	-C6	-C7	-N2	165.7(2)
O4	-C6	-C7	-C8	-71.7(3)

Hydrogen parameters:

O1	-Ni	-O5	-H13	83(3)
O1	-Ni	-O5	-H13'	-137(4)
O3	-Ni	-O5	-H13	-104(3)
O3	-Ni	-O5	-H13'	35(4)
O6	-Ni	-O5	-H13	-12(3)
O6	-Ni	-O5	-H13'	127(4)
N2	-Ni	-O5	-H13	174(3)
N2	-Ni	-O5	-H13'	-47(4)
O1	-Ni	-O6	-H14	-92(3)
O1	-Ni	-O6	-H14'	134(3)
O3	-Ni	-O6	-H14	91(3)
O3	-Ni	-O6	-H14'	-43(3)
O5	-Ni	-O6	-H14	-4(3)
O5	-Ni	-O6	-H14'	-139(3)
N1	-Ni	-O6	-H14	-173(3)
N1	-Ni	-O6	-H14'	53(3)
O1	-Ni	-N1	-H11	-134(2)
O3	-Ni	-N1	-H11	54(2)
O6	-Ni	-N1	-H11	-38(2)
N2	-Ni	-N1	-H11	135(2)
O1	-Ni	-N2	-H12	45(2)

O3	-Ni	-N2	-H12	-138(2)
O5	-Ni	-N2	-H12	-43(2)
N1	-Ni	-N2	-H12	126(2)
Ni	-N1	-C2	-H2	147(2)
C4	-N1	-C2	-H2	31(2)
H11	-N1	-C2	-C1	142(2)
H11	-N1	-C2	-C3	24(3)
H11	-N1	-C2	-H2	-95(3)
Ni	-N1	-C4	-H4	77.9(18)
Ni	-N1	-C4	-H4'	-164(2)
C2	-N1	-C4	-H4	-164.7(18)
C2	-N1	-C4	-H4'	-46(2)
H11	-N1	-C4	-C5	-159(2)
H11	-N1	-C4	-H4	-40(3)
H11	-N1	-C4	-H4'	79(3)
Ni	-N2	-C5	-H5	78.4(16)
Ni	-N2	-C5	-H5'	-158.9(17)
C7	-N2	-C5	-H5	-161.5(16)
C7	-N2	-C5	-H5'	-38.8(17)
H12	-N2	-C5	-C4	-162(2)
H12	-N2	-C5	-H5	-43(3)
H12	-N2	-C5	-H5'	80(3)
Ni	-N2	-C7	-H7	138(2)
C5	-N2	-C7	-H7	21(2)
H12	-N2	-C7	-C6	147(2)
H12	-N2	-C7	-C8	25(2)
H12	-N2	-C7	-H7	-92(3)
O1	-C1	-C2	-H2	-148(2)
O2	-C1	-C2	-H2	35(2)
N1	-C2	-C3	-H3	58(3)
N1	-C2	-C3	-H3'	177(2)
N1	-C2	-C3	-H3"	-65(3)
C1	-C2	-C3	-H3	-63(3)
C1	-C2	-C3	-H3'	56(2)
C1	-C2	-C3	-H3"	174(3)
H2	-C2	-C3	-H3	178(3)
H2	-C2	-C3	-H3'	-64(3)
H2	-C2	-C3	-H3"	54(3)
N1	-C4	-C5	-H5	-62.3(18)
N1	-C4	-C5	-H5'	179.3(17)
H4	-C4	-C5	-N2	-57.7(18)
H4	-C4	-C5	-H5	-177(2)
H4	-C4	-C5	-H5'	64(2)
H4'	-C4	-C5	-N2	-178(2)
H4'	-C4	-C5	-H5	63(3)
H4'	-C4	-C5	-H5'	-56(3)
O3	-C6	-C7	-H7	-137(2)
O4	-C6	-C7	-H7	46(2)
N2	-C7	-C8	-H8	58(2)
N2	-C7	-C8	-H8'	-59(3)
N2	-C7	-C8	-H8"	-173(2)
C6	-C7	-C8	-H8	-65(2)
C6	-C7	-C8	-H8'	178(3)

C6	-C7	-C8	-H8"	64(2)
H7	-C7	-C8	-H8	175(3)
H7	-C7	-C8	-H8'	59(3)
H7	-C7	-C8	-H8"	-56(3)

The sign of the torsion angle is positive if when looking from atom-2 to atom-3 a clockwise motion of atom-1 would superimpose it on atom-4.

Table 4.

Geometry of intra- and intermolecular hydrogen bonds (Å,°) with *s.u.*'s in parentheses

D-H···A			[ARU-code]	D-H(Å)	H···A(Å)	D···A(Å)	D-H··· A(°)
N1	--H11	..O1	[2656.01]	0.81(4)	2.45(3)	3.228(3)	162(3)
N2	--H12	..O4	[2545.01]	0.84(4)	2.25(3)	3.034(2)	156(3)
O5	--H13	..O3	[2546.01]	0.82(3)	2.08(3)	2.885(3)	167(3)
O5	--H13'	..O2	[1455.01]	0.78(4)	1.93(4)	2.679(3)	161(4)
O6	--H14	..O4	[2546.01]	0.88(4)	1.83(4)	2.707(3)	174(4)
O6	--H14'	..O2	[2656.01]	0.67(4)	2.16(4)	2.821(4)	173(3)
C3	--H3	..O6	[ ]	1.01(3)	2.50(3)	3.335(3)	140(2)
C3	--H3"	..O1	[2656.01]	0.84(5)	2.57(4)	3.179(3)	130(3)
C5	--H5'	..O2	[2655.01]	1.05(3)	2.32(3)	3.364(3)	170(2)

Translation of ARU-code to Equivalent Position Code

==========

[2656.] = 1-x,1/2+y,1-z
[1455.] = -1+x,y,z
[2546.] = -x,-1/2+y,1-z
[2545.] = -x,-1/2+y,-z
[2655.] = 1-x,1/2+y,-z

CIF file :

>>>> 932753

(Cambridge Crystallographic Data Centre).

Table S2. Conformations of *N*-membered Rings ($N \leq 8$)

Ring Size *N*	Definition	Descriptive Name	Descriptive Symbol
4	$q_2 \neq 0$	puckered form	
5	$q_2 > 0;\ \phi_2 = 0$	envelope	
	$q_2 > 0;\ \phi_2 = \pi/2$	twist form	
6	$q_3 = 0;\ q_2 > 0;\ \phi_2 = 0$	boat	
	$q_3 = 0;\ q_2 > 0;\ \phi_2 = \pi/2$	twist-boat	
	$q_2 = 0;\ q_3 \neq 0$	chair	
7	$q_3 = 0;\ q_2 > 0;\ \phi_2 = 0$	boat	
	$q_3 = 0;\ q_2 > 0;\ \phi_2 = \pi/2$	twist-boat	
	$q_2 = 0;\ q_3 > 0;\ \phi_3 = 0$	chair	
	$q_2 = 0;\ q_3 > 0;\ \phi_3 = \pi/2$	twist-chair	
8	$q_4 = q_3 = 0;\ q_2 > 0;\ \phi_2 = 0$	boat-boat	
	$q_4 = q_3 = 0;\ q_2 > 0;\ \phi_2 = \pi/2$	twist-boat	
	$q_4 = q_2 = 0;\ q_3 > 0;\ \phi_3 = 0$	long-chair	
	$q_4 = q_2 = 0;\ q_3 > 0;\ \phi_3 = \pi/2$	twist-chair	
	$q_3 = q_2 = 0;\ q_4 \neq 0$	crown	

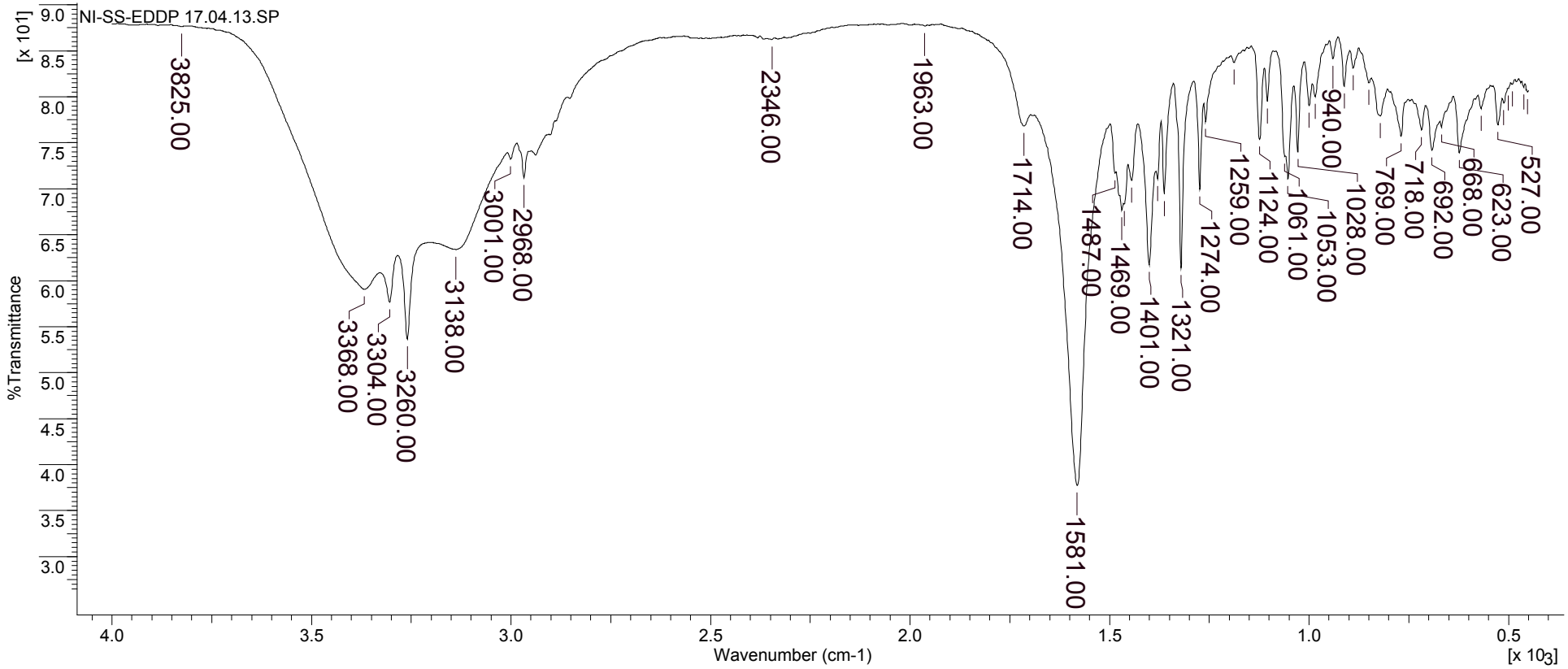
Table 3
3-Center, 2-Electron A:-B-:C Hyperbonds (β orbitals) (A-B :C <=> A: B-C).

Hypobond A:-B-:C	%A-B/%B-C	OCC.	NBOs BD[1] (A-B)	NBOs LP[2] (C)	3-center hybrids h(A)	3-center hybrids h(B)	3-center hybrids h(C)	DA Energy[3]
		edda						
N 20:-Ni 1-: O 2	65.7/34.3	1.9789	2	59	3	4	90	76.63
N 21:-Ni 1-: O 5	64.2/35.8	1.9789	3	61	5	6	92	
		MeOhedda						
N 22:-Ni 1-: O 5	62.3/37.7	1.9683	1	72	1	2	110	
N 24:-Ni 1-: O 2	62.7/37.3	1.9697	2	70	3	4	108	66.42
O 37:-Ni 1-: O 35	50.7/49.3	1.9467	3	80	5	6	118	
		***S,S*-eddp**						
N 18:-Ni 1-: O 5	65.1/34.9	1.9798	1	68	1	2	104	
N 19:-Ni 1-: O 2	65.0/35.0	1.9802	2	66	3	4	102	75
O 32:-Ni 1-: O 34	50.9/49.1	1.9493	3	76	5	6	112	
		edap						
N 14:-Ni 24-: O 1	66.4/33.6	1.9852	15	60	29	30	94	
N 17:-Ni 24-: O 25	54.7/45.3	1.9565	22	73	43	44	107	73.92
O 22:-Ni 24-: O 23	61.6/38.4	1.9772	30	68	59	60	102	
		eddp						
N 25:-Ni 1-: O 3	66.2/33.8	1.9863	1	68	1	2	104	
N 30:-Ni 1-: O 35	58.1/41.9	1.9633	2	76	3	4	112	71.32
O 34:-Ni 1-: O 2	63.7/36.3	1.9815	3	66	5	6	102	
		1,3-pdda						
		no 3-center hyperbonds						74.83
		dacoda						
N 5:-Ni 1-: O 9	53.7/46.3	1.9499	1	76	1	2	115	99.67
		baboc						
O 48:-Ni 1-: O 24	53.1/46.9	1.9553	3	103	5	6	163	95.4

[1]BD means Bonding orbital
[2]LP means Lone Pair

[Ni[S,S-eddp)(H2O)2]_IR

File Name	C:\DOCUMENTS AND SETTINGS\ZORAN MATOVIC\LOCAL SETTINGS\TEMPORARY INTERNET FILES\CONTENT.IE5\OPGPNSLM\NI-SS-EDDP 17.04.13.SP				
Date Stamp	wed apr 17 00:22:41 2013	***Date***	wed apr 17 00:22:41 2013	***Technique***	Infrared
Instrument	Spectrum One	***Spectral Region***	IR	***X Axis***	Wavenumber (cm-1)
Y Axis	%Transmittance	***Spectrum Range***	450.0000 - 4000.0000	***Points Count***	3551
Data Spacing	1.0000				



No	cm-1	%T	Intensity
1	453.00	80.488	W
2	462.00	81.042	W
3	490.00	81.352	W
4	501.00	80.880	W
5	512.00	79.343	W
6	527.00	76.941	W
7	569.00	78.724	W
8	623.00	73.914	W
9	668.00	76.646	W
10	692.00	74.149	W
11	718.00	76.371	W
12	769.00	75.672	W

No	cm-1	%T	Intensity
13	821.00	77.910	W
14	850.00	81.509	W
15	889.00	83.097	VW
16	912.00	81.118	W
17	940.00	84.105	VW
18	985.00	79.994	W
19	1000.00	79.016	W
20	1028.00	74.013	W
21	1053.00	71.042	M
22	1061.00	73.516	W
23	1105.00	79.509	W
24	1124.00	75.371	W
25	1188.00	83.708	VW
26	1259.00	77.255	W
27	1274.00	69.919	M
28	1321.00	61.408	M
29	1363.00	69.444	M
30	1380.00	71.036	M
31	1401.00	61.736	M
32	1445.00	70.889	M
33	1463.00	68.328	M
34	1469.00	67.690	M
35	1487.00	71.657	M
36	1581.00	37.718	VS
37	1714.00	76.826	W
38	1963.00	87.692	VW
39	2346.00	86.197	VW
40	2968.00	71.175	M
41	3001.00	73.226	W
42	3138.00	63.393	M
43	3260.00	53.636	S
44	3304.00	57.661	S
45	3368.00	59.061	M
46	3825.00	87.635	VW

[Ni[edap)(H2O)2]_IR

File Name	E:\NAUKA\SVETLANA-DISERTACIJA\EDAP\IRSPEKTRI\01022008\NI-EDMAMP NEUTRALNO (01.02.08).SP					***Date Stamp***	fri feb 01 14:16:36 2008
Date	Fri Feb 01 14:16:39 2008	***Technique***	Infrared	***Instrument***	Spectrum One	***Spectral Region***	IR
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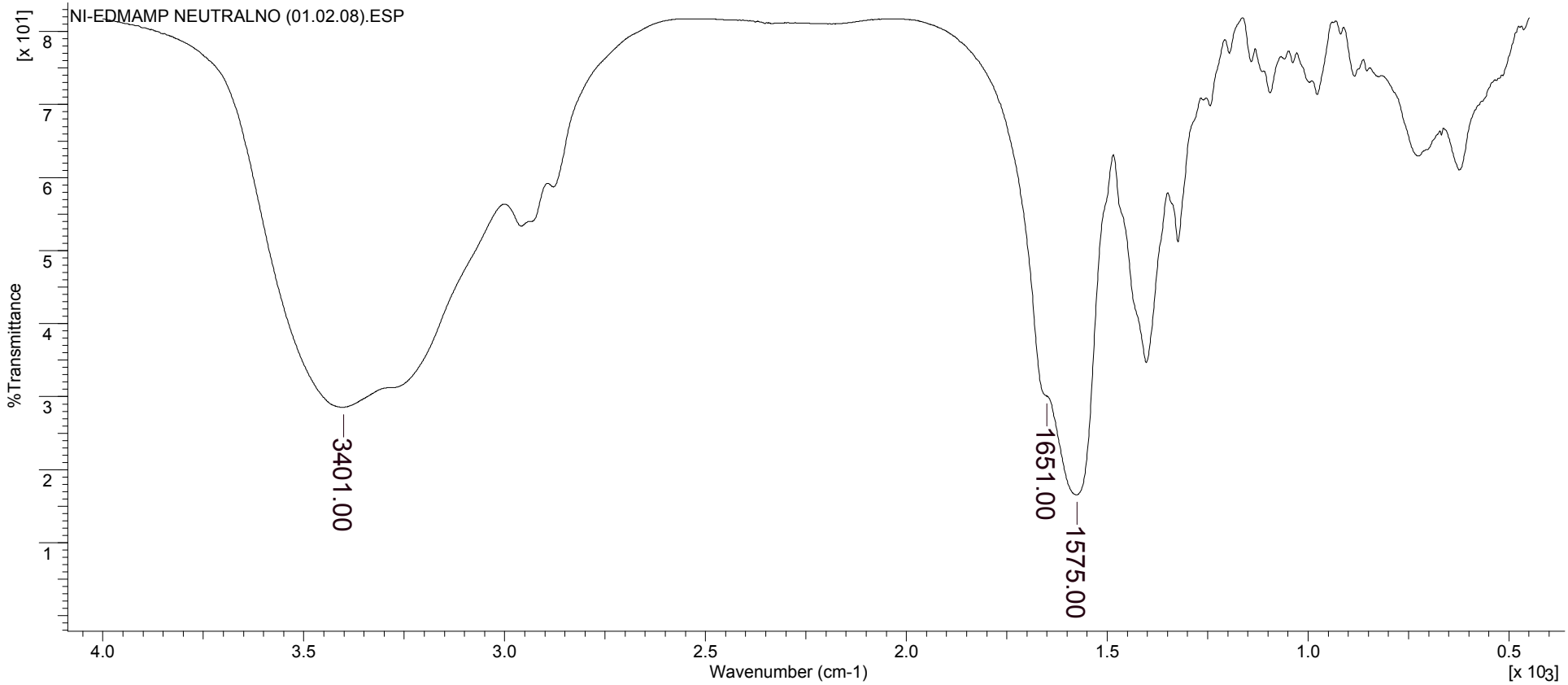

No	cm-1	%T	Intensity
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Ni-SSeddp

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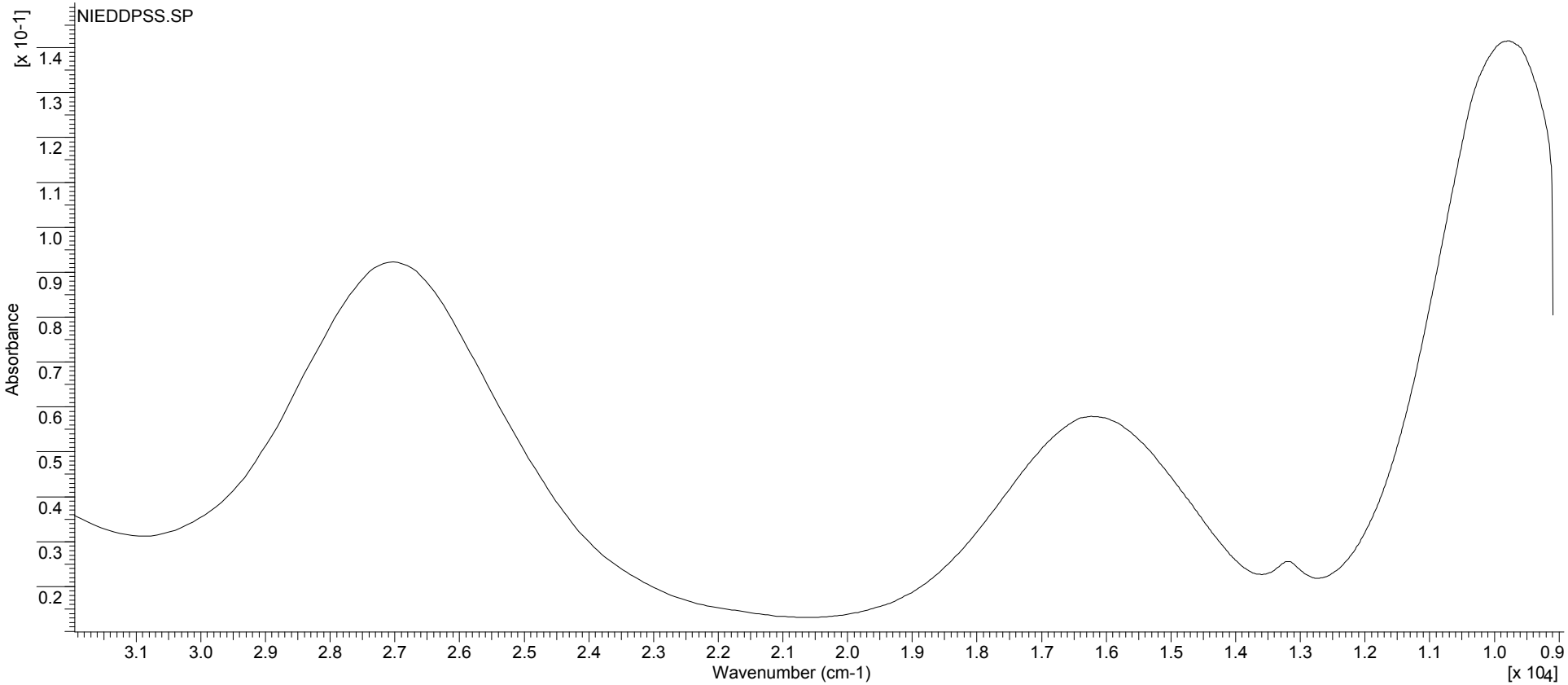
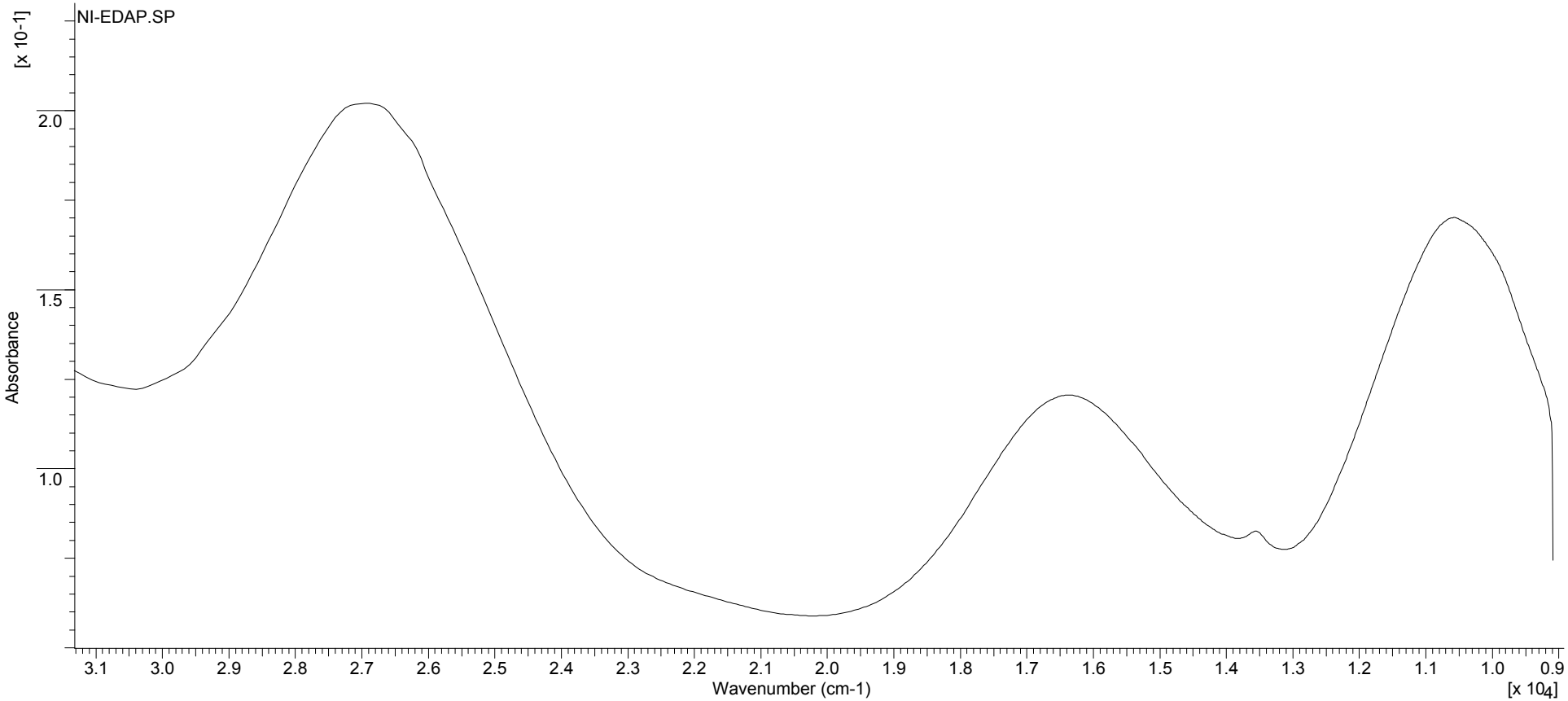
Date Stamp	Mon Mar 18 12:53:54 2013	***Technique***	UV-Visible	***Spectral Region***	UV-Vis-NIR
X Axis	Wavelength (nanometers)	***Y Axis***	Absorbance	***Spectrum Range***	200.0000 - 1100.0000
Points Count	901	***Data Spacing***	1.0000		



No	cm-1	A	FWHH	Asym	Intensity
1	9842.52	0.141	-	-	VW
2	13192.61	0.026	-	-	VW
3	16233.77	0.058	3992.56	-0.04	VW
4	27027.03	0.092	4405.67	0.00	VW

Ni-edap

File Name	H:\ZA STAMPU\SRECKO\NIEDAP\NICKEL\UV\NI-EDAP.SP			***Date Stamp***	Tue May 22 14:53:01 2012		
Technique	UV-Visible	***Spectral Region***	UV-Vis-NIR	***X Axis***	Wavelength (nanometers)		
Y Axis	Absorbance	***Spectrum Range***	200.0000 - 1100.0000	***Points Count***	901	***Data Spacing***	1.0000



No	cm-1	A	FWHH	Asym	Intensity
1	10570.82	0.170	3542.07	0.40	VW
2	13568.52	0.083	-	-	VW
3	16339.87	0.121	-	-	VW
4	26954.18	0.202	-	-	VW