Electronic Supplementary Material for Organic & Biomolecular Chemistry

Supporting Information:

A C_2-Symmetric Nickel Diamine Complex as an Asymmetric Catalyst for Enecarbamate Additions to Butane-2,3-dione

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Experimental Section:

General:

All reactions were carried out under an argon atmosphere in dried glassware. Reaction solvents were distilled prior to use by standard procedures. Preparative thin layer chromatography was carried out using Wakogel B-F5. Butane-2,3-dione **1** was freshly distilled from anhydrous magnesium sulfate under reduced pressure at room temperature prior to use.[1] Enecarbamates **2a-c** were prepared according to the method reported by Kagan *et al*.[2] Diamine ligand **5** was prepared according top a reported procedure.[3] Metal triflate compounds that were not commercially available from the usual suppliers ($Ni(OTf)_2$ & $Co(OTf)_2$) were prepared in accordance with literature methods.[4a-c] High-performance liquid chromatography was conducted using the following apparatus: SHIMADZU LC-10AT (liquid chromatograph), SHIMADZU SPD-10A (UV detector) and SHIMADZU C-R6A Chromatopac (CHIRACEL AD-H column, *i*-propylalcohol:hexanes 1:19 as eluent, 0.5 mL flow rate, unless otherwise stated). Proton and ^{13}C NMR spectra were recorded in $CDCl_3$ on JEOL JNM-LA300, JNM-LA400, JNM-DE400 or JNM-DE600, tetramethyl silane ($\delta = 0$) or residual solvent peak ($CHCl_3 = 7.26$ in $CDCl_3$) served as standards for ^1H NMR, $CDCl_3$ ($\delta = 77.0$) for ^{13}C NMR. Mass spectra were recorded on a BRUKER DIALATONICS® BIOTOF® II machine. Melting points of crystalline materials are uncorrected and were determined on YAZAWA BY-1 apparatus. IR spectra were measured on a JASCO FT/IR-610. X-Ray diffraction analysis was carried out on a Rigaku RAXIS-RAPID diffractometer (see later).

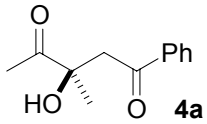
General Procedure for Catalysed Reactions:

Metal triflates were weighed in a dry-box into dried two necked flasks fitted with 3-way taps. Diamine ligand corresponding to 1.0 molar equivalent was added under a flow of argon, the flask was sealed and evacuated-back-filled with argon (3 cycles). Dry solvents were added *via* syringe to give the stated concentration with respect to metal triflate. The solutions were stirred at the specified temperature and enecarbamates (**2a-c)** (corresponding to the given catalyst loading) were added rapidly under a flow a of argon, followed by freshly distilled diketone **1a-b**. Reactions were stirred at that temperature for the stated time. Reactions were quenched at the

[1]Perrin, D.D.; Armarego, W.L.F. *Purification of Laboratory Chemicals*, 3rd ed.; Pergamon Press: Oxford, England, 1998.
[2]Suen, Y.H.; Horeau, A.; Kagan, H.B. *Bull. Soc. Chim. Fr.* **1965**, *5,* 1454.
[3] Kobayashi, S.; Matsubara, R.; Nakamura, Y.; Kitagawa, H.; Sugira, M. *J. Am. Chem. Soc.* **2003**, *125,* 2507.
[4] (a) Boumizane, K.; Herzog-Cance, M.H.; Jones, D.J.; Pascal, J.L.; Potier, J.; Roziere, J. *Polyhedron* **1991**, *10,* 2757.(b) Jansky, M.T.; Yoke, J.T. *J. Inorg. Nucl. Chem.* **1979**, *41,* 1707. (c) Dixon, N.E.; Lawrance, G.A.; Lay, P.A.; Sargeson, A.M.; Taubes, H. *Inorg. Synth.* **1986**, *24,* 243.

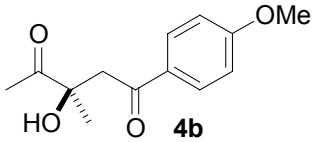
given temperature by addition of 3 N HCl_{aqu} solution, and then stirred at room temperature (exposed to air) for ~1 min, transferred to a separating funnel and extracted with EtOAc (3 times). The organic fractions were combined dried over magnesium sulfate and solvent removed *in vacuo*. These residues were loaded onto preparative TLC plates with DCM and purified by eluting with benzene/acetone 7/1 (rf ~ 0.36).

Analytical Data:



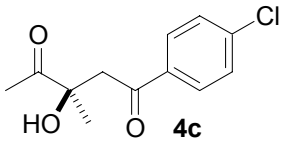
4a

Compound **4a**:[5]
^{1}H NMR (δ; 600 MHz; $CDCl_3$) 1.31 (3H, s, C*H*$_3$-C-OH), 2.31 (3H, s, C*H*$_3$-C=O), 3.17 (1H, d, *J* = 17.9, C*H*H), 3.73 (1H, d, *J* = 17.2, CH*H*), 4.30-4.48 (1H, br s, O*H*), 7.41 (2H, t, *J* = 7.6, Ph 3- and 5-H), 7.53 (1H, t, *J* = 7.6, Ph 4-H), 7.87 (2H, d, *J* = 8.2, Ph 2- and 6-H). ^{13}C NMR (δ; 150 MHz; $CDCl_3$) 24.3 (H_3*C*-C=O), 25.2 (H_3*C*-C-OH), 47.1 (*C*H_2), 78.2 (*C*-OH), 128.3 (Ph 2- and 6-*C*H), 128.7 (Ph 3- and 5-*C*H), 133.9 (Ph 4-*C*H), 136.1 (Ph 1-*C*), 200.6 (O=*C*-Ph), 213.4 (O=*C*-Me). (Found: $[M+H]^+$, *m/z*, (ESI) 207.1029. $C_{12}H_{15}O_3$ requires 207.1021) IR (neat) 1675 (C=O), 1711 (C=O) and 3461 (br O-H). HPLC analysis of ee under stated conditions major enantiomer 26.9 min, minor enantiomer 28.1 min.



4b

Compound **4b**:[5]
^{1}H NMR (δ; 600 MHz; $CDCl_3$) 1.29 (3H, s, C*H*$_3$-C-OH), 2.31 (3H, s, C*H*$_3$-C=O), 3.10 (1H, d, *J* = 17.9, C*H*H), 3.69 (1H, d, *J* = 17.2, CH*H*), 3.81 (3H, *s*, O-C*H*$_3$), 4.41-4.62 (1H, br s, O*H*), 6.86 (2H, d, *J* = 8.9, Ar 2- and 6-H), 7.85 (2H, d, *J* = 8.9, Ar 3- and 5-H). ^{13}C NMR (δ; 150 MHz; $CDCl_3$) 24.4 (H_3*C*-C=O), 25.1 (H_3*C*-C-OH), 46.7 (*C*H_2), 55.5 (O-*C*H_3), 78.3 (*C*-OH), 113.8 (Ar 3- and 5-*C*H), 129.2 (Ar 1-*C*), 130.7 (Ar 2- and 6-*C*H), 164.1 (Ar 4-*C*-OMe), 199.01 (O=*C*-Ph), 213.7 (O=*C*-Me). M.p. 123-124 C. Anal. calcd. for $C_{13}H_{17}O_4$: C, 66.09; H, 6.83. Found: C, 65.99; H, 6.70. (Found: $[M+H]^+$, *m/z*, (ESI) 237.1134. $C_{13}H_{17}O_4$ requires 237.1127). IR (neat) 1171 (C-O), 1668 (C=O), 1715 (C=O) and 3503 (O-H) cm^{-1}. HPLC analysis of ee under stated conditions major enantiomer 53.1 min, minor enantiomer 57.0 min.



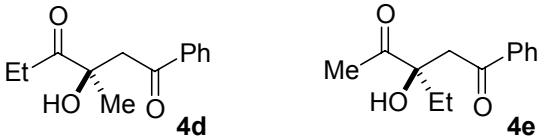
4c

Compound **4c**:
^{1}H NMR (δ; 600 MHz; $CDCl_3$) 1.31 (3H, s, C*H*$_3$-C-OH), 2.30 (3H, s, C*H*$_3$-C=O), 3.14 (1H, d, *J* = 18.0, C*H*H), 3.69 (1H, d, *J* = 18.0, CH*H*), 4.21-4.49 (1H, br s, O*H*), 7.38 (2H, d, *J* = 8.9, Ar 3- and 5-H), 7.81 (2H, d, *J* = 8.2, Ar 2- and 6-H). ^{13}C NMR (δ; 150 MHz; $CDCl_3$) 23.9 (H_3*C*-C=O),

[5] Arime, T.; Takahashi, H.; Kobayashi, S.; Yamaguchi, S.; Mori, N. *Synth. Commun.* **1995**, *25,* 389.

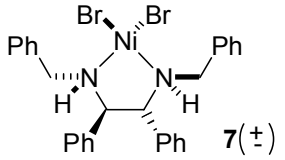
24.8 (H_3C-C-OH), 46.7 (CH_2), 77.9 (*C*-OH), 128.7 (Ar 3- and 5-*C*H), 129.4 (Ar 2- and 6-*C*H), 134.1 (Ar 1-*C*), 140.1 (Ar 4-*C*-Cl), 199.01 (O=*C*-Ph), 212.8 (O=*C*-Me). (Found: $[M+H]^+$, *m/z*, (ESI) 241.0635. $C_{12}H_{14}ClO_3$ requires 241.0631). IR (neat) 1683 (C=O), 1714 (C=O) and 3462 (br O-H) cm^{-1}. HPLC analysis of ee under stated conditions major enantiomer 33.1 min, minor enantiomer 36.8 min.

Compounds **4d** and **4e** *isolated as a mixture*:



4d **4e**

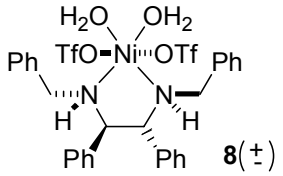
^{1}H NMR spectral data reported together, 1H of compound **4e** is given as 1H integration whereas 1H for **4d** is given as 7H (i.e ratio is 7:1), also se attached spectrum. ^{1}H NMR (δ; 600 MHz; $CDCl_3$) 0.86 (3H, t, *J* = 7.6, **4e** CH_2CH_3), 1.02 (21H, t, *J* = 6.9, **4d**, CH_2CH_3), 1.30 (21H, s, **4d** CH_3-C-OH), 1.69-1.78 (2H, m, **4e** CH_2CH_3), 2.28 (3H, s, **4e** H_3C-C=O), 2.68-2.81 (14H, m, **4d** CH_2CH_3), 3.14 (7H, d, *J* = 17.9, **4d** CH*H*), 3.26 (1H, d, *J* = 17.2, **4e** CH*H*), 3.59 (1H, d, *J* = 17.2, **4e** C*H*H), 3.77 (7H, d, *J* = 17.8, **4d** C*H*H), 4.26-4.48 (7.3H, br with br side peak, **4d** + **4e** O*H*), 7.27-7.33 (2H, br m, **4e** Ph 2- & 5-H), 7.40 (14H, t, *J* = 8.3, **4d** Ph 3- & 5-H), 7.53 (8H, app t, *J* = 7.6, , **4d** + **4e** Ph 4-H), 7.87 (16H, app d, *J* = 7.9, , **4d** + **4e** Ph 2- & 6-H). Copy of ^{13}C spectrum of mixture also included (see later), assignments not given (i.e. mostly major product observed some peaks for minor can be seen but are inconclusive). (Found: $[M+H]^+$, *m/z*, (ESI) 221.1176. $C_{13}H_{17}O_3$ requires 221.1178). IR (neat) 1672 (C=O), 1709 (C=O) and 3462 (br O-H) cm^{-1}. HPLC analysis of ee (eluent = 9:1 hexane:*i*-propyl alcochol, *flow rate* = 0.2 mL/min) **4d** major enantiomer 47.6 min, **4d** minor enantiomer 53.6 min and **4e** major enantiomer 51.4 min, **4e** minor enantiomer 57.2 min.



7(±)

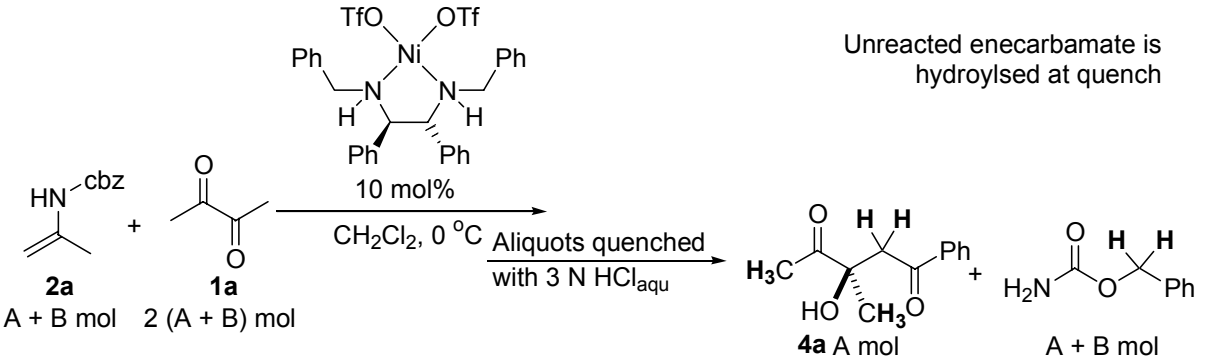
Compound **7**:
Racemic diamine **5** (0.1107 g, 0.282 mmol) was combined with nickel(II) bromide (0.0670 g, 0.307 mmol) under an argon atmosphere. Acetonitrile (20 mL) was added and the mixture heated at reflux for 0.5 hours then stirred at room temperature for 8 hours, resulting in a light blue solution. Solvent was removed *in vacuo* and the brown solid was triturated with hexane, the pink/brown residue was taken up in CH_2Cl_2 and filtered to remove a small amount of insoluble material. Solvent was removed *in vacuo* once more and the brown/pink solid obtained was dried *in vacuo* at 85 C for a further 6 hours. Dissolution in CH_2Cl_2 gave a bright pink/red solution which readily provided deep purple crystals by slow addition of hexane. X-Ray crystal structure analysis failed to give data of sufficient quality for publication but a tetrahedral coordination environment about nickel was tentatively assigned. Upon drying, the purple crystals of **7**, were isolated in 61 % yield (0.1050 g, not including XRD sample). Proton NMR spectroscopy could not provide evidence of structure in a variety of solvents, see attached spectrum recorded in $CDCl_3$ by way of an example. M.p 249-251 C, IR (neat) 3409 (br N-H), 1635, 1456 and 669 cm^{-1}. Anal. calcd. for $C_{28}H_{28}Br_2N_2Ni$: C, 55.04; H, 4.62; N, 4.58. Found: C, 54.67; H, 4.70; N,

4.54. (*m/z*; FAB) 531 (M^+ - Br, 88 %), 451 (M^+ - 2Br, 100 %). (Found: $[M\text{-}2Br+H]^+$, *m/z*, (ESI) 451.486. $C_{28}H_{29}N_2Ni$ requires 451.1684).



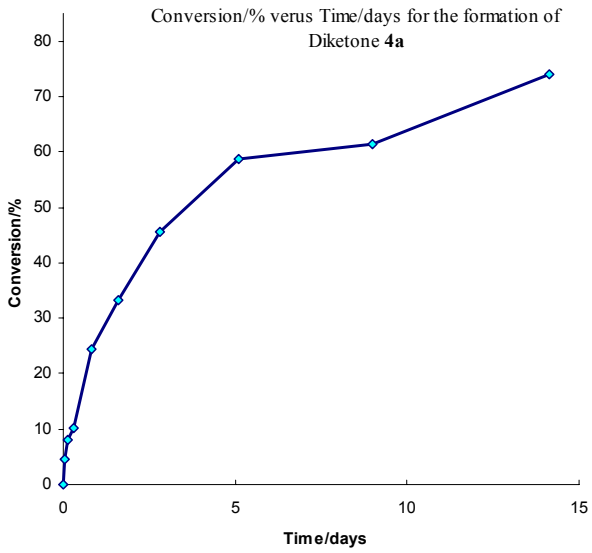
Compound **8**:
Silver triflate (0.0430 g, 0.167 mmol) was stirred in CH_2Cl_2 (10 mL), at room temperature, protected from light. To the resulting mixture purple crystals of compound **7** (0.0453g, 0.074 mmol) were added. Although dry CH_2Cl_2 was used no precautions concerning exclusion of air or moisture were taken after addition of the solid materials. After 10 hours a grey precipitate (consistent with the formation of silver bromide) was removed by filtration and the light blue/green solution was concentrated *in vacuo*. Dissolution in (wet) acetone, filtration through celite, solvent removal *in vacuo*, dissolution in CH_2Cl_2 and subsequent diffusion of diethyl ether gave a light blue precipitate corresponding to 94 % yield of **8** (0.0497 g, 0.069 mmol). The isolated blue powder was split into a number of portion and recrystallisations were attempted. Crystals suitable for XRD conformation of structure **8** were obtained by dissolving **8** in CH_2Cl_2 /Et_2O (10/1) and layering this solution with hexane. No precautions concerning exclusion of light, atmospheric oxygen or water were taken during crystal preparation and the resultant blue crystals were stored in ambient conditions with no apparent decomposition. As with compound **7** no useful NMR data could be obtained the ^1H spectrum recorded in acetone-d_6 is included for comparison. M.p 157-159 C, IR (neat) 3397 (br OH_2+ NH), 1650, 1455, 1250, 1168 (CF_3) and 1029 (SO) cm^{-1}. (Found: $[M+H]^+$, *m/z*, (ESI) 785.5168. $C_{30}H_{33}F_6N_2NiO_8S_2$ requires 785.0936. Found $[M\text{-}OH_2+H]^+$, *m/z*, (ESI) 766.8783. $C_{30}H_{31}F_6N_2NiO_7S_2$ requires 767.0830. Found $[M^+\text{-}OTf+H]^+$, *m/z*, (ESI) 639.4411. $C_{29}H_{33}F_3N_2NiO_5S$ requires 639.1416. Found $[M^+\text{-}2OTf+H]^+$, *m/z*, (ESI) 487.4356. $C_{28}H_{33}N_2NiO_2$ requires 487.1895. Found $[M^+\text{-}2(H_2O+OTf)+H]^+$, *m/z*, (ESI) 451.4564. $C_{28}H_{29}N_2Ni$ requires 451.1684).

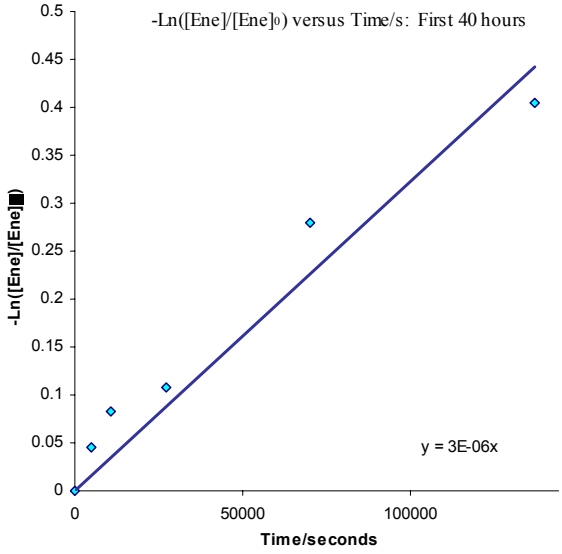
Kinetic Data:
Determination of Observed Rate Constant ($k_{obs.}$) for Reaction of **1a** with **2a**: Calculated conversions are tabulated and represented graphically. k_{obs} Corresponds to the slope of the second plot.



Conversions were calculated with respect to the protons drawn in **bold** type-face, benzyl carbamate serves as internal standard

Time/days	Time/Sec	Conv./%	-Ln([**2a**]/[**2a**]$_0$)
0	0	0	0
0.056	4800	4.4	0.045462
0.125	10800	8.0	0.08325
0.313	27000	10.2	0.107484
0.813	70200	24.4	0.280302
1.583	136800	33.3	0.405465
2.813	243000	45.6	0.609064
5.094	440100	58.7	0.885038
8.997	777300	61.4	0.952009
14.146	1222200	74.1	1.349927



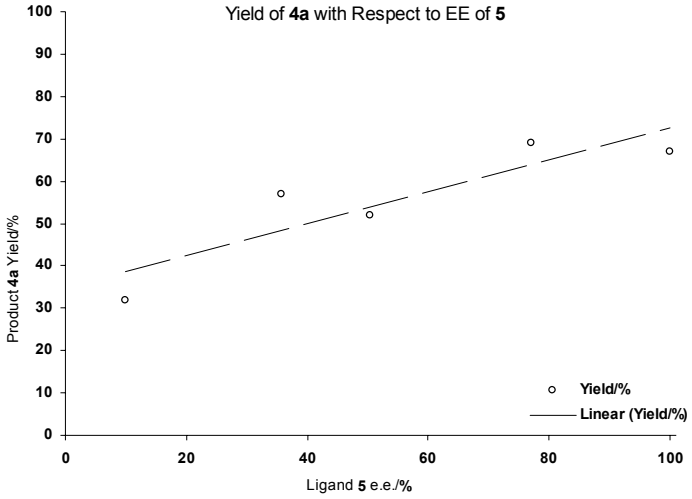


Non-Linear Effect Experiments:

Tabulated here is the data pertaining to Figure 1 in the main text and the associated discussion. The data point for 100% ee ligand is taken from Table 1, entry 1 in the main text. A plot showing approximate correlation between yield of **4a** and ee of **5** is also given.

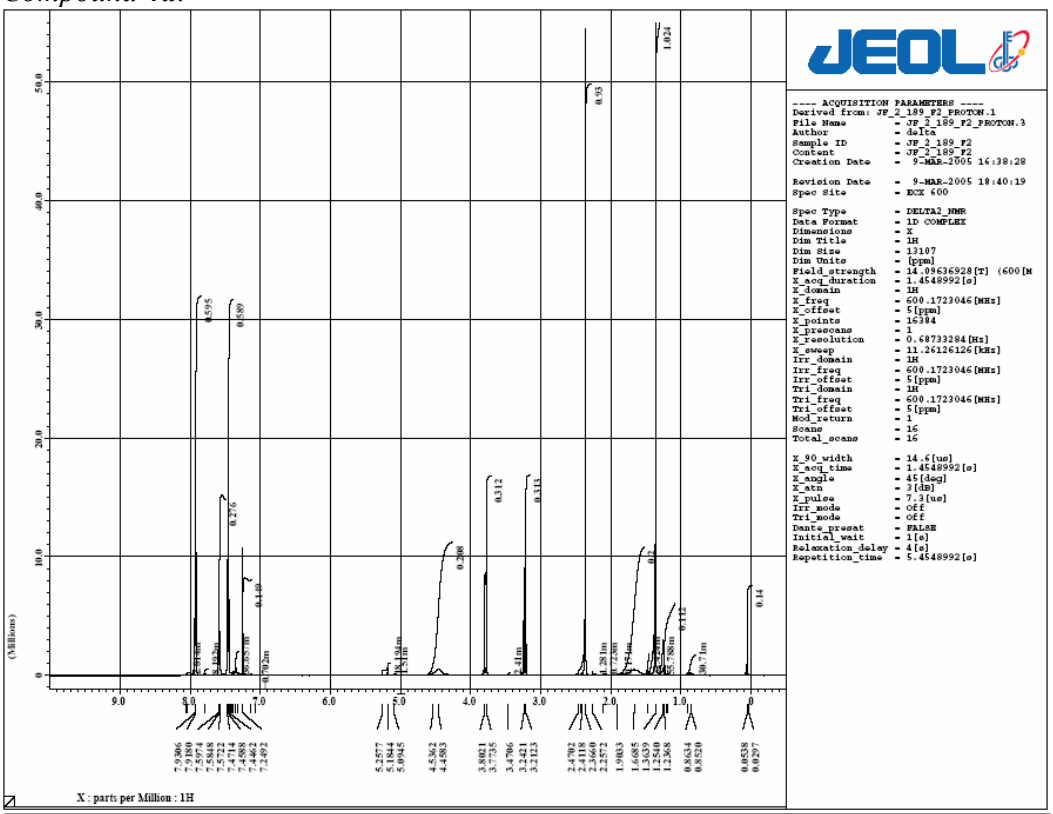
Low ee **5** was prepared by combination of 0.007M stock solutions of *R* **5** and racemic **5**, other experimental conditions match those of Table 1, entry 1 in the main text.

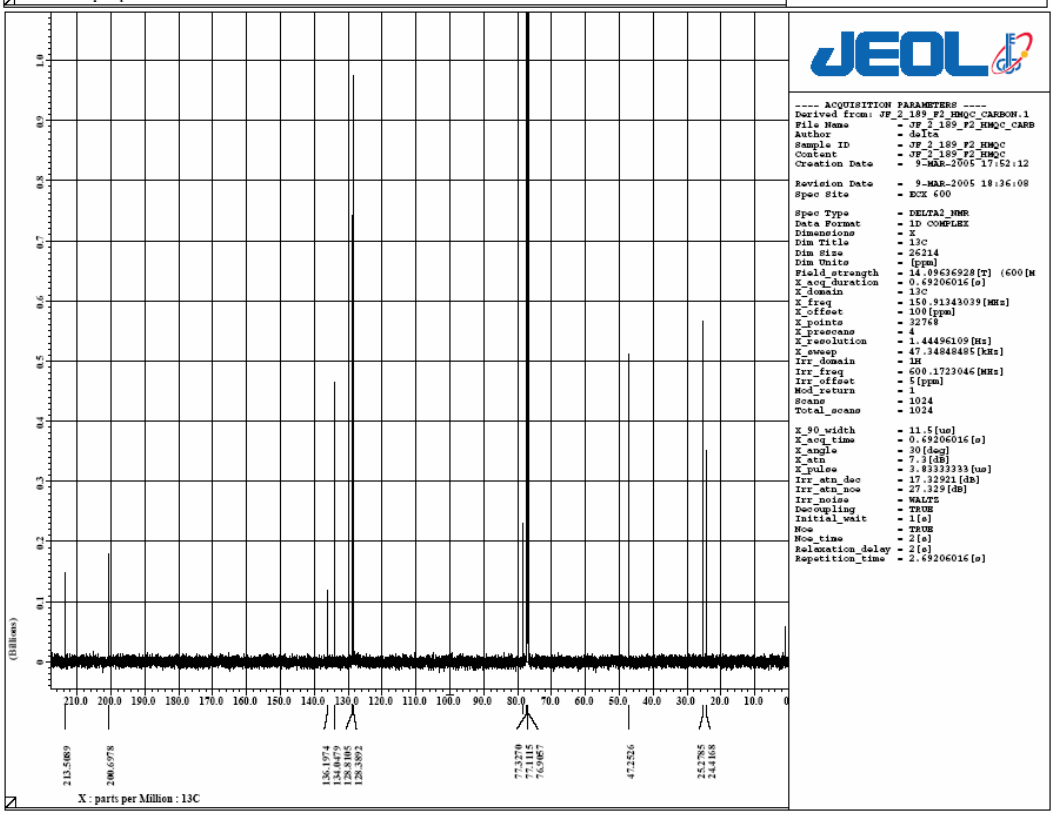
Ligand **5** ee/%	Product **4a** ee/%	Yield **4a**/%
100	76	67
77	76	69
50	79	52
35	78	57
10	58	32



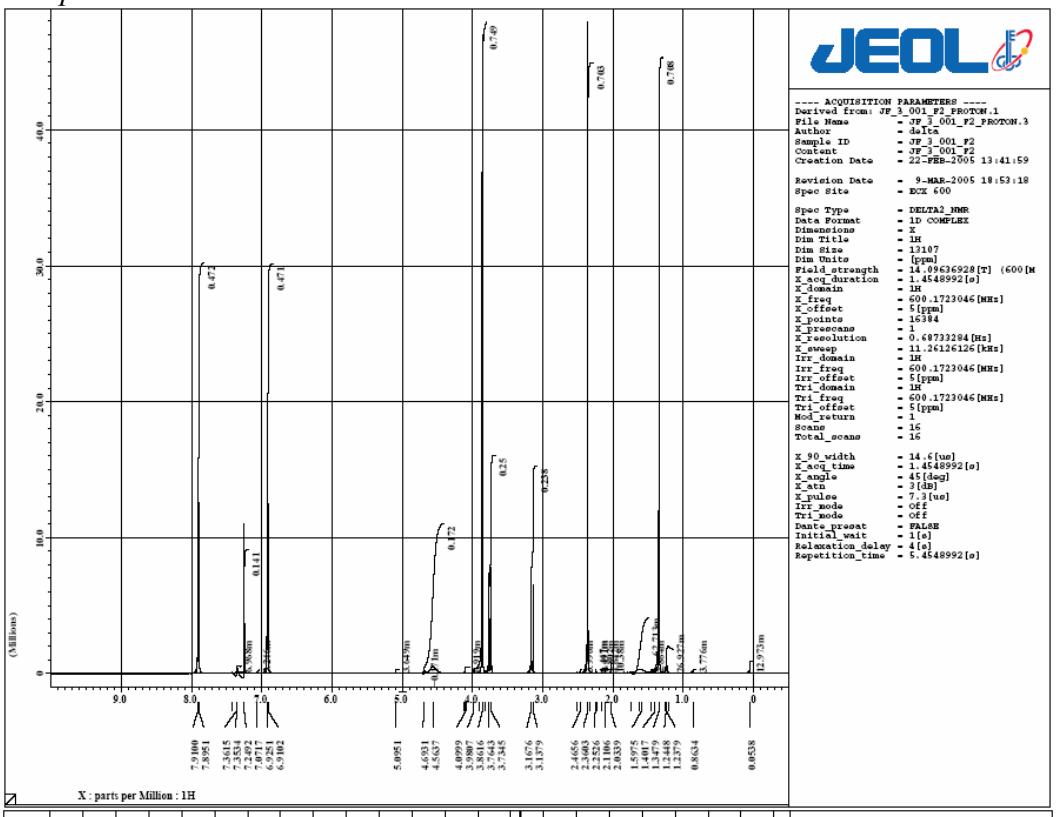
NMR Spectrums ($CDCl_3$)

*Compound **4a**:*





*Compound **4b**:*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Author - delta
Sample ID - JF_3_001_F2
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Revision Date - 9-MAR-2005 18:53:18
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Spec Type - DELTA2_NMR
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X_offset - 5[ppm]
X_points - 16384
X_prescans - 1
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X_sweep - 11.26126126[kHz]
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Irr_freq - 600.1723046[MHz]
Irr_offset - 5[ppm]
Tri_domain - 1H
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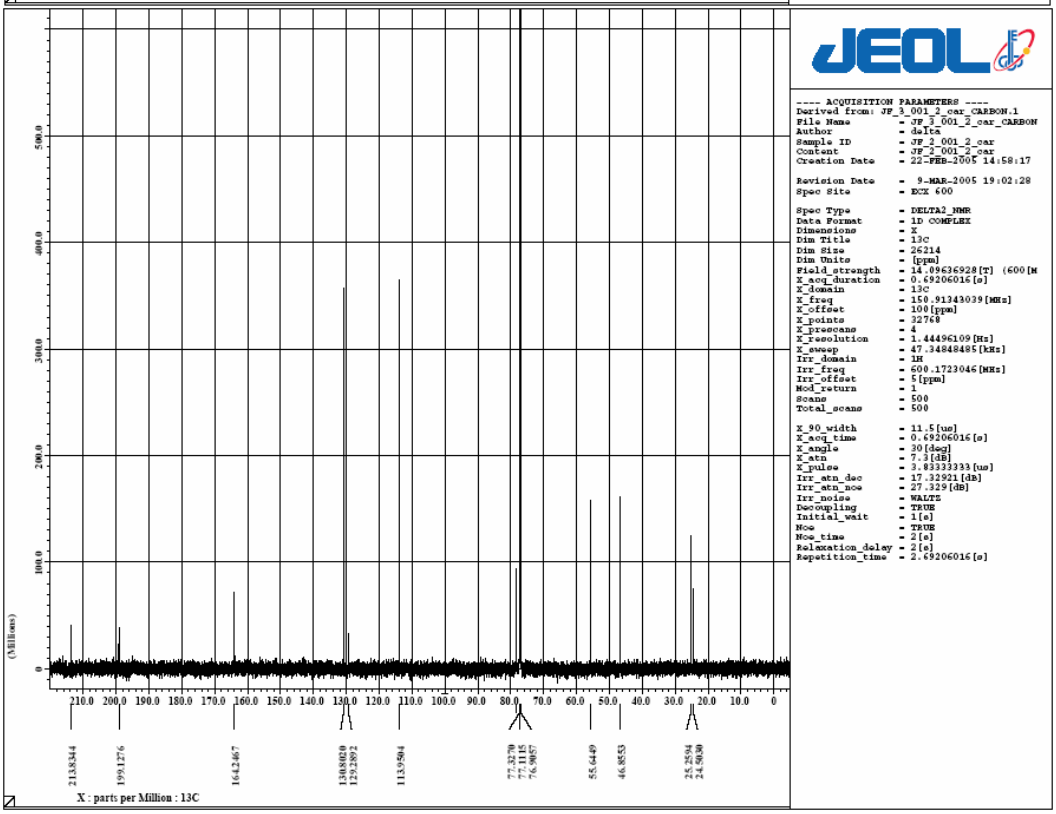
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Revision Date - 9-MAR-2005 19:02:28
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Irr_atn_noe - 27.329[dB]
Irr_noise - WALTZ
Decoupling - TRUE
Initial_wait - 1[s]
Noe - TRUE
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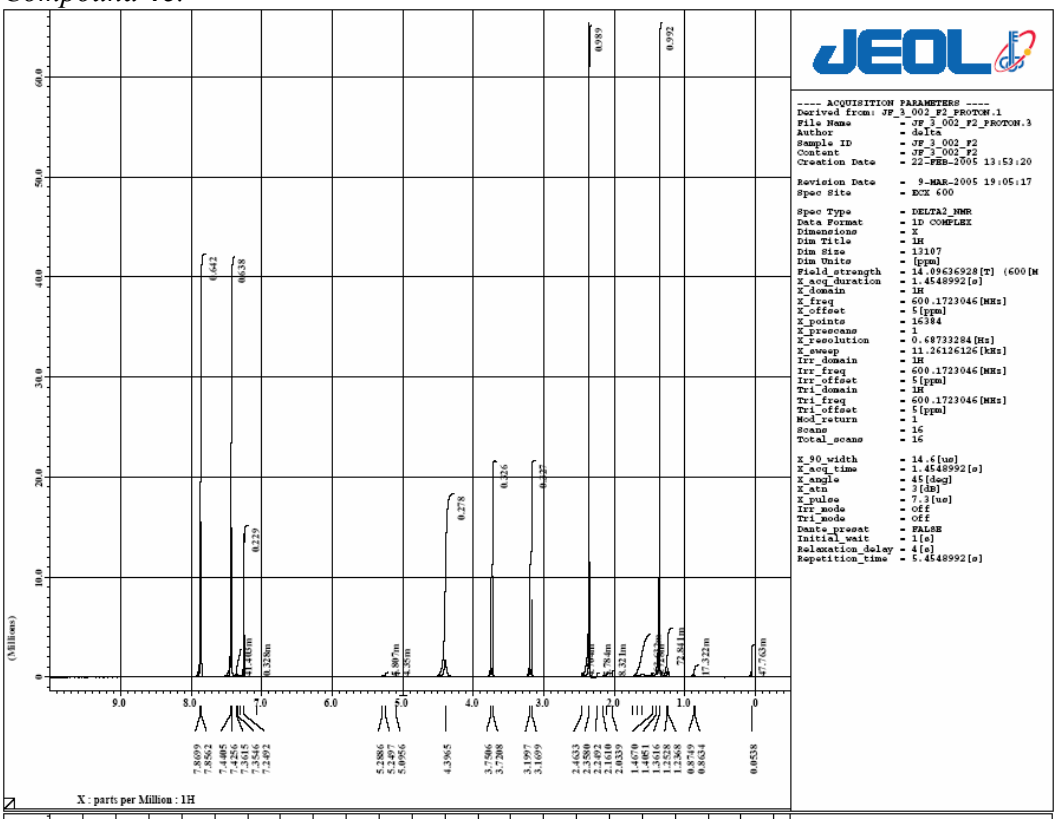
X : parts per Million : 13C

*Compound **4c**:*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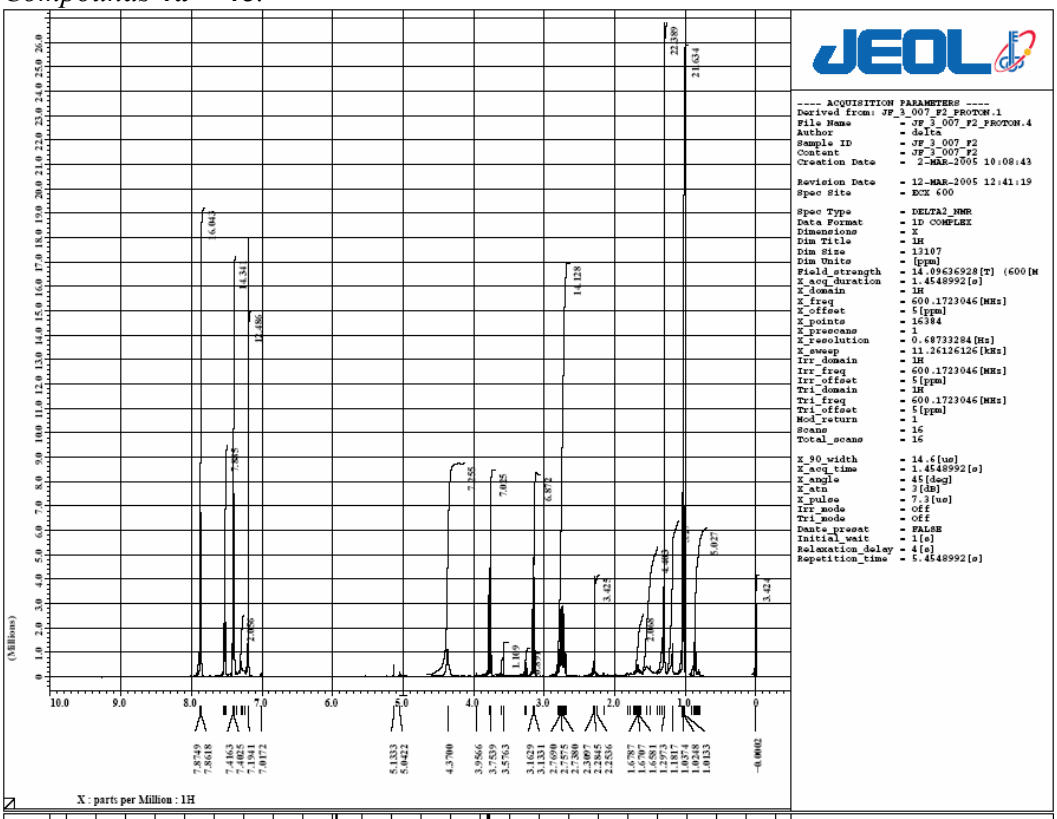
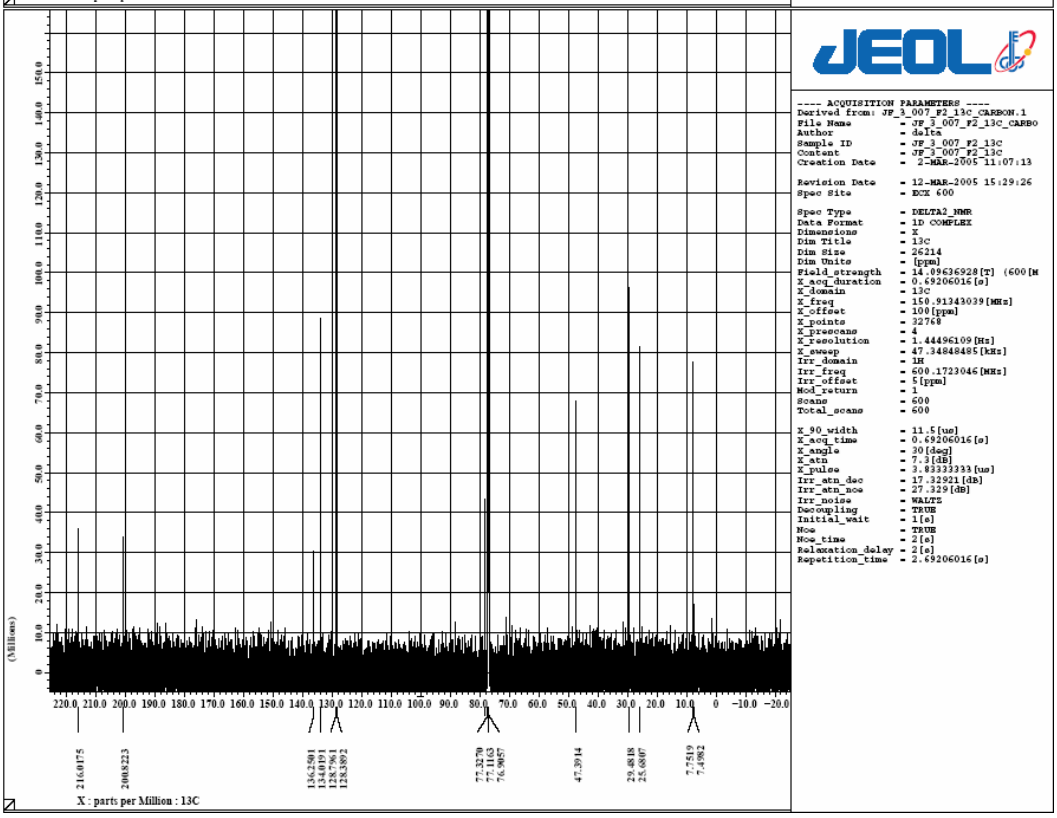
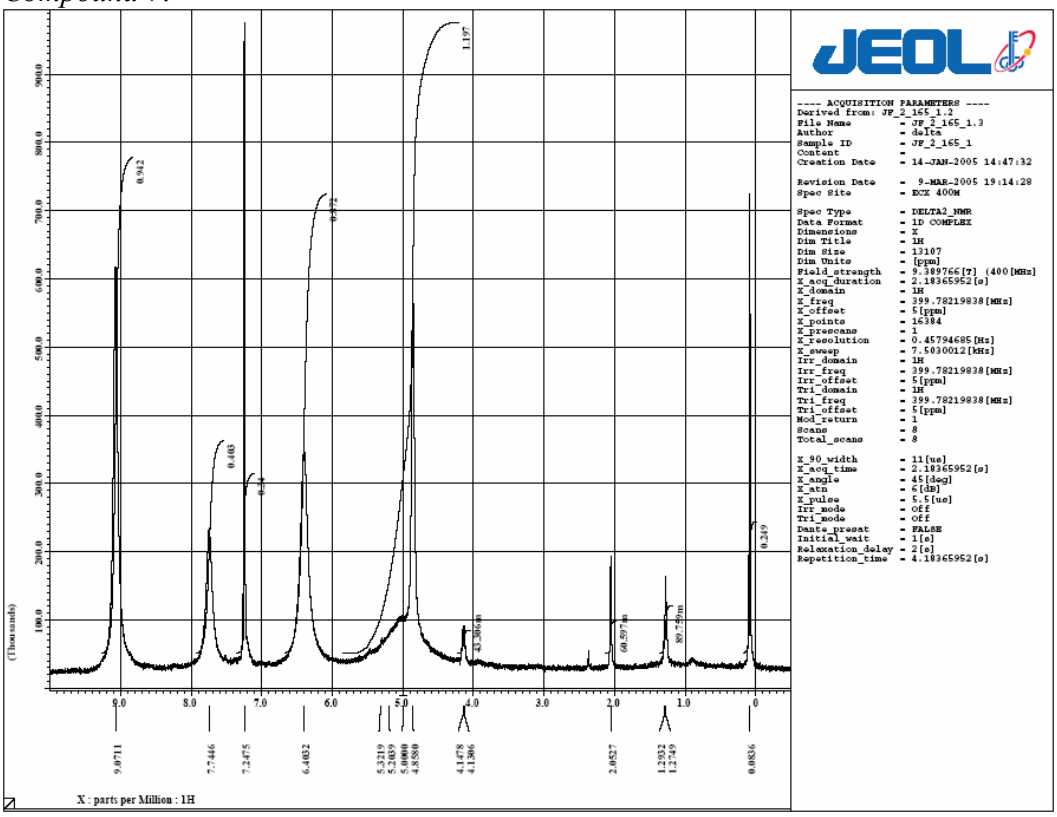
X_sweep - 11.26126126[kHz]
Irr_domain - 1H
Irr_freq - 600.1723046[MHz]
Irr_offset - 5[ppm]
Tri_domain - 1H
Tri_freq - 600.1723046[MHz]
Tri_offset - 5[ppm]
Mod_return - 1
Scans - 16
Total_scans - 16
X_90_width - 14.6[us]
X_acq_time - 1.4548992[s]
X_angle - 45[deg]
X_atn - 3[dB]
X_pulse - 7.3[us]
Irr_mode - Off
Tri_mode - Off
Data_presat - FALSE
Initial_wait - 1[s]
Relaxation_delay - 4[s]
Repetition_time - 5.4548992[s]
X : parts per Million : 1H
JEOL
---- ACQUISITION PARAMETERS ----
Derived from: JF_3_002_F2_car.1
File Name - JF_3_002_F2_car.5
Author - delta
Sample ID - JF_3_002_F2_car
Content -
Creation Date - 22-FEB-2005 16:06:45
Revision Date - 9-MAR-2005 19:08:44
Spec Site - ECX 600
Spec Type - DELTA2_NMR
Data Format - 1D COMPLEX
Dimensions - X
Dim Title - 13C
Dim Size - 26214
Dim Units - [ppm]
Field_strength - 14.09636928[T] (600[M
X_acq_duration - 0.69206016[s]
X_domain - 13C
X_freq - 150.91343039[MHz]
X_offset - 100[ppm]
X_points - 32768
X_prescans - 4
X_resolution - 1.44496109[Hz]
X_sweep - 47.34848485[kHz]
Irr_domain - 1H
Irr_freq - 600.1723046[MHz]
Irr_offset - 5[ppm]
Mod_return - 1
Scans - 1000
Total_scans - 1000
X_90_width - 11.5[us]
X_acq_time - 0.69206016[s]
X_angle - 30[deg]
X_atn - 7.3[dB]
X_pulse - 3.83333333[us]
Irr_atn_dec - 17.32921[dB]
Irr_atn_noe - 27.329[dB]
Irr_noise - WALTZ
Decoupling - TRUE
Initial_wait - 1[s]
Noe - TRUE
Noe_time - 2[s]
Relaxation_delay - 2[s]
Repetition_time - 2.69206016[s]
X : parts per Million : 13C

*Compounds **4d** + **4e**:*

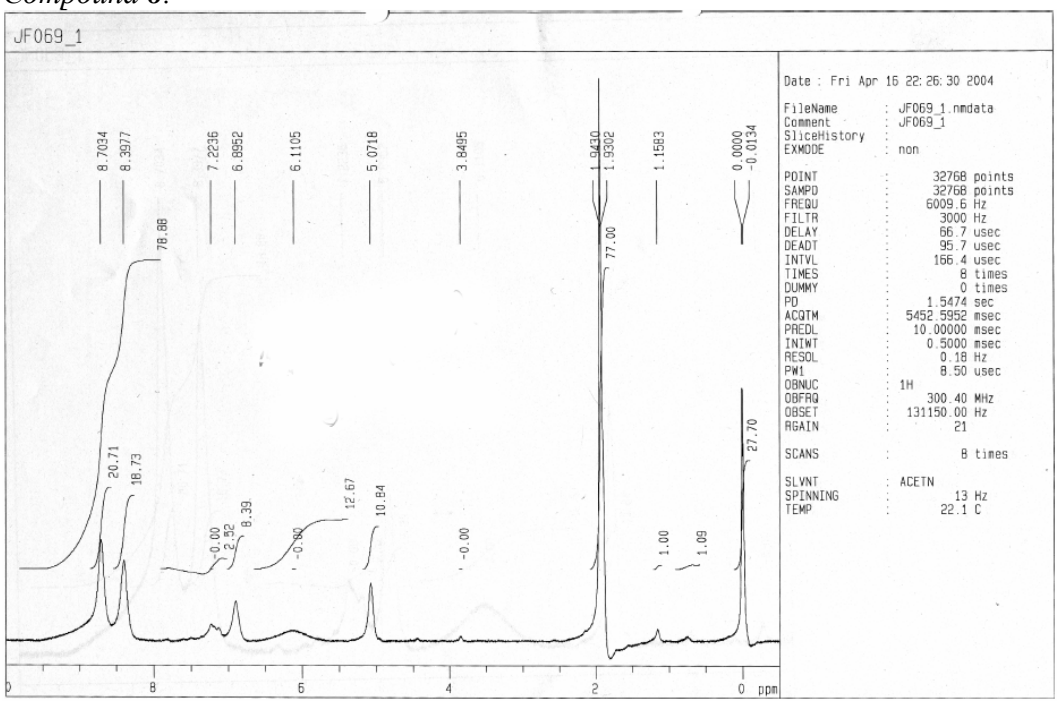




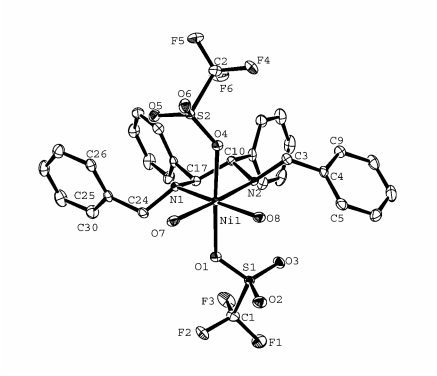
*Compound **7**:*



*Compound **8**:*



Crystal Data for Compound **8:**



Data Collection

A blue block crystal of $C_{30}H_{32}O_8N_2S_2NiF_6$ having approximate dimensions of 0.10 x 0.10 x 0.10 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-Kα radiation.
Indexing was performed from 3 oscillations that were exposed for 90 seconds. The crystal-to-detector distance was 127.40 mm.
Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

a = 11.503(8) Å
b = 15.70(2) Å β = 92.82(3)°
c = 18.53(1) Å
V = 3342(4) Å^3

For Z = 4 and F.W. = 785.40, the calculated density is 1.56 g/cm^3. The systematic absences of:

h0l: h ± 2n
0k0: k ± 2n

uniquely determine the space group to be:

$P2_1/a$ (#14)

The data were collected at a temperature of -173 ± 1°C to a maximum 2θ value of 54.9°. A total of 55 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0° in 3.0° step, at χ=45.0° and ϕ = 0.0°. The exposure rate was 120.0 [sec./°]. A second sweep was performed using ω scans from 0.0 to 160.0° in 4.0° step, at χ=45.0° and ϕ = 180.0°. The exposure rate was 120.0 [sec./°]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 29278 reflections that were collected, 7603 were unique (R_{int} = 0.030); equivalent reflections were merged.

The linear absorption coefficient, μ, for Mo-Kα radiation is 7.9 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.83 to 1.00. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 4965 observed reflections and 492 variable parameters and converged (largest parameter shift was 0.01 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.024$$

$$wR2 = [\Sigma (w (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.030$$

The standard deviation of an observation of unit weight[4] was 1.02. A Robust-resistant weighting scheme was used[5]. Plots of $\Sigma \, w \, (|Fo| - |Fc|)^2$ versus |Fo|, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.31 and -0.34 e$^-$/Å^3, respectively.
Neutral atom scattering factors were taken from Cromer and Waber[6]. Anomalous dispersion effects were included in Fcalc[7]; the values for Δf' and Δf'' were those of Creagh and McAuley[8]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[9]. All calculations were performed using the CrystalStructure[10,11] crystallographic software package.

References

(1) SIR2002: Burla, M.C., Camalli, M., Carrozzini, B., Cascarano, G.L., Giacovazzo, C., Polidori, G., Spagna., R. (2003).
(2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.
(3) Least Squares function minimized:

$\Sigma w(F_o^2 - F_c^2)^2$ where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations

N_v = number of variables

(5) Carruthers, J.R. and Watkin, D.J. (1979), Acta Cryst, A35, 698-699
(6) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).
(7) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).
(8) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).
(9) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).
(10) CrystalStructure 3.6.0: Crystal Structure Analysis Package, Rigaku and Rigaku/MSC (2000-2004). 9009 New Trails Dr. The Woodlands TX 77381 USA.
(11) CRYSTALS Issue 10: Watkin, D.J., Prout, C.K. Carruthers, J.R. & Betteridge, P.W. Chemical Crystallography Laboratory, Oxford, UK. (1996)

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{30}H_{32}O_8N_2S_2NiF_6$
Formula Weight	785.40
Crystal Color, Habit	blue, block
Crystal Dimensions	0.10 X 0.10 X 0.10 mm
Crystal System	monoclinic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 90.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 11.503(8) Å b = 15.70(2) Å c = 18.53(1) Å β = 92.82(3) ° V = 3342(4) Å^3
Space Group	$P2_1/a$ (#14)
Z value	4
D_{calc}	1.561 g/cm^3
F_{000}	1616.00
μ(MoKα)	7.91 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	55 exposures
ω oscillation Range (χ=45.0, ϕ=0.0)	130.0 - 190.0°
Exposure Rate	120.0 sec./°
ω oscillation Range (χ=45.0, ϕ=180.0)	0.0 - 160.0°
Exposure Rate	120.0 sec./°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	54.9°
No. of Reflections Measured	Total: 29278 Unique: 7603 (R_{int} = 0.030)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.8297 - 1.0000)

C. Structure Solution and Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w (Fo^2 - Fc^2)^2
Least Squares Weights	Chebychev polynomial with 3 parameters 6419.4300,8908.6800,3224.1100,
$2\theta_{max}$ cutoff	0.0°
Anomalous Dispersion	All non-hydrogen atoms

No. Observations (I>2.00σ(I))	4965
No. Variables	492
Reflection/Parameter Ratio	10.09
Residuals: R1 (I>2.00σ(I))	0.024
Residuals: wR2 (I>2.00σ(I))	0.030
Goodness of Fit Indicator	1.022
Max Shift/Error in Final Cycle	0.010
Maximum peak in Final Diff. Map	0.31 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.34 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
Ni(1)	0.06505(2)	0.11118(1)	0.80246(1)	0.818(4)
S(1)	-0.20562(3)	0.08024(2)	0.77150(2)	0.968(7)
S(2)	0.33925(3)	0.14390(3)	0.86285(2)	1.012(7)
F(1)	-0.40922(8)	0.05515(7)	0.70709(6)	2.44(2)
F(2)	-0.30717(8)	0.15062(6)	0.65578(5)	1.73(2)
F(3)	-0.2648(1)	0.01788(7)	0.64428(5)	2.46(2)
F(4)	0.39435(9)	0.03194(7)	0.96318(6)	2.87(3)
F(5)	0.53880(8)	0.07594(7)	0.90358(6)	2.53(2)
F(6)	0.4154(1)	-0.01262(7)	0.85459(6)	3.03(3)
O(1)	-0.09678(9)	0.11238(8)	0.74405(5)	1.30(2)
O(2)	-0.2610(1)	0.13947(8)	0.81783(6)	1.62(2)
O(3)	-0.1997(1)	-0.00667(7)	0.79510(6)	1.56(2)
O(4)	0.22119(9)	0.11111(7)	0.86665(5)	1.23(2)
O(5)	0.37492(9)	0.15447(7)	0.78966(6)	1.44(2)
O(6)	0.36785(9)	0.21204(7)	0.91173(6)	1.50(2)
O(7)	0.0709(1)	0.24015(7)	0.78806(6)	1.31(2)
O(8)	-0.0170(1)	0.13066(7)	0.89887(6)	1.21(2)
N(1)	0.1458(1)	0.08805(8)	0.70665(7)	0.91(3)
N(2)	0.0657(1)	-0.02076(8)	0.81399(7)	0.91(3)
C(1)	-0.3017(1)	0.0761(1)	0.68967(9)	1.47(3)
C(2)	0.4275(1)	0.0550(1)	0.89817(9)	1.78(4)
C(3)	0.0919(1)	-0.0562(1)	0.88829(8)	1.16(3)
C(4)	0.0072(1)	-0.1231(1)	0.91178(7)	1.13(3)
C(5)	-0.1018(1)	-0.0993(1)	0.93355(8)	1.54(3)
C(6)	-0.1792(1)	-0.1599(1)	0.95836(9)	2.09(4)
C(7)	-0.1469(2)	-0.2449(1)	0.96148(9)	2.32(4)
C(8)	-0.0389(1)	-0.2691(1)	0.93950(9)	1.90(4)
C(9)	0.0376(1)	-0.2089(1)	0.91378(8)	1.40(3)
C(10)	0.1459(1)	-0.05547(9)	0.75992(8)	0.93(3)
C(11)	0.1303(1)	-0.1500(1)	0.74726(8)	1.28(3)
C(12)	0.0258(1)	-0.1823(1)	0.71728(9)	1.51(3)
C(13)	0.0110(2)	-0.2695(1)	0.7076(1)	2.17(4)
C(14)	0.0996(2)	-0.3247(1)	0.7297(1)	2.43(4)
C(15)	0.2038(2)	-0.2938(1)	0.7595(1)	2.23(4)
C(16)	0.2190(1)	-0.2064(1)	0.76795(9)	1.66(4)
C(17)	0.1303(1)	-0.0037(1)	0.68954(8)	1.02(3)
C(18)	0.2101(1)	-0.0356(1)	0.63259(8)	1.07(3)
C(19)	0.3301(1)	-0.0257(1)	0.64107(8)	1.35(3)
C(20)	0.4018(1)	-0.0515(1)	0.58718(9)	1.64(3)
C(21)	0.3534(1)	-0.0888(1)	0.52466(9)	1.86(4)
C(22)	0.2350(2)	-0.1011(1)	0.51651(9)	2.07(4)
C(23)	0.1634(1)	-0.0745(1)	0.57028(9)	1.66(3)
C(24)	0.1099(1)	0.1451(1)	0.64486(8)	1.22(3)
C(25)	0.1983(1)	0.1577(1)	0.58781(8)	1.14(3)
C(26)	0.3103(1)	0.1862(1)	0.60604(8)	1.34(3)
C(27)	0.3898(1)	0.2000(1)	0.55315(9)	1.75(4)
C(28)	0.3570(2)	0.1872(1)	0.48096(9)	1.97(4)
C(29)	0.2456(2)	0.1600(1)	0.46194(9)	2.21(4)
C(30)	0.1670(1)	0.1447(1)	0.51501(9)	1.83(4)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq}

atom	x	y	z	B_{eq}
H(1)	-0.1239	-0.0410	0.9315	1.866
H(2)	-0.2535	-0.1429	0.9734	2.520
H(3)	-0.1989	-0.2861	0.9792	2.756
H(4)	-0.0174	-0.3275	0.9407	2.296

H(5)	0.1113	-0.2263	0.8980	1.672
H(6)	0.0905	-0.0104	0.9217	1.389
H(7)	0.1676	-0.0805	0.8895	1.363
H(8)	-0.000(1)	-0.038(1)	0.8045(9)	1.0(4)
H(9)	0.2233	-0.0460	0.7785	1.086
H(10)	-0.0354	-0.1442	0.7032	1.821
H(11)	-0.0592	-0.2913	0.6856	2.620
H(12)	0.0893	-0.3845	0.7245	2.977
H(13)	0.2641	-0.3320	0.7748	2.726
H(14)	0.2913	-0.1851	0.7874	2.000
H(15)	0.0523	-0.0114	0.6715	1.241
H(16)	0.0818	-0.0833	0.5648	1.998
H(17)	0.2022	-0.1272	0.4739	2.483
H(18)	0.4021	-0.1064	0.4874	2.304
H(19)	0.4836	-0.0434	0.5926	1.979
H(20)	0.3627	-0.0011	0.6843	1.593
H(21)	0.223(2)	0.100(1)	0.7190(9)	1.9(4)
H(22)	0.0924	0.1994	0.6641	1.474
H(23)	0.0416	0.1214	0.6220	1.458
H(24)	0.3327	0.1949	0.6555	1.614
H(25)	0.4663	0.2191	0.5663	2.100
H(26)	0.4108	0.1974	0.4446	2.410
H(27)	0.2232	0.1510	0.4125	2.685
H(28)	0.0907	0.1255	0.5015	2.159
H(29)	0.012(2)	0.268(1)	0.788(1)	2.6(5)
H(30)	0.123(2)	0.268(1)	0.799(1)	3.0(5)
H(31)	0.028(2)	0.139(1)	0.932(1)	3.0(5)
H(32)	-0.062(2)	0.169(1)	0.899(1)	2.6(5)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 3. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni(1)	0.00985(9)	0.00835(9)	0.01292(9)	0.00019(9)	0.00109(7)	0.00009(9)
S(1)	0.0110(2)	0.0114(2)	0.0142(2)	-0.0002(1)	-0.0015(1)	0.0010(1)
S(2)	0.0114(2)	0.0127(2)	0.0142(2)	-0.0018(1)	-0.0017(1)	0.0026(1)
F(1)	0.0179(5)	0.0348(7)	0.0389(6)	-0.0106(5)	-0.0102(5)	0.0078(5)
F(2)	0.0287(5)	0.0160(5)	0.0204(5)	0.0014(4)	-0.0066(4)	0.0045(4)
F(3)	0.0469(7)	0.0226(6)	0.0228(5)	0.0077(5)	-0.0084(5)	-0.0100(5)
F(4)	0.0271(6)	0.0432(7)	0.0386(6)	0.0075(5)	0.0000(5)	0.0279(5)
F(5)	0.0123(5)	0.0411(7)	0.0422(6)	0.0031(5)	-0.0029(4)	0.0112(5)
F(6)	0.0360(6)	0.0197(6)	0.0584(8)	0.0093(5)	-0.0079(5)	-0.0029(5)
O(1)	0.0133(5)	0.0173(6)	0.0187(5)	-0.0002(5)	-0.0007(4)	0.0025(5)
O(2)	0.0193(6)	0.0234(7)	0.0188(6)	0.0056(5)	0.0013(4)	-0.0018(5)
O(3)	0.0185(6)	0.0150(6)	0.0252(6)	-0.0028(5)	-0.0040(5)	0.0062(5)
O(4)	0.0124(5)	0.0169(6)	0.0173(5)	-0.0011(5)	-0.0012(4)	0.0028(5)
O(5)	0.0158(6)	0.0236(7)	0.0150(5)	-0.0041(5)	0.0000(4)	0.0017(5)
O(6)	0.0211(6)	0.0176(6)	0.0180(6)	-0.0065(5)	-0.0005(5)	0.0008(5)
O(7)	0.0119(6)	0.0128(6)	0.0250(7)	0.0004(6)	0.0017(5)	-0.0012(5)
O(8)	0.0141(6)	0.0170(7)	0.0150(6)	0.0039(5)	0.0007(5)	-0.0008(5)
N(1)	0.0111(7)	0.0107(7)	0.0129(6)	0.0002(5)	0.0006(5)	0.0019(5)
N(2)	0.0085(6)	0.0116(7)	0.0144(7)	-0.0003(5)	-0.0003(5)	0.0001(5)
C(1)	0.0183(9)	0.0161(9)	0.0210(8)	-0.0020(7)	-0.0048(7)	-0.0016(7)
C(2)	0.0156(8)	0.023(1)	0.0283(9)	0.0024(7)	-0.0007(7)	0.0070(8)
C(3)	0.0177(8)	0.0140(8)	0.0124(7)	0.0001(6)	-0.0004(6)	0.0007(6)
C(4)	0.0168(8)	0.0184(9)	0.0076(7)	-0.0029(6)	-0.0008(6)	0.0017(6)
C(5)	0.0212(8)	0.025(1)	0.0125(7)	0.0045(7)	-0.0004(6)	0.0025(7)
C(6)	0.0170(9)	0.046(1)	0.0162(8)	-0.0031(8)	0.0006(7)	0.0059(8)
C(7)	0.031(1)	0.039(1)	0.0180(9)	-0.0188(9)	-0.0056(8)	0.0096(8)
C(8)	0.034(1)	0.0158(9)	0.0217(9)	-0.0066(8)	-0.0045(8)	0.0053(7)
C(9)	0.0200(9)	0.0163(9)	0.0168(8)	0.0005(7)	0.0006(6)	0.0005(7)
C(10)	0.0117(7)	0.0095(8)	0.0139(7)	0.0006(6)	0.0010(6)	0.0001(6)
C(11)	0.0222(9)	0.0124(8)	0.0146(8)	-0.0009(7)	0.0067(6)	0.0014(7)
C(12)	0.0213(9)	0.0156(9)	0.0208(8)	-0.0013(7)	0.0060(7)	-0.0022(7)
C(13)	0.033(1)	0.023(1)	0.028(1)	-0.0122(8)	0.0125(8)	-0.0082(8)
C(14)	0.050(1)	0.0102(9)	0.034(1)	-0.0017(9)	0.0221(9)	-0.0040(8)
C(15)	0.040(1)	0.0148(9)	0.032(1)	0.0099(8)	0.0157(9)	0.0032(8)
C(16)	0.0238(9)	0.0178(9)	0.0220(9)	0.0047(7)	0.0064(7)	0.0023(7)
C(17)	0.0133(8)	0.0084(8)	0.0170(7)	-0.0010(6)	-0.0013(6)	-0.0018(6)
C(18)	0.0178(8)	0.0089(8)	0.0141(7)	0.0000(6)	0.0021(6)	0.0023(6)
C(19)	0.0204(8)	0.0158(9)	0.0150(8)	0.0012(7)	0.0002(6)	-0.0010(6)

C(20)	0.0184(8)	0.0203(9)	0.0240(9)	0.0048(7)	0.0038(7)	0.0013(7)
C(21)	0.030(1)	0.021(1)	0.0217(9)	0.0038(7)	0.0122(7)	-0.0014(7)
C(22)	0.039(1)	0.023(1)	0.0165(8)	-0.0071(8)	0.0051(7)	-0.0067(8)
C(23)	0.0217(9)	0.0205(9)	0.0211(8)	-0.0070(7)	0.0021(7)	-0.0030(7)
C(24)	0.0164(8)	0.0109(7)	0.0187(8)	0.0017(6)	-0.0006(6)	0.0024(7)
C(25)	0.0201(8)	0.0084(8)	0.0151(8)	0.0016(6)	0.0019(6)	0.0018(6)
C(26)	0.0225(9)	0.0120(8)	0.0166(8)	0.0007(7)	0.0022(6)	0.0009(6)
C(27)	0.0243(9)	0.0157(9)	0.0271(9)	-0.0001(7)	0.0058(7)	0.0002(7)
C(28)	0.034(1)	0.020(1)	0.0229(9)	0.0020(8)	0.0153(8)	0.0019(7)
C(29)	0.045(1)	0.026(1)	0.0130(8)	-0.0003(9)	0.0032(7)	-0.0007(7)
C(30)	0.0247(9)	0.0241(9)	0.0205(9)	-0.0029(8)	-0.0018(7)	0.0006(7)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Ni(1)	O(1)	2.107(1)	Ni(1)	O(4)	2.105(1)
Ni(1)	O(7)	2.044(1)	Ni(1)	O(8)	2.084(1)
Ni(1)	N(1)	2.075(1)	Ni(1)	N(2)	2.083(1)
S(1)	O(1)	1.464(1)	S(1)	O(2)	1.436(1)
S(1)	O(3)	1.434(1)	S(1)	C(1)	1.833(2)
S(2)	O(4)	1.457(1)	S(2)	O(5)	1.445(1)
S(2)	O(6)	1.430(1)	S(2)	C(2)	1.827(2)
F(1)	C(1)	1.335(2)	F(2)	C(1)	1.327(2)
F(3)	C(1)	1.326(2)	F(4)	C(2)	1.331(2)
F(5)	C(2)	1.321(2)	F(6)	C(2)	1.337(2)
N(1)	C(17)	1.484(2)	N(1)	C(24)	1.495(2)
N(2)	C(3)	1.502(2)	N(2)	C(10)	1.498(2)
C(3)	C(4)	1.512(2)	C(4)	C(5)	1.387(2)
C(4)	C(9)	1.391(2)	C(5)	C(6)	1.397(3)
C(6)	C(7)	1.386(3)	C(7)	C(8)	1.380(3)
C(8)	C(9)	1.392(2)	C(10)	C(11)	1.511(2)
C(10)	C(17)	1.540(2)	C(11)	C(12)	1.395(2)
C(11)	C(16)	1.391(2)	C(12)	C(13)	1.391(3)
C(13)	C(14)	1.385(3)	C(14)	C(15)	1.382(3)
C(15)	C(16)	1.391(2)	C(17)	C(18)	1.517(2)
C(18)	C(19)	1.391(2)	C(18)	C(23)	1.390(2)
C(19)	C(20)	1.386(2)	C(20)	C(21)	1.390(2)
C(21)	C(22)	1.376(3)	C(22)	C(23)	1.388(2)
C(24)	C(25)	1.516(2)	C(25)	C(26)	1.389(2)
C(25)	C(30)	1.394(2)	C(26)	C(27)	1.390(2)
C(27)	C(28)	1.386(2)	C(28)	C(29)	1.380(3)
C(29)	C(30)	1.389(3)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
O(7)	H(29)	0.81(2)	O(7)	H(30)	0.77(2)
O(8)	H(31)	0.80(2)	O(8)	H(32)	0.80(2)
N(1)	H(21)	0.93(2)	N(2)	H(8)	0.81(2)
C(3)	H(6)	0.9502	C(3)	H(7)	0.9500
C(5)	H(1)	0.9501	C(6)	H(2)	0.9499
C(7)	H(3)	0.9500	C(8)	H(4)	0.9501
C(9)	H(5)	0.9500	C(10)	H(9)	0.9500
C(12)	H(10)	0.9500	C(13)	H(11)	0.9500
C(14)	H(12)	0.9500	C(15)	H(13)	0.9500
C(16)	H(14)	0.9500	C(17)	H(15)	0.9500
C(19)	H(20)	0.9500	C(20)	H(19)	0.9500
C(21)	H(18)	0.9501	C(22)	H(17)	0.9500
C(23)	H(16)	0.9500	C(24)	H(22)	0.9500
C(24)	H(23)	0.9500	C(26)	H(24)	0.9501
C(27)	H(25)	0.9500	C(28)	H(26)	0.9500
C(29)	H(27)	0.9500	C(30)	H(28)	0.9501
H(8)	N(2)	0.81(2)	H(11)	C(13)	0.9500

Table 6. Bond angles (^{O})

atom	atom	atom	angle	atom	atom	atom	angle
Ni(1)	O(1)	S(1)	124.58(6)	O(4)	Ni(1)	O(1)	176.48(4)
O(7)	Ni(1)	O(1)	87.59(5)	O(8)	Ni(1)	O(1)	90.72(4)
N(1)	Ni(1)	O(1)	89.06(4)	N(2)	Ni(1)	O(1)	93.44(5)
Ni(1)	O(4)	S(2)	138.06(6)	O(7)	Ni(1)	O(4)	92.39(5)
O(8)	Ni(1)	O(4)	85.75(4)	N(1)	Ni(1)	O(4)	94.46(4)
N(2)	Ni(1)	O(4)	86.74(5)	O(8)	Ni(1)	O(7)	89.17(5)
N(1)	Ni(1)	O(7)	92.46(5)	N(2)	Ni(1)	O(7)	177.31(5)

N(1)	Ni(1)	O(8)	178.35(5)	N(2)	Ni(1)	O(8)	93.30(5)
N(2)	Ni(1)	N(1)	85.08(5)	Ni(1)	N(1)	C(17)	107.37(9)
Ni(1)	N(1)	C(24)	115.58(9)	Ni(1)	N(2)	C(3)	117.54(9)
Ni(1)	N(2)	C(10)	107.00(9)	O(3)	S(1)	O(1)	113.91(7)
O(2)	S(1)	O(1)	113.36(7)	C(1)	S(1)	O(1)	102.48(7)
O(3)	S(1)	O(2)	116.76(7)	C(1)	S(1)	O(2)	104.48(7)
C(1)	S(1)	O(3)	103.69(7)	S(1)	C(1)	F(1)	109.7(1)
S(1)	C(1)	F(2)	111.9(1)	S(1)	C(1)	F(3)	110.5(1)
O(5)	S(2)	O(4)	113.21(6)	O(6)	S(2)	O(4)	114.82(6)
C(2)	S(2)	O(4)	102.43(7)	O(6)	S(2)	O(5)	116.18(7)
C(2)	S(2)	O(5)	104.20(7)	C(2)	S(2)	O(6)	103.79(7)
S(2)	C(2)	F(4)	110.7(1)	S(2)	C(2)	F(5)	110.9(1)
S(2)	C(2)	F(6)	110.5(1)	F(1)	C(1)	F(2)	108.0(1)
F(1)	C(1)	F(3)	108.2(1)	F(2)	C(1)	F(3)	108.5(1)
F(4)	C(2)	F(5)	108.6(1)	F(4)	C(2)	F(6)	107.7(1)
F(5)	C(2)	F(6)	108.3(1)	N(1)	C(17)	C(10)	108.9(1)
C(24)	N(1)	C(17)	113.1(1)	N(1)	C(17)	C(18)	113.5(1)
N(1)	C(24)	C(25)	116.3(1)	C(10)	N(2)	C(3)	112.1(1)
N(2)	C(3)	C(4)	114.7(1)	N(2)	C(10)	C(17)	108.9(1)
N(2)	C(10)	C(11)	112.9(1)	C(3)	C(4)	C(5)	120.0(1)
C(3)	C(4)	C(9)	121.0(1)	C(9)	C(4)	C(5)	118.9(1)
C(4)	C(5)	C(6)	120.8(2)	C(4)	C(9)	C(8)	120.3(2)
C(5)	C(6)	C(7)	119.7(2)	C(6)	C(7)	C(8)	119.8(2)
C(7)	C(8)	C(9)	120.5(2)	C(17)	C(10)	C(11)	112.3(1)
C(10)	C(11)	C(12)	120.8(1)	C(10)	C(11)	C(16)	120.1(1)
C(10)	C(17)	C(18)	111.4(1)	C(16)	C(11)	C(12)	119.0(2)
C(11)	C(12)	C(13)	120.5(2)	C(11)	C(16)	C(15)	120.7(2)
C(12)	C(13)	C(14)	119.6(2)	C(13)	C(14)	C(15)	120.6(2)
C(14)	C(15)	C(16)	119.6(2)	C(17)	C(18)	C(19)	121.0(1)
C(17)	C(18)	C(23)	120.0(1)	C(23)	C(18)	C(19)	118.9(1)
C(18)	C(19)	C(20)	120.6(1)	C(18)	C(23)	C(22)	120.7(2)
C(19)	C(20)	C(21)	119.7(1)	C(20)	C(21)	C(22)	120.3(2)
C(21)	C(22)	C(23)	119.9(2)	C(24)	C(25)	C(26)	121.3(1)
C(24)	C(25)	C(30)	120.3(1)	C(30)	C(25)	C(26)	118.4(1)
C(25)	C(26)	C(27)	120.9(1)	C(25)	C(30)	C(29)	120.9(2)
C(26)	C(27)	C(28)	120.0(2)	C(27)	C(28)	C(29)	119.7(2)
C(28)	C(29)	C(30)	120.1(2)				

Table 7. Bond angles involving hydrogens (^{o})

atom	atom	atom	angle	atom	atom	atom	angle
Ni(1)	O(7)	H(29)	120(1)	Ni(1)	O(7)	H(30)	124(1)
Ni(1)	O(8)	H(31)	112(1)	Ni(1)	O(8)	H(32)	115(1)
Ni(1)	N(1)	H(21)	102(1)	Ni(1)	N(2)	H(8)	107(1)
H(30)	O(7)	H(29)	109(2)	H(32)	O(8)	H(31)	105(2)
N(1)	C(17)	H(15)	107.4	H(21)	N(1)	C(17)	110(1)
H(21)	N(1)	C(24)	107(1)	N(1)	C(24)	H(22)	107.8
N(1)	C(24)	H(23)	107.4	N(2)	C(3)	H(6)	107.9
N(2)	C(3)	H(7)	108.2	H(8)	N(2)	C(3)	102(1)
H(8)	N(2)	C(10)	109(1)	N(2)	C(10)	H(9)	107.3
H(6)	C(3)	C(4)	107.9	H(7)	C(3)	C(4)	108.5
H(7)	C(3)	H(6)	109.4	C(4)	C(5)	H(1)	119.5
C(4)	C(9)	H(5)	119.8	H(1)	C(5)	C(6)	119.7
C(5)	C(6)	H(2)	120.2	H(2)	C(6)	C(7)	120.1
C(6)	C(7)	H(3)	119.8	C(7)	C(8)	H(4)	119.7
H(3)	C(7)	C(8)	120.4	C(8)	C(9)	H(5)	119.9
H(4)	C(8)	C(9)	119.7	H(9)	C(10)	C(11)	108.2
H(9)	C(10)	C(17)	107.0	C(10)	C(17)	H(15)	107.5
C(11)	C(12)	H(10)	119.5	C(11)	C(16)	H(14)	119.8
H(10)	C(12)	C(13)	120.0	C(12)	C(13)	H(11)	120.5
H(11)	C(13)	C(14)	119.9	C(13)	C(14)	H(12)	120.1
C(14)	C(15)	H(13)	120.3	H(12)	C(14)	C(15)	119.3
C(15)	C(16)	H(14)	119.5	H(13)	C(15)	C(16)	120.2
H(15)	C(17)	C(18)	107.8	C(18)	C(19)	H(20)	119.3
C(18)	C(23)	H(16)	119.4	H(20)	C(19)	C(20)	120.2
C(19)	C(20)	H(19)	120.4	H(19)	C(20)	C(21)	119.9
C(20)	C(21)	H(18)	120.1	C(21)	C(22)	H(17)	120.1
H(18)	C(21)	C(22)	119.7	C(22)	C(23)	H(16)	119.9
H(17)	C(22)	C(23)	120.0	H(22)	C(24)	C(25)	107.8
H(23)	C(24)	C(25)	108.0	H(23)	C(24)	H(22)	109.5
C(25)	C(26)	H(24)	119.1	C(25)	C(30)	H(28)	119.5

H(24)	C(26)	C(27)	120.0	C(26)	C(27)	H(25)	120.2
H(25)	C(27)	C(28)	119.8	C(27)	C(28)	H(26)	120.3
C(28)	C(29)	H(27)	119.8	H(26)	C(28)	C(29)	120.0
C(29)	C(30)	H(28)	119.6	H(27)	C(29)	C(30)	120.1

Table 8. Torsion Angles(°)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O(7)	Ni(1)	O(1)	S(1)	-122.73(8)	O(8)	Ni(1)	O(1)	S(1)	-33.59(8)
N(1)	Ni(1)	O(1)	S(1)	144.77(8)	N(2)	Ni(1)	O(1)	S(1)	59.75(8)
O(7)	Ni(1)	O(4)	S(2)	-50.6(1)	O(8)	Ni(1)	O(4)	S(2)	-139.6(1)
N(1)	Ni(1)	O(4)	S(2)	42.1(1)	N(2)	Ni(1)	O(4)	S(2)	126.9(1)
O(1)	Ni(1)	N(1)	C(17)	-78.53(9)	O(1)	Ni(1)	N(1)	C(24)	48.70(9)
O(4)	Ni(1)	N(1)	C(17)	101.33(9)	O(4)	Ni(1)	N(1)	C(24)	-131.43(9)
O(7)	Ni(1)	N(1)	C(17)	-166.07(8)	O(7)	Ni(1)	N(1)	C(24)	-38.8(1)
N(2)	Ni(1)	N(1)	C(17)	15.01(8)	N(2)	Ni(1)	N(1)	C(24)	142.2(1)
O(1)	Ni(1)	N(2)	C(3)	-130.8(1)	O(1)	Ni(1)	N(2)	C(10)	102.11(9)
O(4)	Ni(1)	N(2)	C(3)	45.6(1)	O(4)	Ni(1)	N(2)	C(10)	-81.42(9)
O(8)	Ni(1)	N(2)	C(3)	-39.9(1)	O(8)	Ni(1)	N(2)	C(10)	-166.97(8)
N(1)	Ni(1)	N(2)	C(3)	140.4(1)	N(1)	Ni(1)	N(2)	C(10)	13.36(8)
O(2)	S(1)	O(1)	Ni(1)	80.90(8)	O(3)	S(1)	O(1)	Ni(1)	-55.81(9)
C(1)	S(1)	O(1)	Ni(1)	-167.11(6)	O(1)	S(1)	C(1)	F(1)	-174.3(1)
O(1)	S(1)	C(1)	F(2)	-54.4(1)	O(1)	S(1)	C(1)	F(3)	66.6(1)
O(2)	S(1)	C(1)	F(1)	-55.8(1)	O(2)	S(1)	C(1)	F(2)	64.0(1)
O(2)	S(1)	C(1)	F(3)	-175.0(1)	O(3)	S(1)	C(1)	F(1)	67.0(1)
O(3)	S(1)	C(1)	F(2)	-173.2(1)	O(3)	S(1)	C(1)	F(3)	-52.2(1)
O(5)	S(2)	O(4)	Ni(1)	-26.5(1)	O(6)	S(2)	O(4)	Ni(1)	110.12(9)
C(2)	S(2)	O(4)	Ni(1)	-138.12(9)	O(4)	S(2)	C(2)	F(4)	-54.9(1)
O(4)	S(2)	C(2)	F(5)	-175.5(1)	O(4)	S(2)	C(2)	F(6)	64.3(1)
O(5)	S(2)	C(2)	F(4)	-173.1(1)	O(5)	S(2)	C(2)	F(5)	66.3(1)
O(5)	S(2)	C(2)	F(6)	-53.9(1)	O(6)	S(2)	C(2)	F(4)	64.8(1)
O(6)	S(2)	C(2)	F(5)	-55.7(1)	O(6)	S(2)	C(2)	F(6)	-175.9(1)
Ni(1)	N(1)	C(17)	C(10)	-40.1(1)	Ni(1)	N(1)	C(17)	C(18)	-164.9(1)
C(24)	N(1)	C(17)	C(10)	-168.8(1)	C(24)	N(1)	C(17)	C(18)	66.4(2)
Ni(1)	N(1)	C(24)	C(25)	153.9(1)	C(17)	N(1)	C(24)	C(25)	-81.8(2)
Ni(1)	N(2)	C(3)	C(4)	131.0(1)	C(10)	N(2)	C(3)	C(4)	-104.4(1)
Ni(1)	N(2)	C(10)	C(11)	-164.0(1)	Ni(1)	N(2)	C(10)	C(17)	-38.6(1)
C(3)	N(2)	C(10)	C(11)	65.8(2)	C(3)	N(2)	C(10)	C(17)	-168.8(1)
N(2)	C(3)	C(4)	C(5)	-76.3(2)	N(2)	C(3)	C(4)	C(9)	105.4(2)
C(3)	C(4)	C(5)	C(6)	-177.2(1)	C(9)	C(4)	C(5)	C(6)	1.1(2)
C(3)	C(4)	C(9)	C(8)	176.3(1)	C(5)	C(4)	C(9)	C(8)	-2.1(2)
C(4)	C(5)	C(6)	C(7)	0.2(2)	C(5)	C(6)	C(7)	C(8)	-0.5(2)
C(6)	C(7)	C(8)	C(9)	-0.4(2)	C(7)	C(8)	C(9)	C(4)	1.7(2)
N(2)	C(10)	C(11)	C(12)	63.9(2)	N(2)	C(10)	C(11)	C(16)	-113.7(2)
C(17)	C(10)	C(11)	C(12)	-59.6(2)	C(17)	C(10)	C(11)	C(16)	122.7(2)
N(2)	C(10)	C(17)	N(1)	53.7(2)	N(2)	C(10)	C(17)	C(18)	179.7(1)
C(11)	C(10)	C(17)	N(1)	179.4(1)	C(11)	C(10)	C(17)	C(18)	-54.6(2)
C(10)	C(11)	C(12)	C(13)	-178.2(2)	C(16)	C(11)	C(12)	C(13)	-0.6(2)
C(10)	C(11)	C(16)	C(15)	177.1(2)	C(12)	C(11)	C(16)	C(15)	-0.5(2)
C(11)	C(12)	C(13)	C(14)	1.7(3)	C(12)	C(13)	C(14)	C(15)	-1.7(3)
C(13)	C(14)	C(15)	C(16)	0.6(3)	C(14)	C(15)	C(16)	C(11)	0.6(3)
N(1)	C(17)	C(18)	C(19)	55.4(2)	N(1)	C(17)	C(18)	C(23)	-123.8(2)
C(10)	C(17)	C(18)	C(19)	-68.0(2)	C(10)	C(17)	C(18)	C(23)	112.8(2)
C(17)	C(18)	C(19)	C(20)	-177.0(1)	C(23)	C(18)	C(19)	C(20)	2.2(2)
C(17)	C(18)	C(23)	C(22)	177.6(2)	C(19)	C(18)	C(23)	C(22)	-1.6(2)
C(18)	C(19)	C(20)	C(21)	-1.1(2)	C(19)	C(20)	C(21)	C(22)	-0.7(3)
C(20)	C(21)	C(22)	C(23)	1.4(3)	C(21)	C(22)	C(23)	C(18)	-0.2(3)
N(1)	C(24)	C(25)	C(26)	-54.9(2)	N(1)	C(24)	C(25)	C(30)	128.1(2)
C(24)	C(25)	C(26)	C(27)	-178.0(1)	C(30)	C(25)	C(26)	C(27)	-1.0(2)
C(24)	C(25)	C(30)	C(29)	176.9(2)	C(26)	C(25)	C(30)	C(29)	-0.3(3)
C(25)	C(26)	C(27)	C(28)	1.4(3)	C(26)	C(27)	C(28)	C(29)	-0.5(3)
C(27)	C(28)	C(29)	C(30)	-0.7(3)	C(28)	C(29)	C(30)	C(25)	1.1(3)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
Ni(1)	S(1)	3.1755(4)	Ni(1)	S(2)	3.3343(4)
Ni(1)	O(3)	3.560(1)	Ni(1)	C(3)	3.080(2)
Ni(1)	C(10)	2.899(1)	Ni(1)	C(17)	2.889(2)
Ni(1)	C(24)	3.037(2)	S(1)	F(1)	2.606(1)
S(1)	F(2)	2.634(1)	S(1)	F(3)	2.611(1)
S(1)	O(8)	3.224(1)	S(1)	N(2)	3.554(1)

S(2)	F(4)	2.614(1)	S(2)	F(5)	2.609(1)
S(2)	F(6)	2.616(1)	S(2)	O(7)[1)]	3.563(1)
F(1)	F(2)	2.154(1)	F(1)	F(3)	2.156(2)
F(1)	O(2)	2.920(2)	F(1)	O(3)	3.004(1)
F(1)	O(5)[2)]	3.363(1)	F(1)	O(7)[3)]	3.559(2)
F(1)	C(19)[2)]	3.426(2)	F(1)	C(20)[2)]	3.463(2)
F(2)	F(3)	2.154(1)	F(2)	O(1)	2.916(1)
F(2)	O(2)	3.028(1)	F(2)	O(7)[3)]	3.355(2)
F(2)	C(21)[4)]	3.496(2)	F(2)	C(22)[4)]	3.427(2)
F(2)	C(24)[3)]	3.351(2)	F(2)	C(25)[3)]	3.264(2)
F(2)	C(26)[3)]	3.060(2)	F(3)	O(1)	3.000(1)
F(3)	O(3)	2.883(1)	F(3)	C(21)[4)]	3.430(2)
F(3)	C(22)[4)]	3.286(2)	F(3)	C(29)[4)]	3.430(2)
F(4)	F(5)	2.154(1)	F(4)	F(6)	2.155(2)
F(4)	O(4)	2.891(1)	F(4)	O(6)	2.995(2)
F(5)	F(6)	2.156(2)	F(5)	O(2)[5)]	3.030(2)
F(5)	O(5)	3.023(1)	F(5)	O(6)	2.913(1)
F(6)	O(4)	2.977(2)	F(6)	O(5)	2.914(2)
F(6)	C(10)	3.550(2)	O(1)	O(2)	2.423(2)
O(1)	O(3)	2.429(2)	O(1)	O(7)	2.873(2)
O(1)	O(8)	2.982(1)	O(1)	N(1)	2.933(2)
O(1)	N(2)	3.050(2)	O(1)	C(1)	2.581(2)
O(1)	C(17)	3.380(2)	O(1)	C(24)	3.118(2)
O(2)	F(5)[2)]	3.030(2)	O(2)	O(3)	2.443(2)
O(2)	O(6)[3)]	3.226(2)	O(2)	O(7)[3)]	2.741(2)
O(2)	O(8)	3.120(2)	O(2)	C(1)	2.595(2)
O(3)	O(8)	3.516(2)	O(3)	N(2)	3.063(2)
O(3)	C(1)	2.580(2)	O(3)	C(5)	3.111(2)
O(3)	C(15)[6)]	3.379(2)	O(4)	O(5)	2.424(1)
O(4)	O(6)	2.432(1)	O(4)	O(7)	2.995(2)
O(4)	O(8)	2.850(2)	O(4)	N(1)	3.069(2)
O(4)	N(2)	2.875(2)	O(4)	C(2)	2.571(2)
O(4)	C(3)	3.055(2)	O(4)	C(10)	3.366(2)
O(5)	F(1)[5)]	3.363(1)	O(5)	O(6)	2.441(2)
O(5)	O(7)[1)]	2.798(2)	O(5)	N(1)	3.163(2)
O(5)	C(2)	2.593(2)	O(5)	C(26)	3.482(2)
O(6)	O(2)[1)]	3.226(2)	O(6)	O(7)[1)]	3.434(2)
O(6)	O(8)[1)]	2.818(2)	O(6)	C(2)	2.575(2)
O(7)	S(2)[3)]	3.563(1)	O(7)	F(1)[1)]	3.559(2)
O(7)	F(2)[1)]	3.355(2)	O(7)	O(2)[1)]	2.741(2)
O(7)	O(5)[3)]	2.798(2)	O(7)	O(6)[3)]	3.434(2)
O(7)	O(8)	2.898(2)	O(7)	N(1)	2.975(2)
O(7)	C(24)	3.096(2)	O(8)	O(6)[3)]	2.818(2)
O(8)	N(2)	3.030(2)	O(8)	C(3)	3.200(2)
N(1)	N(2)	2.811(2)	N(1)	C(10)	2.460(2)
N(1)	C(18)	2.510(2)	N(1)	C(19)	3.069(2)
N(1)	C(25)	2.557(2)	N(1)	C(26)	3.127(2)
N(2)	C(4)	2.538(2)	N(2)	C(5)	3.249(2)
N(2)	C(9)	3.508(2)	N(2)	C(11)	2.507(2)
N(2)	C(12)	3.126(2)	N(2)	C(16)	3.533(2)
N(2)	C(17)	2.472(2)	C(3)	C(5)	2.511(2)
C(3)	C(9)	2.527(2)	C(3)	C(10)	2.488(2)
C(3)	C(11)	3.050(2)	C(4)	C(6)	2.420(2)
C(4)	C(7)	2.794(2)	C(4)	C(8)	2.413(2)
C(4)	C(10)	3.469(2)	C(4)	C(11)	3.449(2)
C(5)	C(7)	2.406(3)	C(5)	C(8)	2.763(2)
C(5)	C(9)	2.393(2)	C(6)	C(8)	2.392(3)
C(6)	C(9)	2.775(2)	C(7)	C(9)	2.407(3)
C(9)	C(11)	3.440(2)	C(9)	C(16)	3.494(2)
C(10)	C(12)	2.528(2)	C(10)	C(16)	2.517(2)
C(10)	C(18)	2.526(2)	C(10)	C(19)	3.165(2)
C(10)	C(23)	3.542(2)	C(11)	C(13)	2.418(3)
C(11)	C(14)	2.783(3)	C(11)	C(15)	2.418(2)
C(11)	C(17)	2.534(2)	C(11)	C(18)	2.962(2)
C(11)	C(23)	3.525(2)	C(12)	C(14)	2.400(3)
C(12)	C(15)	2.777(3)	C(12)	C(16)	2.401(2)
C(12)	C(17)	3.104(2)	C(12)	C(18)	3.548(2)
C(13)	C(15)	2.404(3)	C(13)	C(16)	2.775(3)
C(14)	C(16)	2.397(3)	C(15)	O(3)[7)]	3.379(2)
C(17)	C(19)	2.532(2)	C(17)	C(23)	2.519(2)

C(17)	C(24)	2.485(2)	C(17)	C(25)	3.276(2)
C(18)	C(20)	2.412(2)	C(18)	C(21)	2.782(2)
C(18)	C(22)	2.414(2)	C(18)	C(24)	3.074(2)
C(18)	C(25)	3.148(2)	C(18)	C(30)	3.591(2)
C(19)	F(1)[5)]	3.426(2)	C(19)	C(21)	2.401(2)
C(19)	C(22)	2.770(2)	C(19)	C(23)	2.395(2)
C(19)	C(25)	3.379(2)	C(19)	C(26)	3.395(2)
C(20)	F(1)[5)]	3.463(2)	C(20)	C(22)	2.399(2)
C(20)	C(23)	2.768(2)	C(21)	F(2)[4)]	3.496(2)
C(21)	F(3)[4)]	3.430(2)	C(21)	C(23)	2.392(2)
C(22)	F(2)[4)]	3.427(2)	C(22)	F(3)[4)]	3.286(2)
C(22)	C(28)[8)]	3.489(3)	C(23)	C(30)	3.590(3)
C(24)	F(2)[1)]	3.351(2)	C(24)	C(26)	2.532(2)
C(24)	C(30)	2.525(2)	C(25)	F(2)[1)]	3.264(2)
C(25)	C(27)	2.417(2)	C(25)	C(28)	2.796(2)
C(25)	C(29)	2.421(2)	C(26)	F(2)[1)]	3.060(2)
C(26)	C(28)	2.405(2)	C(26)	C(29)	2.767(2)
C(26)	C(30)	2.390(2)	C(27)	C(29)	2.393(2)
C(27)	C(30)	2.764(2)	C(28)	C(22)[9)]	3.489(3)
C(28)	C(30)	2.399(3)	C(29)	F(3)[4)]	3.430(2)

Symmetry Operators:

(1) X+1/2,-Y+1/2,Z	(2) X-1,Y,Z
(3) X+1/2-1,-Y+1/2,Z	(4) -X,-Y,-Z+1
(5) X+1,Y,Z	(6) X+1/2-1,-Y+1/2-1,Z
(7) X+1/2,-Y+1/2-1,Z	(8) -X+1/2,Y+1/2-1,-Z+1
(9) -X+1/2,Y+1/2,-Z+1	

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
Ni(1)	H(6)	2.9232	Ni(1)	H(7)	3.5863
Ni(1)	H(8)	2.45(2)	Ni(1)	H(9)	3.1115
Ni(1)	H(15)	3.0949	Ni(1)	H(21)	2.45(2)
Ni(1)	H(22)	2.9433	Ni(1)	H(23)	3.3452
Ni(1)	H(29)	2.55(2)	Ni(1)	H(30)	2.56(2)
Ni(1)	H(31)	2.50(2)	Ni(1)	H(32)	2.53(2)
S(1)	H(8)	3.04(2)	S(1)	H(30)[1)]	3.15(2)
S(1)	H(32)	3.14(2)	S(2)	H(9)	3.5929
S(2)	H(21)	3.00(2)	S(2)	H(29)[2)]	2.84(2)
S(2)	H(30)	3.33(2)	S(2)	H(32)[2)]	3.20(2)
F(1)	H(12)[3)]	2.6998	F(1)	H(19)[4)]	2.8555
F(1)	H(20)[4)]	2.7809	F(1)	H(29)[1)]	3.29(2)
F(1)	H(30)[1)]	3.27(2)	F(2)	H(17)[5)]	2.7669
F(2)	H(18)[5)]	2.9033	F(2)	H(22)[1)]	2.6319
F(2)	H(24)[1)]	2.9105	F(2)	H(25)[4)]	3.2047
F(2)	H(29)[1)]	3.52(2)	F(2)	H(30)[1)]	3.09(2)
F(3)	H(12)[3)]	3.1079	F(3)	H(17)[5)]	2.9013
F(3)	H(18)[5)]	3.1625	F(3)	H(19)[4)]	3.1540
F(3)	H(27)[5)]	2.9009	F(4)	H(4)[6)]	3.3984
F(4)	H(7)	3.3810	F(5)	H(29)[2)]	3.26(2)
F(5)	H(30)[2)]	3.29(2)	F(6)	H(4)[6)]	3.0539
F(6)	H(7)	3.1399	F(6)	H(9)	2.6150
F(6)	H(12)[6)]	3.5920	F(6)	H(14)	3.2784
F(6)	H(20)	3.1877	O(1)	H(8)	2.81(2)
O(1)	H(15)	2.9581	O(1)	H(22)	3.0180
O(1)	H(23)	2.8326	O(1)	H(24)[1)]	3.5174
O(1)	H(29)	2.85(2)	O(1)	H(32)	3.01(2)
O(2)	H(29)[1)]	3.01(2)	O(2)	H(30)[1)]	1.99(2)
O(2)	H(32)	2.72(2)	O(3)	H(1)	2.6863
O(3)	H(8)	2.34(2)	O(3)	H(10)	3.3835
O(3)	H(12)[3)]	3.1960	O(3)	H(13)[3)]	2.5922
O(4)	H(6)	2.6627	O(4)	H(7)	3.1040
O(4)	H(9)	2.9594	O(4)	H(21)	2.74(2)
O(4)	H(30)	2.96(2)	O(4)	H(31)	2.62(2)
O(4)	H(32)	3.46(2)	O(5)	H(20)	3.1253
O(5)	H(21)	2.29(2)	O(5)	H(24)	2.5878
O(5)	H(29)[2)]	1.99(2)	O(5)	H(30)	3.41(2)
O(5)	H(30)[2)]	3.10(2)	O(5)	H(32)[2)]	3.48(2)
O(6)	H(29)[2)]	2.92(2)	O(6)	H(30)	3.53(2)
O(6)	H(31)[2)]	2.99(2)	O(6)	H(32)[2)]	2.05(2)
O(7)	H(21)	3.13(2)	O(7)	H(22)	2.4080

O(7)	H(23)	3.5998	O(7)	H(31)	3.17(2)
O(7)	H(32)	2.84(2)	O(8)	H(1)	3.0356
O(8)	H(6)	2.5615	O(8)	H(8)	3.18(2)
O(8)	H(29)	3.02(2)	O(8)	H(30)	3.31(2)
N(1)	H(8)	3.21(2)	N(1)	H(9)	2.6232
N(1)	H(15)	1.9871	N(1)	H(20)	2.9082
N(1)	H(22)	2.0013	N(1)	H(23)	1.9964
N(1)	H(24)	2.9221	N(1)	H(29)	3.59(2)
N(1)	H(30)	3.33(2)	N(2)	H(1)	3.1715
N(2)	H(6)	2.0087	N(2)	H(7)	2.0125
N(2)	H(9)	1.9985	N(2)	H(10)	3.0136
N(2)	H(15)	2.6416	N(2)	H(21)	3.21(2)
N(2)	H(31)	3.37(2)	C(1)	H(12)[3)]	3.3356
C(1)	H(17)[5)]	3.3887	C(1)	H(18)[5)]	3.4572
C(1)	H(19)[4)]	3.5239	C(1)	H(30)[1)]	3.32(2)
C(2)	H(9)	3.5266	C(3)	H(1)	2.6554
C(3)	H(5)	2.6851	C(3)	H(8)	1.86(2)
C(3)	H(9)	2.5982	C(3)	H(31)	3.27(2)
C(4)	H(1)	2.0302	C(4)	H(2)	3.2751
C(4)	H(4)	3.2683	C(4)	H(5)	2.0379
C(4)	H(6)	2.0178	C(4)	H(7)	2.0249
C(4)	H(8)	2.40(2)	C(5)	H(2)	2.0461
C(5)	H(3)	3.2645	C(5)	H(5)	3.2520
C(5)	H(6)	2.6340	C(5)	H(7)	3.2564
C(5)	H(8)	2.88(2)	C(5)	H(13)[3)]	3.4271
C(6)	H(1)	2.0421	C(6)	H(3)	2.0330
C(6)	H(4)	3.2495	C(6)	H(5)[3)]	3.1604
C(6)	H(13)[3)]	3.4328	C(7)	H(1)	3.2625
C(7)	H(2)	2.0359	C(7)	H(4)	2.0264
C(7)	H(5)	3.2609	C(7)	H(5)[3)]	3.0010
C(7)	H(14)[3)]	3.4487	C(8)	H(2)	3.2510
C(8)	H(2)[6)]	3.5881	C(8)	H(3)	2.0331
C(8)	H(5)	2.0391	C(8)	H(14)[3)]	3.4258
C(9)	H(1)	3.2506	C(9)	H(2)[6)]	3.4850
C(9)	H(3)	3.2657	C(9)	H(3)[6)]	3.2094
C(9)	H(4)	2.0375	C(9)	H(6)	3.1780
C(9)	H(7)	2.5630	C(9)	H(8)	3.38(2)
C(10)	H(6)	3.1750	C(10)	H(7)	2.4334
C(10)	H(8)	1.93(2)	C(10)	H(10)	2.6786
C(10)	H(14)	2.6676	C(10)	H(15)	2.0376
C(10)	H(20)	3.0418	C(10)	H(21)	2.72(2)
C(11)	H(5)	3.0561	C(11)	H(7)	2.8651
C(11)	H(8)	2.58(2)	C(11)	H(9)	2.0206
C(11)	H(10)	2.0375	C(11)	H(11)	3.2769
C(11)	H(13)	3.2743	C(11)	H(14)	2.0378
C(11)	H(15)	2.7185	C(11)	H(16)	3.5556
C(12)	H(5)	3.5110	C(12)	H(8)	2.81(2)
C(12)	H(9)	3.2820	C(12)	H(11)	2.0437
C(12)	H(12)	3.2592	C(12)	H(13)[3)]	3.2500
C(12)	H(14)	3.2600	C(12)	H(15)	2.8357
C(12)	H(16)	3.3142	C(13)	H(10)	2.0397
C(13)	H(12)	2.0348	C(13)	H(13)	3.2613
C(13)	H(13)[3)]	3.5355	C(13)	H(14)[3)]	3.0728
C(13)	H(26)[7)]	3.0472	C(14)	H(5)	3.4763
C(14)	H(10)	3.2579	C(14)	H(11)	2.0327
C(14)	H(13)	2.0339	C(14)	H(14)	3.2522
C(14)	H(19)[3)]	3.4912	C(14)	H(26)[7)]	3.2441
C(14)	H(27)[7)]	3.4306	C(15)	H(5)	3.0173
C(15)	H(10)[6)]	3.3693	C(15)	H(11)	3.2585
C(15)	H(11)[6)]	3.3862	C(15)	H(12)	2.0242
C(15)	H(14)	2.0351	C(15)	H(27)[7)]	3.4463
C(16)	H(5)	2.7805	C(16)	H(7)	3.0761
C(16)	H(9)	2.5260	C(16)	H(10)	3.2577
C(16)	H(11)[6)]	3.0366	C(16)	H(12)	3.2520
C(16)	H(13)	2.0413	C(17)	H(8)	2.72(2)
C(17)	H(9)	2.0318	C(17)	H(10)	2.9340
C(17)	H(16)	2.6615	C(17)	H(20)	2.6800
C(17)	H(21)	2.01(2)	C(17)	H(22)	3.2491
C(17)	H(23)	2.5175	C(18)	H(9)	2.7049
C(18)	H(10)	3.5988	C(18)	H(15)	2.0215

C(18)	H(16)	2.0327	C(18)	H(17)	3.2694
C(18)	H(19)	3.2699	C(18)	H(20)	2.0320
C(18)	H(21)	2.66(2)	C(18)	H(23)	3.1354
C(19)	H(9)	2.8988	C(19)	H(11)[6)]	3.2329
C(19)	H(12)[6)]	3.5798	C(19)	H(15)	3.2802
C(19)	H(16)	3.2520	C(19)	H(18)	3.2591
C(19)	H(19)	2.0384	C(19)	H(21)	2.76(2)
C(19)	H(24)	3.4749	C(20)	H(11)[6)]	3.0879
C(20)	H(12)[6)]	3.4051	C(20)	H(17)	3.2571
C(20)	H(18)	2.0390	C(20)	H(20)	2.0366
C(20)	H(26)[8)]	3.2206	C(21)	H(16)	3.2487
C(21)	H(17)	2.0269	C(21)	H(19)	2.0377
C(21)	H(19)[8)]	3.5987	C(21)	H(20)	3.2603
C(21)	H(25)[8)]	3.4154	C(21)	H(26)[8)]	3.2311
C(22)	H(16)	2.0356	C(22)	H(18)	2.0226
C(22)	H(19)	3.2540	C(23)	H(15)	2.5231
C(23)	H(17)	2.0367	C(23)	H(18)	3.2491
C(23)	H(20)	3.2509	C(23)	H(23)	3.5309
C(23)	H(28)	3.4755	C(23)	H(28)[5)]	3.2516
C(24)	H(15)	2.5972	C(24)	H(21)	1.98(2)
C(24)	H(24)	2.6782	C(24)	H(25)[1)]	3.0271
C(24)	H(28)	2.6719	C(24)	H(29)	3.51(2)
C(24)	H(30)	3.45(2)	C(25)	H(15)	3.5395
C(25)	H(20)	3.5575	C(25)	H(21)	2.60(2)
C(25)	H(22)	2.0207	C(25)	H(23)	2.0224
C(25)	H(24)	2.0281	C(25)	H(25)	3.2728
C(25)	H(25)[1)]	3.3049	C(25)	H(27)	3.2763
C(25)	H(28)	2.0374	C(26)	H(17)[9)]	3.2843
C(26)	H(20)	3.3204	C(26)	H(21)	2.72(2)
C(26)	H(22)	2.7841	C(26)	H(23)	3.2809
C(26)	H(25)	2.0403	C(26)	H(26)	3.2638
C(26)	H(28)	3.2490	C(27)	H(17)[9)]	2.9460
C(27)	H(18)[8)]	2.9394	C(27)	H(22)[2)]	3.4178
C(27)	H(23)[2)]	3.5102	C(27)	H(24)	2.0389
C(27)	H(26)	2.0375	C(27)	H(27)	3.2505
C(28)	H(17)[9)]	3.1161	C(28)	H(18)[8)]	3.0783
C(28)	H(19)[8)]	3.2512	C(28)	H(24)	3.2620
C(28)	H(25)	2.0330	C(28)	H(27)	2.0276
C(28)	H(28)	3.2532	C(29)	H(17)[9)]	3.5870
C(29)	H(25)	3.2497	C(29)	H(26)	2.0295
C(29)	H(28)	2.0330	C(30)	H(16)[5)]	3.3002
C(30)	H(22)	3.0564	C(30)	H(23)	2.5346
C(30)	H(24)	3.2470	C(30)	H(25)[1)]	3.3207
C(30)	H(26)	3.2569	C(30)	H(27)	2.0384
H(2)	C(8)[3)]	3.5881	H(2)	C(9)[3)]	3.4850
H(3)	C(9)[3)]	3.2094	H(4)	F(4)[3)]	3.3984
H(4)	F(6)[3)]	3.0539	H(5)	C(6)[6)]	3.1604
H(5)	C(7)[6)]	3.0010	H(8)	Ni(1)	2.45(2)
H(8)	S(1)	3.04(2)	H(8)	O(1)	2.81(2)
H(8)	O(3)	2.34(2)	H(8)	O(8)	3.18(2)
H(8)	N(1)	3.21(2)	H(8)	C(3)	1.86(2)
H(8)	C(4)	2.40(2)	H(8)	C(5)	2.88(2)
H(8)	C(9)	3.38(2)	H(8)	C(10)	1.93(2)
H(8)	C(11)	2.58(2)	H(8)	C(12)	2.81(2)
H(8)	C(17)	2.72(2)	H(10)	C(15)[3)]	3.3693
H(11)	C(11)	3.2769	H(11)	C(12)	2.0437
H(11)	C(14)	2.0327	H(11)	C(15)	3.2585
H(11)	C(15)[3)]	3.3862	H(11)	C(16)[3)]	3.0366
H(11)	C(19)[3)]	3.2329	H(11)	C(20)[3)]	3.0879
H(12)	F(1)[6)]	2.6998	H(12)	F(3)[6)]	3.1079
H(12)	F(6)[3)]	3.5920	H(12)	O(3)[6)]	3.1960
H(12)	C(1)[6)]	3.3356	H(12)	C(19)[3)]	3.5798
H(12)	C(20)[3)]	3.4051	H(13)	O(3)[6)]	2.5922
H(13)	C(5)[6)]	3.4271	H(13)	C(6)[6)]	3.4328
H(13)	C(12)[6)]	3.2500	H(13)	C(13)[6)]	3.5355
H(14)	C(7)[6)]	3.4487	H(14)	C(8)[6)]	3.4258
H(14)	C(13)[6)]	3.0728	H(16)	C(30)[5)]	3.3002
H(17)	F(2)[5)]	2.7669	H(17)	F(3)[5)]	2.9013
H(17)	C(1)[5)]	3.3887	H(17)	C(26)[7)]	3.2843
H(17)	C(27)[7)]	2.9460	H(17)	C(28)[7)]	3.1161

H(17)	C(29)[7)]	3.5870	H(18)	F(2)[5)]	2.9033
H(18)	F(3)[5)]	3.1625	H(18)	C(1)[5)]	3.4572
H(18)	C(27)[8)]	2.9394	H(18)	C(28)[8)]	3.0783
H(19)	F(1)[10)]	2.8555	H(19)	F(3)[10)]	3.1540
H(19)	C(1)[10)]	3.5239	H(19)	C(14)[6)]	3.4912
H(19)	C(21)[8)]	3.5987	H(19)	C(28)[8)]	3.2512
H(20)	F(1)[10)]	2.7809	H(22)	F(2)[2)]	2.6319
H(22)	C(27)[1)]	3.4178	H(23)	C(27)[1)]	3.5102
H(24)	F(2)[2)]	2.9106	H(24)	O(1)[2)]	3.5174
H(25)	F(2)[10)]	3.2047	H(25)	C(21)[8)]	3.4154
H(25)	C(24)[2)]	3.0271	H(25)	C(25)[2)]	3.3049
H(25)	C(30)[2)]	3.3207	H(26)	C(13)[9)]	3.0472
H(26)	C(14)[9)]	3.2441	H(26)	C(20)[8)]	3.2206
H(26)	C(21)[8)]	3.2311	H(27)	F(3)[5)]	2.9009
H(27)	C(14)[9)]	3.4306	H(27)	C(15)[9)]	3.4463
H(28)	C(23)[5)]	3.2516	H(29)	S(2)[1)]	2.84(2)
H(29)	F(1)[2)]	3.29(2)	H(29)	F(2)[2)]	3.52(2)
H(29)	F(5)[1)]	3.26(2)	H(29)	O(2)[2)]	3.01(2)
H(29)	O(5)[1)]	1.99(2)	H(29)	O(6)[1)]	2.92(2)
H(30)	S(1)[2)]	3.15(2)	H(30)	F(1)[2)]	3.27(2)
H(30)	F(2)[2)]	3.09(2)	H(30)	F(5)[1)]	3.29(2)
H(30)	O(2)[2)]	1.99(2)	H(30)	O(5)[1)]	3.10(2)
H(30)	C(1)[2)]	3.32(2)	H(31)	O(6)[1)]	2.99(2)
H(32)	S(2)[1)]	3.20(2)	H(32)	O(5)[1)]	3.48(2)
H(32)	O(6)[1)]	2.05(2)			

Symmetry Operators:

(1) X+1/2-1,-Y+1/2,Z	(2) X+1/2,-Y+1/2,Z
(3) X+1/2-1,-Y+1/2-1,Z	(4) X-1,Y,Z
(5) -X,-Y,-Z+1	(6) X+1/2,-Y+1/2-1,Z
(7) -X+1/2,Y+1/2-1,-Z+1	(8) -X+1,-Y,-Z+1
(9) -X+1/2,Y+1/2,-Z+1	(10) X+1,Y,Z