Spectroscopic characterization

UV-Visible spectrum

The structural characteristics of **1** and the concentration-dependent availability of the compound in aqueous solution were investigated at ambient temperature across the 200-1100 nm wavelength range using a Cary 5000 UV-Vis-NIR spectrophotometer from Agilent Technologies. Solutions of the compound were prepared at concentrations ranging from 0.1 M to 0.7 M. The resulting UV-Vis-NIR spectra are shown in Figs. S1 and S2.

In the visible region of the spectrum, two absorption maxima are observed. The band at λ_{max}=393 nm (25445 cm^{-1}) is assigned to the $^3A_{2g}\rightarrow{}^3T_{1g}(P)$ d-d transition of the nickel cation. The near-infrared absorption at λ_{max}=726 nm (13761 cm^{-1}) is associated with the $^3A_{2g}\rightarrow{}^3T_{2g}(F)$ and $^3A_{2g}\rightarrow{}^1E_g$ d-d transitions of Ni(II) in an octahedral coordination environment (Fig. S1). [1, 2].

The consistent positions of the observed absorption maxima across the tested concentration range indicate that the structural characteristics of the compound remain stable and that the compound maintains its chemical integrity in aqueous solutions.

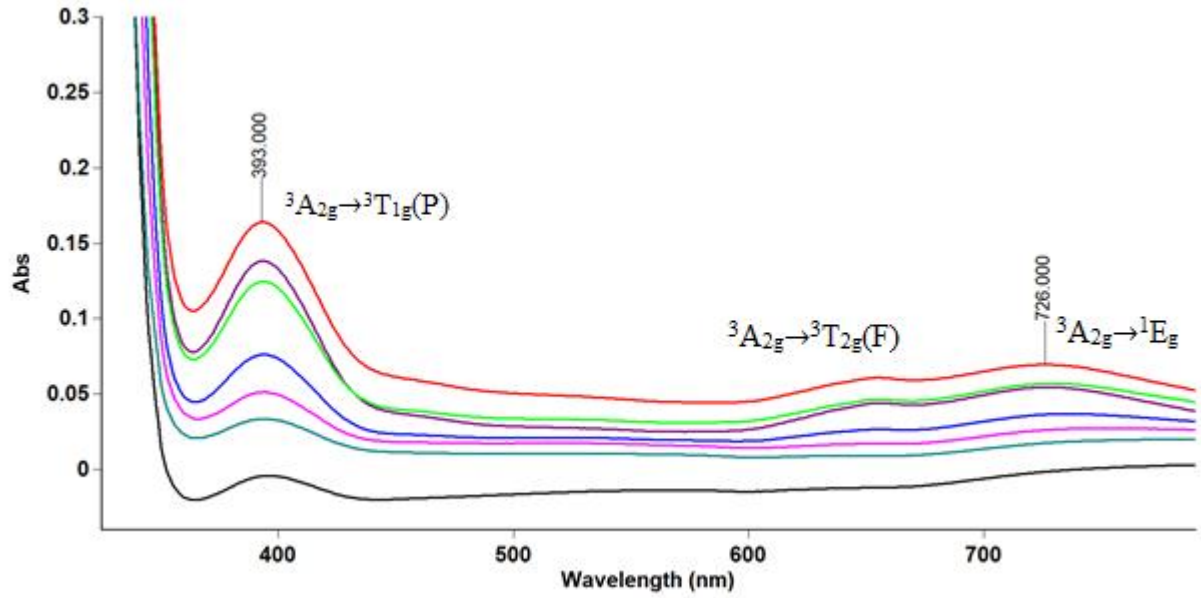


Figure S1. Spectra (350-800 nm) of solutions of **1** in the concentration range between 0.1 M and 0.7 M show the presence of the nickelhexaaqua cation.

The ultraviolet absorption spectrum exhibits two prominent peaks. The maximum at λ_{max}=215 nm (45512 cm^{-1}) is attributed to n→π* electronic transitions within the sulfosalicylic ion moiety, whereas the absorption band centered at λ_{max}=311 nm (32154 cm^{-1}) corresponds to π→π* transitions in the aromatic ring system (Fig. S2).

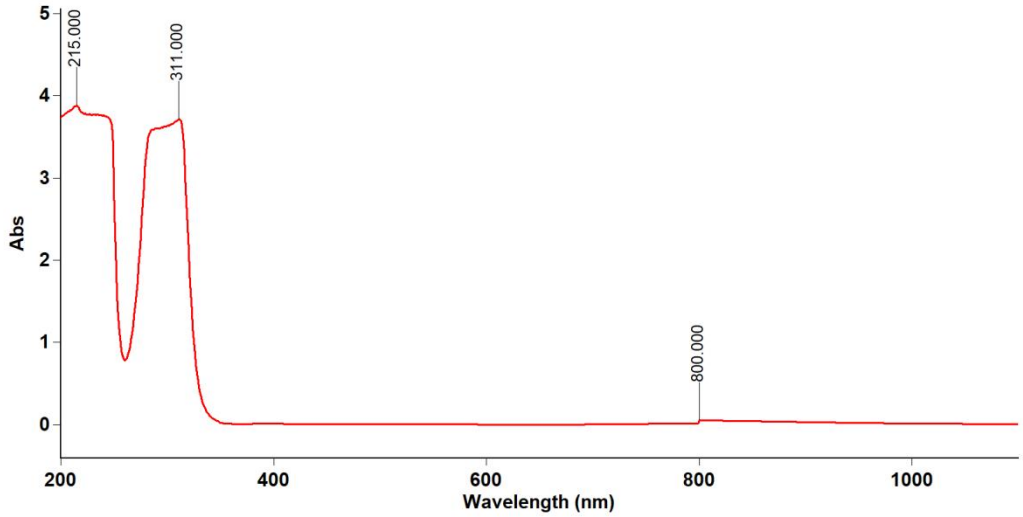


Figure S2. UV absorption maxima characteristic for the organic anion in **1**.

Infrared spectrum

The infrared (IR) spectrum of **1** in the range of 4000-400 cm^{-1} was registered on a Bruker Invenio S-2021 device. For comparison, the IR spectrum of sulfosalicylic acid as measured under the same conditions. Sulfosalicylic acid and the functional groups in the anion of **1** have the same characteristic vibrations, but their intensities differ.

In the IR spectrum of sulfosalicylic acid (Fig. S3), the hydrogen-bonded –OH group undergoes valence vibrations at around 3105 cm^{-1}, while the C-H bonds of the aromatic ring undergo valence vibrations at approximately 3066 cm^{-1}. Both of these functional group vibrations are shown with high intensity in the infrared spectra of sulfosalicylic acid. In the IR spectrum of **1** these vibrations appear with only average absorption intensities (3111 cm^{-1} for -OH, 3071 cm^{-1} for C-H in the aromatic ring) (Fig. S4).

In the IR spectrum of sulfosalicylic acid the carboxylato group is associated with strong bands at 1659 cm^{-1} for the asymmetric and 1604 cm^{-1} for the symmetric vibration. In **1**, the corresponding vibrations are observed at 1660 cm^{-1} (ν_{as}) and 1607 cm^{-1} (ν_s) with inferior intensities.

The sulfonato group in sulfosalicylic acid is characterized by valence vibrations ν_{as} at 1478 cm^{-1} and (ν_s) at 1433 cm^{-1} and by deformation vibrations at 1366 cm^{-1} (δ_{as}) and 1315 cm^{-1} (δ_s). In **1**, the corresponding bands are located at very similar wave numbers of 1479, 1431, 1364 and 1348 cm^{-1}.

In sulfosalicylic acid, the deformation vibrations of the C-H bond are manifested at 1150 cm^{-1} (δ_{as}) and 1117 cm^{-1} (δ_s); the corresponding vibrations in **1** are encountered at 1152 cm^{-1} (δ_{as}) and 1122 cm^{-1} (δ_s).

The main difference between the spectra is caused by the vibrations of the Ni-O bonds; they are associated with a strong band around 500 cm^{-1}.

The comparison between the IR spectra of sulfosalicylic acid and **1** shows that bands associated with the carboxylato and sulfonato groups occur in the same spectral regions. This indicates that the sulfosalicylato anion is present in the external sphere of the coordination compound, rather than being directly coordinated to the metal center.

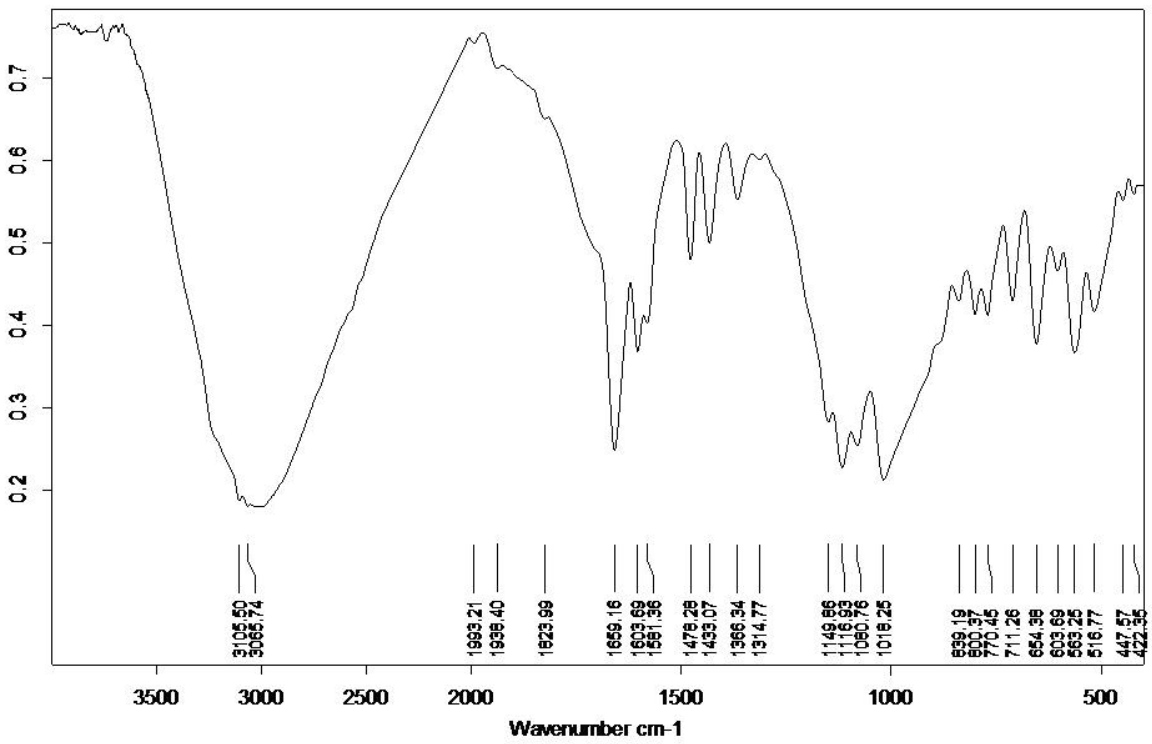


Figure S3. IR spectrum of sulfosalicylic acid

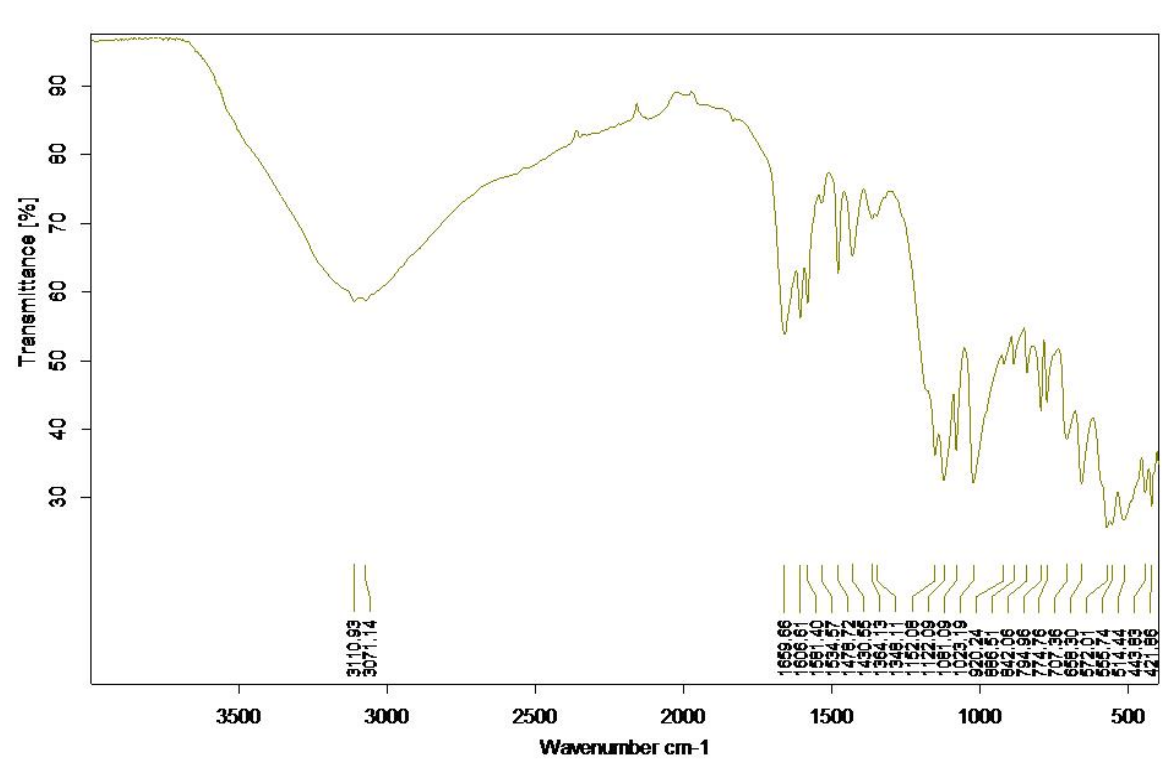


Figure S4. IR spectrum of **1**

1. Liu, W., Migdisov, A., & Williams-Jones, A. (2012). The stability of aqueous nickel (II) chloride complexes in hydrothermal solutions: Results of UV–Visible spectroscopic experiments. *Geochimica et Cosmochimica Acta*, *94*, 276-290. http://dx.doi.org/10.1016/j.gca.2012.04.055

2. Rasyda, Y. A., Rahardjo, S. B., & Nurdiyah, F. (2019, September). Synthesis and characterization complex nickel (II) with diphenylamine. In *IOP Conference Series: Materials Science and Engineering* (Vol. 578, No. 1, p. 012008). IOP Publishing. doi:10.1088/1757-899X/578/1/012008

Data completeness and quality

Table S1: Resolution and completeness statistics (cumulative and Friedel pairs averaged) of **1**

Resolution & Completeness Statistics (Cumulative and Friedel Pairs Averaged)

Theta	sin(th)/Lambda	Complete	Expected	Measured	Total	Missing
20.82	0.500	0.998	1277	1274		3
23.01	0.550	0.998	1691	1688		3
25.24	0.600	0.998	2190	2185		5
27.51	0.650	0.998	2805	2800		5
29.84	0.700	0.999	3499	3494		5
32.21	0.750	0.999	4284	4279		5
34.65	0.800	0.999	5205	5200		5
37.17	0.850	0.999	6249	6244		5
39.77	0.900	0.999	7403	7398		5
42.47	0.950	0.999	8708	8703		5
45.29	1.000	1.000	10173	10168		5
48.27	1.050	0.999	11778	11772		6
51.43	1.100	0.999	13536	13528		8
54.82	1.150	0.999	15430	15418		12
58.50	1.200	0.996	17538	17461		77

Note: The Reported Completeness refers to the Actual H,K,L Index Range

Missing Reflections (Asym. Refl. Unit) below sin(th)/lambda = 0.600

Nr	H	K	L	sin(th)/lambda	Theta	I(calc)	I(calc)/I(max
1	2	0	0	0.156	6.35	11598.50	1.03086
2	0	0	1	0.039	1.60*	134.98	0.01200
3	0	1	3	0.120	4.89	9778.25	0.86908
4	5	-1	9	0.562	23.54	994.03	0.08835
5	6	0	9	0.595	25.00	508.20	0.04517

** Note: I(max) is the maximum I(obs) encountered in the fcf-file **

Starred Reflections have a Theta below Theta(Min) = 2.93

From CIF: Theta(Min) = 2.93

Table S2: *R*-value statistics as a function of resolution (in resolution shells) of **1**

R-Value Statistics as a Function of Resolution (in Resolution Shell)

Theta	sin(Th)/L	#	R1	wR2	S	Rs	av(I/SigW)	av(I)	av(SigW)
12.38	0.302	274	0.057	0.174	3.898	0.010	22.08	1310.34	56.83
15.68	0.380	275	0.048	0.118	2.520	0.008	20.89	712.85	30.58
18.02	0.435	292	0.033	0.094	1.901	0.009	19.48	524.25	23.23
19.90	0.479	279	0.028	0.095	1.808	0.010	17.96	382.26	17.42
21.51	0.516	275	0.023	0.091	1.605	0.014	16.56	298.07	14.43
22.94	0.548	277	0.032	0.093	1.556	0.021	15.58	251.91	13.18
24.22	0.577	289	0.037	0.093	1.518	0.023	15.32	214.78	11.61
25.40	0.603	251	0.032	0.085	1.448	0.017	16.41	163.61	8.42
26.49	0.628	302	0.025	0.069	1.219	0.015	16.89	164.60	8.22
27.52	0.650	287	0.024	0.066	1.143	0.014	16.67	153.02	7.64
28.49	0.671	253	0.020	0.059	1.019	0.014	16.31	130.33	6.51
29.41	0.691	282	0.020	0.056	0.947	0.013	16.01	127.58	6.34
30.28	0.709	303	0.020	0.054	0.906	0.013	15.90	105.66	5.29
31.12	0.727	271	0.019	0.057	0.970	0.013	16.17	101.80	5.11
31.93	0.744	254	0.016	0.046	0.803	0.012	16.61	107.26	5.35
32.71	0.760	289	0.016	0.047	0.802	0.013	16.08	108.90	5.42
33.46	0.776	290	0.016	0.054	0.899	0.013	15.79	109.29	5.42
34.20	0.791	283	0.015	0.047	0.794	0.013	15.70	83.70	4.25
34.91	0.805	276	0.016	0.047	0.784	0.013	15.52	80.31	4.09
58.50	1.200	12159	0.022	0.065	0.884	0.024	11.85	33.41	2.01

R(sig) = sum(sig(I)) / sum(I) = 0.0147

From FCF: R1 = 0.0266(15965), wR2 = 0.0760(17461), S = 1.148
From CIF: R1 = 0.0266(15967), wR2 = 0.0760(17461), S = 1.147, Npar = 209

No (SHELXL) Optimized Weights: wR2 = 0.0657 , S = 3.46

Details of the multipole refinement

Refinement was conducted with all intensity data $I > 0$. ADPs for H Atoms were from obtained from the shade-server [3,4]. The final refinements on F^2 comprised multipoles up to hexadecapoles for non-H atoms, up to bond directed dipoles for the carbon-bonded H atoms and up to quadrupoles for the H atoms bonded to oxygen. Contraction parameters were refined freely, starting with a value of 2.0 for Ni; κ' for the H atoms was fixed to 1.2. Contraction parameters and monopole population coefficients have been compiled in Table S6. The positional parameters of H atoms were refined freely.

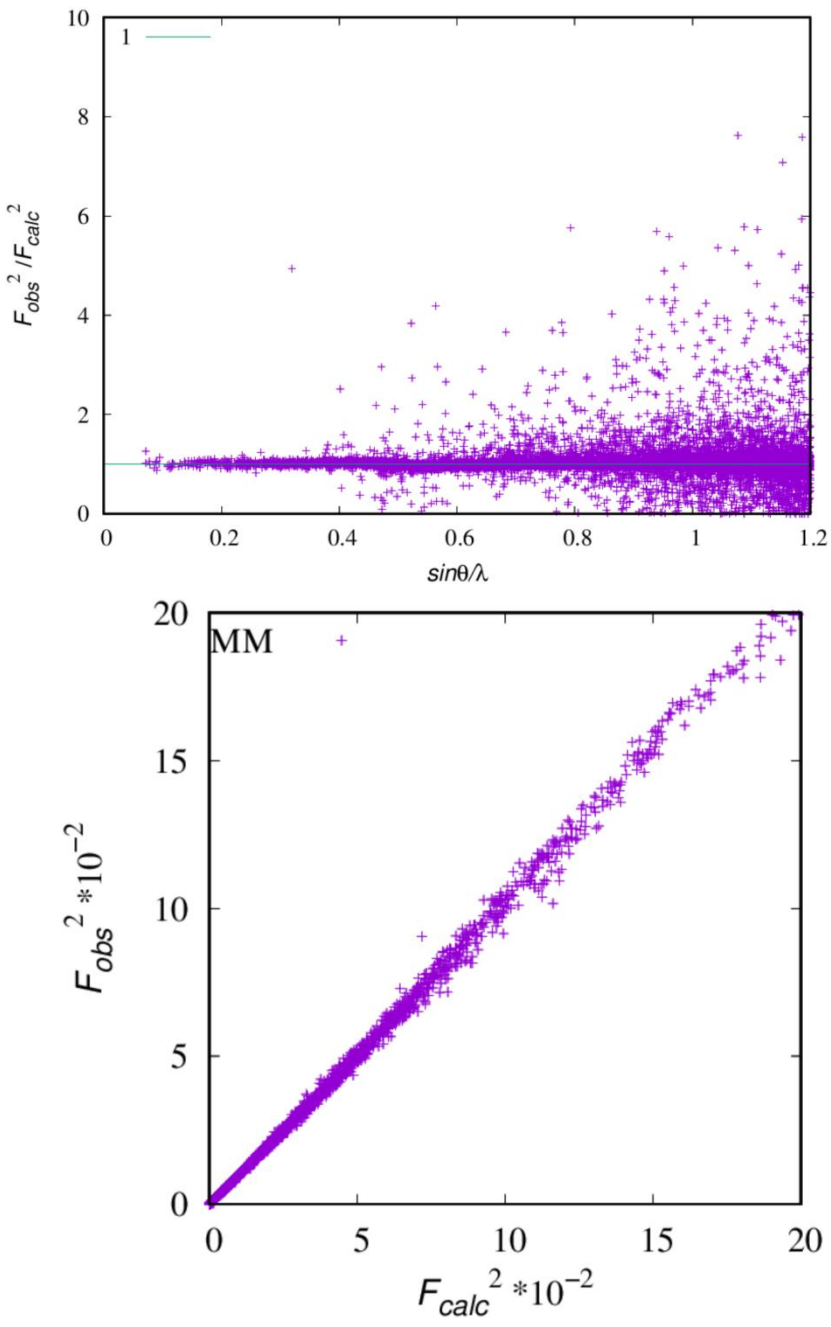


Figure S5: Scatterplot for X-ray refinement result (MM) for 1

Table S3: Final Coordinates and equivalent isotropic displacement parameters of the atoms in **1**

NI(1)	0.5	0.5	0.5	0.008
S(2)	0.217959(7)	0.977102(7)	0.702091(4)	0.008
O(1)	0.33698(3)	0.77873(3)	0.449778(17)	0.012
O(2)	0.40281(3)	0.49397(3)	0.647753(13)	0.011
O(3)	0.23408(3)	0.41368(3)	0.473345(15)	0.012
O(4)	0.15817(5)	0.79303(5)	0.71370(3)	0.012
O(5)	0.43275(5)	0.97011(5)	0.66890(3)	0.013
O(6)	0.07442(5)	1.14096(5)	0.63579(2)	0.013
O(7)	0.26933(4)	1.10457(4)	1.118979(19)	0.015
O(8)	0.31545(5)	0.73831(4)	1.198649(18)	0.014
O(9)	0.27695(4)	0.55174(3)	1.096095(17)	0.015
O(10)	0.22222(3)	0.32025(2)	0.281972(13)	0.012
C(1)	0.21489(3)	1.01161(3)	0.828520(14)	0.009
C(2)	0.19858(3)	1.19806(3)	0.838679(16)	0.011
C(3)	0.21646(3)	1.22636(3)	0.936766(17)	0.012
C(4)	0.25033(3)	1.06876(3)	1.026290(15)	0.010
C(5)	0.26111(3)	0.88185(3)	1.016124(14)	0.010
C(6)	0.24411(3)	0.85505(3)	0.916245(14)	0.010
C(7)	0.28721(3)	0.71848(3)	1.110752(14)	0.011
H(1WA)	0.192428	0.801779	0.41573	0.022
H(1WB)	0.414391	0.876642	0.40967	0.022
H(2WA)	0.332316	0.621994	0.660913	0.021
H(2WB)	0.51067	0.425971	0.707936	0.022
H(3WA)	0.217216	0.377172	0.405909	0.023
H(3WB)	0.170225	0.325862	0.530231	0.024
H(7O)	0.295401	0.975387	1.174825	0.023
H(9O)	0.281911	0.451574	1.169506	0.026
H(2C)	0.171633	1.32085	0.769247	0.026
H(3C)	0.205366	1.37073	0.945	0.027
H(6C)	0.252368	0.710251	0.907922	0.023
H(10A)	0.094136	0.261502	0.281036	0.027
H(10B)	0.349857	0.214131	0.301276	0.027

Table S4: Anisotropic displacement parameters of the non-hydrogen atoms in **1**

Atom Label	U11	U22	U33	U12	U13	U23
NI(1)	0.007891(12)	0.008225(13)	0.006289(11)	-0.002003(9)	-0.000356(8)	-0.000931(8)
S(2)	0.007328(16)	0.009456(17)	0.007553(16)	-0.001934(11)	0.000366(10)	-0.000760(11)
O(1)	0.01051(5)	0.00988(5)	0.01395(6)	-0.00173(4)	-0.00102(4)	0.00014(4)
O(2)	0.01316(6)	0.01245(6)	0.00767(4)	-0.00180(5)	0.00041(4)	-0.00201(4)
O(3)	0.01162(5)	0.01547(6)	0.01003(5)	-0.00614(5)	0.00035(4)	-0.00318(5)
O(4)	0.01365(9)	0.01261(9)	0.01246(9)	-0.00550(8)	0.00257(7)	-0.00428(7)
O(5)	0.00899(8)	0.01407(9)	0.01244(9)	-0.00222(7)	0.00249(7)	-0.00046(8)
O(6)	0.01070(8)	0.01414(9)	0.01039(8)	-0.00052(8)	-0.00212(7)	0.00022(7)
O(7)	0.01733(8)	0.01740(8)	0.01199(6)	-0.00517(7)	-0.00059(6)	-0.00638(6)
O(8)	0.01640(8)	0.01687(8)	0.00831(6)	-0.00297(7)	-0.00223(6)	-0.00130(6)
O(9)	0.02131(8)	0.01110(6)	0.01012(6)	-0.00221(6)	-0.00028(6)	-0.00012(5)
O(10)	0.01258(5)	0.01151(5)	0.01166(5)	-0.00316(4)	-0.00045(4)	-0.00282(4)
C(1)	0.00945(5)	0.00914(5)	0.00829(5)	-0.00230(4)	-0.00001(4)	-0.00087(4)
C(2)	0.01231(6)	0.00943(5)	0.01086(5)	-0.00325(4)	0.00034(4)	-0.00108(4)
C(3)	0.01332(6)	0.01038(6)	0.01271(6)	-0.00394(5)	0.00038(5)	-0.00313(5)
C(4)	0.00971(5)	0.01193(6)	0.01027(5)	-0.00301(4)	-0.00012(4)	-0.00356(4)
C(5)	0.00950(5)	0.01040(5)	0.00813(5)	-0.00179(4)	-0.00048(4)	-0.00141(4)
C(6)	0.01111(5)	0.00917(5)	0.00813(5)	-0.00196(4)	-0.00023(4)	-0.00106(4)
C(7)	0.01058(5)	0.01218(6)	0.00803(5)	-0.00128(4)	-0.00074(4)	-0.00066(4)

Table S5: Anisotropic displacement parameters of the hydrogen atoms in the **1** （SHADEserver）

Atom Label	U11	U22	U33	U12	U13	U23
H(1WA)	0.015685	0.026813	0.021745	-0.003153	-0.00459	-0.003911
H(1WB)	0.023212	0.02057	0.020745	-0.009009	0.003199	-0.000253
H(2WA)	0.022787	0.017326	0.022747	-0.001913	0.00066	-0.006893
H(2WB)	0.022654	0.022863	0.016891	-0.001828	-0.005638	-0.001678
H(3WA)	0.027854	0.029145	0.017022	-0.009703	0.000184	-0.009773
H(3WB)	0.028106	0.026171	0.019396	-0.013853	0.005582	-0.003338
H(7O)	0.028809	0.021468	0.01814	-0.003563	-0.001086	-0.004534
H(9O)	0.039538	0.021724	0.013747	-0.006402	0.002212	-0.001529
H(2C)	0.037258	0.018741	0.018441	-0.005017	0.000068	-0.000128
H(3C)	0.038163	0.018231	0.026319	-0.006785	0.001755	-0.009244
H(6C)	0.032759	0.016943	0.02205	-0.006565	0.00044	-0.006447
H(10A)	0.022778	0.033855	0.02872	-0.013521	0.000459	-0.011394
H(10B)	0.022778	0.033855	0.02872	-0.013521	0.000459	-0.011394

3. A. Ø. Madsen. SHADE web server for estimation of hydrogen anisotropic displacement parameters. *J. Appl. Cryst.* (2006) **39**, 757–758, https://doi.org/10.1107/S0021889806026379

4. A. Ø. Madsen, A. A. Hoser. SHADE3 server: a streamlined approach to estimate H-atom anisotropic displacement parameters using periodic *ab initio* calculations or experimental information. *J. Appl. Cryst.* (2014). **47**, 2100–2104, https://doi.org/10.1107/S1600576714022973

Table S6: Monopole populations, radial parameters and net atomic charges for atoms in **1**

Atom	Pval	Kappa	P00	Kappa'	Atomic charge
NI(1)	3.137(17)	1.834	6.27(3)	1.088	+0.59(5)
S(2)	5.45(5)	1.122	0.000	0.790	+0.55(5)
O(1)	6.83(3)	0.958	0.000	0.852	-0.83(3)
O(2)	6.92(3)	0.958	0.000	0.852	-0.92(3)
O(3)	6.91(3)	0.958	0.000	0.852	-0.90(3)
O(4)	6.88(3)	0.956	0.000	1.484	-0.88(3)
O(5)	6.93(3)	0.956	0.000	1.484	-0.93(3)
O(6)	6.88(3)	0.956	0.000	1.484	-0.88(3)
O(7)	6.52(3)	0.972	0.000	0.992	-0.51(3)
O(8)	6.45(3)	0.972	0.000	1.092	-0.45(3)
O(9)	6.65(4)	0.964	0.000	0.898	-0.65(4)
O(10)	7.03(3)	0.953	0.000	0.550	-1.03(3)
C(1)	3.89(7)	1.015	0.000	0.885	+0.11(7)
C(2)	4.01(8)	1.008	0.000	0.721	-0.01(8)
C(3)	3.92(9)	1.008	0.000	0.721	+0.08(9)
C(4)	3.67(8)	1.014	0.000	0.735	+0.33(8)
C(5)	3.63(8)	1.021	0.000	0.775	+0.37(8)
C(6)	3.93(7)	1.008	0.000	0.721	+0.07(7)
C(7)	3.89(6)	1.006	0.000	0.759	+0.11(6)
H(1WA)	0.63(2)	1.229	0.000	1.200	+0.37(2)
H(1WB)	0.64(3)	1.229	0.000	1.200	+0.36(3)
H(2WA)	0.68(3)	1.229	0.000	1.200	+0.32(3)
H(2WB)	0.62(3)	1.229	0.000	1.200	+0.38(3)
H(3WA)	0.71(3)	1.229	0.000	1.200	+0.29(3)
H(3WB)	0.64(3)	1.229	0.000	1.200	+0.36(3)
H(7O)	0.466(19)	1.505	0.000	1.200	+0.534(19)
H(9O)	0.306(18)	1.803	0.000	1.200	+0.694(18)
H(2C)	0.42(2)	1.799	0.000	1.200	+0.58(2)
H(3C)	0.37(2)	1.799	0.000	1.200	+0.63(2)
H(6C)	0.46(2)	1.799	0.000	1.200	+0.54(2)
H(10A)	0.46(3)	1.302	0.000	1.200	+0.54(3)
H(10B)	0.51(3)	1.302	0.000	1.200	+0.49(3)

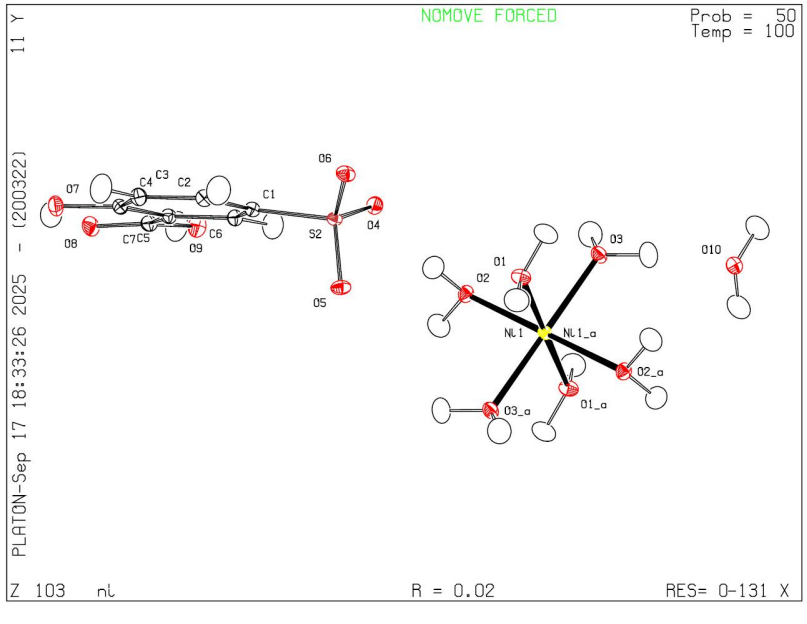
Table S7

Topological properties of interactions at their bond critical point (3,–1)

$d_1(d_2)$ is the distance from the first (second) atom to (3,-1) critical point, $R_{ij} = d_1 + d_2$, ρ is the electron density, $\nabla^2\rho$ is the Laplacian of the electron density. $G(a.u.)$ is the kinetic energy density, $G/\rho(a.u.)$ the ratio between kinetic energy density and electron density, $V(a.u.)$ the potential energy density and $E(a.u.)$ the total energy density in the bond critical point .

Bond	R_{ij}(Å)	d_1(Å)	d_2(Å)	$\rho(e\cdot Å^{-3})$	$\nabla^2\rho(e\cdot Å^{-5})$	$G(a.u.)$	$\frac{G}{\rho}(a.u.)$	$V(a.u.)$	$E(a.u.)$
H(1WA)···O(6)^{iii}	1.8257	0.5595	1.2663	0.16(4)	1.6(2)	0.0163	0.70	-0.0163	0.0000
H(1WB)···O(5)^{iv}	1.7855	0.5555	1.2300	0.25(5)	-0.4(2)	0.0094	0.25	-0.0224	-0.0130
H(2WA)···O(4)	1.8466	0.5522	1.2944	0.12(5)	2.0(2)	0.0170	0.98	-0.0135	0.0035
H(2WB)···O(8)^{v}	1.8310	0.5601	1.2709	0.09(5)	2.8(2)	0.0213	1.63	-0.0137	0.0075
H(3WA)···O(10)	1.8035	0.5031	1.3004	0.16(6)	4.8(2)	0.0387	1.67	-0.0274	0.0113
H(3WB)···O(6)^{vi}	1.8760	0.5955	1.2805	0.14(7)	1.8(2)	0.0168	0.82	-0.0151	0.0018
H(70)···O(8)	1.7489	0.5888	1.1601	0.18(5)	4.6(2)	0.0389	1.45	-0.0298	0.0091
H(9O)···O(10)^{vii}	1.6989	0.4848	1.2141	0.20(7)	4.9(2)	0.0421	1.42	-0.0332	0.0088
H(10A)···O(4)^{ii}	1.7903	0.5296	1.2606	0.18(6)	1.4(2)	0.0164	0.63	-0.0181	-0.0017
H(10B)···O(5)^{i}	1.8196	0.5776	1.2420	0.15(5)	2.2(2)	0.0203	0.90	-0.0180	0.0023
NI(1)—O(1)	2.0510	0.9917	1.0593	0.384(6)	11.672(9)				
NI(1)—O(2)	2.0385	0.9872	1.0513	0.391(6)	11.985(8)				
NI(1)—O(3)	2.0530	0.9923	1.0607	0.375(6)	11.576(8)				
S(2)—O(4)	1.4620	0.6717	0.7904	2.43(3)	-10.85(7)				
S(2)—O(5)	1.4645	0.6686	0.7959	2.50(2)	-12.47(7)				
S(2)—O(6)	1.4543	0.6641	0.7902	2.51(3)	-10.91(7)				
S(2)—C(1)	1.7628	0.9686	0.7942	1.52(2)	-6.98(5)				
O(7)—C(4)	1.3407	0.8444	0.4964	2.19(2)	-20.64(6)				
O(8)—C(7)	1.2372	0.8201	0.4172	2.91(2)	-21.13(9)				
O(9)—C(7)	1.3148	0.8566	0.4583	2.37(2)	-23.35(8)				
C(1)—C(2)	1.4025	0.7295	0.6730	2.20(3)	-19.75(6)				
C(1)—C(6)	1.3847	0.7256	0.6591	2.17(3)	-19.96(6)				
C(2)—C(3)	1.3843	0.6925	0.6918	2.22(2)	-20.90(4)				
C(3)—C(4)	1.4038	0.6679	0.7359	2.18(2)	-20.53(4)				
C(4)—C(5)	1.4123	0.6927	0.7195	2.11(2)	-19.74(4)				
C(5)—C(6)	1.4027	0.7303	0.6723	2.10(2)	-18.84(4)				
C(5)—C(7)	1.4711	0.6838	0.7873	1.96(2)	-17.99(5)				

$i = 1-x, 1-y, 1-z : ii = -x, 1-y, 1-z : iii = -x, 2-y, 1-z; iv = 1-x, 2-y, 1-z;$
$v = 1-x, 1-y, 2-z; vi = -x, -1-y, -z;\ vii = x, y, 1+z$



a=1-x, 1-y,1-z

Residual electron density distribution in the MM model of 1

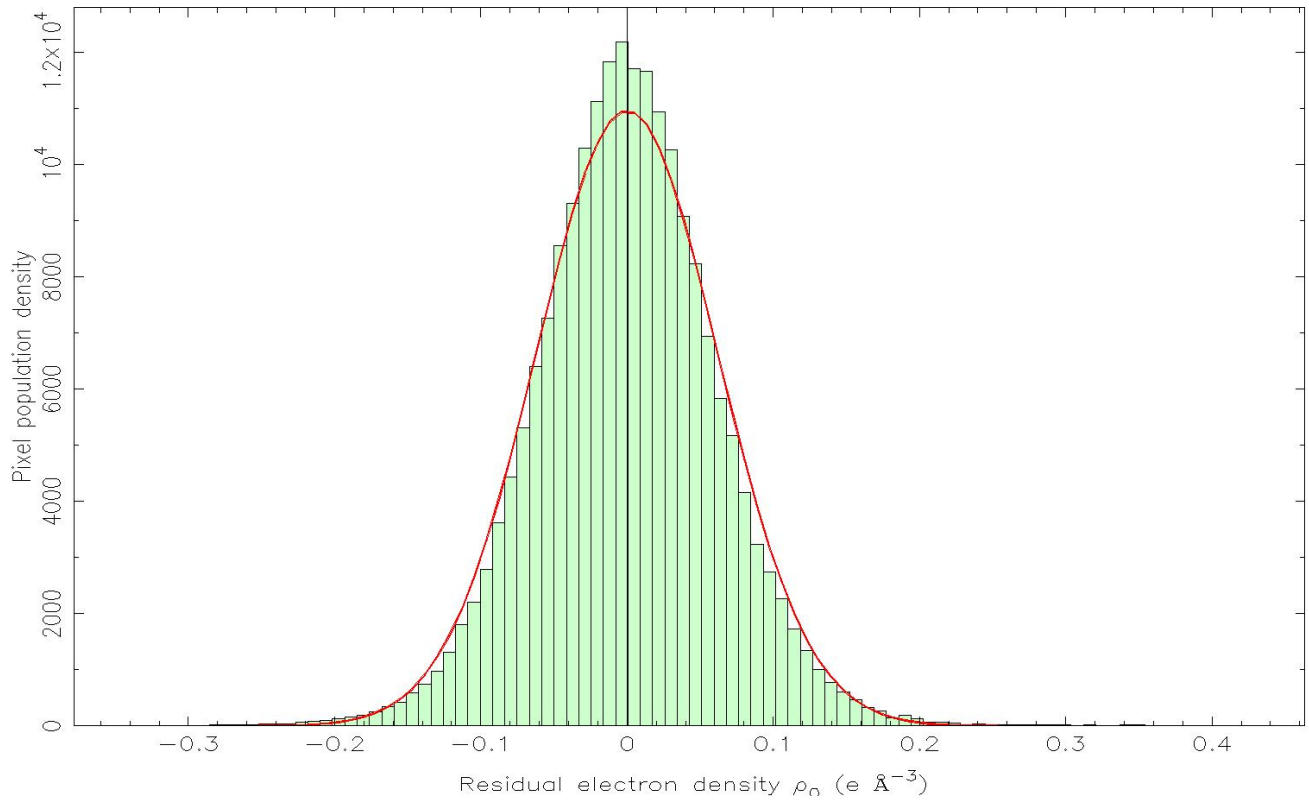


Figure S6: Probabillity distribution histogram of residual electron density of **1**

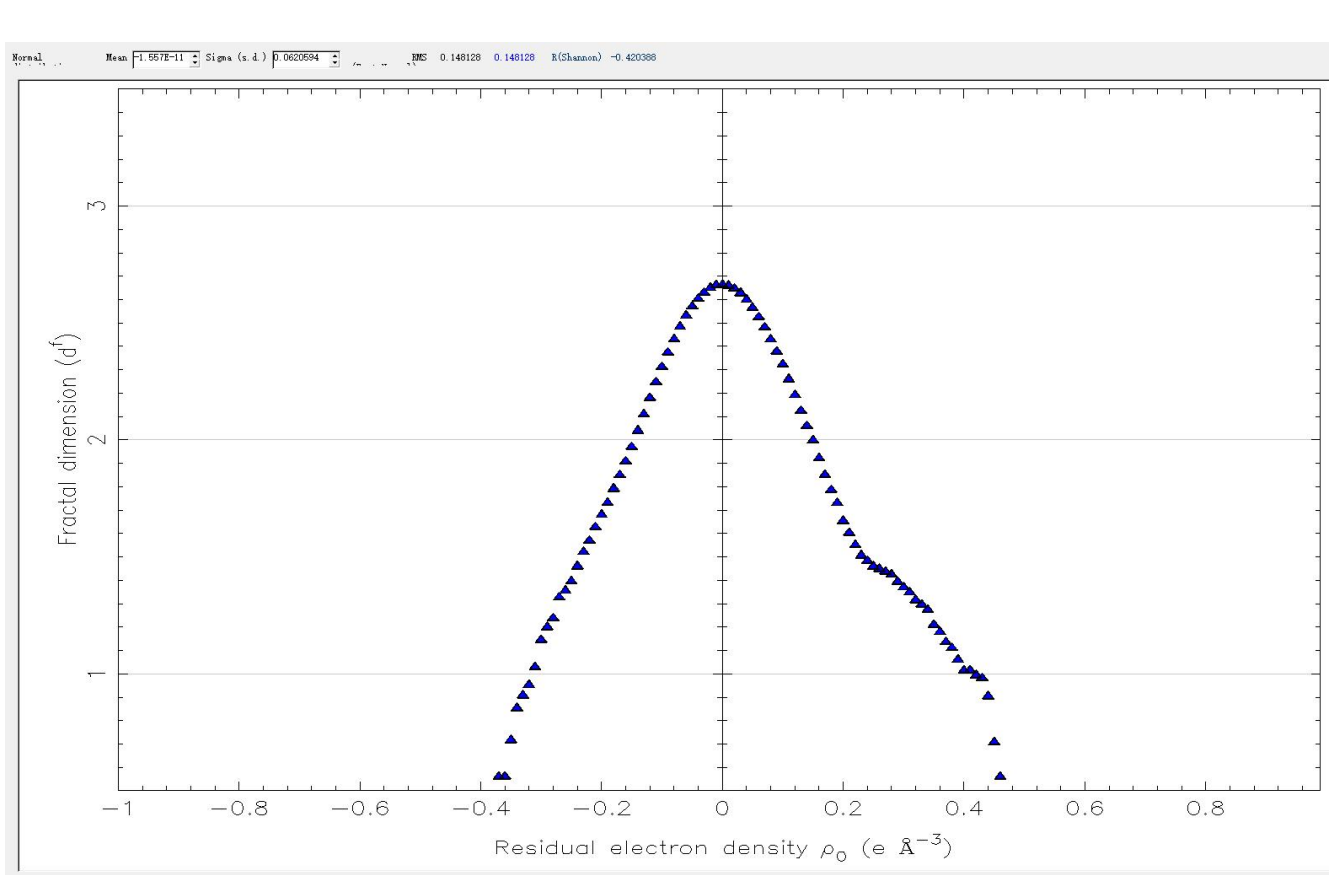


Figure S7: Fractal dimension plot of residual electron density of **1**

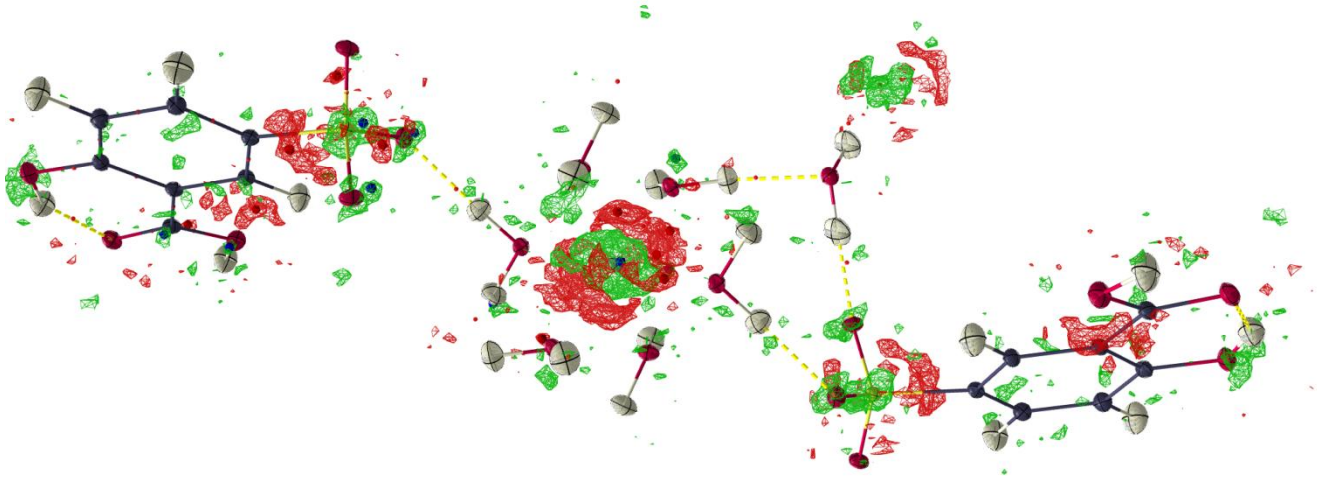


Figure S8: Residual electron density (MM) of **1,** on isosurfaces of ρ = 0.17 e·Å^{-3} (green） and ρ = -0.17 e·Å^{-3} （red)

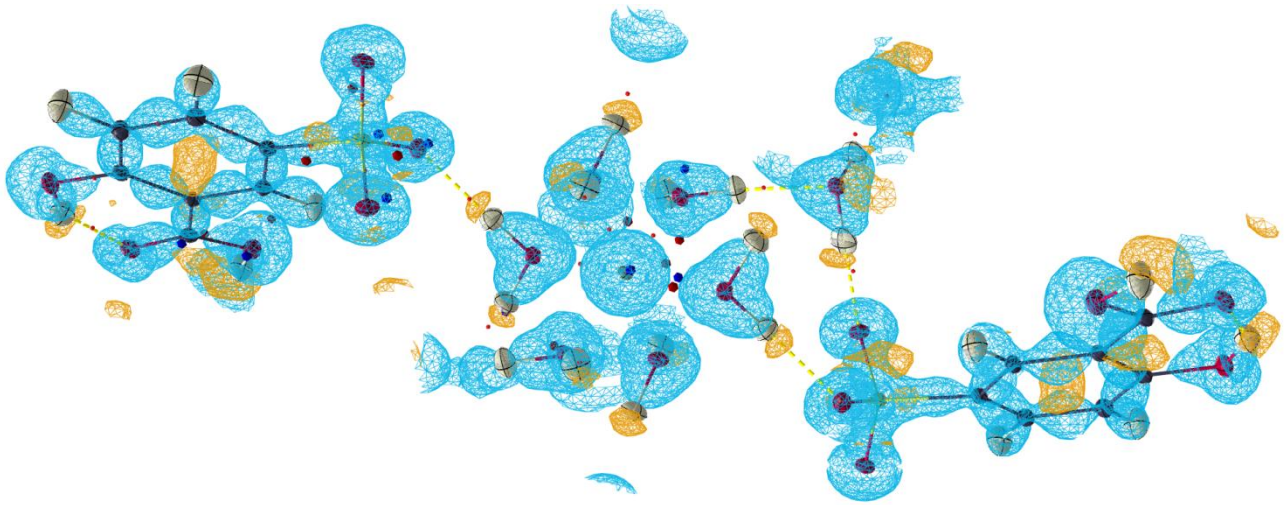


Figure S9: Deformation density ρ_{MM} - ρ_{IAM} of **1,** on isosurfaces of ρ = 0.23 e·Å^{-3} (blue） and ρ = -0.23 e·Å^{-3} （orange）

Table S8: *d* orbital population at Ni1 in **1**, assuming an overall d^8 electron configuration.

d orbital	experimentally observed occupancy [electrons]	idealized occupancy according to the CF model
d_{z2}	1.20	1
d_{x2-y2}	1.21	1
d_{xy}	1.86	2
d_{xz}	1.89	2
d_{yz}	1.83	2