Supplementary Information for

Paramagnetic ^{1}H NMR Assignments in a Ni^{II} Pincer Electrocatalyst for Aqueous H_2 Production

Oana R. Luca,[†] *Steven J. Konezny,*[*,†,§] *Eric K. Paulson,*[†] *Fatemah Habib,*[‡] *Kurt M. Luthy,*[†]

Muralee Murugesu,[*,‡] *Robert H. Crabtree,*[*,†,§] *Victor S. Batista*[*,†,§]

[†]Department of Chemistry, Yale University, P.O. Box 208107, New Haven, CT, 06520-8107, USA; [§]Energy Sciences Institute, Yale University, P.O. Box 27394, West Haven, CT 06516-7394, USA; [‡]Department of Chemistry, University of Ottawa, 10 Marie-Curie, Ottawa, Canada, K1N 6N5.Centre for Catalysis Research and Innovation, University of Ottawa, 30 Marie Curie, Ottawa, Canada, K1N 6N5

[*]E-mail: robert.crabtree@yale.edu; eric.paulson@yale.edu; m.murugesu@uottawa.ca; steven.konenzy@yale.edu; victor.batista@yale.edu

Contents

S1: SQUID measurements

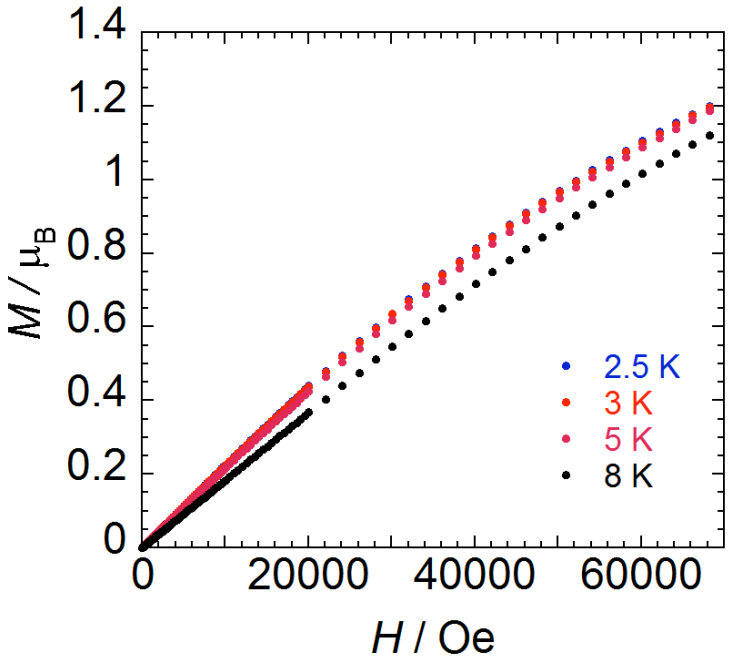


Figure S1-1. Magnetization plot, *M vs. H*, for NiNNN.

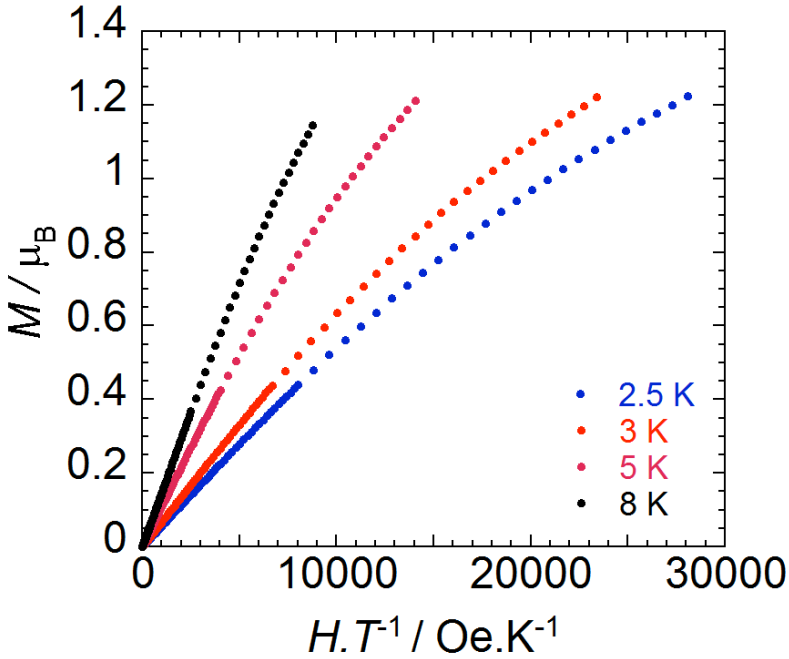


Figure S1-2. Magnetization plot, *M vs. H/T*, for NiNNN.

S2: Preparative experimental details

T1 Measurements

CD_2Cl_2 T1 data collected on a 500MHz Bruker instrument at -40°C. Data worked up using the Bruker Topspin T1/T2 relaxation module and MestReNova 5.2.4-3824.
d1=2s, SW=50, 64 scans per datapoint.

Stacked spectra available in the SI.

Synthesis of Bis[1-(2,6-dimethylphenylimino)ethyl]pyridine (NNN) ligand

NNN ligand was prepared by a Schiff's base reaction using a modified literature methodology. 2,6-diacetylpyridine (0.443 g, 2.7 mmol) and p-toluenesulfonic acid (0.027 g, 0.2 mmol) were added to a 100 mL round bottom flask with a magnetic stir bar and toluene was added via syringe (10 mL) forming a heterogeneous solution. 2,6-dimethylaniline was purified by passing it through a column of basic alumina and then an aliquot was added via microsyringe to the reaction mixture (0.74 mL, 6.0 mmol), quickly forming a homogeneous yellow solution. The reaction mixture was stirred at reflux for 10 hours under nitrogen, and water that formed was removed with a Dean-Starke trap.
After cooling to room temperature, the brown reaction mixture was washed with a solution of $NaCO_3$ and then twice with water. The organic layer was separated and the combined aqueous layers were washed twice with diethyl ether. All organic layers were combined and dried with $MgSO_4$ and rotary evaporation of the resulting solution yielded a brown residue. After adding EtOH to the residue, the mixture was stored at -24 °C for 3 hr. The solid was isolated by vacuum filtration and washed several times with cold EtOH. The filtrate was stored overnight at -24 °C, and more yellow crystals were isolated and washed in the same manner. Overall yield: 0.485 mg (48.5 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (d, J = 7.8 Hz, 2H), 7.92 (t, J = 7.8 Hz, 1H), 7.08 (d, J = 7.5 Hz, 4H), 6.99 – 6.90 (m, 2H), 2.24 (s, 6H), 2.06 (s, 12H).

Synthesis of **ZnNNN**

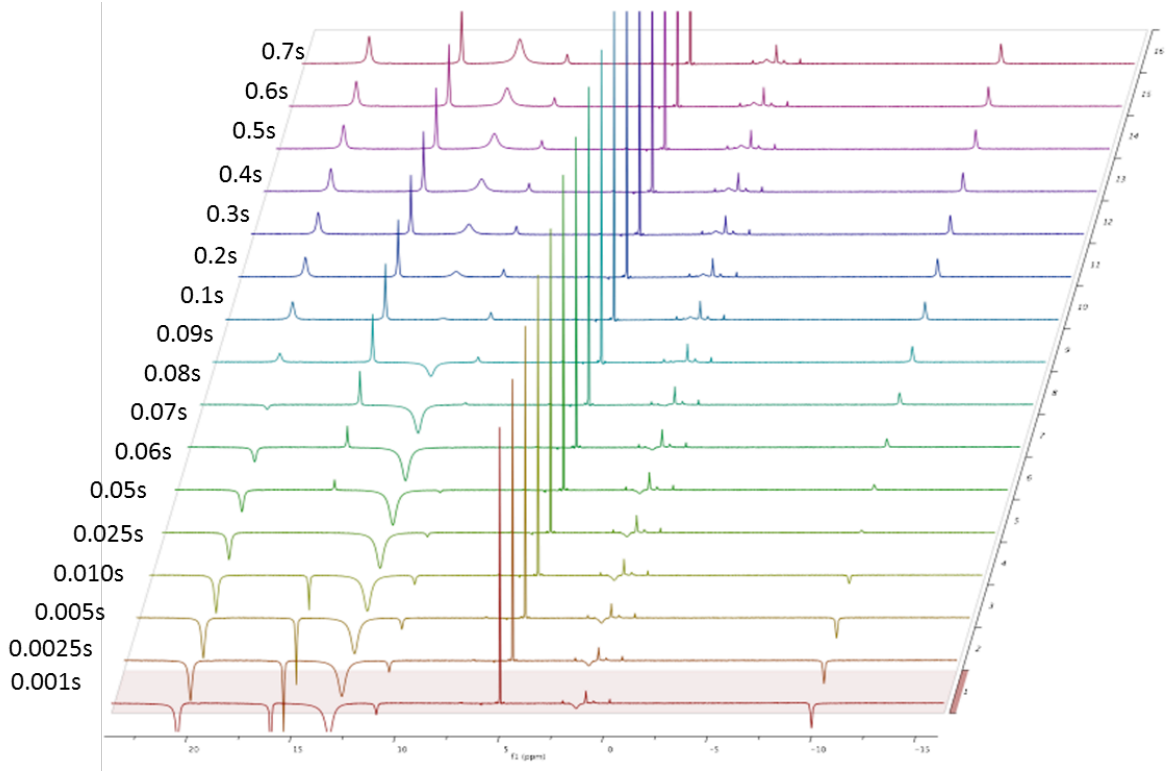
ZnNNN was prepared by a known synthesis for a zinc chloride analog[i]

Bis[1-(2,6-dimethylphenylimino)ethyl]pyridine (217 mg, 0.59 mmol) and $ZnBr_2$ (132 mg, 0.59 mmol) were combined with a magnetic stir bar in a 100 mL round bottom flask. Acetonitrile (45 mL) was added via syringe and the reaction was allowed to proceed under nitrogen for 18 hours with stirring. Solvent was removed from the resulting yellow solution by rotary evaporation. The isolated yellow product was heated in a mixture of 2:1 acetonitrile:dichloromethane until dissolved. The yellow solution was removed from heat, allowed to cool to room temperature, and then stored in a freezer for 2 days. The resulting yellow crystals were isolated by vacuum filtration and dried on a Schlenk line. Yield: 286 mg (81.5%) ^{1}H NMR (400 MHz, CD_2Cl_2) δ 8.50 (m, 1H), 8.31 (d, J = 7.8 Hz, 2H), 7.16 – 7.03 (m, 6H), 2.33 (s, 6H), 2.23 (s, 12H). ^{13}C NMR (126 MHz, CD_2CL_4) δ 164.04 (s), 148.21 (s), 144.50 (s), 143.52 (s), 128.12 (s), 127.99 (s), 126.39 (s), 125.46 (s), 19.45 (s), 17.18 (s). Calcd for C25H27Br2N3Zn (%): C, 50.49; H, 4.58; N, 7.07. Found (%): C, 50.22; H, 4.54; N, 7.34. HR FT-ICR MS: Found (calcd for $C_{25}H_{27}Br_2N_3Zn$): m/z = $(M\text{-}Br)^+$ 514.0594 (514.0659).

S3: T1 experiment stacked spectra

CD_2Cl_2 T1 data collected on a 500MHz Bruker instrument at -40°C. Data worked up using the Bruker Topspin T1/T2 relaxation module and MestReNova 5.2.4-3824.

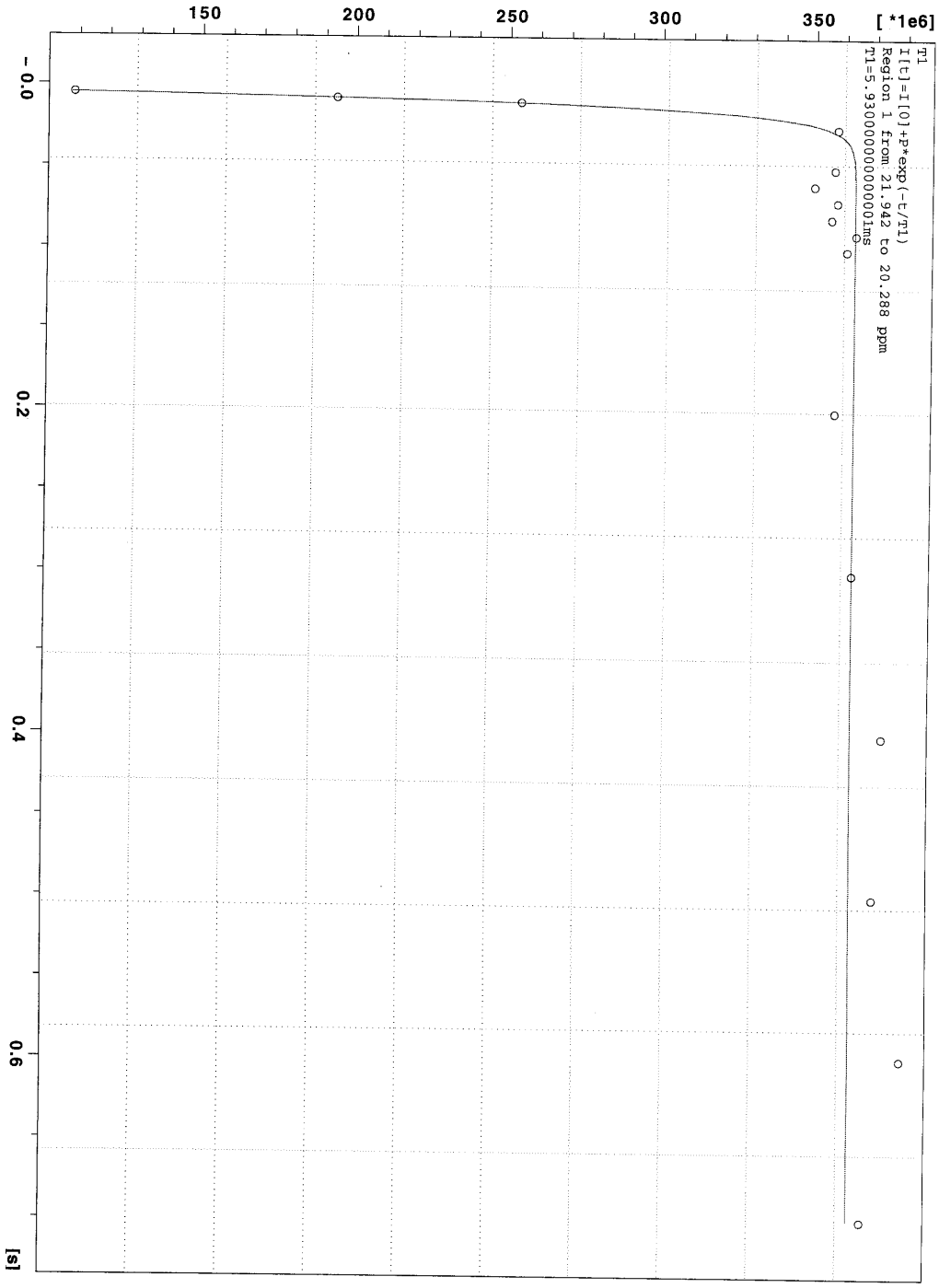
d1=2s, SW=50, 64 scans per datapoint.
Delays: 0.7s, 0.6s, 0.5s, 0.4s, 0.3s, 0.2s, 0.1s, 0.09s, 0.08s, 0.07s, 0.06s, 0.05s, 0.025s, 0.010s, 0.005s, 0.0025s,0.001 Stacked spectra below.

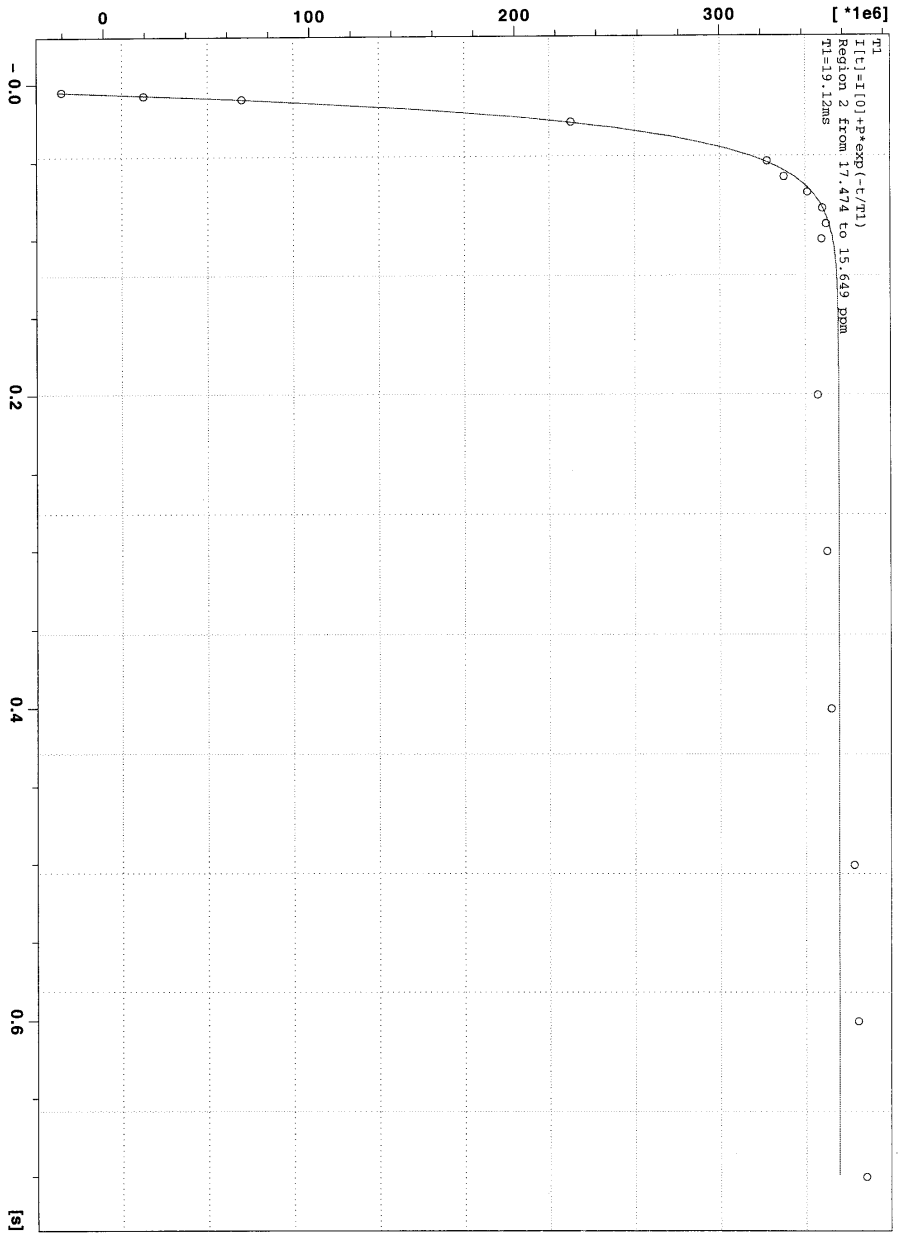


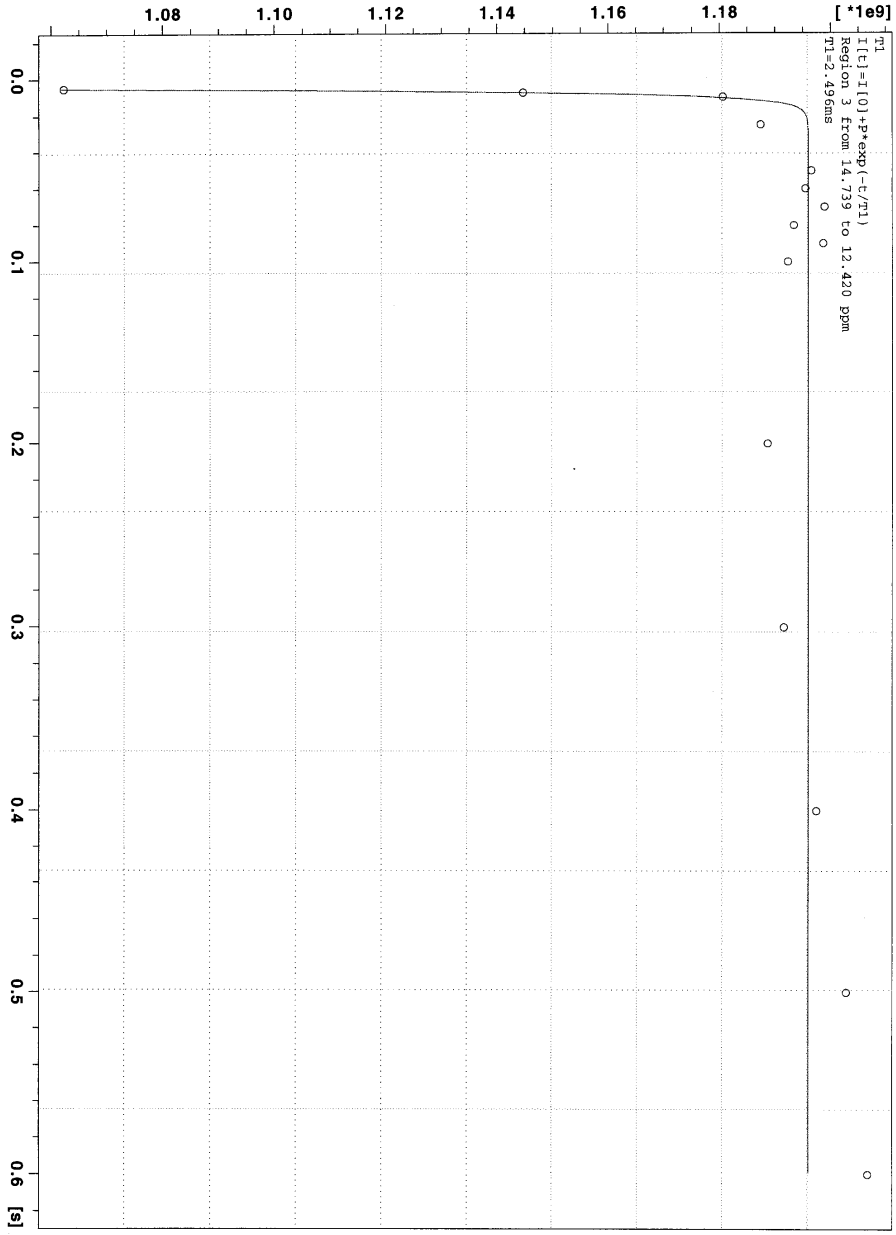
S4: T1 experiment exponential fits

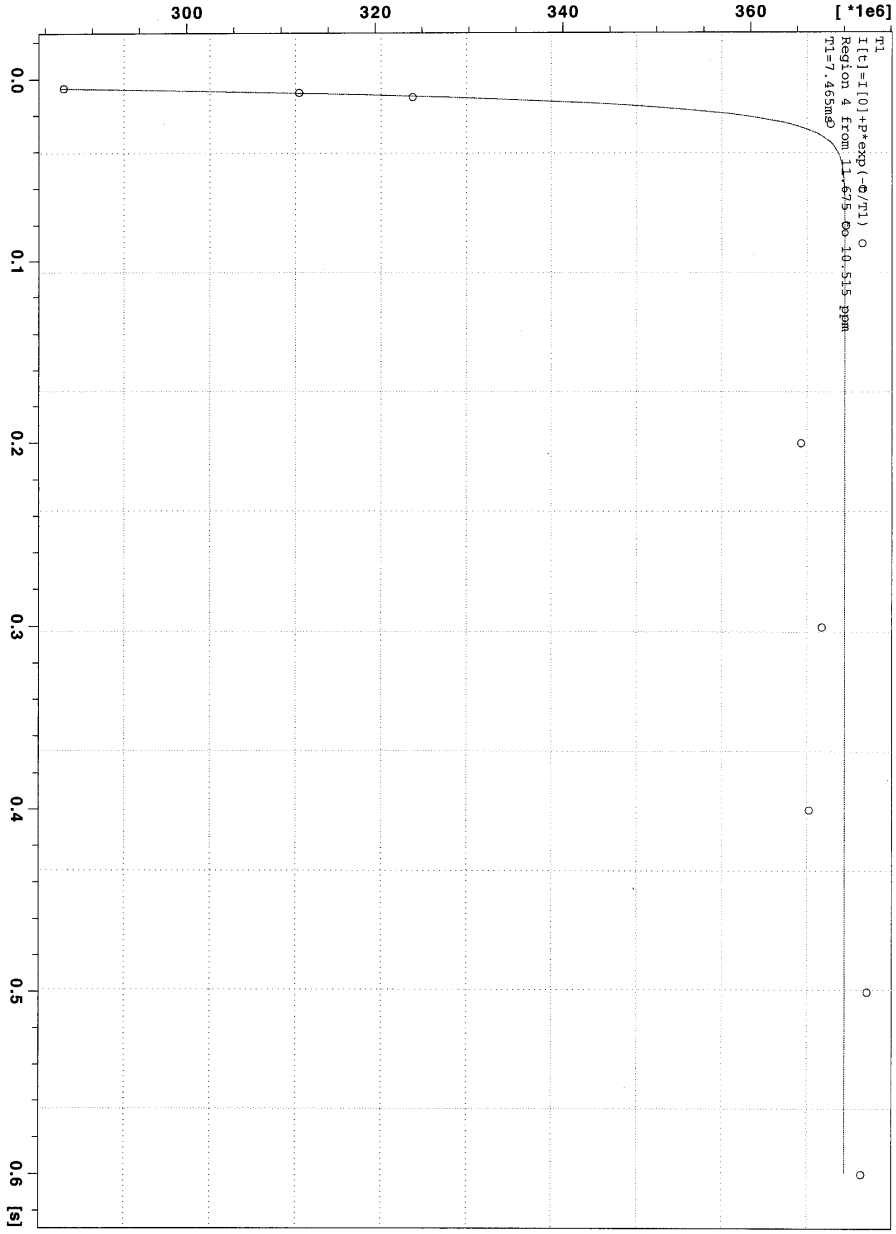
	T1(ms)	r(Å)	r^6(Å^6)
Ha	115	6.44	71337.06
Hb	5.9	4.92	14183.74
Hc	7.4	5.4	24794.91
Hd	2.5	4.3	6321.363
He	19.12	5.88	41329.86
Hf	34	6.63	84933.92

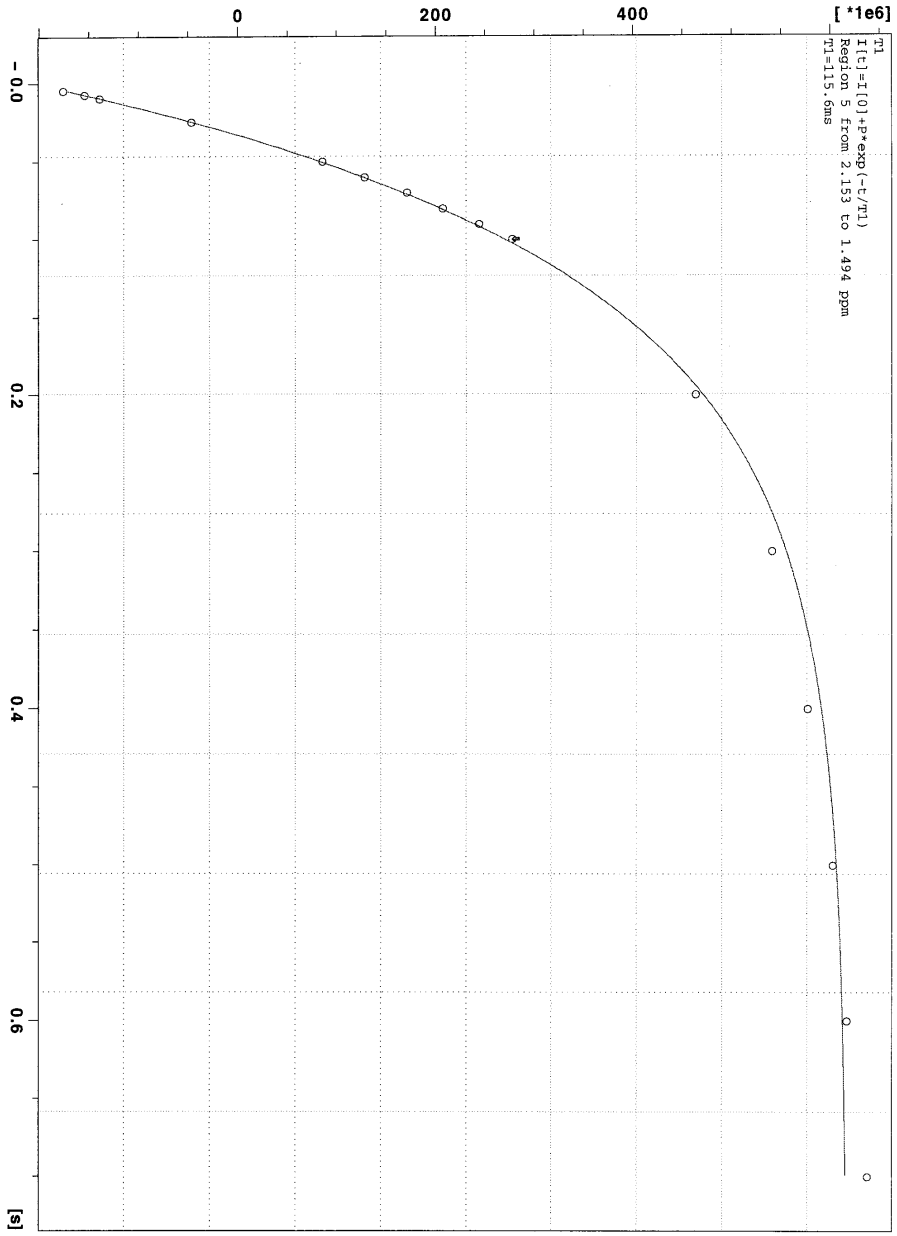
Table S4. Input data for Figure 3 plot. r: average distances between H_x and Ni center from the XRay structure[3], T1: relaxation time determined by exponential fits at 16 delay timepoints.

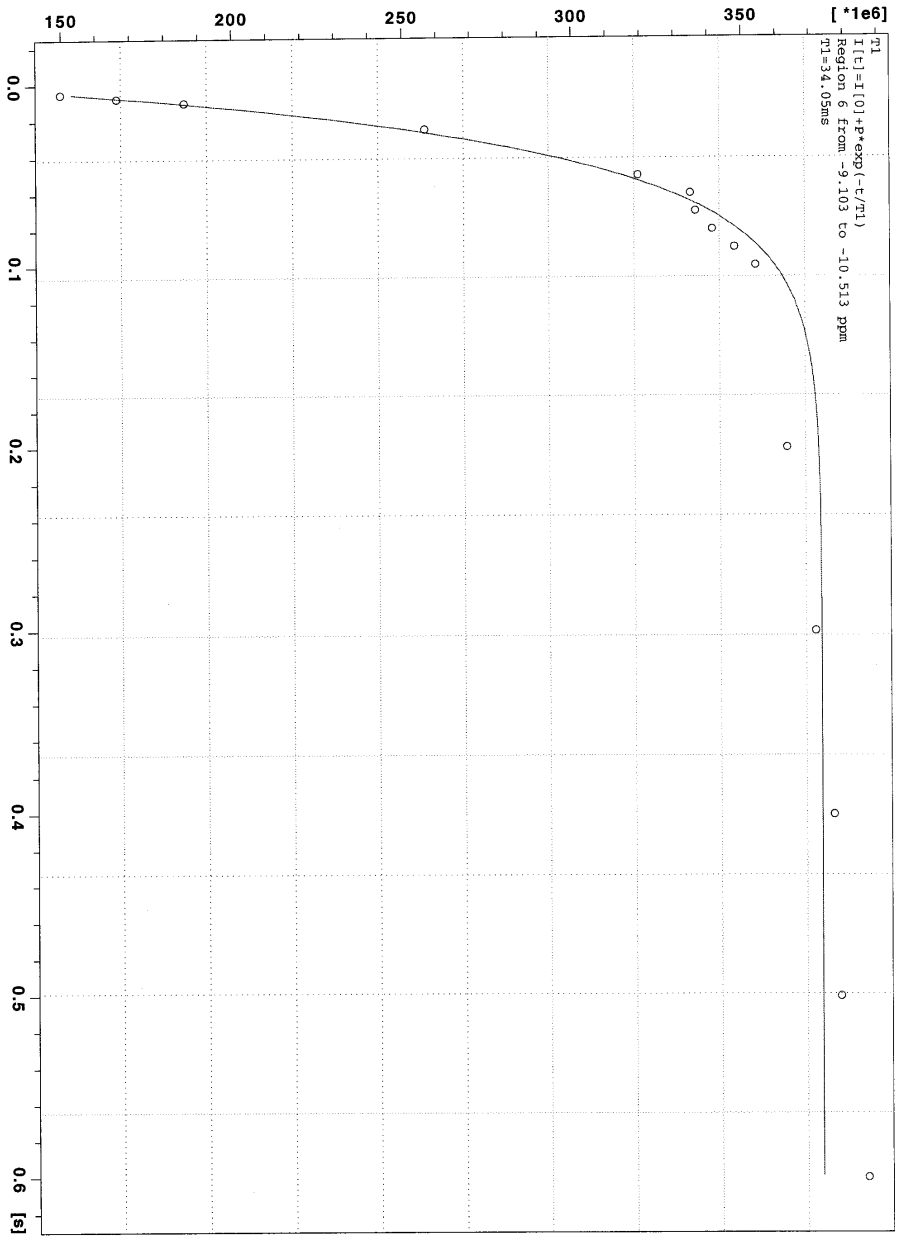



[*1e6]
300
200
100
0
T1
I[t]=I[0]+P*exp(-t/T1)
Region 2 from 17.474 to 15.649 ppm
T1=19.12ms
0.0
0.2
0.4
0.6
[s]

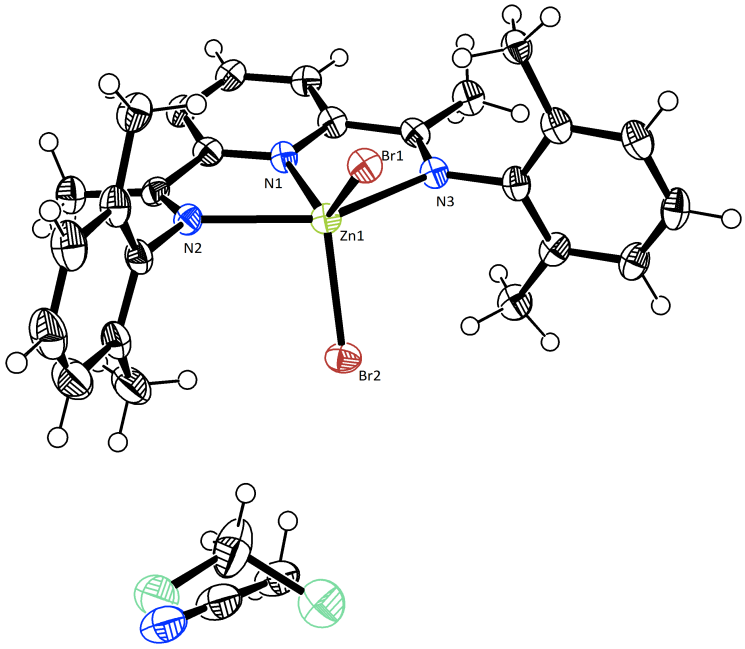

T1
I[t]=I[0]+P*exp(-t/T1)
Region 3 from 14.739 to 12.420 ppm
T1=2.496ms
[*1e9]
1.08
1.10
1.12
1.14
1.16
1.18
0.0
0.1
0.2
0.3
0.4
0.5
0.6
[s]


T1
I[t]=I[0]+P*exp(-t/T1)
Region 4 from 11.675 to 10.515 ppm
T1=7.465ms
[*1e6]
360
340
320
300
0.0
0.1
0.2
0.3
0.4
0.5
0.6
[s]


T1
I[t]=I[0]+P*exp(-t/T1)
Region 5 from 2.153 to 1.494 ppm
T1=115.6ms
[*1e6]
400
200
0
0.0
0.2
0.4
0.6
[s]


T1
I[t]=I[0]+P*exp(-t/T1)
Region 6 from -9.103 to -10.513 ppm
T1=34.05ms
[*1e6]
350
300
250
200
150
0.0
0.1
0.2
0.3
0.4
0.5
0.6
[s]

S5: X-ray crystal structure of (NNN)$ZnBr_2$·0.5(MeCN)·0.5(CH_2Cl_2)



A. Crystal Data

Empirical Formula	$Br_2ZnN_{3.50}C_{26.50}H_{29.50}Cl$	
Formula Weight	657.69	
Crystal Color, Habit	colorless, prism	
Crystal Dimensions	0.10 X 0.09 X 0.04 mm	
Crystal System	monoclinic	
Lattice Type	Primitive	
Indexing Images	8 images @ 1.0 second	
Detector Position	50.00 mm	
Lattice Parameters	a = 13.0658(2) Å	α = 90°
	b = 14.9486(3) Å	β = 107.768(8)°
	c = 14.4262(10) Å	γ = 90°
	V = 2683.3(2) $Å^3$	
Space Group	$P2_1/n$ (#14)	
Z value	4	
D_{calc}	1.628 g/cm^3	
F_{000}	1320.00	
μ(CuKα)	58.361 cm^{-1}	

B. Intensity Measurements

Diffractometer	Rigaku Saturn944+ CCD
Radiation	CuKα (λ = 1.54187 Å)
Detector Aperture	94 mm x 94 mm
Data Images	777 images (1° width)
Exposure Rate (detector angle = 42°)	1.0 sec./°
Exposure Rate (detector angle = 90°)	2.0 sec./°
Detector Position	50.00 mm
$2\theta_{max}$	130.1°
No. of Reflections Measured	Total: 12790 Unique: 4421 (R_{int} = 0.0717)
Corrections	Lorentz-polarization, Absorption (trans. factors: 0.543 - 0.792)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\Sigma\ w\ (Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/ [\ \sigma^2 (Fo^2) + (0.0563 \cdot P)^2 + 7.2757 \cdot P\ ]$
	where $P = (Max(Fo^2,0) + 2Fc^2)/3$
$2\theta_{max}$ cutoff	130.1°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	4421
No. Variables	328
Reflection/Parameter Ratio	13.48
Residuals: R1 (I>2.00σ(I))	0.0480
Residuals: R (All reflections)	0.0511
Residuals: wR2 (All reflections)	0.1279
Goodness of Fit Indicator	1.030
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.72 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.74 $e^-/Å^3$

D. Experimental Details

The crystal sample was mounted in a Hampton Research loop with immersion oil. All measurements were made on a Rigaku Saturn944+ CCD diffractometer with filtered Cu-Kα radiation at a temperature of -180°C. The structure was solved by direct methods[b] and expanded using Fourier techniques.[c] The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The disordered solvent (50:50 MeCN/DCM) was modeled with restraints placed on the N4-C26 (1.14 0.02), C26-C28 (1.43 0.02), Cl2-C27, and Cl1-C27 bond distances (1.73 0.02). The thermal parameters of the atoms in the acetonitrile molecule were constrained to be equal. (EADP C28 N4 C26). The final cycle of full-matrix least-squares refinement[d] on F^2 was based on 4421 observed reflections and 328 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

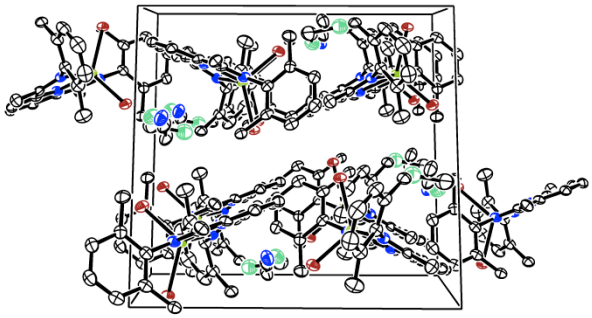
$$R1 = \Sigma\ ||Fo| - |Fc|| / \Sigma\ |Fo| = 0.0480$$

$$wR2 = [\ \Sigma\ (\ w\ (Fo^2 - Fc^2)^2\ )/\ \Sigma\ w(Fo^2)^2]^{1/2} = 0.1279$$

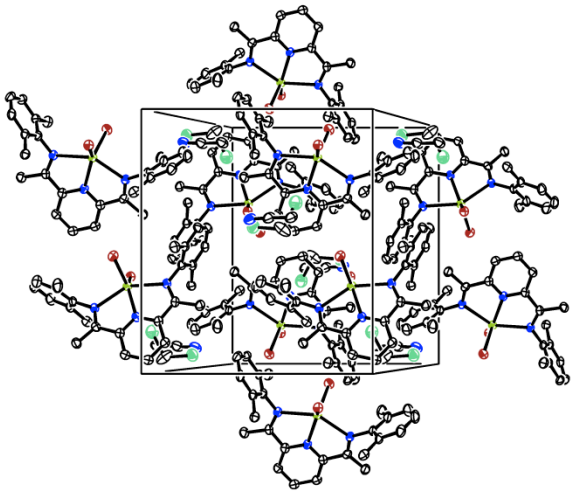
The standard deviation of an observation of unit weight[e] was 1.03. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.72 and -0.74 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber.[f] Anomalous dispersion effects were included in Fcalc[g]; the values for Δf' and Δf" were those of Creagh and McAuley.[h] The values for the mass attenuation coefficients are those of Creagh and Hubbell.[i] All calculations were performed using the CrystalStructure[j] crystallographic software package except for refinement, which was performed using SHELXL-97.[b]

E. Additional Images



View of packing along a (H atoms omitted)



View of packing along c (H atoms omitted)

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

atom	x	y	z	B_{eq}[k]	Occ
Br(1)	0.75974(3)	0.36784(3)	0.56788(3)	2.119(13)	
Br(2)	0.80677(4)	0.44688(3)	0.84427(3)	2.411(13)	
Zn(1)	0.82199(4)	0.33102(4)	0.73672(4)	1.865(14)	
Cl(1)	0.6440(2)	0.6488(2)	0.9365(2)	4.47(6)	1/2
Cl(2)	0.4370(3)	0.5581(2)	0.8787(3)	3.91(8)	1/2
N(1)	0.8886(3)	0.2119(2)	0.7992(2)	1.86(6)	
N(2)	0.6801(3)	0.2451(2)	0.7482(2)	1.90(6)	
N(3)	1.0045(3)	0.3407(2)	0.7545(2)	1.86(6)	
N(4)	0.4252(11)	0.5880(9)	0.8829(13)	3.60(14)	1/2
C(1)	0.9945(3)	0.1954(3)	0.8156(3)	1.99(7)	
C(2)	1.0388(4)	0.1137(3)	0.8516(3)	2.12(7)	
C(3)	0.9739(4)	0.0483(3)	0.8737(3)	2.23(8)	
C(4)	0.8668(4)	0.0671(3)	0.8593(3)	2.20(7)	
C(5)	0.8256(3)	0.1494(3)	0.8208(3)	2.00(7)	
C(6)	0.7096(3)	0.1732(3)	0.7974(3)	2.00(7)	
C(7)	0.6385(4)	0.1111(3)	0.8298(4)	2.66(8)	
C(8)	1.0569(3)	0.2701(3)	0.7901(3)	1.96(7)	
C(9)	1.1749(4)	0.2567(3)	0.8082(3)	2.54(8)	
C(10)	0.5679(3)	0.2707(3)	0.7140(3)	2.14(7)	
C(11)	0.5217(4)	0.3188(3)	0.7747(4)	2.77(8)	
C(12)	0.4162(5)	0.3490(4)	0.7342(5)	3.94(11)	
C(13)	0.3577(4)	0.3298(4)	0.6391(4)	3.68(11)	
C(14)	0.4039(4)	0.2783(4)	0.5831(4)	3.17(9)	
C(15)	0.5091(3)	0.2477(3)	0.6182(3)	2.33(8)	
C(16)	0.5568(4)	0.1897(3)	0.5573(3)	2.57(8)	
C(17)	0.5798(5)	0.3332(4)	0.8804(4)	3.88(11)	
C(18)	1.0585(3)	0.4141(3)	0.7247(3)	2.05(7)	
C(19)	1.0998(3)	0.4844(3)	0.7897(3)	2.14(7)	
C(20)	1.1444(4)	0.5578(3)	0.7550(3)	2.48(8)	
C(21)	1.1483(4)	0.5596(3)	0.6611(4)	2.67(8)	
C(22)	1.1115(3)	0.4880(3)	0.5996(3)	2.48(8)	
C(24)	1.0349(4)	0.3332(3)	0.5649(3)	2.29(8)	
C(25)	1.0992(4)	0.4810(4)	0.8934(3)	2.80(8)	
C(26)	0.5181(9)	0.5922(8)	0.9100(8)	3.60(14)	1/2
C(27)	0.5773(7)	0.5482(7)	0.9120(8)	5.4(3)	1/2
C(28)	0.6318(8)	0.5924(7)	0.9560(7)	3.60(14)	1/2

Table 2. Atomic coordinates and B_{iso} of hydrogen atoms

atom	x	y	z	B_{iso}	Occ
H(2)	1.1128	0.1022	0.8611	2.55	
H(3)	1.0030	-0.0083	0.8983	2.68	
H(4)	0.8216	0.0241	0.8756	2.63	
H(7A)	0.6689	0.1002	0.8999	3.19	
H(7B)	0.6329	0.0543	0.7946	3.19	
H(7C)	0.5670	0.1378	0.8163	3.19	
H(9A)	1.2085	0.2391	0.8761	3.05	
H(9B)	1.2072	0.3126	0.7951	3.05	
H(9C)	1.1862	0.2096	0.7651	3.05	
H(12)	0.3839	0.3836	0.7730	4.73	
H(13)	0.2865	0.3518	0.6126	4.41	
H(14)	0.3625	0.2633	0.5185	3.81	
H(16A)	0.5024	0.1776	0.4947	3.08	
H(16B)	0.5808	0.1331	0.5913	3.08	
H(16C)	0.6182	0.2204	0.5461	3.08	
H(17A)	0.6507	0.3591	0.8875	4.65	
H(17B)	0.5884	0.2758	0.9147	4.65	
H(17C)	0.5384	0.3742	0.9080	4.65	
H(20)	1.1721	0.6068	0.7971	2.98	
H(21)	1.1767	0.6106	0.6382	3.20	
H(22)	1.1160	0.4899	0.5351	2.98	
H(24A)	1.0322	0.3497	0.4985	2.75	
H(24B)	0.9638	0.3127	0.5654	2.75	
H(24C)	1.0874	0.2850	0.5882	2.75	
H(25A)	1.1398	0.4286	0.9257	3.36	
H(25B)	1.0249	0.4767	0.8951	3.36	
H(25C)	1.1323	0.5355	0.9273	3.36	
H(27A)	0.6015	0.5102	0.9696	6.54	1/2
H(27B)	0.5966	0.5185	0.8595	6.54	1/2
H(28A)	0.6593	0.6523	0.9552	4.32	1/2
H(28B)	0.6657	0.5526	0.9215	4.32	1/2
H(28C)	0.6473	0.5725	1.0226	4.32	1/2

Table 3. Anisotropic displacement parameters[l]

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br(1)	0.0254(3)	0.0332(3)	0.0184(2)	0.00059(17)	0.00144(18)	0.00122(16)
Br(2)	0.0289(3)	0.0350(3)	0.0249(3)	0.00248(18)	0.00396(19)	-0.00555(17)
Zn(1)	0.0222(3)	0.0270(3)	0.0186(3)	0.0009(2)	0.0016(2)	0.0008(2)
Cl(1)	0.0473(16)	0.0630(20)	0.0534(17)	0.0017(14)	0.0062(13)	0.0014(14)
Cl(2)	0.045(2)	0.054(2)	0.0498(16)	0.0013(16)	0.0149(14)	-0.0072(18)
N(1)	0.0202(18)	0.0298(20)	0.0183(16)	-0.0023(14)	0.0021(13)	0.0019(14)
N(2)	0.0188(18)	0.0289(20)	0.0215(17)	-0.0031(14)	0.0018(13)	-0.0028(14)
N(3)	0.0197(18)	0.0298(19)	0.0187(17)	-0.0004(14)	0.0020(14)	0.0022(14)
N(4)	0.041(4)	0.037(4)	0.051(4)	0.006(3)	0.003(3)	-0.010(3)
C(1)	0.022(2)	0.033(2)	0.0170(19)	0.0007(18)	0.0008(16)	0.0030(17)
C(2)	0.022(2)	0.032(2)	0.022(2)	0.0020(18)	-0.0006(17)	0.0004(17)
C(3)	0.029(2)	0.028(2)	0.022(2)	-0.0011(18)	-0.0001(18)	0.0015(17)
C(4)	0.027(2)	0.029(2)	0.023(2)	-0.0024(18)	0.0014(17)	0.0012(17)
C(5)	0.024(2)	0.031(2)	0.0177(19)	-0.0003(18)	0.0027(16)	-0.0016(17)
C(6)	0.026(2)	0.028(2)	0.021(2)	-0.0070(17)	0.0047(17)	-0.0063(17)
C(7)	0.027(2)	0.035(3)	0.039(3)	-0.0029(20)	0.0085(20)	0.004(2)
C(8)	0.020(2)	0.035(2)	0.0180(19)	0.0011(18)	0.0038(16)	0.0002(17)
C(9)	0.026(2)	0.036(3)	0.032(2)	0.0056(19)	0.0052(18)	0.0084(19)
C(10)	0.018(2)	0.031(2)	0.033(2)	-0.0010(17)	0.0081(17)	0.0024(18)
C(11)	0.024(2)	0.041(3)	0.042(3)	-0.0051(20)	0.014(2)	-0.002(2)
C(12)	0.044(3)	0.050(3)	0.065(4)	0.005(3)	0.030(3)	0.002(3)
C(13)	0.022(3)	0.055(3)	0.061(4)	0.005(2)	0.010(2)	0.013(3)
C(14)	0.026(3)	0.048(3)	0.042(3)	-0.000(2)	0.004(2)	0.015(2)
C(15)	0.017(2)	0.036(3)	0.031(2)	-0.0009(18)	0.0006(17)	0.0073(19)
C(16)	0.029(2)	0.037(3)	0.024(2)	-0.0057(20)	-0.0017(18)	-0.0008(19)
C(17)	0.042(3)	0.067(4)	0.045(3)	-0.003(3)	0.024(3)	-0.010(3)
C(18)	0.017(2)	0.031(2)	0.028(2)	0.0010(17)	0.0037(16)	0.0058(18)
C(19)	0.016(2)	0.029(2)	0.030(2)	0.0003(17)	-0.0023(17)	0.0010(18)
C(20)	0.022(2)	0.031(2)	0.035(2)	-0.0047(18)	0.0002(18)	0.0023(19)
C(21)	0.024(2)	0.034(3)	0.040(3)	-0.0064(19)	0.0046(19)	0.008(2)
C(22)	0.019(2)	0.043(3)	0.031(2)	0.0021(19)	0.0057(18)	0.0074(20)
C(23)	0.018(2)	0.036(3)	0.027(2)	0.0031(18)	0.0032(16)	0.0062(18)
C(24)	0.030(2)	0.032(2)	0.026(2)	-0.0010(19)	0.0103(18)	0.0031(18)
C(25)	0.029(2)	0.043(3)	0.029(2)	-0.004(2)	0.0015(19)	-0.003(2)
C(26)	0.041(4)	0.037(4)	0.051(4)	0.006(3)	0.003(3)	-0.010(3)
C(27)	0.083(11)	0.067(10)	0.052(8)	-0.039(9)	0.015(7)	-0.004(7)
C(28)	0.041(4)	0.037(4)	0.051(4)	0.006(3)	0.003(3)	-0.010(3)

Table 4. Bond lengths (Å)

atom	atom	distance
Br(1)	Zn(1)	2.3850(7)
Zn(1)	N(1)	2.065(3)
Zn(1)	N(3)	2.324(4)
Cl(2)	C(27)	1.753(10)
N(1)	C(5)	1.343(6)
N(2)	C(10)	1.448(5)
N(3)	C(18)	1.440(6)
C(1)	C(2)	1.383(6)
C(2)	C(3)	1.393(7)
C(4)	C(5)	1.389(6)
C(6)	C(7)	1.486(7)
C(10)	C(11)	1.404(7)
C(11)	C(12)	1.398(7)
C(12)	C(13)	1.381(8)
C(14)	C(15)	1.390(6)
C(18)	C(19)	1.402(6)
C(19)	C(20)	1.404(7)
C(20)	C(21)	1.372(7)
C(22)	C(23)	1.390(7)
C(26)	C(28)	1.431(14)

atom	atom	distance
Br(2)	Zn(1)	2.3738(8)
Zn(1)	N(2)	2.303(4)
Cl(1)	C(27)	1.719(10)
N(1)	C(1)	1.353(6)
N(2)	C(6)	1.281(5)
N(3)	C(8)	1.277(5)
N(4)	C(26)	1.158(17)
C(1)	C(8)	1.495(6)
C(3)	C(4)	1.380(7)
C(5)	C(6)	1.491(6)
C(8)	C(9)	1.496(6)
C(10)	C(15)	1.403(6)
C(11)	C(17)	1.497(7)
C(13)	C(14)	1.382(9)
C(15)	C(16)	1.499(7)
C(18)	C(23)	1.400(7)
C(19)	C(25)	1.500(7)
C(21)	C(22)	1.380(7)
C(23)	C(24)	1.504(6)

S6: Computational data and full Gaussian09 reference

S6-T1. Atomic coordinates of NiNNN obtained at the DFT UB3LYP/LANL2DZ/6-311++G(d,p) level. Structure is shown in Figure 1 of the main text.

Br	-9.2E-05	-2.47002	0.08858
Br	-0.00016	0.71534	2.50375
Ni	-0.00038	-0.01307	0.073778
N	-0.00018	1.902111	-0.5808
N	2.15538	0.426582	-0.31788
N	-2.1557	0.427114	-0.31669
C	1.167872	2.536029	-0.71275
C	1.204898	3.908278	-0.96628
C	-0.00002	4.595822	-1.08179
C	-1.20504	3.908613	-0.96535
C	-1.16817	2.53637	-0.71187
C	2.374745	1.658497	-0.60953
C	3.716701	2.267086	-0.89097
C	-2.37512	1.659056	-0.60811
C	-3.71718	2.267663	-0.88896
C	3.22663	-0.52796	-0.28023
C	3.434243	-1.32455	-1.42157
C	4.44086	-2.29201	-1.37744
C	5.214265	-2.47183	-0.2343
C	4.993313	-1.67004	0.880456
C	4.003503	-0.68117	0.881217
C	2.608354	-1.13436	-2.66889
C	3.812489	0.198953	2.090975
C	-3.22664	-0.52778	-0.27992
C	-3.43357	-1.32308	-1.42223
C	-4.43922	-2.29158	-1.37939
C	-5.21254	-2.47338	-0.2365
C	-4.9924	-1.67269	0.879219
C	-4.0034	-0.68298	0.881288
C	-2.60804	-1.12963	-2.66924
C	-3.81309	0.195922	2.09203
H	2.145076	4.431248	-1.07068
H	0.00005	5.661963	-1.27102
H	-2.14517	4.431815	-1.06904
H	4.502613	1.515889	-0.88071
H	3.711523	2.761946	-1.86558

H	3.9484	3.02812	-0.13974
H	-3.94843	3.028866	-0.13777
H	-3.71256	2.762293	-1.86369
H	-4.50313	1.516508	-0.87808
H	4.611539	-2.9119	-2.25109
H	5.984853	-3.23417	-0.21253
H	5.596531	-1.80469	1.771972
H	1.545696	-1.28429	-2.46051
H	2.723337	-0.12806	-3.08396
H	2.907164	-1.84845	-3.43768
H	4.194522	1.210316	1.911675
H	2.760068	0.294658	2.362781
H	4.356984	-0.20712	2.945049
H	-4.60911	-2.91073	-2.25373
H	-5.98236	-3.23653	-0.21563
H	-5.59552	-1.80899	1.770548
H	-2.73314	-0.12616	-3.08843
H	-1.54419	-1.26725	-2.4589
H	-2.89841	-1.84977	-3.4356
H	-2.76068	0.292844	2.363437
H	-4.19664	1.206948	1.914024
H	-4.35663	-0.21192	2.94587

The full Gaussian09 refernce is given below:

Gaussian 09, Revision A.1, Frisch, M. J.; Trucks, G. W.; Schlegel, H. B.; Scuseria, G. E.; Robb, M. A.; Cheeseman, J. R.; Scalmani, G.; Barone, V.; Mennucci, B.; Petersson, G. A.; Nakatsuji, H.; Caricato, M.; Li, X.; Hratchian, H. P.; Izmaylov, A. F.; Bloino, J.; Zheng, G.; Sonnenberg, J. L.; Hada, M.; Ehara, M.; Toyota, K.; Fukuda, R.; Hasegawa, J.; Ishida, M.; Nakajima, T.; Honda, Y.; Kitao, O.; Nakai, H.; Vreven, T.; Montgomery, Jr., J. A.; Peralta, J. E.; Ogliaro, F.; Bearpark, M.; Heyd, J. J.; Brothers, E.; Kudin, K. N.; Staroverov, V. N.; Kobayashi, R.; Normand, J.; Raghavachari, K.; Rendell, A.; Burant, J. C.; Iyengar, S. S.; Tomasi, J.; Cossi, M.; Rega, N.; Millam, N. J.; Klene, M.; Knox, J. E.; Cross, J. B.; Bakken, V.; Adamo, C.; Jaramillo, J.; Gomperts, R.; Stratmann, R. E.; Yazyev, O.; Austin, A. J.; Cammi, R.; Pomelli, C.; Ochterski, J. W.; Martin, R. L.; Morokuma, K.; Zakrzewski, V. G.; Voth, G. A.; Salvador, P.; Dannenberg, J. J.; Dapprich, S.; Daniels, A. D.; Farkas, Ö.; Foresman, J. B.; Ortiz, J. V.; Cioslowski, J.; Fox, D. J. Gaussian, Inc., Wallingford CT, **2009**.

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[i] Fan, Rui-Qang; Chen, Hong; Wang, Ping; Yang, Yu-Lin; Yin, Yan-Bing; Hasi, Wuliji. *J. Coord. Chem.* **2010,** *63*, 1514-1530.

[b] "A short history of SHELX". Sheldrick, G.M. *Acta Cryst.* **2008**, A64, 112-122.

[c] 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.

[d] Least Squares function minimized: (SHELXL97) $\Sigma w(Fo^2-Fc^2)^2$ where w = Least Squares weights.

[e] Standard deviation of an observation of unit weight: $[\Sigma w(Fo^2-Fc^2)^2/(No-Nv)]^{1/2}$
where: No = number of observations, Nv = number of variables

[f] Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

[g] Ibers, J. A. & Hamilton, W. C.; *Acta Cryst*. **1964,** 17, 781.

[h] 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).

[i] 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).

[j] CrystalStructure 3.8: Crystal Structure Analysis Package, Rigaku and Rigaku Americas (2000-2007). 9009 New Trails Dr. The Woodlands TX 77381 USA.

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

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