Supporting Information

A new tool for validating theoretically derived anisotropic displacement parameters with experiment: directionality of prolate displacement ellipsoids.

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Table S1 Crystal data, data collection parameters and convergence results for **2**.

Crystal data	
Chemical formula	$C_6H_{14}N_4NiO_2S_2$
M (g mol^{-1})	297.04
Crystal system, space group (No.)	Monoclinic, $P2_1/c$ (14)
Temperature (K)	100
a,b,c (Å)	8.505(4), 9.781(4), 7.345(3)
α,β,γ (°)	90, 96.148(7), 90
V (Å^3)	607.6(5)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	1.927
Crystal size (mm)	0.25 × 0.25 × 0.07
Data collection	
Diffractometer	Bruker APEX CCD
Absorption correction	multi-scan *SADABS* (Bruker, 2008)
T_{min}, T_{max}	0.548, 0.746
No. of measured, independent, observed [I>2σ(I)] reflections	9002, 1816, 1596
R_{int}	0.0355
Scan range (θ_{min}/ θ_{max})	2.4/30.9
Refinement results	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.0277, 0.0625, 1.047
No. of reflections	1816
No of parameters	82
No. of restraints	0
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.558, – 0.470

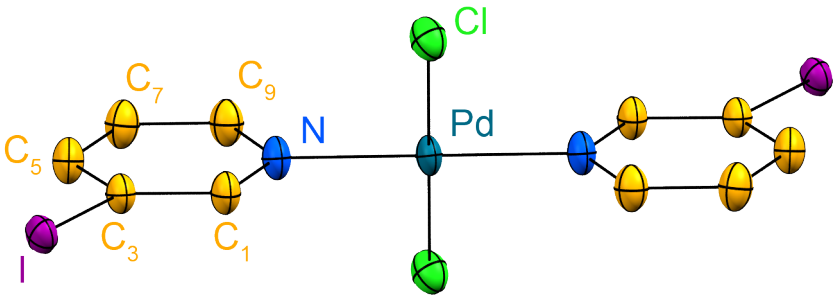

Fig. S1 Displacement ellipsoids in the harmonic approximation for compound **1** at 100 K, drawn at the 90% probability level.

Table S2 Theoretical ADPs (Å^2) for **1** at 100 K.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
I	0.010412	0.010608	0.007304	0.002111	0.003070	0.002558
Pd	0.011035	0.007139	0.005234	0.001531	0.002131	0.001766
Cl	0.013123	0.011192	0.008184	0.000380	0.001894	0.000829
N	0.013076	0.008264	0.006677	0.001835	0.002323	0.002336
C1	0.012880	0.008685	0.007250	0.002025	0.002555	0.003105
C3	0.010879	0.009058	0.007047	0.002063	0.001859	0.002120
C5	0.014773	0.008766	0.008541	0.002410	0.001978	0.003032
C7	0.016463	0.008882	0.009182	0.001027	0.002102	0.004219
C9	0.015275	0.009549	0.007827	0.000973	0.002890	0.003495

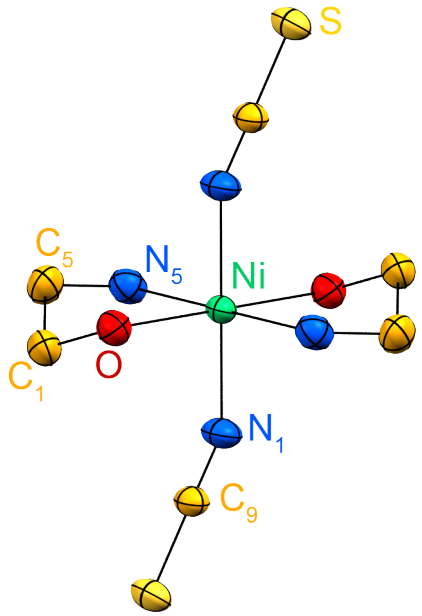

Fig. S2 As in Fig. S1 but for **2**.

Table S3 As in Table S2 but for **2**.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Ni1	0.009243	0.008550	0.010579	– 0.000073	0.001210	– 0.001420
S1	0.012490	0.014980	0.015485	0.001733	– 0.000993	– 0.004551
N1	0.014504	0.011550	0.017578	0.000080	0.001978	– 0.004210
N5	0.013180	0.014970	0.013242	– 0.002363	0.002181	– 0.001773
O1	0.012388	0.013200	0.013591	– 0.000179	0.000290	– 0.000228
C1	0.010967	0.014300	0.017172	– 0.001582	0.000498	0.000319
C5	0.014803	0.014980	0.018658	– 0.004470	0.002092	0.001694
C9	0.011553	0.009970	0.013133	0.000779	0.000992	– 0.001788

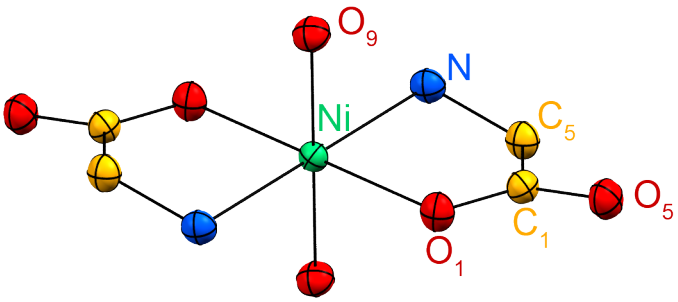

Fig. S3 As in Fig. S1 but for **3**.

Table S4 As in Table S2 but for **3**.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Ni1	0.006242	0.006420	0.006547	0.000060	0.003447	– 0.000045
O1	0.009291	0.009130	0.010793	0.001730	0.006175	0.000949
O5	0.009984	0.009490	0.011195	0.002910	0.004096	0.001142
O9	0.009939	0.009520	0.009106	– 0.001230	0.004828	– 0.000493
N1	0.007802	0.008780	0.009118	0.000482	0.004471	– 0.000431
C1	0.007712	0.007680	0.007519	0.000510	0.003463	0.000057
C5	0.008418	0.009270	0.009405	0.000529	0.005029	0.000621

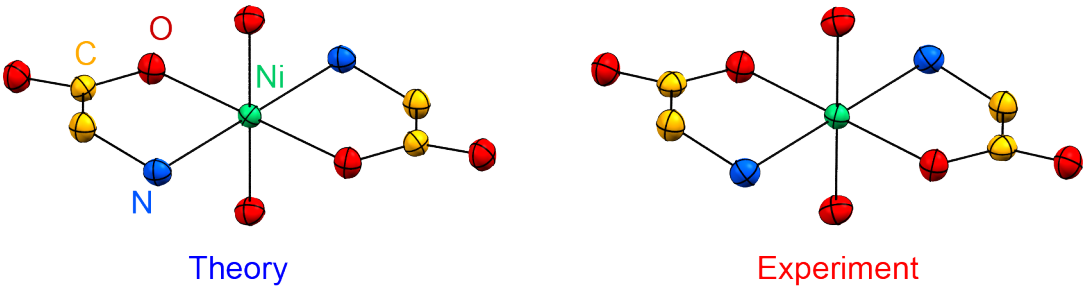

Fig. S4 Comparison of theoretical (left) and experimental (right) thermal ellipsoids of **3** at 100 K. All ellipsoids are drawn at the 90% probability level.

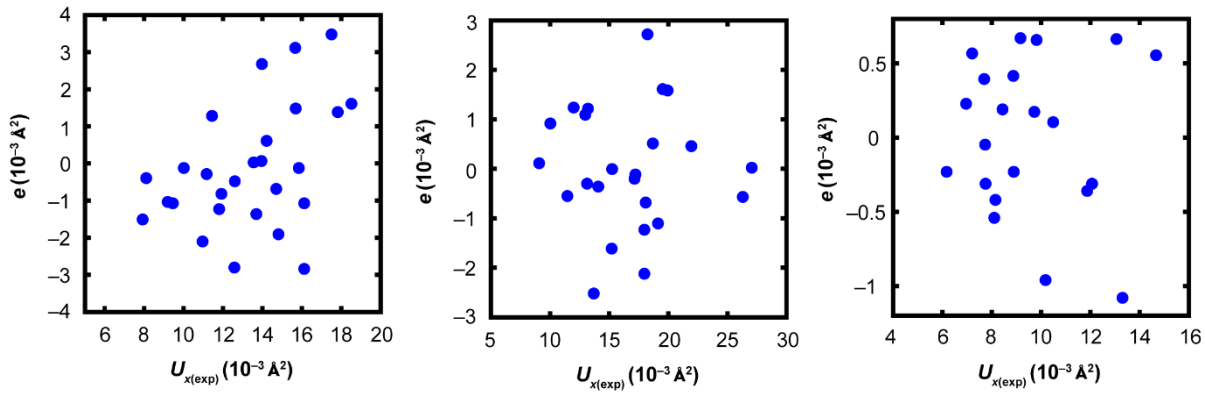

Fig. S5 Residuals for compound **1** (left), **2** (centre) and **3** (right).

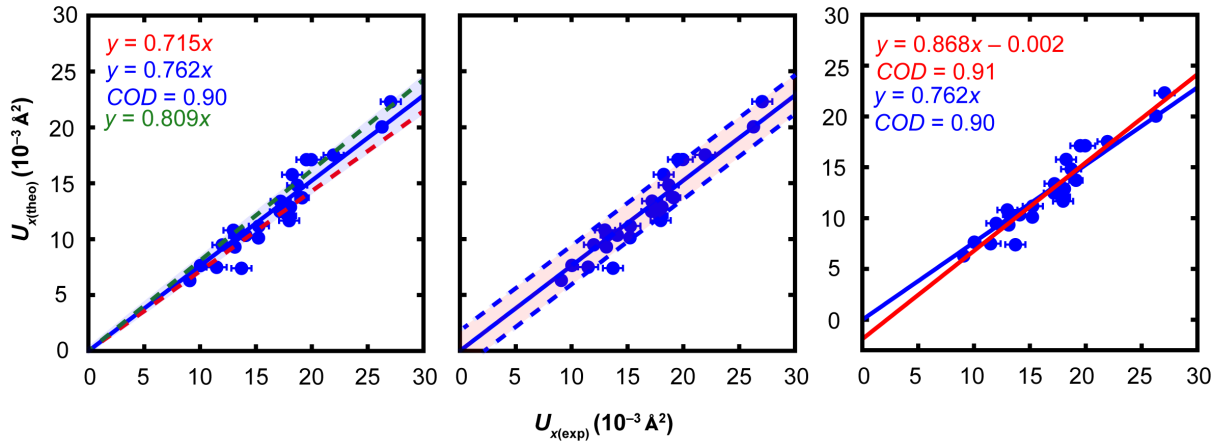

Fig. S6 Left: Scatter plot of theoretical main-axes components for all non-hydrogen atoms in **2** (theoretical ADPs based on the D2 correction) against the experimental values at 100 K, including a linear fit (blue) with its coefficient of determination (*COD*) and the two limiting slopes for three standard uncertainties (*su*) (red - lower limit, green - upper limit), corresponding to 99.7 % of the values in a Gaussian distribution. Centre: Scatter plot for **2** (blue) with tolerance interval representing a population of 90 % with a confidence of 99 % (light red, enclosed by dotted blue lines). Right: Comparison of the origin fit (blue) and a non-origin fit (red) for **1** with their *CODs*. In all plots, horizontal lines represent error bars of three *sus in* experimental data.

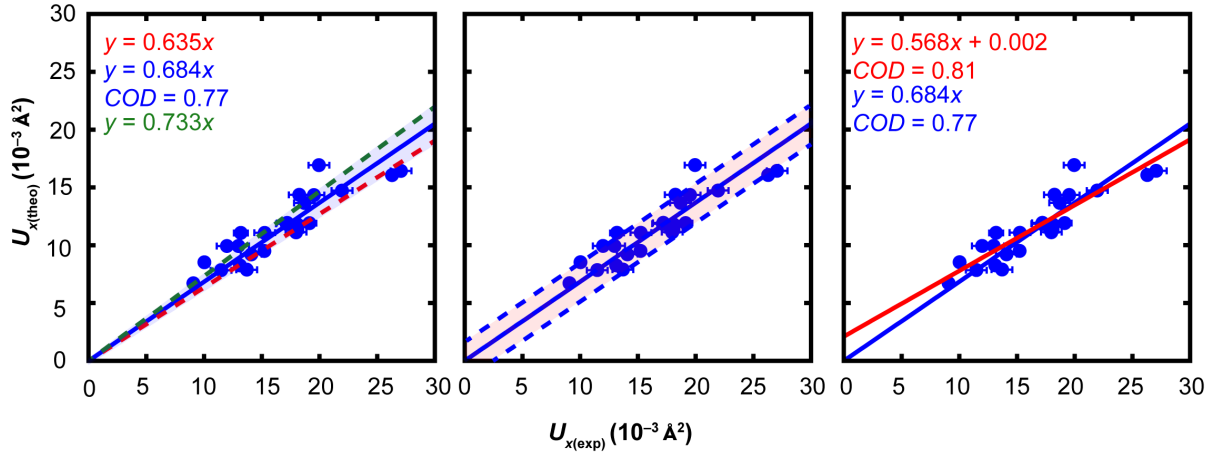

Fig. S7 As in Fig. S6 but for theoretical ADPs based on the TS correction.

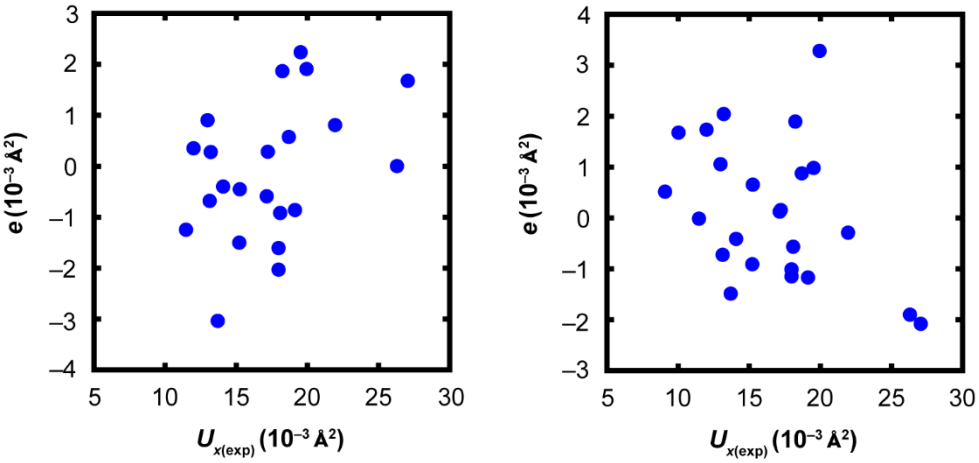

Fig. S8 Left: Residuals for compound **2** (D2 correction). Right: Residuals for compound **2** (TS correction).