Supplementary material for the article

Temperature resolved anisotropic displacement parameters from theory and experiment: a case study

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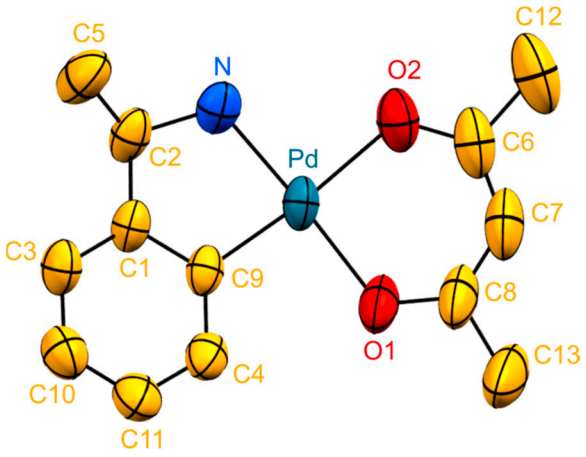

Figure S1 Displacement ellipsoids in the harmonic approximation for compound **1** at 130 K, drawn at the 90% probability level.

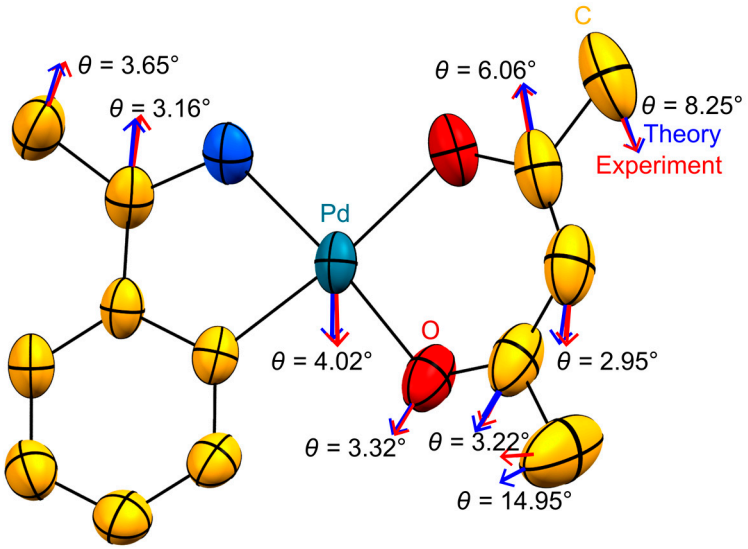

Figure S2 Comparison of the orientation of theoretical (blue) and experimental (red) main axis components for sufficiently anisotropic ($U_{max}/U_{min} > 2$) thermal ellipsoids, given for the high resolution control experiment at 100 K (90% probability).

The structure is drawn in the theoretically predicted crystallographic coordinate system. The deviation angles are given next to the corresponding atom.

Table S1 Deviation angles between experiment and theory (harmonic approximation) in the temperature range 150 - 250 K for the atoms shown in Fig. S1.

Angle for atom (°)	150 K	170 K	190 K	210 K	230 K	250 K
Pd	3.11	2.98	2.99	3.02	2.93	2.97
O1	4.86	4.82	5.41	5.79	5.95	5.84
O2	2.78	2.57	2.44	3.48	3.14	3.40
N1	5.53	5.09	5.86	4.98	5.60	4.29
C1	2.98	1.89	2.22	1.86	2.91	2.72
C2	1.36	1.03	1.80	2.110	2.62	2.80
C3	1.16	1.07	1.72	0.65	0.89	1.53
C4	10.53	9.9	-	9.71	-	12.04
C5	3.12	3.58	4.28	6.21	7.92	8.79
C6	3.44	4.09	3.82	4.42	4.16	5.04
C7	4.02	4.06	3.34	3.28	3.87	3.44
C8	5.51	5.78	5.19	4.63	7.18	6.19
C9	4.71	4.29	4.35	4.33	5.29	5.01
C10	-	-	10.52	10.50	9.27	8.05
C11	-	-	-	-	-	-
C12	9.75	9.08	8.78	7.51	6.76	6.35
C13	11.87	11.49	11.99	10.82	11.20	10.91

Table S2 As in Table S1, but for the quasi-harmonic approximation at 170 K.

Angle for atom (°)	170 K
Pd	1.94
O1	1.56
O2	2.13
N1	1.72
C1	0.47
C2	3.52
C3	2.18
C4	5.06
C5	1.28
C6	4.82
C7	3.52
C8	1.19
C9	1.61
C10	-
C11	-
C12	9.46
C13	12.04

Table S3 Summary of crystal data, data collection parameters and convergence results for all diffraction data on **1**.

Common features for refinements at all temperatures: chemical composition $C_{13}H_{17}NO_2Pd$, tetragonal space group $P4_1$ (76), $Z = 4$, 157 refined variables.

T [K]	100	130	150	170
crystal size [mm^3]	0.22·0.07·0.04	0.12·0.04·0.04	0.12·0.04·0.04	0.12·0.04·0.04
a [Å]	10.5607(17)	10.56720(18)	10.57390(18)	10.57980(18)
c [Å]	11.9201(9)	11.9841(2)	12.0054(2)	12.0233(2)
V [Å]	1329.4(4)	1338.21(5)	1342.29(5)	1345.79(5)
$\sin\theta_{max}/\lambda$ [Å^{-1}]	1.111	0.832	0.833	0.831
total no. of refls.	174274	40794	41033	41211
indep refls.	15292	5979	6038	6031
refls. $I > 2\sigma(I)$	12239	5872	5912	5877
$R1$	0.0274	0.0175	0.0170	0.0165
w$R2$	0.0594	0.0478	0.0465	0.0464
GOF	1.066	1.118	1.039	1.084
Flack parameter[a]	0.029(2)	0.049(6)	0.027(6)	0.046(7)
CCDC dep. no.	2143154	1997131	1997132	1997133

T [K]	190	210	230	250
crystal size [mm^3]	0.12·0.04·0.04	0.12·0.04·0.04	0.12·0.04·0.04	0.12·0.04·0.04
a [Å]	10.58700(18)	10.59500(18)	10.60060(18)	10.60730(18)
c [Å]	12.0458(2)	12.0688(2)	12.0908(2)	12.1138(2)
V [Å]	1350.15(5)	1354.77(5)	1358.68(5)	1362.98(5)
$\sin\theta_{max}/\lambda$ [Å^{-1}]	0.830	0.830	0.829	0.828
total no. of refls.	41031	41303	41530	41908
indep refls.	5978	5997	6024	6044
refls. $I > 2\sigma(I)$	5806	5769	5753	5730
$R1$	0.0166	0.0162	0.0165	0.0163
w$R2$	0.0474	0.0473	0.0462	0.0453
GOF	1.057	1.044	1.116	1.050
Flack parameter[a]	0.045(8)	0.041(7)	0.042(7)	0.052(8)
CCDC dep. no.	1997134	1997135	1997136	1997137

[a] H. D. Flack, *Acta Crystallogr.* **1983**, *A39*, 876–881.