Electronic Supplementary Material (ESI) for CrystEngComm


# On the high-temperature phase of barbituric acid

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## Supporting information

**Table S1.** Experimental[a] and calculated[b] $^1$H chemical shift tensors (ppm) and shielding anisotropy (ppm) of BAc HT-form. The shielding anisotropy is defined as $\Delta\sigma = \sigma_{33} - (\sigma_{11} + \sigma_{22})/2$ with $\sigma_{11} \leq \sigma_{22} \leq \sigma_{33}$ and $\delta_{ii} = -\sigma_{ii}$.

| $^1$H δ | hydrogen atom | $^1$H $\delta_{11}$ | $^1$H $\delta_{22}$ | $^1$H $\delta_{33}$ | Δσ |
|---|---|---|---|---|---|
| Experimental | | | | | |
| 9.6 | H1/H3 | 16.0 | 16.0 | -3.2 | 19.2 |
| 4.4 | H5 | 10.3 | 10.3 | -7.4 | 17.8 |
| Computed | | | | | |
| 6.8 | H1/H3 | 9.6 | 6.8 | 4.1 | 4.2 |
| 3.3 | H5a | 6.4 | 3.3 | 0.1 | 4.8 |
| 3.3 | H5b | 6.3 | 3.6 | 0.1 | 4.9 |

[a] Chemical shifts and principal tensor values refer to spectra aquired at 233 °C during the phase transition. The chemical shifts of HT-form are reported in the article. [b] Gas phase, DFT at the B3LYP/6-311++G-(d,p) level.

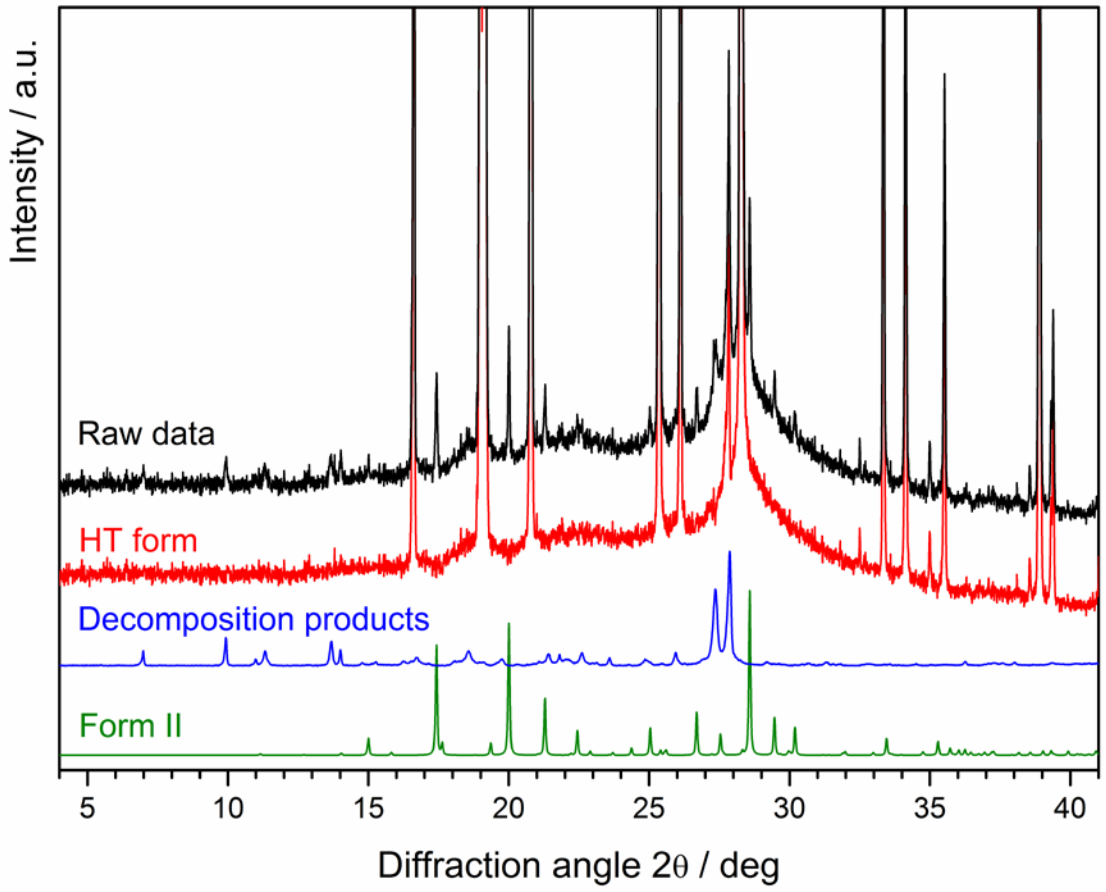


**Figure S2.** X-ray powder diffraction pattern (Cu $K\alpha_1$ radiation) of the HT-form of barbituric acid at 236(1) °C. This form nearly always contains small fractions of form II, of decomposition products, or both. Contributions of these impurities have to be deducted from the raw XRPD data.

**Table S3.** Crystal structure of HT-form of barbituric acid at T = 236(1) °C.

**Symmetry and unit cell**

| | |
|---|---|
| Crystal system | Monoclinic |
| Space group | *C*2/*c* (15) |
| *a* | 8.5302(3) Å |
| *b* | 6.8167(3) Å |
| *c* | 9.3304(4) Å |
| *β* | 89.865(2)° |
| Volume | 542.54(4) Å$^3$ |

**Fractional coordinates, isotropic or equivalent displacement factors, site occupation factors**

| Atom | Type | Wyckoff | *x* | *y* | *z* | U / Å$^2$ | Occupation | Utype |
|---|---|---|---|---|---|---|---|---|
| N1 | N | 4*e* | 0.0 | 0.4925(4) | 0.25 | 0.093(2) | 1.0 | Uiso |
| H1 | H | 4*e* | 0.0 | 0.6406(4) | 0.25 | 0.093(2) | 1.0 | Uiso |
| C2 | C | 8*f* | -0.0967(6) | 0.4004(3) | 0.3469(5) | 0.078(4) | 0.5 | Uiso |
| N3 | N | 8*f* | -0.0846(6) | 0.1970(5) | 0.3504(5) | 0.043(3) | 0.5 | Uiso |
| H3 | H | 8*f* | -0.1421(6) | 0.1266(5) | 0.4298(5) | 0.043(3) | 0.5 | Uiso |
| C4 | C | 4*e* | 0.0 | 0.0798(3) | 0.25 | 0.108(8) | 1.0 | Uani |
| C5 | C | 8*f* | 0.0846(6) | 0.1970(5) | 0.1496(5) | 0.238(16) | 0.5 | Uani |
| H5a | H | 8*f* | 0.0394(6) | 0.1599(5) | 0.0447(5) | 0.238(16) | 0.5 | Uani |
| H5b | H | 8*f* | 0.2040(6) | 0.1432(5) | 0.1521(5) | 0.238(16) | 0.5 | Uani |
| C6 | C | 8*f* | 0.0967(6) | 0.4004(3) | 0.1531(5) | 0.154(7) | 0.5 | Uani |
| O7 | O | 8*f* | -0.1805(4) | 0.4850(4) | 0.4326(3) | 0.098 | 0.5 | Uiso |
| O8 | O | 4*e* | 0.0 | -0.0995(3) | 0.25 | 0.250(10) | 1.0 | Uani |
| O9 | O | 8*f* | 0.1805(4) | 0.4850(4) | 0.0674(3) | 0.154(7) | 0.5 | Uani |

**Anisotropic displacement factors**

| Atom | $u_{11}$/ Å$^2$ | $u_{22}$ /Å$^2$ | $u_{33}$ / Å$^2$ | $u_{12}$ / Å$^2$ | $u_{13}$ / Å$^2$ | $u_{23}$ / Å$^2$ |
|---|---|---|---|---|---|---|
| C4 | 0.136(10) | 0.056(4) | 0.132(11) | 0.0 | 0.009(7) | 0.0 |
| C5 | 0.51(3) | 0.056(4) | 0.148(14) | 0.0 | 0.077(12) | 0.0 |
| H5a | 0.51(3) | 0.056(4) | 0.148(14) | 0.0 | 0.077(12) | 0.0 |
| H5b | 0.51(3) | 0.056(4) | 0.148(14) | 0.0 | 0.077(12) | 0.0 |
| C6 | 0.265(9) | 0.056(4) | 0.139(7) | 0.0 | 0.121(6) | 0.0 |
| O8 | 0.379(12) | 0.056(4) | 0.315(13) | 0.0 | -0.049(8) | 0.0 |
| O9 | 0.265(9) | 0.056(4) | 0.139(7) | 0.0 | 0.121(6) | 0.0 |

Note that due to the site symmetry 4*e* of the molecule as a whole the following pairs of atoms occupy a common site: C2/C6, O7/O9, and N3/C5. Also the isotropic and anisotropic displacement factors of hydrogen atoms were refined with the same values as the bonded heavy atom.

**Bond distances and angles**

| Atoms | Bond length / Å |
|---|---|
| N1-H1 | 1.010(4) |
| N1-C2 | 1.374(5) |
| N1-C6 | 1.374(5) |
| C2-N3 | 1.391(4) |
| C2-O7 | 1.217(5) |
| N3-C4 | 1.426(5) |
| N3-H3 | 1.009(6) |
| C4-C5 | 1.426(5) |
| C4-O8 | 1.222(3) |
| C5-C6 | 1.391(4) |
| C5-H5a | 1.083(7) |
| C5-H5b | 1.083(7) |
| C6-O9 | 1.217(5) |

| Atoms | Bond angle / deg |
|---|---|
| C2-N1-H1 | 117.2(4) |
| C6-N1-H1 | 117.2(4) |
| C2-N1-C6 | 125.6(6) |
| N1-C2-N3 | 115.2(4) |
| N1-C2-O7 | 124.5(4) |
| N3-C2-O7 | 120.0(5) |
| C2-N3-C4 | 125.5(4) |
| C2-N3-H3 | 117.1(6) |
| C4-N3-H3 | 117.4(6) |
| N3-C4-C5 | 111.9(5) |
| N3-C4-O8 | 124.1(4) |
| C5-C4-O8 | 124.1(4) |
| C4-C5-C6 | 125.5(4) |
| C4-C5-H5a | 106.4(5) |
| C4-C5-H5b | 105.7(6) |
| C6-C5-H5a | 106.3(5) |
| C6-C5-H5b | 105.5(5) |
| H5a-C5-H5b | 106.1(8) |
| N1-C6-C5 | 115.2(4) |
| N1-C6-O9 | 124.5(4) |
| C5-C6-O9 | 120.0(5) |

**Table S4.** List of crystal structure information files.

| | |
|---|---|
| HT-BAc-509K-X-ray_diffraction.cif | Observed & calculated X-ray powder diffraction pattern of HT-BAc |
| HT-BAc-509K-Neutron_diffraction.cif | Observed & calculated neutron powder diffraction pattern of HT-BAc |
| HT-BAc-C2c-disorder_Xn.cif | Experimental structure of HT-BAc |
| BAc-form_I-BLYP.cif | BLYP-optimized structure of BAc form I |
| BAc-form_II-BLYP.cif | BLYP-optimized structure of BAc form II |
| HT-BAc-BLYP-C2c-ordered.cif | BLYP-optimized structure of BAc in the unit cell of HT-BAc with an ordered arrangement in space group *C*2/*c* |
| HT-BAc-BLYP-Cc-ordered.cif | BLYP-optimized structure of BAc in the unit cell of HT-BAc with an ordered arrangement in space group *Cc* |
| HT-BAc-BLYP-P21c-ordered.cif | BLYP-optimized structure of BAc in the unit cell of HT-BAc with an ordered arrangement in space group $P2_1/c$ |

**Figure S5.** Thermal expansion of BAc II and HT-BAc. The direct plot of the experimental lattice parameters shows discontinuities at the phase transition, which do not appear for the properly related structural parameters (figure 8 of main paper).

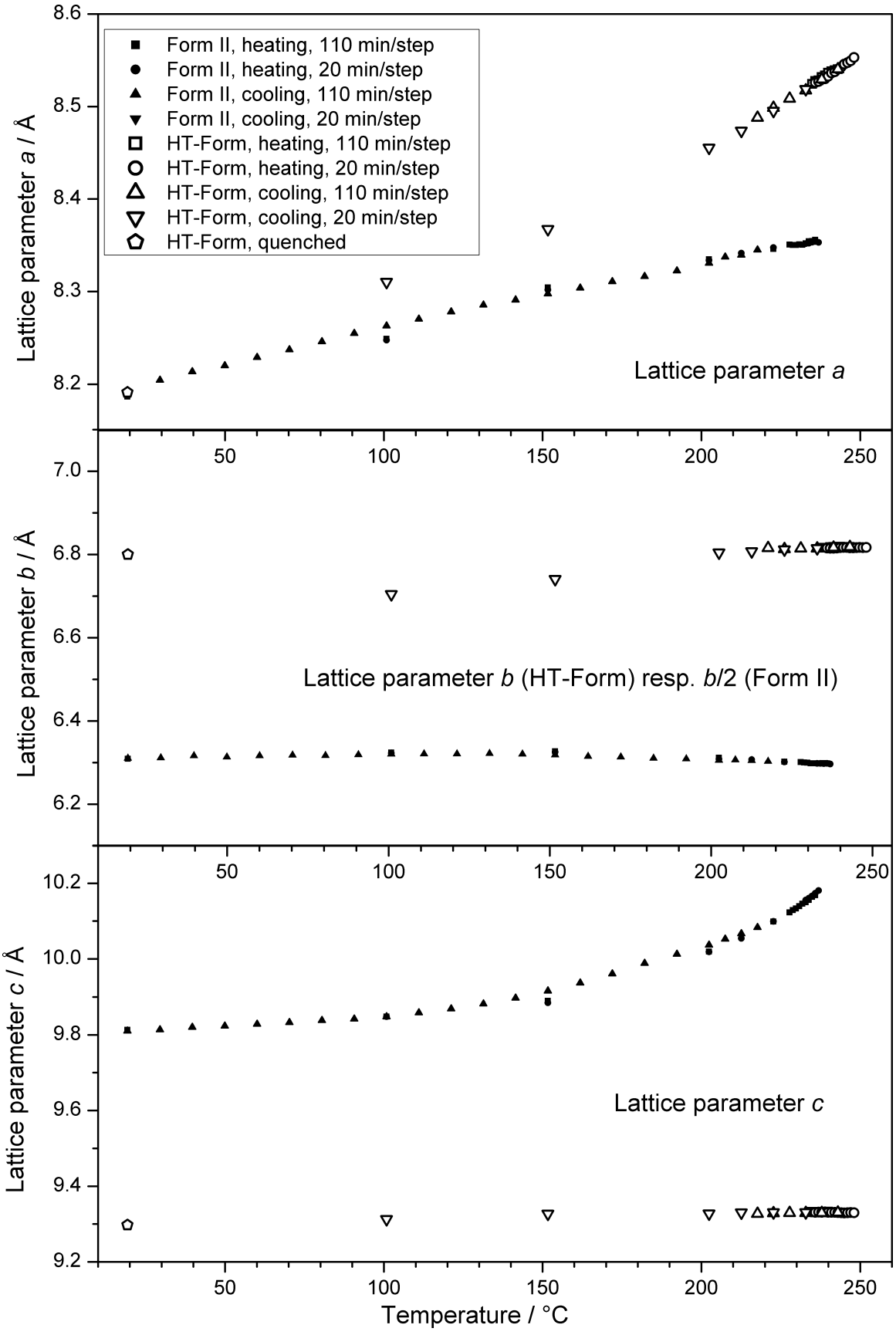

**Table S6.** Thermal expansion of BAc II and HT-BAc. Experimental and calculated lattice parameters and structural parameters at various temperatures and heating ranges.

| Temperature / °C | *a* / Å | s.u. | *b* / Å | s.u. | *c* / Å | s.u. | *β* / deg | s.u. | Volume / Å$^3$ | s.u. | *d*1 / Å | s.u. | *d*2 / Å | s.u. | *d*3 / Å | s.u. | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 101 | 8.248 | 0.004 | 12.640 | 0.008 | 9.848 | 0.007 | 94.77 | 0.04 | 1023.5 | 1.1 | 6.058 | 0.005 | 6.680 | 0.006 | 6.323 | 0.004 | Form II, heating, 20 min/step |
| 152 | 8.302 | 0.003 | 12.651 | 0.006 | 9.885 | 0.004 | 94.37 | 0.03 | 1035.1 | 0.8 | 6.114 | 0.003 | 6.692 | 0.004 | 6.325 | 0.003 | Form II, heating, 20 min/step |
| 202 | 8.334 | 0.003 | 12.617 | 0.005 | 10.019 | 0.004 | 94.08 | 0.03 | 1050.8 | 0.7 | 6.178 | 0.003 | 6.740 | 0.004 | 6.308 | 0.003 | Form II, heating, 20 min/step |
| 213 | 8.341 | 0.003 | 12.614 | 0.005 | 10.054 | 0.004 | 94.03 | 0.02 | 1055.3 | 0.7 | 6.193 | 0.003 | 6.754 | 0.003 | 6.307 | 0.002 | Form II, heating, 20 min/step |
| 223 | 8.348 | 0.003 | 12.603 | 0.005 | 10.099 | 0.004 | 93.98 | 0.02 | 1059.9 | 0.7 | 6.210 | 0.003 | 6.771 | 0.003 | 6.301 | 0.002 | Form II, heating, 20 min/step |
| 233 | 8.352 | 0.002 | 12.596 | 0.004 | 10.155 | 0.003 | 93.93 | 0.02 | 1065.8 | 0.5 | 6.229 | 0.002 | 6.792 | 0.003 | 6.298 | 0.002 | Form II, heating, 20 min/step |
| 234 | 8.352 | 0.002 | 12.596 | 0.004 | 10.160 | 0.003 | 93.92 | 0.02 | 1066.4 | 0.6 | 6.231 | 0.003 | 6.793 | 0.003 | 6.298 | 0.002 | Form II, heating, 20 min/step |
| 235 | 8.353 | 0.002 | 12.595 | 0.004 | 10.166 | 0.003 | 93.92 | 0.02 | 1067.1 | 0.6 | 6.234 | 0.003 | 6.796 | 0.003 | 6.298 | 0.002 | Form II, heating, 20 min/step |
| 236 | 8.354 | 0.003 | 12.596 | 0.006 | 10.174 | 0.004 | 93.91 | 0.03 | 1068.0 | 0.7 | 6.236 | 0.003 | 6.799 | 0.003 | 6.298 | 0.003 | Form II, heating, 20 min/step |
| 237 | 8.353 | 0.006 | 12.593 | 0.012 | 10.181 | 0.007 | 93.92 | 0.06 | 1068.4 | 1.5 | 6.237 | 0.006 | 6.802 | 0.007 | 6.297 | 0.006 | Form II, heating, 20 min/step |
| 19 | 8.187 | 0.002 | 12.618 | 0.004 | 9.813 | 0.004 | 95.31 | 0.02 | 1009.4 | 0.6 | 5.992 | 0.003 | 6.674 | 0.003 | 6.309 | 0.002 | Form II, heating, 110 min/step |
| 101 | 8.249 | 0.002 | 12.648 | 0.004 | 9.848 | 0.004 | 94.78 | 0.02 | 1023.9 | 0.6 | 6.058 | 0.003 | 6.682 | 0.003 | 6.324 | 0.002 | Form II, heating, 110 min/step |
| 152 | 8.304 | 0.001 | 12.653 | 0.003 | 9.889 | 0.002 | 94.38 | 0.01 | 1036.1 | 0.4 | 6.115 | 0.002 | 6.695 | 0.002 | 6.327 | 0.001 | Form II, heating, 110 min/step |
| 202 | 8.335 | 0.001 | 12.623 | 0.002 | 10.019 | 0.002 | 94.10 | 0.01 | 1051.4 | 0.3 | 6.178 | 0.001 | 6.742 | 0.002 | 6.311 | 0.001 | Form II, heating, 110 min/step |
| 223 | 8.346 | 0.001 | 12.604 | 0.002 | 10.099 | 0.002 | 93.98 | 0.01 | 1059.7 | 0.3 | 6.209 | 0.001 | 6.770 | 0.002 | 6.302 | 0.001 | Form II, heating, 110 min/step |
| 228 | 8.351 | 0.001 | 12.602 | 0.002 | 10.123 | 0.002 | 93.96 | 0.01 | 1062.7 | 0.3 | 6.219 | 0.001 | 6.780 | 0.001 | 6.301 | 0.001 | Form II, heating, 110 min/step |
| 229 | 8.350 | 0.001 | 12.600 | 0.002 | 10.129 | 0.002 | 93.95 | 0.01 | 1063.1 | 0.3 | 6.221 | 0.001 | 6.782 | 0.001 | 6.300 | 0.001 | Form II, heating, 110 min/step |
| 230 | 8.350 | 0.001 | 12.599 | 0.002 | 10.133 | 0.002 | 93.94 | 0.01 | 1063.5 | 0.3 | 6.223 | 0.001 | 6.783 | 0.001 | 6.299 | 0.001 | Form II, heating, 110 min/step |
| 231 | 8.351 | 0.001 | 12.596 | 0.002 | 10.139 | 0.002 | 93.93 | 0.01 | 1064.0 | 0.3 | 6.225 | 0.001 | 6.785 | 0.001 | 6.298 | 0.001 | Form II, heating, 110 min/step |
| 232 | 8.350 | 0.001 | 12.596 | 0.002 | 10.146 | 0.001 | 93.93 | 0.01 | 1064.7 | 0.3 | 6.226 | 0.001 | 6.788 | 0.001 | 6.298 | 0.001 | Form II, heating, 110 min/step |
| 233 | 8.352 | 0.001 | 12.596 | 0.002 | 10.149 | 0.002 | 93.93 | 0.01 | 1065.2 | 0.3 | 6.228 | 0.001 | 6.789 | 0.001 | 6.298 | 0.001 | Form II, heating, 110 min/step |
| 234 | 8.354 | 0.001 | 12.596 | 0.002 | 10.155 | 0.001 | 93.93 | 0.01 | 1066.1 | 0.3 | 6.230 | 0.001 | 6.792 | 0.001 | 6.298 | 0.001 | Form II, heating, 110 min/step |
| 235 | 8.354 | 0.001 | 12.597 | 0.003 | 10.164 | 0.002 | 93.92 | 0.01 | 1067.1 | 0.3 | 6.233 | 0.001 | 6.795 | 0.002 | 6.298 | 0.001 | Form II, heating, 110 min/step |
| 236 | 8.356 | 0.002 | 12.596 | 0.004 | 10.168 | 0.002 | 93.91 | 0.02 | 1067.7 | 0.4 | 6.235 | 0.002 | 6.797 | 0.002 | 6.298 | 0.002 | Form II, heating, 110 min/step |
| | | | | | | | | | | | | | | | | | |
| 218 | 8.345 | 0.002 | 12.606 | 0.004 | 10.083 | 0.003 | 93.99 | 0.02 | 1058.2 | 0.5 | 6.205 | 0.002 | 6.765 | 0.002 | 6.303 | 0.002 | Form II, cooling, 110 min/step |
| 213 | 8.339 | 0.002 | 12.610 | 0.003 | 10.067 | 0.002 | 94.01 | 0.02 | 1056.1 | 0.4 | 6.197 | 0.002 | 6.757 | 0.002 | 6.305 | 0.002 | Form II, cooling, 110 min/step |
| 208 | 8.337 | 0.002 | 12.613 | 0.003 | 10.053 | 0.002 | 94.04 | 0.02 | 1054.5 | 0.4 | 6.191 | 0.002 | 6.752 | 0.002 | 6.307 | 0.002 | Form II, cooling, 110 min/step |
| 202 | 8.331 | 0.002 | 12.611 | 0.004 | 10.037 | 0.003 | 94.06 | 0.02 | 1051.8 | 0.5 | 6.183 | 0.002 | 6.745 | 0.002 | 6.305 | 0.002 | Form II, cooling, 110 min/step |
| 192 | 8.322 | 0.002 | 12.618 | 0.004 | 10.013 | 0.003 | 94.12 | 0.02 | 1048.7 | 0.5 | 6.170 | 0.002 | 6.736 | 0.002 | 6.309 | 0.002 | Form II, cooling, 110 min/step |
| 182 | 8.316 | 0.002 | 12.621 | 0.004 | 9.988 | 0.003 | 94.16 | 0.02 | 1045.6 | 0.5 | 6.158 | 0.002 | 6.726 | 0.003 | 6.311 | 0.002 | Form II, cooling, 110 min/step |
| 172 | 8.311 | 0.002 | 12.627 | 0.004 | 9.961 | 0.003 | 94.20 | 0.02 | 1042.4 | 0.6 | 6.146 | 0.002 | 6.716 | 0.003 | 6.313 | 0.002 | Form II, cooling, 110 min/step |
| 162 | 8.304 | 0.002 | 12.630 | 0.004 | 9.937 | 0.003 | 94.25 | 0.02 | 1039.2 | 0.6 | 6.134 | 0.003 | 6.707 | 0.003 | 6.315 | 0.002 | Form II, cooling, 110 min/step |
| 152 | 8.298 | 0.002 | 12.636 | 0.004 | 9.916 | 0.003 | 94.33 | 0.02 | 1036.7 | 0.6 | 6.122 | 0.003 | 6.700 | 0.003 | 6.318 | 0.002 | Form II, cooling, 110 min/step |
| 142 | 8.291 | 0.002 | 12.641 | 0.005 | 9.897 | 0.003 | 94.39 | 0.02 | 1034.3 | 0.6 | 6.111 | 0.003 | 6.695 | 0.003 | 6.320 | 0.002 | Form II, cooling, 110 min/step |
| 131 | 8.286 | 0.002 | 12.644 | 0.005 | 9.882 | 0.003 | 94.45 | 0.02 | 1032.1 | 0.6 | 6.101 | 0.003 | 6.690 | 0.003 | 6.322 | 0.002 | Form II, cooling, 110 min/step |
| 121 | 8.278 | 0.002 | 12.642 | 0.005 | 9.869 | 0.003 | 94.51 | 0.02 | 1029.6 | 0.6 | 6.091 | 0.003 | 6.685 | 0.003 | 6.321 | 0.002 | Form II, cooling, 110 min/step |
| 111 | 8.270 | 0.002 | 12.642 | 0.005 | 9.859 | 0.003 | 94.58 | 0.02 | 1027.5 | 0.6 | 6.081 | 0.003 | 6.682 | 0.003 | 6.321 | 0.003 | Form II, cooling, 110 min/step |
| 101 | 8.263 | 0.002 | 12.641 | 0.005 | 9.848 | 0.003 | 94.65 | 0.02 | 1025.2 | 0.6 | 6.072 | 0.003 | 6.679 | 0.003 | 6.320 | 0.002 | Form II, cooling, 110 min/step |
| 91 | 8.255 | 0.002 | 12.638 | 0.005 | 9.842 | 0.003 | 94.73 | 0.02 | 1023.2 | 0.6 | 6.062 | 0.003 | 6.678 | 0.003 | 6.319 | 0.002 | Form II, cooling, 110 min/step |
| 80 | 8.246 | 0.002 | 12.634 | 0.005 | 9.838 | 0.003 | 94.80 | 0.02 | 1021.3 | 0.6 | 6.053 | 0.003 | 6.678 | 0.003 | 6.317 | 0.002 | Form II, cooling, 110 min/step |
| 70 | 8.237 | 0.002 | 12.636 | 0.005 | 9.833 | 0.003 | 94.90 | 0.02 | 1019.7 | 0.6 | 6.042 | 0.003 | 6.678 | 0.003 | 6.318 | 0.002 | Form II, cooling, 110 min/step |
| 60 | 8.229 | 0.002 | 12.633 | 0.005 | 9.828 | 0.003 | 94.98 | 0.02 | 1017.9 | 0.6 | 6.033 | 0.003 | 6.677 | 0.003 | 6.316 | 0.002 | Form II, cooling, 110 min/step |
| 50 | 8.220 | 0.002 | 12.626 | 0.005 | 9.823 | 0.003 | 95.04 | 0.02 | 1015.6 | 0.6 | 6.025 | 0.003 | 6.676 | 0.003 | 6.313 | 0.003 | Form II, cooling, 110 min/step |
| 40 | 8.214 | 0.002 | 12.633 | 0.005 | 9.820 | 0.003 | 95.13 | 0.02 | 1014.9 | 0.6 | 6.016 | 0.002 | 6.677 | 0.003 | 6.316 | 0.002 | Form II, cooling, 110 min/step |
| 29 | 8.204 | 0.002 | 12.623 | 0.005 | 9.813 | 0.003 | 95.19 | 0.02 | 1012.1 | 0.6 | 6.007 | 0.003 | 6.674 | 0.003 | 6.312 | 0.002 | Form II, cooling, 110 min/step |
| 19 | 8.193 | 0.002 | 12.621 | 0.005 | 9.810 | 0.003 | 95.26 | 0.02 | 1010.1 | 0.6 | 5.997 | 0.002 | 6.673 | 0.003 | 6.310 | 0.002 | Form II, cooling, 110 min/step |
| 236 | 8.526 | 0.005 | 6.816 | 0.004 | 9.331 | 0.006 | 89.87 | 0.05 | 542.2 | 0.6 | 6.301 | 0.005 | 6.816 | 0.004 | 6.313 | 0.005 | HT-Form, heating, 20 min/step |
| 237 | 8.527 | 0.003 | 6.815 | 0.002 | 9.331 | 0.003 | 89.87 | 0.03 | 542.3 | 0.3 | 6.302 | 0.003 | 6.815 | 0.002 | 6.313 | 0.003 | HT-Form, heating, 20 min/step |
| 238 | 8.529 | 0.002 | 6.815 | 0.002 | 9.331 | 0.003 | 89.86 | 0.02 | 542.4 | 0.3 | 6.303 | 0.002 | 6.815 | 0.002 | 6.313 | 0.002 | HT-Form, heating, 20 min/step |
| 239 | 8.530 | 0.004 | 6.816 | 0.003 | 9.332 | 0.004 | 89.84 | 0.03 | 542.5 | 0.4 | 6.305 | 0.004 | 6.816 | 0.003 | 6.313 | 0.004 | HT-Form, heating, 20 min/step |
| 240 | 8.533 | 0.004 | 6.816 | 0.003 | 9.331 | 0.004 | 89.83 | 0.03 | 542.7 | 0.4 | 6.306 | 0.004 | 6.816 | 0.003 | 6.313 | 0.004 | HT-Form, heating, 20 min/step |
| 241 | 8.536 | 0.004 | 6.817 | 0.003 | 9.331 | 0.004 | 89.83 | 0.03 | 543.0 | 0.4 | 6.308 | 0.004 | 6.817 | 0.003 | 6.314 | 0.004 | HT-Form, heating, 20 min/step |
| 242 | 8.538 | 0.004 | 6.817 | 0.003 | 9.331 | 0.004 | 89.81 | 0.03 | 543.1 | 0.4 | 6.309 | 0.004 | 6.817 | 0.003 | 6.314 | 0.004 | HT-Form, heating, 20 min/step |
| 243 | 8.540 | 0.004 | 6.816 | 0.003 | 9.330 | 0.004 | 89.80 | 0.03 | 543.1 | 0.4 | 6.311 | 0.004 | 6.816 | 0.003 | 6.313 | 0.004 | HT-Form, heating, 20 min/step |
| 244 | 8.543 | 0.004 | 6.816 | 0.003 | 9.329 | 0.004 | 89.80 | 0.03 | 543.2 | 0.4 | 6.311 | 0.004 | 6.816 | 0.003 | 6.314 | 0.004 | HT-Form, heating, 20 min/step |
| 245 | 8.546 | 0.004 | 6.816 | 0.003 | 9.329 | 0.004 | 89.78 | 0.03 | 543.4 | 0.4 | 6.314 | 0.004 | 6.816 | 0.003 | 6.314 | 0.004 | HT-Form, heating, 20 min/step |
| 246 | 8.547 | 0.004 | 6.817 | 0.003 | 9.330 | 0.004 | 89.77 | 0.03 | 543.6 | 0.4 | 6.315 | 0.004 | 6.817 | 0.003 | 6.314 | 0.004 | HT-Form, heating, 20 min/step |
| 247 | 8.549 | 0.004 | 6.817 | 0.003 | 9.330 | 0.004 | 89.76 | 0.03 | 543.7 | 0.4 | 6.317 | 0.004 | 6.817 | 0.003 | 6.314 | 0.004 | HT-Form, heating, 20 min/step |
| 248 | 8.553 | 0.004 | 6.817 | 0.003 | 9.329 | 0.004 | 89.75 | 0.03 | 544.0 | 0.4 | 6.318 | 0.004 | 6.817 | 0.003 | 6.314 | 0.004 | HT-Form, heating, 20 min/step |
| 235 | 8.524 | 0.008 | 6.816 | 0.002 | 9.332 | 0.006 | 89.92 | 0.07 | 542.2 | 0.6 | 6.298 | 0.007 | 6.816 | 0.002 | 6.315 | 0.007 | HT-Form, heating, 110 min/step |
| 236 | 8.527 | 0.004 | 6.816 | 0.001 | 9.330 | 0.003 | 89.92 | 0.03 | 542.3 | 0.3 | 6.299 | 0.004 | 6.816 | 0.001 | 6.316 | 0.004 | HT-Form, heating, 110 min/step |
| 237 | 8.528 | 0.002 | 6.817 | 0.001 | 9.332 | 0.002 | 89.90 | 0.02 | 542.6 | 0.2 | 6.301 | 0.002 | 6.817 | 0.001 | 6.315 | 0.002 | HT-Form, heating, 110 min/step |
| 238 | 8.532 | 0.002 | 6.818 | 0.001 | 9.332 | 0.002 | 89.89 | 0.02 | 542.8 | 0.2 | 6.303 | 0.002 | 6.818 | 0.001 | 6.316 | 0.002 | HT-Form, heating, 110 min/step |
| 239 | 8.534 | 0.002 | 6.818 | 0.001 | 9.332 | 0.002 | 89.87 | 0.02 | 543.0 | 0.2 | 6.304 | 0.002 | 6.818 | 0.001 | 6.316 | 0.002 | HT-Form, heating, 110 min/step |
| 240 | 8.536 | 0.002 | 6.818 | 0.001 | 9.332 | 0.002 | 89.87 | 0.02 | 543.2 | 0.2 | 6.306 | 0.002 | 6.818 | 0.001 | 6.316 | 0.002 | HT-Form, heating, 110 min/step |
| 241 | 8.538 | 0.002 | 6.817 | 0.001 | 9.330 | 0.002 | 89.86 | 0.02 | 543.1 | 0.2 | 6.307 | 0.002 | 6.817 | 0.001 | 6.316 | 0.002 | HT-Form, heating, 110 min/step |
| 242 | 8.539 | 0.002 | 6.818 | 0.001 | 9.330 | 0.002 | 89.84 | 0.02 | 543.2 | 0.2 | 6.308 | 0.002 | 6.818 | 0.001 | 6.315 | 0.002 | HT-Form, heating, 110 min/step |
| 243 | 8.540 | 0.002 | 6.818 | 0.001 | 9.330 | 0.002 | 89.83 | 0.02 | 543.3 | 0.2 | 6.309 | 0.002 | 6.818 | 0.001 | 6.315 | 0.002 | HT-Form, heating, 110 min/step |
| 233 | 8.519 | 0.003 | 6.815 | 0.003 | 9.331 | 0.004 | 89.93 | 0.03 | 541.7 | 0.4 | 6.295 | 0.003 | 6.815 | 0.003 | 6.313 | 0.003 | HT-Form, cooling, 20 min/step |
| 223 | 8.495 | 0.003 | 6.812 | 0.003 | 9.331 | 0.004 | 90.04 | 0.03 | 540.0 | 0.4 | 6.280 | 0.003 | 6.812 | 0.003 | 6.312 | 0.004 | HT-Form, cooling, 20 min/step |
| 213 | 8.474 | 0.003 | 6.807 | 0.003 | 9.330 | 0.004 | 90.15 | 0.03 | 538.2 | 0.4 | 6.265 | 0.003 | 6.807 | 0.003 | 6.310 | 0.004 | HT-Form, cooling, 20 min/step |
| 202 | 8.455 | 0.003 | 6.805 | 0.003 | 9.328 | 0.004 | 90.26 | 0.03 | 536.7 | 0.4 | 6.250 | 0.004 | 6.805 | 0.003 | 6.309 | 0.004 | HT-Form, cooling, 20 min/step |
| 152 | 8.367 | 0.003 | 6.741 | 0.003 | 9.327 | 0.003 | 91.16 | 0.03 | 526.0 | 0.4 | 6.165 | 0.003 | 6.741 | 0.003 | 6.328 | 0.003 | HT-Form, cooling, 20 min/step |
| 101 | 8.310 | 0.003 | 6.704 | 0.003 | 9.313 | 0.003 | 91.79 | 0.03 | 518.6 | 0.3 | 6.104 | 0.003 | 6.704 | 0.003 | 6.337 | 0.003 | HT-Form, cooling, 20 min/step |
| 243 | 8.540 | 0.002 | 6.818 | 0.001 | 9.330 | 0.002 | 89.83 | 0.02 | 543.3 | 0.2 | 6.309 | 0.002 | 6.818 | 0.001 | 6.315 | 0.002 | HT-Form, cooling, 110 min/step |
| 238 | 8.529 | 0.003 | 6.816 | 0.001 | 9.329 | 0.002 | 89.88 | 0.02 | 542.4 | 0.2 | 6.302 | 0.003 | 6.816 | 0.001 | 6.313 | 0.003 | HT-Form, cooling, 110 min/step |
| 233 | 8.517 | 0.005 | 6.814 | 0.003 | 9.329 | 0.005 | 89.93 | 0.05 | 541.5 | 0.5 | 6.294 | 0.005 | 6.814 | 0.003 | 6.312 | 0.005 | HT-Form, cooling, 110 min/step |
| 228 | 8.508 | 0.003 | 6.814 | 0.002 | 9.329 | 0.003 | 89.98 | 0.03 | 540.9 | 0.3 | 6.287 | 0.003 | 6.814 | 0.002 | 6.312 | 0.003 | HT-Form, cooling, 110 min/step |
| 223 | 8.499 | 0.003 | 6.813 | 0.001 | 9.329 | 0.003 | 90.04 | 0.03 | 540.1 | 0.3 | 6.280 | 0.003 | 6.813 | 0.001 | 6.312 | 0.003 | HT-Form, cooling, 110 min/step |
| 218 | 8.488 | 0.014 | 6.816 | 0.004 | 9.327 | 0.009 | 90.08 | 0.11 | 539.6 | 1.1 | 6.273 | 0.012 | 6.816 | 0.004 | 6.310 | 0.012 | HT-Form, cooling, 110 min/step |
| 19 | 8.191 | 0.007 | 6.800 | 0.004 | 9.298 | 0.007 | 91.64 | 0.28 | 517.6 | 2.5 | 6.058 | 0.007 | 6.800 | 0.004 | 6.283 | 0.007 | HT-Form, quenched |
| -123 | 8.083 | 0.001 | 12.583 | 0.002 | 9.764 | 0.001 | 96.15 | 0.01 | 987.4 | 0.3 | 5.888 | 0.001 | 6.663 | 0.001 | 6.291 | 0.001 | Form II, BARBAC02 [7] |
| - | 8.108 | | 12.597 | | 9.877 | | 96.55 | | 1002.1 | | 5.904 | | 6.737 | | 6.298 | | Form II, *ab initio* |
| - | 8.056 | | 6.823 | | 9.264 | | 92.11 | | 508.9 | | 5.967 | | 6.823 | | 6.249 | | HT-form, *Cc* type, *ab initio* |
| - | 8.218 | | 6.870 | | 9.124 | | 93.66 | | 514.1 | | 5.909 | | 6.870 | | 6.332 | | HT-form, $P2_1/c$ type, *ab initio* |
| - | 8.149 | | 7.245 | | 9.272 | | 88.83 | | 547.3 | | 6.183 | | 7.245 | | 6.109 | | HT-form, *C*2/*c* ordered, *ab initio* |