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**Completing the picture of tolazamide polymorphism under extreme conditions: a low-temperature study**

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**Table S1** Crystal data, data collection and refinement parameters for tolazamide form I at different temperatures.

For all structures:  $C_{14}H_{21}N_3O_3S$ ,  $M_r = 311.40$ , triclinic,  $P\bar{1}$ ,  $Z = 2$ . Experiments were carried out at different temperatures with Mo- $K\alpha$  radiation using an Oxford Diffraction Gemini R Ultra X-ray diffractometer, Absorb-7 and Absorb-GUI (Angel *et al.*, 2013). H-atom parameters were constrained.

Temperature	275 K	250 K	225 K	200 K
<i>Crystal data</i>				
<i>a, b, c</i> (Å)	6.3453(3), 9.1929(5), 13.4917(7)	6.3346(3), 9.1641(5), 13.4642(7)	6.3186(5), 9.1502(8), 13.4244(11)	6.3064(5), 9.1298(7), 13.3984(11)
$\alpha, \beta, \gamma$ (°)	79.103(5), 87.266(4), 85.857(5)	79.222(4), 87.305(4), 85.936(4)	79.314(7), 87.303(7), 85.948(7)	79.397(7), 87.344(7), 85.986(6)
<i>V</i> (Å <sup>3</sup> )	770.32(7)	765.43(7)	760.32(11)	755.95(11)
$\mu$ (mm <sup>-1</sup> )	0.22	0.23	0.23	0.23
<b>Crystal size (mm)</b>	$0.40 \times 0.10 \times 0.05$	$0.40 \times 0.10 \times 0.05$	$0.40 \times 0.10 \times 0.05$	$0.40 \times 0.10 \times 0.05$
<i>Data collection</i>				
<i>T</i> <sub>min</sub> , <i>T</i> <sub>max</sub>	0.915, 1.000	0.919, 1.000	0.926, 1.000	0.893, 1.000
<b>No. of measured, independent and observed [<math>I &gt; 2\sigma(I)</math>] reflections</b>	12387, 3149, 2245	12313, 3131, 2355	12225, 3111, 2425	12156, 3090, 2455
<i>R</i> <sub>int</sub>	0.044	0.042	0.041	0.040
(sin θ / λ) <sub>max</sub> (Å <sup>-1</sup> )	0.625	0.625	0.625	0.625
<i>Refinement</i>				
<i>R</i> [ $F^2 > 2 \sigma(F^2)$ ], <i>wR</i> ( $F^2$ ), <i>S</i>	0.048, 0.115, 1.02	0.046, 0.111, 1.05	0.043, 0.100, 1.05	0.041, 0.097, 1.03
<b>No. of reflections</b>	3149	3131	3111	3090
<b>No. of parameters</b>	223	223	223	223
<b>No. of restraints</b>	2	2	2	2
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	0.27, -0.22	0.30, -0.22	0.27, -0.29	0.32, -0.32

Temperature	175 K	150 K	125 K	100 K
<b>Crystal data</b>				
<i>a, b, c</i> (Å)	6.2891(3), 9.0960(4), 13.3659(5)	6.2738(3), 9.0824(4), 13.3414(5)	6.2586(2), 9.0748(4), 13.3249(5)	6.2433(2), 9.0724(4), 13.3119(5)
$\alpha, \beta, \gamma$ (°)	79.436(4), 87.381(3), 86.026(4)	79.449(4), 87.415(3), 86.013(4)	79.432(3), 87.442(3), 85.975(3)	79.359(4), 87.461(3), 85.933(3)
<i>V</i> (Å <sup>3</sup> )	749.41(6)	745.14(6)	741.73(5)	738.80(5)
$\mu$ (mm <sup>-1</sup> )	0.23	0.23	0.23	0.23
Crystal size (mm)	0.40 × 0.10 × 0.05	0.40 × 0.10 × 0.05	0.40 × 0.10 × 0.05	0.40 × 0.10 × 0.05
<b>Data collection</b>				
<i>T</i> <sub>min</sub> , <i>T</i> <sub>max</sub>	0.851, 1.000	0.927, 1.000	0.894, 1.000	0.934, 1.000
No. of measured, independent and observed [ <i>I</i> >2σ( <i>I</i> )] reflections	12090, 3064, 2504	12009, 3045, 2540	11961, 3037, 2585	11890, 3021, 2604
<i>R</i> <sub>int</sub>	0.039	0.038	0.038	0.037
(sin θ / λ) <sub>max</sub> (Å <sup>-1</sup> )	0.625	0.625	0.625	0.625
<b>Refinement</b>				
<i>R</i> [ <i>F</i> <sup>2</sup> >2 σ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.039, 0.095, 1.03	0.038, 0.093, 1.06	0.036, 0.086, 1.03	0.036, 0.086, 1.04
No. of reflections	3064	3045	3037	3021
No. of parameters	223	223	218	213
No. of restraints	2	2	2	0
Δρ <sub>max</sub> , Δρ <sub>min</sub> (e Å <sup>-3</sup> )	0.33, -0.36	0.43, -0.39	0.40, -0.38	0.44, -0.40

Computer programs: *CrysAlis PRO* (Agilent technologies UK Ltd, 2013), *SHELXT* (Sheldrick & IUCr, 2015b), *SHELXL* (Sheldrick & IUCr, 2015a), *Olex2 Version 1.2.9* (Dolomanov *et al.*, 2009), *Mercury 3.10* (Macrae *et al.*, 2008), *PLATON* (Spek, 2003).

**Table S2** Crystal data, data collection and refinement parameters for tolazamide form II at different temperatures.

For all structures:  $C_{14}H_{21}N_3O_3S$ ,  $M_r = 311.40$ , triclinic,  $P\bar{1}$ ,  $Z = 2$ . Experiments were carried out at different temperatures with Mo- $K\alpha$  radiation using an Oxford Diffraction Gemini R Ultra X-ray diffractometer, Absorb-7 and Absorb-GUI (Angel *et al.*, 2013). H-atom parameters were constrained.

Temperature	250 K	200 K	150 K	100 K
<i>Crystal data</i>				
<i>a, b, c</i> (Å)	6.4468(3), 9.0141(5), 13.4850(7)	6.3939(3), 8.9809(4), 13.5213(7)	6.3358(8), 8.9455(11), 13.5482(15)	6.3091(10), 8.9270(15), 13.576(3)
$\alpha, \beta, \gamma$ (°)	82.360(4), 85.368(4), 85.553(4)	82.129(4), 85.602(4), 85.341(4)	81.976(10), 85.919(10), 85.273(10)	81.882(16), 86.073(16), 85.167(13)
<i>V</i> (Å <sup>3</sup> )	772.38(7)	764.89(6)	756.38(15)	753.0(2)
$\mu$ (mm <sup>-1</sup> )	0.22	0.23	0.23	0.23
Crystal size (mm)	0.45 × 0.30 × 0.05	0.45 × 0.30 × 0.05	0.45 × 0.30 × 0.05	0.45 × 0.30 × 0.05
<i>Data collection</i>				
<i>T</i> <sub>min</sub> , <i>T</i> <sub>max</sub>	0.837, 1.000	0.805, 1.000	0.949, 1.000	0.753, 1.000
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	11517, 3154, 2336	11396, 3115, 2445	10897, 3073, 2345	10088, 3057, 2081
<i>R</i> <sub>int</sub>	0.058	0.055	0.068	0.094
(sin $\theta$ / $\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )	0.625	0.625	0.625	0.625
<i>Refinement</i>				
<i>R</i> [ $F^2 > 2 \sigma(F^2)$ ], <i>wR</i> ( $F^2$ ), <i>S</i>	0.048, 0.122, 1.07	0.045, 0.113, 1.06	0.050, 0.127, 1.04	0.069, 0.173, 1.03
No. of reflections	3154	3115	3073	3057
No. of parameters	219	219	209	200
No. of restraints	3	3	3	0
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	0.23, -0.41	0.31, -0.39	0.31, -0.47	0.47, -0.63

Computer programs: *CrysAlis PRO* (Agilent technologies UK Ltd, 2013), *SHELXT* (Sheldrick & IUCr, 2015*b*), *SHELXL* (Sheldrick & IUCr, 2015*a*), *Olex2 Version 1.2.9* (Dolomanov *et al.*, 2009), *Mercury 3.10* (Macrae *et al.*, 2008), *PLATON* (Spek, 2003).

**Table S3** Unit-cell parameters of tolazamide form II at different temperatures.

For all structures:  $C_{14}H_{21}N_3O_3S$ ,  $M_r = 311.40$ , triclinic,  $P\bar{1}$ ,  $Z = 2$ . Experiments were carried out at different temperatures with Mo- $K\alpha$  radiation using an Oxford Diffraction Gemini R Ultra X-ray diffractometer, Absorb-7 and Absorb-GUI (Angel *et al.*, 2013). Data collection and cell determination were performed using the *CrysAlis PRO* (Agilent technologies UK Ltd, 2013).

Temperature	275 K	225 K	175 K	125 K
<i>Crystal data</i>				
<i>a, b, c</i> (Å)	6.4810(14), 9.0464(19), 13.485(3)	6.4311(16), 9.011(2), 13.528(4)	6.3681(5), 8.9660(6), 13.5459(9)	6.3176(8), 8.9325(12), 13.5451(16)
<i>α, β, γ</i> (°)	82.481(18), 85.368(18), 85.778(17)	82.38(2), 85.36(2), 85.51(2)	82.031(6), 85.714(6), 85.257(6)	81.877(10), 85.965(10), 85.199(11)
<i>V</i> (Å <sup>3</sup> )	779.6(3)	772.7(3)	761.76(9)	752.72(17)

**Table S4** Unit-cell parameters, hydrogen-bond distances and distances between the stacked benzene rings in tolazamide form I at 295 K: previously reported data (Boldyreva *et al.*, 2015) and the data obtained in the present study before and after a cooling-heating cycle.

For all structures:  $C_{14}H_{21}N_3O_3S$ ,  $M_r = 311.40$ , triclinic,  $P\bar{1}$ ,  $Z = 2$ . Experiments were carried out with Mo- $K\alpha$  radiation using an Oxford Diffraction Gemini R Ultra X-ray diffractometer, Absorb-7 and Absorb-GUI (Angel *et al.*, 2013).

Reference	[Boldyreva <i>et al.</i> (2015)]	present study (start point)	present study (final point)
$a, b, c$ (Å)	6.3570(4), 9.2186(9), 13.5146(13)	6.3506(6), 9.2241(9), 13.4984(13)	6.3558(3), 9.2184(5), 13.5140(7)
$\alpha, \beta, \gamma$ (°)	78.997(8), 87.202(7), 85.811(7)	79.091(8), 87.215(8), 85.815(8)	78.983(5), 87.259(5), 85.808(5)
$V$ (Å <sup>3</sup> )	774.86(12)	773.87(13)	774.66(8)
$D\cdots A$ distance in N2-H2 $\cdots$ O3 (-x, 1-y, 1-z) hydrogen bond (Å)	2.917(5)	2.912(3)	2.917(3)
Distance between stacked benzene rings (Å)	4.108	4.094(2)	4.099(2)

**Table S5** Unit-cell parameters and hydrogen-bond distances in tolazamide form II at 295 K: previously reported data (Boldyreva *et al.*, 2015) and the data obtained in the present study before and after a cooling-heating cycle.

For all structures:  $C_{14}H_{21}N_3O_3S$ ,  $M_r = 311.40$ , triclinic,  $P\bar{1}$ ,  $Z = 2$ . Experiments were carried out with Mo- $K\alpha$  radiation using an Oxford Diffraction Gemini R Ultra X-ray diffractometer, Absorb-7 and Absorb-GUI (Angel *et al.*, 2013).

Reference	[Boldyreva <i>et al.</i> (2015)]	present study (start point)	present study (final point)
$a, b, c$ (Å)	6.5041(3), 9.0679(6), 13.4841(10)	6.4948(3), 9.0520(4), 13.4605(6)	6.5046(16), 9.067(2), 13.474(4)
$\alpha, \beta, \gamma$ (°)	82.510(6), 85.260(5), 85.839(5)	82.467(4), 85.251(4), 85.836(4)	82.59(2), 85.19(2), 85.90(2)
$V$ (Å <sup>3</sup> )	784.25(9)	780.31(6)	783.7(3)
$D\cdots A$ distance in $N1-H1\cdots O1(I-x, 2-y, I-z)$ hydrogen bond (Å)	3.107(2)	3.105(2)	not defined
$D\cdots A$ distance in $N2-H2\cdots O3 (-x, I-y, I-z)$ hydrogen bond (Å)	3.043(2)	3.036(2)	not defined

**Table S6** Parameters of the Salje thermal equations of state for forms I and II of tolazamide.

Form	I	II
$V_0$ (Å <sup>3</sup> )	733.9(2)	749.0(4)
$p_1 \cdot 10^5$ (Å/K)	89.3(9)	87.0(17)
$\theta_{\text{sat}}$ (K)	132.6(17)	179(3)
$\alpha_{(295 \text{ K}, 1 \text{ atm})} \cdot 10^4$ (K <sup>-1</sup> )	2.79(11)	2.5(2)

**Table S7** The values of linear coefficients of thermal expansion along the principal axes of the strain ellipsoids ( $\alpha_l = (1/l_0)(\partial l/\partial T)$ ) and angles between the principal axes and unit-cell directions in forms I and II. The strain ellipsoids were calculated between the structures corresponding to 295 and 100 K. The results are obtained using Win\_Strain (Angel, 2011) in an infinitesimal lagrangian approximation.

Form	Mode	Axis	$\alpha_l$ (MK <sup>-1</sup> )	Angle to unit-cell direction		
				<i>a</i>	<i>b</i>	<i>c</i>
I	cooling	1	96	45.2(14)	50.7(12)	57.4(5)
		2	80	48.9(15)	134.5(14)	99.7(12)
		3	60	74.6(9)	70.9(11)	145.7(5)
	heating	1	102	134.2(9)	126.5(6)	125.9(4)
		2	84	135.1(10)	55.0(8)	66.2(6)
		3	59	96.3(6)	124.3(6)	45.4(5)
II	cooling	1	168	25.7(4)	108.6(3)	77.2(2)
		2	66	114.7(4)	157.4(5)	89.6(4)
		3	-55	83.2(3)	102.3(4)	167.1(1)
	heating	1	186	28.8(7)	110.1(5)	76.3(3)
		2	75	117.5(7)	154.9(4)	88.9(6)
		3	-52	97.7(3)	75.7(5)	13.8(6)

**Table S8** The values of linear strain along the directions of the principal axes of the strain ellipsoids in forms I and II in relation to the structures at 295 K on cooling and at 100 K on re-heating. Angles between the principal axes and unit-cell directions are given in Table S7. The results are obtained using WinStrain (Angel, 2011) in an infinitesimal lagrangian approximation.

Form	Mode	Temperature (K)	$\Delta l/l (\%)$		
			1	2	3
I	cooling	295	0	0	0
		250	-0.684(11)	-0.243(12)	-0.167(11)
		200	-1.178(11)	-0.629(13)	-0.526(14)
		150	-1.723(10)	-1.125(11)	-0.911(11)
		100	-1.881(9)	-1.558(11)	-1.162(11)
	heating	100	0	0	0
		125	0.262(5)	0.135(6)	-0.001(6)
		175	0.778(6)	0.410(6)	0.242(6)
		225	1.251(9)	0.872(10)	0.763(10)
		275	1.736(6)	1.415(7)	1.056(8)
		295	1.987(6)	1.638(7)	1.152(8)
II	cooling	295	0	0	0
		250	-0.911(7)	-0.310(7)	0.204(7)
		200	-1.857(7)	-0.662(7)	0.540(7)
		150	-2.795(13)	-1.106(13)	0.828(12)
		100	-3.285(17)	-1.291(17)	1.07(2)
	heating	100	0	0	0
		125	0.17(2)	0.05(2)	-0.26(3)
		175	1.039(19)	0.452(19)	-0.33(2)
		225	2.26(3)	0.95(3)	-0.61(4)
		275	3.13(3)	1.27(3)	-0.87(3)
		295	3.62(3)	1.45(3)	-1.01(4)

**Table S9** Angles between the principal axes of the strain ellipsoid and directions corresponding to stacking interactions and hydrogen bonds in form I (calculated as angles between the principal axes and vectors formed by centroids of the stacked benzene rings or by donor and acceptor atoms of hydrogen bonds).

<b>Mode</b>	<b>Axis</b>	<b>Angle between the principal axis and the particular direction (°)</b>	
		<b>Direction corresponding to stacking interactions</b>	<b>Direction of hydrogen bond</b>
<b>cooling</b>	<b>1</b>	12.1	62.0
	<b>2</b>	82.8	25.4
	<b>3</b>	83.2	76.7
<b>heating</b>	<b>1</b>	10.4	62.3
	<b>2</b>	85.1	24.7
	<b>3</b>	85.8	84.2
<b>hydrostatic compression*</b>	<b>1</b>	64.4	46.2
	<b>2</b>	34.2	67.6
	<b>3</b>	59.0	72.6

\*the angles are calculated using the data published by Fedorov *et al.* (2017).

**Table S10** Angles between the principal axes of the strain ellipsoid and the directions of hydrogen bonds in form II (calculated as angles between the principal axes and vectors formed by donor and acceptor atoms of hydrogen bonds).

Mode	Axis	Angle between the principal axis and the direction of particular hydrogen bond (°)	
		Hydrogen bond N1-H1…O1 (-x+1, -y+2, -z+1)	Hydrogen bond N2-H2…O3 (-x, -y+1, -z+1)
cooling	1	30.1	69.8
	2	55.3	17.1
	3	82.8	88.6
heating	1	32.0	71.8
	2	52.9	14.3
	3	83.4	87.7
hydrostatic compression*	1	61.4	80.7
	2	33.3	8.4
	3	69.4	63.8

\*the angles are calculated using the data published by Fedorov *et al.* (2017).

**Table S11** Geometrical parameters of stacking interactions between two benzene rings and N2-H2···O3 (-x, 1-y, 1-z) hydrogen bond in form I at different temperatures.

T (K)	Distance between benzene rings (Å)	Interplanar distance between the planes containing benzene rings (Å)	Hydrogen bond N2-H2···O3 (-x, 1-y, 1-z)			
			D–H (Å)	H···A (Å)	D···A (Å)	D–H ··· A (°)
275	4.082(2)	3.809	0.84(3)	2.08(3)	2.911(3)	172(2)
250	4.063(2)	3.794	0.83(2)	2.08(2)	2.909(2)	175(2)
225	4.045(2)	3.779	0.82(2)	2.09(2)	2.906(2)	174(2)
200	4.029(2)	3.768	0.82(2)	2.10(2)	2.905(2)	172(2)
175	4.011(2)	3.755	0.84(2)	2.06(2)	2.899(2)	174(2)
150	4.000(1)	3.750	0.83(2)	2.07(2)	2.896(2)	173(2)
125	3.990(1)	3.743	0.85(2)	2.05(2)	2.896(2)	173(2)
100	3.985(1)	3.746	0.83(2)	2.07(2)	2.898(2)	173(2)

**Table S12** Geometrical parameters of hydrogen bonds in form II at different temperatures.

T (K)	Hydrogen bond N1-H1···O1 (-x+1, -y+2, -z+1)				Hydrogen bond N2-H2···O3 (-x, -y+1, -z+1)			
	D–H (Å)	H···A (Å)	D···A (Å)	D–H ··· A (°)	D–H (Å)	H···A (Å)	D···A (Å)	D–H ··· A (°)
250	0.75(2)	2.48(2)	3.083(2)	139(2)	0.80(3)	2.22(3)	3.025(2)	174(3)
200	0.74(2)	2.45(2)	3.048(2)	140(2)	0.84(2)	2.17(3)	3.011(2)	173(2)
150	0.76(3)	2.41(3)	3.013(3)	137(3)	0.82(3)	2.18(3)	2.995(3)	172(3)
100	0.78(4)	2.37(4)	2.991(3)	137(4)	0.85(4)	2.15(4)	2.985(3)	168(4)

**Table S13** Root-mean-square deviations (RMSD) of the positions of non-hydrogen atoms between the structures of form I/II at 295 K and at other temperature points (as implemented in Mercury 3.10) (Macrae *et al.*, 2008).

Form I		Form II	
Temperature (K)	RMSD (Å)	Temperature (K)	RMSD (Å)
275	0.0070	250	0.0160
250	0.0128	200	0.0360
225	0.0185	150	0.0552
200	0.0235	100	0.0650
175	0.0293		
150	0.0348		
125	0.0397		
100	0.0453		

**Table S14** Site occupancy factors for two possible conformations of azepane ring in tolazamide polymorphs.

T (K)	Form I		Form II	
	Conformation A	Conformation B	Conformation A	Conformation B
275	0.823(6)	0.177(6)		
250	0.835(6)	0.165(6)	0.569(7)	0.431(7)
225	0.838(5)	0.162(5)		
200	0.854(5)	0.146(5)	0.725(7)	0.275(7)
175	0.869(5)	0.131(5)		
150	0.894(5)	0.106(5)	0.904(7)	0.096(7)
125	0.927(5)	0.073(5)		
100	1	0	1	0

**Table S15** The relative energy differences between tolazamide molecules with two possible conformations (*A* and *B*) of the azepane ring extracted from the corresponding crystal structures of forms I and II determined at 295 and 150 K. The root-mean-square deviations (RMSD) of the positions of different atoms indicating small geometry changes during the optimization process are shown in Tables S16 and S17.

Temperature at which the initial geometry was determined	$\Delta E_{\text{conf}} = E(A) - E(B)$ (kJ/mol)	
	Form I	Form II
295 K	1.2	-2.3
150 K	1.5	-1.9

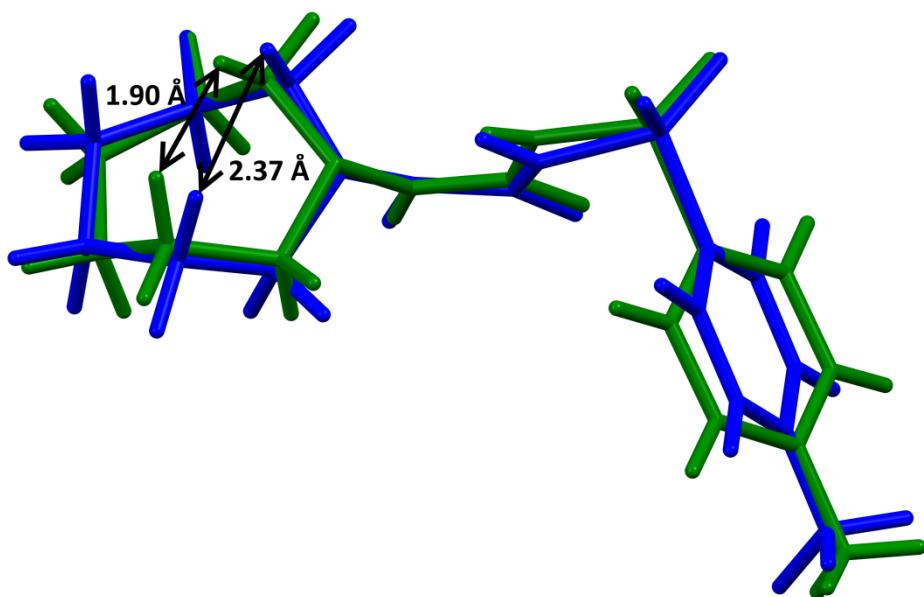
**Table S16** The root-mean-square deviations (RMSD) of the positions of different atoms (as implemented in ChemCraft v.1.8 (<https://www.chemcraftprog.com>)) in the different structures of form I.

RMSD calculated for the positions of ...	RMSD (Å) between the experimental and calculated structures of conformations ...			
	<i>A</i> at 150 K	<i>B</i> at 150 K	<i>A</i> at 295 K	<i>B</i> at 295 K
all atoms	0.352	0.507	0.578	0.585
non-hydrogen atoms	0.230	0.347	0.422	0.415
atoms of azepane fragment	0.170	0.280	0.224	0.213
disordered C and H atoms	0.129	0.135	0.159	0.167
all atoms except the disordered C and H atoms	0.373	0.468	0.554	0.538
non-hydrogen atoms except the disordered C and H atoms	0.232	0.354	0.412	0.423

**Table S17** The root-mean-square deviations (RMSD) of the positions of different atoms (as implemented in ChemCraft v.1.8 (<https://www.chemcraftprog.com>)) in the different structures of form II.

RMSD calculated for the positions of ...	RMSD (Å) between the structures of conformations ...			
	A at 295 and 150 K defined experimentally	A at 295 and 150 K defined computationally	B at 295 and 150 K defined experimentally	B at 295 and 150 K defined computationally
all atoms	0.593	0.770	0.685	0.794
non-hydrogen atoms	0.035	0.325	0.038	0.174
atoms of azepane fragment	0.838	0.982	1.017	1.138
disordered C and H atoms	0.835	0.881	1.106	1.328
all atoms except the disordered C and H atoms	0.531	0.675	0.531	0.619
non-hydrogen atoms except the disordered C and H atoms	0.035	0.313	0.035	0.170

**Figure S1** Overlay of the initial (in green) and the optimized (in blue) molecular conformations as an illustration of modifying unfavourable contacts during the process of gas-phase optimization. Particularly, increasing the distance between H10C and H14A atoms is shown quantitatively in the figure. The initial molecular geometry was extracted from the crystal structure of tolazamide form I determined at 150 K. The optimized geometry was obtained after gas-phase optimization of the initial molecular geometry. The molecular overlay was performed as implemented in Mercury 3.10 (Macrae *et al.*, 2008).



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