Electronic Supplementary Material (ESI) for CrystEngComm.
This journal is © The Royal Society of Chemistry 2020

Supplementary Information

Polymorphism of 1,3-X-adamantanes (X=Br, OH, CH_3) and the crystal plastic phase formation ability

Philippe Negrier,[a] Bacem Ben Hassine,[a,b] Maria Barrio[b], Michela Romanini[b], Denise Mondieig[a] and Josep-Lluis Tamarit[b,*]

[a] Université de de Bordeaux, LOMA, UMR 5798, F-33400 Talence, France, CNRS, LOMA, UMR 5798, F-33400 Talence, France.
[b] Grup de Caracterització de Materials, Departament de Física and Barcelona Research Center in Multiscale Science and Engineering, Universitat Politècnica de Catalunya,EEBE, Campus Diagonal-Besos, Av. Eduard Maristany 10-14, 08019 Barcelona, Catalonia

Table S1. Lattice parameters as a function of temperature for the monoclinic (*$P2_1/c$*) phase II of 1,3-dimethyladamantane.

T (K)	a (Å)	b (Å)	c (Å)	α (°)	β (°)	γ (°)	V (Å^3)
90	7.7679(13)	12.0782(24)	11.7020(16)	90	112.217(8)	90	1016.4(3)
110	7.7752(15)	12.0974(20)	11.7214(15)	90	112.251(9)	90	1020.4(3)
130	7.7843(17)	12.1212(20)	11.7431(15)	90	112.293(9)	90	1025.2(3)
150	7.7886(16)	12.1382(21)	11.7644(15)	90	112.322(10)	90	1028.9(3)
170	7.7967(17)	12.1607(23)	11.7885(16)	90	112.359(11)	90	1033.7(3)
180	7.7983(14)	12.1688(24)	11.7985(18)	90	112.371(8)	90	1035.4(3)
190	7.7994(17)	12.1792(23)	11.8099(17)	90	112.386(10)	90	1037.3(3)
200	7.7969(17)	12.1852(24)	11.8191(17)	90	112.403(10)	90	1038.2(3)
210	7.7967(16)	12.1965(23)	11.8332(19)	90	112.416(10)	90	1040.2(3)
215	7.7976(15)	12.2022(23)	11.8404(18)	90	112.425(10)	90	1041.4(3)

Table S2. Lattice parameters as a function of temperature for the hexagonal (*$P6_3/mmc$*) phase I of 1,3-dimethyladamantane. (*) metastable lattice parameters obtained from cooling phase I.

T (K)	a (Å)	b (Å)	c (Å)	α (°)	β (°)	γ (°)	V (Å^3)
160*	24.942(13)	24.942(13)	12.983(7)	90	90	120	6995(6)
170*	24.998(12)	24.998(12)	13.005(6)	90	90	120	7038(6)
180*	25.056(13)	25.056(13)	13.026(7)	90	90	120	7082(6)
190*	25.096(12)	25.096(12)	13.036(6)	90	90	120	7110(6)
200*	25.139(12)	25.139(12)	13.050(6)	90	90	120	7142(6)
210*	25.205(14)	25.205(14)	13.078(7)	90	90	120	7195(7)
220	25.281(12)	25.281(12)	13.108(6)	90	90	120	7255(6)
230	25.338(11)	25.338(11)	13.131(5)	90	90	120	7301(5)
240	25.438(10)	25.438(10)	13.183(5)	90	90	120	7388(5)

Table S3. Lattice parameters as a function of temperature for the orthorhombic (*Pnma)* phase I of 1,3-dibromoadamantane.

T (K)	a (Å)	b (Å)	c (Å)	α (°)	β (°)	γ (°)	V (Å^3)
90	14.7526(5)	7.2611(3)	9.6498(3)	90	90	90	1033.69(6)
120	14.7813(5)	7.2628(3)	9.6644(3)	90	90	90	1037.51(6)
140	14.8017(5)	7.2638(3)	9.6745(3)	90	90	90	1040.17(6)
160	14.8226(6)	7.2653(3)	9.6848(4)	90	90	90	1042.96(7)
170	14.8333(6)	7.2662(3)	9.6907(4)	90	90	90	1044.48(7)
180	14.8447(6)	7.2666(4)	9.6960(4)	90	90	90	1045.91(8)
190	14.8559(6)	7.2675(3)	9.7021(3)	90	90	90	1047.49(7)
200	14.8678(6)	7.2684(3)	9.7082(4)	90	90	90	1049.12(7)
210	14.8794(6)	7.2694(3)	9.7143(4)	90	90	90	1050.74(7)
230	14.9037(7)	7.2711(3)	9.7268(4)	90	90	90	1054.06(8)
260	14.9401(7)	7.2742(4)	9.7470(4)	90	90	90	1059.28(9)
300	14.9898(8)	7.2781(4)	9.7760(5)	90	90	90	1066.54(10)
340	15.0459(8)	7.2824(4)	9.8096(5)	90	90	90	1074.84(10)
375	15.0947(8)	7.2856(5)	9.8404(5)	90	90	90	1082.19(11)

Table S4. Lattice parameters as a function of temperature for the hexagonal (*P6$_3$/mcm*) phase II of 1,3-adamantanediol.

T (K)	a (Å)	b (Å)	c (Å)	α (°)	β (°)	γ (°)	V (Å^3)
90	11.8476(4)	11.8476(4)	10.8396(5)	90	90	120	1317.66(9)
110	11.8520(4)	11.8520(4)	10.8486(5)	90	90	120	1319.74(9)
130	11.8562(4)	11.8562(4)	10.8576(5)	90	90	120	1321.77(9)
150	11.8606(4)	11.8606(4)	10.8666(5)	90	90	120	1323.85(9)
170	11.8654(4)	11.8654(4)	10.8766(5)	90	90	120	1326.14(9)
190	11.8707(4)	11.8707(4)	10.8871(5)	90	90	120	1328.60(9)
210	11.8758(5)	11.8758(5)	10.8977(5)	90	90	120	1331.04(10)
230	11.8812(5)	11.8812(5)	10.9087(5)	90	90	120	1333.60(10)
250	11.8877(5)	11.8877(5)	10.9227(6)	90	90	120	1336.77(11)
270	11.8940(5)	11.8940(5)	10.9353(6)	90	90	120	1339.73(11)
290	11.8998(5)	11.8998(5)	10.9481(6)	90	90	120	1342.61(11)
310	11.9062(5)	11.9062(5)	10.9607(6)	90	90	120	1345.60(11)
330	11.9124(5)	11.9124(5)	10.9741(6)	90	90	120	1348.65(11)
350	11.9186(5)	11.9186(5)	10.9880(6)	90	90	120	1351.76(11)
370	11.9245(5)	11.9245(5)	11.0010(6)	90	90	120	1354.70(11)
297	11.9016(4)	11.9016(4)	10.9509(4)	90	90	120	1343.36(8)
320	11.9105(4)	11.9105(4)	10.9669(5)	90	90	120	1347.33(9)
387.2	11.9346(4)	11.9346(4)	11.0167(5)	90	90	120	1358.93(9)
410	11.9447(4)	11.9447(4)	11.0372(5)	90	90	120	1363.77(9)
424.3	11.9537(4)	11.9537(4)	11.0531(5)	90	90	120	1367.79(9)
440	11.9590(5)	11.9590(5)	11.0655(5)	90	90	120	1370.54(10)
445	11.9597(6)	11.9597(6)	11.0682(5)	90	90	120	1371.03(12)
449.3	11.9618(5)	11.9618(5)	11.0704(6)	90	90	120	1371.79(11)

Table S5. Lattice parameters as a function of temperature for the high-temperature cubic ($Fm\bar{3}m$) phase I of 1,3-adamantanediol.

T (K)	a (Å)	b (Å)	c (Å)	α (°)	β (°)	γ (°)	V (Å^3)
456.7	9.9616(9)	9.9616(9)	9.9616(9)	90	90	90	988.52(15)
461.3	9.9675(15)	9.9675(15)	9.9675(15)	90	90	90	990.3(3)
470.6	9.9781(17)	9.9781(17)	9.9781(17)	90	90	90	993.4(3)
479.8	9.9891(17)	9.9891(17)	9.9891(17)	90	90	90	996.7(3)
486.3	9.9962(17)	9.9962(17)	9.9962(17)	90	90	90	998.9(3)

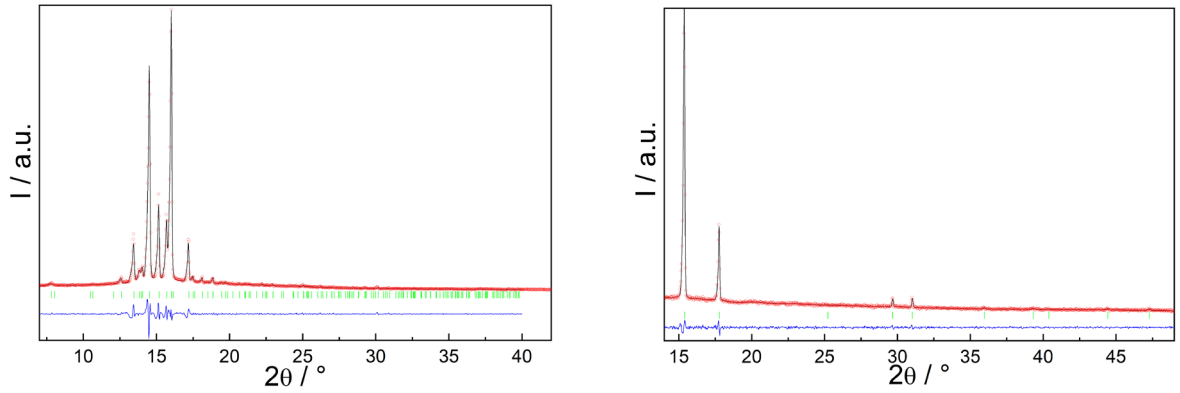

Figure S1. Experimental (red circles) diffraction pattern and calculated (black line) diffraction pattern by Pawley fitting procedure along with difference profile (blue line) and Bragg reflections (vertical sticks) of the hexagonal ($P6_3/mmc$) phase I of 1,3-dimethyladamantane (13DMA) at 220 K (left panel), R_{wp}=6.07 and R_p=4.37, and of the cubic ($Fm\bar{3}m$) phase of 1,3-adamantanediol (13DOHA) at 479.8 K (right panel), R_{wp}=7.08 and R_p=5.55

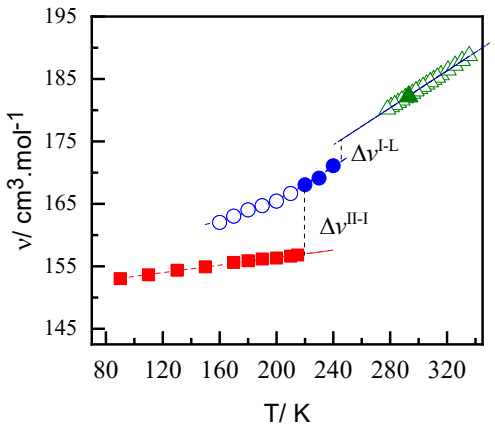

Figure S2. Molar volume for the monoclinic (II), hexagonal (I) and liquid (*L*) phases of 1,3-dimethyladamantane as a function of temperature. Stable phase II, full red squares, stable phase I, full blue circles. Metastable phase I, open circles. Liquid phase densitometry

measurements, open green triangles (△). Full green triangle corresponds to the liquid density according to ref. [1], the value of density at T=293 K, D=0.9016 g/cm^3.

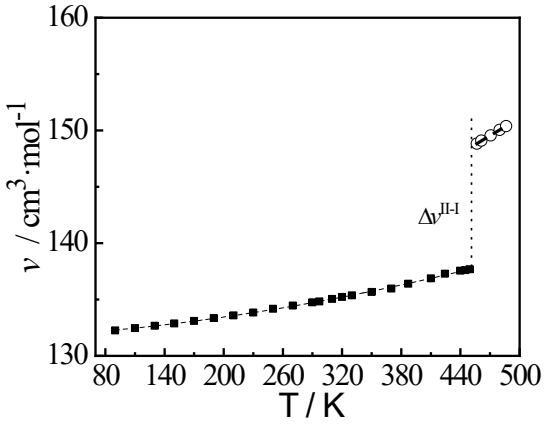

Figure S3. Molar volume for the hexagonal (II, full squares) and face-centered cubic (I, open circles) phases of 1,3-adamantanediol as a function of temperature.

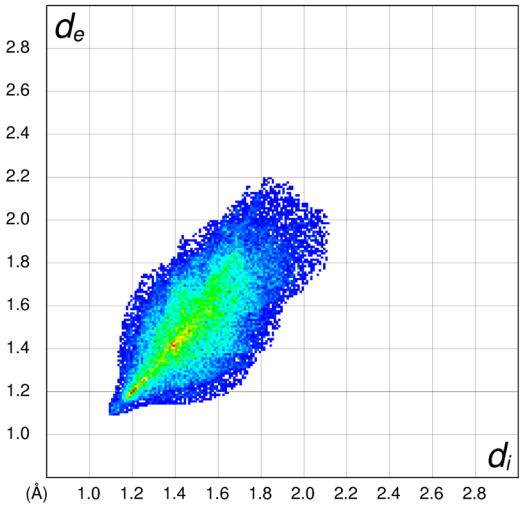

Figure S4. Fingerprint plot of 1,3-dimethyladamantane H….H contacts at 120 K. Blue points correspond to the low frequency of occurrence, green points to more frequency and red points correspond to the high frequency of the surface points.

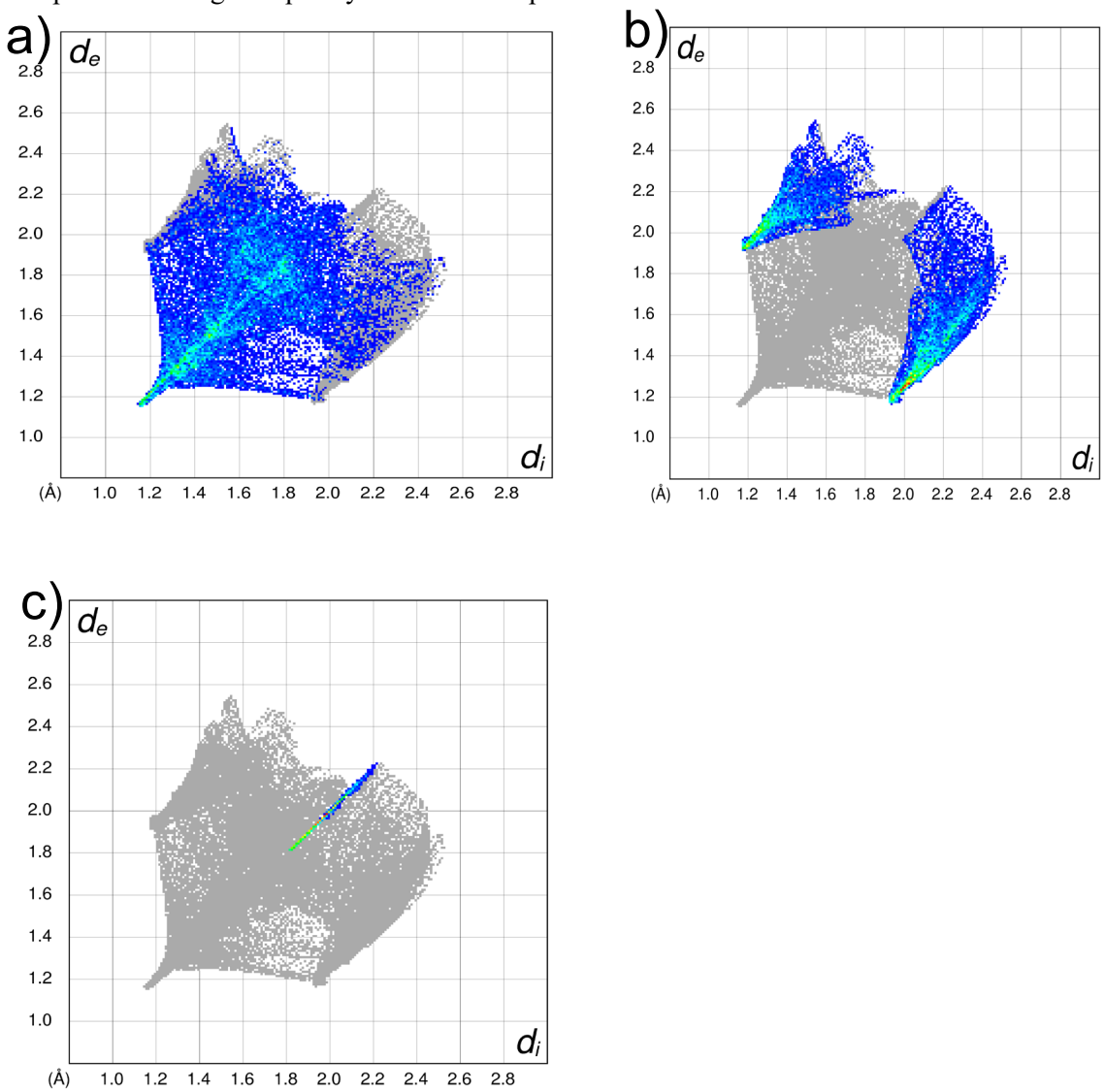

Figure S5. Fingerprint plots of 1,3-dibromoadamantane at 294 K: a) H….H, b) Br…H and c) Br….Br contacts. Blue points correspond to the low frequency of occurrence, green points to more frequency and red points correspond to the high frequency of the surface points.

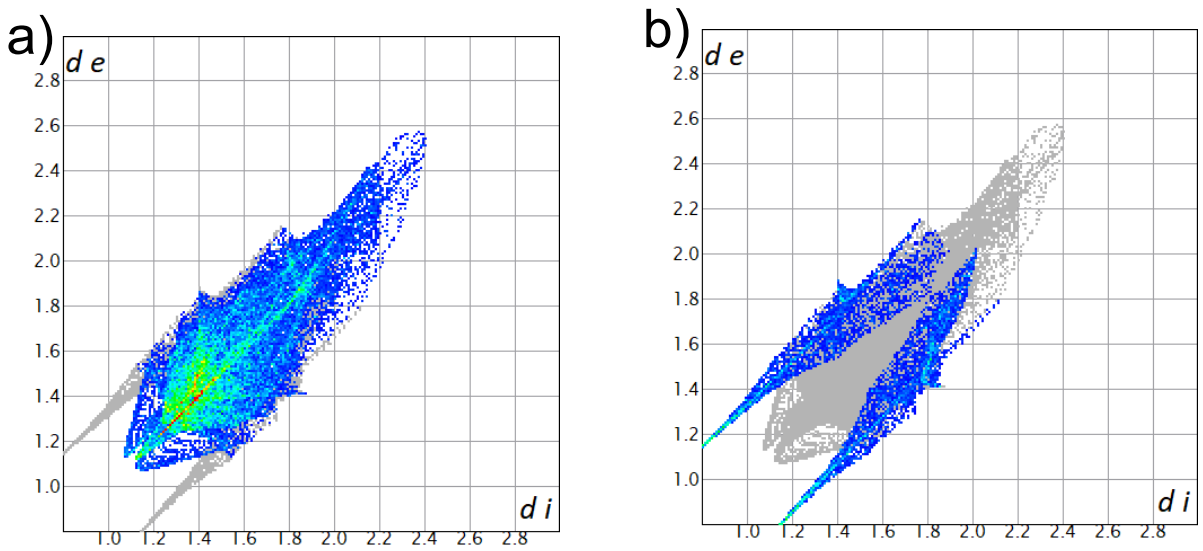

Figure S6. Fingerprint plots of 1,3-adamantanediol at 294 K: a) H….H and b) O…H contacts. Blue points correspond to the low frequency of occurrence, green points to more frequency and red points correspond to the high frequency of the surface points.

References

[1] R.M. Varushchenko, L.L. Pashchenko, A.I. Druzhinina, A.V. Abramenkov, A.A. Pimersin, *J. Chem. Thermodyn.,* 2001, **7**, 733-744.