Electronic Supplementary Material (ESI) for RSC Advances.

Structural features evolution – from fluids to solid phase – and crystal morphology study of LASSBio 1601: a cyclohexyl-*N*-acylhydrazone derivative

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Supporting Information

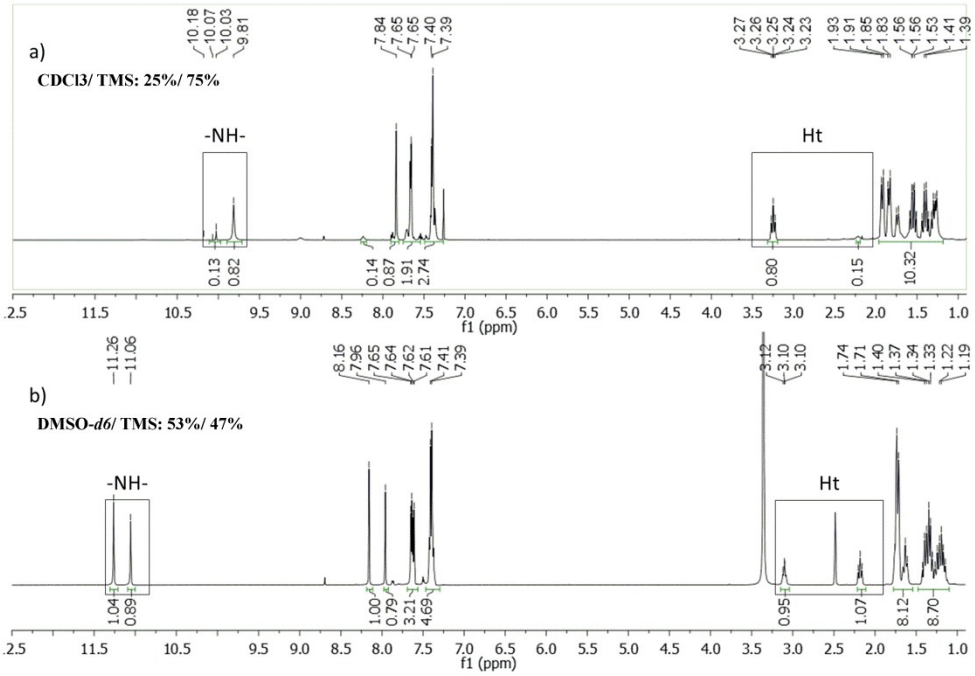

Figure S1. Change in signal integration at ^{1}H-NMR (200 MHz) spectra of LASSBio-1601 solvent induced , using (a) $CDCl_3$/TMS and (b) DMSO-d_6/TMS. The hydrogen attached to the tertiary carbon of the cyclohexyl subunit (Ht) and hydrogen attached to nitrogen amide (-NH-) are highlighted.

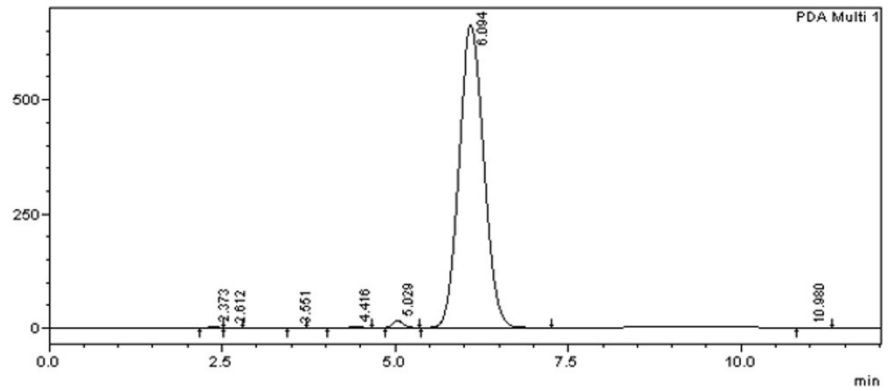

Figure S2. LASSBio-1601 chromatogram by internal normalization with 98,90% of area. Kromasil column 100-5-C18 (4.6 mm x 250 mm); detector SPD-M20A (Diode Array); 254 nm; 1 mL min^{-1}; injection of 20 µL; 70/30 ACN/H_2O.

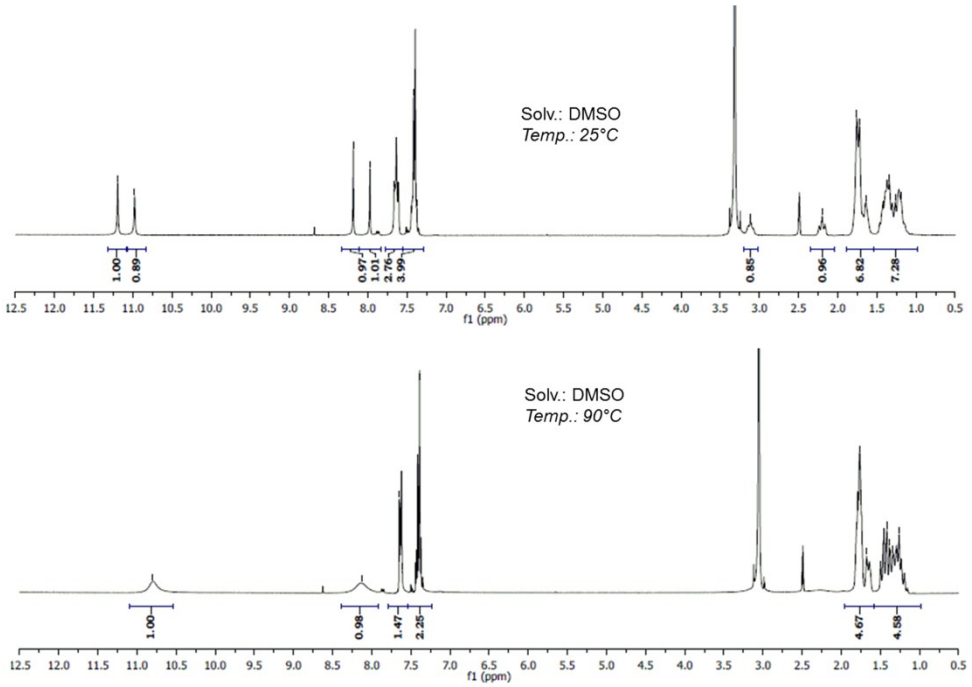

Figure S3. LASSBio-1601 ^{1}H-NMR spectra at 25 °C and 90 °C (300 MHz, DMSO-d_6/TMS) showing the coalescence of signals that were previously duplicated.

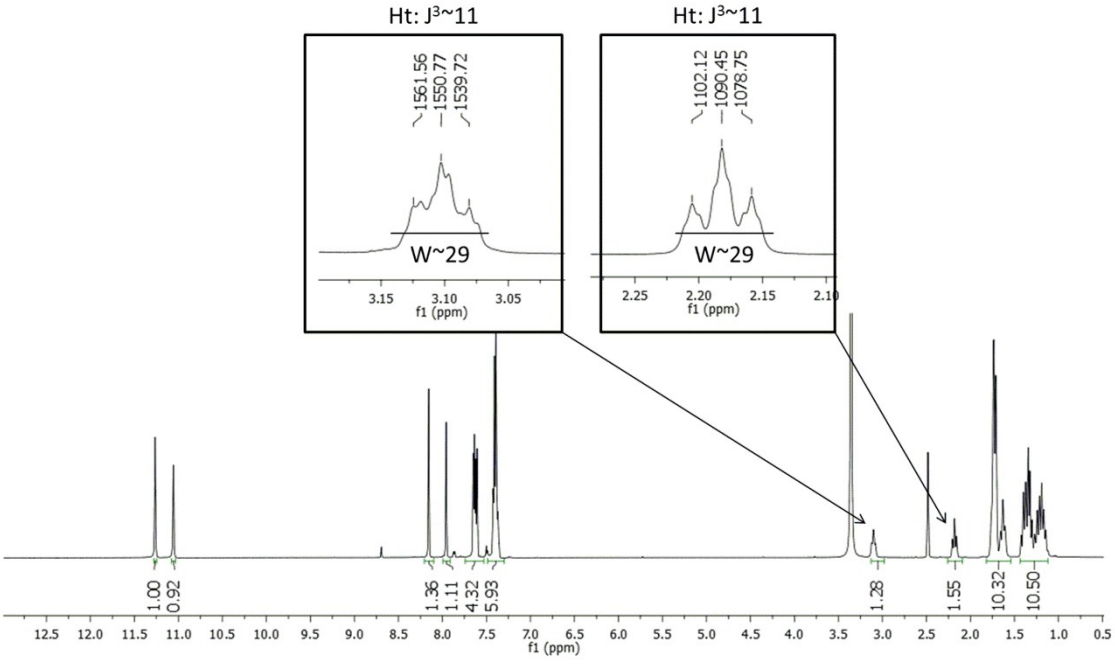

Figure S4. ^{1}H-NMR Spectra of the LASSBio-1601 compound including the H-H couplings (500 MHz, DMSO-d6/TMS).

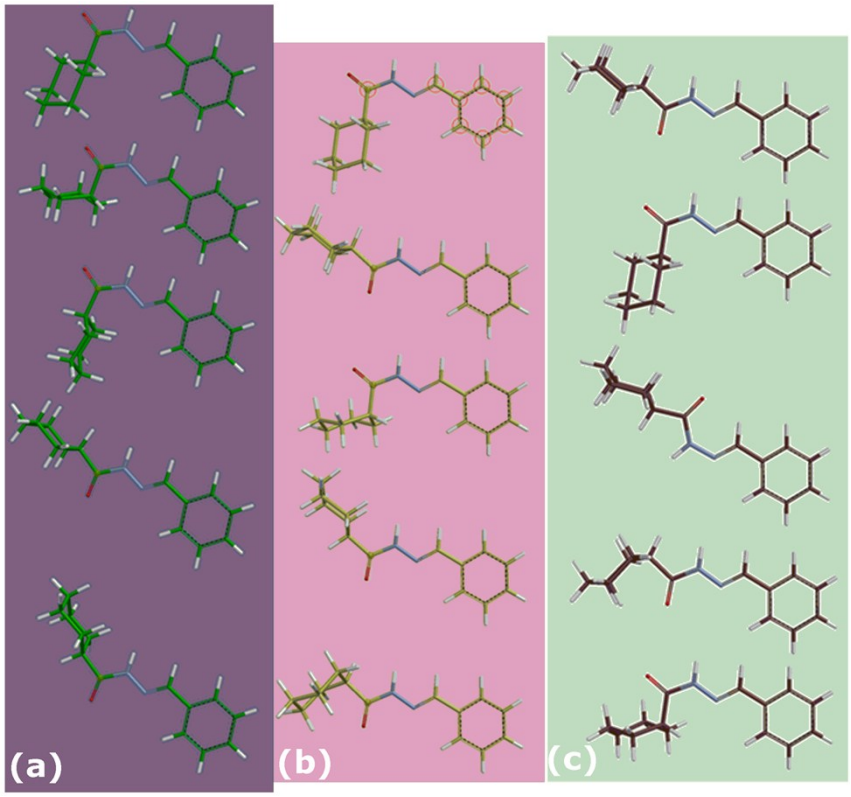

Figure S5. The five more stable conformers of LASSBio-1601 compound (i*n Silico*) in different solvents – (a) vacuum, (b) DMSO and (c) water.

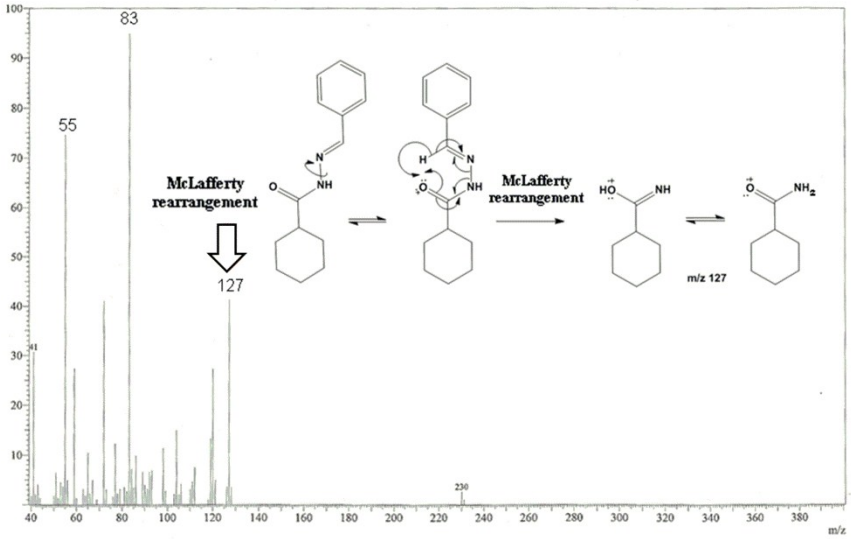

Figure S6. Mass spectrum of LASSBio-1601 showing a McLafferty rearrangement exclusive of relative configuration *E*.

Table S1. Final coordinates and equivalent isotropic displacement parameters (U_{iso} = $B_{iso}/8\pi^2$) for all atoms in the LASSBio-1601 crystal structure.

Atom	*x*	*y*	*z*	U_{iso} (Å^2)
C(1)	0.780(5)	0.547(4)	0.357(2)	0.03812
C(2)	0.605(5)	0.591(3)	0.2555(18)	0.03812
O(3)	0.943(3)	0.444(2)	0.4055(12)	0.03812
N(4)	0.724(3)	0.617(2)	0.4004(18)	0.03812
C(5)	0.741(4)	0.745(3)	0.1603(15)	0.03812
C(6)	0.595(5)	0.458(2)	0.2466(18)	0.03812
N(8)	0.527(3)	0.724(2)	0.3512(16)	0.03812
C(10)	0.592(4)	0.788(3)	0.0529(16)	0.03812
C(13)	0.449(4)	0.503(3)	0.1392(15)	0.03812
C(16)	0.501(4)	0.785(3)	0.397(2)	0.03812
C(17)	0.585(4)	0.654(2)	0.0450(15)	0.03812
C(22)	0.305(5)	0.907(4)	0.350(2)	0.03812
C(26)	0.185(4)	0.977(3)	0.2462(19)	0.03812
C(27)	0.270(4)	0.964(3)	0.4053(16)	0.03812
C(28)	-0.001(4)	1.092(3)	0.2009(18)	0.03812
C(30)	0.078(4)	1.075(3)	0.3632(18)	0.03812
C(32)	-0.057(4)	1.140(3)	0.2604(19)	0.03812
H(7)	0.422(5)	-0.389(3)	-0.7468(18)	0.04574
H(9)	0.802(3)	-0.420(2)	-0.5338(18)	0.04574
H(11)	0.929(4)	-0.261(3)	-0.8348(15)	0.04574
H(12)	0.693(4)	-0.176(3)	-0.8370(15)	0.04574
H(14)	0.470(5)	-0.628(2)	-0.6976(18)	0.04574
H(15)	0.769(5)	-0.574(2)	-0.7439(18)	0.04574
H(18)	0.728(4)	-0.134(3)	-0.9995(16)	0.04574

H(19)	0.421(4)	-0.182(3)	-0.9607(16)	0.04574
H(20)	0.245(4)	-0.489(3)	-0.8656(15)	0.04574
H(21)	0.457(4)	-0.586(3)	-0.8638(15)	0.04574
H(23)	0.601(4)	-0.244(3)	-0.535(2)	0.04574
H(24)	0.764(4)	-0.375(2)	-0.9536(15)	0.04574
H(25)	0.467(4)	-0.327(2)	-1.0170(15)	0.04574
H(29)	0.232(4)	-0.068(3)	-0.7888(19)	0.04574
H(31)	0.338(4)	-0.073(3)	-0.5233(16)	0.04574
H(33)	-0.077(4)	0.129(3)	-0.8716(18)	0.04574
H(34)	0.028(4)	0.124(3)	-0.6075(18)	0.04574
H(35)	-0.178(4)	0.224(3)	-0.7806(19)	0.04574

Table S2. Some selected bond distances for LASSBio-1601.

Bonds	Lengths (Å)
C(1)-C(2)	1.50(4)
C(1)-O(3)	1.22(4)
C(1)-N(4)	1.37(6)
C(2)-C(5)	1.54(3)
C(2)-C(6)	1.52(5)
N(4)-N(8)	1.37(2)
C(5)-C(10)	1.53(3)
C(6)-C(13)	1.52(3)
N(8)-C(16)	1.27(5)
C(10)-C(17)	1.53(5)
C(13)-C(17)	1.52(2)
C(16)-C(22)	1.47(4)
C(22)-C(26)	1.38(4)
C(22)-C(27)	1.38(6)

C(26)-C(28)	1.39(4)
C(27)-C(30)	1.39(4)
C(28)-C(32)	1.40(5)
C(30)-C(32)	1.40(3)

Table S3. Some selected bond angles for LASSBio-1601.

Bonds	Angles (°)
C(2)-C(1)-O(3)	123(3)
C(2)-C(1)-N(4)	116(3)
O(3)-C(1)-N(4)	120(3)
C(1)-C(2)-C(5)	109(2)
C(1)-C(2)-C(6)	108(2)
C(5)-C(2)-C(6)	112(2)
C(1)-N(4)-N(8)	121(2)
C(2)-C(5)-C(10)	111(2)
C(2)-C(6)-C(13)	111(2)
N(4)-N(8)-C(16)	115(2)
C(5)-C(10)-C(17)	111(2)
C(6)-C(13)-C(17)	112(2)
N(8)-C(16)-C(22)	121(3)
C(10)-C(17)-C(13)	111(2)
C(16)-C(22)-C(26)	120(3)
C(16)-C(22)-C(27)	119(3)
C(26)-C(22)-C(27)	120(3)
C(22)-C(26)-C(28)	121(3)
C(22)-C(27)-C(30)	120(3)
C(26)-C(28)-C(32)	119(2)

C(27)-C(30)-C(32)	120(2)
C(28)-C(32)-C(30)	120(2)