SUPPORTING INFORMATION FOR

From mannose to small amphiphilic polyol – perfect linearity leads to spontaneous aggregation

Tiina Saloranta,[†] *Anssi Peuronen,*[‡] *Johannes Dieterich,*[§] *Janne Ruokolainen,*[ǀ] *Manu Lahtinen,*[‡] *Reko Leino**[†]

[†] Johan Gadolin Process Chemistry Centre, c/o Laboratory of Organic Chemistry, Åbo Akademi University, Turku, Finland

[‡] Department of Chemistry, University of Jyväskylä, Jyväskylä, Finland

[§] Institut für Physikalische Chemie, Universität Göttingen, Göttingen, Germany

[ǀ] Department of Applied Physics, Nanomicroscopy Center (Aalto-NMC), Aalto University School of Science, Finland

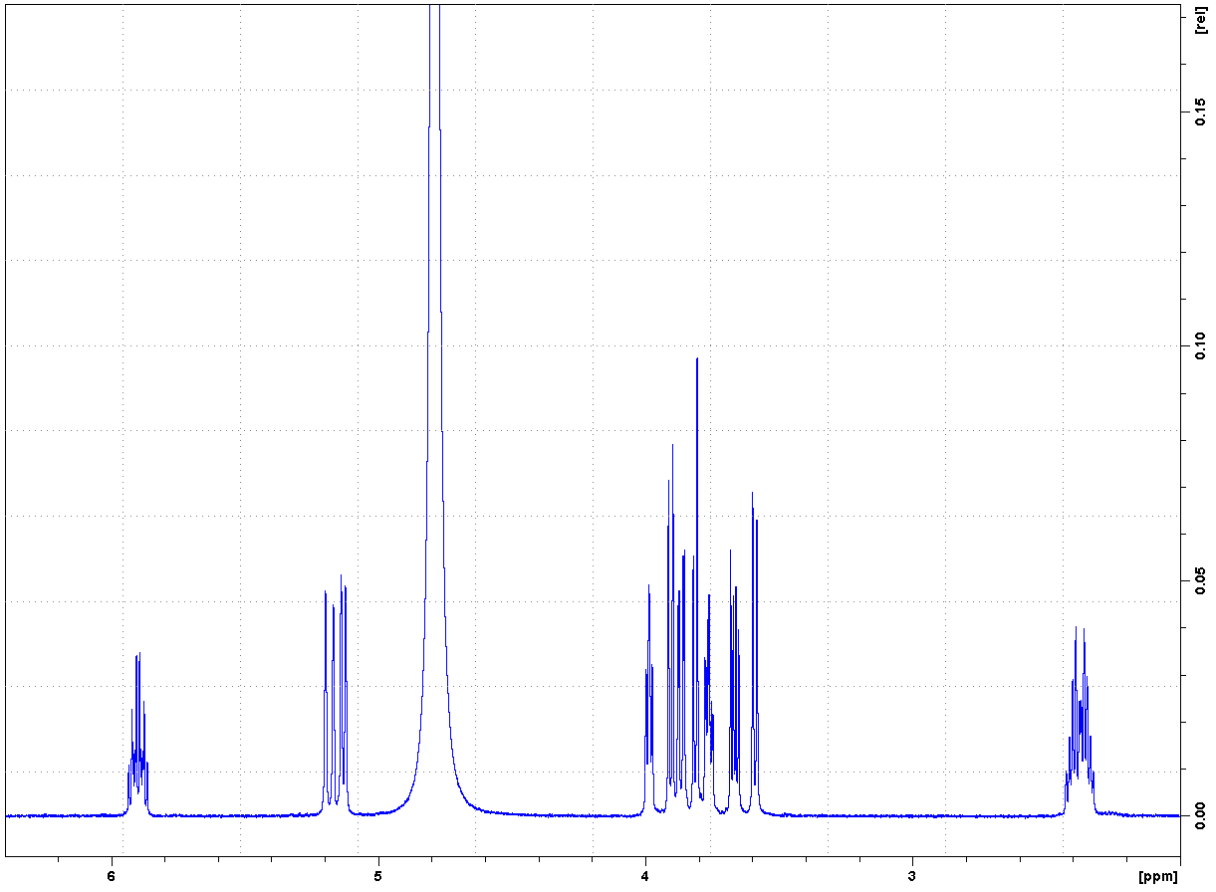
* reko.leino@abo.fi

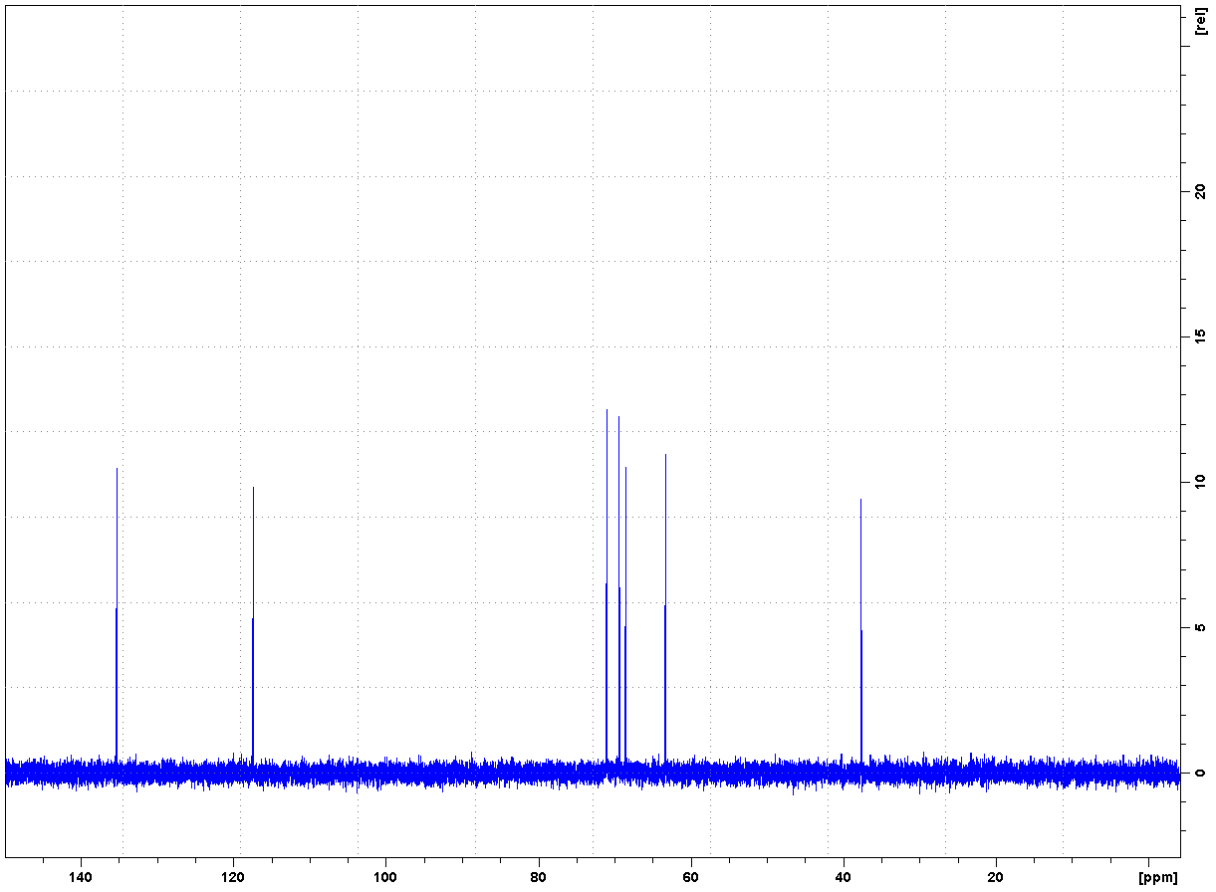
Synthesis of homoallylic polyols 1a, 2a and 3a

General procedure for the allylation of unprotected monosaccharides (details given apply for the allylation of D-mannose): D-Mannose (5 g, 27.8 mmol) was dissolved in 600 mL of EtOH:H_2O (10:1) at room temperature. Tin powder (6.7 g, 55.5 mmol, 2 equiv) and allyl bromide (7.2 mL, 83.4 mmol, 3 equiv) were added. The reaction mixture was stirred under Ar atmosphere at room temperature for 20 min after which the temperature was gradually raised to 60 °C. A greyish suspension was obtained after stirring for ~2 h. The color of the reaction mixture turned gradually to yellow as the stirring was continued for 24 h. The conversion of the starting material was followed by TLC (MeOH/acetone, 1:1). After cooling to room temperature, the reaction mixture was neutralized by adding 5 M NaOH (18 mL). Dichloromethane (300 mL) and water (300 mL) were added, and the phases were separated. The aqueous phase was washed with dichloromethane (2 x 150 mL). The combined organic layers were washed with water (2 x 200 mL). The combined aqueous layers were filtered through celite. The colorless filtrate was concentrated under reduced pressure to obtain the crude product as a white solid. ^{1}H NMR spectroscopic analysis of the crude product: conversion 100%, diastereomeric ratio 3:1 (threo:erythro). The crude product was dissolved in EtOH (350 mL) at 60 °C. Upon cooling, the major diastereomer **1a** precipitated as a white crystalline solid. Typically, 2.8 g (45%) of pure diastereomer was obtained.

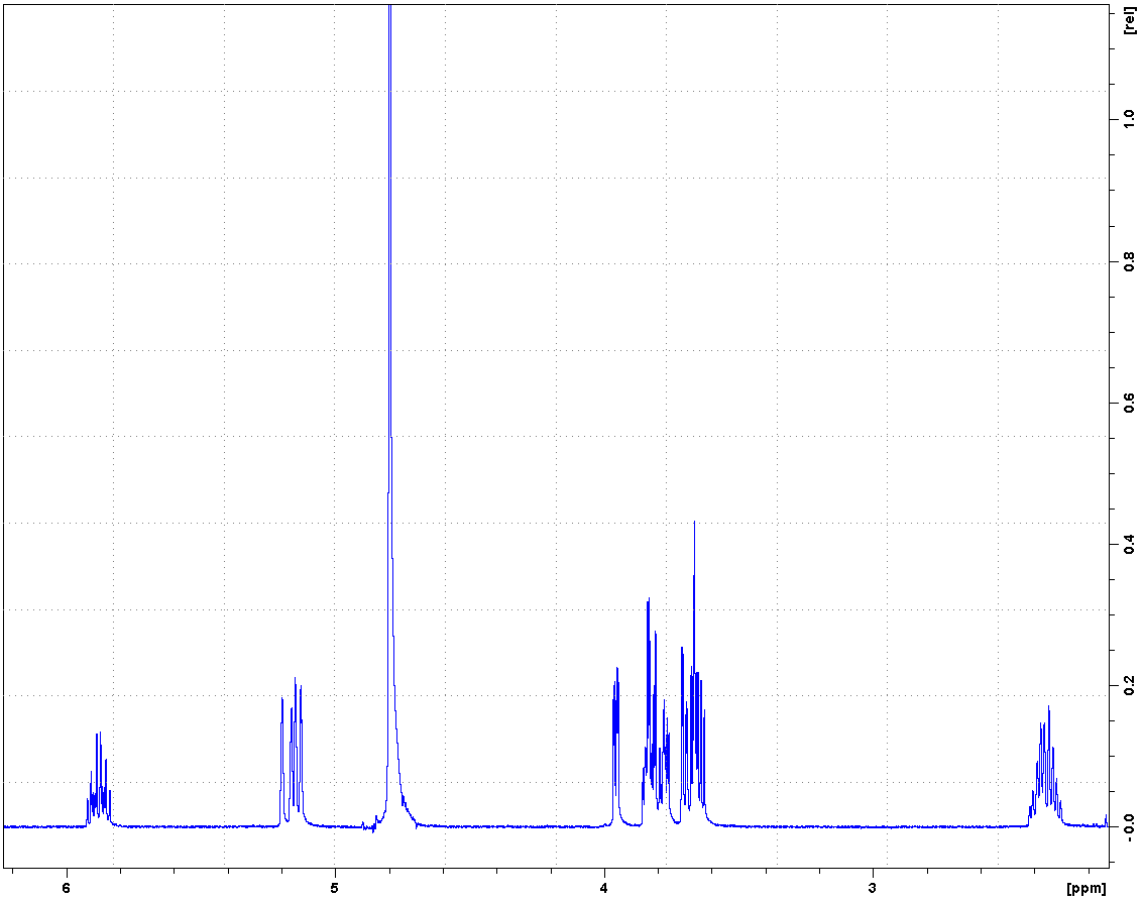
For obtaining diastereomerically pure **2a** and **3a**, a peracetylation – chromatography – deacetylation procedure was pursued.

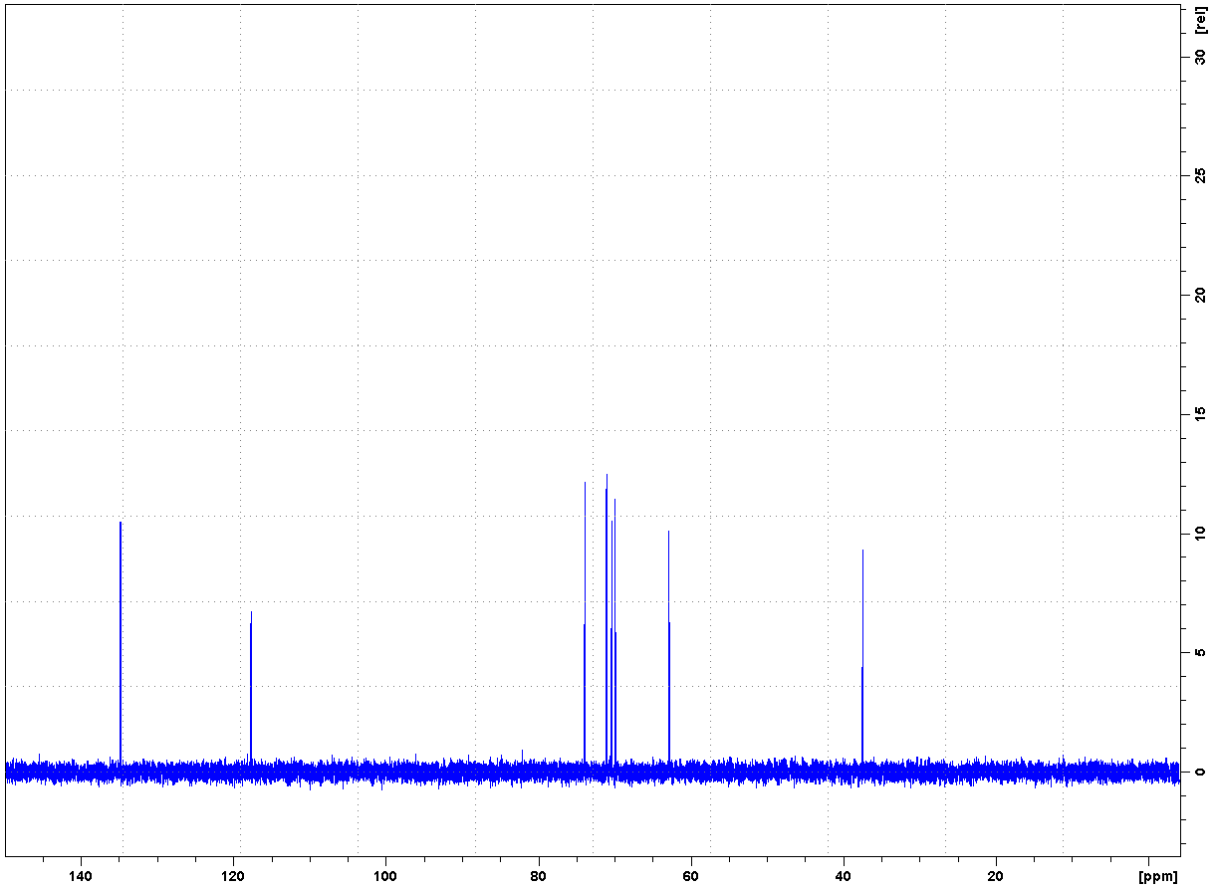
^{1}H and ^{13}C NMR spectra of compound 1a



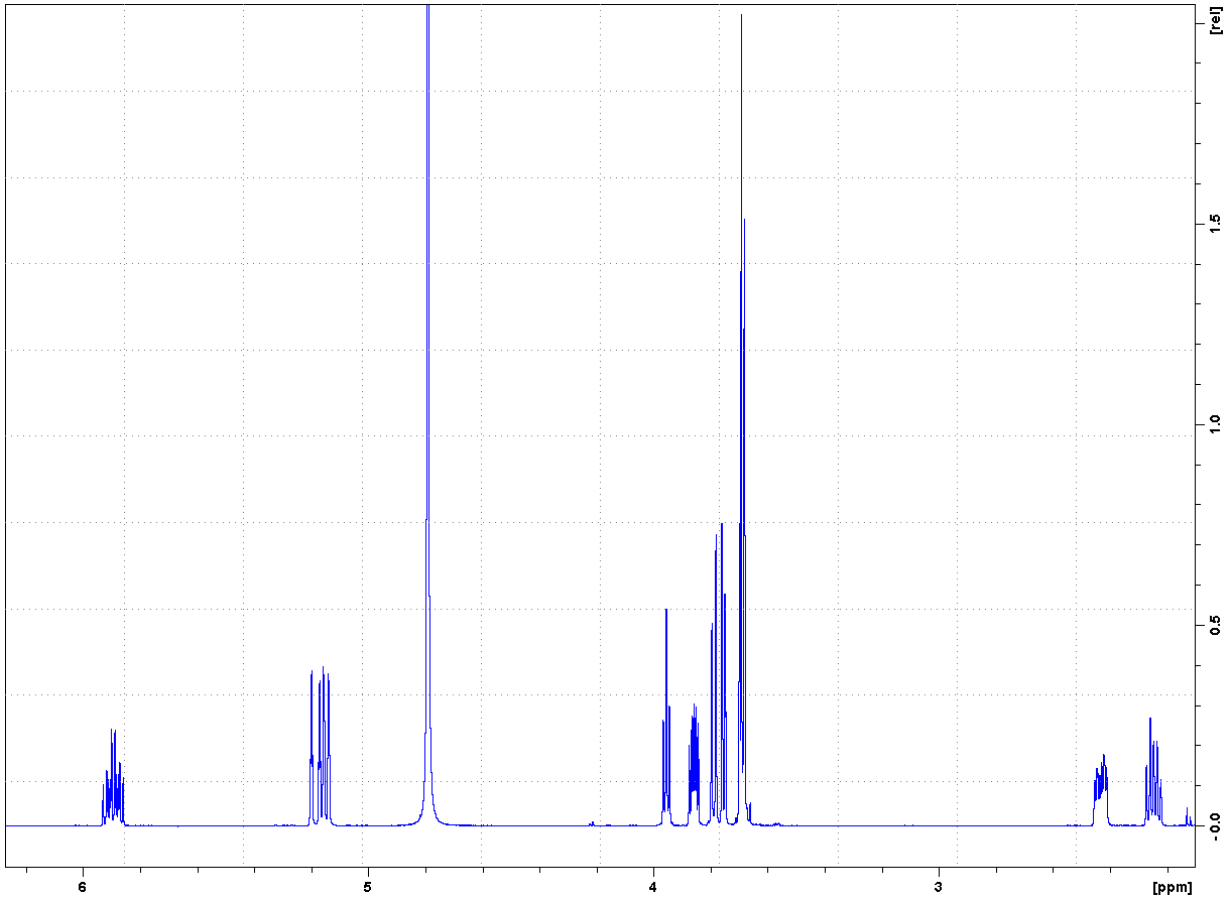


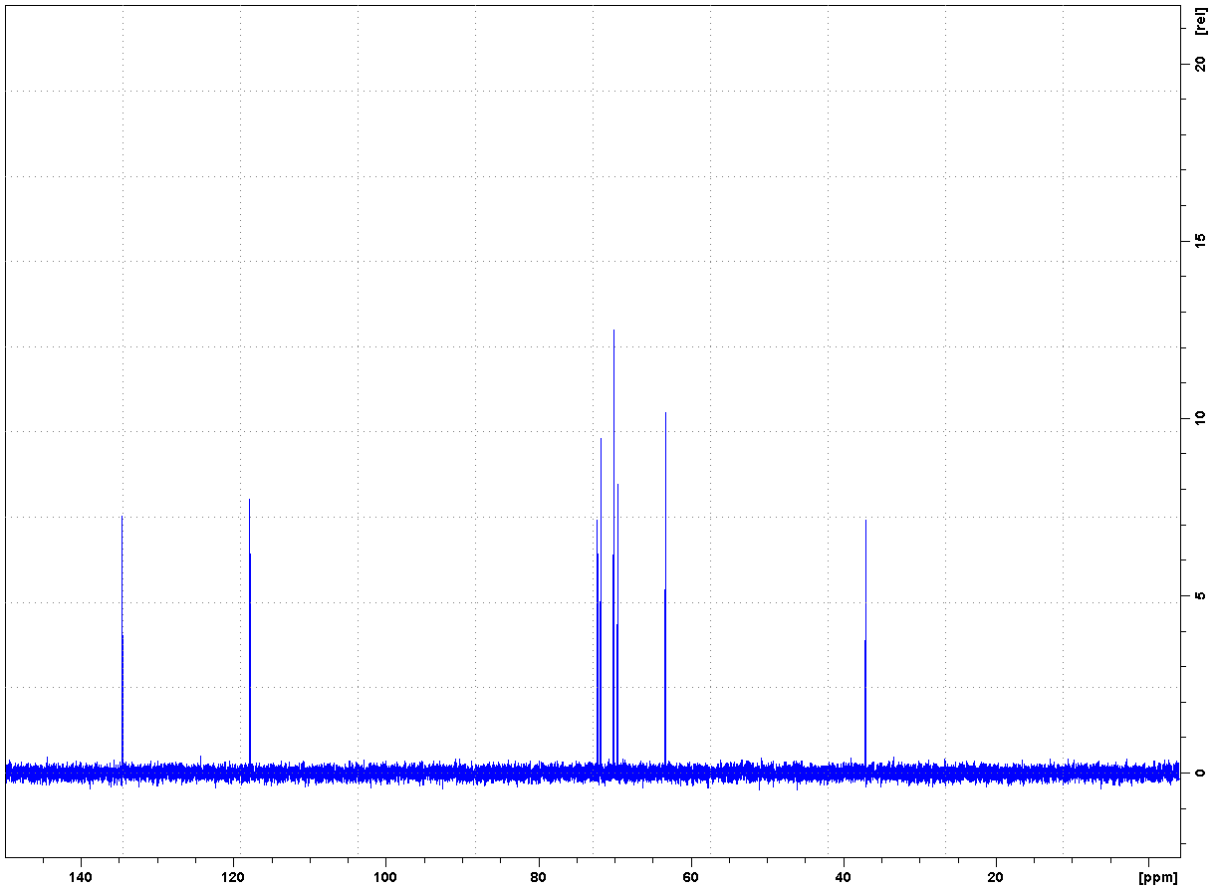
^{1}H and ^{13}C NMR spectra of compound 2a



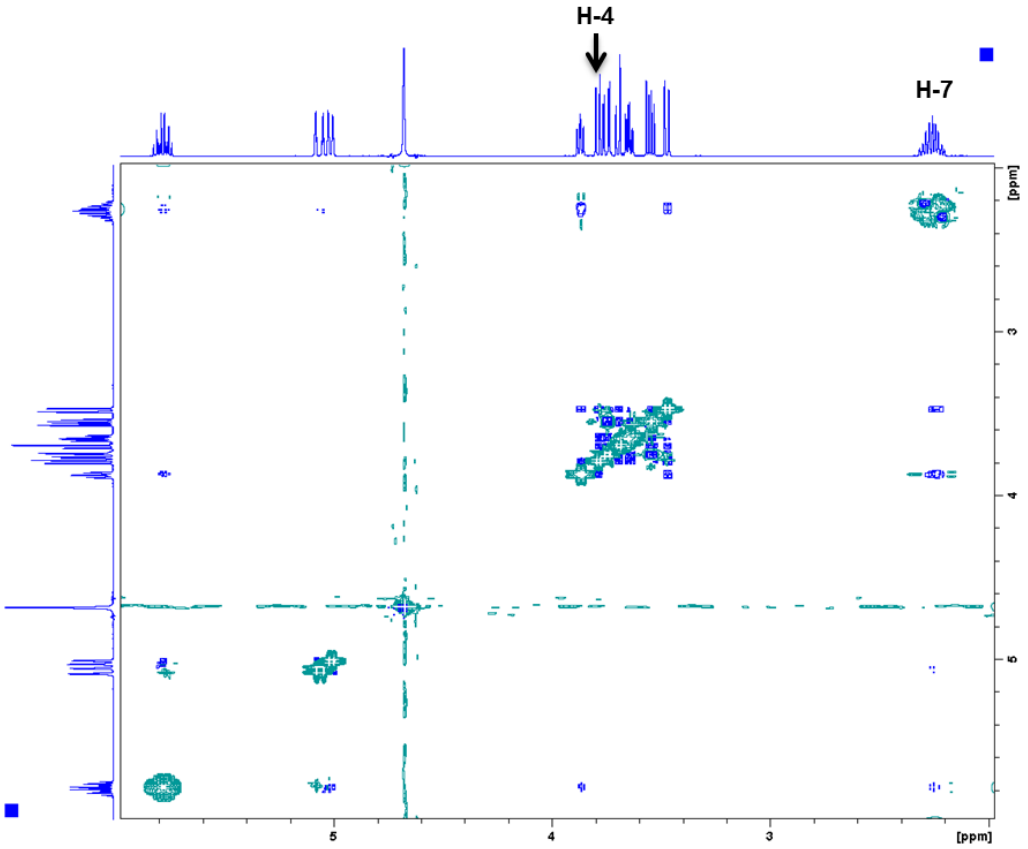


^{1}H and ^{13}C NMR spectra of compound 3a

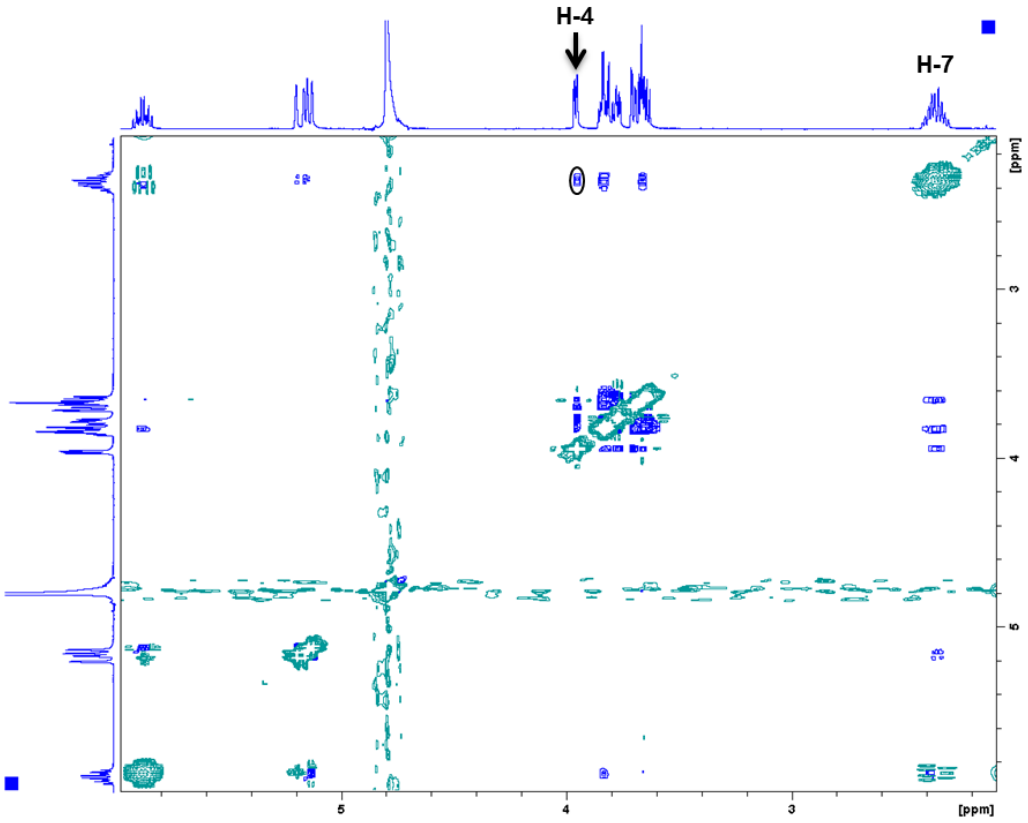




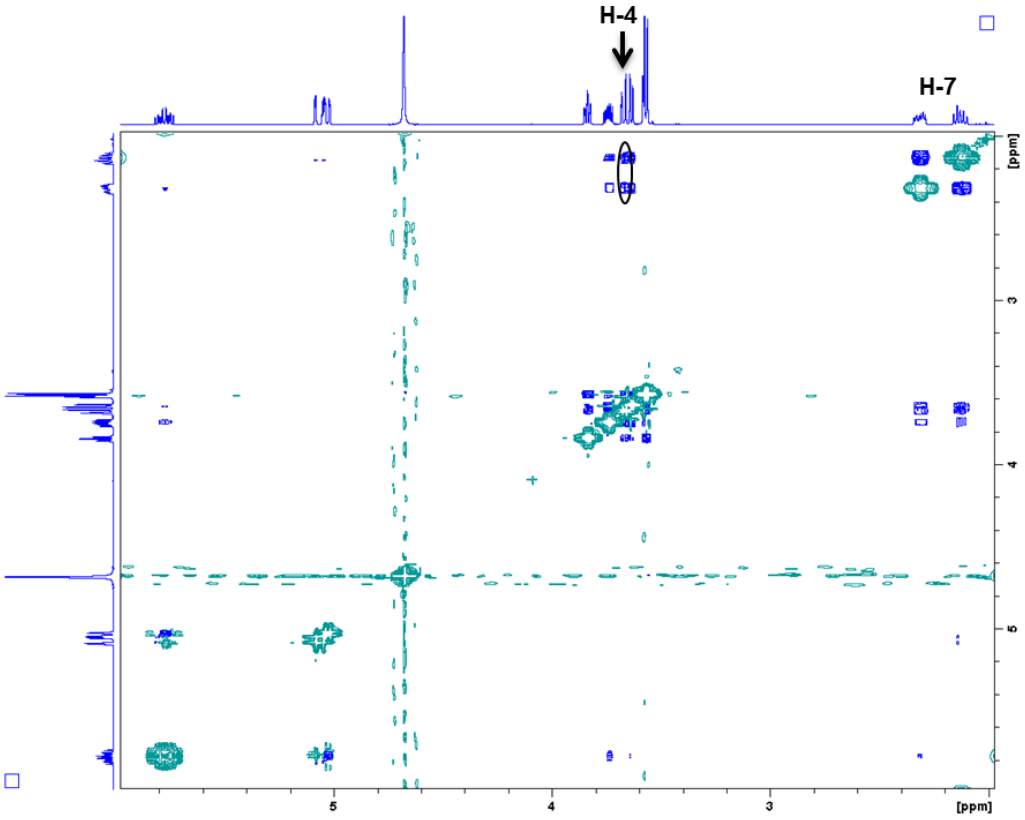
NOESY spectrum of compound 1a



NOESY spectrum of compound 2a



NOESY spectrum of compound 3a



DSC measurements

The samples were characterized by power compensated Perkin Elmer hyper-DSC 8500 differential scanning calorimeter. Measurements were carried out under nitrogen atmosphere (flow rate 40 ml/min) using 50 μl aluminum pan with 30 μl aluminum pan (pinholes) crimped on top as a lid. Temperature calibration was made using two standard materials (*n*–decane and In) and the energy calibrations by an indium standard (28.45 J g^{-1}). Samples were heated from 5 to 200 °C with a heating rate of 10 °C/min, cooled back with a same rate and then heating-cooling cycle was repeated. The melting (T_m), crystallization, (T_c), and solid-solid state phase transition (T_{ss}) temperatures were obtained as an extrapolated onset value. The sample weights used on the measurements were about 1.4 mg.

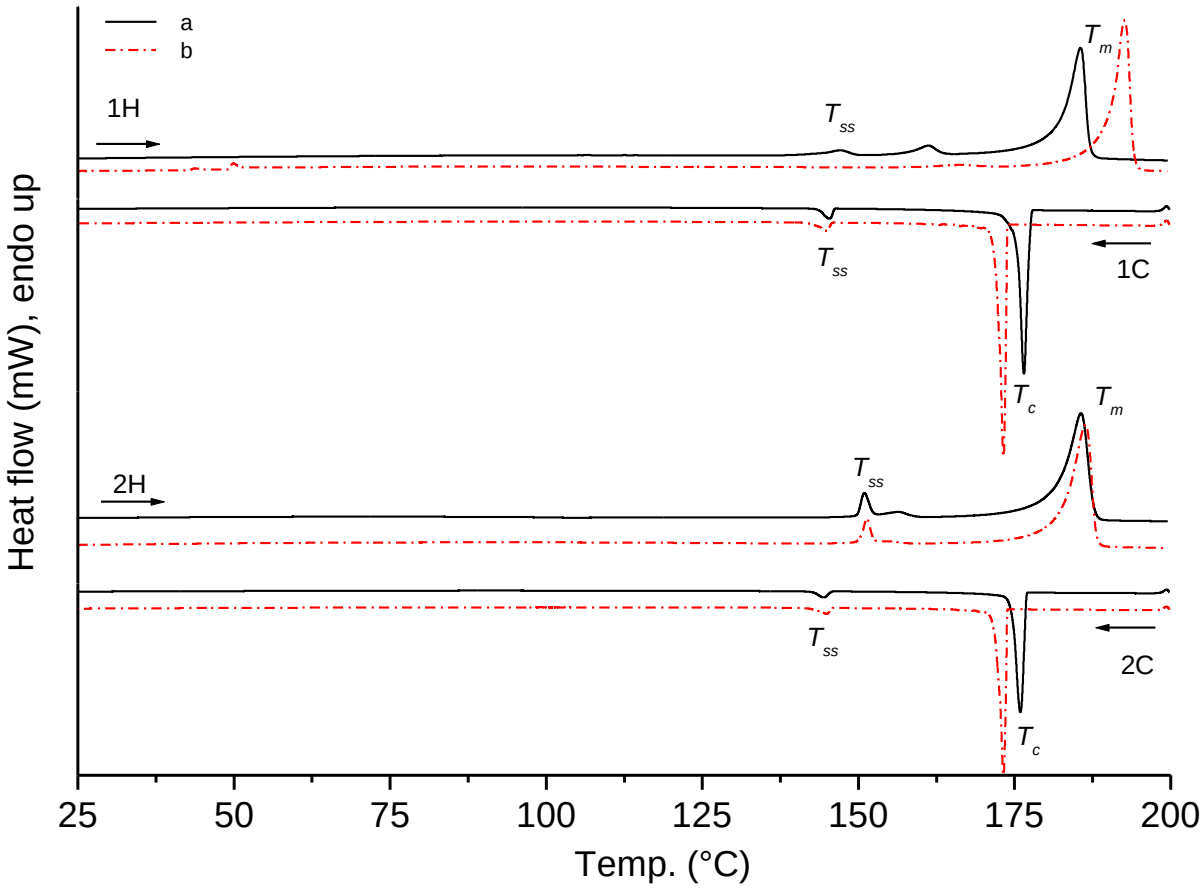


Figure S1. DSC scans of compound a) **1a** (black solid line) and b) aggregation product of **1a** (red dashed line). Heating (H) and cooling (C) scans are indicated by sequential number and direction of propagation.

Table S1. Thermal properties of compound **1a**.

Comp.	1st heat	1st cool	2nd heat	2nd cool
	°C, (J/g)	°C, (J/g)	°C, (J/g)	°C, (J/g)
Bulk	T_{ss} 142.9, (13.34)	T_c 177.4, (-206.22)	T_{ss} 149.9, (15.04)	T_c 176.9, (-204.08)
	T_m 181.9 (181.19)	T_{ss} 146.1 (-12.71)	T_m 181.4, (201.33)	T_{ss} 145.5, (-12.49)
aggregate	T_m 189.9 (246.20)	T_c 174.3 (-212.46)	T_{ss} 150.2 (14.70)	T_c 173.8 (-209.42)
		T_{ss} 145.9 (-12.89)	T_m 181.9 (182.19)	T_{ss} 145.7 (-12.60)

T_m = melting, T_c = crystallization, and T_{ss} = solid-solid state phase transition.

xyz-coordinates for optimized geometries

Compound 1a (gas-phase)

```
  33
DF-LMP2/AUG-CC-PVTZ,H=CC-PVTZ  ENERGY=-803.70746389
C          -4.6625955617       -0.6769164559        0.1572703675
C          -3.3061608579        0.0013928482        0.1201324314
H          -4.8180208800       -1.2573200818       -0.7524695435
O          -5.6220125173        0.3840453124        0.2675604399
H          -4.7150933025       -1.3349536482        1.0302027222
C          -2.1682776734       -1.0108760709        0.1161046092
C          -0.8015166990       -0.3255776160        0.0991924574
O          -2.3214725817       -1.8016547031       -1.0761227461
H          -2.2429589138       -1.6344634092        1.0090115342
C           0.3447941687       -1.3292343665        0.2939157416
H          -0.7870517151        0.4096473119        0.9062507707
O          -0.6165089829        0.3884365128       -1.1199866070
C           1.7020945654       -0.6216758029        0.3807808669
H           0.3613455850       -2.0096814825       -0.5634744496
O           0.1315457848       -2.1296309579        1.4520258326
C           2.8661541237       -1.6063128447        0.3103130635
O           1.7899085162        0.0423650107        1.6561259557
H           1.7791605430        0.1044094058       -0.4312365632
C           4.1958198384       -0.9159539400        0.3492275008
H           2.7765818893       -2.1865901485       -0.6113450666
H           2.7710604088       -2.3003338003        1.1495659982
C           5.0923228169       -0.9653132549       -0.6412524376
H           4.4154012155       -0.3373971744        1.2397136716
H           4.9010197473       -1.5336746566       -1.5439142570
H           6.0364124795       -0.4421390141       -0.5706528600
O          -3.1276391294        0.8029591603        1.2842720329
H          -3.2450199865        0.6208791332       -0.7812888935
H          -6.4951479182       -0.0029425583        0.3856071653
H          -3.9501535055        1.3005676406        1.3890130307
H          -1.9281479279       -2.6654580329       -0.9081357481
H          -0.9614103016       -0.1873319203       -1.8180316315
H           0.4606044509       -1.6003744511        2.1945281074
H           1.4002623209        0.9196240552        1.5685265039
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Compound 1a (COSMO-solvated)

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  33
DF-LMP2/AUG-CC-PVTZ,H=CC-PVTZ  ENERGY=-803.73634925
C          -4.6520421307       -0.7432467670        0.1381662724
C          -3.3248544196       -0.0104301279        0.1180630989
H          -4.7715991312       -1.3322785415       -0.7711708840
O          -5.6679293048        0.2677227220        0.2300036182
H          -4.6918513479       -1.4018892214        1.0098003994
C          -2.1515788280       -0.9829400369        0.0591387791
C          -0.7982154779       -0.2696985180        0.1499798726
O          -2.2411393981       -1.6516869196       -1.2081085361
H          -2.2366461159       -1.6971657258        0.8803416183
C           0.3455944220       -1.2762900158        0.3584682134
H          -0.8199636244        0.4193636422        0.9960881105
O          -0.5815281244        0.5160720618       -1.0199946473
C           1.7192418257       -0.5989447472        0.3862114161
H           0.3336084871       -2.0006041312       -0.4612199039
O           0.1324592701       -2.0119625421        1.5622902532
C           2.8530935180       -1.6217216166        0.3614612216
O           1.8467806853        0.1506313514        1.6085977040
H           1.8071918483        0.0683526011       -0.4730413191
C           4.2014752093       -0.9676019776        0.3256479179
H           2.7250885493       -2.2592696072       -0.5163166744
H           2.7616306001       -2.2509974338        1.2509430720
C           5.0578599386       -1.0719183074       -0.6985330515
H           4.4761136784       -0.3647571072        1.1851386650
H           4.8146074660       -1.6637484529       -1.5740700541
H           6.0173457611       -0.5711107734       -0.6803768842
O          -3.1758930381        0.7644076085        1.3098143270
H          -3.2918182968        0.6434205857       -0.7606055379
H          -6.5055857575       -0.1673571703        0.4320173583
H          -4.0264880289        1.2067839118        1.4477254568
H          -1.9307238898       -2.5591837818       -1.0992427974
H          -0.8390711262       -0.0421202274       -1.7702034048
H           0.4369102000       -1.4360183723        2.2811447070
H           1.4332265812        1.0147076374        1.4832716126
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Compound 2a (gas-phase)

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  33
DF-LMP2/AUG-CC-PVTZ,H=CC-PVTZ  ENERGY=-803.70473248
C          -4.5497136867       -0.8344251620        0.7720126102
C          -3.2709578299       -0.1333574472        0.3554061123
H          -4.9464194866       -1.4228890391       -0.0554303869
O          -5.4576550372        0.2094968802        1.1528121103
H          -4.3415564321       -1.4921392396        1.6215029543
C          -2.1648266452       -1.1329163684        0.0212256744
C          -0.8724626194       -0.4141385454       -0.3712200585
O          -2.5923650009       -2.0552526630       -0.9730956344
H          -1.9650052548       -1.7261451297        0.9183430529
C           0.3302288121       -1.3730403301       -0.5415055824
H          -0.6555152279        0.3033350069        0.4223910229
O          -1.0562496270        0.3368979882       -1.5734797881
C           1.6530727333       -0.6719222582       -0.2113099122
O           0.3631485571       -1.8669614739       -1.8846306565
H           0.2001352439       -2.2288334544        0.1253161249
C           2.8302647330       -1.6399006925       -0.1214648184
H           1.5441253217       -0.1563665287        0.7495641427
O           1.9692728753        0.2665851899       -1.2478900708
C           4.1027587226       -0.9403485133        0.2531489474
H           2.9438816422       -2.1393957601       -1.0877351103
H           2.5951947687       -2.4064037143        0.6206262660
C           4.7926198805       -1.2003099549        1.3677078544
H           4.4528583610       -0.1719241846       -0.4269570758
H           4.4644923957       -1.9603705140        2.0669036227
H           5.7012549737       -0.6628454657        1.6031486374
O          -2.7859334472        0.6740217732        1.4260401704
H          -3.4747996728        0.4878890378       -0.5249191935
H          -3.5557136623        1.1490267381        1.7681916840
H          -2.5882275524       -1.5874242633       -1.8179153189
H          -0.7768775420       -0.2700152482       -2.2790070190
H           1.1057002521       -1.4123193740       -2.3113790567
H           1.2223862279        0.8781748200       -1.3326282301
H          -6.2491367760       -0.1931621098        1.5233569251
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Compound 2a (COSMO-solvated)

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  33
DF-LMP2/AUG-CC-PVTZ,H=CC-PVTZ  ENERGY=-803.73835459
C          -4.6970399434       -0.8183123012        0.4121909924
C          -3.4032606207       -0.0516429042        0.2471857544
H          -4.7863744922       -1.5545909189       -0.3869072251
O          -5.7822464782        0.1143832071        0.3635942877
H          -4.6764780764       -1.3358928274        1.3758704234
C          -2.2387285404       -1.0219747054        0.0120709850
C          -0.8766533873       -0.3301100640        0.0277439079
O          -2.4699399023       -1.6210800024       -1.2679371827
H          -2.2460837917       -1.7808410625        0.8041931173
C           0.2794674334       -1.3421090276        0.0543654614
H          -0.7923239345        0.2603004799        0.9421259866
O          -0.7631514318        0.5875606270       -1.0682315659
C           1.6477772896       -0.6560292051        0.1081135739
O           0.2186718661       -2.2482604765       -1.0569083808
H           0.1554535605       -1.9436628278        0.9586635311
C           2.7628903562       -1.6287907680        0.4824768977
H           1.6002447866        0.1444826181        0.8553423545
O           1.9657101922       -0.1104735657       -1.1792187830
C           4.0830980799       -0.9342772980        0.6205259033
H           2.8253660346       -2.4182605988       -0.2697066319
H           2.4885181035       -2.0901151897        1.4355685256
C           5.1743876067       -1.2299420555       -0.0953764004
H           4.1360701854       -0.1392056547        1.3602028819
H           5.1560529733       -2.0160149240       -0.8417869838
H           6.1037519676       -0.6949702027        0.0517363052
O          -3.2002390727        0.7055753342        1.4420856184
H          -3.4882967879        0.6141465499       -0.6157809992
H          -2.6967421873        1.4982501419        1.2199602675
H          -1.6835961116       -2.1642541708       -1.4546804404
H          -1.2042049474        0.1720983763       -1.8259622321
H           0.7485277510       -1.8467603192       -1.7643735457
H           1.2281767388        0.4794887296       -1.4105196492
H          -6.6008252199       -0.3860949940        0.4705032450
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Compound 3a (gas-phase)

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  33
DF-LMP2/AUG-CC-PVTZ,H=CC-PVTZ  ENERGY=-803.71150282
C          -4.6686613944       -0.6972067333        0.3189193408
C          -3.3168780122       -0.0099787381        0.3529471655
H          -4.8572139041       -1.0869995736       -0.6851758098
O          -5.6305641084        0.3077714222        0.6669491299
H          -4.6856140656       -1.5192820050        1.0371960460
C          -2.1713459631       -0.9985272943        0.1545084525
C          -0.8362182181       -0.2740797924       -0.0752349671
H          -2.3755416989       -1.6297815392       -0.7154638711
O          -2.0711149957       -1.8644884650        1.2802162421
C           0.3083502673       -1.2722285155       -0.2878417196
H          -0.6295089878        0.3520019262        0.7957521878
O          -0.9269279050        0.6161767319       -1.1939367140
C           1.6866526247       -0.6051738236       -0.2066608034
O           0.1346387645       -1.8994308585       -1.5634305481
H           0.2343007230       -2.0443193289        0.4798128699
C           2.8202166123       -1.6239432192       -0.1174542104
H           1.7139225011        0.0357077465        0.6823552828
O           1.9229374002        0.1750733407       -1.3860973412
C           4.1614152263       -0.9646664234        0.0023028261
H           2.7851019283       -2.2526386625       -1.0117096995
H           2.6436263233       -2.2678487988        0.7471858827
C           4.9751023905       -1.1199587331        1.0514281984
H           4.4590440880       -0.3172933624       -0.8150378886
H           4.7029769653       -1.7592909299        1.8832188194
H           5.9312033010       -0.6163429608        1.0994926442
O          -3.1024536989        0.5863580672        1.6356969663
H          -3.2799412745        0.7560536019       -0.4258713612
H          -6.4733558772       -0.1214036362        0.8450840631
H          -3.9334583689        1.0233213784        1.8685017496
H          -2.2441181940       -1.3009005283        2.0496449144
H          -0.9930466925        0.0385949325       -1.9692555771
H           0.7875612490       -1.4831689462       -2.1468471200
H           1.1983529944        0.8168937203       -1.4439751505
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Compound 3a (COSMO-solvated)

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  33
DF-LMP2/AUG-CC-PVTZ,H=CC-PVTZ  ENERGY=-803.73804787
C          -4.7032046166       -0.6954751104        0.1496444690
C          -3.3559462720       -0.0006466997        0.1647422137
H          -4.7978255436       -1.2927577943       -0.7604967384
O          -5.6931102491        0.3438196132        0.1830766534
H          -4.8042328945       -1.3431607690        1.0212979007
C          -2.2008047081       -0.9960096290        0.1895754129
C          -0.8418090312       -0.2967768699        0.0302775720
H          -2.3270785785       -1.7088394856       -0.6290614043
O          -2.2199813355       -1.7532708695        1.3991682816
C           0.3195938061       -1.2947538468        0.0549369442
H          -0.7145314641        0.4054320573        0.8572137805
O          -0.8115840822        0.4972497909       -1.1635036105
C           1.6840623041       -0.5963179541        0.1052388077
O           0.2416835569       -2.1686598653       -1.0752066235
H           0.2123774155       -1.9084637363        0.9504212313
C           2.8136126962       -1.5679219356        0.4384435356
H           1.6417317999        0.1853597603        0.8721871321
O           1.9880932719       -0.0156972760       -1.1716371797
C           4.1333954011       -0.8700430554        0.5572081783
H           2.8626171677       -2.3454714972       -0.3269810393
H           2.5624446021       -2.0457335059        1.3897366745
C           5.2069795433       -1.1448648486       -0.1935908757
H           4.2014917092       -0.0868313881        1.3083958758
H           5.1734432393       -1.9179361041       -0.9533140106
H           6.1366905140       -0.6062498525       -0.0623305129
O          -3.2244340220        0.8037054095        1.3444112079
H          -3.2719837029        0.6285085375       -0.7253096896
H          -6.5361106791       -0.0538108295        0.4337273088
H          -4.0759205117        1.2499058063        1.4665592385
H          -2.3990273516       -1.1200478304        2.1111890298
H          -0.9859259203       -0.0956065143       -1.9096569363
H           0.8093839183       -1.7800887940       -1.7585096094
H           1.2453500173        0.5704550865       -1.3906332181
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X-ray diffraction measurements

Single crystal X-ray

Single crystal analysis of crystals of **1a** was carried out with a dual source (Cu/Mo) Agilent SuperNova diffractometer using monochromated (multilayer optics) Cu Kα radiation. A carefully selected needle-shaped colorless crystal was mounted in a MiTeGen MicroMount™ which was kept under a stream of nitrogen at a steady temperature of 123 K during the course of the data collection. High redundancy data collection, reduction and absorption correction were all made using CrysAlis^{PRO} program[1] wherein Friedel pairs were kept separate. The crystal structure was solved with Superflip[2] and refined on F^2 by full matrix least squares techniques with ShelXL[3] program as implemented in Olex[2] (v.1.2.5) program package.[4] All atoms heavier than hydrogen were refined with anisotropic displacement parameters, whereas hydrogen atoms were treated as follows: C-H hydrogens were calculated into their ideal positions using isotropic displacement parameters 1.2 times of the host atom. All O-H atoms were located on a difference Fourier map and refined without geometrical restraints, except the disordered H atoms of O5 and O6 for which mild distance restraints were applied in order to ensure reasonable hydrogen bonding geometries. The lack of atoms heavier than oxygen increases the uncertainty of correct assignment of absolute structure as reflected in the Flack and Hooft parameters (see Table S2). Therefore, a Bijvoet-Pair analysis was additionally performed using Platon program [5] where a probability of 1.0 (two enantiomorph model with Friedel pair coverage of 98%) was afforded as an indication of a correct assignment of the absolute structure.

Powder X-ray diffraction

Powder samples of compound **1a** were examined by powder X-ray diffraction (PXRD) using a PANalytical X´Pert PRO alpha 1 diffractometer with Cu $K_{\alpha 1}$ radiation (1.5406 Å; 45kV, 30mA). Each sample was prepared onto a silicon-made (zero-background signal) sample holder using petrolatum jelly as an adhesive. Diffraction intensities were recorded by an X´Celerator detector at room temperature with 2θ-range of 3–70° with a step size of 0.017° and counting time of 120 s per step. Data processing was made with X´pert HighScore Plus v. 4.1 program. ICDD PDF-4+ powder diffraction database[6] was used for the qualitative phase analyses. The unit cell parameters of the aggregated powder of **1a** were determined with Pawley analysis using the single crystal structure parameters as basis of the refinement (variable parameters were as follows: zero-offset, unit cell and peak profile parameters).

Table S2. Crystallographic data for structure **1a**.

CCDC no.	1406085
Empirical formula	$C_9H_{18}O_6$
Formula weight (g/mol)	222.23
Temperature/K	123.0(1)
Crystal system	Orthorhombic
Space group	$P2_12_12$
a/Å	21.1909(5)
b/Å	8.8667(2)
c/Å	5.55223(12)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1043.22(4)
Z	4
ρ_{calc} g/cm^3	1.415
μ/mm^{-1}	1.014
F(000)	480.0
Crystal size/mm^3	0.275 × 0.052 × 0.032
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	8.346 to 153.47
Index ranges	$-25 \le h \le 26$, $-10 \le k \le 11$, $-6 \le l \le 6$
Reflections collected	10568
Independent reflections	2160 [R_{int} = 0.0291, R_{sigma} = 0.0198]
Data/restraints/parameters	2160/4/166
Goodness-of-fit on F^2	1.087
Final R indexes [I>=2σ (I)]	R_1 = 0.0307, wR_2 = 0.0749
Final R indexes [all data]	R_1 = 0.0321, wR_2 = 0.0758
Largest diff. peak/hole / e Å^{-3}	0.19/-0.21
Flack parameter	-0.09(10)
Hooft parameter	-0.04(9)

Table S3. Bond lengths for crystal structure of **1a**.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O1	C1	1.433(2)	C2	C3	1.535(2)
O2	C2	1.430(2)	C3	C4	1.526(2)
O3	C3	1.435(2)	C4	C5	1.541(2)
O4	C4	1.430(2)	C5	C6	1.524(2)
O5	C5	1.419(2)	C6	C7	1.538(3)
O6	C6	1.427(2)	C7	C8	1.496(3)
C1	C2	1.516(2)	C8	C9	1.316(3)

Table S4. Bond angles for crystal structure of **1a**.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O1	C1	C2	111.58(15)	C3	C4	C5	111.38(15)
O2	C2	C1	107.38(15)	O5	C5	C4	108.84(15)
O2	C2	C3	110.00(14)	O5	C5	C6	110.26(16)
C1	C2	C3	112.49(14)	C6	C5	C4	112.15(15)
O3	C3	C2	110.30(14)	O6	C6	C5	108.79(16)
O3	C3	C4	110.26(14)	O6	C6	C7	111.11(15)
C4	C3	C2	112.86(14)	C5	C6	C7	112.27(16)
O4	C4	C3	107.68(14)	C8	C7	C6	111.23(18)
O4	C4	C5	109.90(14)	C9	C8	C7	124.4(2)

Table S5. Torsion angles for crystal structure of **1a**.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
O1	C1	C2	O2	-61.65(18)	C1	C2	C3	O3	57.24(19)
O1	C1	C2	C3	59.5(2)	C1	C2	C3	C4	-178.95(15)
O2	C2	C3	O3	176.88(14)	C2	C3	C4	O4	-60.21(18)
O2	C2	C3	C4	-59.30(18)	C2	C3	C4	C5	179.23(15)
O3	C3	C4	O4	63.63(18)	C3	C4	C5	O5	-58.9(2)
O3	C3	C4	C5	-56.93(18)	C3	C4	C5	C6	178.86(15)
O4	C4	C5	O5	-178.11(16)	C4	C5	C6	O6	60.3(2)
O4	C4	C5	C6	59.62(19)	C4	C5	C6	C7	-176.26(16)
O5	C5	C6	O6	-61.1(2)	C5	C6	C7	C8	177.28(17)
O5	C5	C6	C7	62.3(2)	C6	C7	C8	C9	110.5(2)
O6	C6	C7	C8	-60.6(2)					

Table S6. Hydrogen bonds for crystal structure of **1a**.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
O1	H1	O4[1]	0.84(3)	1.94(3)	2.746(2)	163(2)
O2	H2	O1[2]	0.80(3)	1.94(3)	2.7259(19)	167(3)
O3	H3	O2[3]	0.82(3)	1.99(3)	2.7767(19)	159(2)
O4	H4	O3[2]	0.85(3)	1.88(3)	2.7172(18)	170(3)
O5	H5A	O5[4]	0.77(4)	2.02(5)	2.729(3)	154(7)
O5	H5B	O6[5]	0.72(5)	2.01(5)	2.726(3)	172(8)
O6	H6A	O5[2]	0.77(5)	1.99(5)	2.726(3)	160(7)
O6	H6B	O6[4]	0.73(4)	2.07(5)	2.771(3)	162(7)

[1]1/2-X,-1/2+Y,2-Z; [2]+X,+Y,-1+Z; [3]1/2-X,1/2+Y,2-Z; [4]-X,-Y,+Z; [5]+X,+Y,1+Z

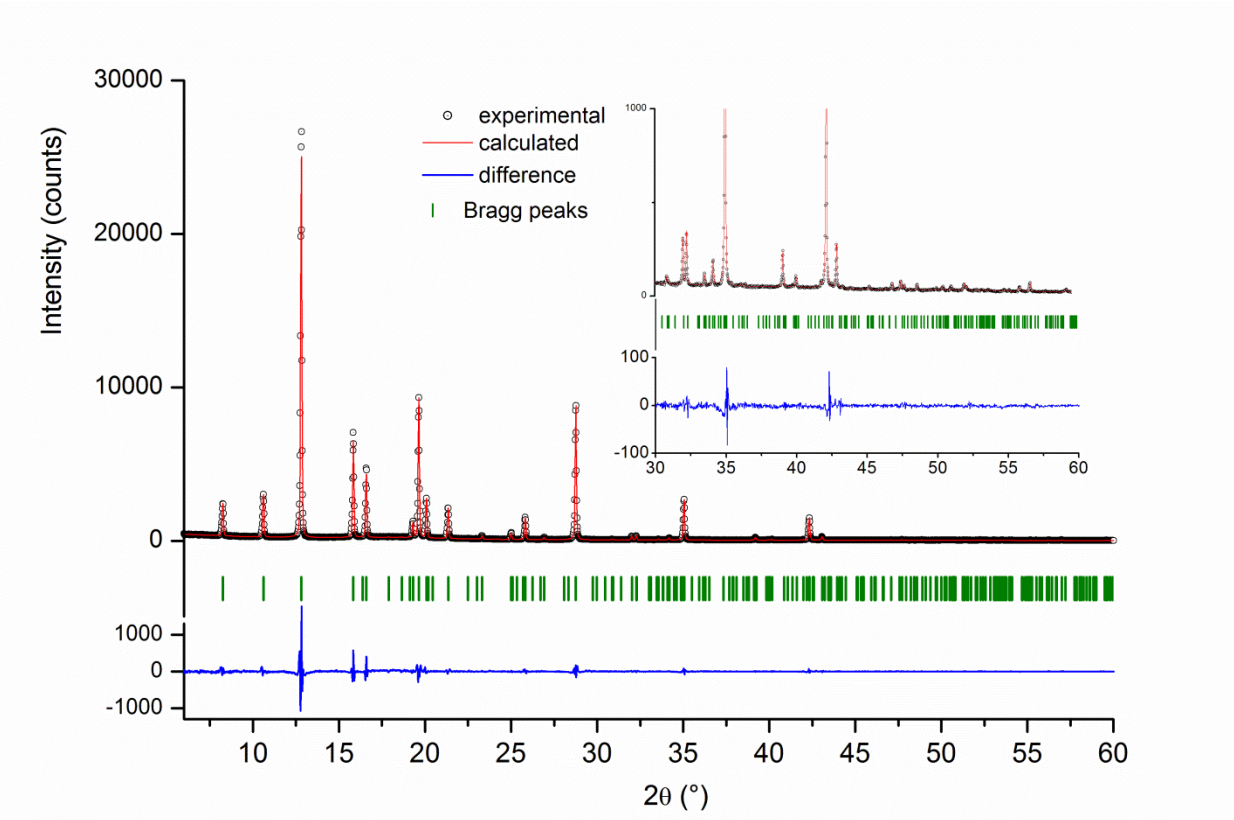

Figure S2. Pawley refinement plot of compound **1a** with 2θ-range magnification of 30-60° (up right inlay). The experimental and calculated patterns with Bragg peak positions and difference curve are indicated by solid red line, black circle, green bar and solid blue curve, respectively.

Table S7. Comparison of unit cell parameters determined for the aggregated powder and the single crystal of **1a**.

	Aggregation	Single crystal
Temperature/K	293(1)	123.0(1)
Crystal system	Orthorhombic	Orthorhombic
Space group	$P2_12_12$	$P2_12_12$
a/Å	21.3157(2)	21.1909(5)
b/Å	9.0158(1)	8.8667(2)
c/Å	5.5799(7)	5.55223(12)
α/°	90	90
β/°	90	90
γ/°	90	90
Volume/Å^3	1072.34	1043.22(4)
Radiation	Cu Kα (λ = 1.5406)	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	3 to 70	8.346 to 153.47
Goodness-of-fit	1.689	1.087
R expected	0.0391	
R profile	0.0494	

Cryo-Transmission Electron Microscopy (Cryo-TEM)

Glow discharge (Emitech KX100, 2 min/25 mA) treated Quantifoil R2/1 holey carbon copper grid with the hole size of 2 μm was transferred into an environmental chamber of FEI Vitrobot having room temperature and 100 % humidity. 3 μl of sample solution was applied on the grid which was blotted for 1,5 seconds and then shot to 1/1 mixture of liquid ethane and propane of temperature -180 °C. The grid with vitrified sample film was cryotransferred into a FEI Tecnai 12 transmission electron microscope with Gatan 910 cryotransfer holder at temperature ca. -185 °C. Bright-field TEM was performed using an acceleration voltage of 120 kV and images were recorded on Gatan Ultrascan 1000 CCD camera.

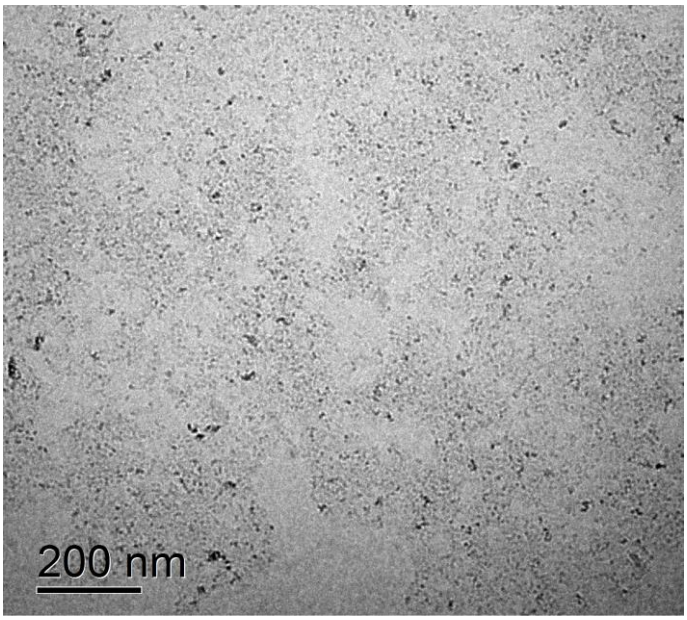

Figure S3 Cryo-TEM image of sample which was vitrified 37 min after the initial dissolution.

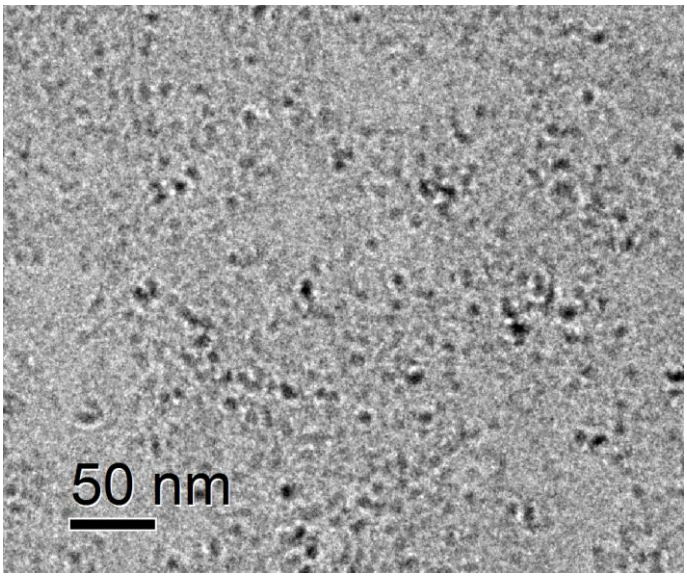

Figure S4 higher magnification cryo-TEM image showing small spherical aggregates with diameter approximately 7-8 nm.

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