Electronic Supplementary Material (ESI) for CrystEngComm.

Plastically bendable Pregabalin multi-component systems with improved tabletability and compressibility

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Supporting Information

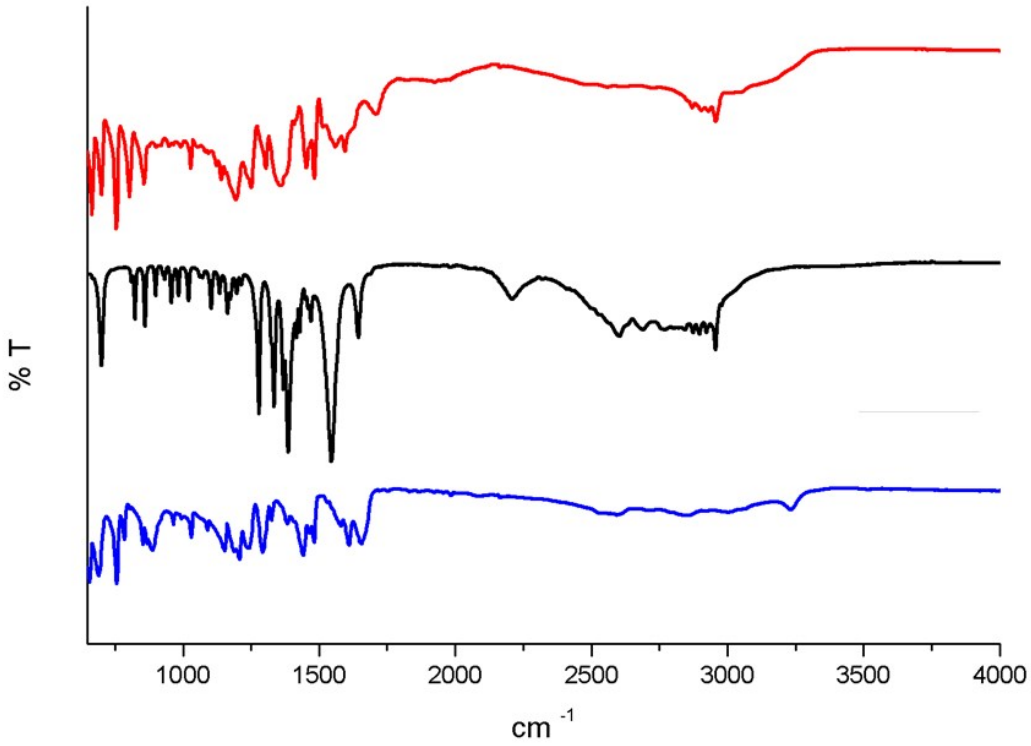

Figure 1. IR spectra comparison for SPG (red), SPG^{+}-SA^{-} (black) and SA (blue).

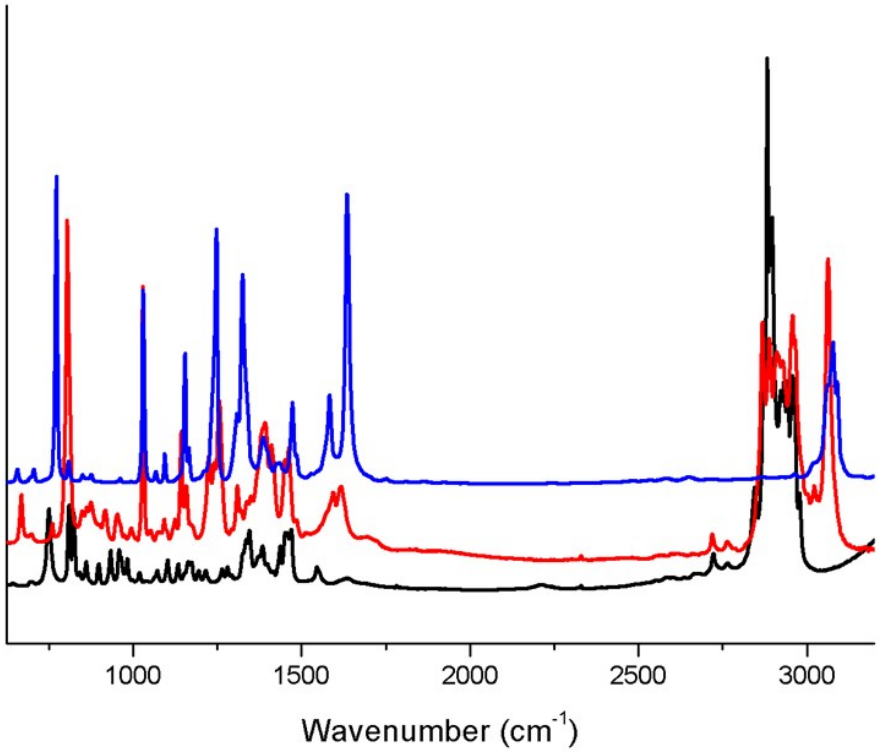

Figure 2. Raman spectra comparison for SPG (black), SPG^{+}-SA^{-} (red) and SA (blue).

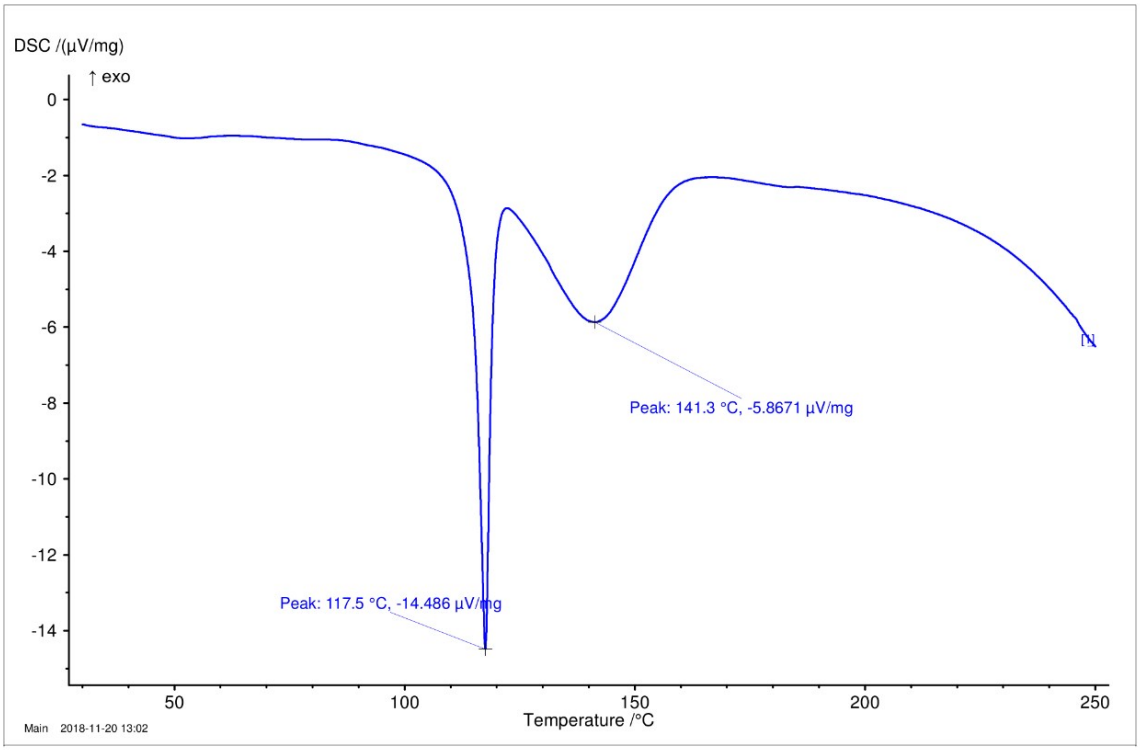

Figure 3. DSC data for SPG^{+}-SA^{-}.

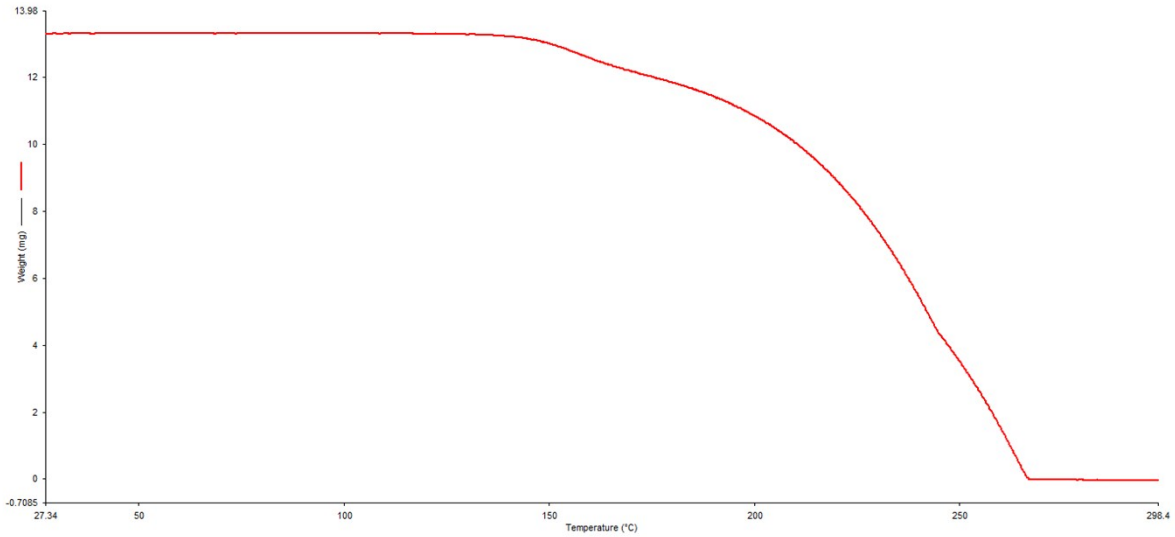

Figure 4. TGA data for SPG^{+}-SA^{-}.

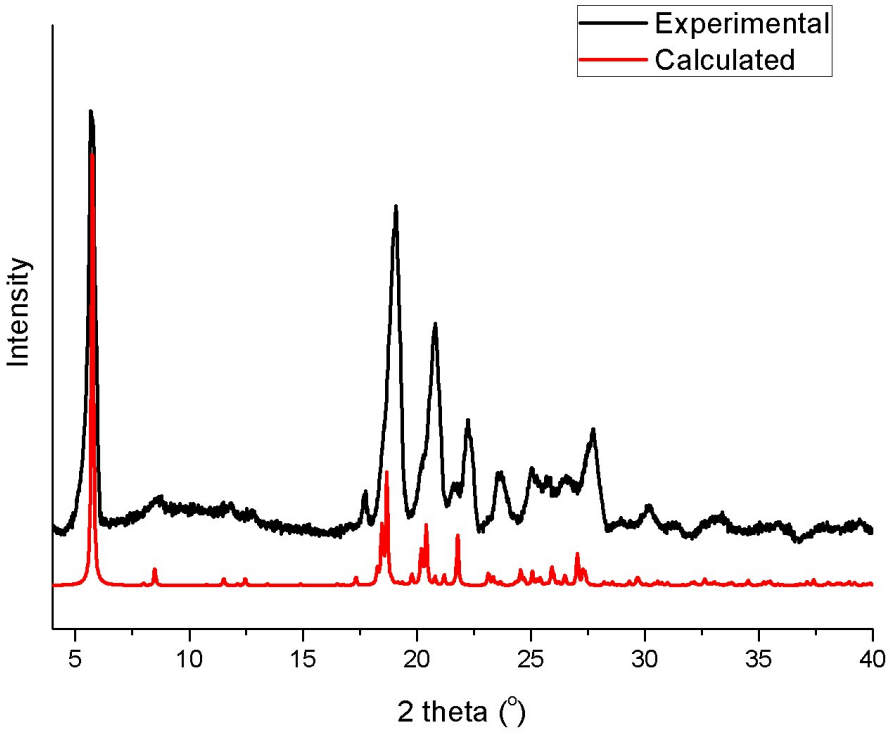

Figure 5. PXRD of for SPG^{+}-SA^{-}.

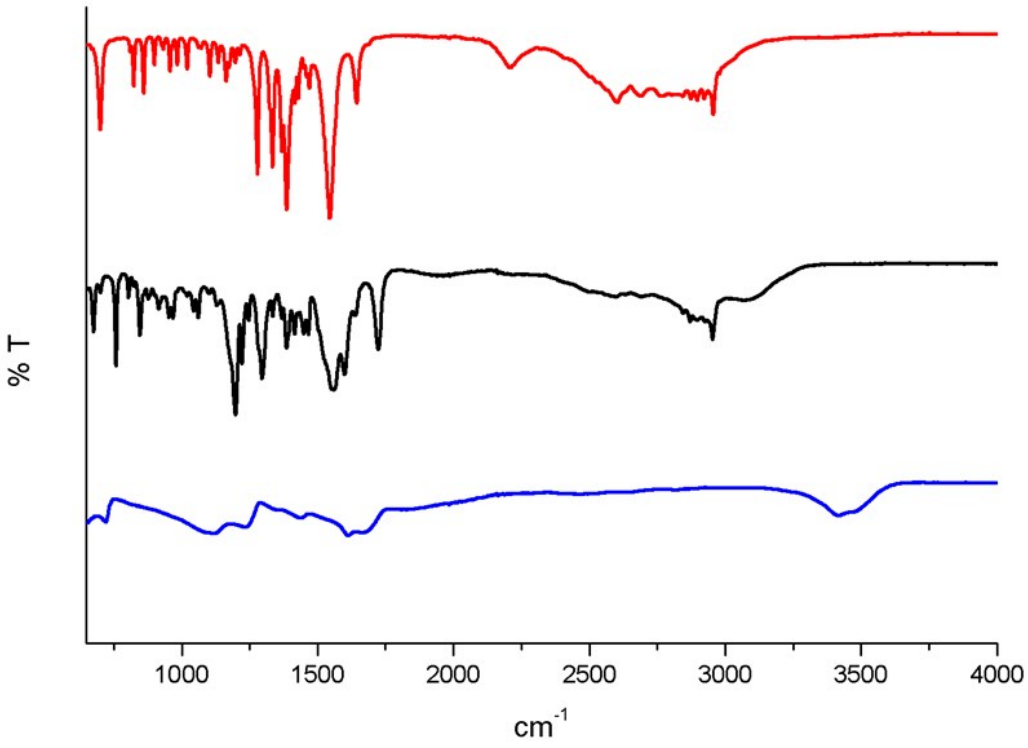

Figure 6. IR spectra comparison for SPG (red), SPG^{+}-OX^{-} (black) and OX (blue).

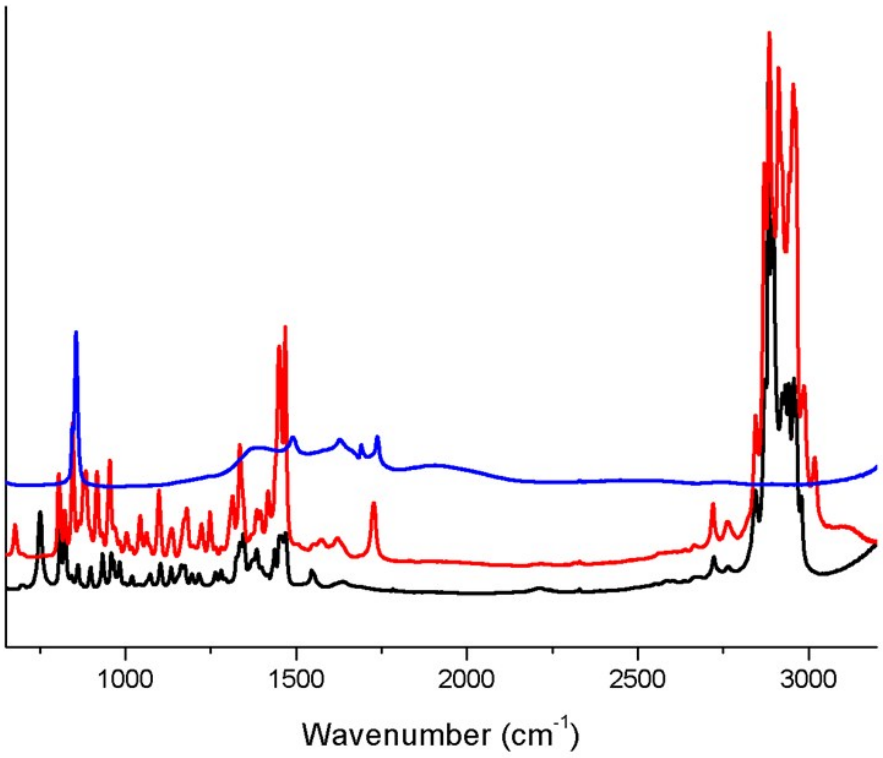

Figure 7. Raman spectra comparison for SPG (black), SPG^{+}-OX^{-} (red) and OX (blue).

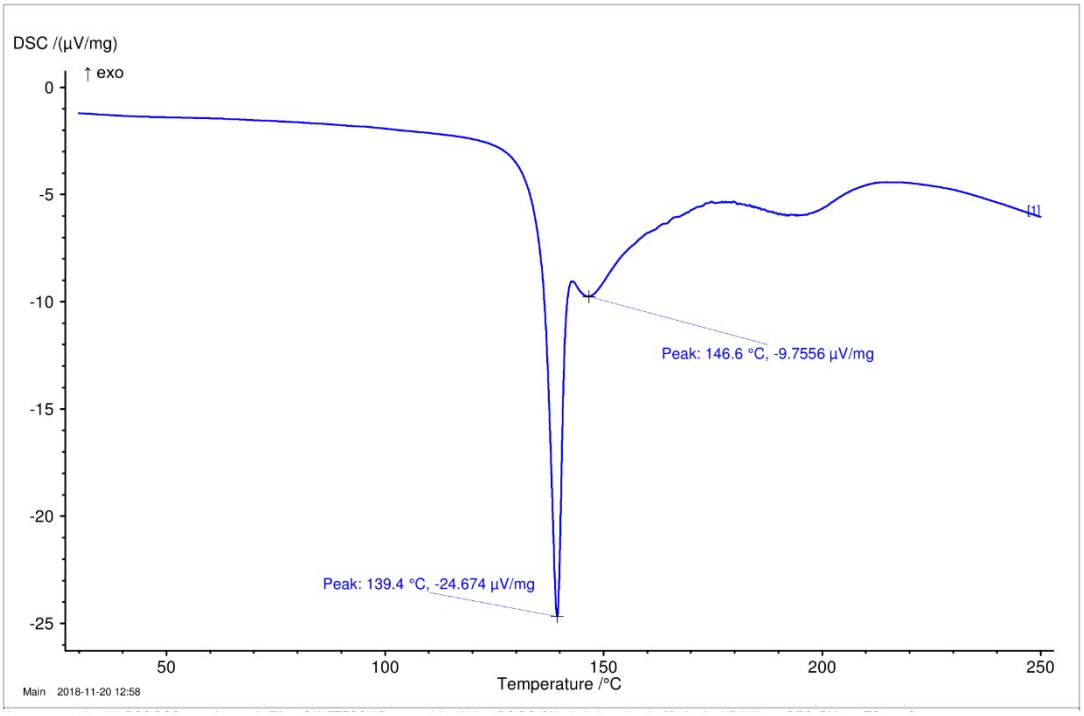

Figure 8. DSC data for SPG^{+}-OX^{-}.

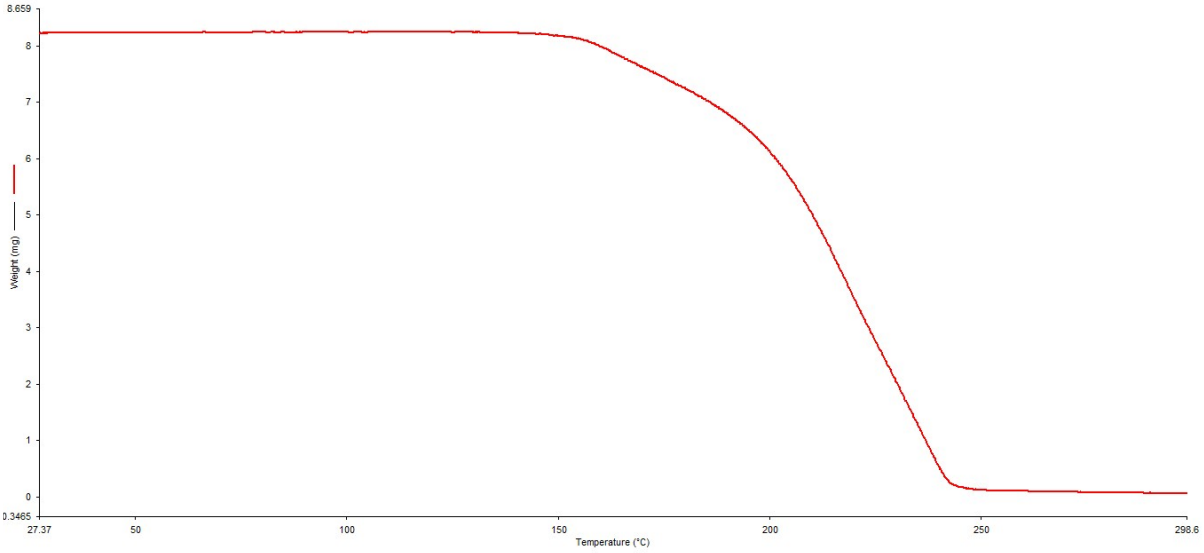

Figure 9. TGA data for SPG^{+}-OX^{-}.

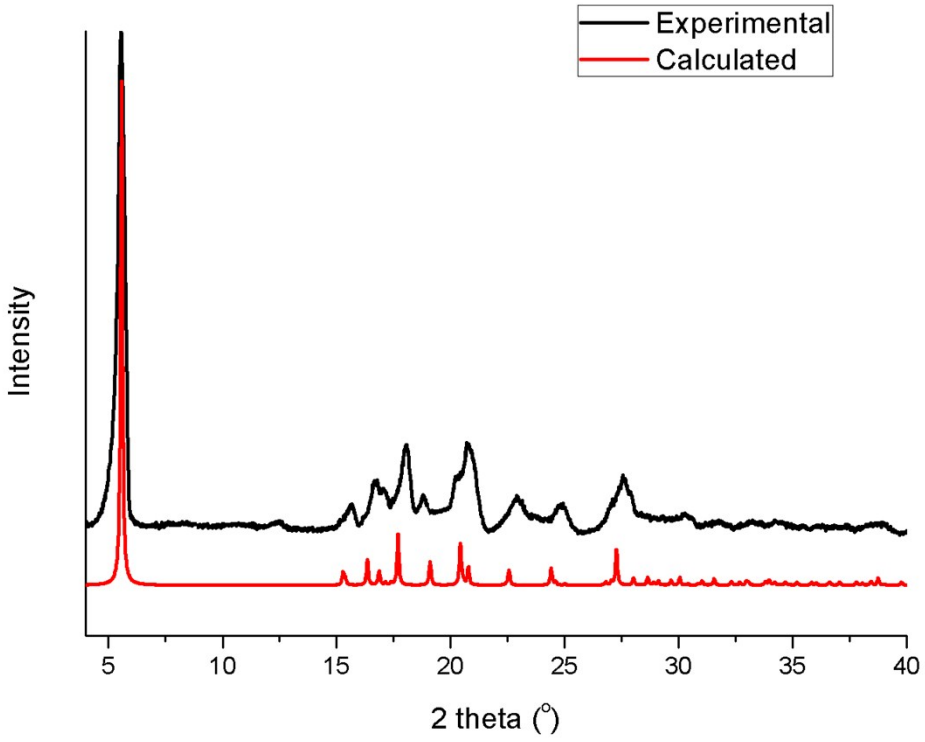

Figure 10. PXRD of SPG^{+}-OX^{-}.

Methodology

Pregabalin (SPG) were purchased from Flourochem and the coformers purchased from Sigma-Aldrich. Methanol was purchased from the Sigma-Aldrich and Milli-Q water was used for the experiments.

Sieving:

The milled powder samples were sieved for 10 minutes using a vibrational sieve shaker (Retsch AS200) at an amplitude of 1 mm. The sieve fraction between 227 and 477 µm was collected and used for further processing.

Powder compression:

Powder compression was performed using a benchtop compaction simulator (Gamlen Tableting D series). A 6 mm round and flat-faced punch and die set was used. The compaction load (200 – 500 kg) was applied at a controlled upper punch velocity of 1 mm s^{-1}. For each compression 70 mg of sample was manually filled into the die and compacted, detached and ejected. Triplicates of each condition were performed. The applied compression force was converted to compaction pressure.

Tensile Strength:

The tensile strength σ_t was calculated using *equation 1* (USP 1217 tablet breaking force). The tablet diameter D and thickness h were measured with callipers. The diametrical tablet breaking force (hardness) F was measured using a texture analyser (TA.XT.plus, Stable Micro Systems) in compression test mode with a plunger (SMSP/20) at fixed distance and a test speed of 2 mm s^{-1}.

$$\sigma_t = \frac{2F}{\pi D h} \tag{1}$$

Tabletability is the relationship between tensile strength σ_t and compaction pressure P. Tabletability defines the change in tensile strength of a compressed body under applied pressure (USP 1062). Tabletability may be described as a linear function of applied pressure P and constants a and b:

$$\sigma_t = aP + b \tag{2}$$

Crystallization of SPG^+-SA^-: SPG^+-SA^- salt crystals were obtained from commercially available SPG and SA (1:1) dissolved in methanol and water mixture (1:1) to saturation and left in fridge for 7 days.

Crystallization of SPG^+-OX^-: SPG^+-OX^- salt crystals were obtained from commercially available SPG and OX (1:0.5) dissolved in methanol and water mixture (1:1) to saturation and left in fridge for 10 days.

IR spectroscopy: IR spectra for SPG^{+}-SA^{-} and SPG^{+}-OX^{-} were collected on a PerkinElmer Spectrum 100 F T-IR Spectrometer equipped with a PerkinElmer Universal ATR Sampling Accessory.

Raman spectroscopy: Raman spectra for SPG^{+}-SA^{-} and SPG^{+}-OX^{-} were collected on a Horiba Jobin Yvon LabRam Aramis spectrometer with a 532 nm laser source. The spectrometer was coupled with an Olympus BX40 confocal microscope with a CCD camera cooled by a thermoelectric Peltier device. Raman maps were processed using the LabSPEC 5 software package.

Powder X-ray diffraction: Powder X-ray diffraction data for SPG^{+}-SA^{-} and SPG^{+}-OX^{-} were collected on an Empyrean diffractometer (PANalytical, Philips) using Cu Kα1,2 radiation (λ = 0.1541 nm) at room temperature operated at 40 kV and 40 mA. The samples were scanned over the range 4–40° 2θ using a step size of 0.02° 2θ and a scan speed of 0.02° 2θ s^{-1}.

Differential scanning calorimetry: calorimetric measurements of SPG^{+}-SA^{-} and SPG^{+}-OX^{-} were performed on a DSC 214 Polyma, NETZSCH instrument. Typically, 3–5 mg of sample was accurately weighed into a hermetically sealed aluminium pan and heated to 250 °C at a 10 °C min^{-1} heating rate under a nitro-gen gas flow of 40 ml min^{-1}.

Thermogravimetric analysis: Thermograms of SPG^{+}-SA^{-} and SPG^{+}-OX^{-} were measured with a Perkin-Elmer TGA 4000 instrument at a heating rate of 10 °C min^{-1} under a nitrogen stream of 20 ml min^{-1}.

X-ray crystallography: An Oxford Diffraction Xcalibur system was used to collect X-ray diffraction data at 150 K using Mo Kα radiation. The crystal structures were solved using ShelxT (2018/2) and refined using Shelxl (2018/3) within the Oscail package.[1-3]

Solubility Test Procedure:

The solubility measurement of three different compounds in water were performed in a 20 ml vial which was stirred using a magnetic stirrer bar. Using 10 ml of water, the sample was prepared for each compound with different concentrations to determine the solubility over a range of saturation temperatures. A 20 ml vial was completely submerged in a transparent poly carbonate bath filled with deionized water. A TX150 thermostat was programmed using LabWise 2.1.2 software to control the temperature of water bath in combination with an immersion cooler. The detection of the solubility point was performed based on laser diffraction principle using an MRL-III-635L-30Mw laser which illuminates a focused beam of red light passing through the slurry. A laser detector measured the intensity of laser power once focused on its length. The investigations were performed by heating the vial at the rate of 0.1 $^{o}Cmin^{-1}$ at 300 rpm. As the heating initiated from a lower temperature, the laser beam was transmitted through the stirring vial and data was logged with respect to time. Upon heating the vial in the setup, the transmission of the light through solution became 100 % at a certain

temperature. The time required for the transmitted light to reach its maximum 100% value through the vial was used in combination with heating rate to determine the saturation temperature of the sample at a given concentration. The laser diode technique offers a good solubility detection and is considered non-intrusive, making it a well-suited process. The MRL-III-635L-30Mw laser has been found to be reliable method which was experimentally proven in previous studies.[2, 3]

Crystallography information:

SPG^{+}-SA^{-} room temperature data:

Table 1. Crystal data and structure refinement for RT-SPG^{+}-SA^{-}

Identification code	RT-SPG^{+}-SA^{-}	
Empirical formula	C15 H23 N O5	
Formula weight	297.34	
Temperature	296.4(8) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12$	
Unit cell dimensions	a = 30.706(2) Å	α = 90°.
	b = 9.6913(7) Å	β = 90°.
	c = 11.0426(9) Å	γ = 90°.
Volume	3286.1(4) $Å^3$	
Z	8	
Density (calculated)	1.202 Mg/m^3	
Absorption coefficient	0.090 mm^{-1}	
F(000)	1280	
Crystal size	0.50 x 0.40 x 0.10 mm^3	
Theta range for data collection	3.750 to 29.121°.	
Index ranges	-41<=h<=20, -13<=k<=7, -8<=l<=15	
Reflections collected	10258	
Independent reflections	6786 [R(int) = 0.0330]	
Completeness to theta = 25.242°	99.5 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.00000 and 0.55958	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6786 / 255 / 389	
Goodness-of-fit on F^2	0.961	
Final R indices [I>2sigma(I)]	R1 = 0.0687, wR2 = 0.1680	
R indices (all data)	R1 = 0.1443, wR2 = 0.2141	

Absolute structure parameter	-0.3(10)
Extinction coefficient	n/a
Largest diff. peak and hole	0.255 and -0.213 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for SPG^+-SA^-. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	2263(1)	6248(4)	1381(4)	78(1)
O(43)	2896(1)	9004(4)	713(4)	68(1)
O(21)	2329(1)	1593(4)	3857(4)	78(1)
O(41)	3076(1)	10553(5)	2102(4)	83(1)
N(7)	2082(2)	9647(4)	-213(4)	65(1)
O(3)	2548(2)	7023(4)	3089(4)	86(1)
O(50)	3444(1)	7310(4)	-74(4)	76(1)
O(23)	2404(1)	1991(5)	1904(3)	74(1)
N(27)	2243(1)	4283(5)	5276(4)	66(1)
O(51)	3144(2)	5295(5)	2667(4)	99(2)
C(2)	2274(2)	7099(5)	2182(5)	59(1)
O(53)	3036(2)	3979(5)	4222(5)	107(2)
O(60)	3643(2)	2519(6)	5057(6)	122(2)
C(25)	1606(2)	3353(5)	4123(5)	56(1)
C(4)	1986(2)	8345(5)	2253(5)	58(1)
C(49)	3738(2)	8001(5)	604(5)	54(1)
C(22)	2211(2)	2178(5)	2941(5)	52(1)
C(42)	3165(2)	9579(6)	1397(5)	55(1)
C(44)	3624(2)	9079(5)	1341(5)	53(1)
C(6)	1776(2)	8488(5)	23(5)	60(1)
C(24)	1839(2)	3165(6)	2919(5)	64(1)
C(52)	3277(2)	4444(6)	3409(5)	64(1)
C(54)	3735(2)	4003(5)	3387(5)	57(1)
C(45)	3948(2)	9742(7)	2019(6)	77(2)
C(26)	1799(2)	4548(5)	4848(6)	68(2)
C(11)	570(2)	6176(7)	1144(8)	107(3)
C(48)	4174(2)	7555(7)	534(6)	79(2)
C(28)	1123(2)	3654(7)	3937(7)	84(2)
C(59)	3895(2)	3054(6)	4194(6)	70(2)
C(5)	1623(2)	8439(5)	1321(5)	57(1)
C(47)	4484(2)	8229(9)	1236(8)	99(2)
C(46)	4370(2)	9302(9)	1955(8)	105(2)
C(55)	4016(2)	4539(8)	2532(6)	90(2)

C(58)	4329(3)	2679(8)	4171(8)	102(2)
C(8)	1284(2)	7294(6)	1522(6)	73(2)
C(56)	4444(3)	4152(12)	2504(8)	136(4)
C(9)	855(2)	7433(7)	893(8)	94(2)
C(57)	4598(3)	3229(11)	3338(9)	127(3)
C(29)	847(3)	2503(11)	3558(11)	141(4)
C(10)	608(3)	8712(9)	1196(13)	157(5)
C(30)	778(7)	1590(20)	4790(20)	143(7)
C(30A)	915(6)	1078(16)	4067(17)	111(5)
C(31)	346(7)	3420(30)	3910(20)	157(8)
C(31A)	389(6)	2761(18)	3226(17)	110(5)

SPG$^+$-SA$^-$ Low temperature data:

Table 3. Crystal data and structure refinement for LT-SPG$^+$-SA$^-$

Identification code	LT-SPG$^+$-SA$^-$	
Empirical formula	C15 H23 N O5	
Formula weight	297.34	
Temperature	153.0(7) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12$	
Unit cell dimensions	a = 9.5719(7) Å	α = 90°.
	b = 30.798(2) Å	β = 90°.
	c = 10.9331(9) Å	γ = 90°.
Volume	3223.1(4) Å^3	
Z	8	
Density (calculated)	1.226 Mg/m^3	
Absorption coefficient	0.092 mm^{-1}	
F(000)	1280	
Crystal size	0.50 x 0.40 x 0.10 mm^3	
Theta range for data collection	3.727 to 29.134°.	
Index ranges	-11<=h<=13, -38<=k<=36, -14<=l<=8	
Reflections collected	9425	
Independent reflections	6070 [R(int) = 0.0366]	
Completeness to theta = 25.242°	99.5 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.00000 and 0.60087	

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6070 / 4 / 401
Goodness-of-fit on F^2	1.094
Final R indices [I>2sigma(I)]	R1 = 0.0638, wR2 = 0.1403
R indices (all data)	R1 = 0.0883, wR2 = 0.1609
Absolute structure parameter	-0.8(10)
Extinction coefficient	n/a
Largest diff. peak and hole	0.241 and -0.303 e.$Å^{-3}$

Table 4. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for SPG^+-SA^-. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(53)	-1058(3)	2108(1)	732(3)	34(1)
O(21)	1575(3)	2688(1)	3866(3)	35(1)
O(3)	6948(3)	2436(1)	3119(3)	37(1)
O(1)	6168(3)	2737(1)	1404(3)	35(1)
O(51)	563(3)	1935(1)	2110(3)	40(1)
O(60)	-2722(3)	1547(1)	-74(3)	38(1)
O(23)	1951(3)	2619(1)	1867(3)	37(1)
O(41)	5205(4)	1851(1)	2633(3)	44(1)
N(7)	9610(3)	2922(1)	-185(3)	28(1)
O(50)	2455(4)	1309(1)	5090(4)	50(1)
N(27)	4270(3)	2743(1)	5256(3)	28(1)
O(43)	3822(4)	1940(1)	4232(3)	49(1)
C(2)	7035(4)	2718(1)	2210(4)	25(1)
C(4)	8297(4)	3009(1)	2307(4)	26(1)
C(5)	8412(4)	3383(1)	1381(4)	24(1)
C(6)	8444(4)	3232(1)	57(4)	26(1)
C(8)	7243(4)	3718(1)	1576(4)	31(1)
C(9)	7379(5)	4146(1)	881(4)	40(1)
C(10)	8716(5)	4382(2)	1169(6)	66(2)
C(11)	6128(5)	4431(2)	1160(5)	47(1)
C(22)	2141(4)	2805(1)	2931(4)	27(1)
C(24)	3117(4)	3191(1)	2894(4)	32(1)
C(25)	3367(4)	3404(1)	4137(4)	26(1)

C(26)	4568(4)	3195(1)	4833(4)	29(1)
C(28)	3690(4)	3891(1)	4001(4)	34(1)
C(29)	2498(5)	4173(1)	3543(5)	50(1)
C(30)	1187(6)	4127(2)	4274(7)	94(3)
C(31)	2961(8)	4650(2)	3501(8)	99(3)
C(42)	4347(4)	1708(1)	3406(4)	30(1)
C(44)	3938(4)	1244(1)	3342(4)	25(1)
C(45)	4505(5)	973(2)	2459(4)	40(1)
C(46)	4163(7)	536(2)	2417(5)	66(2)
C(47)	3260(7)	364(2)	3277(5)	60(2)
C(48)	2673(5)	623(2)	4152(5)	44(1)
C(49)	3016(4)	1067(1)	4187(4)	31(1)
C(52)	-419(4)	1839(1)	1409(4)	28(1)
C(54)	-891(4)	1374(1)	1352(4)	26(1)
C(55)	-185(5)	1058(1)	2026(4)	38(1)
C(56)	-594(5)	624(2)	1960(5)	50(1)
C(57)	-1709(5)	507(1)	1222(5)	45(1)
C(58)	-2409(5)	813(2)	548(4)	38(1)
C(59)	-2011(4)	1251(1)	614(4)	28(1)

SPG^{+}-OX^{-} Room temperature data:

Table 5. Crystal data and structure refinement for RT-SPG^{+}-OX^{-}

Identification code	RT-SPG^{+}-OX^{-}	
Empirical formula	C9 H18 N O4	
Formula weight	204.24	
Temperature	294.0(3) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2	
Unit cell dimensions	a = 11.7250(10) Å	α = 90°.
	b = 6.1271(4) Å	β= 99.628(9)°.
	c = 16.0527(15) Å	γ = 90°.
Volume	1136.99(16) $Å^3$	
Z	4	
Density (calculated)	1.193 Mg/m^3	
Absorption coefficient	0.093 mm^{-1}	
F(000)	444	

Crystal size	0.50 x 0.40 x 0.20 mm^3
Theta range for data collection	3.525 to 29.202°.
Index ranges	-14<=h<=15, -8<=k<=7, -21<=l<=20
Reflections collected	5043
Independent reflections	2580 [R(int) = 0.0253]
Completeness to theta = 25.242°	99.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.00000 and 0.51318
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2580 / 1 / 131
Goodness-of-fit on F^2	1.088
Final R indices [I>2sigma(I)]	R1 = 0.0429, wR2 = 0.1064
R indices (all data)	R1 = 0.0510, wR2 = 0.1141
Absolute structure parameter	0.7(6)
Extinction coefficient	n/a
Largest diff. peak and hole	0.235 and -0.188 e.$Å^{-3}$

Table 6. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for RT-SPG$^+$-OX$^-$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(6)	7655(1)	4115(3)	3999(1)	38(1)
O(7)	6006(2)	5027(3)	3170(1)	43(1)
O(12)	5821(2)	11073(3)	4476(1)	52(1)
O(11)	5520(2)	7499(4)	4345(2)	62(1)
N(1)	8304(2)	-502(3)	4322(1)	41(1)
C(5)	6935(2)	3796(3)	3380(2)	31(1)
C(14)	5388(2)	9348(4)	4658(1)	31(1)
C(2)	8327(2)	-1049(4)	3426(2)	38(1)
C(3)	8166(2)	873(4)	2817(2)	32(1)
C(8)	8346(2)	4(5)	1953(2)	45(1)
C(4)	6983(2)	1990(4)	2760(2)	39(1)
C(9)	8436(3)	1704(7)	1268(2)	67(1)
C(11)	8573(6)	540(11)	460(3)	122(2)
C(10)	9425(6)	3294(10)	1535(3)	118(2)

SPG$^+$-OX$^-$ Low temperature data:

Table 7. Crystal data and structure refinement for LT-SPG$^+$-OX$^-$

Identification code	LT-SPG$^+$-OX$^-$	
Empirical formula	C18 H36 N2 O8	
Formula weight	408.49	
Temperature	150.0(1) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2	
Unit cell dimensions	a = 11.5328(10) Å	α = 90°.
	b = 6.1123(5) Å	β = 99.780(9)°.
	c = 16.0138(14) Å	γ = 90°.
Volume	1112.44(17) Å^3	
Z	2	
Density (calculated)	1.219 Mg/m^3	
Absorption coefficient	0.095 mm^{-1}	
F(000)	444	
Crystal size	0.50 x 0.40 x 0.20 mm^3	
Theta range for data collection	3.599 to 29.069°.	
Index ranges	-15<=h<=9, -7<=k<=4, -19<=l<=21	
Reflections collected	2690	
Independent reflections	1838 [R(int) = 0.0269]	
Completeness to theta = 25.242°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.00000 and 0.74806	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1838 / 1 / 131	
Goodness-of-fit on F^2	1.084	
Final R indices [I>2sigma(I)]	R1 = 0.0398, wR2 = 0.0997	
R indices (all data)	R1 = 0.0427, wR2 = 0.1026	
Absolute structure parameter	-1.5(10)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.252 and -0.228 e.Å^{-3}	

Table 8. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for LT-SPG$^+$-OX$^-$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)

O(6)	7679(1)	4112(3)	3985(1)	21(1)
O(7)	6006(1)	5067(3)	3143(1)	23(1)
O(12)	5848(1)	11123(3)	4474(1)	24(1)
O(11)	5512(1)	7534(3)	4329(1)	28(1)
N(1)	8325(2)	-448(3)	4333(1)	22(1)
C(5)	6938(2)	3793(4)	3360(1)	18(1)
C(14)	5395(2)	9377(4)	4649(1)	18(1)
C(2)	8331(2)	-1058(4)	3434(1)	21(1)
C(3)	8174(2)	826(4)	2807(1)	18(1)
C(8)	8338(2)	-110(4)	1949(1)	25(1)
C(4)	6975(2)	1970(4)	2744(1)	21(1)
C(9)	8409(2)	1530(6)	1240(2)	37(1)
C(11)	8556(3)	307(8)	442(2)	65(1)
C(10)	9401(4)	3168(7)	1482(2)	63(1)

Table 9. Multi-component form screening with various organic and inorganic compounds in different ratios at different temperatures

Coformer	Molar Ratio w.r.t. SPG	Solvent (v/v)	Temperature	Result
Oxalic acid	1:1 and 1:2	1:1 of Water and Methanol	RT and ~4°C	(1:0.5) Salt
L-Tartaric acid	1:1 and 1:2	1:1 of Water and Methanol	RT and ~4°C	Pasty material
Adipic acid	1:1 and 1:2	1:1 of Water and Methanol	RT and ~4°C	Pasty material
Malonic Acid	1:1 and 1:2	1:1 of Water and Methanol	RT and ~4°C	Pasty material
Benzoic acid	1:1	1:1 of Water and Methanol	RT and ~4°C	Pasty material
Salicylic acid	1:1	1:1 of Water and Methanol	RT and ~4°C	(1:1) Salt
L-Aspartic acid	1:1 and 1:2	1:1 of Water and Methanol	RT and ~4°C	Pasty material
Glutaric acid	1:1 and 1:2	1:1 of Water and Methanol	RT and ~4°C	Pasty material
Nicotinic acid	1:1.1	1:1 of Water and Methanol	RT and ~4°C	Pasty material
Phosphoric acid	1:1.1	1:1 of Water and Methanol	RT and ~4°C	Pasty material
Nitric acid	1:1.1	Water	RT	Pasty material
Sulphuric acid	1:1.1	Water	RT	Pasty material
Acetic acid	1:1.1	Water	RT	Pasty material
Formic acid	1:1.1	Water	RT	Pasty material
Nicotinamide	1:1	1:1 of Water and Methanol	RT and ~4°C	Separated
Isonicotinamide	1:1	1:1 of Water and Methanol	RT and ~4°C	Separated
Benzamide	1:1	1:1 of Water and Methanol	RT and ~4°C	Separated
Saccharine	1:1	1:0.5 of Water and Methanol	RT and ~4°C	Separated
4, 4'-bipyridine	1:1 and 1:2	1:1 of Water and Methanol	RT and ~4°C	Separated

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