SUPPORTING INFORMATION

Spontaneous resolution of RS-Fluoxetine through tetrafluoroborate conglomerate salt and the racemic kryptoracemate formation via sulfate one.

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COMPLEMENTARY FIGURES AND TABLES

Figure S1. Representative outcomes from the crystallization of Fluoxetine (Flx) with various inorganic and organic salt formers investigated in this study. Shown are oil phases and a degradation phase.
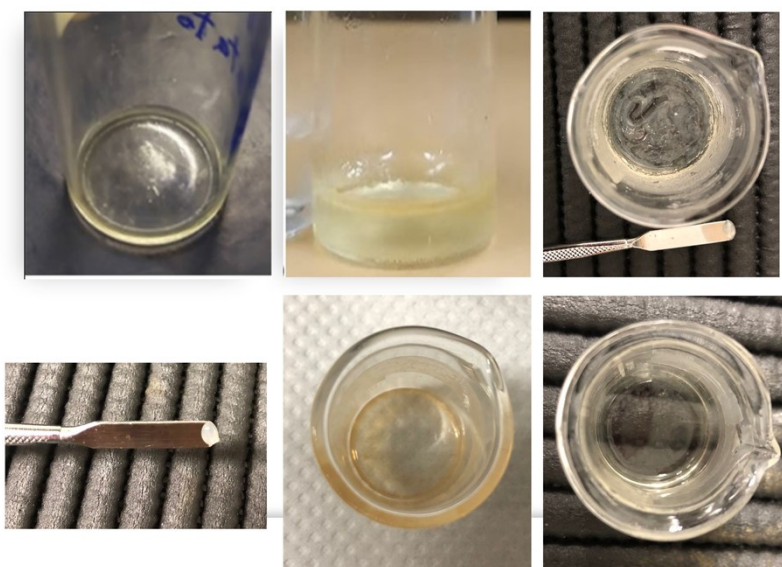
Figure S2. Asymmetric unit of Flx-SO4-h. The water molecules are incorporated in the Flx-SO4-*h* structure H-bonded to Flx-enantiomers.
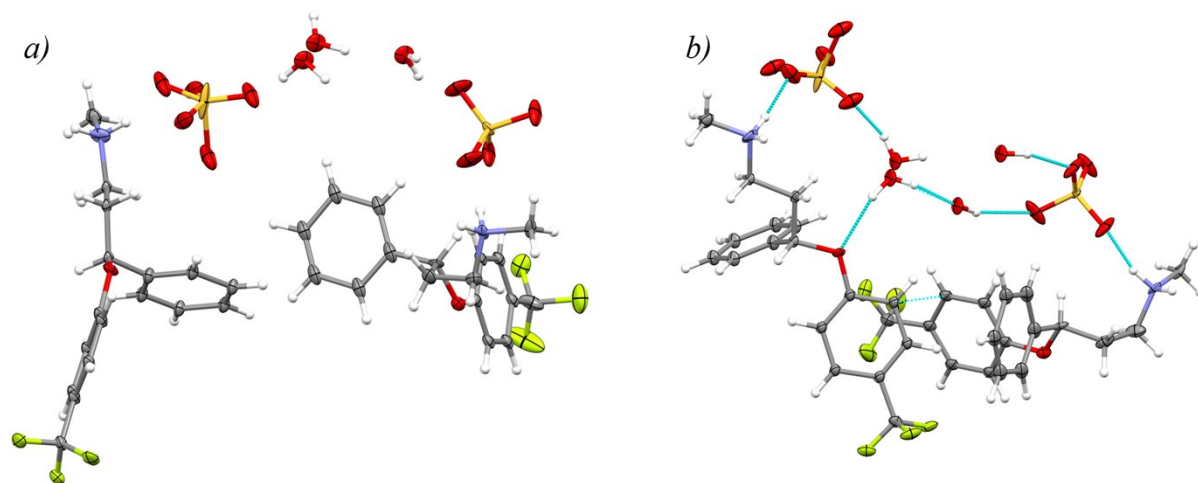
Figure S3. Racemic chains of the ionic unit of Flx-SO4. The R- (in blue) and S-Flx (in red) are alternately arranged along the motif.
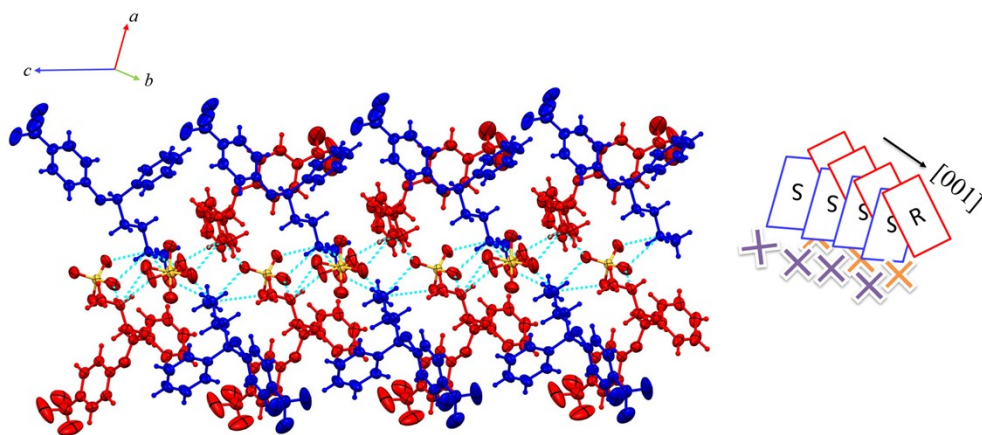
Figure S4. Crystal of Flx-BF4 obtained by (a) protonation of the racemic Flx free base or (b) AE methods.
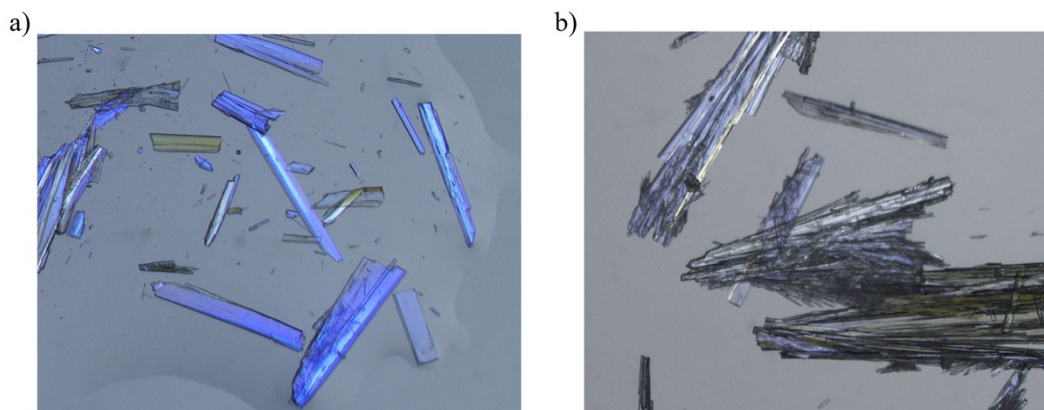
a)

b)

Figure S5. Diffractograms overlay for a freshly prepared sample of Flx-BF4 and from the solid residue remaining after the solubility study.
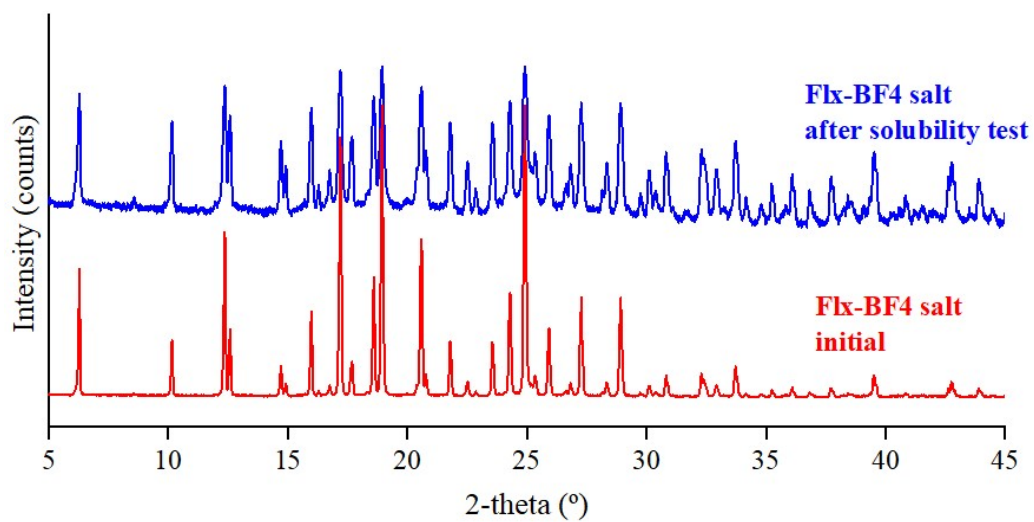
Figure S6. Integration of the Flx enantiomers (R- and S-) peaks using (a) automatic integration of Waters-Empower software, and (b) after deconvolution through the Origin program. In both integration strategies, the peak areas correspond to approximately 50%.

(a)

Component Summary Table
Name: R-Flx

	Name	RT	Area	% Area
1	R-Flx	10.463	6295916	51.14
Mean			6295916	
Std. Dev.				
% RSD				

Component Summary Table
Name: S-Flx

	Name	RT	Area	% Area
1	S-Flx	9.303	6014080	48.86
Mean			6014080	
Std. Dev.				
% RSD				

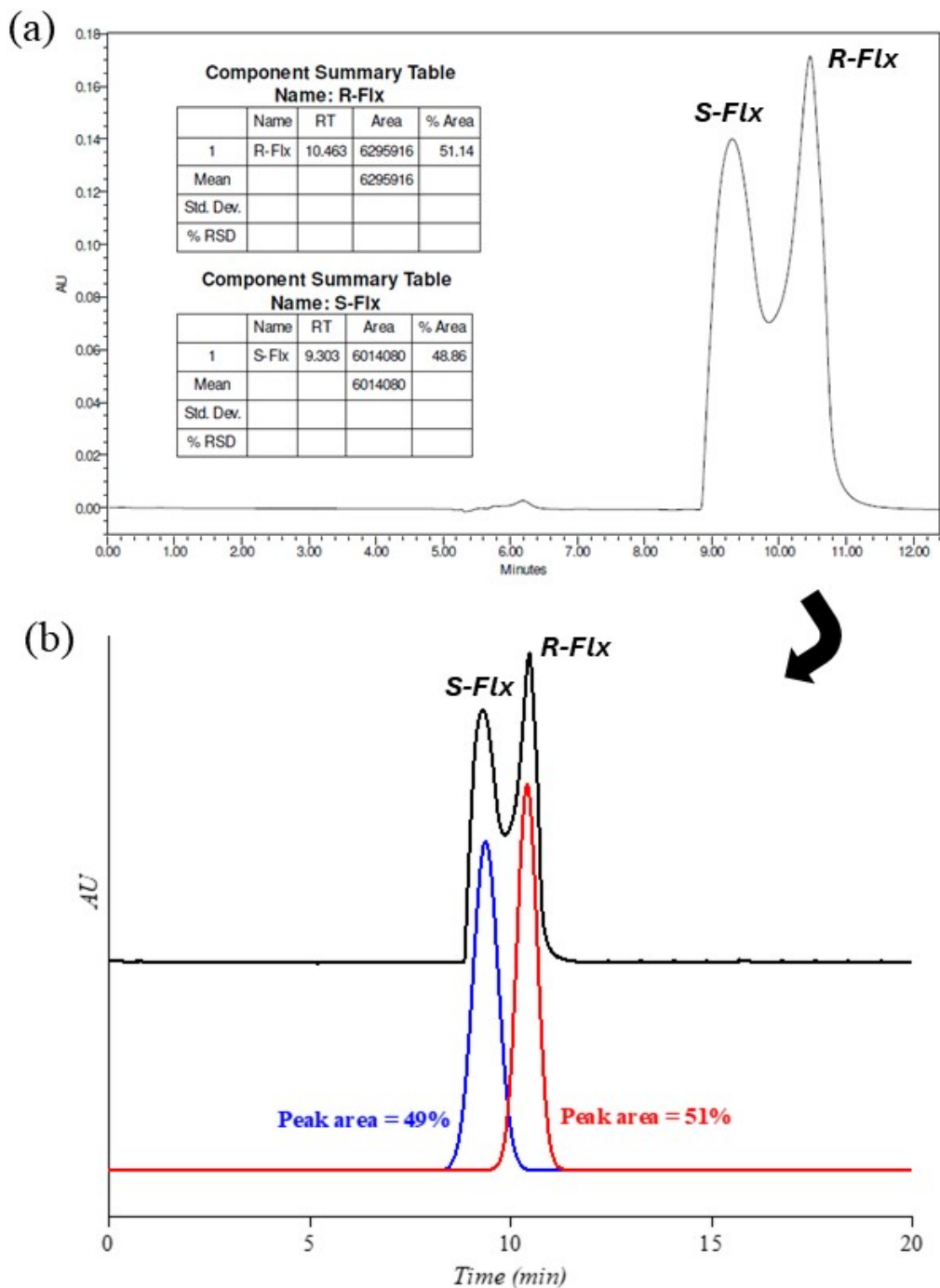(b)

Peak area = 49%

Peak area = 51%

Table 1. Crystallographic data of FlxBF4 collected using Cu Kα radiation (1.54184 Å). Data collection was collected at 100K on Bruker X8 Proteum diffractometer. 7

	Flx-BF4
Empirical formula	$C_{17}H_{19}BF_7NO$
Formula weight	397.14
Temperature/K	100.0
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
a/Å	6.0816(3)
b/Å	10.8836(5)
c/Å	27.6129(13)
Volume/Å^3	1827.69(15)
Z, Z'	4, 1
$\rho_{calc}g/cm^3$	1.443
μ/mm^{-1}	1.214
F(000)	816.0
Crystal size/mm^3	0.40 × 0.177 × 0.07
Radiation	CuKα (λ = 1.54178)
2Θ range for collection/°	6.402 to 144.594
Index ranges	-7 ≤ h ≤ 7
	-13 ≤ k ≤ 13
	-31 ≤ l ≤ 33
Reflections collected	65291
Independent reflections	3550 [R_{int} = 0.0316, R_{sigma} = 0.0113]
Data/restraints/parameters	3550/0/245
Goodness-of-fit on F^2	1.093
Final R indexes [I>=2σ (I)]	R_1 = 0.0409, wR_2 = 0.1006
Final R indexes [all data]	R_1 = 0.0428, wR_2 = 0.1040
Largest diff. peak/hole / e Å^{-3}	0.21/-0.27
Flack parameter	0.411(19)