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Supporting information for article:

Solvomorphs of tyraminium 5,5-diethylbarbiturate: a rare example of the barbiturate *R*3^3(12) hydrogen-bond motif and a crystal structure with *Z*′ = 4

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Annex

Solvomorphs of tyraminium 5,5-diethylbarbiturate: a rare example of the barbiturate $R_3^3(12)$ ring motif and a crystal structure with Z' = 4

Table A1 The crystal data for (II) before SQUEEZE was applied is as followed:

	(II) before SQUEEZE
Crystal data	
Chemical formula	$C_{16.20}H_{23.73}N_3O_{4.17}$ (with disordered solvent 0.100 C_2H_6O·0.066 H_2O)
M_r	327.18
Crystal system, space group	Trigonal, $R\bar{3}c$:*H*
Temperature (K)	120
a, *c* (Å)	17.3372 (3), 62.3663 (12)
V (Å^3)	16234.5 (6)
Z	36
Radiation type	Cu *K*α
μ (mm^{-1})	0.72
Crystal size (mm)	0.60 × 0.35 × 0.05
Data collection	
Diffractometer	Rigaku Oxford Diffraction SuperNova Dual Source diffractometer with an Atlas detector
Absorption correction	Multi-scan *CrysAlis PRO* 1.171.38.34a (Rigaku Oxford Diffraction, 2015) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
T_{min}, T_{max}	0.762, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	104082, 3829, 3432
R_{int}	0.069
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.633
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.057, 0.185, 1.07
No. of reflections	3829
No. of parameters	266
No. of restraints	0
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
	$w = 1/[\sigma^2(F_o^2) + (0.1283P)^2 + 19.3147P]$ where $P = (F_o^2 + 2F_c^2)/3$
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.30, −0.31

Optical Indicatrix of (II)

Optical character and optical activity of (II) single crystals were determined using Zeiss Axio Scope.A1 polarizing microscope. Conoscopic image (Figure A1a) demonstrates uniaxial interference figure of (II) crystal approximately along [001]. Figure A1b, c and d present reactions with accessory plates: gypsum,

mica and quartz wedge, respectively. Characteristic blue hue in negative quadrants (Figure A1b) and black dots in positive ones (Figure A1c) definitely proved the negative optical character of (II) crystals.

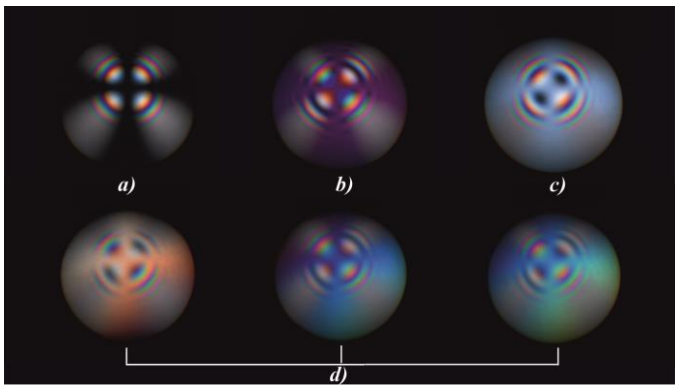

Figure A1 a) Conoscopic image of (II) crystal oriented almost along to the optical axis; the reaction of the crystal with the gypsum plate, the mica plate and the quartz wedge (b, c and d, respectively) showing uniaxial interference figures and negative optical character of (II).

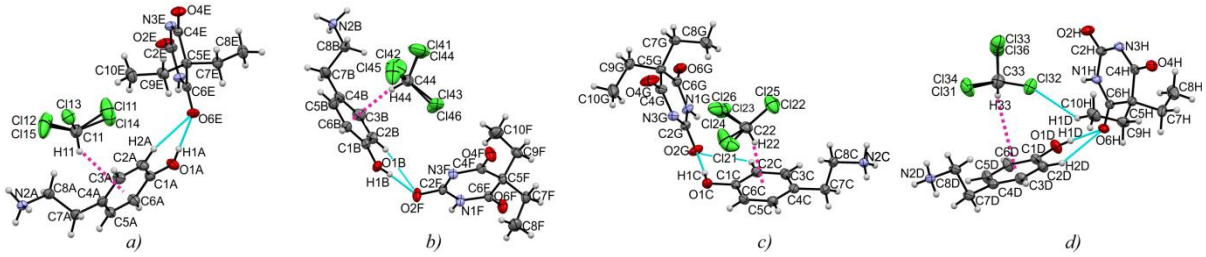

Figure A2 Four independent moieties of tyraminium barbitalate ions and chloroform molecules in the asymmetric unit of (I) (a-d).

Table A2 The geometry of C-H···π interactions (Å,°) for (I).

C – H···π	D – H	H – A	D – A	∢D – H···A
C11-H11···Cg(C1A^C6A)	0.93	3.23	3.890	130
C22-H22··· Cg(C1C^C6C)	0.93	2.54	3.447	165
C33-H33··· Cg(C1D^C6D)	0.97	3.57	4.177	123
C44-H44··· Cg(C1B^C6B)	0.98	2.49	3.449	168

C6A-H6A··· Cg(C1B^C6B)	0.95	3.05	3.730	130
C2D-H2D··· Cg(C1C^C6C)	0.95	3.08	3.774	131

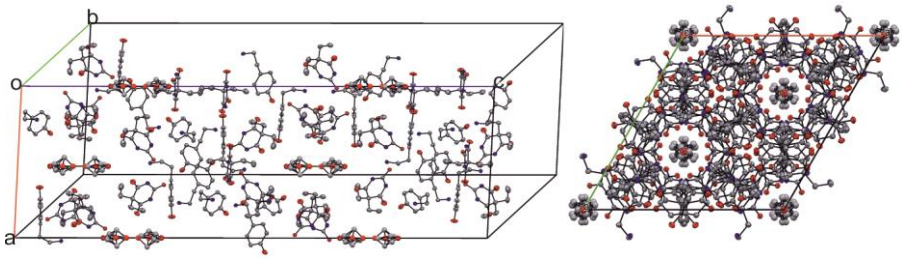

Figure A3 Packing of structural components in (II) with disordered solvent molecules located in cavities (left – general view, right – projection along [001]).

Table A3 Hydrogen-bond geometry (Å,°) for (II).

C-H···π	D-H (Å)	H···A (Å)	D-A (Å)	D-H-A (°)
C7B-H7B1···Cg(C1B-^C6B)	0.98	2.51	3.434	156
C10A-H10B··· Cg(C1B-^C6B)	0.98	3.28	4.095	141

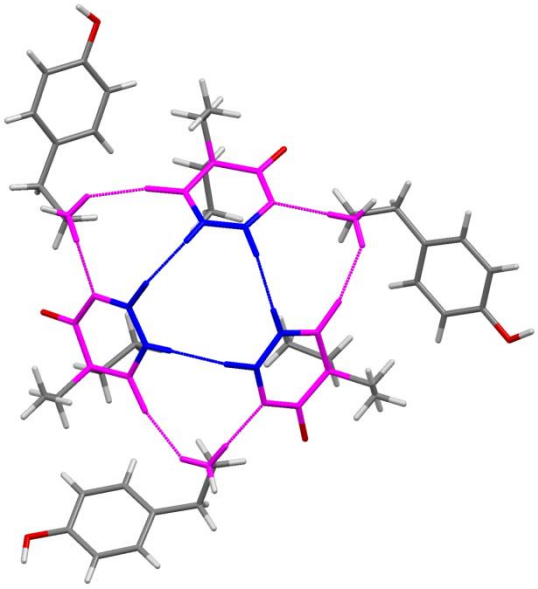

Figure A4 $R_3^3(12)$ and $R_6^6(24)$ ring motifs in (II).