crystals

MDPI

Supplementary Materials

Salification Controls the In-Vitro Release of Theophylline

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Crystal data	
Empirical formula	$C_{11}H_{16}N_4O_9$
Formula weight	348.28
Temperature/K	150.0
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	14.8686(7)
b/Å	15.1485(7)
c/Å	6.5200(3)
α/°	90
β/°	97.598(4)
γ/°	90
Volume/Å^3	1455.65(12)
Z	4
ϱ_{calc}g/cm^3	1.589
μ/mm^{-1}	1.217
F(000)	728.0
Crystal size/mm^3	0.21 × 0.11 × 0.05
Radiation	CuKα (λ = 1.54184 Å)
2Θ range for data collection/°	5.834 to 141.292
Index ranges	$-18 \le h \le 18$, $-18 \le k \le 18$, $-7 \le l \le 7$
Reflections collected	27139
Independent reflections	2695 [R_{int} = 0.1182, R_{sigma} = 0.0519]
Data/restraints/parameters	2695/1/256
Goodness-of-fit on F^2	1.054
Final R indexes [I>=2σ (I)]	R_1 = 0.0790, wR_2 = 0.1973
Largest diff. peak/hole/e Å^{-3}	0.49/−0.37

Table S1. Summary of crystallographic data for Theophylline Squarate trihydrate (**TS3w**).

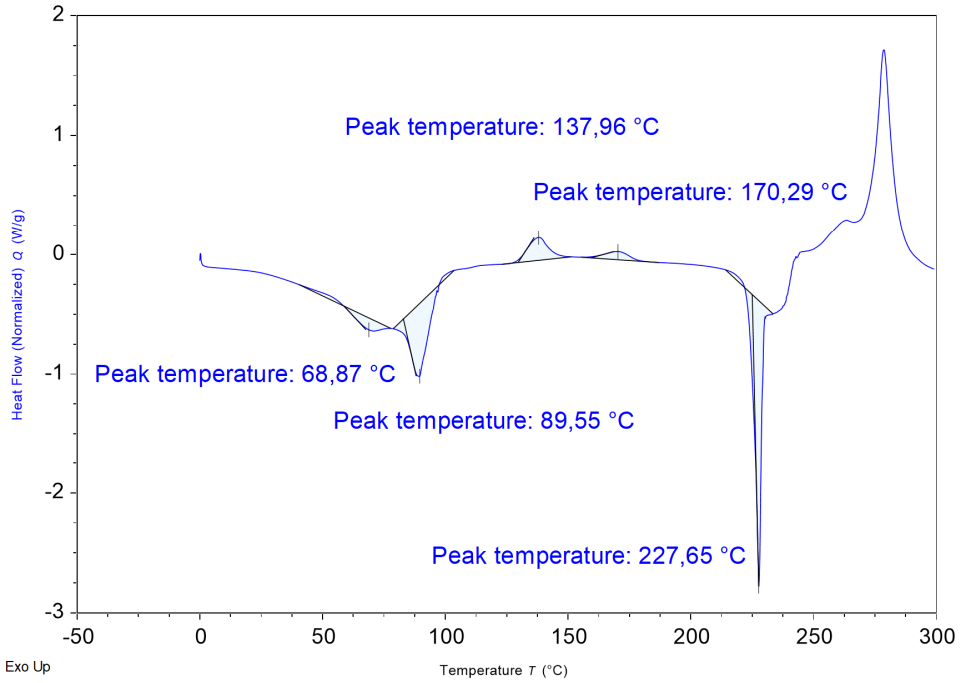

Figure S1. DSC thermogram of **TS3w** (10°C/min).

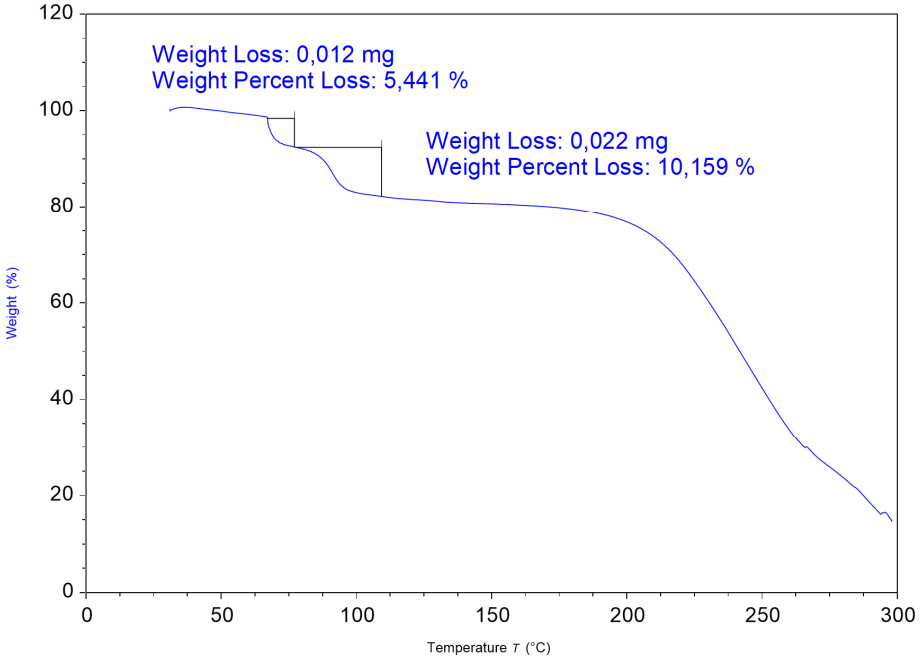

Figure S2. TGA thermogram of **TS3w** (10°C/min).

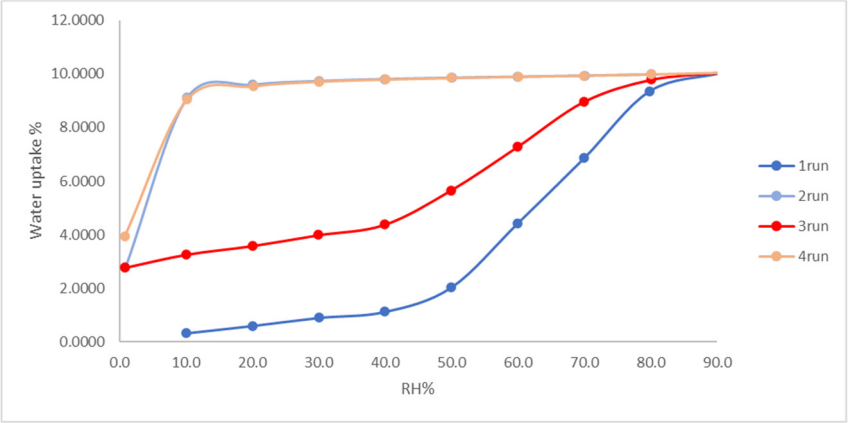

Figure S3. DVS of **TS3w**. The hysteresis represents the gain and the loss of the channel water molecule.

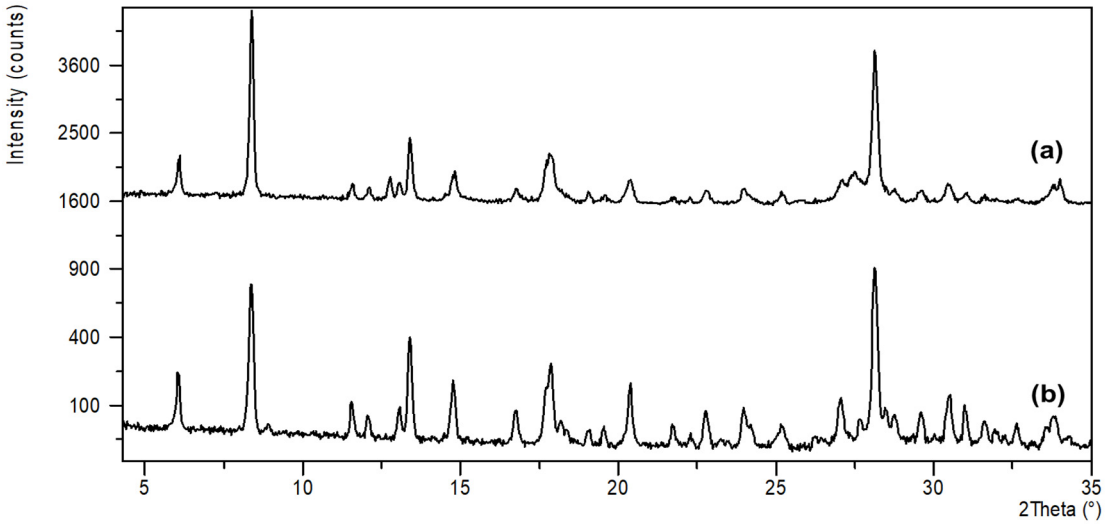

Figure S4. XRPD of TS3w after DVS (**a**) compared to its pattern before DVS (**b**).

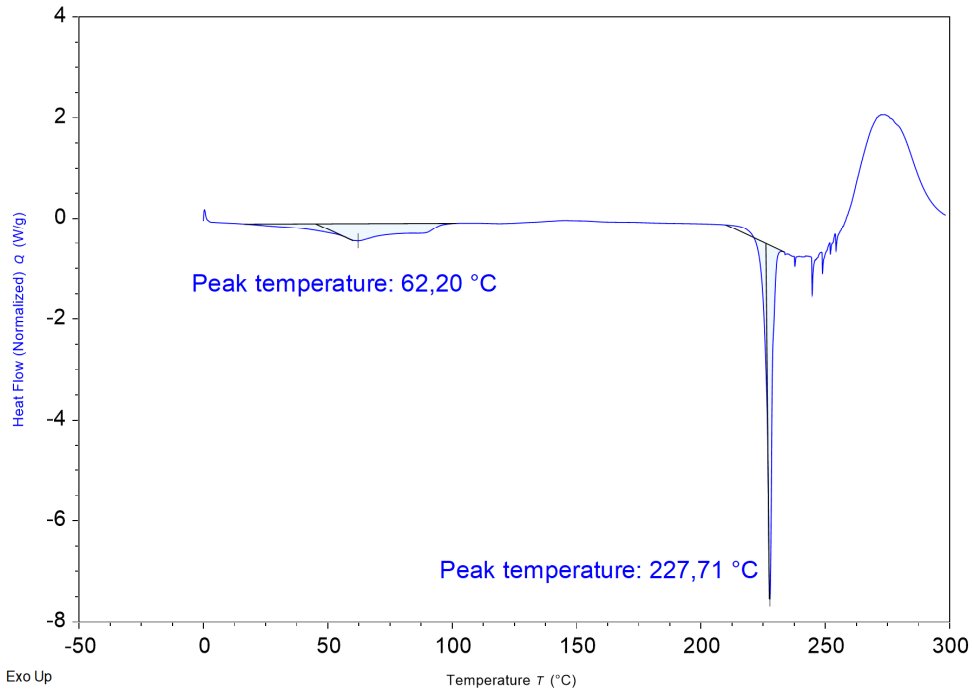

Figure S5. DSC thermogram of **TSan** (10°C/min).

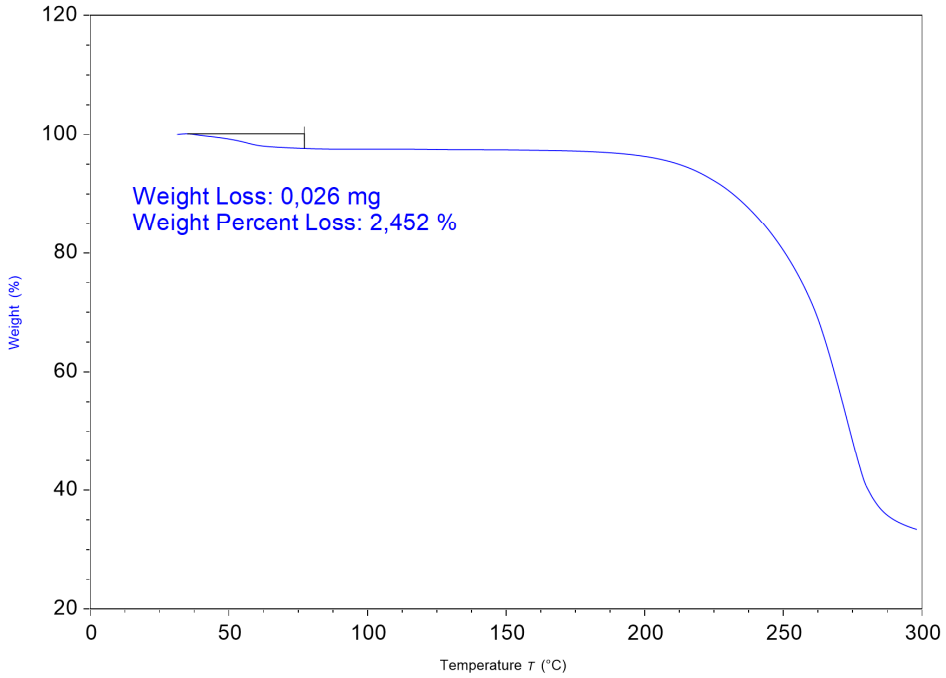

Figure S6. TGA thermogram of **TSan** (10°C/min).

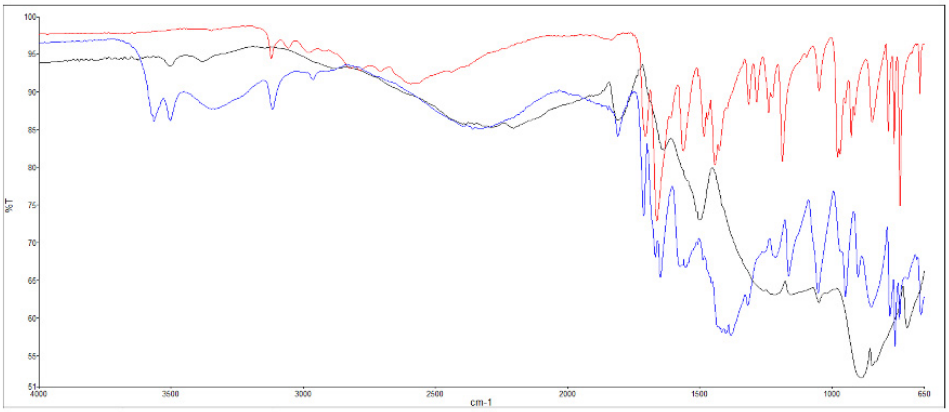

Figure S7. FTIR spectra of Theophylline (red), Squaric Acid (black) and Theophylline squarate trihydrate (blue).

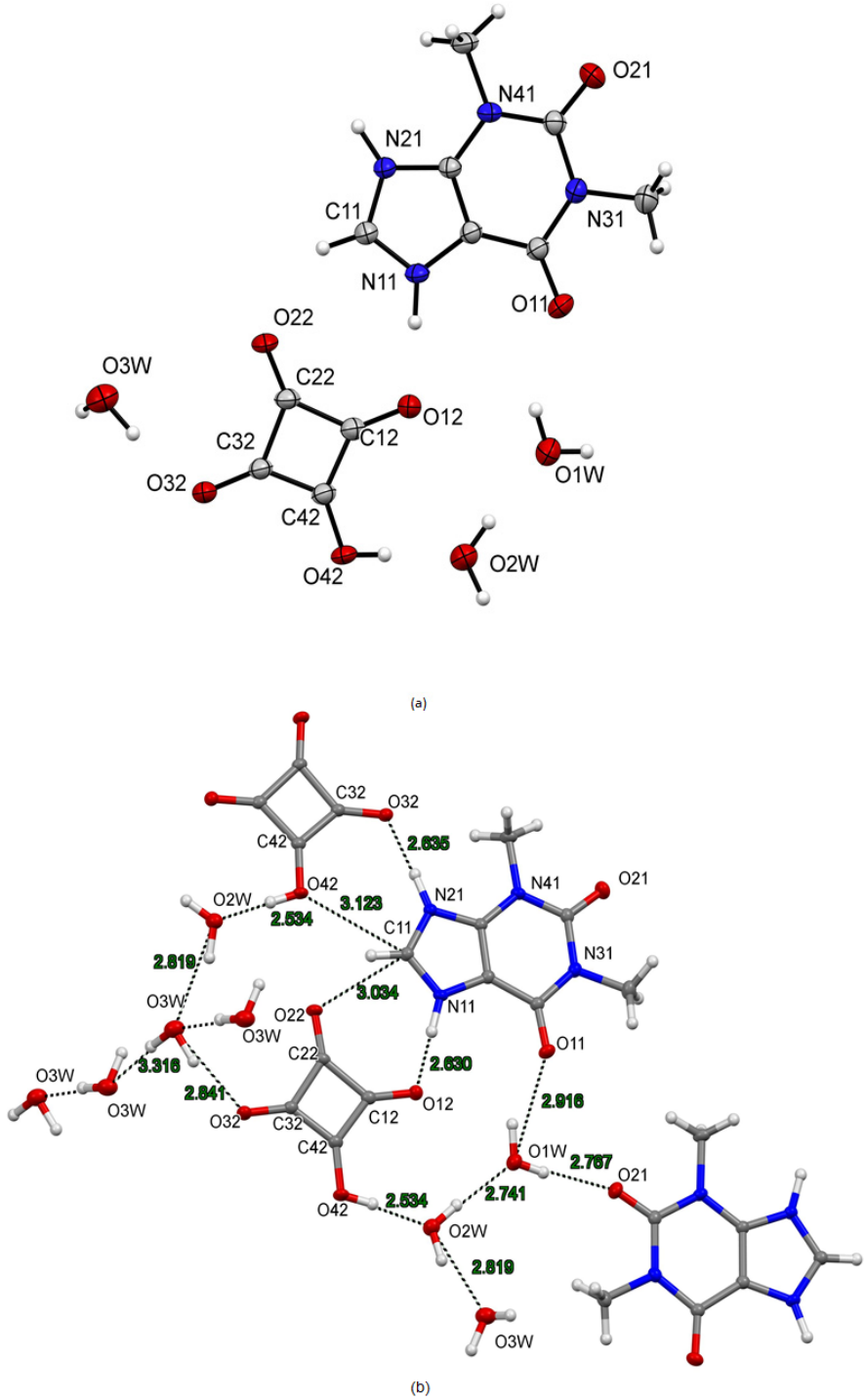

Figure S8. Asymmetric unit of Theophylline squarate trihydrate (**a**) and molecular structure highlighting the HB connections and distances (**b**).

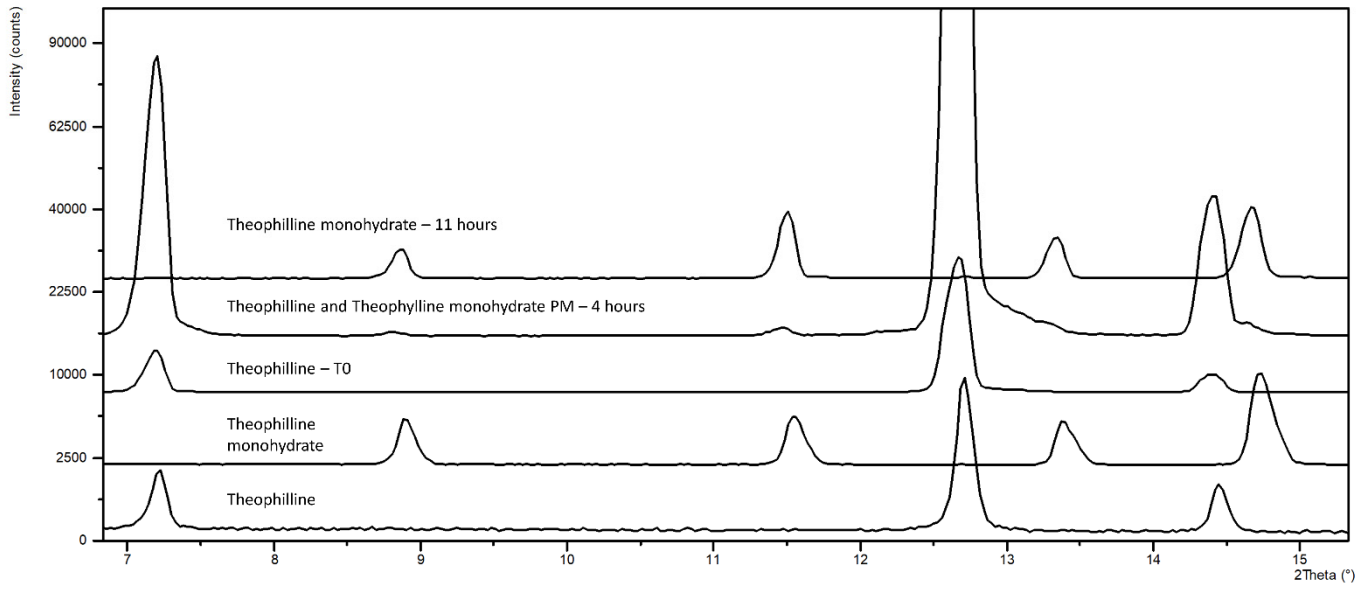

Figure S9. VH-XRPD of pure Theophylline.