Thiocarbamoyl disulfides as inhibitors of urease and ammonia monooxygenase: crystal engineering for novel materials

SUPPORTING INFORMATION (8 pages)

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[**Note**: In the following β-CD, β-CD·TMTD, $(\beta\text{-CD})_2$·TETD, and $(\beta\text{-CD})_2$·TIPTD stay for β-CD·$8H_2O$, β-CD·TMTD·$5H_2O$, $(\beta\text{-CD})_2$·TETD·$14H_2O$ and $(\beta\text{-CD})_2$·TIPTD·$12H_2O$, respectively.]

Single Crystal X-ray Diffraction

Table S1. Crystal data and details of measurement for TIPTD Form II.

	TIPTD Form II
Chemical formula	C14 H28 N2 S4
M_r, g mol^{-1}	352.62
T / K	298
Morphology, colour	Block, colourless
Crystal system	Monoclinic
Space group	P $2_1/c$
a / Å	12.152(1)
b / Å	13.1870(6)
c / Å	14.0049(13)
α / °	90
β / °	114.146(11)
γ / °	90
V / $Å^3$	2047.9(3)
Z	4
d / mg cm^{-3}	1.144
μ / mm^{-1}	0.458
Reflections collected/unique	9323/4701
R_{int}	0.0423
Threshold expression	> 2σ(I)
R_1 (obs)	0.0698
wR_2 (all)	0.2461

Powder X-ray Diffraction

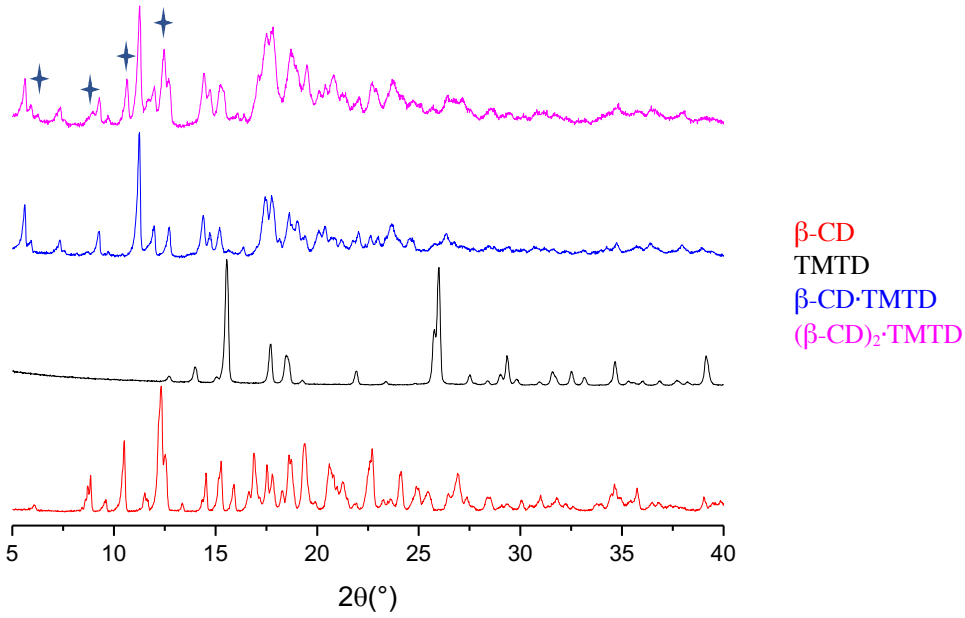


Figure S1: comparison between the reagents β-CD (red line) and TMTD (black line) with the product of the 1:1 (blue line) and the 2:1 (pink line) mechanochemical reactions. The presence of extra-peaks in $(\beta\text{-CD})_2$·TMTD is due to unreacted β-CD, thus confirming the 1:1 stoichiometry of the complex.

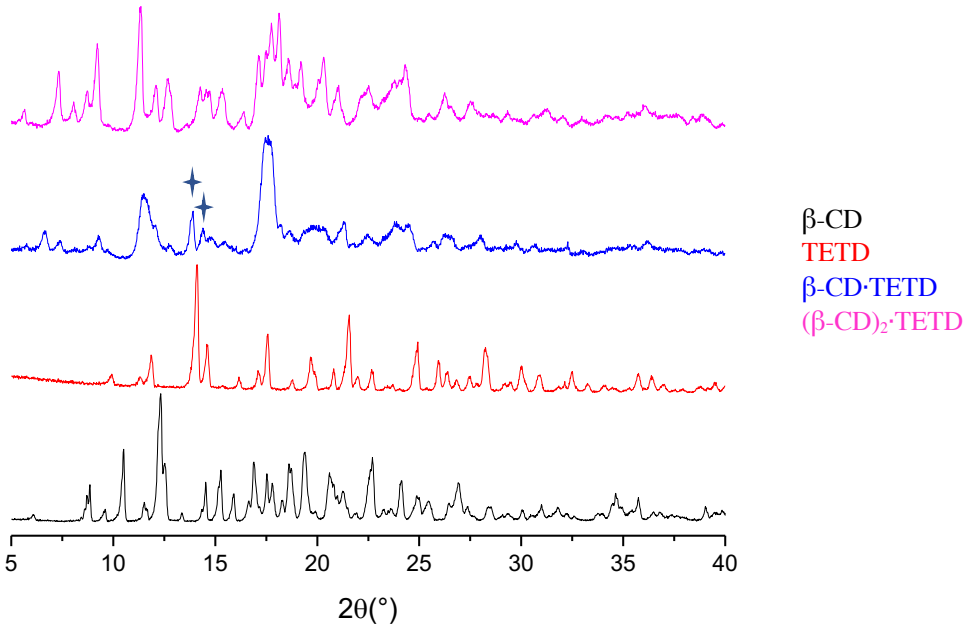


Figure S2: comparison between the reagents β-CD (red line) and TETD (black line) with the product of the 1:1 (blue line) and the 2:1 (pink line) mechanochemical reactions. The presence of extra-peaks in β-CD·TETD is due to unreacted TETD, thus confirming the 2:1 stoichiometry of the complex.

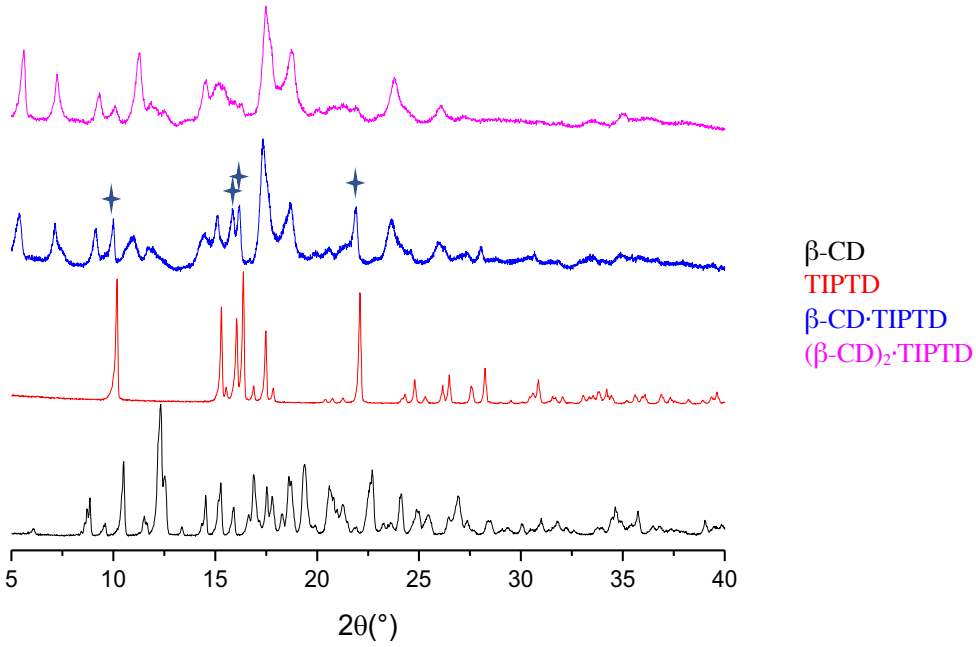


Figure S3: comparison between the reagents β-CD (black line) and TIPTD (red line line) with the product of the 1:1 (blue line) and the 2:1 (pink line) mechanochemical reactions. The presence of extra-peaks in β-CD·TIPTD is due to unreacted TIPTD, thus confirming the 2:1 stoichiometry of the complex.

Pawley Refinement

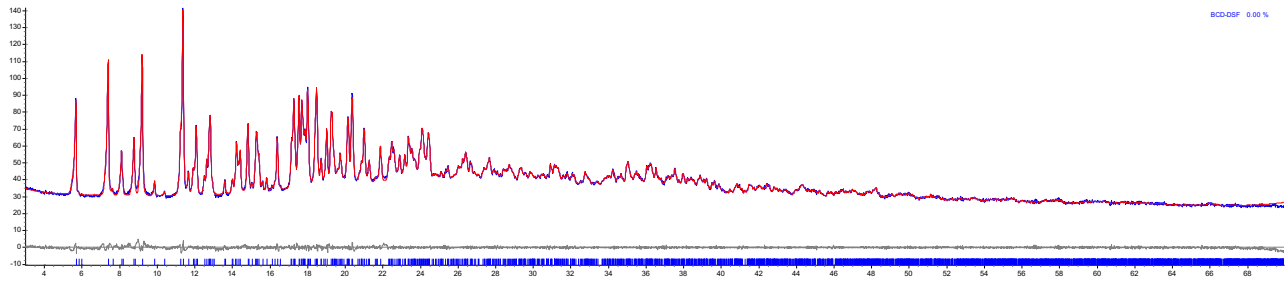
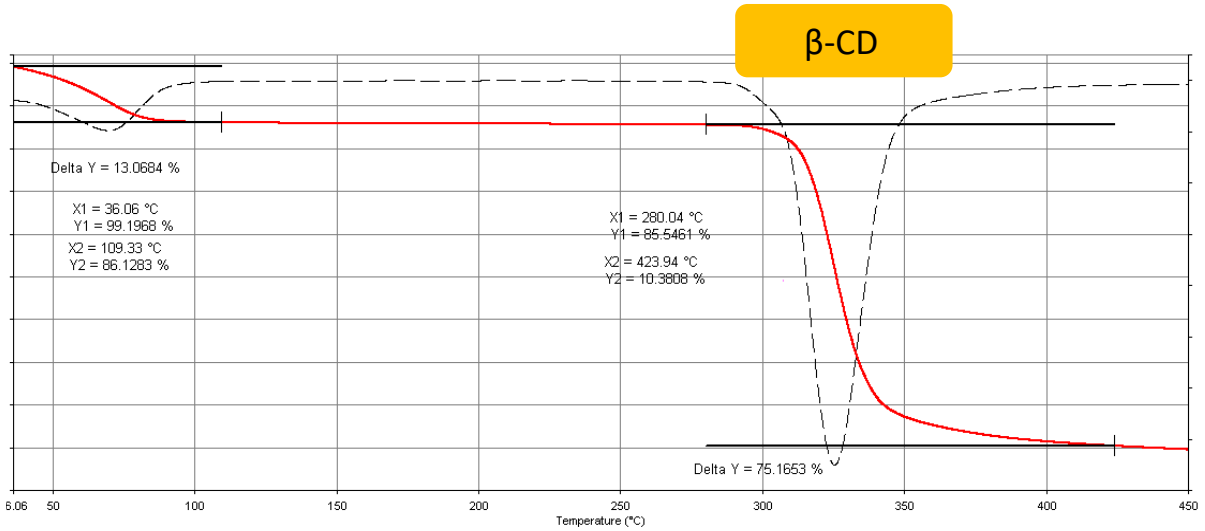


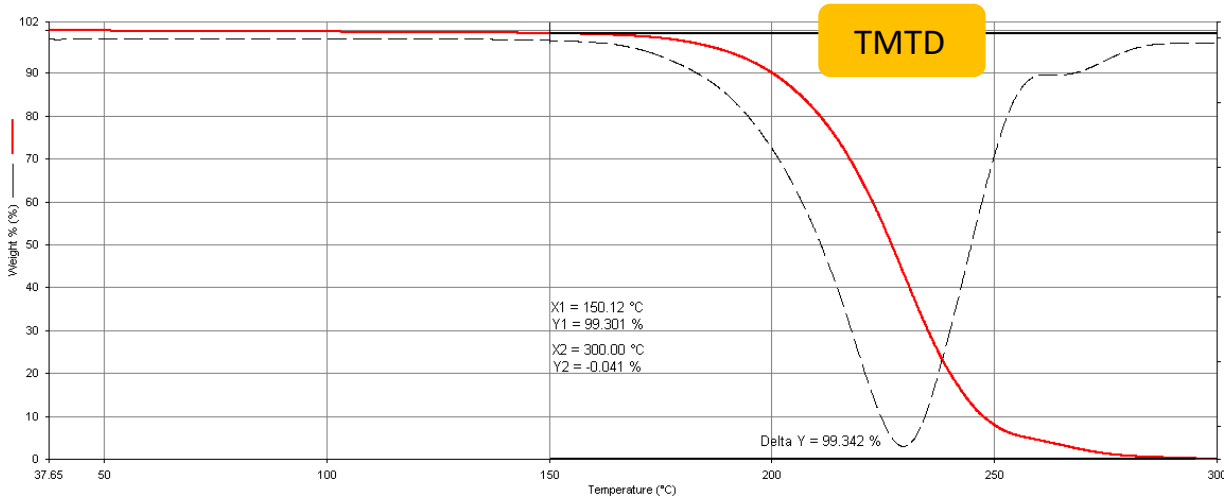
Figure S4: Pawley refinement on the experimental pattern of $(\beta\text{-CD})_2$·TETD.

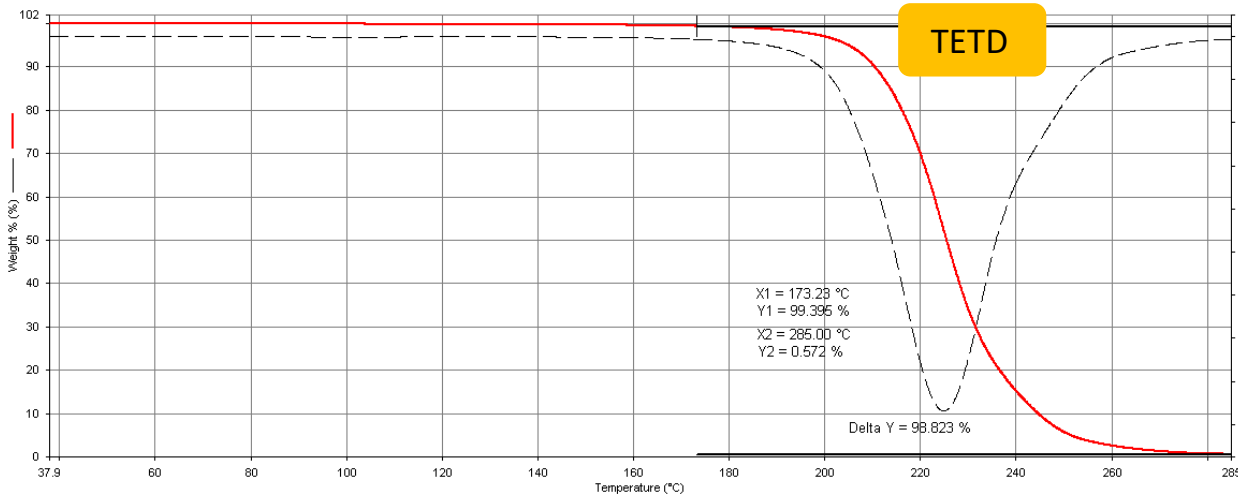
Table S2: Pawley refinement data

S.G.	a (Å)	b (Å)	c (Å)	α (°)	β (°)	γ (°)	V $(Å)^3$	**Rwp**
P1	15.282492	15.531621	15.711671	87.68418	81.52082	77.38594	3599.427	3.164

TGA (Thermogravimetric Analysis)







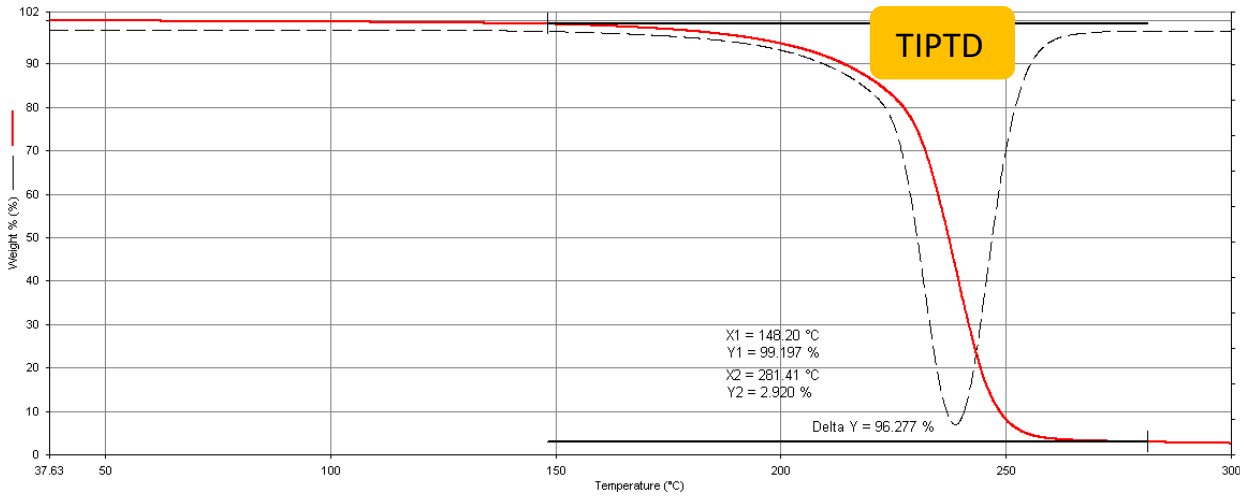
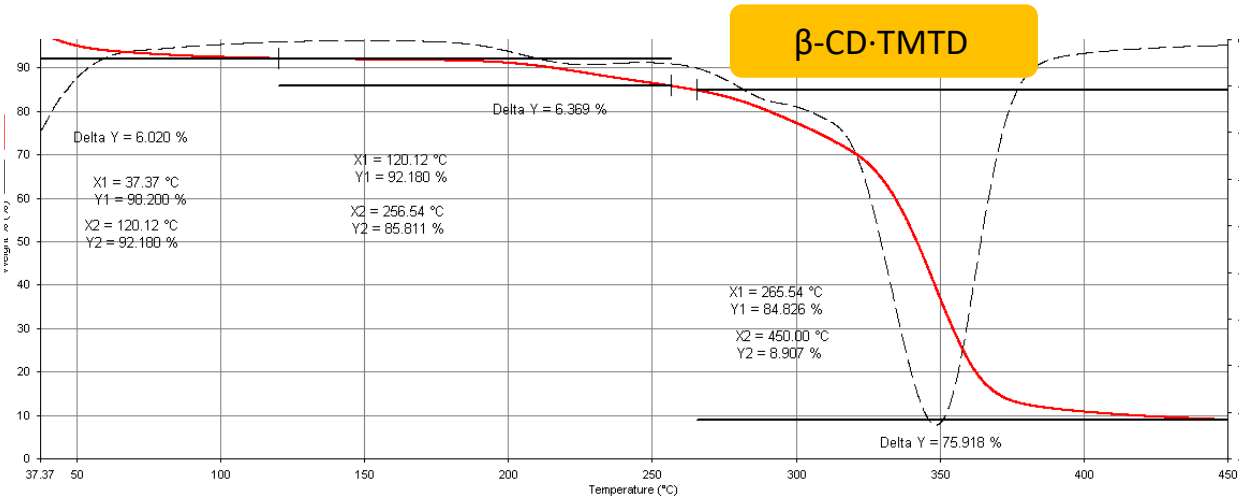
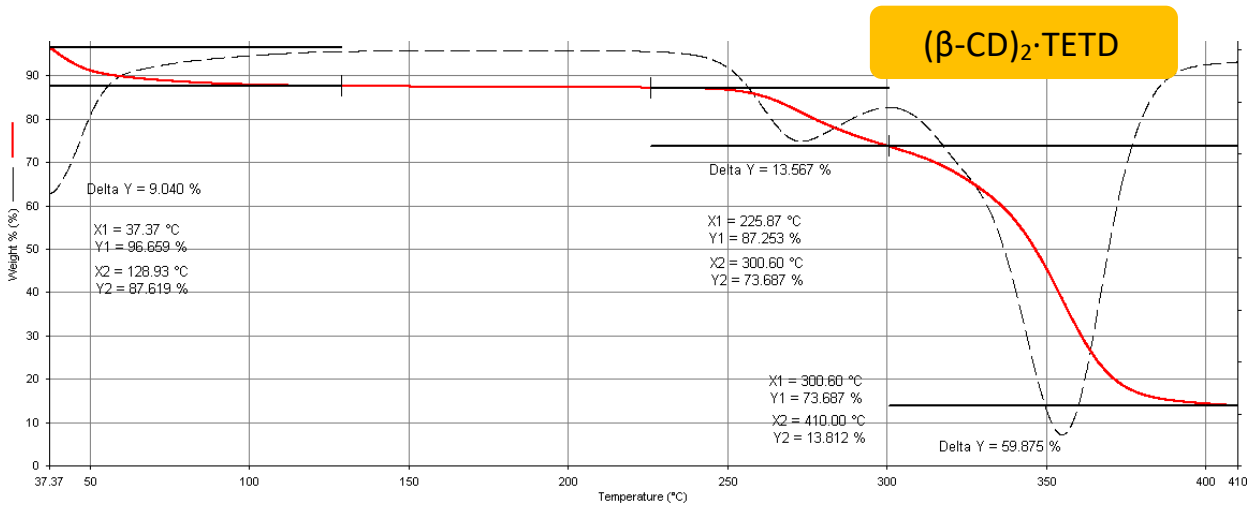


Figure S5: TGA traces of the reagents (from top to bottom): β-CD, TMTD, TETD and TIPTD.





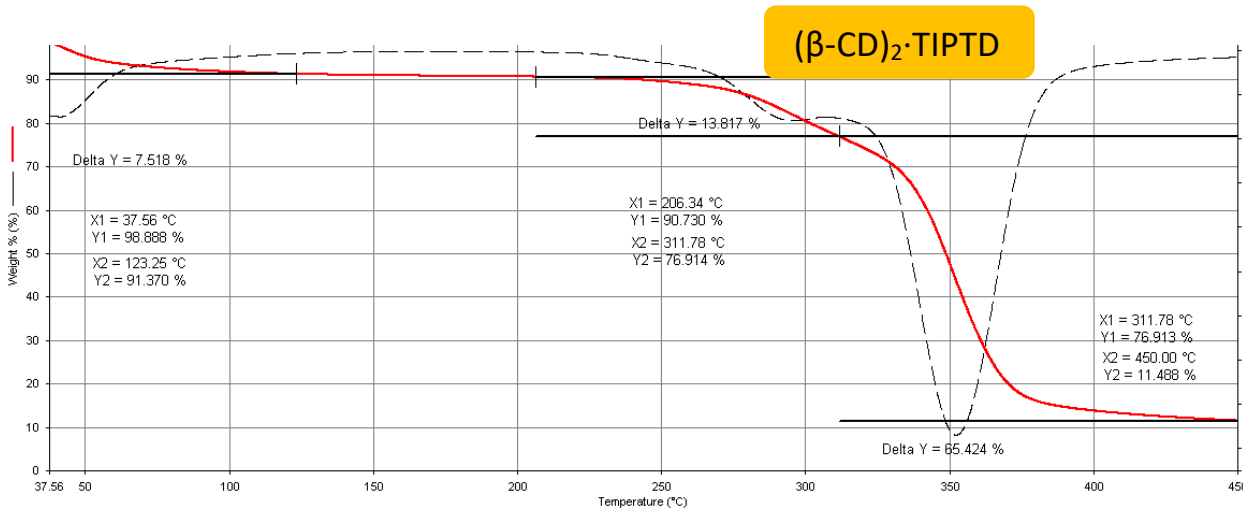


Figure S6: TGA traces of the complexes. From top to bottom: β-CD·TMTD, $(\beta\text{-CD})_2$·TETD and $(\beta\text{-CD})_2$·TIPTD.

DSC trace for TIPTD

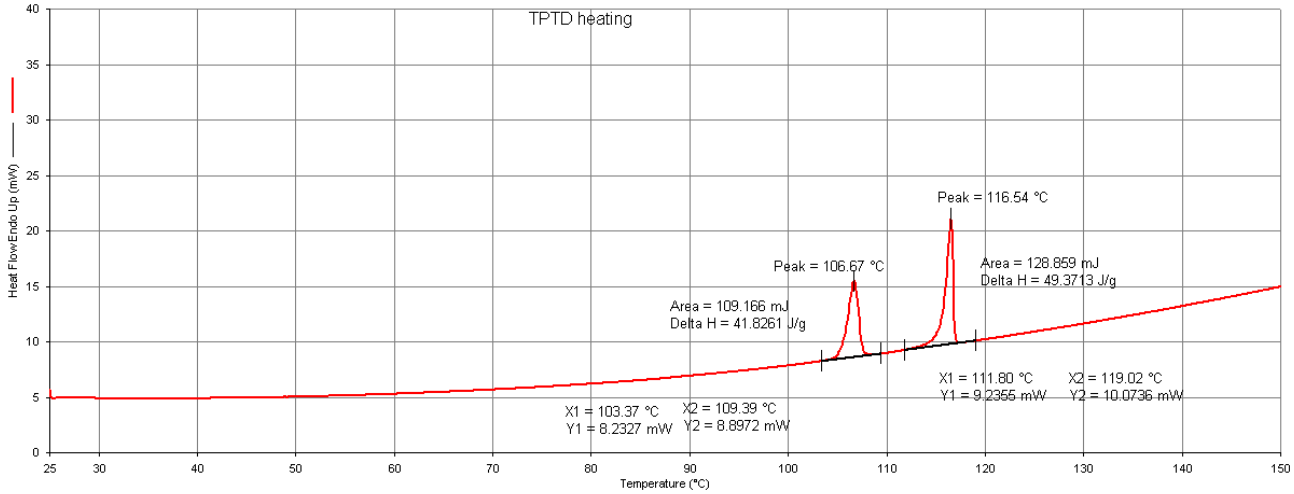


Figure S7: DSC trace for TIPTD, showing the phase transition to Form II at ca. 107 °C, before melting at ca. 117 °C.

Solubility data

First repeat

	Water (mL)	Solute (mg)	Solute (mol)	M_Thiuram (mg)	Solubility (mg/L)	Solubility (mol/L)
β-CD·TMTD	60	14.3	9.8797E-06	2.3754	39.5901	1.6466E-04
(β-CD)$_2$·TETD	52	9.9	3.5125E-06	1.0416	20.0306	6.7548E-05
(β-CD)$_2$·TIPTD	55	2.6	9.2178E-07	3.2507	5.9103	1.6760E-05

Second repeat

	Water (mL)	Solute (mg)	Solute (mol)	M_Thiuram (mg)	Solubility (mg/L)	Solubility (mol/L)
β-CD·TMTD	60	13	8.9815E-06	2.1594	35.9908	1.4969E-04
(β-CD)$_2$·TETD	55	9	3.1932E-06	9.4691	17.2165	5.8058E-05
(β-CD)$_2$·TIPTD	60	3	1.0636E-06	3.7508	6.2513	1.7727E-05

Third repeat

	Water (mL)	Solute (mg)	Solute(mol)	M(Thiuram/mg)	Solubility(mg/L)	Solubility(mol/L)
β-CD·TMTD	60	12.7	8.7743E-06	2.1096	35.1605	1.4624E-04
(β-CD)$_2$·TETD	65	9.5	3.3706E-06	9.9951	15.3771	5.1855E-05
(β-CD)$_2$·TIPTD	60	2.8	9.9269E-07	3.5007	5.8345	1.6545E-05

Solubility average values (RMS error in parenthesis)

	Solubility (mg/L)	Solubility (mol/L)
β-CD·TMTD	37(2)	1.537E-04
(β-CD)$_2$·TETD	18(2)	5.917E-05
(β-CD)$_2$·TIPTD	6.0(2)	1.700E-05

As specified in the introduction in main text, for sake of conciseness the formulae β-CD·TMTD, (β-CD)$_2$·TETD, and (β-CD)$_2$·TIPTD stay for β-CD·TMTD·5H$_2$O, (β-CD)$_2$·TETD·14H$_2$O, and (β-CD)$_2$·TIPTD·12H$_2$O, respectively.