

**Studies on Polymorph Conversion in a New Cyclodextrin
Inclusion Compound**

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Electronic Supplementary Information

Thermogravimetric Studies

The crystals of fraction B of β CD·PPNO were studied by thermogravimetric analysis (TGA) and compared to the profiles of β CD, PPNO and an equimolar physical mixture of β CD and PPNO, prepared by combining the two components and gently mixing with a spatula (no pressure added to the system). The results are depicted in the Figure S1.

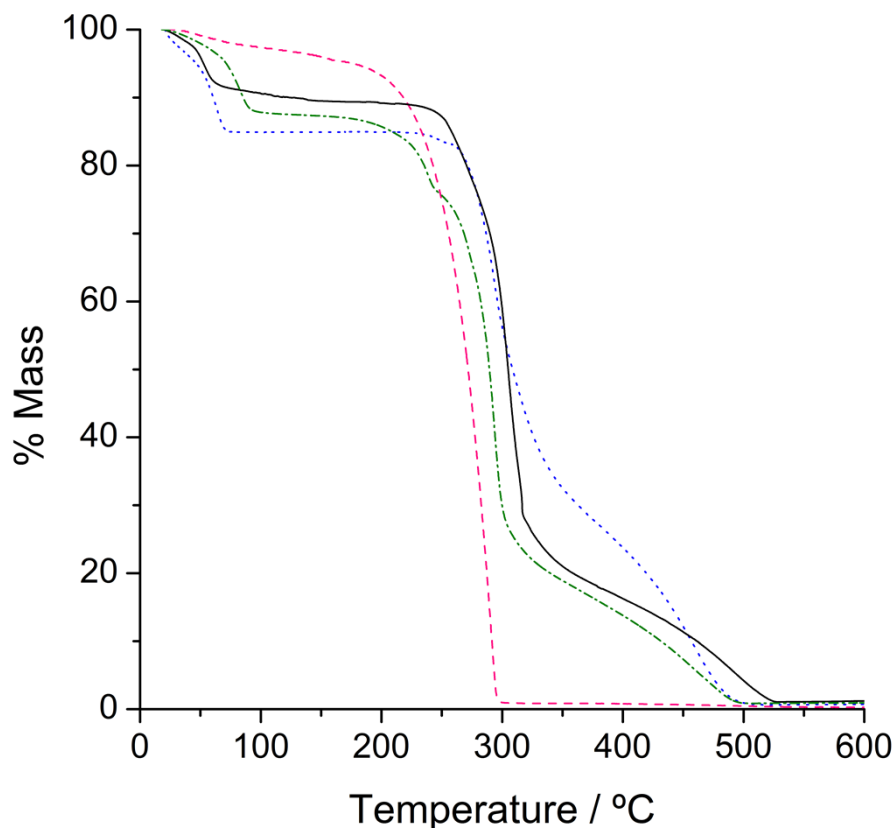


Figure S1. Thermogravimetric traces for fraction B (—), β CD (- - -), PPNO (- - -) and their 1 : 1 physical mixture (· · ·).

The first representative feature is water loss, occurring between ambient temperature and 120 °C. Pure β CD hydrate exhibits a 14% dehydration step, corresponding to 10 hydration waters *per* CD molecule; water loss (around 13%) is slightly reduced in the 1 : 1 physical mixture, and even lower in β CD·PPNO. Furthermore, mass loss from dehydration of the inclusion compound is observed up to 140 °C, with a total step of *ca.* 10%, corresponding to 8 hydration water molecules *per* adduct. In the second stage (120–300 °C) a plateau region appears and no further mass loss can be seen in the TG curves; in the mix, there is the step ascribed to PPNO evaporation at 200 °C.

The thermal degradation of the hosts becomes remarkable over 280 °C, with an abrupt mass loss in both the mix and β CD·PPNO. Degradation is believed to start in the solid phase and to comprise a simultaneous melting/decomposition effect.¹

Spectroscopic Studies

Vibrational Spectroscopy

The solid products of ten different fractions of β CD·PPNO (A or B – see main text for details) were studied by infrared spectroscopy. The full FT-IR spectra for PPNO, β CD and β CD·PPNO are depicted in the Figure S2 (on the next page). Even though this technique does not allow distinguishing material belonging to fraction A from that belonging to fraction B, it has helped us to confirm inclusion by the observation of vibrational bands typically assigned to the PPNO. Some of these had their maxima slightly shifted in comparison to those of pure PPNO, as listed in Table S1. These changes are coherent with the inclusion of PPNO into the cavity of β CD.

Table S1. Relevant ATR-FTIR bands for β CD·PPNO (in cm^{-1}).

PPNO	β CD·PPNO	Approximate description ²
1471, 1427	1475, 1423	ν Ph, ν Py
1340	1330	δ Ph, ν CN
1290, 1234	1299, 1232	δ Ph, ν Py-Ph, ν NO
1182	Not observed	δ Py
931	935	γ CH(Ph)
842	844	γ CH(Py)

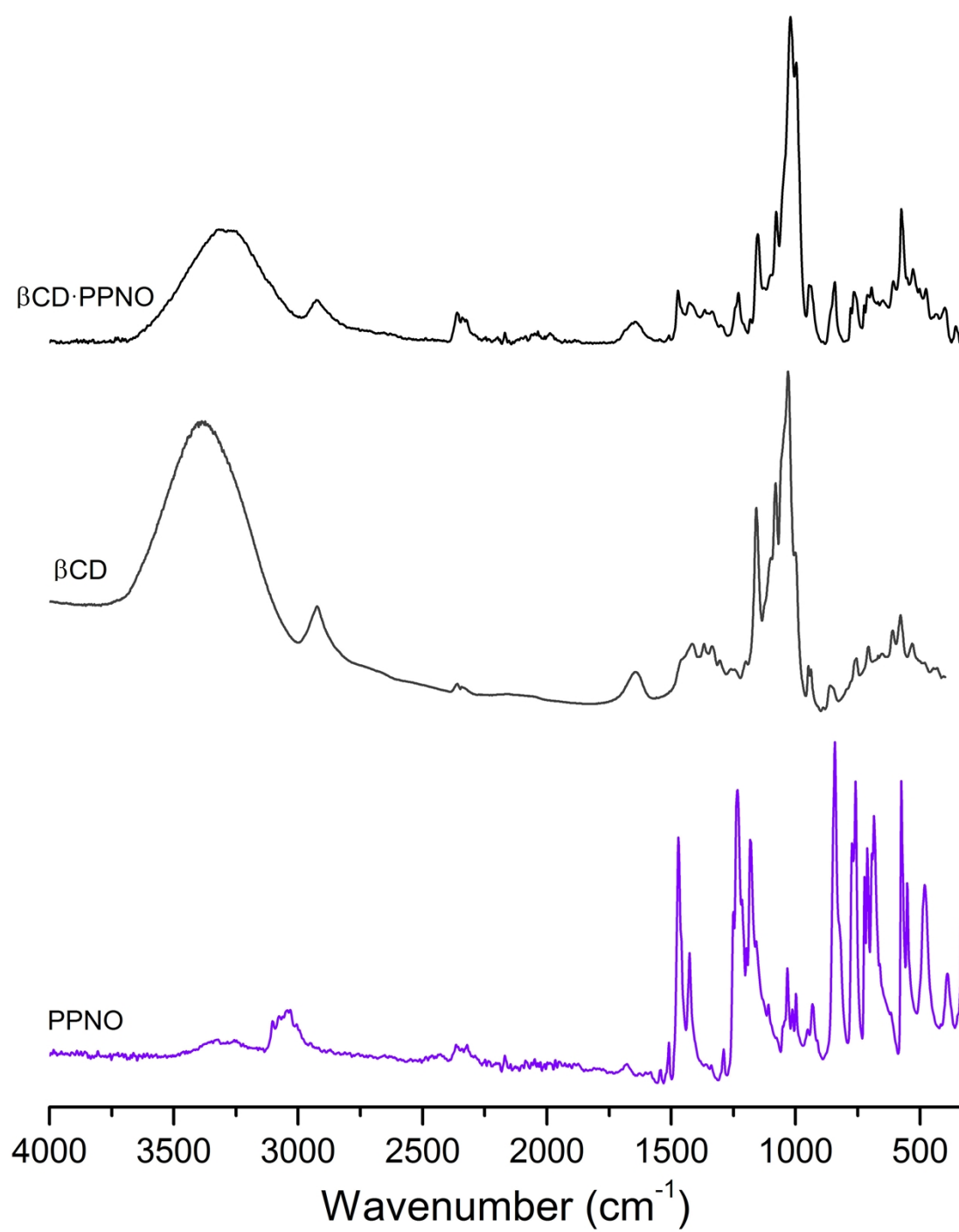


Figure S2. Fourier-transform Infrared spectra for PPNO, βCD and $\beta\text{CD}\cdot\text{PPNO}$, collected as KBR pellets in the 4000-300 cm^{-1} range.

References

1. Z. Éhen, F. Giordano, J. Sztatiz, L. Jicsinszky and C. Novák, *J. Therm. Anal. Calorim.*, 2005, **80**, 419-424.
2. K. Nakamoto, *Infrared and Raman spectra of inorganic and coordination compounds*, 5th edn., Wiley-Interscience Publications, New York, 1997.