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Supporting information for article:

Controlled usage of H/D-exchange to circumvent concomitant polymorphs of ROY

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S1. Synthesis of d₁-ROY

d₁-ROY was synthesized *via* a deuterated solvent reflux reaction. A saturated solution at RT of ROY in d₄-methanol, d₆-ethanol or d₈-1-propanol was stirred and heated under reflux for 30 minutes. The solution was slowly cooled down to -7 °C. The product was obtained as a yellow crystalline product. IR (ν [cm⁻¹]): 2442, 2230, 1613, 1568, 1552, 1364, 1228, 1263, 930, 839.

S2. Temperature Screening

ROY and d₁-ROY were dissolved until saturation in d₆-methanol at temperatures between RT and 60 °C and subsequently the solution was slowly cooled down to -7 °C. For comparative reasons, a temperature screening of ROY was carried out similarly. To reproduce the results of the crystallization investigations, the experiments were repeated. The obtained crystalline materials were characterized by powder X-Ray diffraction (PXRD), measured on a Bruker AXS Advance diffractometer in flat mode and Bragg-Brentano geometry using filtered CuK α radiation.

Table S1. Crystallographic data of the Y polymorph of d₁-ROY and ROY.

	d ₁ -ROY Y polymorph	ROY Y polymorph (Yu <i>et al.</i> , 2000)
identification code	CCDC 1828199	CCDC 1241884
empirical formula	C ₁₂ H ₈ D ₁ N ₃ O ₂ S ₁	C ₁₂ H ₉ N ₃ O ₂ S ₁
formula weight	260.28	259.28
Temperature	170	273
crystalline system	monoclinic	monoclinic
space group	<i>P2₁/n</i>	<i>P2₁/n</i>
description	yellow prism	yellow prism
<i>a</i> [Å]	8.537(5)	8.5001(1)
<i>b</i> [Å]	16.466(5)	16.413(2)
<i>c</i> [Å]	8.569(5)	8.5371(1)
β [°]	91.905(5)	91.767(7)
<i>V</i> [Å ³]	1203.9(11)	1190.5(4)
<i>Z</i>	4	4
<i>D</i> _{calcd} , [g/cm ³]	1.407	1.447
<i>F</i> (000)	536	536
2θ _{max}	50	48
reflections measured	14600	2095
Unique reflections	2837	1956
parameters	199	167
<i>RI</i> with $I > 2\sigma(I)$	0.0400	0.033
<i>wR2</i> for all reflections	0.0829	0.044
GOF	0.761	1.415

Figure S1. Temperature crystallization screening of ROY from methanol at different temperatures and fast cooling down to -7°C

