Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information - Cadmium phenylphosphonates: preparation, characterisation and *in situ* investigation

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Table S1: Selected bond lengths [Å] and angles [°] for (**2**).

Atoms	Contacts [Å]	Atoms	Angles [°]
Cd1-O3	2x 2.2262(2)	O3-Cd1-O1	107.414(2)
-O1	2x 2.5319(3)		72.586(2)
-O2	2x 2.5762(3)	O3-Cd1-O2	102.551(3)
			77.449(2)
		O1-Cd1-O2	97.625(2)
			82.375(2)
P1-O2	1.4196(1)	O2-P1-O3	107.355(4)
-O3	1.5388(1)	O2-P1-O1	105.184(3)
-O1	1.5796(2)	O3-P1-O1	112.061(3)

Table S2: Selected bond lengths [Å] and angles [°] for (**3**)

Atoms	Contacts [Å]	Atoms	Angles [°]
Cd1-O4	2.2439(7)	O4-Cd1-O2	88.883(16)
-O2	2.3328(5)	O4-Cd1-O7	97.065(15)
-O7	2.3706(5)	O4-Cd1-O1	108.637(26)
-O2	2.4066(6)	O4-Cd1-O6	90.491(13)
-O1	2.4510(7)	O7-Cd1-O1	101.328(15)
-O6	2.5202(5)	O7-Cd1-O2	90.393(14)
		O7-Cd1-O6	94.845(13)
		O2-Cd1-O1	83.732(14)
		O2-Cd1-O6	77.548(12)
		O2-Cd1-O2	81.845(13)
		O2-Cd1-O6	72.681(16)
		O2-Cd1-O1	85.573(15)
P1-O3	1.5271(6)	O3-P1-O2	111.586(21)
-O1	1.5333(4)	O3-P1-O1	108.832(21)
-O2	1.5337(4)	O2-P1-O1	107.854(19)
P2-O4	1.5220(4)	O4-P2-O5	111.726(18)
-O5	1.5609(4)	O4-P2-O6	113.024(19)
-O6	1.5708(5)	O5-P2-O6	103.310(24)
P3-O9	1.5299(7)	O9-P3-O8	95.607(20)
-O8	1.5422(4)	O9-P3-O7	113.216(40)
-O7	1.5525(6)	O8-P3-O7	118.683(21)

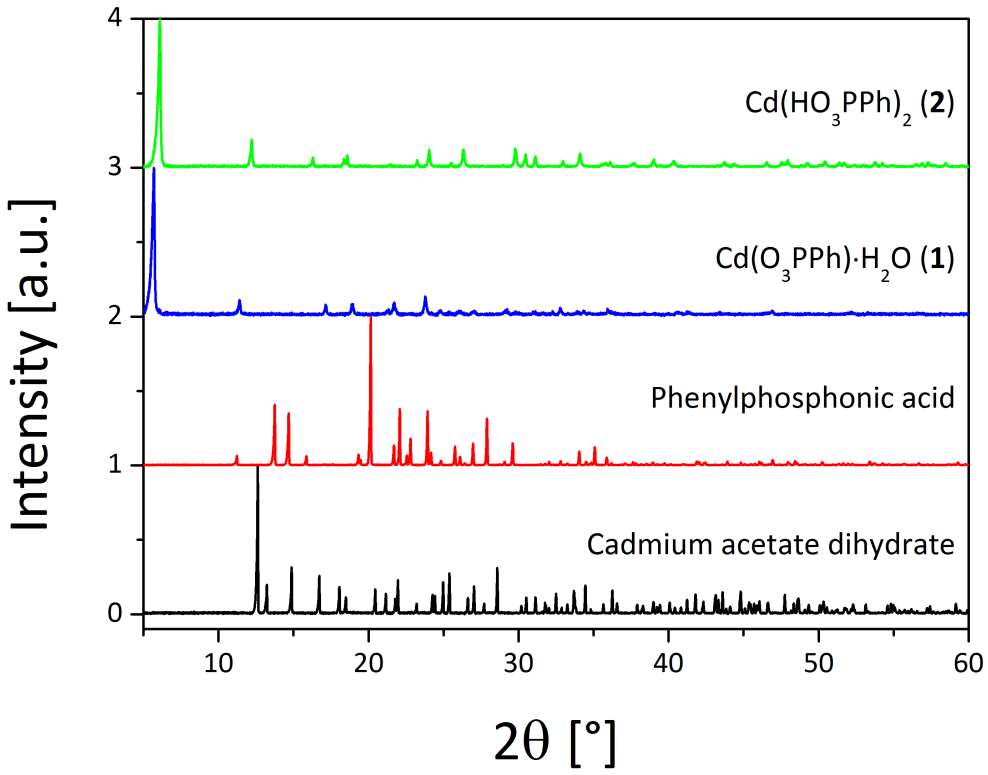

Figure S1: Comparison of the measured PXRD patterns of (**1**) (blue) and (**2**) (green) with the measured PXRD patterns of the starting materials cadmium acetate dihydrate (black) and phenylphosphonic acid (red).

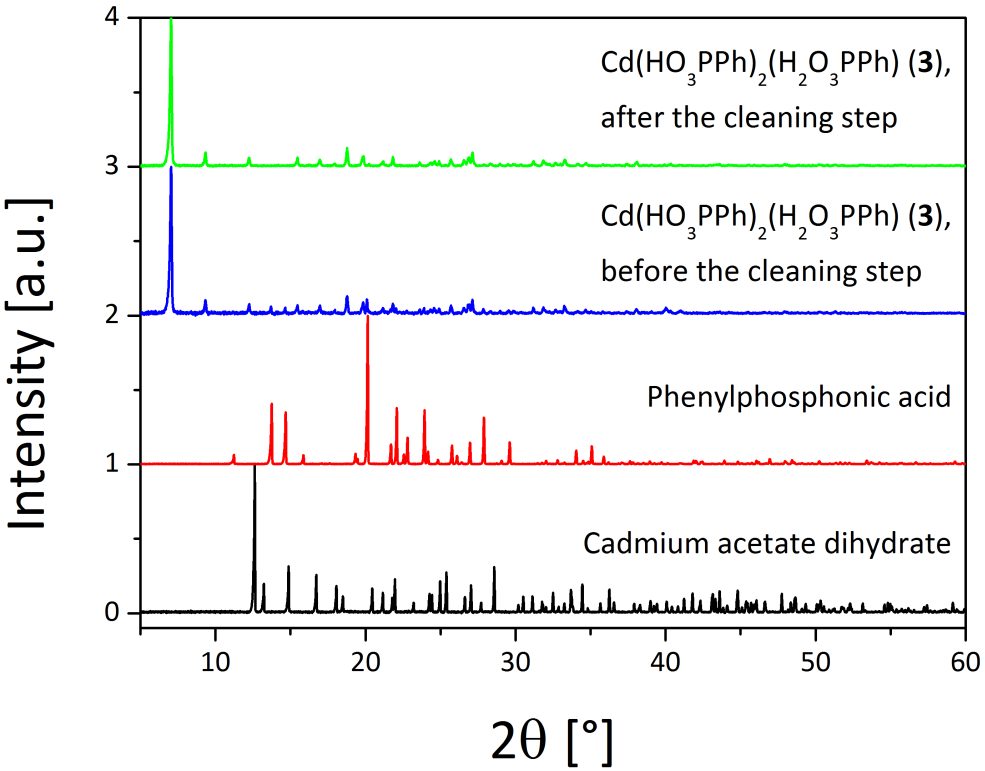

Figure S2: Comparison of the measured PXRD patterns of (**3**) after the mechanochemical synthesis, before the cleaning step (blue) and (**3**) after stirring in diethyl ether (green) with the measured PXRD patterns of the starting materials cadmium acetate dihydrate (black) and phenylphosphonic acid (red).

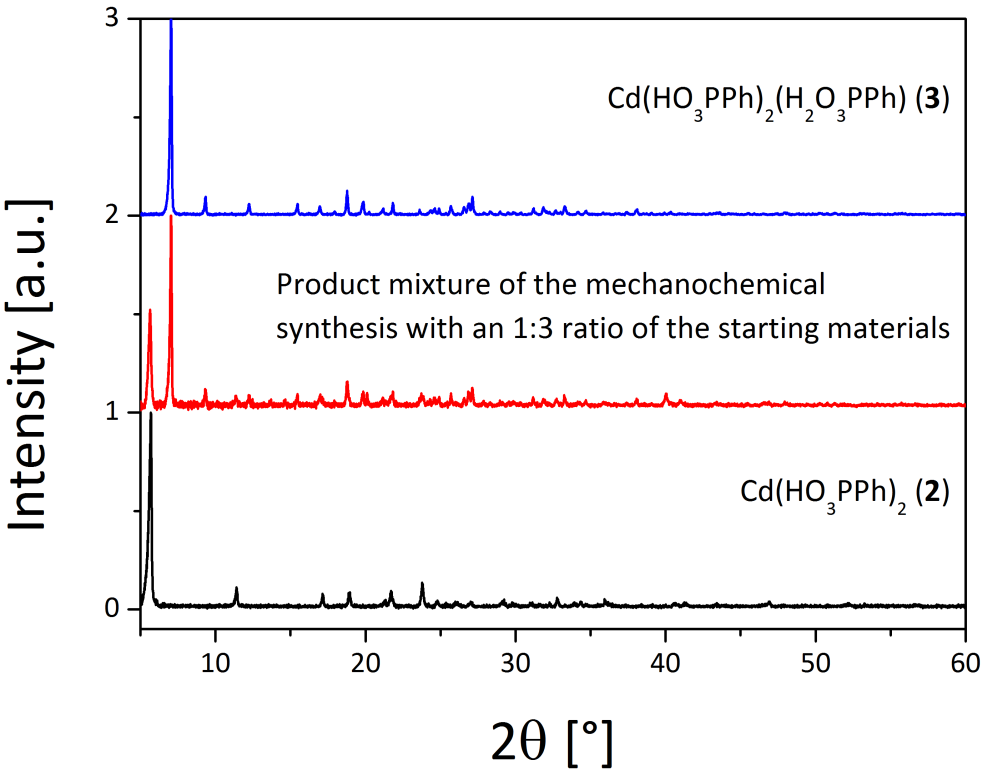

Figure S3: Comparison of the measured PXRD pattern of the product mixture of the mechanochemical synthesis of cadmium acetate with phenylphosphonic acid in a 1:3 ratio (red) with the measured PXRD patterns of (**2**) (blue) and (**3**) (black).

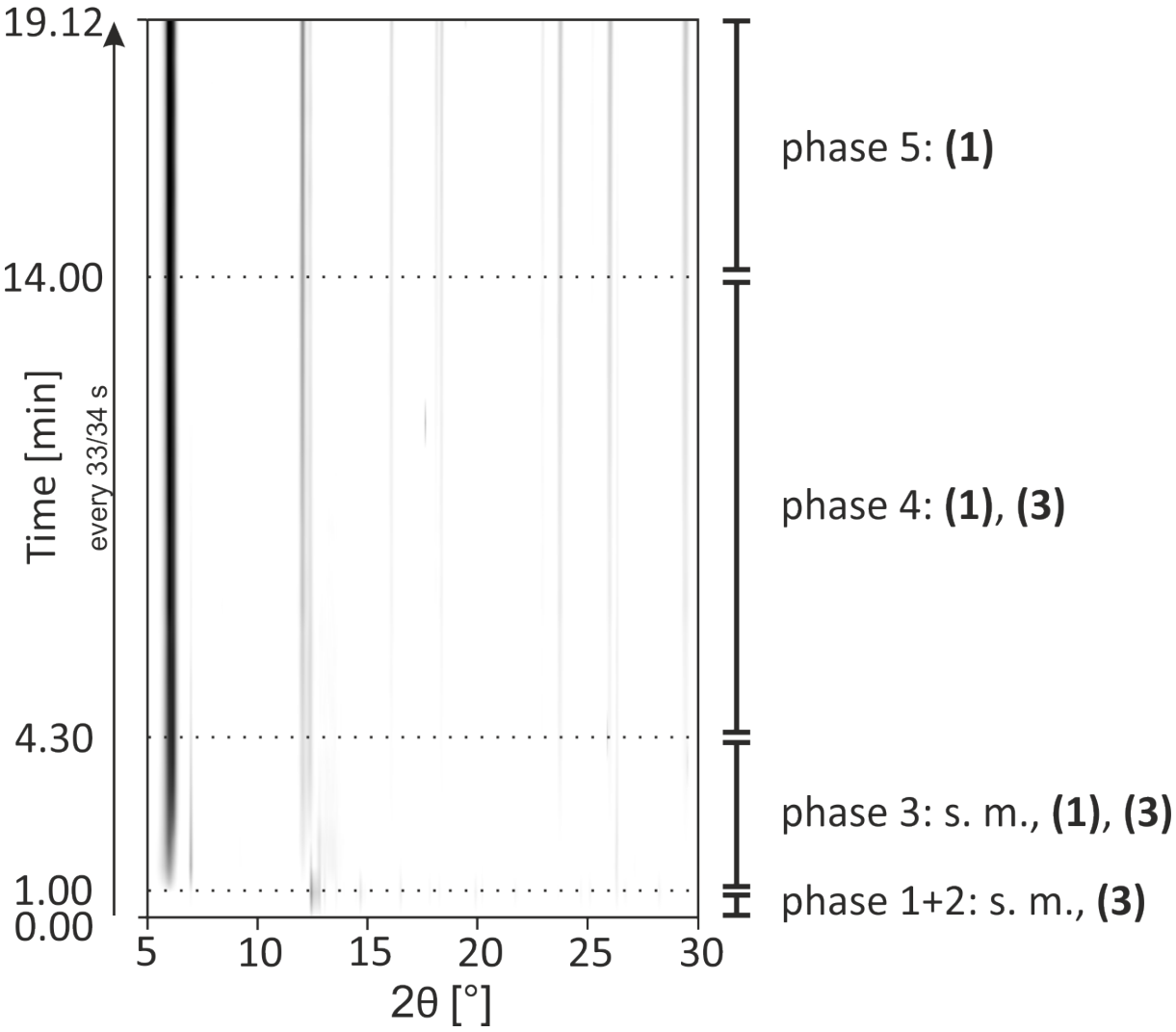

Figure S4: 2D plot of synchrotron XRD data for the synthesis of (**1**).

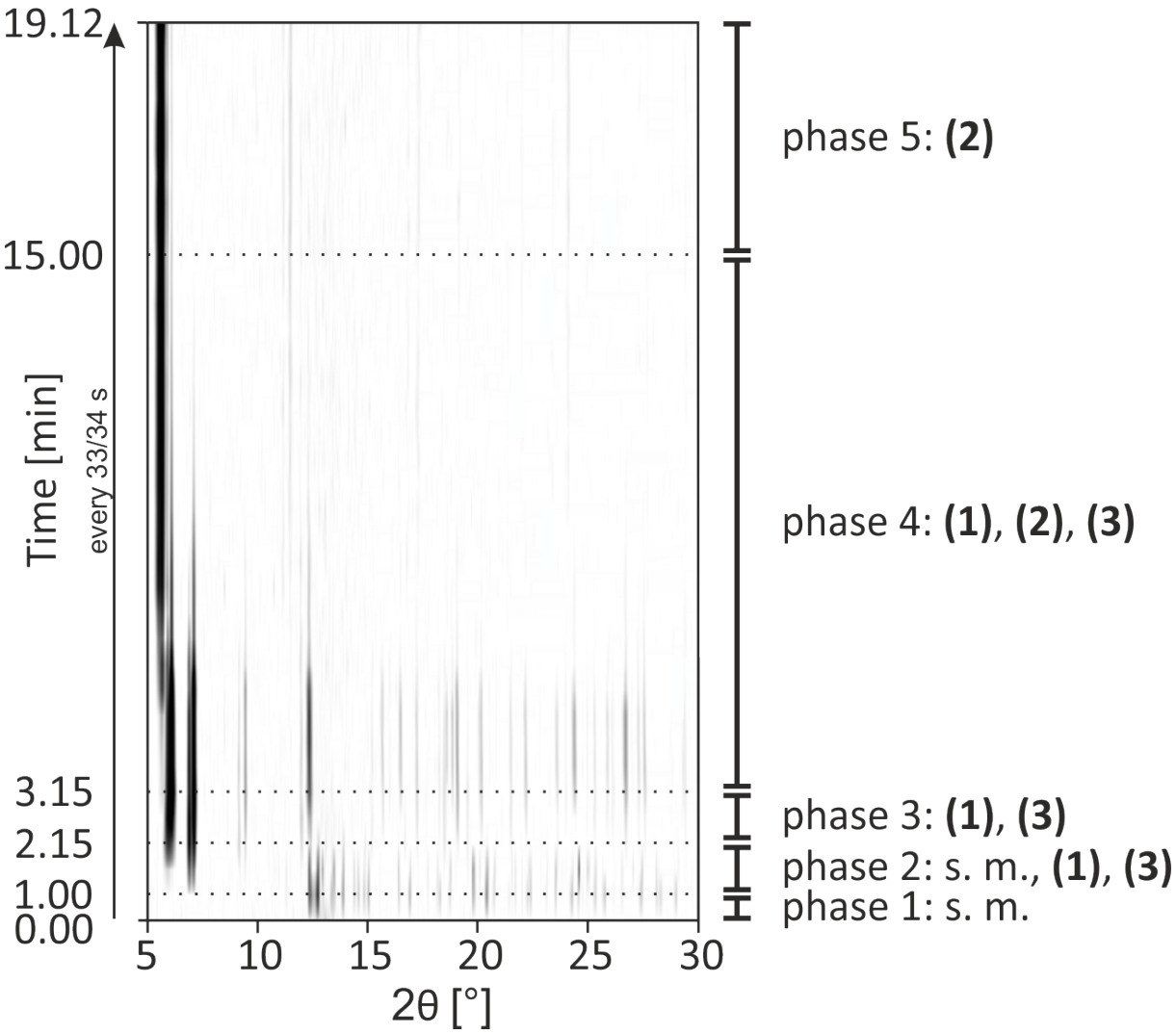

Figure S5: 2D plot of synchrotron XRD data for the synthesis of (**1**).

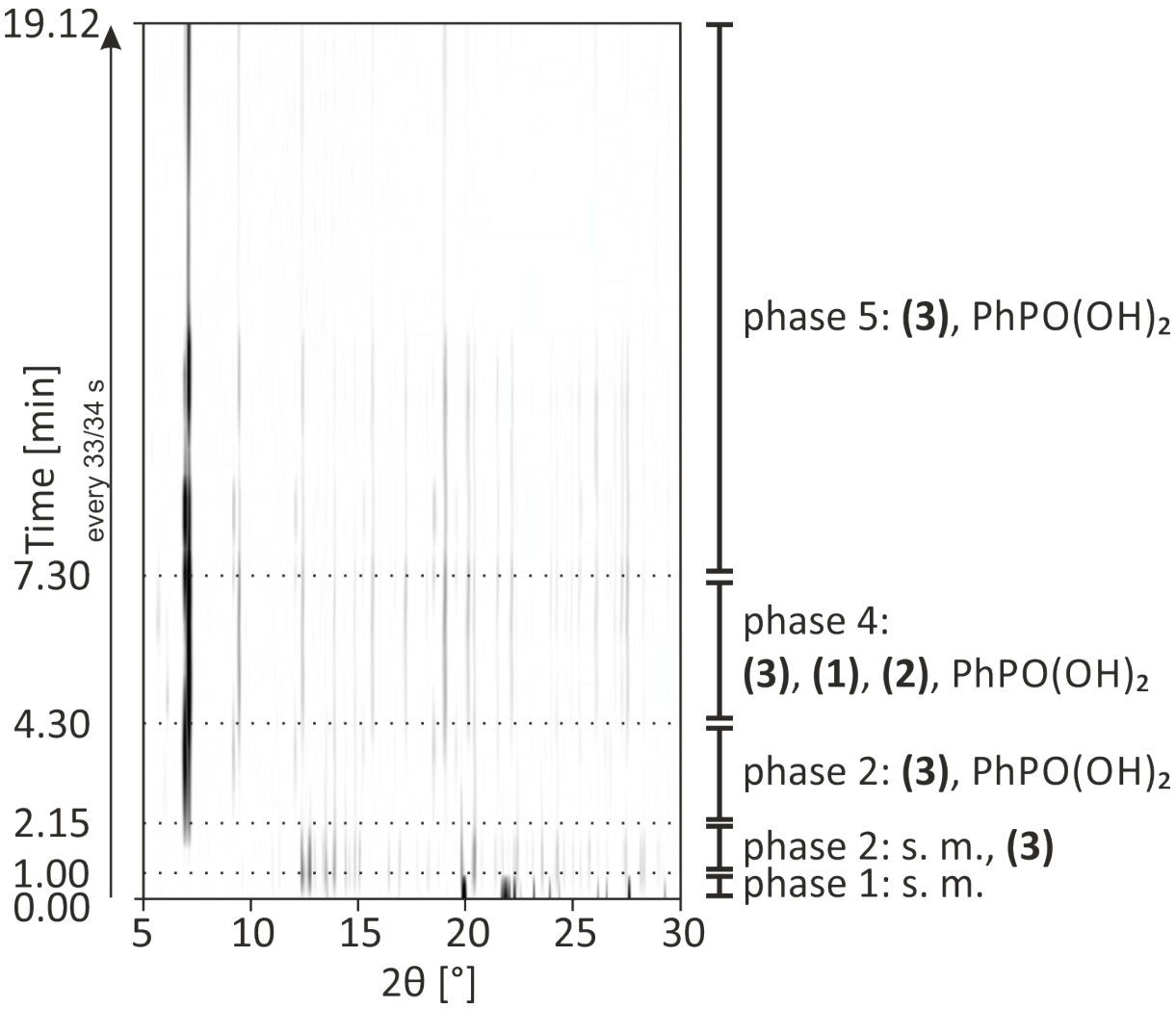

Figure S6: 2D plot of synchrotron XRD data for the synthesis of (**1**).

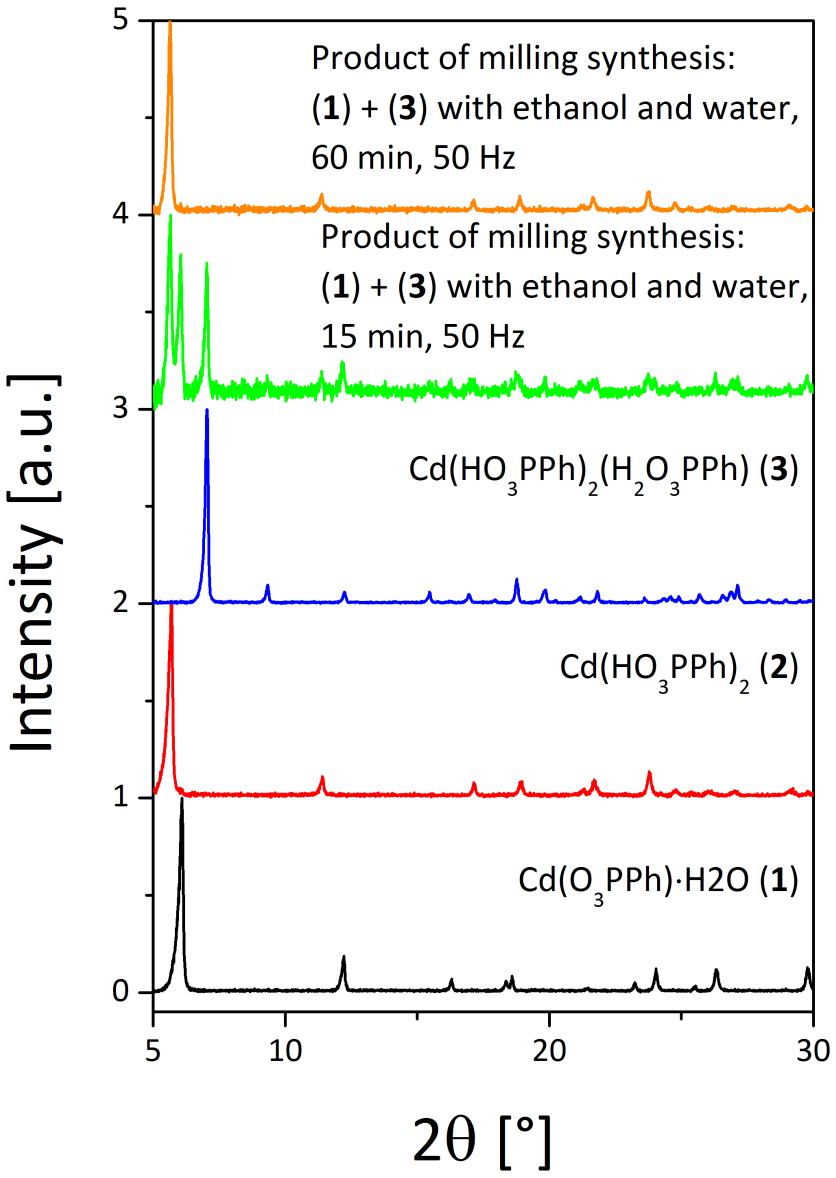

Figure S7: XRD patterns of the products of the syntheses milling compound (**1**) and (**3**) together for 15 min (green) or 60 min (orange) at 50 Hz. Reactions were carried out by milling (**1**) and (**3**) in a molar ratio of 1:1 and a total load of 1 g together with acetic acid (200 L) and water (50 L) as liquid assistants.

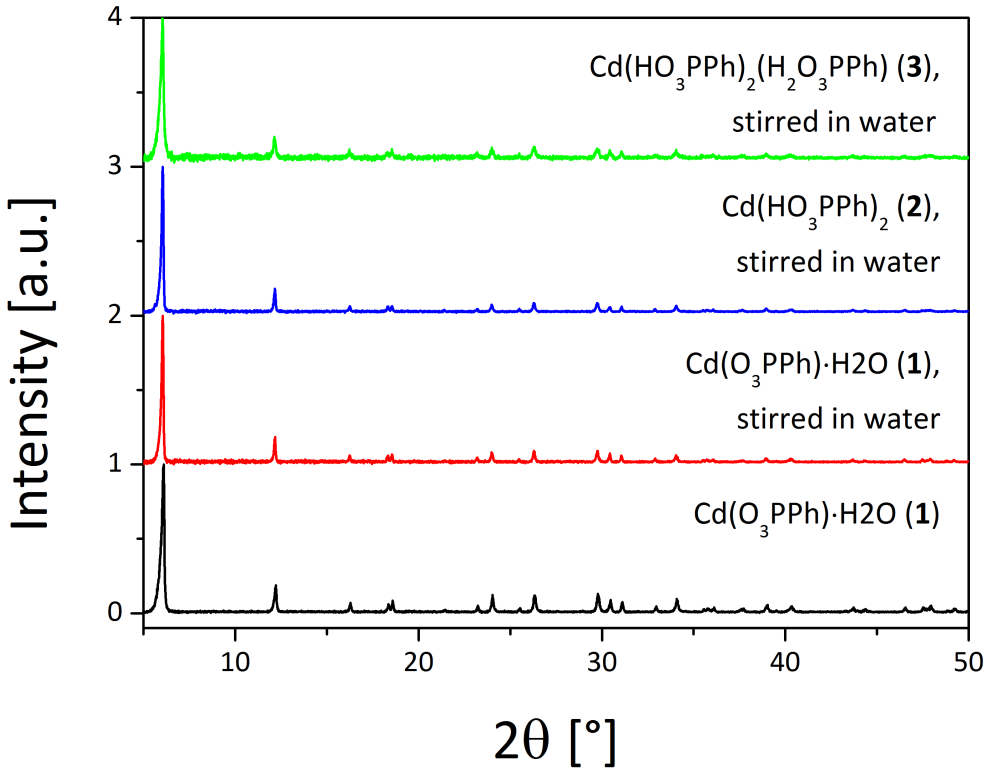

Figure S8: XRD patterns after slurry experiments to obtain the relative stability of the compounds (**1**)-(**3**). Each compound (250 mg) was stirred for 24 h in water (25 ml)) under ambient conditions. The solid phase was gained by filtration and analyzed by powder X-ray diffraction (PXRD). Compound 1 was obtained as single product in all three cases.