Pharmaceutical nanocrystals confined in porous host systems – Interfacial effects and amorphous interphases

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Characterization of controlled porous glasses

Structural properties have been determined using mercury intrusion measurements with a mercury contact angle of 141.3°. Filling degree of the samples has been calculated from mass increase after infiltration and porosity.

Table A1. Characteristics of the CPG samples.

CPG label	d_p nm	O_{sp} m^2g^{-1}	V_{cum} cm^3g^{-1}	P %	X_{ACE} %
8	8.4	163.2	0.34	41	100
11	11.1	150.0	0.53	36	84
14	14.4	107.2	0.39	48	98
16	15.7	114.4	0.45	45	80
39	38.5	46.5	0.40	40	86
69	69.1	37.6	0.49	51	89
8	89.0	25.8	0.40	48	91

d_p – pore diameter, O_{sp} – specific surface area, V_{cum} - cumulative pore volume, P - porosity, X_{ACE} - filling degree

Melting behaviour of ACE nanocrystals

Melting temperatures T_m have been determined based on the maximum of the melting peak in DSC heating scans measured on a PerkinElmer Pyris Diamond. Typical DSC sample masses have been 3-10 mg. The values for the melting enthalpy ΔH_m are taken from the integrated area of the melting peak after applying a tangent construction.

Table A2. Thermodynamic properties of ACE in nanopores.

d_p nm	form	T_m °C	ΔH_m $J{\cdot}g^{-1}$	f_c %	t_0 nm	F [§]
8.4	I	135.2	95.6	51	1.19	0.31
	III	- (103.4)[#]	- (70.9)[#]	- (43)[#]	- (1.44)[#]	-
11.1	I	137.8	104.7	56	1.37	0.35
	III	- (107,5)[#]	- (67.0)[#]	- (41)[#]	- (2.01)[#]	
14.4	I	147.9	125.7	68	1.28	0.34
	III	120.3	80.7	49	2.16	
15.7	I	155.6	122.5	66	1.48	0.32
	III	125.1	96.1	58	1.86	
38.5	I	163.8	147.2	79	2.13	0.31
	III	137.7	130.3	79	2.14	
69.1	I	165,3	132,7	71	5.36	0.36
	III	139,4	121,9	74	4.85	
89.0	I	167.8	168.1	90	2.20	0.32
	III	142.2	133.1	81	4.54	
mean	**I**				**2.14**	
	III				**2.71**	

[§] F – ACE mass fraction in the host guest system
[#] maximum value of $\Delta H_{m,III}$ measured after nucleation at lower temperature and growth at 80°C.