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APPENDICES

APPENDIX A

Qualitative and Quantitative XRPD analysis.

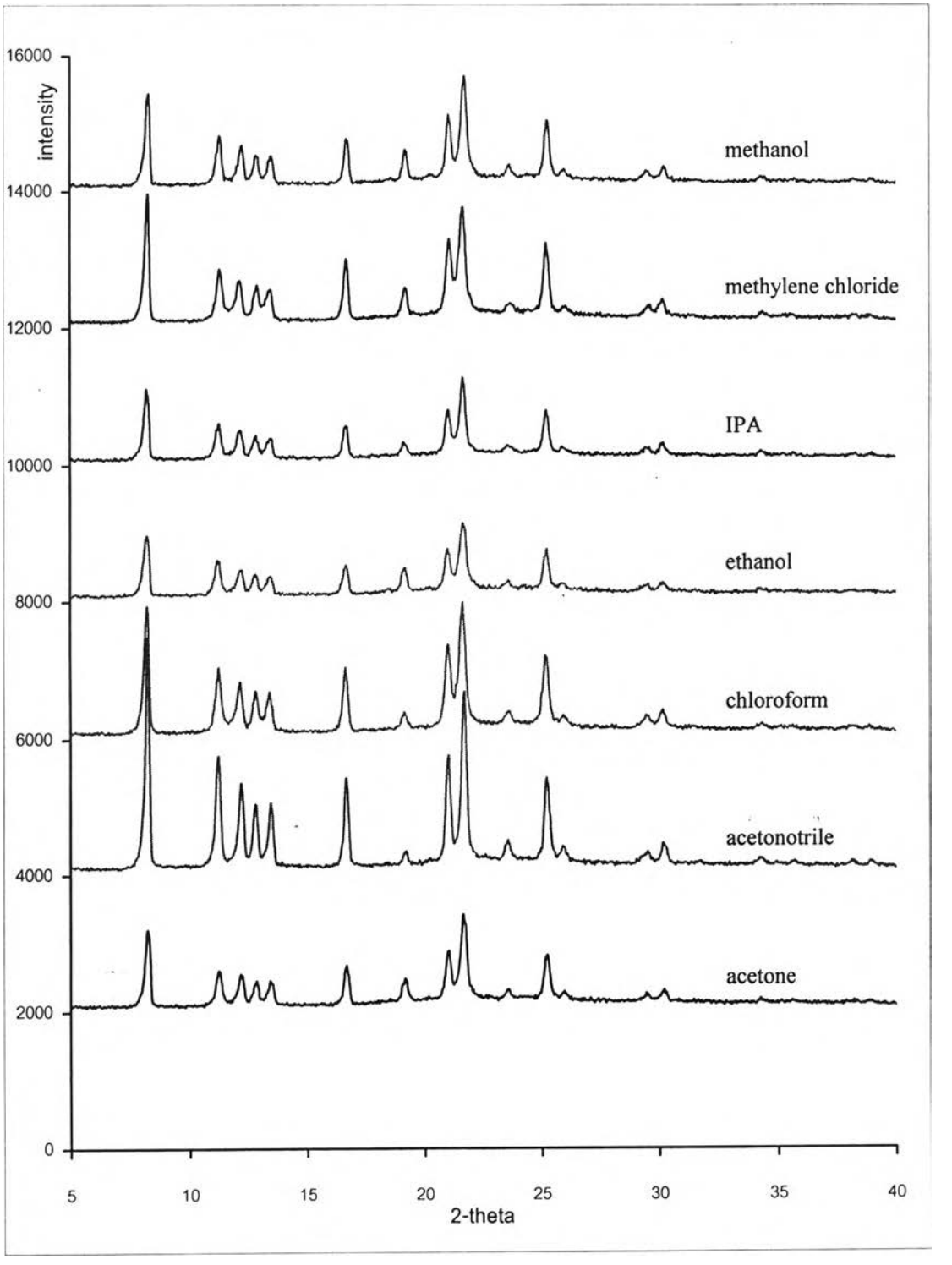

Figure 53: XRPD patterns of VPU products obtained from various solvents by method I

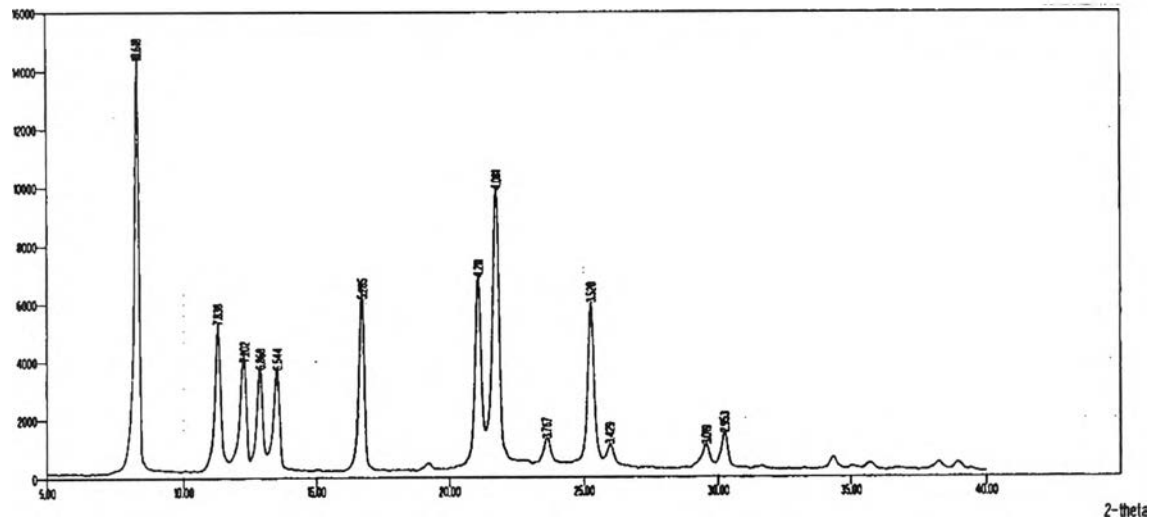

Figure 54: XRPD pattern of VPU product obtained from benzene by method II collected in damp mass

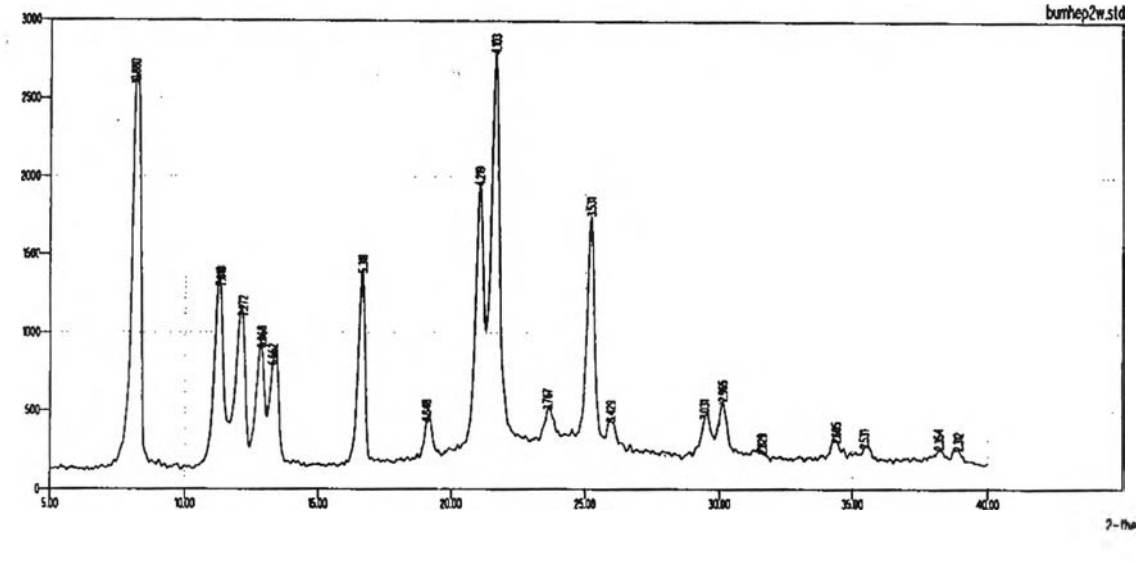

Figure 55: XRPD pattern of VPU product obtained from heptane by method II collected in damp mass

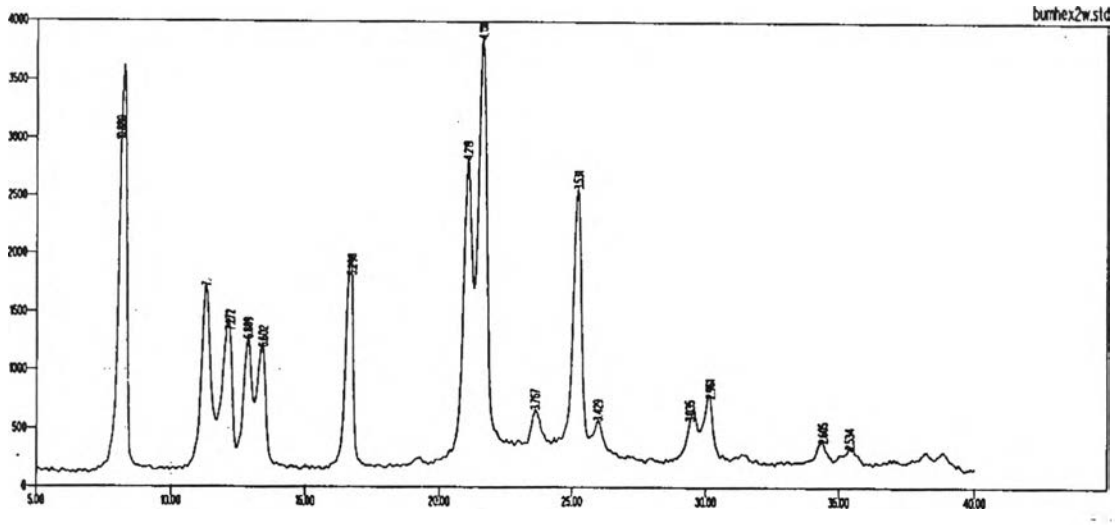

Figure 56: XRPD pattern of VPU product obtained from hexane by method II collected in damp mass

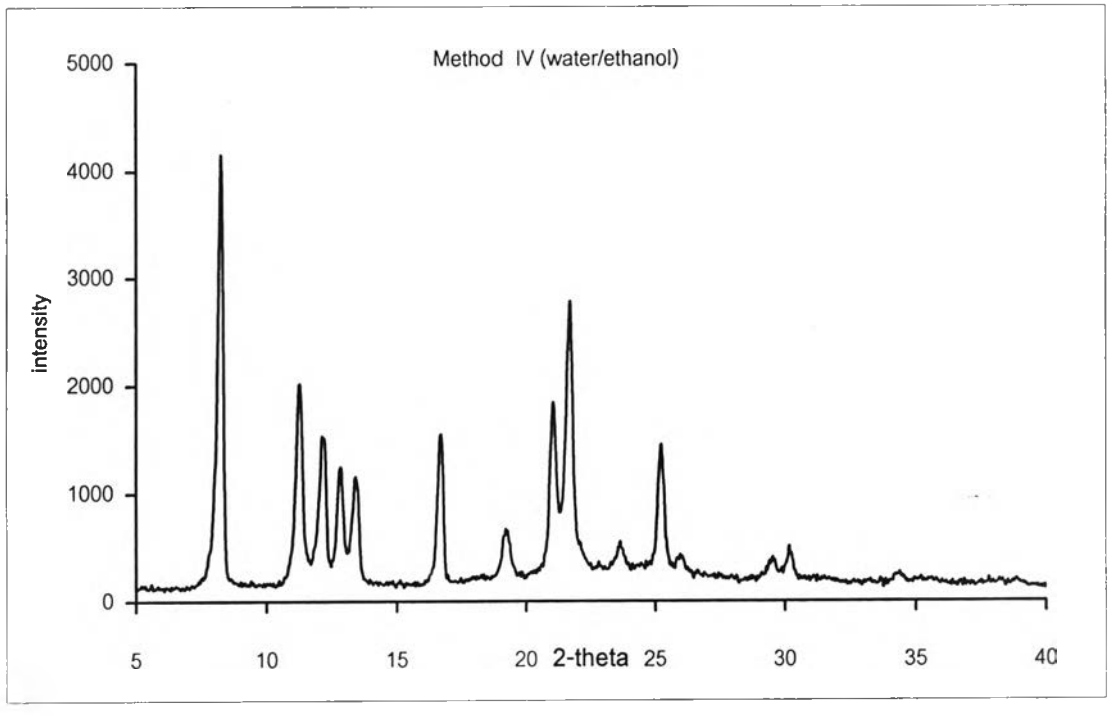

Figure 57: XRPD pattern of VPU product obtained from recrystallized by ethanol/water (method II).

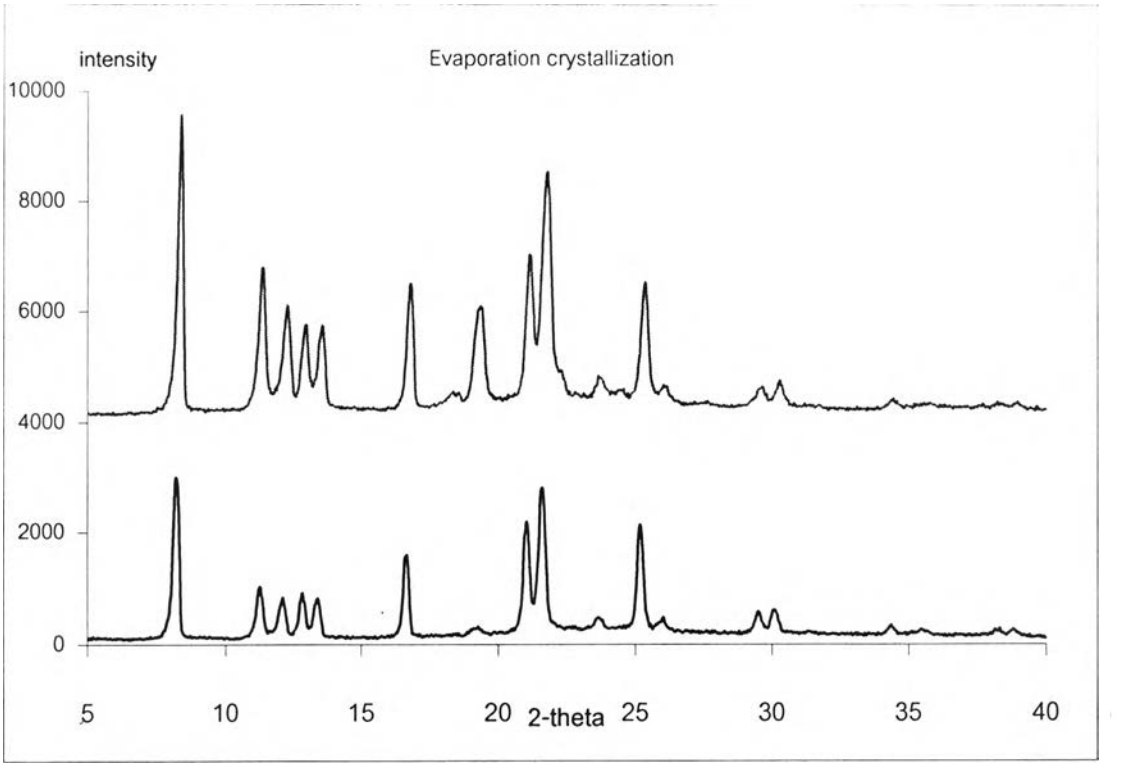

Figure 58: XRPD patterns of VPU products obtained by evaporation crystallization method (Method V)

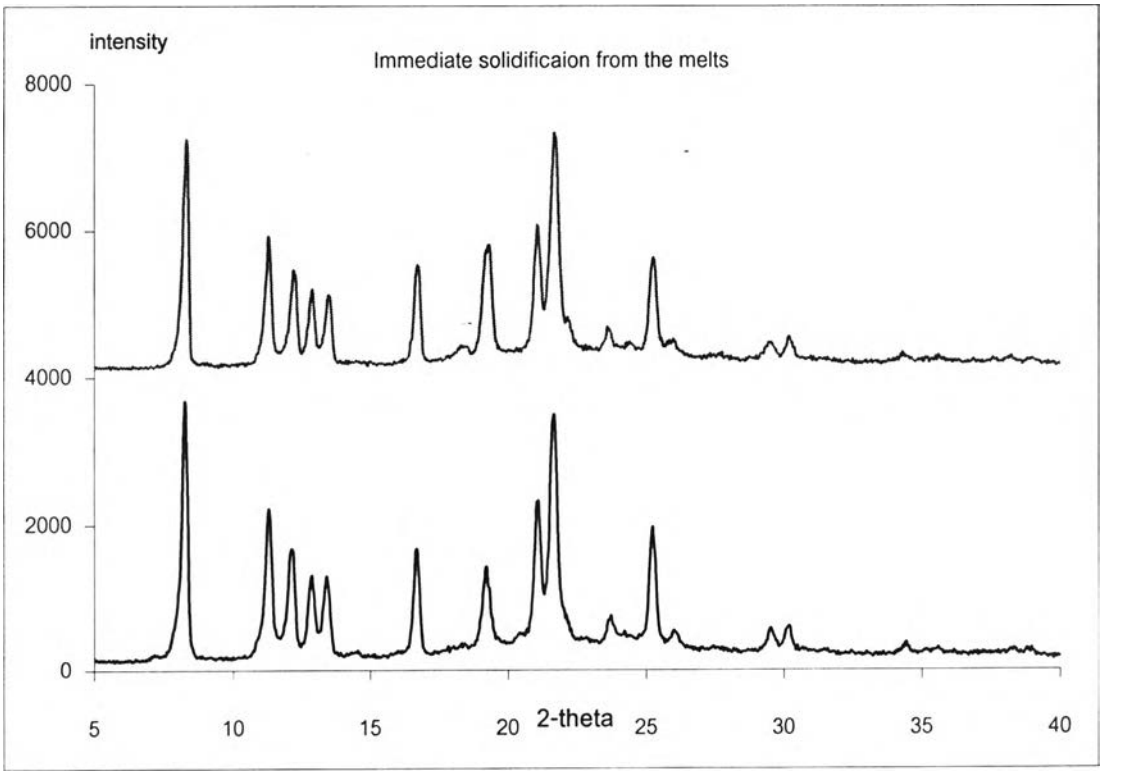

Figure 59: XRPD patterns of VPU products obtained by immediate solidification from the melt (Method VI)

Table 10: Intensity ratio of samples obtained from stability study at 50, 60, 70 and 80 °C for 0, 2 and 4 weeks

Time interval	Temperature		Peak area			Average intensity ratio	Standard deviation
			I_{VPU} $2\theta = 8.4$	I_{NaCl} $2\theta = 31.7$	I_{VPU}/I_{NaCl}		
Begin	1		274.35	300.10	0.9142	1.0661	1.4358×10^{-1}
	2		341.83	284.96	1.1996		
	3		475.20	438.17	1.0845		
2 weeks	50C	1	243.82	231.09	1.0551	1.0484	5.5564×10^{-2}
		2	375.30	379.18	0.9898		
		3	363.48	330.35	1.1003		
	60C	1	333.13	409.33	0.8138	1.0107	4.0545×10^{-1}
		2	404.65	273.96	1.4770		
		3	289.15	390.04	0.7413		
	70C	1	292.23	264.40	1.1053	8.9264×10^{-1}	1.8413×10^{-1}
		2	228.30	290.41	0.7861		
		3	256.41	326.00	0.7865		
	80C	1	276.89	315.31	0.8782	8.5238×10^{-1}	4.2619×10^{-2}
		2	160.50	183.26	0.8758		
		3	288.20	358.82	0.8032		
4 weeks	50C	1	445.72	429.16	1.0386	1.0428	7.9711×10^{-2}
		2	327.34	339.09	0.9653		
		3	492.17	437.64	1.1246		
	60C	1	203.24	239.04	0.8502	9.4781×10^{-1}	9.5740×10^{-2}
		2	181.06	190.27	0.9516		
		3	319.72	306.95	1.0416		
	70C	1	200.06	241.92	0.8270	8.4752×10^{-1}	2.1475×10^{-2}
		2	166.33	196.66	0.8458		
		3	470.69	541.14	0.8698		
	80C	1	221.94	276.96	0.8013	7.5791×10^{-1}	4.2111×10^{-2}
		2	189.48	250.92	0.7551		
		3	442.34	616.71	0.7173		

APPENDIX B

DSC thermograms of VPU products obtained from various methods

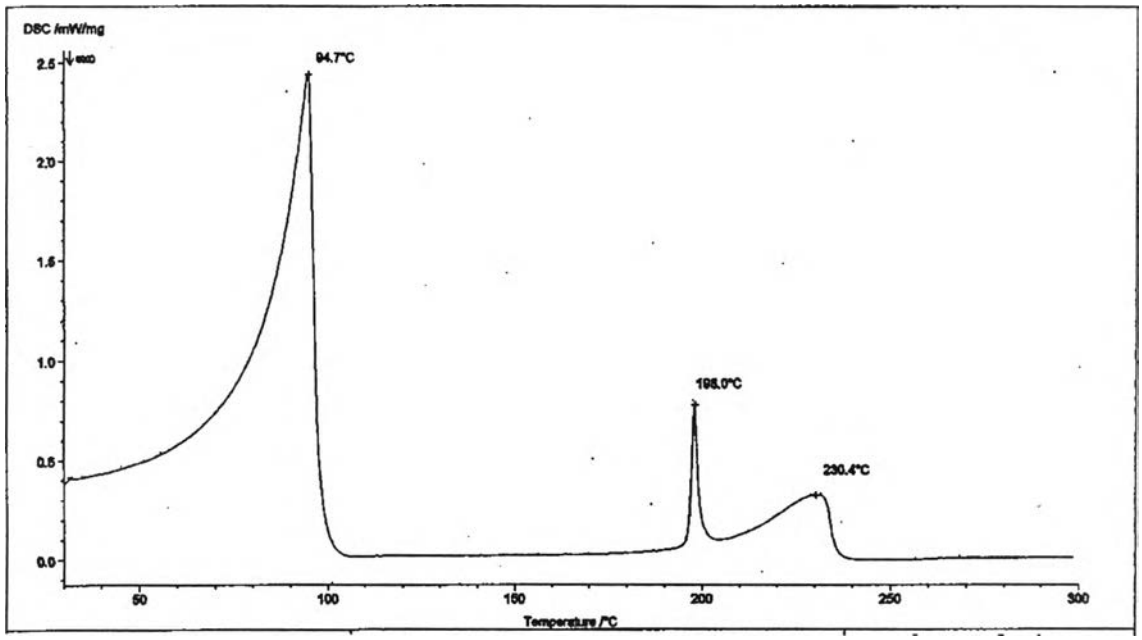

Figure 60: DSC thermogram (10°C/min) of the product obtained from heptane by method II collected in damp mass.

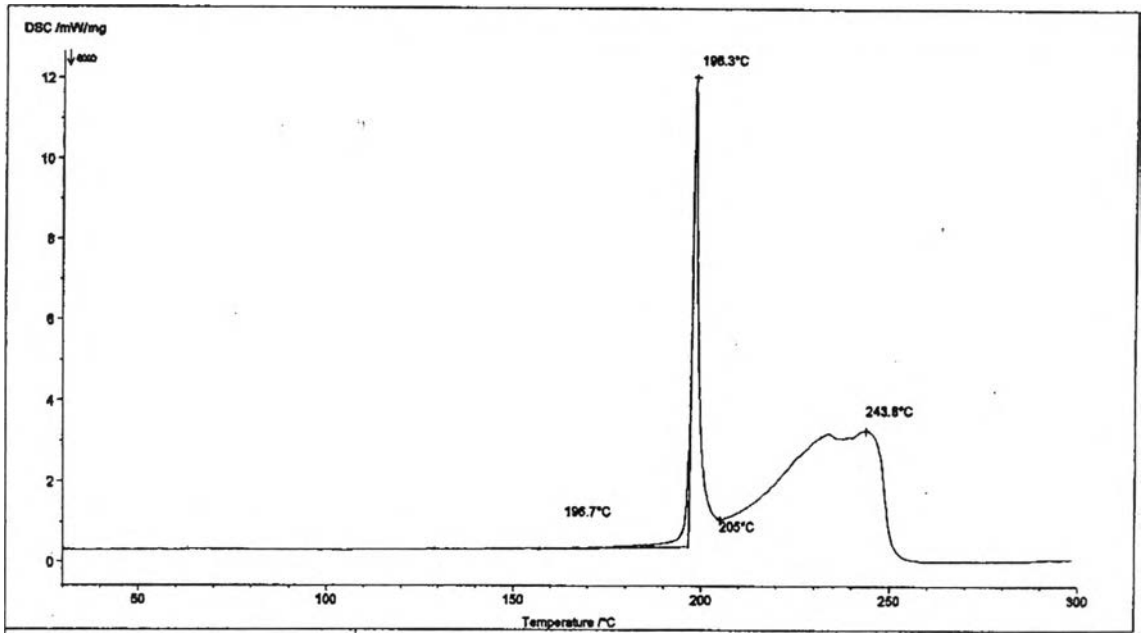

Figure 61: DSC thermogram (10°C/min) of the product obtained from heptane by method II collected in dry form.

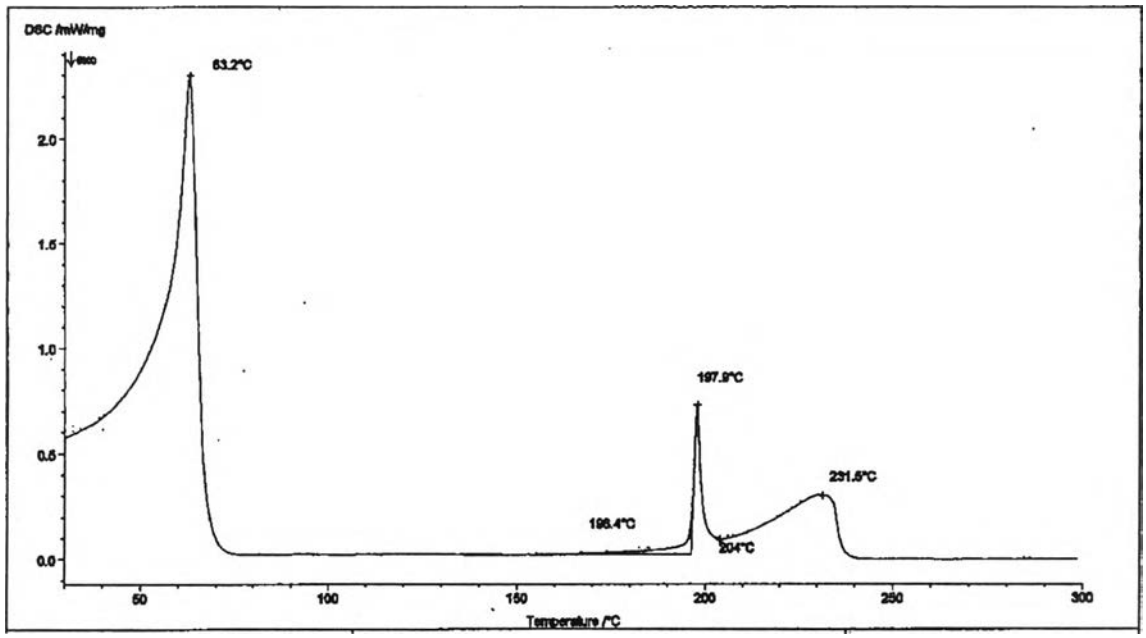

Figure 62: DSC thermogram (10°C/min) of the product obtained from hexane by method II collected in damp mass.

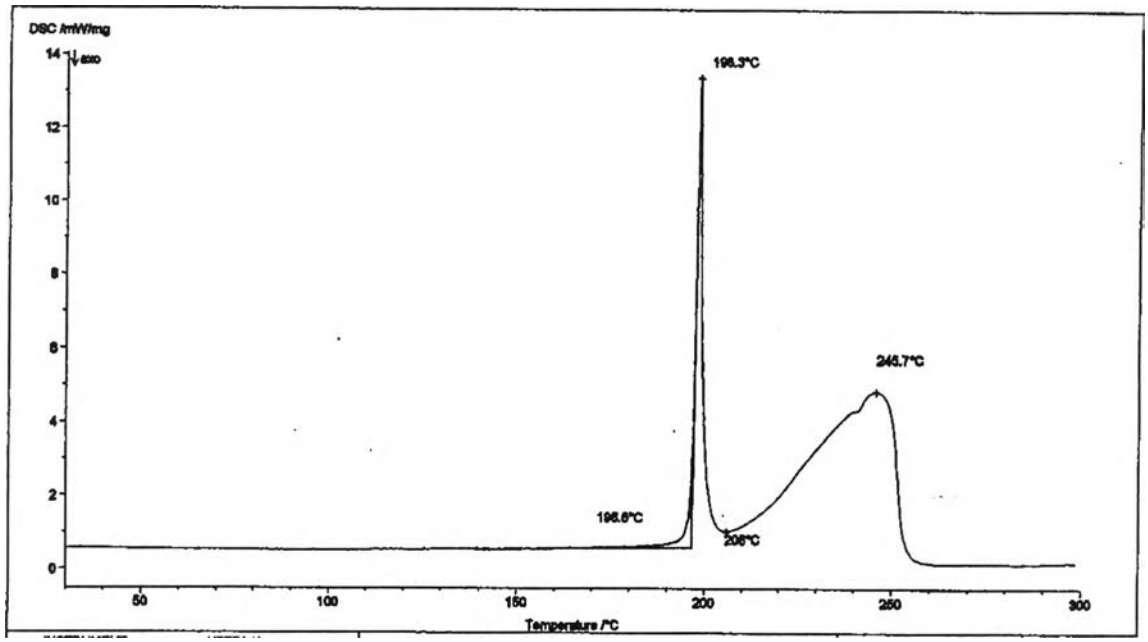

Figure 63: DSC thermogram (10°C/min) of the product obtained from hexane by method II collected in dry form.

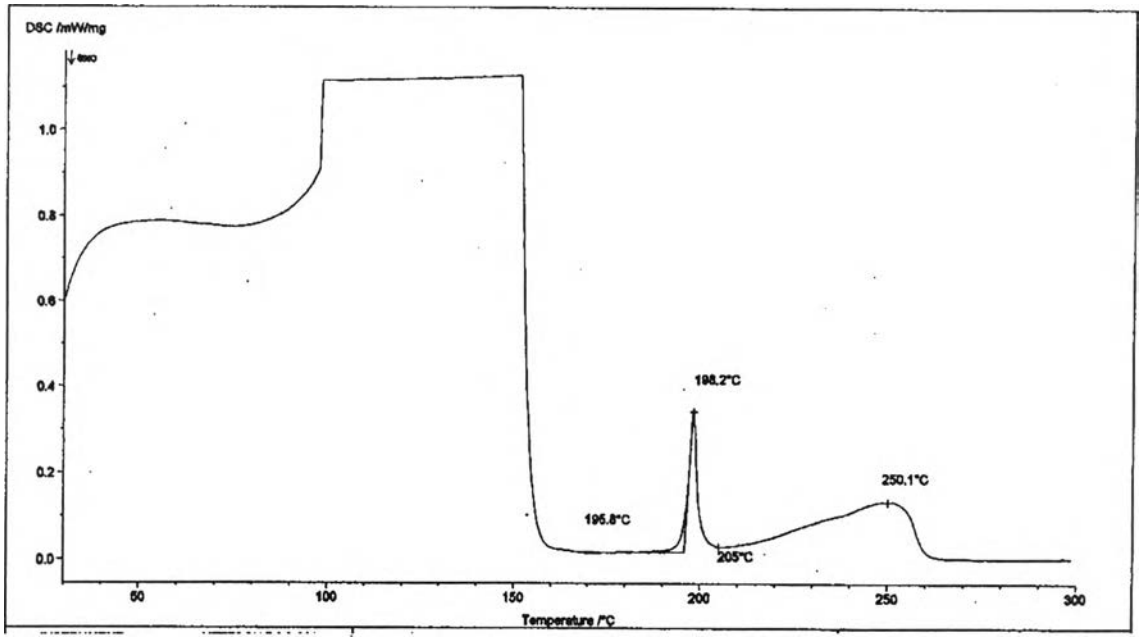

Figure 64: DSC thermogram (10°C/min) of the product obtained from water by method II collected in damp mass.

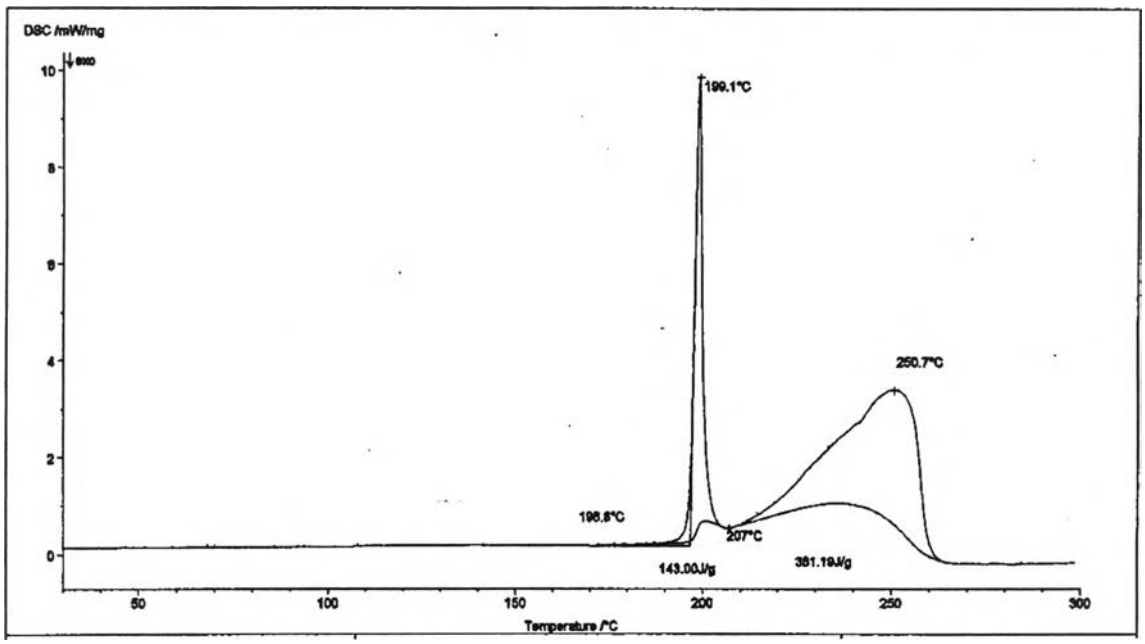

Figure 65: DSC thermogram (10°C/min) of the product obtained from water by method II collected in dry form.

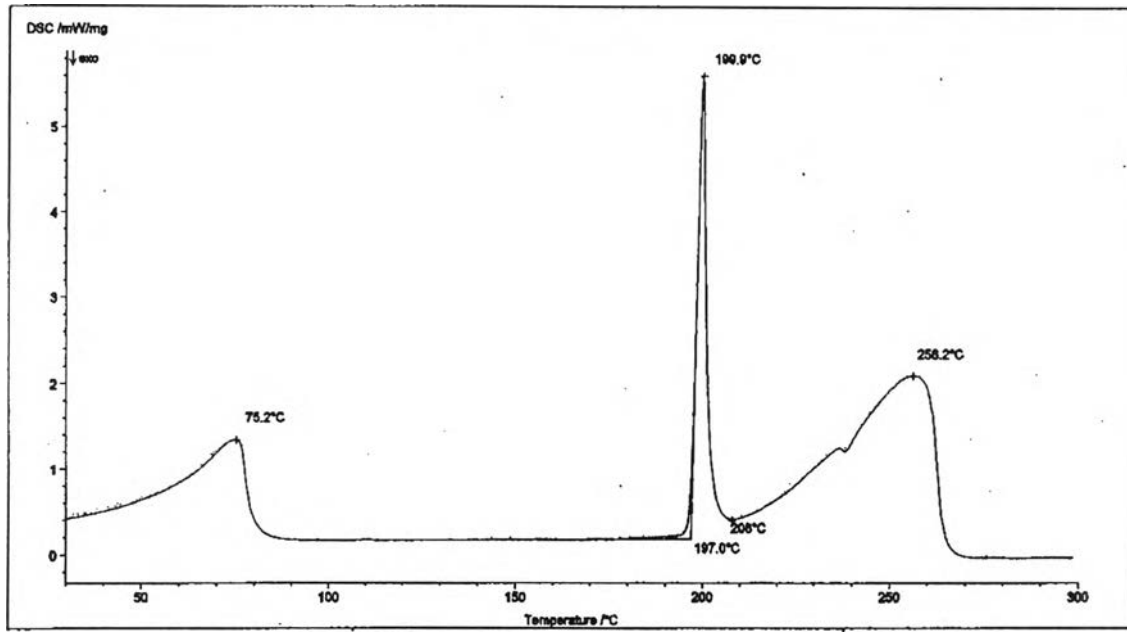

Figure 66: DSC thermogram (10°C/min) of the product obtained from benzene by method II collected in dry form.

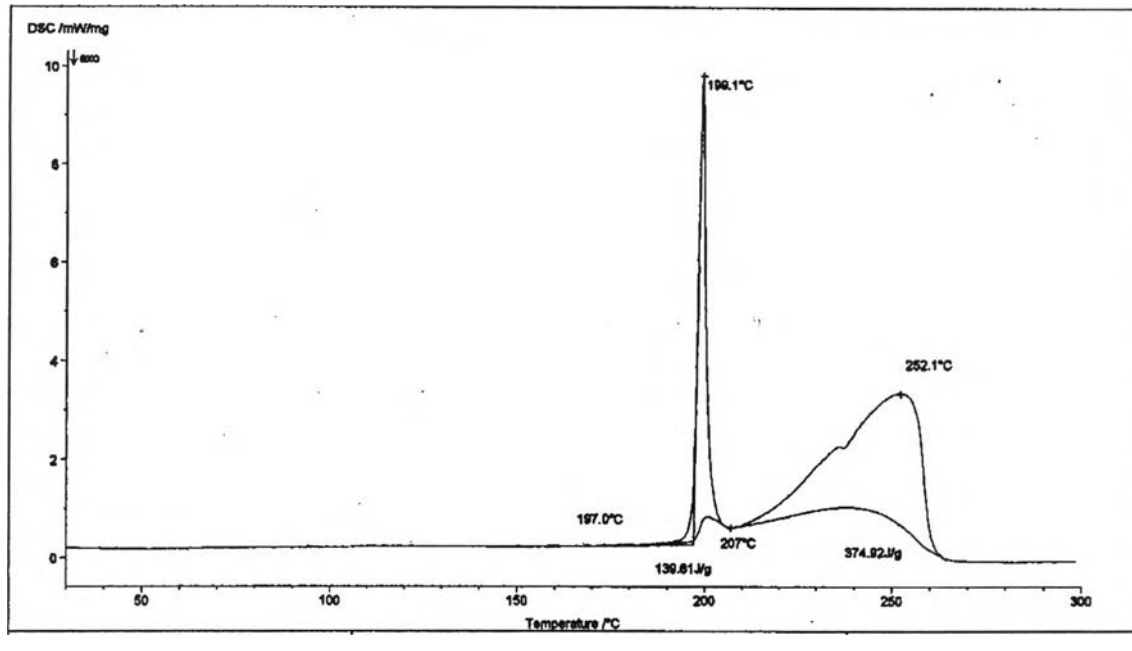

Figure 67: DSC thermogram (10°C/min) of the product obtained from benzene by thermal treatment (Method VII)

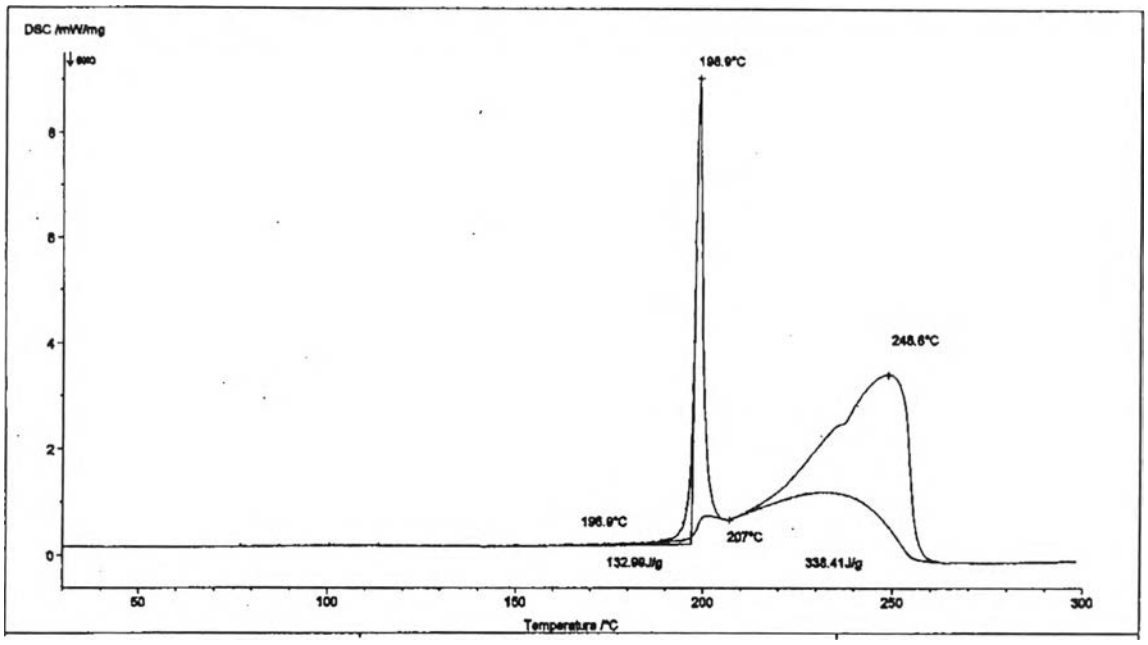

Figure 68: DSC thermogram (10°C/min) of the product obtained from diethyl ether by thermal treatment (Method VII)

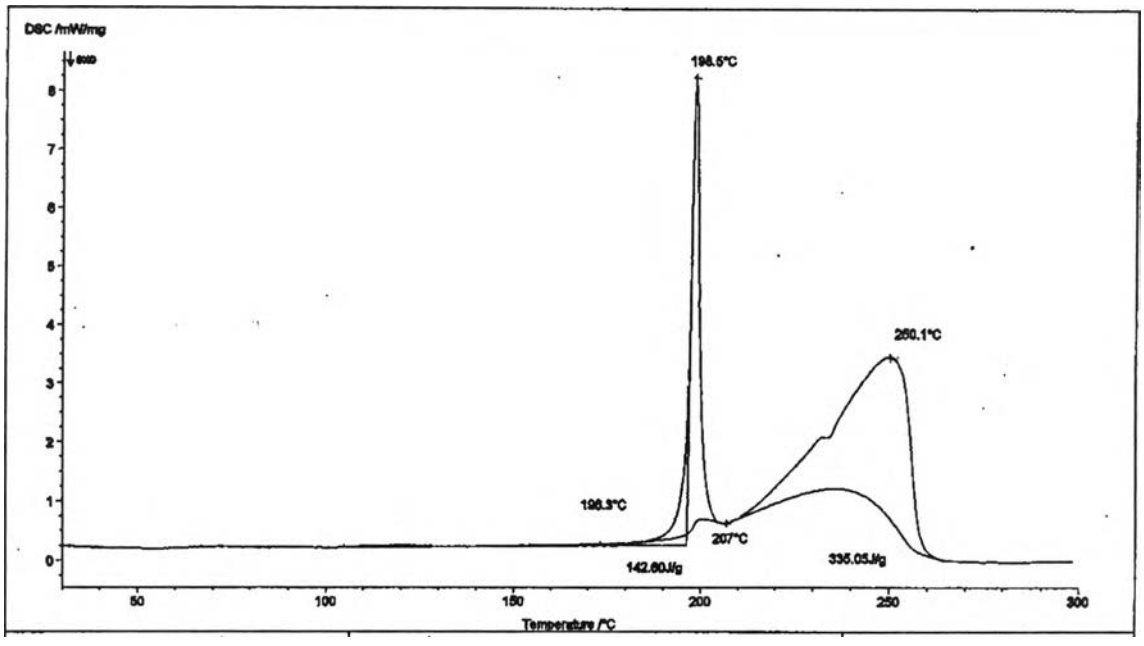

Figure 69: DSC thermogram (10°C/min) of the product obtained from heptane by thermal treatment (Method VII)

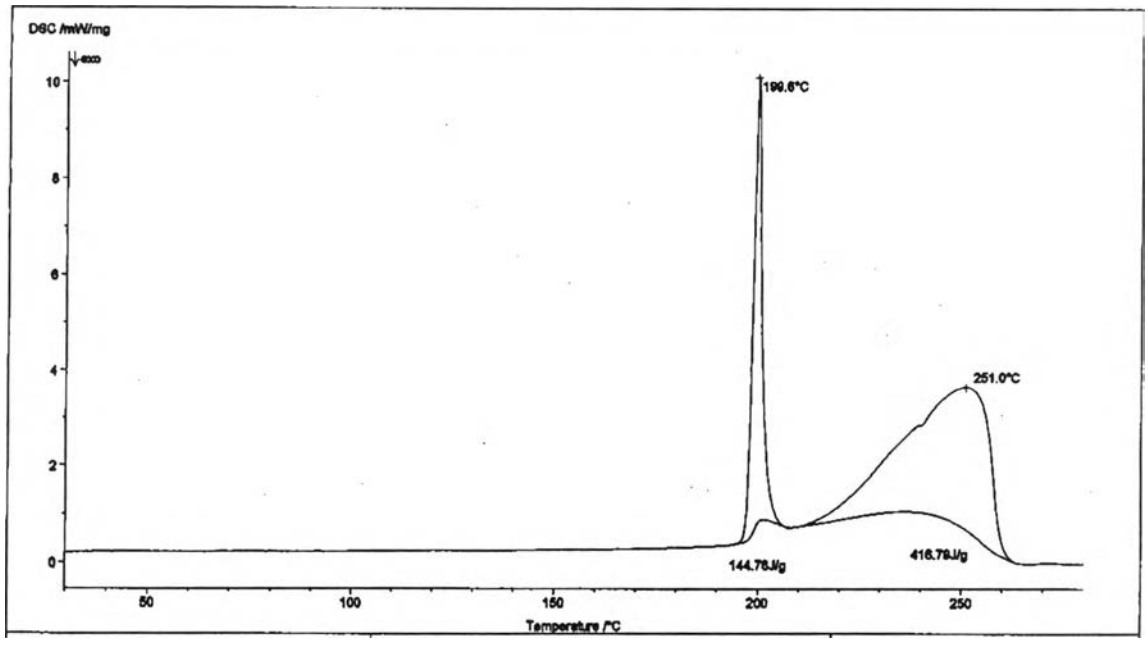

Figure 70: DSC thermogram (10°C/min) of the product obtained from stability study at 80 °C for 4 weeks.

APPENDIX C

Solubility data

Table 11: Concentration and absorbance data for calibration curve of the reference VPU in acetonitrile:water = 7:3.

conc. (mg/ml)	Absorbance λ=225			Average absorbance	SD
0.0899	0.112	0.141	0.113	0.122	16.46
0.1198	0.163	0.154	0.157	0.158	4.58
0.1498	0.212	0.203	0.206	0.207	4.58
0.2996	0.461	0.461	0.462	0.461	0.58
0.4494	0.706	0.681	0.687	0.691	13.05

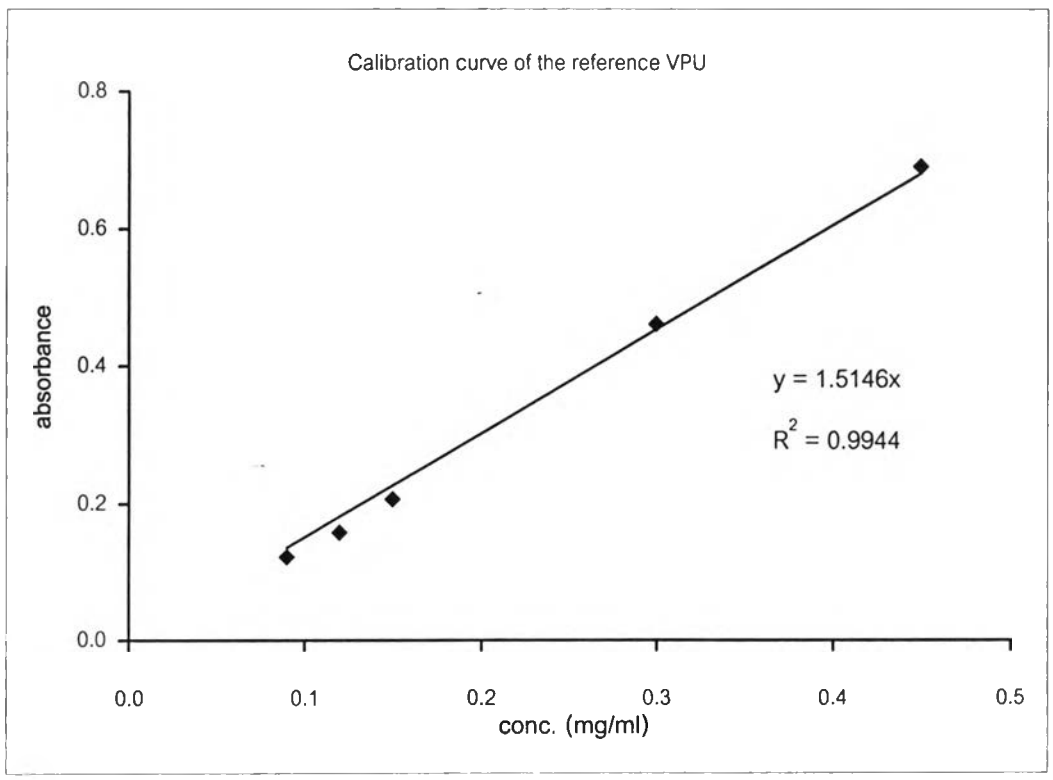

Figure 71: Calibration curve for the reference VPU in acetonitrile:water = 7:3 using UV absorption spectroscopy at λ = 225 nm.

Table 12: Solubility data of the reference VPU and the samples obtained from stability study at 80 °C for 4 weeks.

Samples	Time (hr)	Absorbance at λ = 225 nm			Average absorbance	SD	Concentration (mg/ml)
Reference VPU	1 hr	0.172	0.172	0.157	0.167	8.66	0.1103
	2 hr	0.160	0.166	0.158	0.161	4.16	0.1065
	3 hr	0.163	0.155	0.166	0.161	5.69	0.1065
	4 hr	0.157	0.166	0.157	0.160	5.20	0.1056
	6hr	0.155	0.162	0.158	0.158	3.51	0.1062
	9 hr	0.187	0.177	0.166	0.177	10.50	0.1166
	24 hr*	0.165	0.162	--	0.164	2.12	0.1079
	32 hr	0.144	0.143	0.140	0.142	2.08	0.0940
Sample from stability study at 80°C, 4 weeks	1 hr	0.248	0.213	0.216	0.226	19.40	0.1490
	2 hr*	0.241	0.223	--	0.232	12.73	0.1532
	3 hr	0.218	0.211	0.221	0.217	5.13	0.1431
	4 hr	164	0.173	0.293	0.210	72.02	0.1387
	6 hr	0.158	0.167	0.154	0.160	6.66	0.1054
	9 hr	0.163	0.160	0.151	0.158	6.24	0.1043
	24 hr	0.150	0.146	0.149	0.148	2.08	0.0979
	32 hr	0.149	0.142	0.157	0.149	7.51	0.0986

*n=2

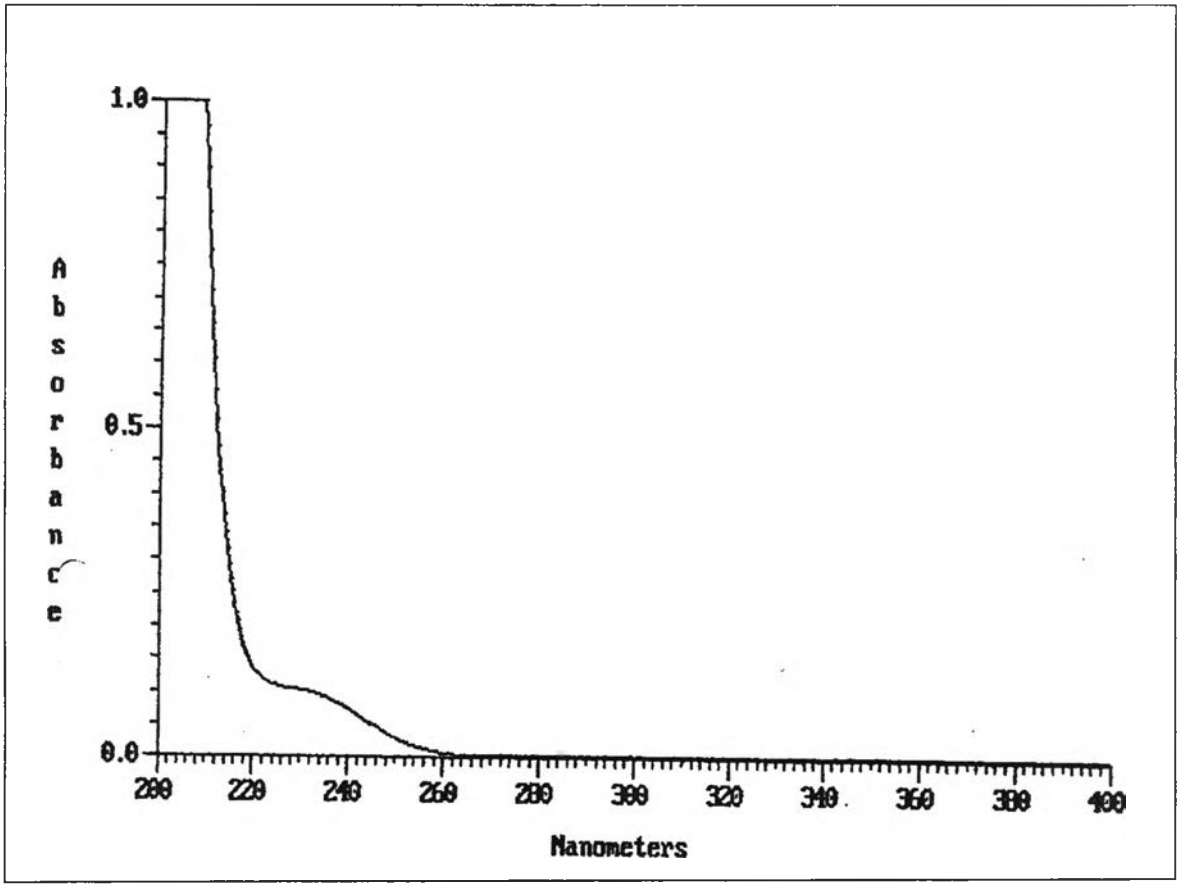

Figure 72: UV absorption spectrum of the reference VPU 0.0600 mg/ml in acetonitrile:water = 7:3

APPENDIX D

Table 13: Dielectric constant of solvents used in recrystallization (74).

Solvent	Formula	Boiling point (°C)	Solvent Strength	Dielectric constant (at 20 °C)
Distilled water	H_2O	100		78.54(25)
Acetone	CH_3COCH_3	56.5		20.7(25)
Acetonitrile	CH_3CN	81.6	0.52	37.5
Benzene	C_6H_6	80.1		2.284
Chloroform	$CHCl_3$	61-62	0.26	4.806
Diethyl ether	$C_2H_5OC_2H_5$	34.6	0.38	4.335
Ethanol	C_2H_5OH	78.5		24.30(25)
Ethyl acetate	$CH_3COOC_2H_5$	77	0.48	6.02(25)
Heptane	$CH_3(CH_2)_5CH_3$	98.4	0.00	1.92
Hexane	$CH_3(CH_2)_4CH_3$	69	0.00	1.890
Isopropyl alcohol	$CH_3CHOHCH_3$	82.5	0.60	18.3(25)
Methyl alcohol	CH_3OH	64.7	0.70	32.63(25)
Methylene Chloride	CH_2Cl_2	39.75	0.30	9.08

VITA

Miss Pasharin Siriaroonrat was born on July 21, 1973 in Nakornpathom, Thailand, She received her Bachelor of Science in Pharmacy from the Faculty of Pharmaceutical Sciences, Chulalongkorn University, Bangkok, Thailand in 1996. After graduation, she worked at Sirindhorn College of Public Health, Pitsanulok, for one years. Since then, she has been working at Chaopraya Yomaraj Hospital, Supanburi. In 1998, she entered the Master's Degree program in Manufacturing Pharmacy at Chulalongkorn University.