

Supporting Information

Alditols in complexing with boronophenyl alanine for improving aqueous solubility for boron neutron capture therapy

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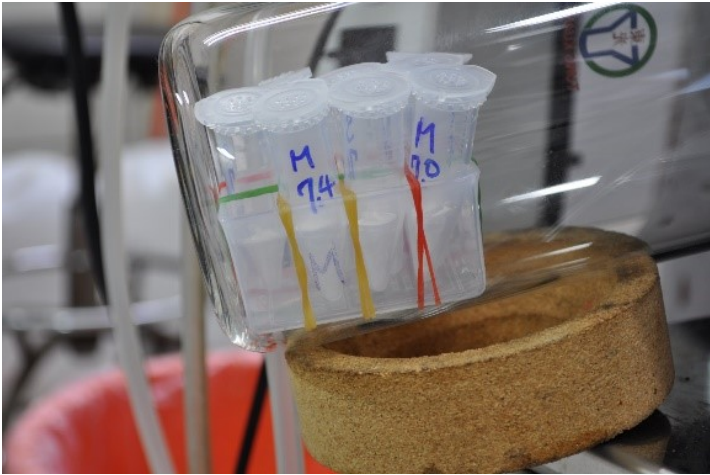
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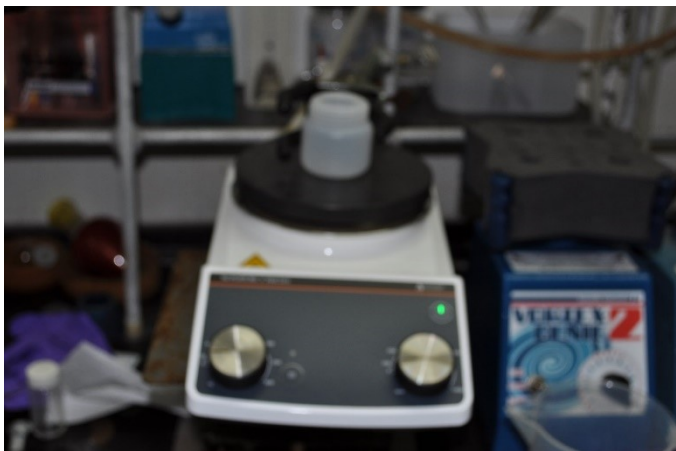
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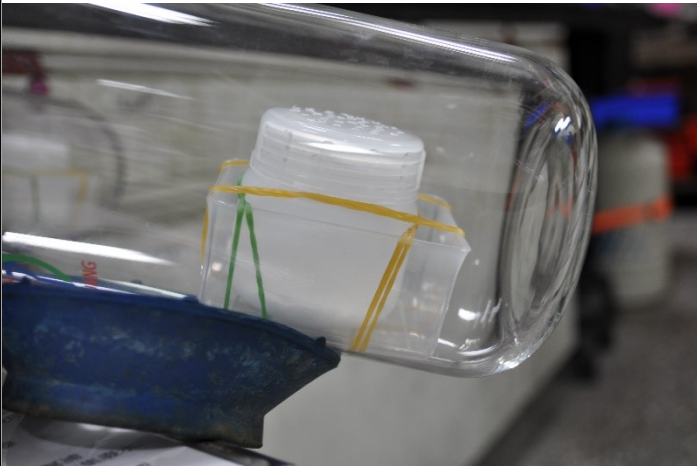
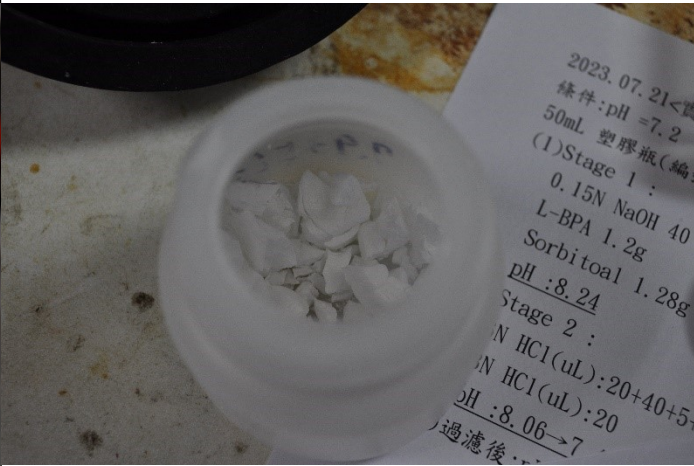
1. Experimental setup and work flow

1.1 60-mg scale preparation

 A photograph showing the experimental setup for 60-mg scale preparation. Two blue Vortex Genie 2 vortex mixers are visible on a lab bench, surrounded by various glassware, tubes, and a green bucket.	 A photograph showing several small, clear vials containing samples being immersed in a large, clear container filled with liquid nitrogen (N2).
a) setup for 60-mg scale preparation	b) immersion of the samples in liquid N2
 A photograph showing a clear plastic container holding several small, clear vials. The vials are labeled with handwritten text, including 'M 7.4' and 'M 9.0'. The container is resting on a wooden block.	
c) appearance of the lyophilized samples after 18 h	

1.2 1-gram scale preparation

 A photograph showing the experimental setup for 1-gram scale preparation. A white vortex mixer is visible on a lab bench, surrounded by various glassware and equipment.	 A photograph showing several small, clear vials containing samples being distributed over flasks. The vials are labeled with handwritten text, including 'M 7.4' and 'M 9.0'.
a) setup	b) samples distributed over flasks

	
c) lyophilization	d) appearance of the lyophilized samples

1.3 3-gram scale preparation

	
a) samples were distributed over 10 PP bottles of 50mL	b) the similar physical appearance as that of 1-gram scale samples

2. Tabulation for experimental protocol and spectroscopic and chromatographic data

2.1 Conditions and analyses for groups stored at -20 °C

Table 1. Summary of experimental protocol and physical data

	-20 °C stored						
Sample ID	1	2	3	4	5	6	Saline
Prepared date	0330 (0402)	0424	0330 (0402)	0403 (0404)	0405 (0406)	0405 (0406)	
BPA	60 mg	60 mg	60 mg	60 mg	60 mg	60 mg	
saccharide	sorb 1	sorb 2	man	xyl	sorb 5	xyl	
specification	Unfilter ed	normal	Unfilter ed	Unfilter ed	antioxid ation	antioxid ation	

					Na ₂ S ₂ O ₅	Na ₂ S ₂ O ₅	
ratio		100%	100%	100%	100%	100%	
Lyophi. Cond.		19 hr	21 °C, 20 h	19 °C 19 h	22 °C, 20 h	23 °C, 20 h	
Time elapsed		1205 250 d	1205 226 d	1205 250 d	1205 246 d	1205 244 d	
Physc. Prop.		Soft as foam	Soft solid	Soft as foam	Soft but easily stick on spoon	Soft but easily stick on spoon	Like chewing gum
Yield (%)	1 st		78.2			79.4	80.6
	2 nd	67.0	78.4	67.3	75.2	78.6	75.2
Dissolution (sec.) [§]		1	6	1	33	3	60
pH	designed	7.0	7.0	7.0	7.0	7.0	7.0
		6.95*	7.0	7.05†	6.99	6.93	7.09
	real	7.21	7.16	7.36	7.17	7.06	7.20
Durability (10 min)		clear	clear	clear	clear	clear	clear
Durability (2 days)		clear	Less precipit ate on tube	Less precipit ate on tube	Less precipit ate on tube	clear	Less precipit ate on tube
Durability (6 days)		clear	Ppt increase d	Ppt increase d	More Ppt	clear	Less ppt
Osmotic presseure	mOsm/kg	213	245	220	257	234	252
	ratio	0.73	0.84	0.76	0.89	0.81	0.87
¹ H-NMR		7.04	7.43	5.49	3.59	7.35	3.87
HPLC (purity, %)		96.38	98.04	95.97	98.68	97.79	98.95
Tyrosine (%)		1.13	0.08	0.35	0	0	0
Phenylalanine (%)		0	0	0	0	0	0

* acidified, inconsistent result

†a little bit turbid

Table 2. Summary of experimental protocol and physical data

-20 °C stored								
Sample ID	7	8	9	10	11	12	13	saline
Prepared date	0424	0405 (0406)	0423 (0424)	0330 (0402)	0403 (0404)	0405 (0406)	0423 (0424)	
BPA	60 mg	60 mg	60 mg	60 mg	60 mg	60 mg	60 mg	
saccharide	sorb 2	sorb 5	sorb 6	man	xyl	xyl	xyl	
specification	normal	antioxida tion Na ₂ S ₂ O ₅	-82°C	unfiltered	unfiltered	antioxida tion Na ₂ S ₂ O ₅ xyl 64+7.6 mg	-82°C	
ratio	100%	100%	100%	100%	100%	100%	100%	
Lyophi. Cond.	21 °C, 20 h	23 °C, 20 h	23 °C, 20 h	19 °C, 19 h	22 °C, 20 h	22 °C, 20 h	23 °C, 20 h	
Time elapsed	1016 175 d	1016 194 d	1016 176 d	1016 200 d	1016 196 d	1016 194 d	1016 176 d	
Physc. Prop.	crispy	crunchy	Crispy foam	Soft ice cream	Sticky candy	Maltose like	Clear sticky	
Yield (%)	1 st	78.2	71.5	75.8		76.0	84.7	

	2 nd	80.7	77.7	78.1	78.2	81.5	85.4	85.8	
Dissolution (sec.) [§]		2	2	3	2	50	80	10	
pH	designed	7.4	7.4	7.4	7.4	7.4	7.4	7.4	
		7.39	7.35	7.36	7.42	7.42	7.39	7.39	
	real	7.53	7.45	7.56	7.61	7.71	7.5	7.54	
Durability (6 days)		clear	clear	clear	clear	Clear but few plaque solid on tube	clear	Clear but few plaque solid on tube	
Osmotic presseure	mOsm/kg	243	218	226	252	240	247	275	290
	ratio	0.84	0.75	0.78	0.87	0.83	0.85	0.95	1
¹ H-NMR		15.75	14.60	15.87	7.60	7.04	6.49	7.27	
HPLC (purity, %)		97.94	95.70	93.94	5.63+90.97	95.48	93.47	96.50	
Tyrosine (%)		0	0	0	0	0	0	0	
Phenylalanine (%)		0.39	0	0	0	0	0	0	

Table 3. Summary of experimental protocol and physical data

-20 °C stored									
Sample ID		14	15	16	17	18	19	20	21
Prepared date		0326	0326	0326	0326	0611	0611	0611	0611
BPA		60 mg	60 mg	60 mg	60 mg	60 mg	60 mg	60 mg	60 mg
saccharide		fru	fru	xyl	xyl	sorb 4	sorb 4	man	xyl
specification		unfiltered fru 64+8 mg	unfiltered Antioxid ation Na ₂ S ₂ O ₅	unfiltered xyl 64+9 mg	unfiltered Antioxid ation Na ₂ S ₂ O ₅	SA (2M)	normal	SA (2M)	SA (2M)
ratio		100%	100%	100%	100%	100%	100%	100%	100%
Lyophi. Cond.		18 °C, 20 h	18 °C, 20 h	18 °C, 20 h	18 °C, 20 h	28 °C, 20h	28 °C, 20h	28 °C, 20h	28 °C, 20h
Time elapsed		1016 204 d	1016 204 d	1016 204 d	1016 204 d	1016 127 d	1016 127 d	1016 127 d	1016 127 d
Physc. Prop.		crunchy	crunchy	Maltose like	Candy like	crunchy	Softly crunchy	Crunchy with soft interior	Sticky crunchy
Yield (%)	1 st					87.3	88.1	78.5	88.3
	2 nd	82.7	77.7	104.8	95.0	89.0	87.5	80.6	89.8
Dissolution (sec.) [§]		5 Not very clear	10 Slightly turbid	60 suspensio n	70	10	20	3	15
pH	designed					7.6	7.0-7.4	7.6	7.6
		7.43	7.69	7.15	7.13	7.55	7.50	7.56	7.63
	real	7.78	7.32	7.88	7.30	7.67	7.68	7.78	7.71
Durability (day)		Turbid in 10 min Ppt after 1hr	Cloudy in 10 min Ppt in 1hr	Ppt in 10 min Cloudy in 1d	Cloudy in 1d	clear	clear	clear	clear
Durability (6 days)		White ppt, at least >=10 mg	White ppt, at least >=10 mg	Turbid more plaque solids	Turbid white solid on tube	clear	clear	clear	clear
Osmotic pressure	mOsm/kg	249	241	341	303	251	255	226	269
	ratio	0.86	0.83	1.18	1.05	0.87	0.88	0.78	0.93
¹ H-NMR		4.23	4.54	5.87	4.88	18.18	15.27	9.17	7.78

HPLC (purity, %)	94.00	95.26	91.90	93.00	97.40	96.30	97.32	97.52
Tyrosine (%)	0	0	0	0	0	0	0	0
Phenylalanine (%)	0	0	0	0	0	0	0	0

Table 4. Summary of experimental protocol and physical data

-20 °C stored									
Sample ID	22	23	24	25	26	27	28 (-1)	28 (-2)	28 (-3)
Prepared date	0330 (0402)	0405 (0406)	0611	0611	0423 (0424)	0423 (0424)	0905(09 25)		
BPA	60 mg	60 mg	60 mg	60 mg	60 mg	60 mg	1.2 g		
saccharide	man	sorb 5	sorb 4	sorb 4	sorb 6	sorb 6	sorb-SA		
specification	unfiltered	antioxidat ion Na ₂ S ₂ O ₅	SA (2M)	SA (2M)	-82°C	-82°C	1.2 g (1/20)		
ratio	100%	100%	100%	100%	100%	100%	5.4:1		
Lyophi. Cond.	19 °C, 19 h	23 °C, 20 h	28 °C, 20h	28 °C, 20h	23 °C, 20 h	23 °C, 20 h	20°C (18 hr) 25°C (6 hr)		
Time elapsed	0117 293 d	0117 287 d	0117 220 d	0117 220 d	0117 269 d	0117 269 d	0117 135 d		
Physc. Prop.	Soft foam	crunchy	Crunchy with soft interior	Crunchy with soft interior	Crunchy with soft interior	Crunchy with soft interior	Crunchy with soft interior		
Yield (%)	1 st	77.3	90.4	86	79.0	83.0	83.2		
	2 nd	76.7	77.7	91.7	86.8	81.9	84.7	83.5 86.3	
Dissolution (sec.) [§]		1	1	10	10	5	4	3	
pH	designed	7.2	7.2	7.4	7.4 7.0	7.2	7.0	7.2	
	real	7.24	7.12	7.37	7.04	7.25	7.03	7.28	
Durability (10 min)		clear	clear	clear	clear	clear	clear	clear	
Durability (2 days)		Less ppt	clear	clear	clear	clear	clear	clear	
Durability (6 days)		Less ppt	clear	clear	clear	clear	clear	clear	
Osmoti c presseu re	mOsm/ kg	239	246	258	245	249	263	260	
	ratio	0.82	0.85	0.89	0.84	0.86	0.91	0.90	
¹ H-NMR		5.71	10.05	14.93	8.93	12.27	10.10	10.31	
HPLC (purity, %)	1.63+0.70+9 4.97	0.78+94. 80	0.70+97 .82	0.68+98 .54	0.71+96 .58	0.91+95 .49	0.48+98. 35	0.51+98 .43	0.59+98 .12
Tyrosine (%)	0.28	0	0	0	0	0	0.42	0.32	0.31
Phenylalanine (%)	0	0	0	0	0	0	0	0	0

1.2 Conditions and analyses for groups stored at room temperature

Table 5. Summary of experimental protocol and physical data

Room-temperature stored																	
Sample	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	sa

ID																		line	
Prepared date		0709				0730								0810	0818			0721	
BPA		60 mg																1.2 g	
saccharide		Sorb+xyl				Sorb+SA								Sorbitol only	Sorb + SA		Sorbitol only		
specification		Sorb/xyl												double conc	Repeats (no. 7)	double conc	100 %		
ratio		1:2	1:1	2:1	3:1	2:1	5:2	3:1	4:1	5.4:1	7:1	3:1 (3.6:1)	7:1	100 %	3:1 (3.6:1)	3:1 (3.6:1)	1.28 g		
Lyophi. Cond.		26 °C for 20 h				20°C for 24 h								20 °C for 18 h and 25 °C for 6 h			20 °C 24 h		
Time elapsed		43 d				22 d								11 d	3 d		31 d		
Physc. Prop.		Solid as rock, hard to break				Ice cream like	foam	Soft foam	Soft foam	crunchy	Fluffy crunchy	Relatively solid foam	Fluffy crunchy	foam	Solid foam	crunchy	Crunchy foam		
Yield (%)		99.7	100.6	100.6	101.1	61.7	67.2	68.5	70.6	74.9	78.5	74.3	72.6	69.5	76.9	80.1	74.7		
Dissolution (sec.)§		120+120	120+120	120	120+60	1	1	5	2	7	10	2	3	15	1	5	3		
pH	designed	7.27.0	7.47.2	7.47.2	7.47.2	7.4			7.4	7.4				7.0	7.4	7.4	7.2		
								7.2¥						6.87§	7.23€	7.12€			
	real	7.38	7.56	7.49	7.55	7.68	7.60	7.52	7.46	7.59	7.55	7.54	7.62	6.99	7.31	7.20	7.41		
Durability (day)		>7														3	>7		
Osmotic pressure	mOsm/kg	366	365	345	342	200	210	219	224	234	237	249	222	482	224	511	306	284	
	ratio	1.289	1.285	1.215	1.204	0.70	0.74	0.77	0.79	0.82	0.83	0.88	0.78	1.70	0.79	1.80	1.08	1.0	
¹H-NMR		8.26	7.27	7.91	9.57	7.25	8.00	7.75	8.81	10.25	9.80	14.08	NA	7.87	7.46	7.47	13.07	NA	
HPLC (%)		NA	98.40	NA	99.44	5.90+90.57	NA	NA	NA	NA	3.75+96.20	5.97+93.19	NA	0.43+97.65	0.70+98.94	0.33+96.04	97.95	NA	

Tyrosine (%)		0		0.25	2.73					0	0.44		0.40	0.20	0	0.77	
Phenylalanine (%)		0		0	0.77					0	0		0.95	0	2.53	0	
SEM	NA															√	NA

Group of SA+sorb is relatively unstable and 96-97% purity left after 22 day-storage

Table 6. Summary of experimental protocol and physical data

Room-temperature stored								
Sample ID		45	46		47		48	49
Prepared date		0624	06 26	06 24	06 26	06 24	0624	0624
BPA		60 mg						
saccharide		Sorb+man*						
specification		Sorb/man						
ratio		2:3	1:1		2:1		3:1	4:1
Lyophi. Cond.		25 °C 18 h						
Time elapsed		74 d (RT)						
Physec. Prop.		Fragile, can be btoken	Bett er than that of 2:3	Soft foa m	As a ston e- like	Soft foa m	Fragile, can be broken	Fragile, can be broken
Yield (%)	1 st	89.6	83.7	82.8	86.9	94.0	90.7	96.8
	2 nd	73.2	85.0	85.3	88.7	94.8	91.2	90.9
Dissolution (sec.) [§]		30	>30 sec	22	60	10	50	30
pH	designed	7.4						
	real	7.53	7.44	7.40	7.18	7.45	7.20	7.55
Durability (day)		>2 d						
Osmotic pressure (74 d)	mOsm/kg	242	258	257	282	274	262	269
	ratio	0.88	0.94	0.94	1.02	1.00	0.95	0.98
¹ H-NMR (74d)		8.13	10.1 5			11.2 4	8.81	13.24
HPLC (%) 12/08 (166 d)		95.19		95.8 6	93.8 7	98.4 7	97.28	97.07
Tyrosine (%)		0		1.26	0.90	0	0	0
Phenylalanine (%)		0.05		0.05	0	0	0.27	0.09

Sorb+man is 95-97% purity and 0-0.27 Phe and 0-1.26 tyr appeared after 74-d storage.

Table 7. Summary of experimental protocol and physical data

Room-temperature stored								
Sample ID	50	51	52	53	54	55	56	57
Prepared date	0905	0908			0914	0915		
BPA	1.28 g	60 mg						
saccharide	Sorb+SA	Sorb	Man	Fru	Sorb	Sorb+ Man	Sorb+ Xyli	Sorb+ SA
specification	Precooling with Liq. N2 for 5 min				-82°C (24 hr)	Precooling with Liq. N2 for 5 min		
ratio	5.4:1	100%	100%	fru	100%	4:1	3:1	5.4:1

				65+3.3 mg							
Lyophi. Cond.		20 °C 18 h + 25 °C 6 h									
		4 x 50 mL bottles	1 x 50 mL bottle								
Time elapsed		0219 168 d	0219 164 d				0219 159 d	0219 158 d			
Physc. Prop.		Soft foam	Solid foam	Hygroscopic foam		Hard candy like	Shining with crunchy interior	Soft foam	Crunchy foam	Crunchy solid	
Yield (%)	1 st	83.7	68.8	68.6		53.9	46.9	60.9	54.2	73.7	
	2 nd	86.9	77.8	74.9		59.5	51.8	62.5	59.9	83.5	
Dissolution (sec.) [§]		2	53	5		30	25	5	14	35	
pH	designed	7.2	7.4	7.4		7.4	7.4	7.4	7.4	7.4	
		7.28	7.43	7.42		7.42	7.39	7.44	7.45	7.50	
	real	7.67	7.91	7.98		7.98	7.86	7.81	7.86	7.74	
Durability (10 min)		clear	clear	clear		clear	clear	clear	clear	clear	
Durability (2 days)		clear	clear	clear		clear	clear	clear	clear	clear	
Durability (6 days)		clear	clear	clear		clear	clear	clear	clear	clear	
Osmotic pression	mOsm/ kg	247	174	152		142	104	133	129	189	
	ratio	0.85	0.60	0.52		0.49	0.36	0.46	0.44	0.65	
¹ H-NMR		10.93	19.61	7.81		8.03	14.60	14.49	14.71	12.35	
HPLC (purity, %)		3.03+94.48	2.56+93.71	28-1	28-2	28-3	1.47+97.96	0.88+97.74	0.41+99.03	0.65+98.11	3.58+95.98
				1.89+93.70	1.60+94.84	1.30+95.02					
Tyrosine (%)		0.83	0	2.25	2.21	1.96	0	0	0	0	0.09
Phenylalanine (%)		0	0	0	0	0	0	0	0	0	0

Table 8. Summary of experimental protocol and physical data

Room-temperature stored					
Sample ID	58	59	60	61	Saline
Prepared date	0429				
BPA	60 mg				
saccharide	Sorb				
specification	antioxidation Na ₂ S ₂ O ₅				
ratio	100%				
Lyophi. Cond.	Lyophilized thrice				
Time elapsed	0722 (81 d)				
Physc. Prop.	Fragile, can be	Fragile, can be	As a rock	As a rock	

		broken	easily broken			
Yield (%)	1 st	>100	79.8	88.7	>100	
	2 nd	87.9	90.3	84.7	89.5	
	3 rd	86.2	89.5	81.4	86.3	
	4 th	89.8	92.4	86.0	90.2	
Dissolution (sec.) [§]		31	22	24	30	
pH	designed	7.6	7.4	7.2	7.0	
		7.47	7.32	7.21	7.02	
	real	7.80	7.66	7.54	7.28	
Durability (day)		>2 d				
Osmotic pression (81 d)	mOsm/kg	225	245	206	245	290
	ratio	0.81	0.89	0.75	0.89	1
¹ H-NMR 12/06 (222 d)		21.98	18.02	18.69	11.70	
HPLC 12/08 (224 d)		98.96	98.22	98.36	97.46	
Tyrosine (%)		0	0	0	0	
Phenylalanine (%)		0.09	0.05	0.06	0.05	

Table 9. Summary of experimental protocol and physical data

Room-temperature stored										
Sample ID		62	63	64	65	66	67	68	69	70
Prepared date		0429 (0502)	0721	0828	0430	0430	0710	0710	0922	0829
BPA		60 mg	1.2g	120 mg	60 mg	60 mg	60 mg	60 mg	60 mg	60 mg
saccharide		Sorb	sorb	sorb	sorb	xyl	sorb+xy l	sorb+xy l	sorb+xy l	Sorb + SA
specification		normal	100 %	double conc.	test	test	-196 °C	-82 °C	sorb/xyl	5.4:1
ratio		100%	1.28g	100%	100%	100%	1:1	1:1	1:1	5.4:1
Lyophi. Cond.		Lyophil ized thrice	20 °C 24 h	20 °C for 15 h and 25 °C for 4 h			16°C 18 hr	16°C 18 hr	20 °C for 18 h and 25 °C for 6 h	20 °C for 21 h and 25 °C for 4 h
Time elapsed		1205 221 d	1205 138 d	1205 100 d	1205 221 d	1205 221 d	1205 149 d	1205 149 d	1205 75 d	1205 99 d
Physc. Prop.		Yellow white solid	Fragile foam	crunchy	Pale yellow solidified candy	Yellow foam like	Maltose like	Solidified candy	Crunchy chip	Crunchy foam
Yield (%)	1 st	81.4	74.7	58.5	88.7	80.6	83.9	83.9	79.8	68.1
	2 nd	83.0	78.6	61.3	95.3	88.8	86.3	86.3	83.2	74.6
	3 rd	80.6					91.0	89.8		
	4 th	97.5								
Dissolution (sec.) [§]		60+25	3	3	60+80	50	60+180	60+90	5	3
pH	designed	7.4	7.2	7.4	7.0	7.0	7.2	7.2	7.4	7.4
		7.42	7.29	7.46	7.0	7.04	7.19	7.17	7.41	7.38
	real	7.35	7.42	7.65	7.15	7.19	7.56	7.55	7.58	7.56
Durability (10 min)		clear	clear	clear	clear	Pale yellow	clear	clear	clear	clear
Durability (2 days)		clear	clear	clear	clear	Less plaque	clear	clear	clear	clear
Durability (6 days)		clear	clear	clear	plaque	Ppt on tube	clear	clear	clear	clear
Osmotic	mOsm/kg	241	258	364	280	287	267	265	259	207

pression	ratio	0.83	0.89	1.26	0.97	0.99	0.92	0.91	0.89	0.71
¹ H-NMR		11.17	12.58	18.87	7.38	3.40	10.81	8.55	10.81	9.17
HPLC (purity, %)		90.86	96.97	95.61	91.61	92.88	90.37	96.39	97.28	90.39
Tyrosine (%)		0	0	0	0	0	0	0	0	1.77
Phenylalanine (%)		6.36	0	0	6.85	0.27	6.23	0.34	0	0

§ shaking in 60 sec followed by vibration

Table 10. Summary of experimental protocol and physical data

Room-temperature stored										
Sample ID		71	72	73	74	75 (-1)	75 (-2)	76	77	78
Prepared date		0829	0829	0710	0429 (0502)	0908		1007	1102	1201
BPA		60 mg	60 mg	60 mg	60 mg	60 mg		60 mg	60 mg	60 mg
saccharide		Sorb + SA	Sorb + SA	sorb+xy l	Sorb	Xyli		sorb	sorb	sorb
specification		5.4:1	5.4:1	-196 °C; lyophilized twice	normal	Precooling with Liq. N2 for 5 min		6.4 M GA	6.4 M GA	Antioxi dation AA
ratio		5.4:1	5.4:1	3:1	100%	100%		100%	100%	100%
Lyophi. Cond.		20 °C for 21 h and 25 °C for 4 h	20 °C for 21 h and 25 °C for 4 h	16°C 18 hr	Lyophilized three times	20 °C 18 h + 25 °C 6 h 1 x 50 mL bottle		20°C (18 hr) 25°C (6 hr)	20°C (18 hr) 25°C (6 hr)	20°C (18 hr) 25°C (6 hr)
Time elapsed		0117 141 d	0117 141 d	0117 191 d	0117 263 d	0117 131 d		0117 102 d	0117 76 d	0117 47 d
Physc. Prop.		Shrinkage, crunch	Soft cake	Pale yellow candy	Pale yellow candy	Sticky solid, hygroscopic		Crunchy foam	Crunchy foam	fragile
Yield (%)	1 st	73.5	73.3	62.93	84.7	62.7		84.1	71.2	73.7
	2 nd	85.0	82.0	68.5	84.7	58.9		89.5	74.6	76.3
	3 rd			72.7	81.4					
	4 th				77.3					
Dissolution (sec.)§		45	1	60+240	60+300	2		1	1	1
pH	designed	7.4	7.4	7.4	7.2	7.4		7.4	7.4	7.4
		7.40	7.42	7.24 (6.99)	7.22	7.43		7.36*	7.40	7.43
	real	7.65	7.68	7.54	7.63	7.90		7.47	7.47	7.56
Durability (10 min)		clear	clear	clear	clear	Pale yellow		clear	clear	clear
Durability (2 days)		clear	clear	clear	clear	Pale yellow		clear	clear	clear
Durability (6 days)		clear	clear	clear	clear	Pale yellow		clear	clear	clear
Osmotic pression	mOsm/kg	224	225	207	232	162		259	214	233
	ratio	0.77	0.78	0.71	0.80	0.56		0.89	0.74	0.80
¹ H-NMR		10.58	10.47	9.85	12.20	3.96		12.20	11.90	13.61
HPLC (purity, %)		4.54+9	1.39+ 9	3.20+88	2.31+90	8.90+0.	8.77+0.	0.37+98	0.20+98	3.17+96

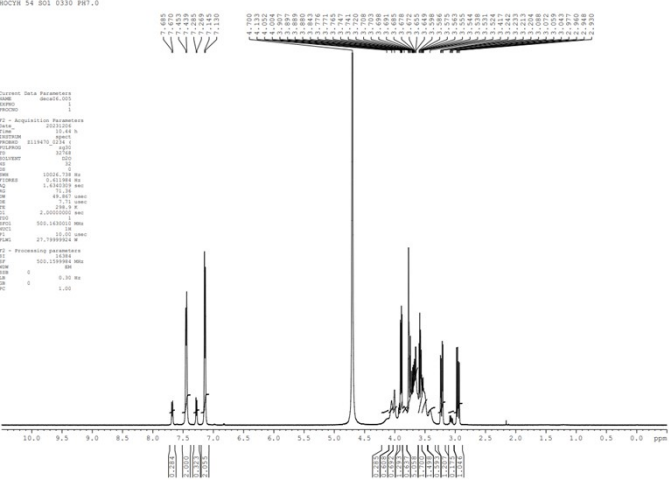
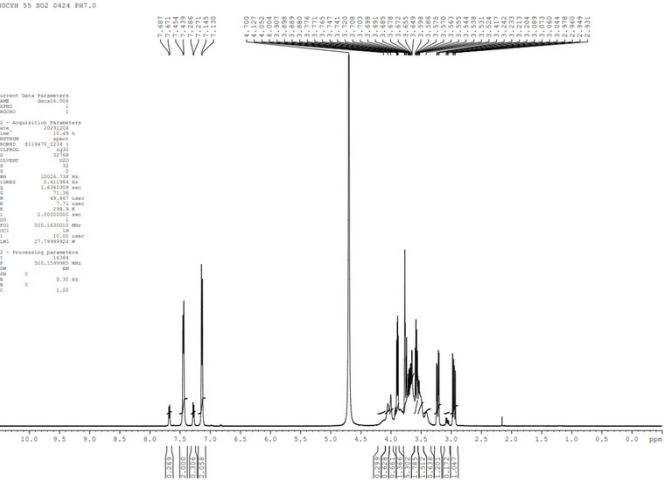
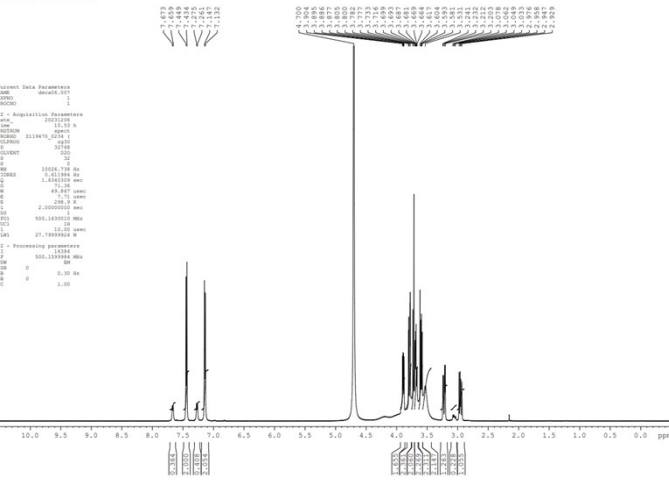
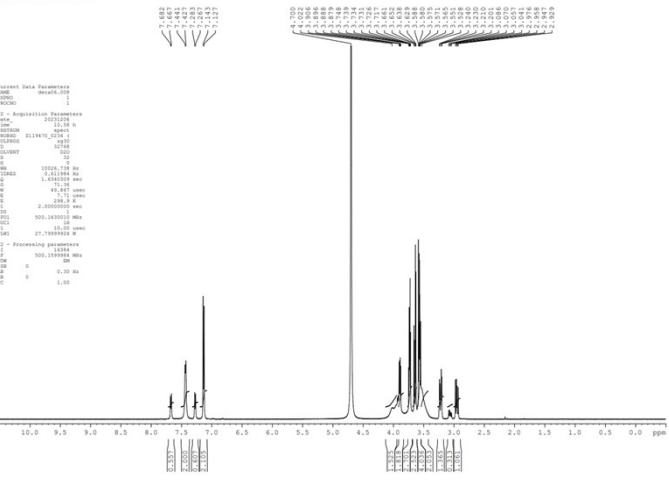
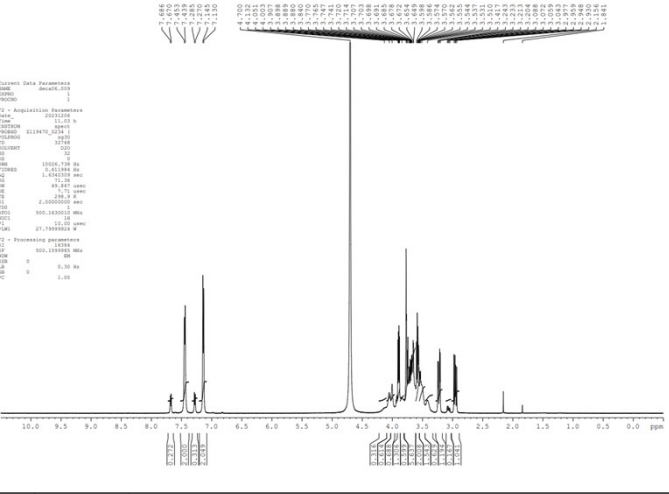
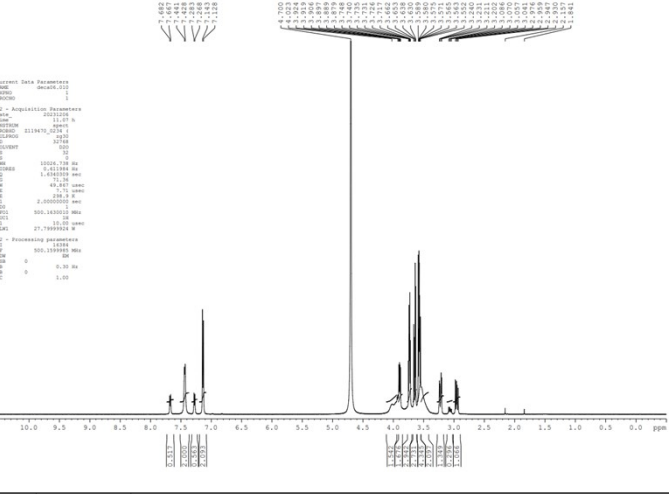
	0.48	2.38	.98	.26	04+0.04 +90.51	11+89.8 8	.39	.47	.17
Tyrosine (%)	2.97	2.59	0	0	0	0	0	0	0.08
Phenylalanine (%)	0	0	6.07	6.36	0	0	0	0	0
SEM					√				

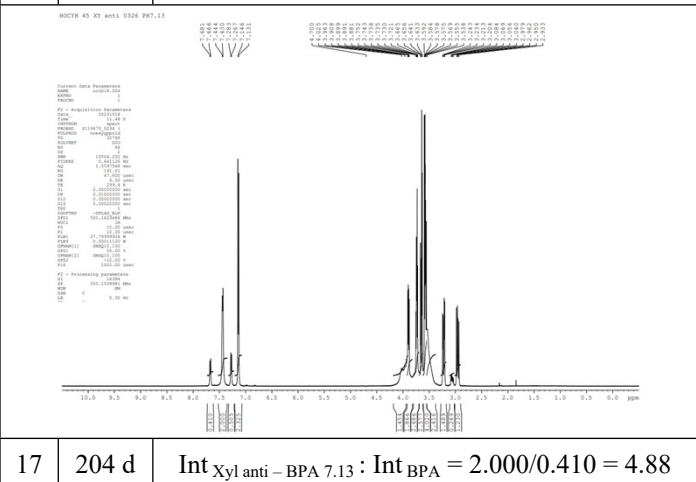
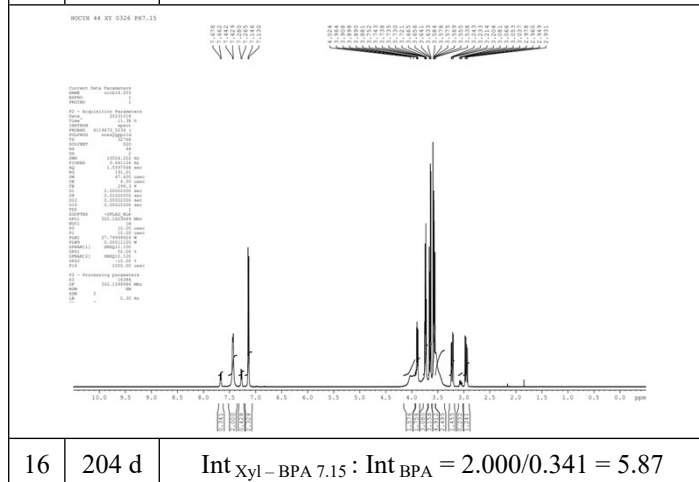
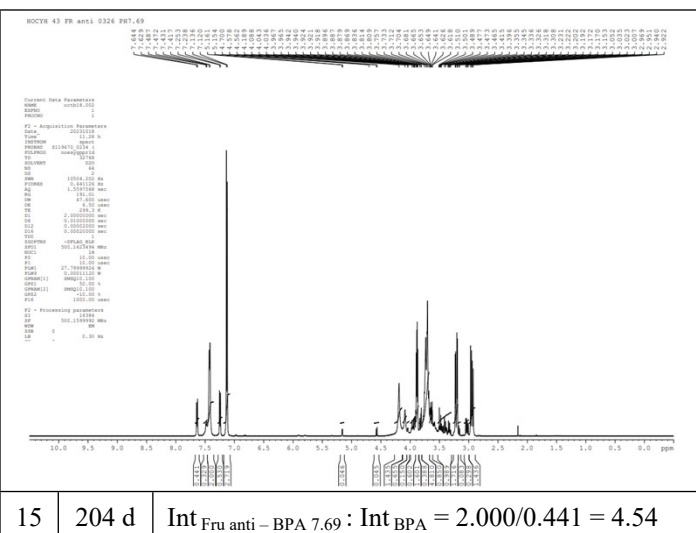
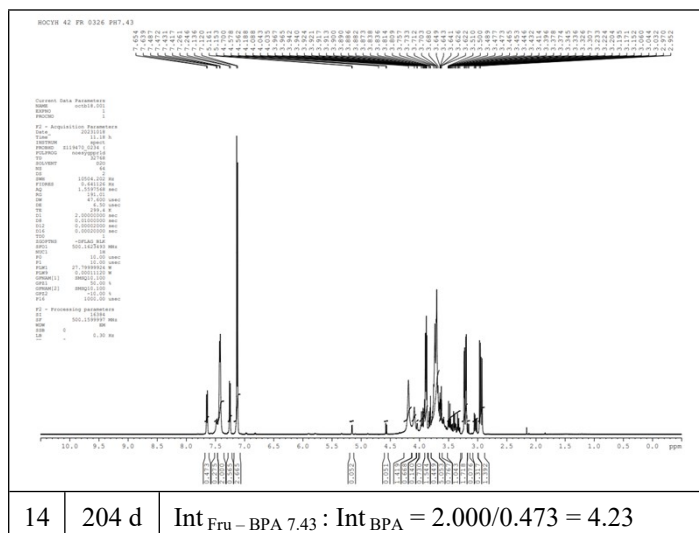
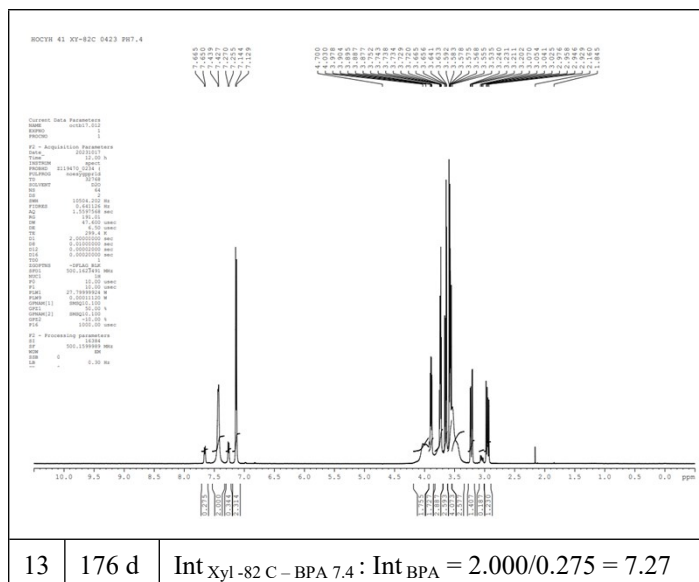
Table 11. Summary of experimental protocol and physical data

Room-temperature stored									
Sample ID		79		80		1	82	83	
Prepared date		0818		1201		1102	0828	0828	
BPA		120 mg		60 mg		60 mg	120 mg	120 mg	
saccharide		Sorb + SA		sorb		sorb	sorb	sorb	
specification		double conc		Antioxidatio n AA		6.4 M GA	double conc.	double conc.	
ratio		3:1 (3.6:1)		100%		100%	100%	100%	
Lyophi. Cond.		20 °C (18 hr) 25 °C (6 hr)		20°C (18 hr) 25°C (6 hr)		20°C (18 hr) 25°C (6 hr)	20°C (15 hr) 25°C (4 hr)	20°C (15 hr) 25°C (4 hr)	
Time elapsed		0219 187 d		0219 81 d		0219 110 d	0219 177 d	0219 177 d	
Physc. Prop.		Crunchy candy		chunchy		Solid foam	Crunch y foam	Solidifie d foam	
Yield (%)	1 st	77.2		64.7		70.3	72.3	72.9	
	2 nd	77.6		68.4		74.7	76.8	77.1	
Dissolution (sec.) [§]		3		1		1	4	5	
pH	designed	7.4		7.6		7.6	7.6	7.2	
		7.08 ^ε		7.54		7.57	7.57	7.23	
	real	7.34		7.69		7.67	7.84	7.50	
Durability (10 min)		clear		clear		clear	clear	clear	
Durability (2 days)		clear		clear		clear	clear	clear	
Durability (6 days)		clear		clear		clear	clear	clear	
Osmotic presseure	mOsm/kg	383		193		212	423	435	
	ratio	1.32		0.67		0.73	1.46	1.5	
¹ H-NMR		9.09		15.75		12.99	23.53	15.50	
HPLC (purity, %)		85-1	85-2	86-1	86-2	0.39+99. 24	0.09+9 9.48	0.38+98. 25	
		2.30+	2.26+	2.09	2.09				
		0.12+	0.12+	+97.	+97.				
		97.03	97.40	58	58				
Tyrosine (%)		0	0	0		0	0	0	
Phenylalanine (%)		0	0	0		0	0	0	

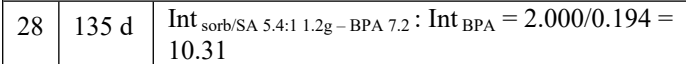
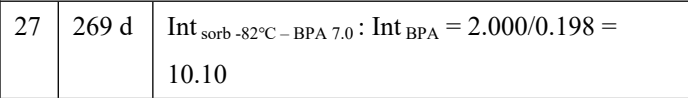
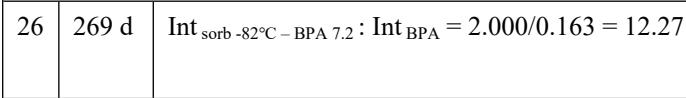
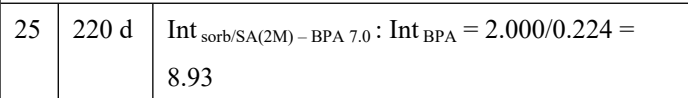
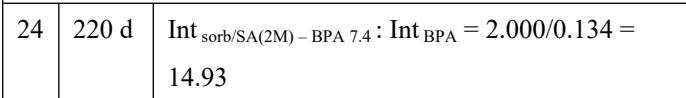
3. ¹H-NMR analyses of the lyophilized samples

2.1 samples that were stored at - 20 °C

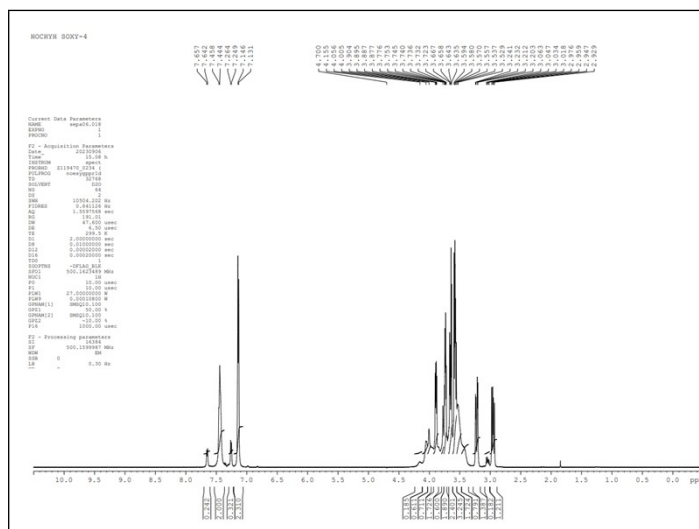
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1	250 d	$\text{Int}_{\text{sorb1}} - \text{BPA } 7.0 : \text{Int}_{\text{BPA}} = 2.000/0.284 = 7.04$	2	266 d	$\text{Int}_{\text{sorb2}} - \text{BPA } 7.0 : \text{Int}_{\text{BPA}} = 2.000/0.269 = 7.43$
NOEYR 56 8A 0330 PWT.0  Current Data Parameters NAME: 8A0330 EXPNO: 1 PROCNO: 1 F2 - Acquisition Parameters Date_: 20220228 Time: 12:45 INSTRUM: spect PROBHD: BBO-5mm PULPROG: zgpg30 TD: 65536 SOLVENT: DMS AQ: 0.02000000 RG: 327.68 DA: 10204.738 Hz FIDRES: 0.411384 Hz AQ: 1.4021028 sec RG: 49.407 GB: 0 PC: 2.0000000 sec SFO: 500.1350013 MHz P1: 27.7995000 sec P2 - Processing parameters SI: 32768 SF: 500.1350013 MHz WDW: EM SSB: 0 LB: 0.30 Hz GB: 0 PC: 0.00			NOEYR 57 XY 0405 PWT.0  Current Data Parameters NAME: XY0405 EXPNO: 1 PROCNO: 1 F2 - Acquisition Parameters Date_: 20220228 Time: 12:45 INSTRUM: spect PROBHD: BBO-5mm PULPROG: zgpg30 TD: 65536 SOLVENT: DMS AQ: 0.02000000 RG: 327.68 DA: 10204.738 Hz FIDRES: 0.411384 Hz AQ: 1.4021028 sec RG: 49.407 GB: 0 PC: 2.0000000 sec SFO: 500.1350013 MHz P1: 27.7995000 sec P2 - Processing parameters SI: 32768 SF: 500.1350013 MHz WDW: EM SSB: 0 LB: 0.30 Hz GB: 0 PC: 0.00		
3	250 d	$\text{Int}_{\text{man}} - \text{BPA } 7.0 : \text{Int}_{\text{BPA}} = 2.000/0.364 = 5.49$	4	246 d	$\text{Int}_{\text{xy1}} - \text{BPA } 7.0 : \text{Int}_{\text{BPA}} = 2.000/0.557 = 3.59$
NOEYR 58 805 anti 0405 PWT.0  Current Data Parameters NAME: 805anti EXPNO: 1 PROCNO: 1 F2 - Acquisition Parameters Date_: 20220228 Time: 12:45 INSTRUM: spect PROBHD: BBO-5mm PULPROG: zgpg30 TD: 65536 SOLVENT: DMS AQ: 0.02000000 RG: 327.68 DA: 10204.738 Hz FIDRES: 0.411384 Hz AQ: 1.4021028 sec RG: 49.407 GB: 0 PC: 2.0000000 sec SFO: 500.1350013 MHz P1: 27.7995000 sec P2 - Processing parameters SI: 32768 SF: 500.1350013 MHz WDW: EM SSB: 0 LB: 0.30 Hz GB: 0 PC: 0.00			NOEYR 59 XY anti 0405 PWT.0  Current Data Parameters NAME: XYanti EXPNO: 1 PROCNO: 1 F2 - Acquisition Parameters Date_: 20220228 Time: 12:45 INSTRUM: spect PROBHD: BBO-5mm PULPROG: zgpg30 TD: 65536 SOLVENT: DMS AQ: 0.02000000 RG: 327.68 DA: 10204.738 Hz FIDRES: 0.411384 Hz AQ: 1.4021028 sec RG: 49.407 GB: 0 PC: 2.0000000 sec SFO: 500.1350013 MHz P1: 27.7995000 sec P2 - Processing parameters SI: 32768 SF: 500.1350013 MHz WDW: EM SSB: 0 LB: 0.30 Hz GB: 0 PC: 0.00		
5	244 d	$\text{Int}_{\text{sorb5 anti}} - \text{BPA } 7.0 : \text{Int}_{\text{BPA}} = 2.000/0.272 = 7.35$	6	244 d	$\text{Int}_{\text{xy1 anti}} - \text{BPA } 7.0 : \text{Int}_{\text{BPA}} = 2.000/0.517 = 3.87$



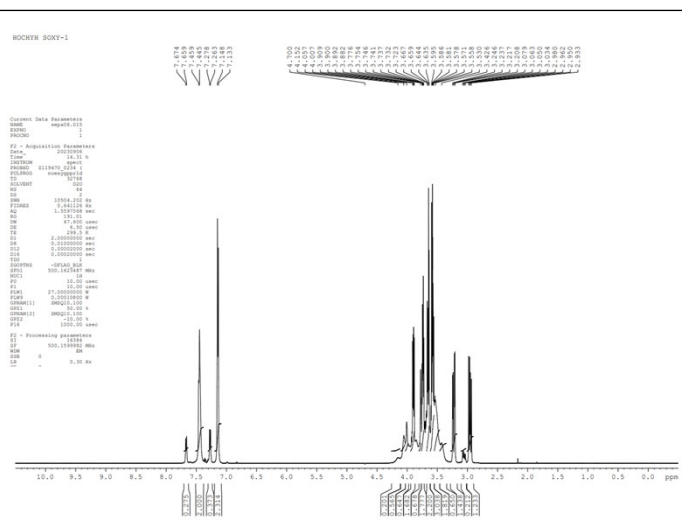
NOCTH 46 304 SA 0611 087.6 Current Data Parameters NAME: NOCTH 46 304 SA 0611 087.6 EXPNO: 1 PROCNO: 1 PROCNAME: NOCTH 46 304 SA 0611 087.6 PROBHD: 5 mm QNP 1H/13 PULPROG: zgpg30 TD: 65536 RG: 320 SD: 1.00000000 DE: 0.00000000 TE: 300.2 K F2 - Acquisition Parameters NAME: NOCTH 46 304 SA 0611 087.6 EXPNO: 1 PROCNO: 1 PROCNAME: NOCTH 46 304 SA 0611 087.6 PROBHD: 5 mm QNP 1H/13 PULPROG: zgpg30 TD: 65536 RG: 320 SD: 1.00000000 DE: 0.00000000 TE: 300.2 K F2 - Processing parameters NAME: NOCTH 46 304 SA 0611 087.6 EXPNO: 1 PROCNO: 1 PROCNAME: NOCTH 46 304 SA 0611 087.6 PROBHD: 5 mm QNP 1H/13 PULPROG: zgpg30 TD: 65536 RG: 320 SD: 1.00000000 DE: 0.00000000 TE: 300.2 K			NOCTH 47 304 SA 0611 087.6 Current Data Parameters NAME: NOCTH 47 304 SA 0611 087.6 EXPNO: 1 PROCNO: 1 PROCNAME: NOCTH 47 304 SA 0611 087.6 PROBHD: 5 mm QNP 1H/13 PULPROG: zgpg30 TD: 65536 RG: 320 SD: 1.00000000 DE: 0.00000000 TE: 300.2 K F2 - Acquisition Parameters NAME: NOCTH 47 304 SA 0611 087.6 EXPNO: 1 PROCNO: 1 PROCNAME: NOCTH 47 304 SA 0611 087.6 PROBHD: 5 mm QNP 1H/13 PULPROG: zgpg30 TD: 65536 RG: 320 SD: 1.00000000 DE: 0.00000000 TE: 300.2 K F2 - Processing parameters NAME: NOCTH 47 304 SA 0611 087.6 EXPNO: 1 PROCNO: 1 PROCNAME: NOCTH 47 304 SA 0611 087.6 PROBHD: 5 mm QNP 1H/13 PULPROG: zgpg30 TD: 65536 RG: 320 SD: 1.00000000 DE: 0.00000000 TE: 300.2 K		
18	127 d	$\text{Int}_{\text{Sorb4 SA} - \text{BPA } 7.6} : \text{Int}_{\text{BPA}} = 2.000/0.110 = 18.18$	19	127 d	$\text{Int}_{\text{Sorb4} - \text{BPA } 7.5} : \text{Int}_{\text{BPA}} = 2.000/0.131 = 15.27$
NOCTH 48 304 SA 0611 087.6 Current Data Parameters NAME: NOCTH 48 304 SA 0611 087.6 EXPNO: 1 PROCNO: 1 PROCNAME: NOCTH 48 304 SA 0611 087.6 PROBHD: 5 mm QNP 1H/13 PULPROG: zgpg30 TD: 65536 RG: 320 SD: 1.00000000 DE: 0.00000000 TE: 300.2 K F2 - Acquisition Parameters NAME: NOCTH 48 304 SA 0611 087.6 EXPNO: 1 PROCNO: 1 PROCNAME: NOCTH 48 304 SA 0611 087.6 PROBHD: 5 mm QNP 1H/13 PULPROG: zgpg30 TD: 65536 RG: 320 SD: 1.00000000 DE: 0.00000000 TE: 300.2 K F2 - Processing parameters NAME: NOCTH 48 304 SA 0611 087.6 EXPNO: 1 PROCNO: 1 PROCNAME: NOCTH 48 304 SA 0611 087.6 PROBHD: 5 mm QNP 1H/13 PULPROG: zgpg30 TD: 65536 RG: 320 SD: 1.00000000 DE: 0.00000000 TE: 300.2 K			NOCTH 49 304 SA 0611 087.6 Current Data Parameters NAME: NOCTH 49 304 SA 0611 087.6 EXPNO: 1 PROCNO: 1 PROCNAME: NOCTH 49 304 SA 0611 087.6 PROBHD: 5 mm QNP 1H/13 PULPROG: zgpg30 TD: 65536 RG: 320 SD: 1.00000000 DE: 0.00000000 TE: 300.2 K F2 - Acquisition Parameters NAME: NOCTH 49 304 SA 0611 087.6 EXPNO: 1 PROCNO: 1 PROCNAME: NOCTH 49 304 SA 0611 087.6 PROBHD: 5 mm QNP 1H/13 PULPROG: zgpg30 TD: 65536 RG: 320 SD: 1.00000000 DE: 0.00000000 TE: 300.2 K F2 - Processing parameters NAME: NOCTH 49 304 SA 0611 087.6 EXPNO: 1 PROCNO: 1 PROCNAME: NOCTH 49 304 SA 0611 087.6 PROBHD: 5 mm QNP 1H/13 PULPROG: zgpg30 TD: 65536 RG: 320 SD: 1.00000000 DE: 0.00000000 TE: 300.2 K		
20	127 d	$\text{Int}_{\text{Man SA} - \text{BPA } 7.6} : \text{Int}_{\text{BPA}} = 2.000/0.218 = 9.17$	21	127 d	$\text{Int}_{\text{Xyl SA} - \text{BPA } 7.6} : \text{Int}_{\text{BPA}} = 2.000/0.257 = 7.78$
NOCTH 69 304 SA 0611 087.6 Current Data Parameters NAME: NOCTH 69 304 SA 0611 087.6 EXPNO: 1 PROCNO: 1 PROCNAME: NOCTH 69 304 SA 0611 087.6 PROBHD: 5 mm QNP 1H/13 PULPROG: zgpg30 TD: 65536 RG: 320 SD: 1.00000000 DE: 0.00000000 TE: 300.2 K F2 - Acquisition Parameters NAME: NOCTH 69 304 SA 0611 087.6 EXPNO: 1 PROCNO: 1 PROCNAME: NOCTH 69 304 SA 0611 087.6 PROBHD: 5 mm QNP 1H/13 PULPROG: zgpg30 TD: 65536 RG: 320 SD: 1.00000000 DE: 0.00000000 TE: 300.2 K F2 - Processing parameters NAME: NOCTH 69 304 SA 0611 087.6 EXPNO: 1 PROCNO: 1 PROCNAME: NOCTH 69 304 SA 0611 087.6 PROBHD: 5 mm QNP 1H/13 PULPROG: zgpg30 TD: 65536 RG: 320 SD: 1.00000000 DE: 0.00000000 TE: 300.2 K			NOCTH 70 304 SA 0611 087.6 Current Data Parameters NAME: NOCTH 70 304 SA 0611 087.6 EXPNO: 1 PROCNO: 1 PROCNAME: NOCTH 70 304 SA 0611 087.6 PROBHD: 5 mm QNP 1H/13 PULPROG: zgpg30 TD: 65536 RG: 320 SD: 1.00000000 DE: 0.00000000 TE: 300.2 K F2 - Acquisition Parameters NAME: NOCTH 70 304 SA 0611 087.6 EXPNO: 1 PROCNO: 1 PROCNAME: NOCTH 70 304 SA 0611 087.6 PROBHD: 5 mm QNP 1H/13 PULPROG: zgpg30 TD: 65536 RG: 320 SD: 1.00000000 DE: 0.00000000 TE: 300.2 K F2 - Processing parameters NAME: NOCTH 70 304 SA 0611 087.6 EXPNO: 1 PROCNO: 1 PROCNAME: NOCTH 70 304 SA 0611 087.6 PROBHD: 5 mm QNP 1H/13 PULPROG: zgpg30 TD: 65536 RG: 320 SD: 1.00000000 DE: 0.00000000 TE: 300.2 K		
22	293 d	$\text{Int}_{\text{Man} - \text{BPA } 7.2} : \text{Int}_{\text{BPA}} = 2.000/0.350 = 5.71$	23	287 d	$\text{Int}_{\text{sorb anti} - \text{BPA } 7.2} : \text{Int}_{\text{BPA}} = 2.000/0.199 = 10.05$



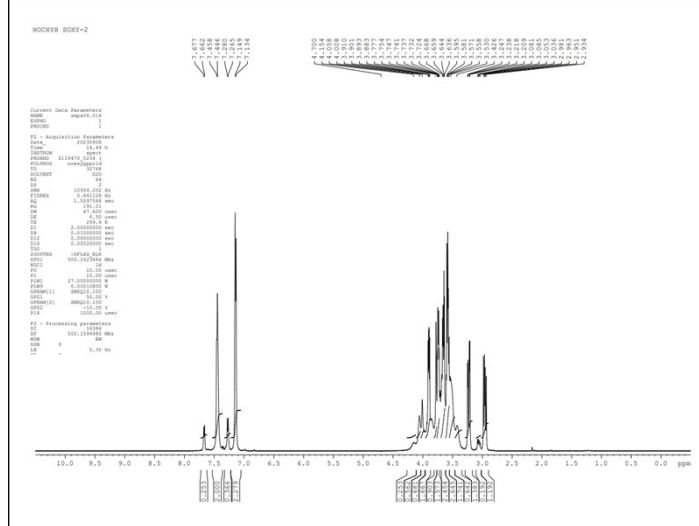
2.2 ¹H-NMR data of lyophilized samples from storage at room temperature.



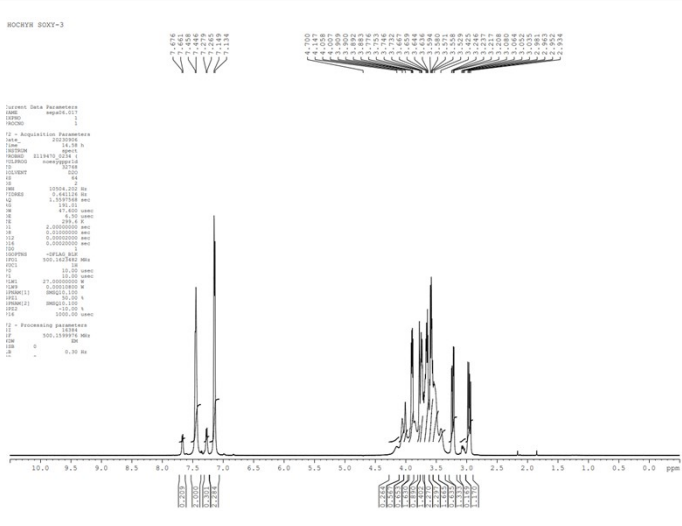
29	43 d	$\text{Int}_{\text{Sorb} + \text{Xyli (1:2)} - \text{BPA}} : \text{Int}_{\text{BPA}} =$ $2.000/0.242 = 8.26$
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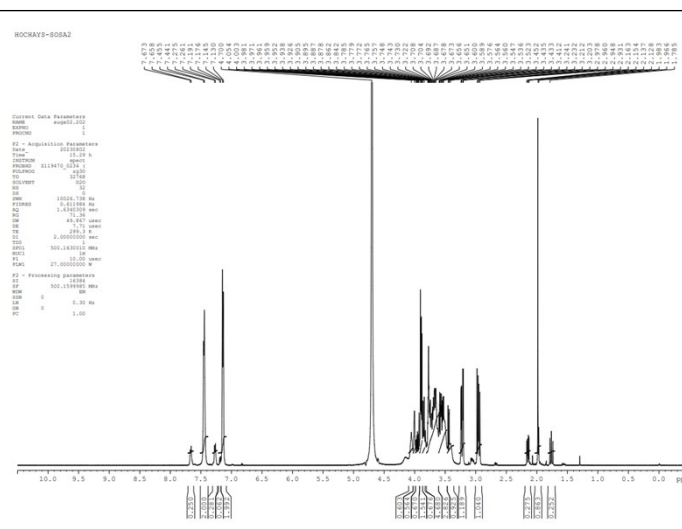
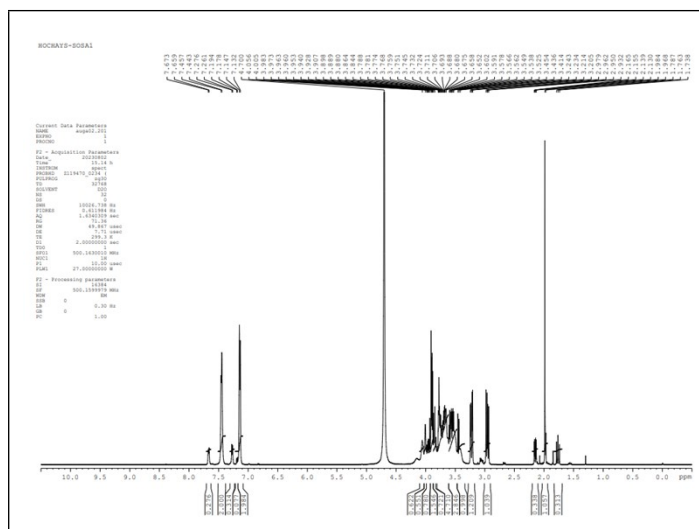
30	43 d	$\text{Int}_{\text{Sorb} + \text{Xyli (1:1)} - \text{BPA}} \frac{7.2}{7.0} : \text{Int}_{\text{BPA}} =$ $2.000/0.275 = 7.27$
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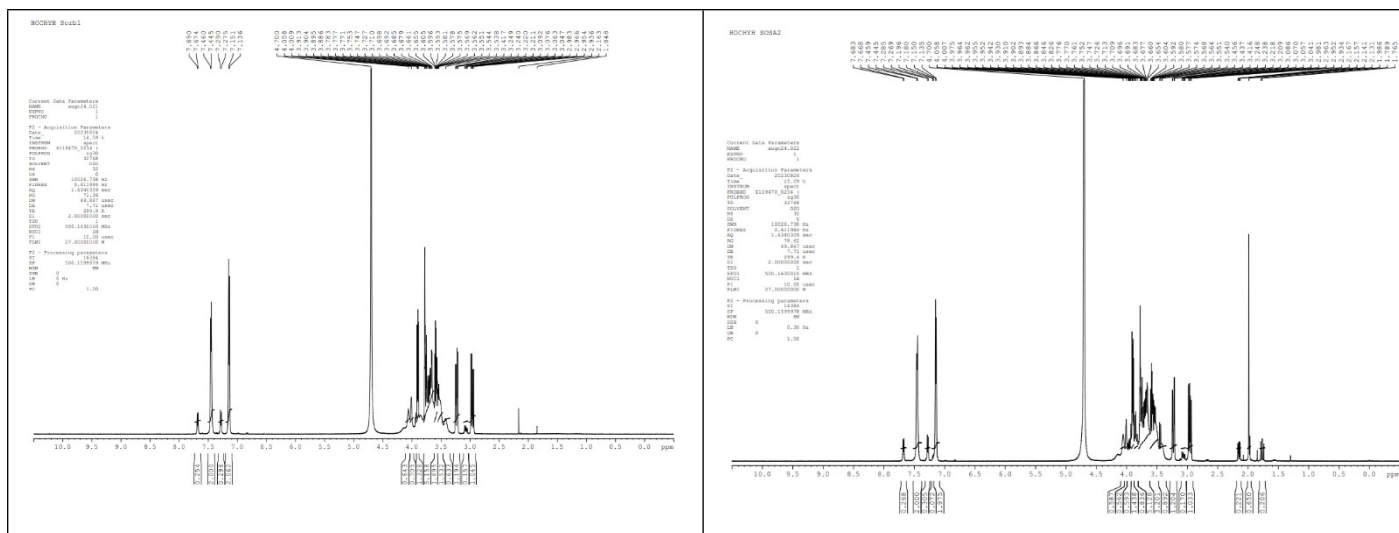


19	43 d	$\text{Int}_{\text{Sorb} + \text{Xyli (2:1)} - \text{BPA}} \frac{7.2}{7.0} : \text{Int}_{\text{BPA}} =$ $2.000/0.253 = 7.91$
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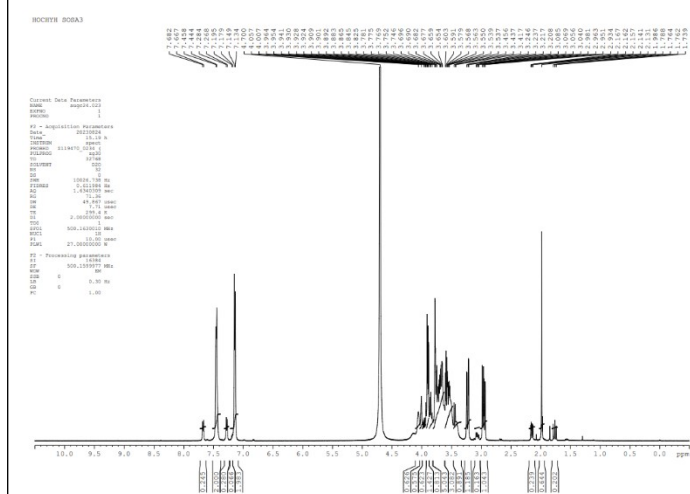
20	43 d	$\text{Int}_{\text{Sorb} + \text{Xyli (3:1)} - \text{BPA}} \frac{7.2}{7.0} : \text{Int}_{\text{BPA}} =$ $2.000/0.209 = 9.57$
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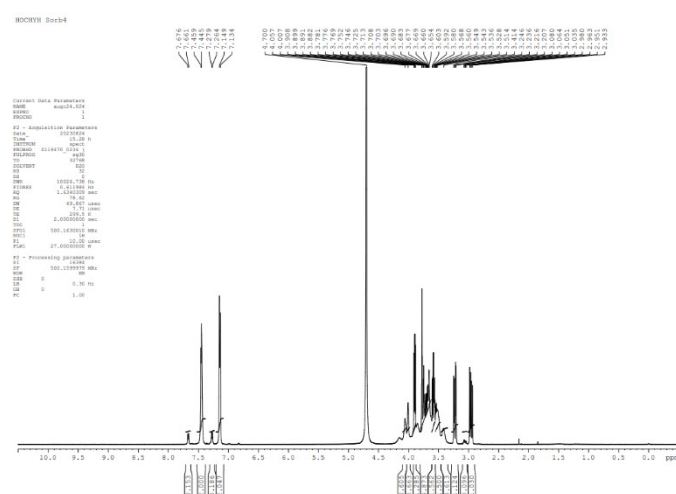


41 11 d Int Sorb only double conc – BPA 7.0 : Int BPA = 2.000 / 0.254 = 7.87

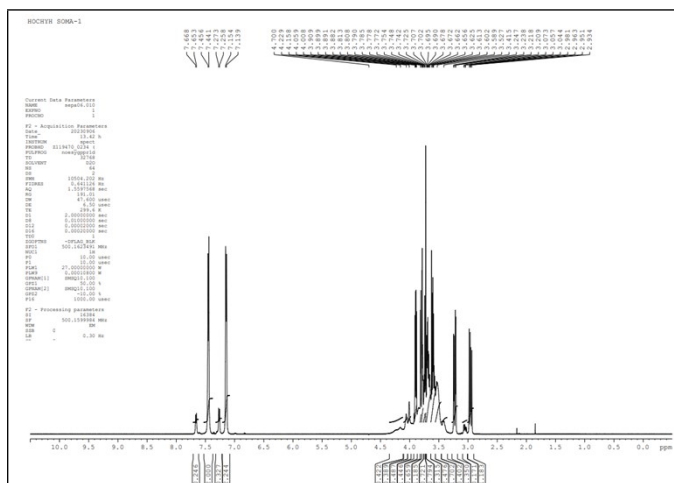
42 3 d Int Sorb SA (3.6:1) repeat – BPA 7.4 : Int BPA = 2.000 / 0.268 = 7.46



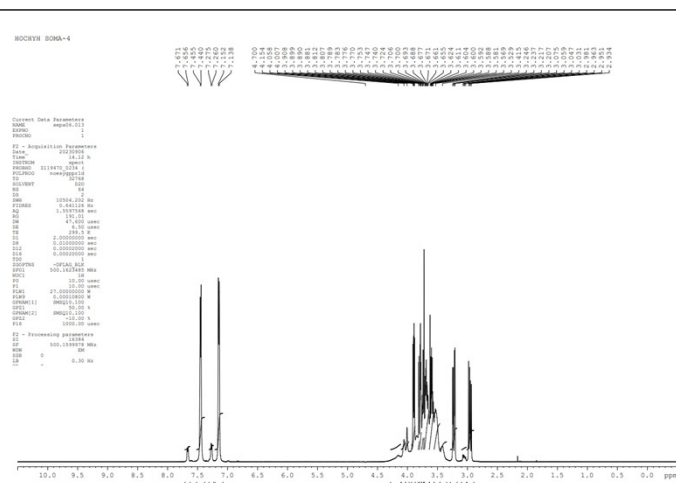
43 3 d Int Sorb SA (3.6:1) double conc – BPA 7.4 : Int BPA = 2.000 / 0.245 = 7.46



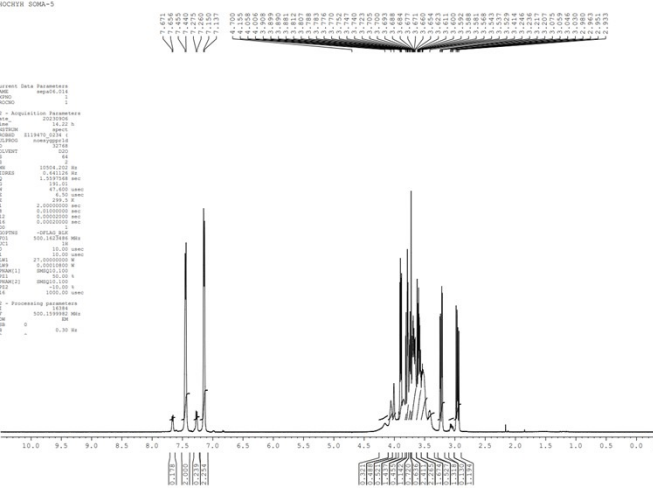
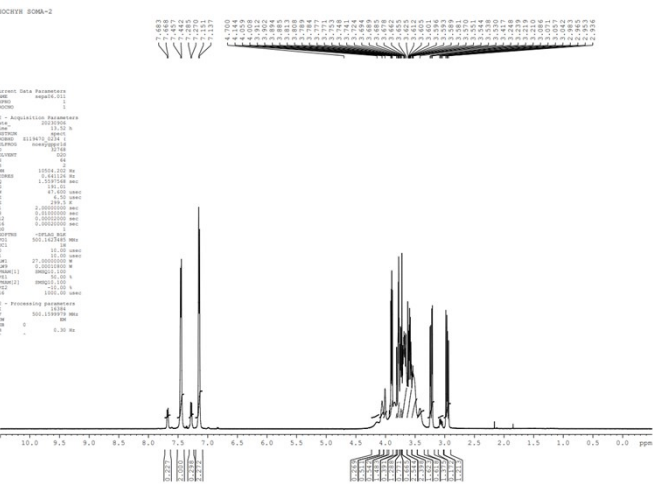
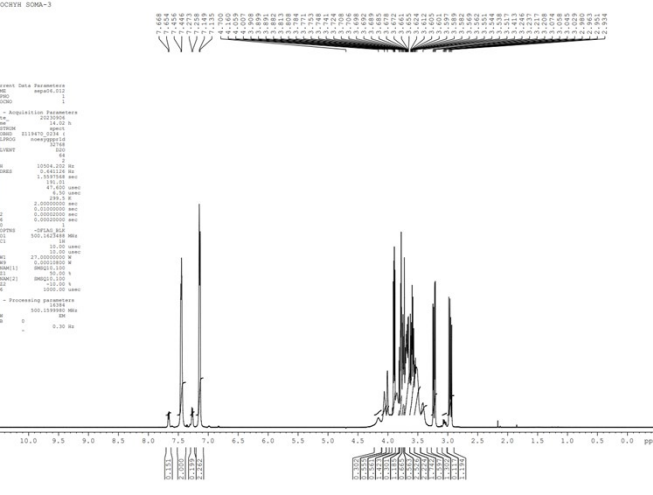
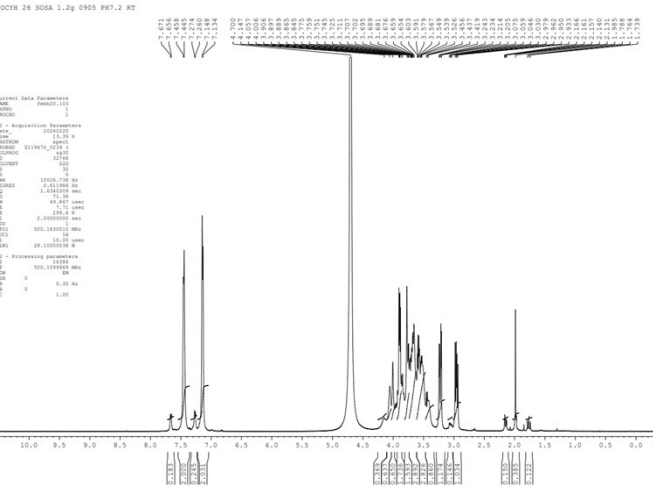
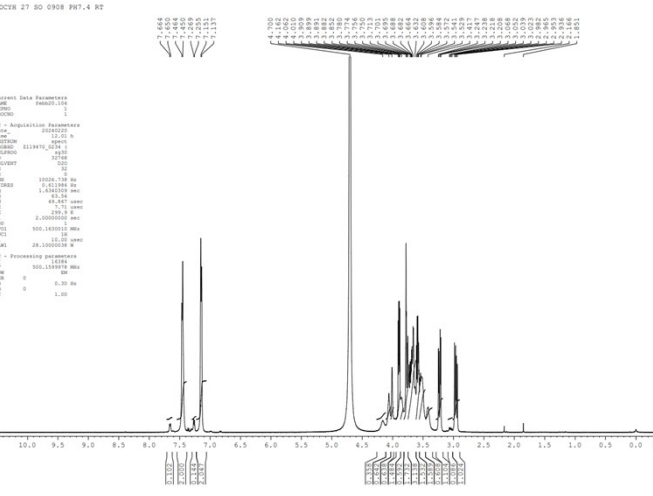
44 31 d Int Sorb 1.2 g – BPA 7.2 : Int BPA = 2.000 / 0.153 = 13.07

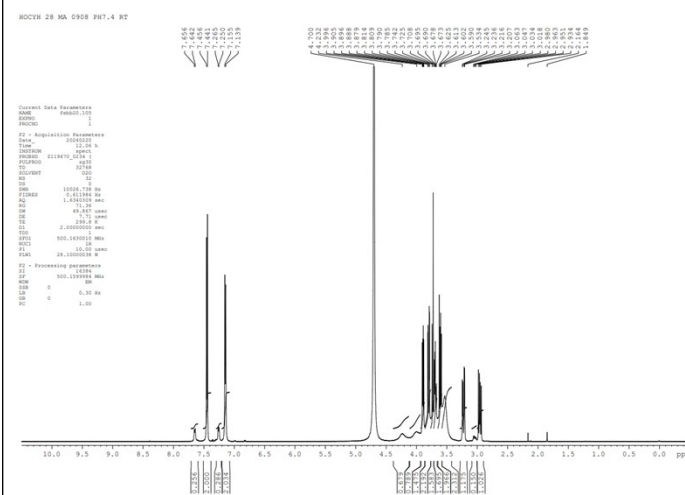
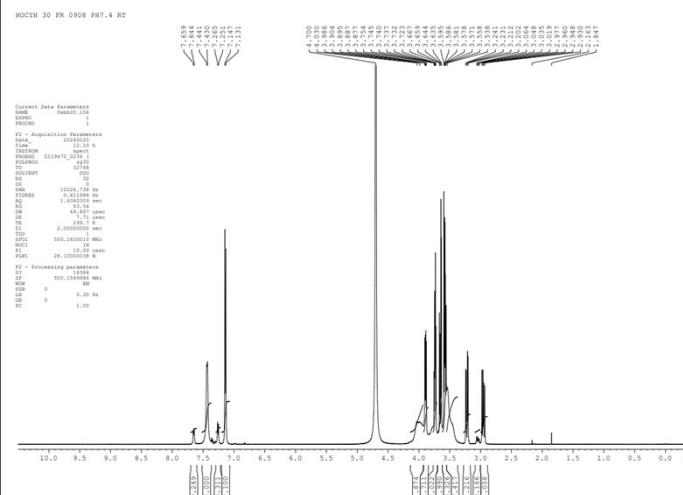
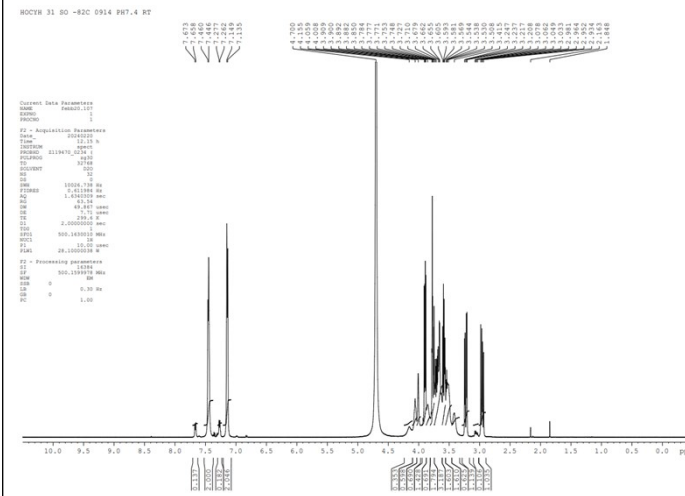
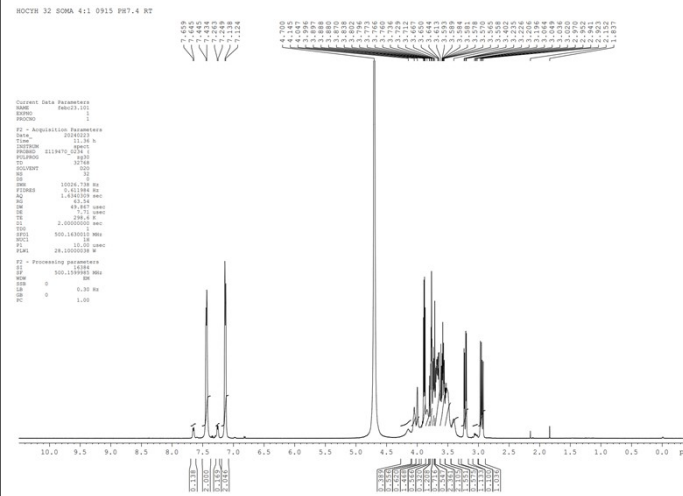
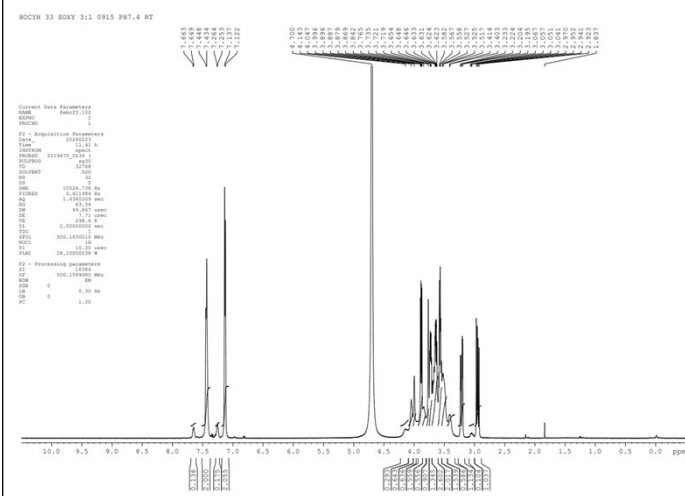
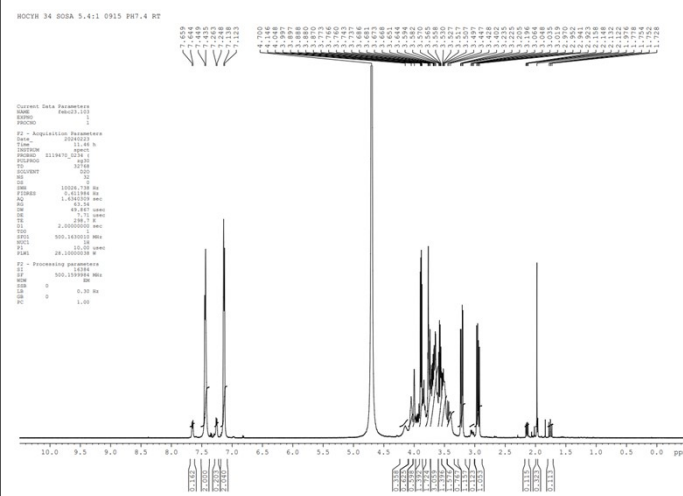


45 74 d Int Sorb + Man (2:3) – BPA 7.4 : Int BPA = 2.000/0.246 =



46 74 d Int Sorb + Man (1:1) – BPA 7.4 : Int BPA = 2.000/0.197 =

8.13			10.15		
ROCKYR SORA-5  Current Data Parameters NAME: sample111 PROCNO: 1 F2 - Acquisition Parameters Date_: 20230901 Time: 14.22 INSTRUM: spect PROBHD: 5mmQNP1H1 PULPROG: zgpg30 PC: 100.000000 FIDRES: 0.000000 AQ: 0.000000 RG: 655.36 SD: 0.000000 SI: 32768 SF: 500.135000 WDW: EM SSB: 0 LB: 0.000000 GB: 0 PC: 1.000000 F2 - Processing parameters SI: 32768 SF: 500.135000 WDW: EM SSB: 0 LB: 0.000000 GB: 0 PC: 1.000000			ROCKYR SORA-2  Current Data Parameters NAME: sample111 PROCNO: 1 F2 - Acquisition Parameters Date_: 20230901 Time: 14.22 INSTRUM: spect PROBHD: 5mmQNP1H1 PULPROG: zgpg30 PC: 100.000000 FIDRES: 0.000000 AQ: 0.000000 RG: 655.36 SD: 0.000000 SI: 32768 SF: 500.135000 WDW: EM SSB: 0 LB: 0.000000 GB: 0 PC: 1.000000 F2 - Processing parameters SI: 32768 SF: 500.135000 WDW: EM SSB: 0 LB: 0.000000 GB: 0 PC: 1.000000		
47	74 d	$\text{Int}_{\text{Sorb} + \text{Man} (2:1) - \text{BPA } 7.4} : \text{Int}_{\text{BPA}} = 2.000/0.178 = 11.24$	48	74 d	$\text{Int}_{\text{Sorb} + \text{Man} (3:1) - \text{BPA } 7.4} : \text{Int}_{\text{BPA}} = 2.000/0.227 = 8.81$
ROCKYR SORA-3  Current Data Parameters NAME: sample111 PROCNO: 1 F2 - Acquisition Parameters Date_: 20230901 Time: 14.22 INSTRUM: spect PROBHD: 5mmQNP1H1 PULPROG: zgpg30 PC: 100.000000 FIDRES: 0.000000 AQ: 0.000000 RG: 655.36 SD: 0.000000 SI: 32768 SF: 500.135000 WDW: EM SSB: 0 LB: 0.000000 GB: 0 PC: 1.000000 F2 - Processing parameters SI: 32768 SF: 500.135000 WDW: EM SSB: 0 LB: 0.000000 GB: 0 PC: 1.000000					
49	74 d	$\text{Int}_{\text{Sorb} + \text{Man} (4:1) - \text{BPA } 7.4} : \text{Int}_{\text{BPA}} = 2.000/0.151 = 13.24$			
ROCKYR 26 SORA 1.2g 0905 PNT.2 MZ  Current Data Parameters NAME: sample111 PROCNO: 1 F2 - Acquisition Parameters Date_: 20230901 Time: 14.22 INSTRUM: spect PROBHD: 5mmQNP1H1 PULPROG: zgpg30 PC: 100.000000 FIDRES: 0.000000 AQ: 0.000000 RG: 655.36 SD: 0.000000 SI: 32768 SF: 500.135000 WDW: EM SSB: 0 LB: 0.000000 GB: 0 PC: 1.000000 F2 - Processing parameters SI: 32768 SF: 500.135000 WDW: EM SSB: 0 LB: 0.000000 GB: 0 PC: 1.000000			ROCKYR 27 SO 0908 PNT.4 MZ  Current Data Parameters NAME: sample111 PROCNO: 1 F2 - Acquisition Parameters Date_: 20230901 Time: 14.22 INSTRUM: spect PROBHD: 5mmQNP1H1 PULPROG: zgpg30 PC: 100.000000 FIDRES: 0.000000 AQ: 0.000000 RG: 655.36 SD: 0.000000 SI: 32768 SF: 500.135000 WDW: EM SSB: 0 LB: 0.000000 GB: 0 PC: 1.000000 F2 - Processing parameters SI: 32768 SF: 500.135000 WDW: EM SSB: 0 LB: 0.000000 GB: 0 PC: 1.000000		
50	168 d	$\text{Int}_{\text{sorb}/\text{SA } 5.4:1 \text{ 1.2g} - \text{BPA } 7.2} : \text{Int}_{\text{BPA}} = 2.000/0.183 =$	51	164 d	$\text{Int}_{\text{sorb} - \text{BPA } 7.4} : \text{Int}_{\text{BPA}} = 2.000/0.102 = 19.61$

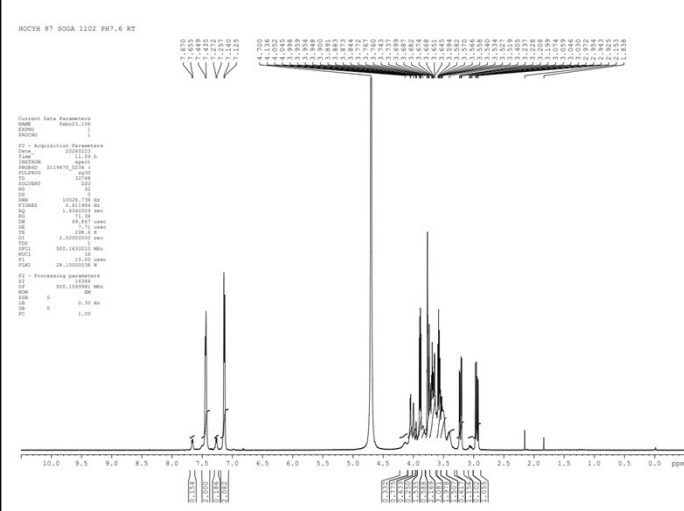
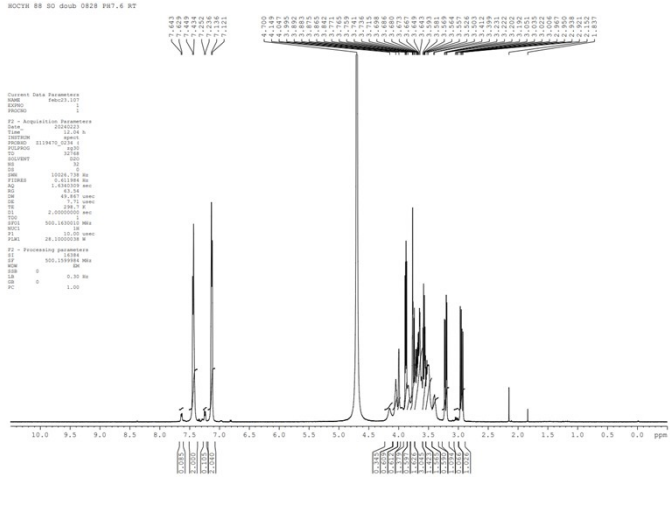
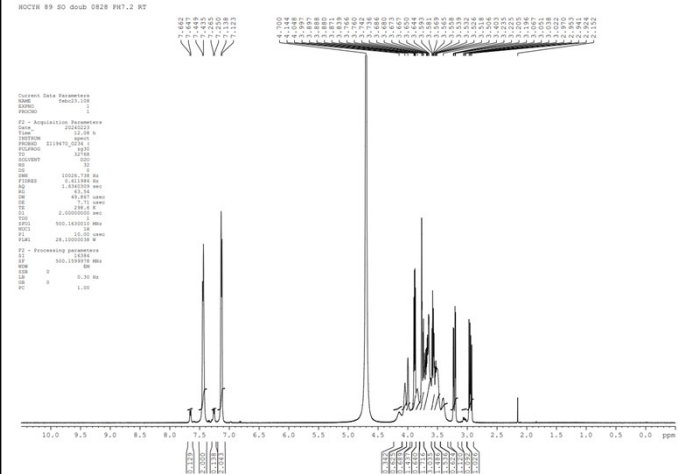
10.93					
 Current Data Parameters NAME: P00023-001 PROCNO: 1 F2 - Acquisition Parameters Date_: 20200220 Time: 12:10 INSTRUM: spect PROBHD: 513670_20mm 1 PULPROG: zgpg30 SFO: 300 AQ: 32.64 RG: 32 FIDRES: 0.001280 Hz F2 - Processing parameters SI: 32768 SF: 300.1350000 MHz WDW: EM SSB: 0 LB: 0.30 Hz GB: 0 PC: 1.00			 Current Data Parameters NAME: P00023-001 PROCNO: 1 F2 - Acquisition Parameters Date_: 20200220 Time: 12:10 INSTRUM: spect PROBHD: 513670_20mm 1 PULPROG: zgpg30 SFO: 300 AQ: 32.64 RG: 32 FIDRES: 0.001280 Hz F2 - Processing parameters SI: 32768 SF: 300.1350000 MHz WDW: EM SSB: 0 LB: 0.30 Hz GB: 0 PC: 1.00		
52	164 d	$\text{Int}_{\text{man}} - \text{BPA } 7.4 : \text{Int}_{\text{BPA}} = 2.000/0.256 = 7.81$	53	164 d	$\text{Int}_{\text{fru}} - \text{BPA } 7.0 : \text{Int}_{\text{BPA}} = 2.000/0.249 = 8.03$
 Current Data Parameters NAME: P00023-001 PROCNO: 1 F2 - Acquisition Parameters Date_: 20200220 Time: 12:10 INSTRUM: spect PROBHD: 513670_20mm 1 PULPROG: zgpg30 SFO: 300 AQ: 32.64 RG: 32 FIDRES: 0.001280 Hz F2 - Processing parameters SI: 32768 SF: 300.1350000 MHz WDW: EM SSB: 0 LB: 0.30 Hz GB: 0 PC: 1.00			 Current Data Parameters NAME: P00023-001 PROCNO: 1 F2 - Acquisition Parameters Date_: 20200220 Time: 12:10 INSTRUM: spect PROBHD: 513670_20mm 1 PULPROG: zgpg30 SFO: 300 AQ: 32.64 RG: 32 FIDRES: 0.001280 Hz F2 - Processing parameters SI: 32768 SF: 300.1350000 MHz WDW: EM SSB: 0 LB: 0.30 Hz GB: 0 PC: 1.00		
54	159 d	$\text{Int}_{\text{sorb}-82^{\circ}\text{C}} - \text{BPA } 7.2 : \text{Int}_{\text{BPA}} = 2.000/0.137 = 14.60$	55	158 d	$\text{Int}_{\text{sorb/man } 4:1} - \text{BPA } 7.4 : \text{Int}_{\text{BPA}} = 2.000/0.138 = 14.49$
 Current Data Parameters NAME: P00023-001 PROCNO: 1 F2 - Acquisition Parameters Date_: 20200220 Time: 12:10 INSTRUM: spect PROBHD: 513670_20mm 1 PULPROG: zgpg30 SFO: 300 AQ: 32.64 RG: 32 FIDRES: 0.001280 Hz F2 - Processing parameters SI: 32768 SF: 300.1350000 MHz WDW: EM SSB: 0 LB: 0.30 Hz GB: 0 PC: 1.00			 Current Data Parameters NAME: P00023-001 PROCNO: 1 F2 - Acquisition Parameters Date_: 20200220 Time: 12:10 INSTRUM: spect PROBHD: 513670_20mm 1 PULPROG: zgpg30 SFO: 300 AQ: 32.64 RG: 32 FIDRES: 0.001280 Hz F2 - Processing parameters SI: 32768 SF: 300.1350000 MHz WDW: EM SSB: 0 LB: 0.30 Hz GB: 0 PC: 1.00		
56	158 d	$\text{Int}_{\text{sorb/xyl } 3:1} - \text{BPA } 7.4 : \text{Int}_{\text{BPA}} = 2.000/0.136 = 14.71$	57	158 d	$\text{Int}_{\text{sorb/SA } 5.4:1} - \text{BPA } 7.4 : \text{Int}_{\text{BPA}} = 2.000/0.162 = 12.35$

5881 dInt _{sorb anti} – BPA 7.6 : Int _{BPA} = 2.000/0.091 = 21.98	5981 dInt _{sorb anti} – BPA 7.4 : Int _{BPA} = 2.000/0.111 = 18.02
6081 dInt _{sorb anti} – BPA 7.2 : Int _{BPA} = 2.000/0.107 = 18.69	6181 dInt _{sorb anti} – BPA 7.0 : Int _{BPA} = 2.000/0.171 = 11.70
62221 dInt _{sorb} – BPA 7.4 : Int _{BPA} = 2.000/0.179 = 11.17	63138 dInt _{sorb 1.2g} – BPA 7.2 : Int _{BPA} = 2.000/0.159 = 12.58

NOEYR 62 80 double 0828 P87.4 RT Current Data Parameters NAME: 0828P87.4 PROCNO: 1 F2 - Acquisition Parameters Date_: 20231227 Time: 15.45 s INSTRUM: spect PROBHD: B1H400_20mm PULPROG: zgpg30 SFO: 400 SOLVENT: DMS DS: 2 SWH: 10024.736 Hz F2FREQ: 101.621384 MHz AQ: 1.6721028 sec RG: 65.427 WDW: EM SS: 2.0000000 sec LB: 2.0000000 Hz GB: 0 PC: 500.1450012 MHz P1: 150.00 usec PL1: 27.78959024 dB F2 - Processing parameters SI: 32768 SF: 500.135958 MHz WDW: EM SS: 0 LB: 0.30 Hz GB: 0 PC: 1.00			NOEYR 63 80 test 0430 P87.0 RT Current Data Parameters NAME: 0430P87.0 PROCNO: 1 F2 - Acquisition Parameters Date_: 20231227 Time: 15.45 s INSTRUM: spect PROBHD: B1H400_20mm PULPROG: zgpg30 SFO: 400 SOLVENT: DMS DS: 2 SWH: 10024.736 Hz F2FREQ: 101.621384 MHz AQ: 1.6721028 sec RG: 65.427 WDW: EM SS: 2.0000000 sec LB: 2.0000000 Hz GB: 0 PC: 500.1450012 MHz P1: 150.00 usec PL1: 27.78959024 dB F2 - Processing parameters SI: 32768 SF: 500.135958 MHz WDW: EM SS: 0 LB: 0.30 Hz GB: 0 PC: 1.00		
64	100 d	$\text{Int}_{\text{sorb double conc.}} - \text{BPA } 7.4 : \text{Int}_{\text{BPA}} = 2.000/0.106 = 18.87$	65	221 d	$\text{Int}_{\text{sorb test}} - \text{BPA } 7.0 : \text{Int}_{\text{BPA}} = 2.000/0.271 = 7.38$
NOEYR 64 XY test 0430 P87.0 RT Current Data Parameters NAME: 0430P87.0 PROCNO: 1 F2 - Acquisition Parameters Date_: 20231227 Time: 15.45 s INSTRUM: spect PROBHD: B1H400_20mm PULPROG: zgpg30 SFO: 400 SOLVENT: DMS DS: 2 SWH: 10024.736 Hz F2FREQ: 101.621384 MHz AQ: 1.6721028 sec RG: 65.427 WDW: EM SS: 2.0000000 sec LB: 2.0000000 Hz GB: 0 PC: 500.1450012 MHz P1: 150.00 usec PL1: 27.78959024 dB F2 - Processing parameters SI: 32768 SF: 500.135958 MHz WDW: EM SS: 0 LB: 0.30 Hz GB: 0 PC: 1.00			NOEYR 65 SX 1:1 -196C 0710 P87.2 RT Current Data Parameters NAME: 0710P87.2 PROCNO: 1 F2 - Acquisition Parameters Date_: 20231227 Time: 15.45 s INSTRUM: spect PROBHD: B1H400_20mm PULPROG: zgpg30 SFO: 400 SOLVENT: DMS DS: 2 SWH: 10024.736 Hz F2FREQ: 101.621384 MHz AQ: 1.6721028 sec RG: 65.427 WDW: EM SS: 2.0000000 sec LB: 2.0000000 Hz GB: 0 PC: 500.1450012 MHz P1: 150.00 usec PL1: 27.78959024 dB F2 - Processing parameters SI: 32768 SF: 500.135958 MHz WDW: EM SS: 0 LB: 0.30 Hz GB: 0 PC: 1.00		
66	221 d	$\text{Int}_{\text{xy test}} - \text{BPA } 7.0 : \text{Int}_{\text{BPA}} = 2.000/0.589 = 3.40$	67	149 d	$\text{Int}_{\text{sorb/xy 1:1 -196C}} - \text{BPA } 7.2 : \text{Int}_{\text{BPA}} = 2.000/0.185 = 10.81$
NOEYR 66 SX 1:1 -82C 0710 P87.2 RT Current Data Parameters NAME: 0710P87.2 PROCNO: 1 F2 - Acquisition Parameters Date_: 20231227 Time: 15.45 s INSTRUM: spect PROBHD: B1H400_20mm PULPROG: zgpg30 SFO: 400 SOLVENT: DMS DS: 2 SWH: 10024.736 Hz F2FREQ: 101.621384 MHz AQ: 1.6721028 sec RG: 65.427 WDW: EM SS: 2.0000000 sec LB: 2.0000000 Hz GB: 0 PC: 500.1450012 MHz P1: 150.00 usec PL1: 27.78959024 dB F2 - Processing parameters SI: 32768 SF: 500.135958 MHz WDW: EM SS: 0 LB: 0.30 Hz GB: 0 PC: 1.00			NOEYR 67 SX 1:1 0922 P87.4 RT Current Data Parameters NAME: 0922P87.4 PROCNO: 1 F2 - Acquisition Parameters Date_: 20231227 Time: 15.45 s INSTRUM: spect PROBHD: B1H400_20mm PULPROG: zgpg30 SFO: 400 SOLVENT: DMS DS: 2 SWH: 10024.736 Hz F2FREQ: 101.621384 MHz AQ: 1.6721028 sec RG: 65.427 WDW: EM SS: 2.0000000 sec LB: 2.0000000 Hz GB: 0 PC: 500.1450012 MHz P1: 150.00 usec PL1: 27.78959024 dB F2 - Processing parameters SI: 32768 SF: 500.135958 MHz WDW: EM SS: 0 LB: 0.30 Hz GB: 0 PC: 1.00		
68	149 d	$\text{Int}_{\text{sorb/xy 1:1 -82C}} - \text{BPA } 7.2 : \text{Int}_{\text{BPA}} = 2.000/0.234 = 8.55$	69	75 d	$\text{Int}_{\text{sorb/xy 1:1 -BPA } 7.4} : \text{Int}_{\text{BPA}} = 2.000/0.185 = 10.81$

NOEYR 68 SS 5.411 0829 PWT.4 RT Current Data Parameters NAME: NOEYR 68 SS PROCNO: 1 F2 - Acquisition Parameters Date_: 20221207 Time: 12.45 N INSTRUM: spect PROBHD: 513670_200-1 PULPROG: zgpg30 AQ: 3.782 RG: 32768 SD: 0.0000000 SI: 32768 SF: 500.1350000 MHz FIDRES: 0.1000000 F2 - Processing parameters SI: 32768 SF: 500.1350000 MHz WDW: EM SSB: 0 GB: 0.30 Hz PC: 1.00		
70	99 d	$\text{Int}_{\text{sorb/SA 5.4:1 - BPA 7.4}} : \text{Int}_{\text{BPA}} = 2.000/0.218 = 9.17$
NOEYR 76 SO5A 5.411 0829 PWT.4 RT Current Data Parameters NAME: NOEYR 76 PROCNO: 1 F2 - Acquisition Parameters Date_: 20221210 Time: 10:55:19 N INSTRUM: spect PROBHD: 513670_200-1 PULPROG: zgpg30 AQ: 3.782 RG: 32768 SD: 0.0000000 SI: 32768 SF: 500.1350000 MHz FIDRES: 0.1000000 F2 - Processing parameters SI: 32768 SF: 500.1350000 MHz WDW: EM SSB: 0 GB: 0.30 Hz PC: 1.00		
71	141 d	$\text{Int}_{\text{sorb/SA 5.4:1 - BPA 7.4}} : \text{Int}_{\text{BPA}} = 2.000/0.189 = 10.58$
NOEYR 77 SO5A 5.411 0829 PWT.4 RT Current Data Parameters NAME: NOEYR 77 PROCNO: 1 F2 - Acquisition Parameters Date_: 20221210 Time: 10:55:19 N INSTRUM: spect PROBHD: 513670_200-1 PULPROG: zgpg30 AQ: 3.782 RG: 32768 SD: 0.0000000 SI: 32768 SF: 500.1350000 MHz FIDRES: 0.1000000 F2 - Processing parameters SI: 32768 SF: 500.1350000 MHz WDW: EM SSB: 0 GB: 0.30 Hz PC: 1.00		
72	141 d	$\text{Int}_{\text{sorb/SA 5.4:1 - BPA 7.4}} : \text{Int}_{\text{BPA}} = 2.000/0.191 = 10.47$
NOEYR 78 SOXY 3:1 0710 PWT.4 RT Current Data Parameters NAME: NOEYR 78 PROCNO: 1 F2 - Acquisition Parameters Date_: 20221210 Time: 10:55:19 N INSTRUM: spect PROBHD: 513670_200-1 PULPROG: zgpg30 AQ: 3.782 RG: 32768 SD: 0.0000000 SI: 32768 SF: 500.1350000 MHz FIDRES: 0.1000000 F2 - Processing parameters SI: 32768 SF: 500.1350000 MHz WDW: EM SSB: 0 GB: 0.30 Hz PC: 1.00		
73	191 d	$\text{Int}_{\text{sorb/xy1 3:1 - BPA 7.4}} : \text{Int}_{\text{BPA}} = 2.000/0.203 = 9.85$
NOEYR 79 SO 0429 PWT.2 RT Current Data Parameters NAME: NOEYR 79 PROCNO: 1 F2 - Acquisition Parameters Date_: 20221210 Time: 10:55:19 N INSTRUM: spect PROBHD: 513670_200-1 PULPROG: zgpg30 AQ: 3.782 RG: 32768 SD: 0.0000000 SI: 32768 SF: 500.1350000 MHz FIDRES: 0.1000000 F2 - Processing parameters SI: 32768 SF: 500.1350000 MHz WDW: EM SSB: 0 GB: 0.30 Hz PC: 1.00		
74	263 d	$\text{Int}_{\text{sorb - BPA 7.2}} : \text{Int}_{\text{BPA}} = 2.000/0.164 = 12.20$

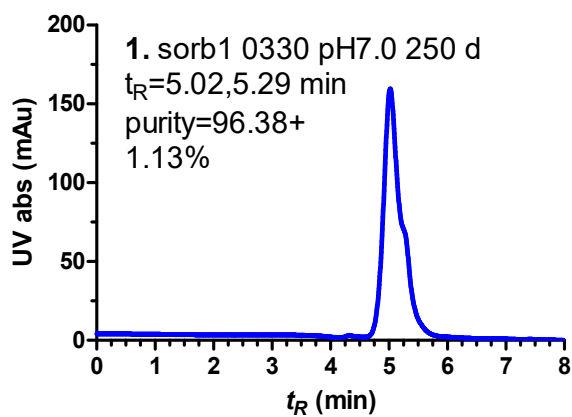
NOETH 29 XY bottle 0908 PHT.4 HT Current Data Parameters NAME: 09081102 PROCNO: 1 F2 - Acquisition Parameters Date_: 20201119 Time_: 12.28 INSTRUM: spect PROBHD: 0136476_2016 1 PULPROG: zgpg30 SI: 32768 SFO: 500.1360912 MHz AQ: 1.00000000 sec RG: 327.68 FIDRES: 0.0001158 Hz AQRES: 0.0001158 Hz RGRES: 0.0001158 Hz F2 - Processing parameters SI: 32768 SFO: 500.1360912 MHz RG: 327.68 FIDRES: 0.0001158 Hz AQRES: 0.0001158 Hz RGRES: 0.0001158 Hz			NOETH 80 SOGA 1007 PHT.4 HT Current Data Parameters NAME: 10071102 PROCNO: 1 F2 - Acquisition Parameters Date_: 20201119 Time_: 12.28 INSTRUM: spect PROBHD: 0136476_2016 1 PULPROG: zgpg30 SI: 32768 SFO: 500.1360912 MHz AQ: 1.00000000 sec RG: 327.68 FIDRES: 0.0001158 Hz AQRES: 0.0001158 Hz RGRES: 0.0001158 Hz F2 - Processing parameters SI: 32768 SFO: 500.1360912 MHz RG: 327.68 FIDRES: 0.0001158 Hz AQRES: 0.0001158 Hz RGRES: 0.0001158 Hz		
75	131 d	$\text{Int}_{\text{xyli bottle}} - \text{BPA 7.4} : \text{Int}_{\text{BPA}} = 2.000/0.505 = 3.96$	76	102 d	$\text{Int}_{\text{sorb/GA}} - \text{BPA 7.4} : \text{Int}_{\text{BPA}} = 2.000/0.164 = 12.20$
NOETH 81 SOGA 1102 PHT.4 HT Current Data Parameters NAME: 11021102 PROCNO: 1 F2 - Acquisition Parameters Date_: 20201119 Time_: 12.28 INSTRUM: spect PROBHD: 0136476_2016 1 PULPROG: zgpg30 SI: 32768 SFO: 500.1360912 MHz AQ: 1.00000000 sec RG: 327.68 FIDRES: 0.0001158 Hz AQRES: 0.0001158 Hz RGRES: 0.0001158 Hz F2 - Processing parameters SI: 32768 SFO: 500.1360912 MHz RG: 327.68 FIDRES: 0.0001158 Hz AQRES: 0.0001158 Hz RGRES: 0.0001158 Hz			NOETH 82 SOGA 1201 PHT.4 HT Current Data Parameters NAME: 12011102 PROCNO: 1 F2 - Acquisition Parameters Date_: 20201119 Time_: 12.28 INSTRUM: spect PROBHD: 0136476_2016 1 PULPROG: zgpg30 SI: 32768 SFO: 500.1360912 MHz AQ: 1.00000000 sec RG: 327.68 FIDRES: 0.0001158 Hz AQRES: 0.0001158 Hz RGRES: 0.0001158 Hz F2 - Processing parameters SI: 32768 SFO: 500.1360912 MHz RG: 327.68 FIDRES: 0.0001158 Hz AQRES: 0.0001158 Hz RGRES: 0.0001158 Hz		
77	76 d	$\text{Int}_{\text{sorb/GA}} - \text{BPA 7.4} : \text{Int}_{\text{BPA}} = 2.000/0.168 = 11.90$	78	47 d	$\text{Int}_{\text{sorb/AA}} - \text{BPA 7.4} : \text{Int}_{\text{BPA}} = 2.000/0.147 = 13.61$
NOETH 85 SOGA doub 3.6:1 CRIS PHT.6 HT Current Data Parameters NAME: 09081102 PROCNO: 1 F2 - Acquisition Parameters Date_: 20201119 Time_: 12.28 INSTRUM: spect PROBHD: 0136476_2016 1 PULPROG: zgpg30 SI: 32768 SFO: 500.1360912 MHz AQ: 1.00000000 sec RG: 327.68 FIDRES: 0.0001158 Hz AQRES: 0.0001158 Hz RGRES: 0.0001158 Hz F2 - Processing parameters SI: 32768 SFO: 500.1360912 MHz RG: 327.68 FIDRES: 0.0001158 Hz AQRES: 0.0001158 Hz RGRES: 0.0001158 Hz			NOETH 86 SOGA 1201 PHT.6 HT Current Data Parameters NAME: 12011102 PROCNO: 1 F2 - Acquisition Parameters Date_: 20201119 Time_: 12.28 INSTRUM: spect PROBHD: 0136476_2016 1 PULPROG: zgpg30 SI: 32768 SFO: 500.1360912 MHz AQ: 1.00000000 sec RG: 327.68 FIDRES: 0.0001158 Hz AQRES: 0.0001158 Hz RGRES: 0.0001158 Hz F2 - Processing parameters SI: 32768 SFO: 500.1360912 MHz RG: 327.68 FIDRES: 0.0001158 Hz AQRES: 0.0001158 Hz RGRES: 0.0001158 Hz		
79	187 d	$\text{Int}_{\text{sorb/SA 3.6:1 double conc.}} - \text{BPA 7.4} : \text{Int}_{\text{BPA}} = 2.000/0.220 = 9.09$	80	81 d	$\text{Int}_{\text{sorb/AA}} - \text{BPA 7.6} : \text{Int}_{\text{BPA}} = 2.000/0.127 = 15.75$

					
81	110 d	$\text{Int}_{\text{sorb/GA-BPA 7.6}} : \text{Int}_{\text{BPA}} = 2.000/0.154 = 12.99$	82	177 d	$\text{Int}_{\text{sorb double conc. - BPA 7.6}} : \text{Int}_{\text{BPA}} = 2.000/0.085 = 23.53$
					
83	177 d	$\text{Int}_{\text{sorb double conc. - BPA 7.2}} : \text{Int}_{\text{BPA}} = 2.000/0.129 = 15.50$			

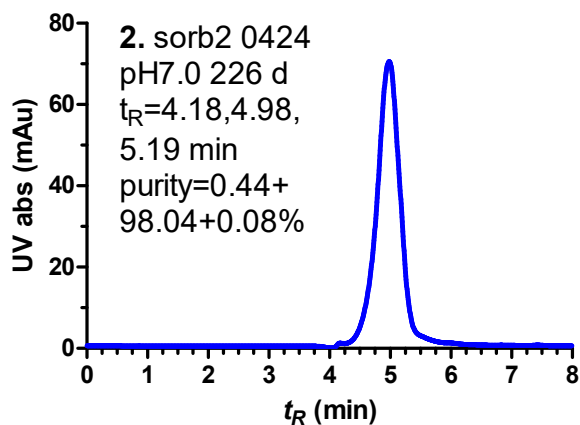
4. RP-HPLC analyses of the lyophilized samples:

Instrumentation using Agilent, Chemcopack, Column Chemcosorb 7-ODS-H 5 μm 10 $\times$ 250 mm assembled with Quatpump G1311A, coupled with UV detector G1314A through a flow cell G1314-60086 4 MPa, and equipped with a stand of G1328B manual injector and Rheodyne injector 7725i with a 500 μL loop with the condition of isocratic eluents: 80% CH_3OH (aq.) at a flowrate of 3 mL/min.

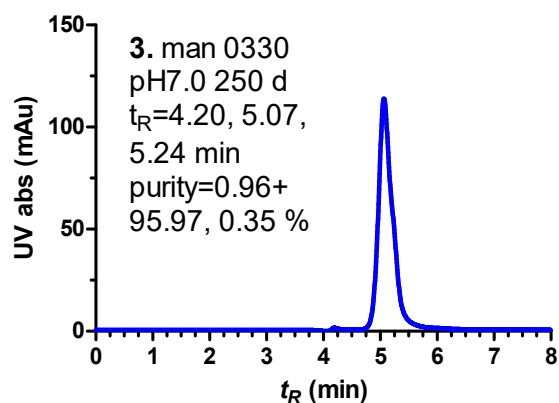
3.1 HPLC chromatogram of lyophilized samples stored at -20 $^{\circ}\text{C}$



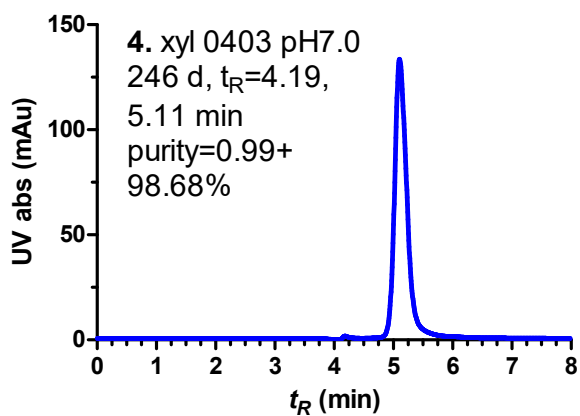
(a) $t_R = 5.02$ min, sorb-BPA: 6.1 mg / 0.2 mL, 10 uL



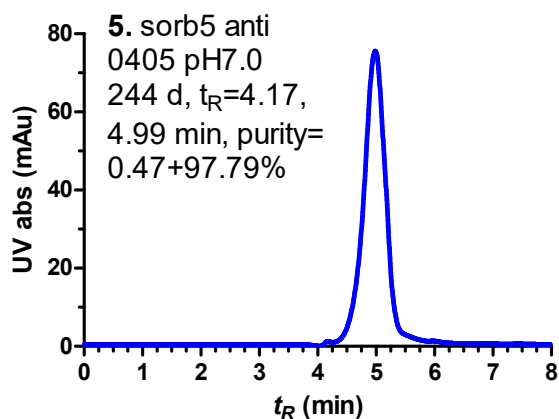
(b) $t_R = 4.98$ min, sorb-BPA: 6.1 mg/0.2 mL, 10 uL



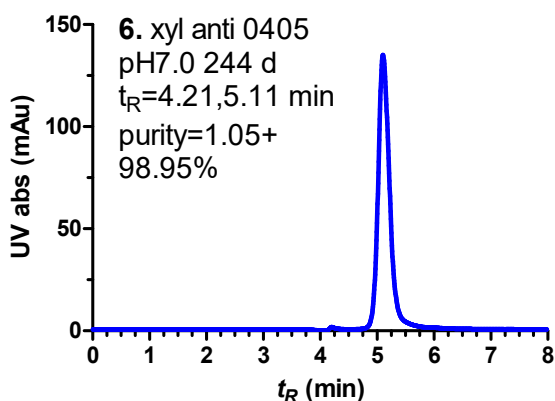
(c) $t_R = 5.07$ min, man-BPA: 6.2 mg / 0.2 mL, 10 uL



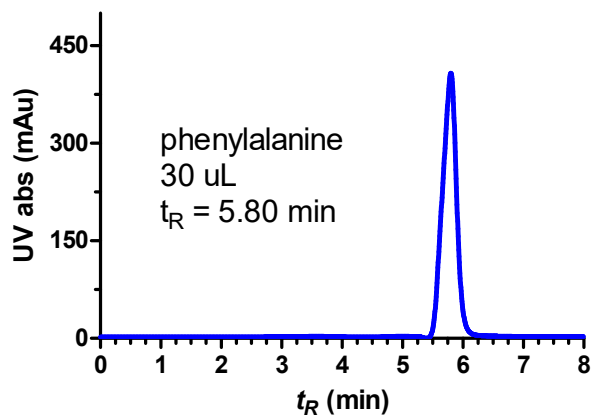
(d) $t_R = 5.11$ min, xyl-BPA: 6.0 mg / 0.2 mL, 10 uL



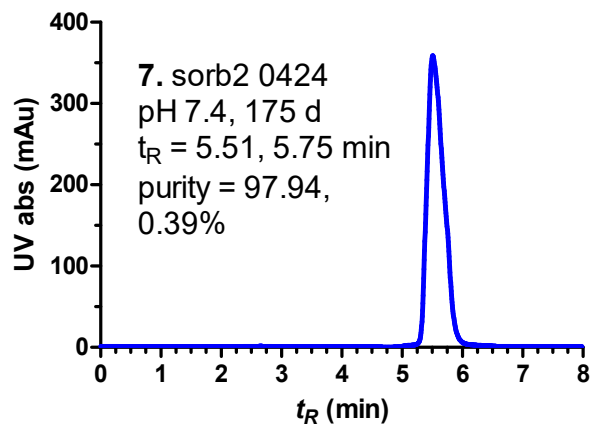
(e) $t_R = 4.99$ min, sorb-BPA anti $\text{Na}_2\text{S}_2\text{O}_5$: 5.8 mg / 0.2 mL, 10 uL



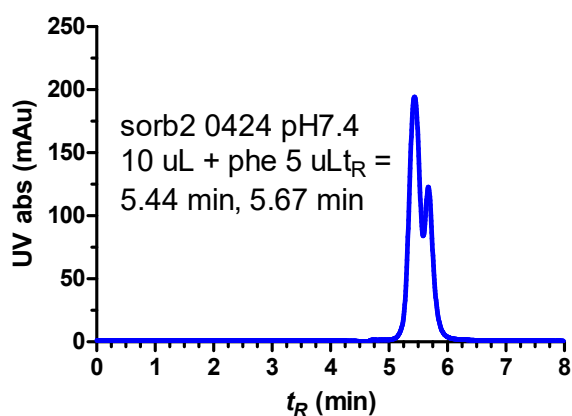
(f) $t_R = 5.11$ min, xyl-BPA anti $\text{Na}_2\text{S}_2\text{O}_5$: 6.8 mg / 0.2 mL, 10 uL



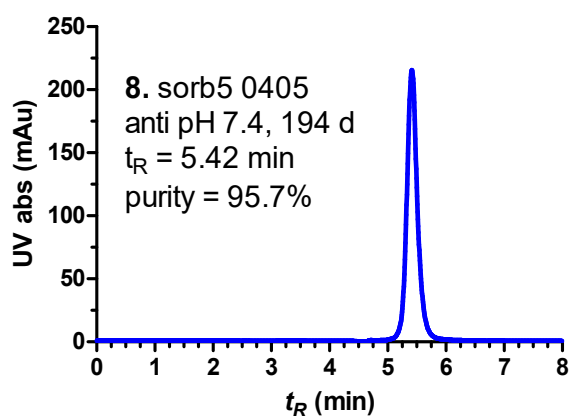
a) $t_R = 5.80$ min, phe: 5 mg/0.5 mL, 10 uL



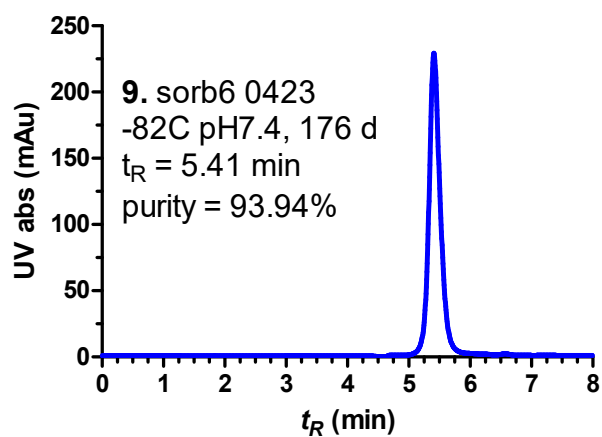
b) $t_R = 5.51$ min, sorb-BPA: 6 mg/0.2 mL, 20 uL



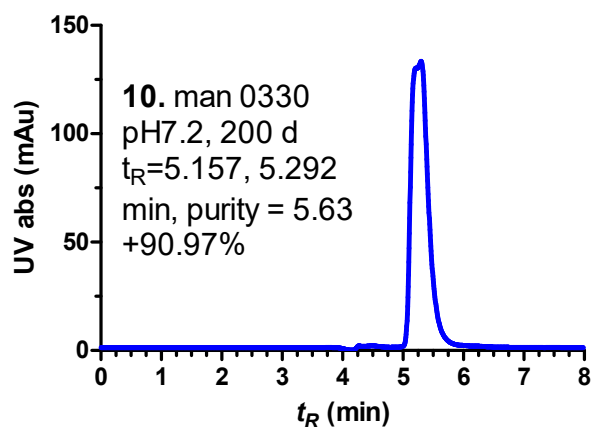
c) $t_R = 5.44$ min; 5.67 min, sorb-BPA: 6 mg/0.2 mL, 10 uL, phe: 5 mg/0.5 mL, 10 uL



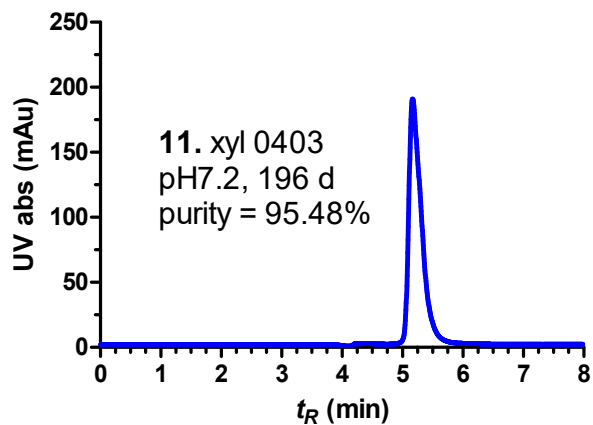
d) $t_R = 5.42$ min, sorb anti-BPA: 6 mg/0.2 mL, 10 uL



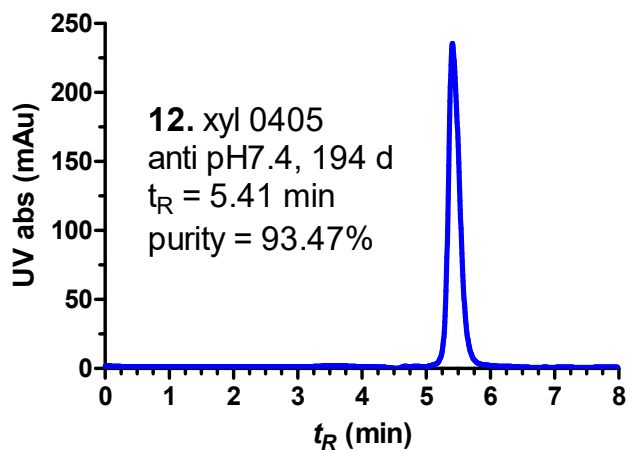
e) $t_R = 5.41$ min, sorb -82 °C-BPA: 6 mg/0.2 mL, 10 uL



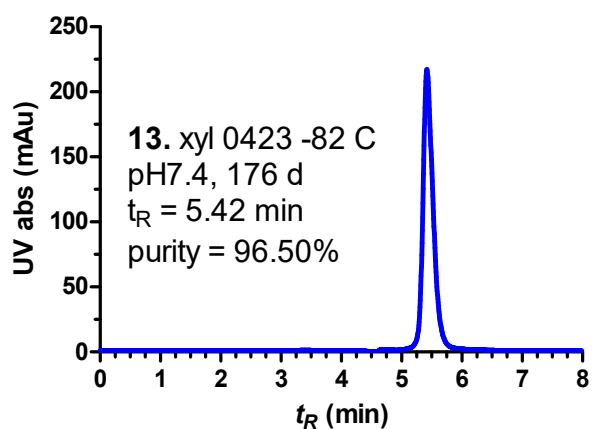
f) $t_R = 5.292$ min, man-BPA: 5.9 mg/0.2 mL, 10 uL



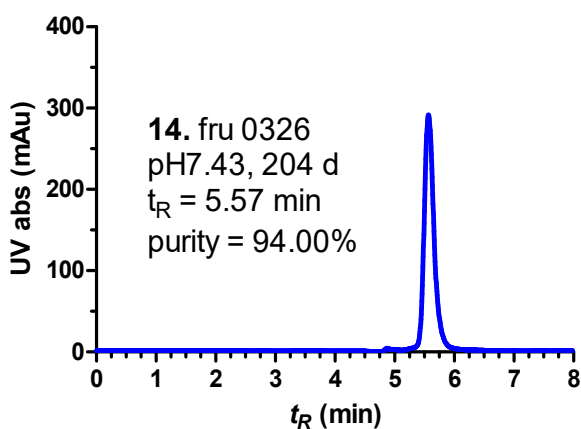
g) $t_R = 5.171$ min, xyl-BPA: 5.9 mg/0.2 mL, 10 uL



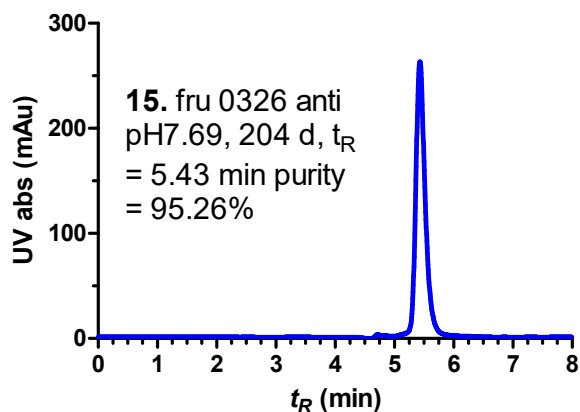
h) $t_R = 5.41$ min, xyl anti-BPA: 7.0 mg/0.2 mL, 10 uL



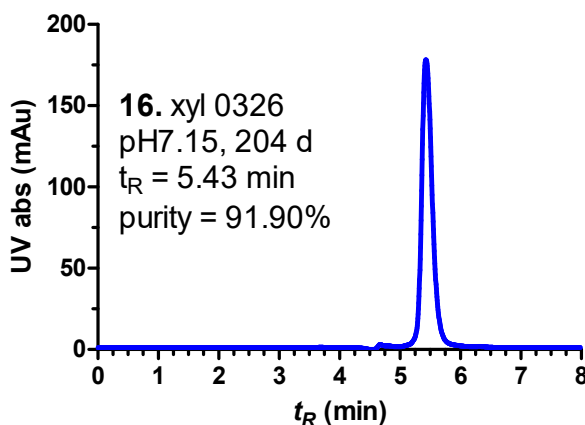
i) $t_R = 5.42$ min, xyl -82 °C-BPA: 6 mg/0.2 mL, 10 uL



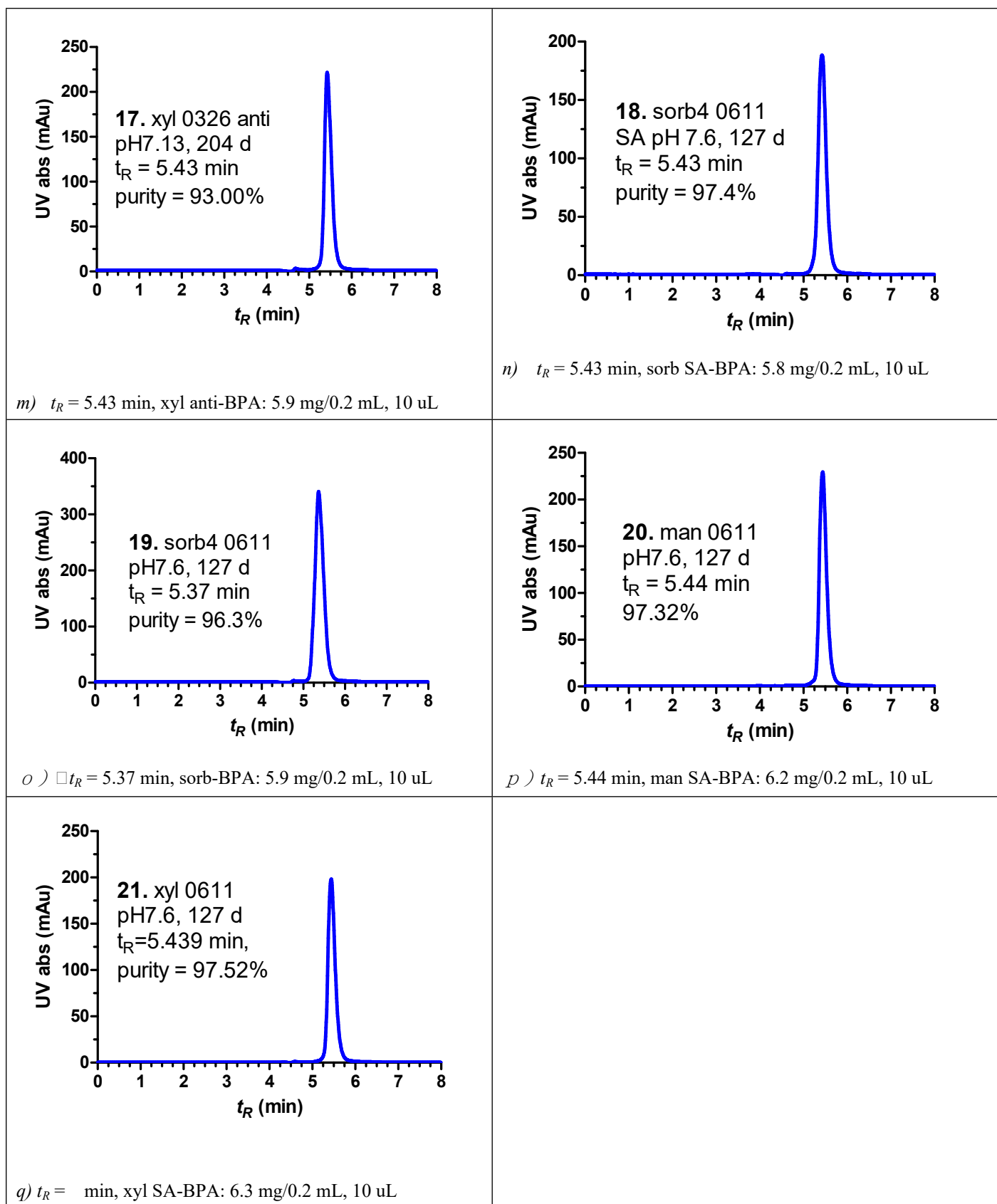
j) $t_R = 5.57$ min, fru-BPA: 6.2 mg/0.2 mL, 10 uL



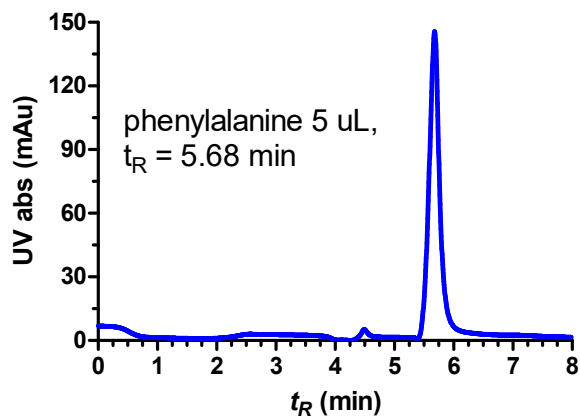
k) $t_R = 5.43$ min, fru anti-BPA: 6.1 mg/0.2 mL, 10 uL



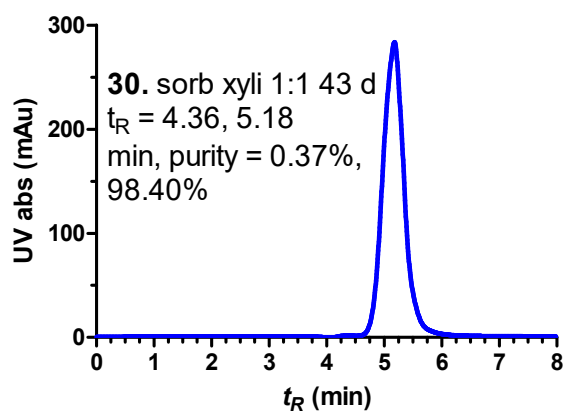
l) $t_R = 5.43$ min, xyl-BPA: 5.5 mg/0.2 mL, 10 uL



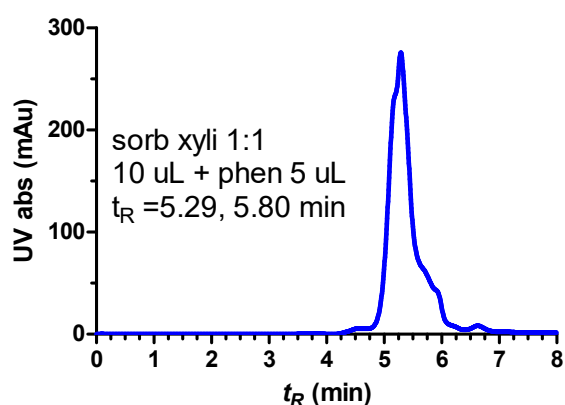
3.2 HPLC chromatogram of lyophilized samples stored at room temperature



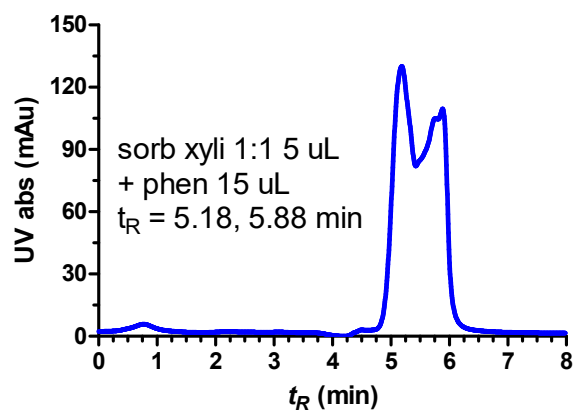
(c) $t_R = 5.678$ min, phe: 5 mg/0.5 mL, 5 uL



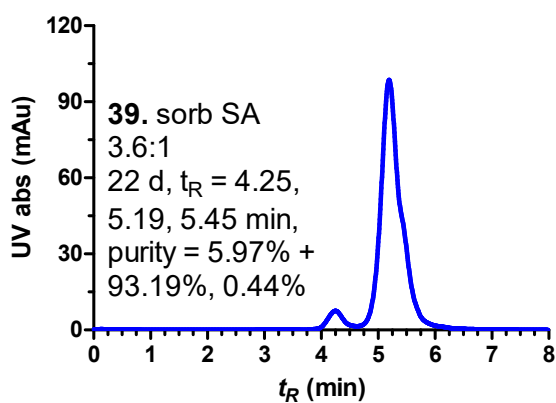
(d) $t_R = 5.178$ min, sorb+xyl-BPA:110 mg/2.2 mL, 10 uL



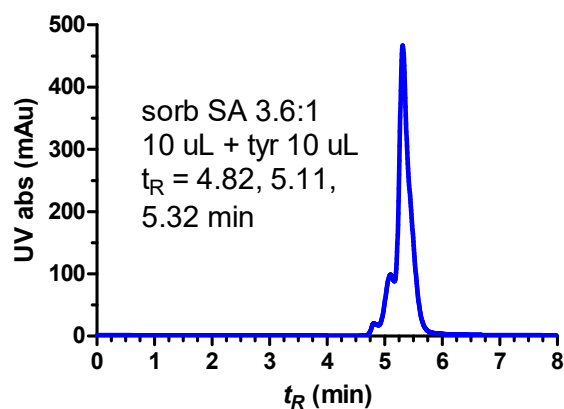
(c) $t_R = 5.290$ (sorb complex), 5.80 min (phen), sorb+xyl-BPA: 110 mg/2.2 mL, 10 uL, phe: 5 mg/0.5 mL, 5 uL



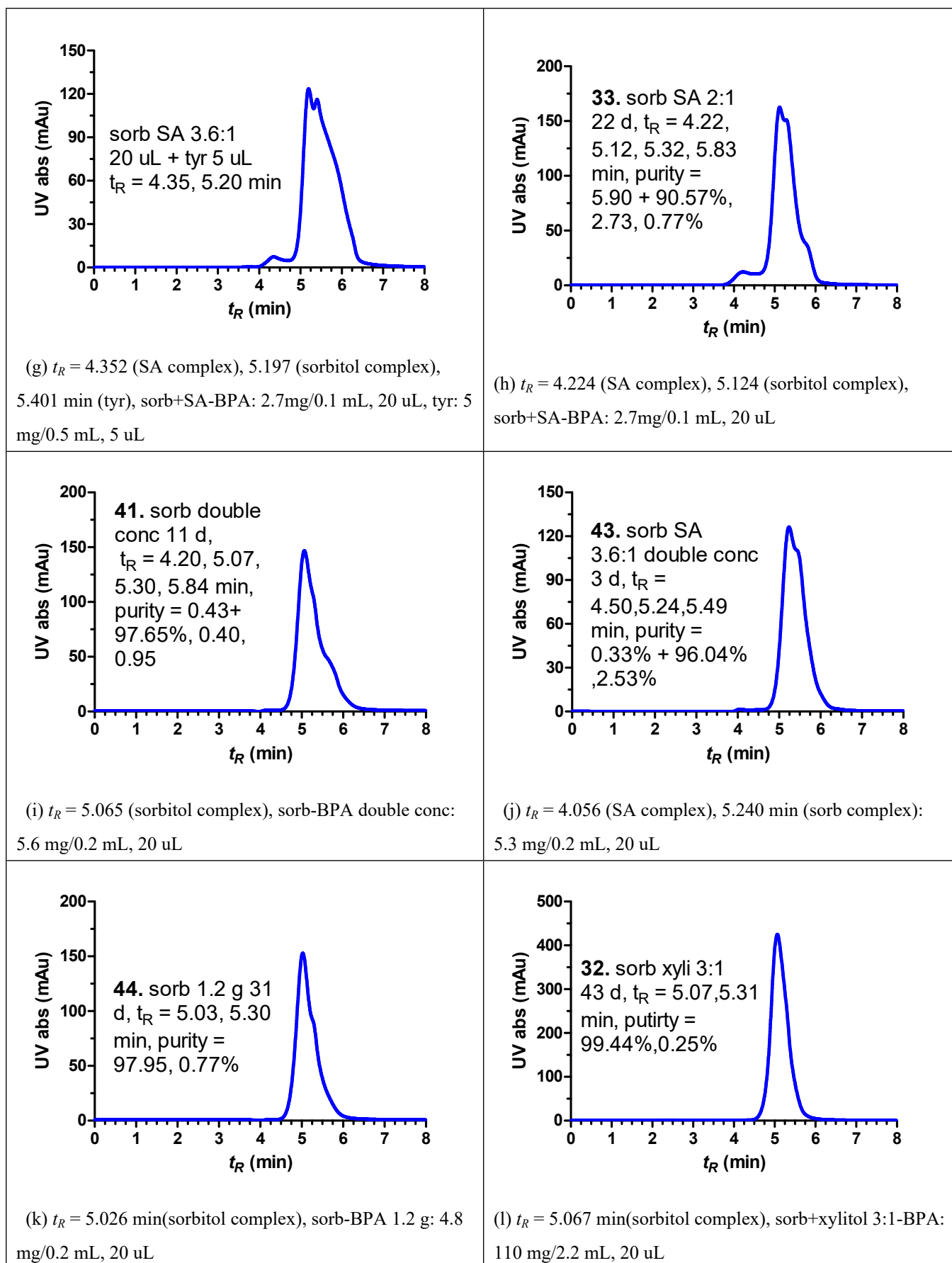
(d) $t_R = 5.184$ (sorb complex), 5.879 min (phen), sorb+xyl-BPA: 110 mg / 2.2 mL, 10 uL, phe: 5 mg/0.5 mL, 5 uL

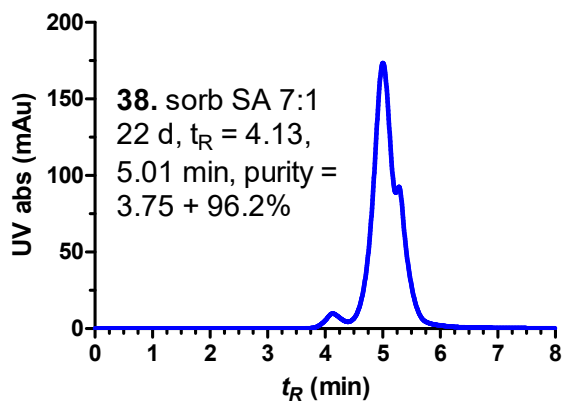


(e) $t_R = 4.251$ (SA complex), 5.193 min (sorb complex), sorb+SA-BPA: 2.7 mg/0.1 mL, 10 uL

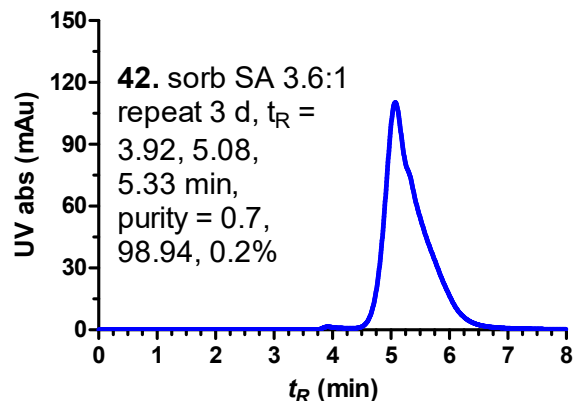


(f) $t_R = 4.819$ (SA complex), 5.105 (sorbitol complex), 5.316 min (tyr), sorb+SA-BPA: 2.7mg/0.1 mL, 10 uL, tyr: 5 mg/0.5 mL, 10 uL

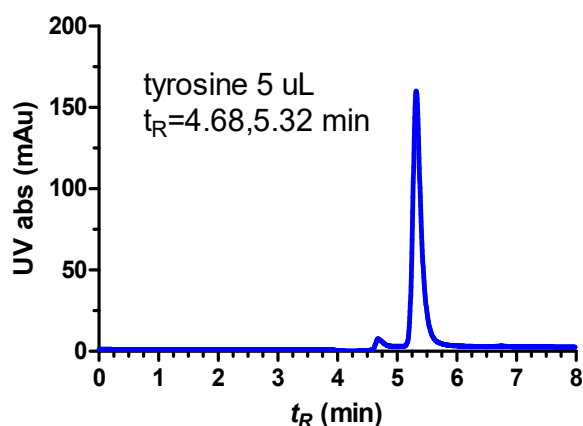




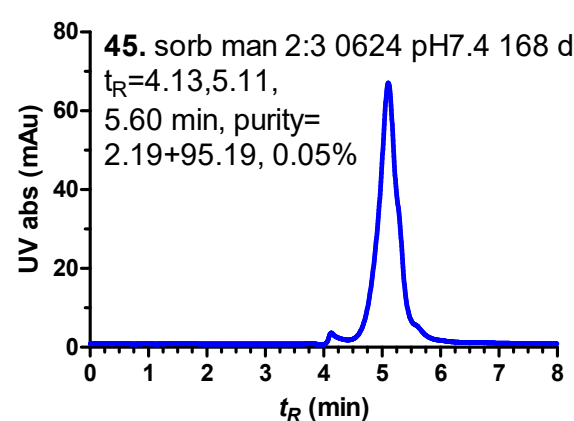
(m) $t_R = 4.133$ (SA complex), 5.005 min (sorb cmplx.),
sorb+SA-BPA: 5.3 mg/0.2 mL, 20 uL



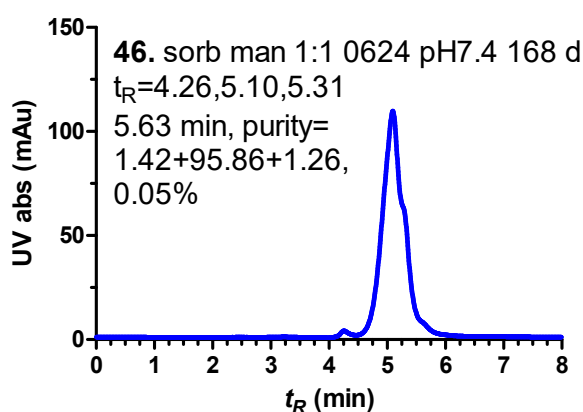
(n) $t_R = 3.924$ (SA complex), 5.075 min (sorb complex),
sorb+SA-BPA: 5.2 mg/0.1 mL, 20 uL



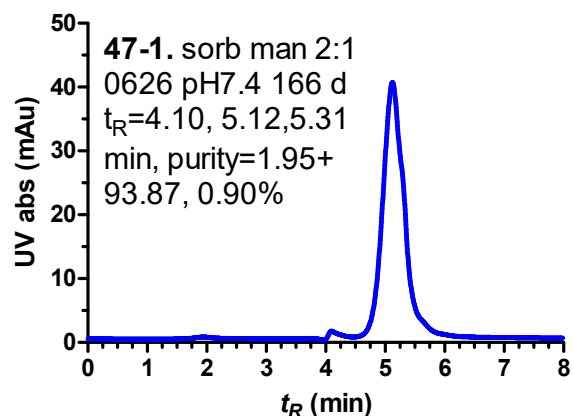
(g) $t_R = 4.68, 5.32$ min, tyrosine: 5 mg/0.5 mL, 5 uL



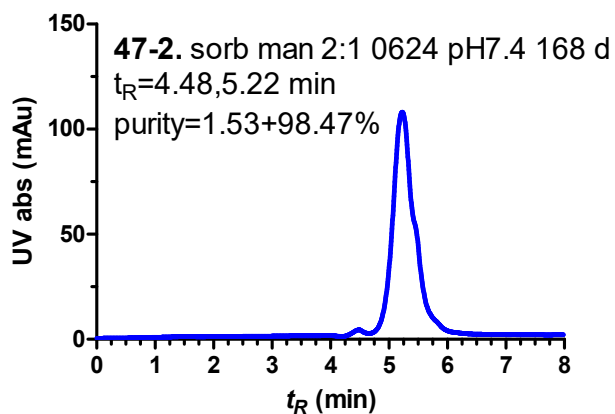
(h) $t_R = 4.13, 5.11$ min, sorb+man 2:3 -BPA: 3.7 mg/0.1 mL,
10 uL



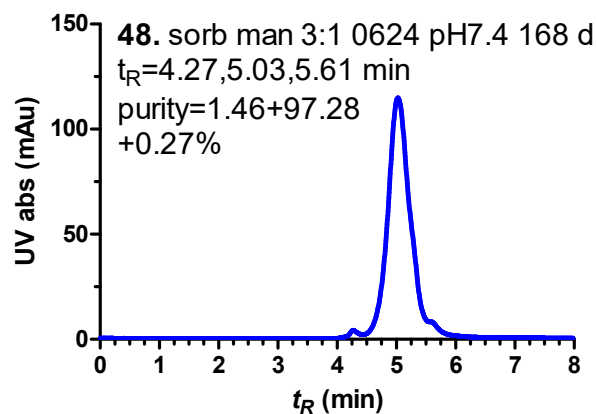
(i) $t_R = 4.26, 5.10$ min, sorb+man 1:1 -BPA: 4.2 mg / 0.1
mL, 10 uL



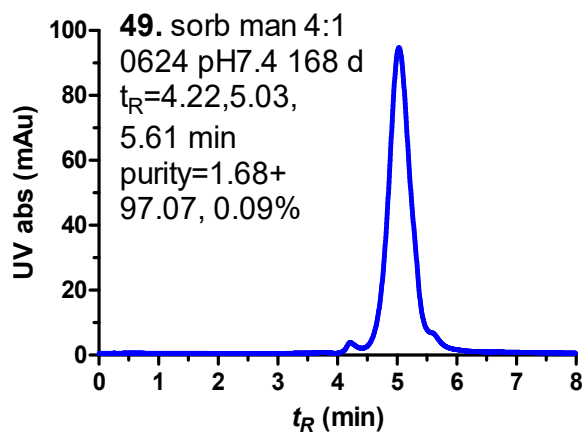
(j) $t_R = 4.10, 5.12$ min, sorb+man 2:1 -BPA: 6.7 mg / 0.2
mL, 10 uL



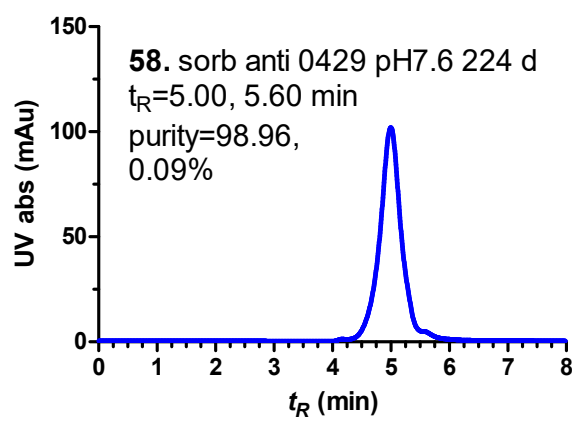
(k) $t_R = 4.48, 5.22$ min, sorb+man 2:1 -BPA: 7.0 mg / 0.2 mL, 10 uL



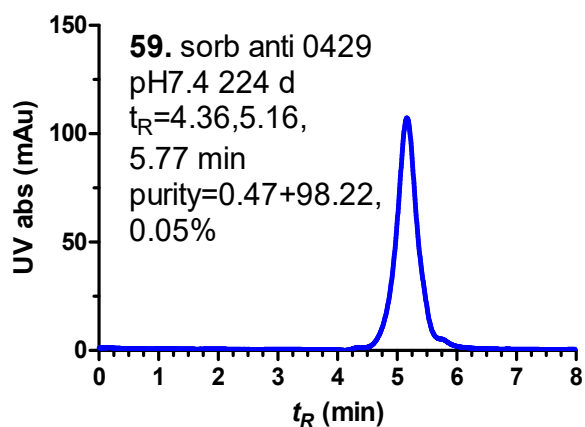
(l) $t_R = 4.27, 5.03, 5.58$ min, sorb+man 3:1 -BPA: 2.7 mg / 0.1 mL, 10 uL



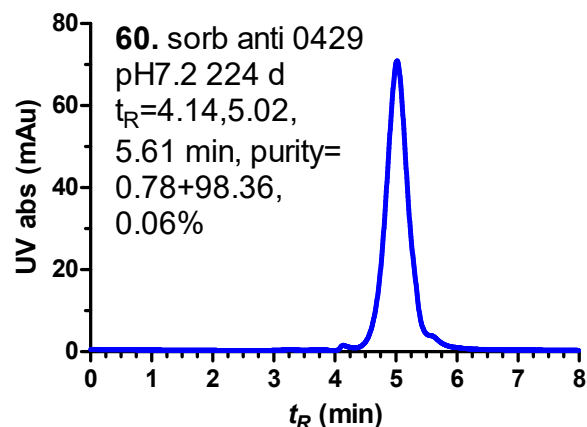
(m) $t_R = 4.22, 5.03$ min, sorb+man 4:1 -BPA: 3.3 mg / 0.1 mL, 10 uL



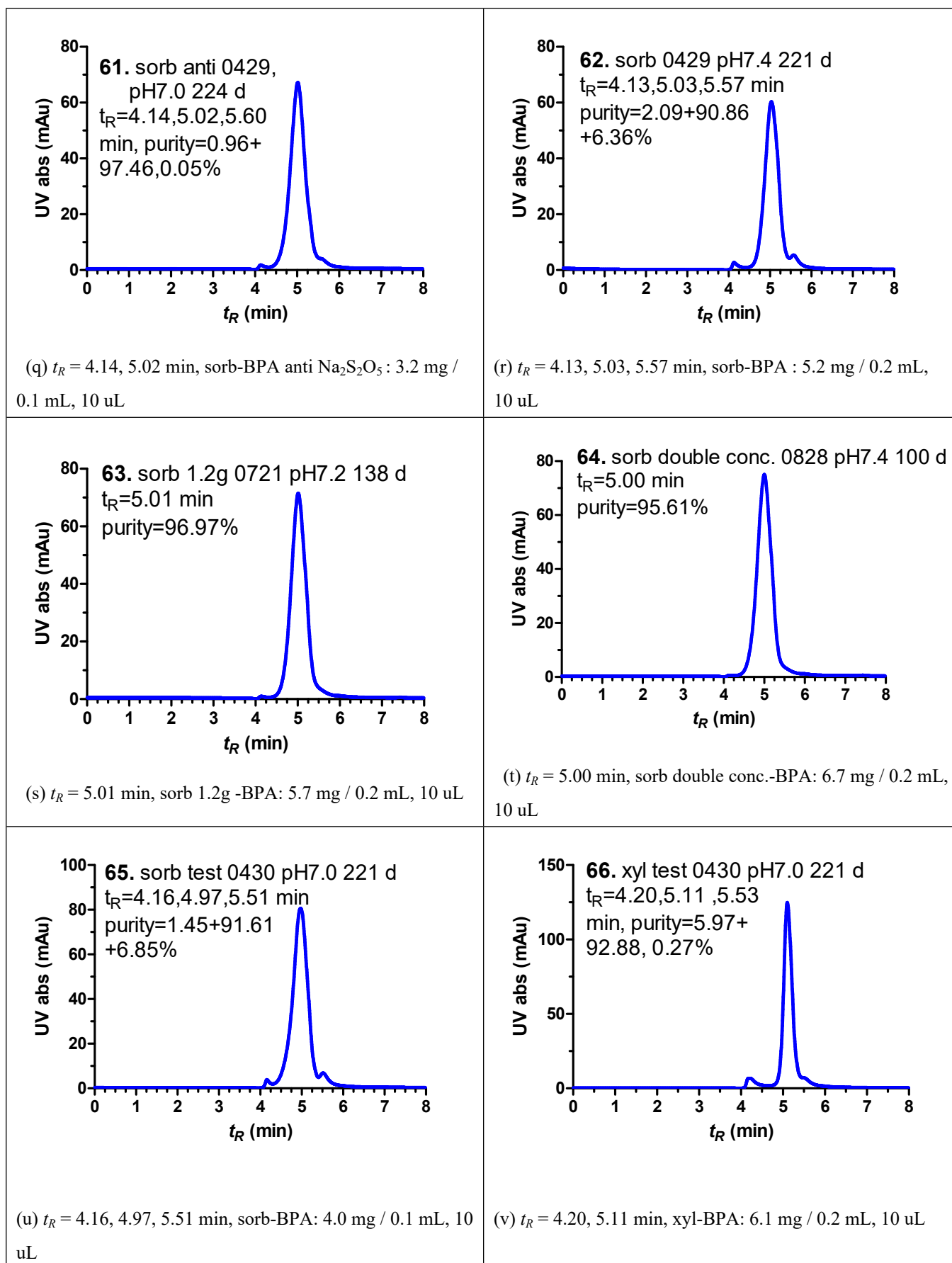
(n) $t_R = 5.00$ min, sorb-BPA anti $\text{Na}_2\text{S}_2\text{O}_5$: 4.2 mg / 0.1 mL, 10 uL



(o) $t_R = 4.36, 5.16$ min, sorb-BPA anti $\text{Na}_2\text{S}_2\text{O}_5$: 4.3 mg / 0.1 mL, 10 uL



(p) $t_R = 4.14, 5.02$ min, : sorb-BPA anti $\text{Na}_2\text{S}_2\text{O}_5$: 6.2 mg / 0.2 mL, 10 uL



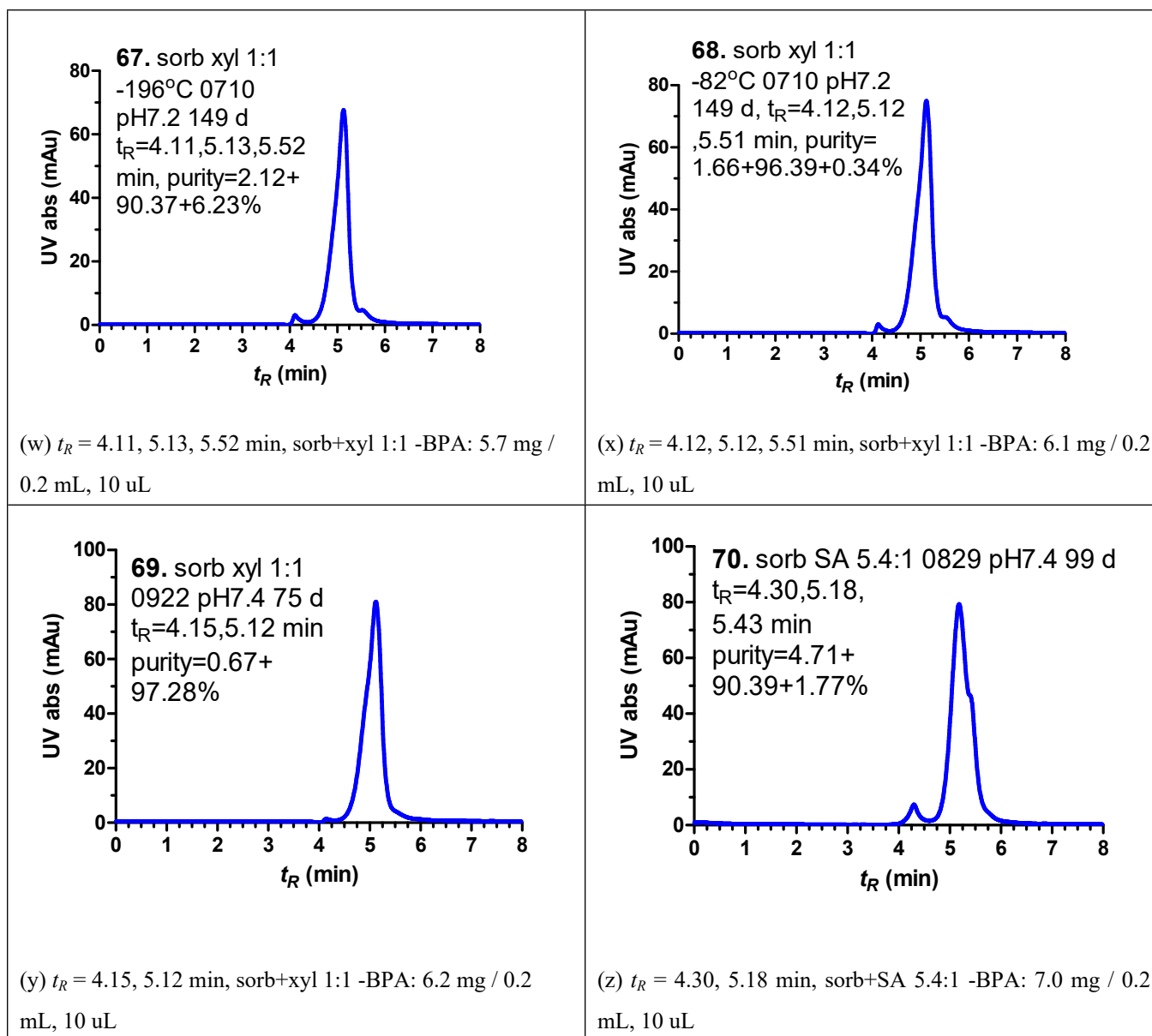


Table 12. Comparison of the stability of the BPA-saccharide lyophilized samples from different formula within 270-day storage.

Condition [§]			Intact BPA			Degraded form					
						tyrosine			phenylalanine		
formulation	storage	saccharide [§]	Mean (%)	SEM (%)	N	Mean (%)	SEM (%)	N	Mean (%)	SEM (%)	N
Single formula	RT	Sorb	94.38	1.24	6	0.13	0.13	6	3.26	1.46	6
		Man	96.12	0.27	3	2.14	0.09	3	0.00	0.00	3
		Xyl RT	97.04	2.09	3	0.00	0.00	3	0.09	0.09	3

		Fru RT	99.43	0.00	1	0.00	0.00	1	0.00	0.00	1
	-20 °C	Sorb	97.17	0.48	4	0.31	0.28	4	0.10	0.01	4
		Man	96.62	0.38	3	0.21	0.11	3	0.00	0.00	3
		Xyl	95.35	1.95	3	0.00	0.00	3	0.00	0.00	3
		Fru	94.00	0.00	1	0.00	0.00	1	0.00	0.00	1
Precooling effect (-82 °C)	-20 °C	Sorb	95.87	1.00	3	0.00	0.00	3	0.00	0.00	3
		Xyl	97.70	0.61	3	0.00	0.00	3	0.00	0.00	3
Dual formula	-20 °C	Sorb/SA	98.82	0.06	3	0.35	0.03	3	0.00	0.00	3
	RT	Sorb/SA	95.25	1.57	5	1.65	0.53	5	0.00	0.00	5
		Sorb/xyl	96.11	1.32	7	0.03	0.03	7	1.80	1.12	7
		Sorb/man	96.74	0.72	7	0.30	0.20	7	0.06	0.03	7
Acid adjuster	-20 °C	Sorb SA 2M	98.38	0.53	3	0.00	0.00	3	0.00	0.00	3
	RT	Sorb GA 6.4M	99.02	0.30	3	0.00	0.00	3	0.00	0.00	3
Antioxidation	RT	Sorb	98.25	0.30	4	0.00	0.00	4	0.06	0.01	4
		Sorb (AA)	99.56	0.11	3	0.02	0.02	3	0.00	0.00	3
	-20 °C	Sorb	96.35	0.71	3	0.00	0.00	3	0.00	0.00	3
		Xyl	95.14	1.90	3	0.00	0.00	3	0.00	0.00	3

The presence of degraded byproducts: tyrosine and phenylalanine were analyzed through RP-HPLC chromatogram. * Denoting the condition. Antioxidation:

Na₂S₂O₅, RT: room-temperature storage, -20 °C: -20 °C storage, -82 °C: formation of the complex at -196 °C unless specified at -82 °C, § Sorb: sorbitol, SA: sialic

acid, Xyl: xylitol, Man: mannitol, Fru: fructose, GA: gluconic acid, AA: ascorbic acid as antioxidant, Dual formula: sorbitol+sialic acid ranging from 2:1 to 7:1,

sorbitol+xylitol ranging from 1:2 to 3:1 and sorbitol+mannitol ranging from 2:3 to 4:1. Mean: average of the data taken for samples with similar conditions, SEM:

standard error of mean, N: number of samples involved for statistics.

Table 13. Osmotic pressure ratio of the BPA-saccharide lyophilized samples after dissolution

Condition ^v			Osmotic pressure ratio ^s		
	storage		Mean	SEM	N
Single formula	RT	Sorb	0.86	0.07	6
		Man	0.52	0.00	1
		Xyl RT	0.78	0.22	2
		Fru RT	0.49	0.00	1
	-20 °C	Sorb	0.83	0.03	4
		Man	0.82	0.03	3
		Xyl	0.97	0.11	3
		Fru	0.86	0.00	1
Precooling effect (-82 °C)	-20 °C	Sorb	0.85	0.04	3
		Xyl	0.99	0.02	3
Dual formula	-20 °C	Sorb/SA	0.90	0.00	1
	RT	Sorb/SA	0.75	0.03	5
		Sorb/xyl	0.98	0.10	9
		Sorb/man	0.90	0.06	8

Acid adjuster	-20 °C	Sorb SA 2M	0.87	0.01	3
	RT	Sorb GA 6.4M	0.79	0.05	3
Antioxidation	RT	Sorb	0.84	0.03	4
		Sorb (AA)	0.74	0.07	2
	-20 °C	Sorb	0.80	0.03	3
		Xyl	0.92	0.06	3

‡ Denoting the condition. Antioxidation: Na₂S₂O₅, RT: room-temperature storage, -20 °C: -20 °C storage, -82 °C: formation of the complex at -196 °C unless

specified at -82 °C, § Sorb: sorbitol, SA: sialic acid, Xyl: xylitol, Man: mannitol, Fru: fructose, GA: gluconic acid, AA: ascorbic acid as antioxidant, Dual formula:

sorbitol+sialic acid ranging from 2:1 to 7:1, sorbitol+xylitol ranging from 1:2 to 3:1 and sorbitol+mannitol ranging from 2:3 to 4:1. Mean: average of the data

taken for samples with similar conditions, SEM: standard error of mean, N: number of samples involved for statistics. §Osmotic pressure of sample in unit of

mOsm/kg comparing to saline (292 mOsm/kg) to derive the ratio.

Table 14. Anti-oxidation effects on BPA-saccharide complexation ratio derived from ¹H-NMR integral of the lyophilized samples from various conditions

Condition[‡]			complex to free BPA ratio		
formulation	storage	saccharide [§]	Mean	SEM	N
Single formula	RT	Sorb	12.66	1.62	6
		Man	7.81	0.00	1
		Xyl RT	3.68	0.28	2
		Fru RT	8.03	0.00	1

	-20 °C	Sorb	11.37	2.39	4
		Man	6.26	0.66	3
		Xyl	5.50	1.01	3
		Fru	4.23	0.00	1
Precooling effect (-82 °C)	-20 °C	Sorb	12.74	1.68	3
		Xyl	5.72	0.88	3
Dual formula	-20 °C	Sorb/SA	10.31	0.00	1
	RT	Sorb/SA	10.70	0.50	5
		Sorb/xyl	9.74	0.74	9
		Sorb/man	11.01	1.01	6
Acid adjuster	-20 °C	Sorb SA 2M	14.01	2.70	3
	RT	Sorb GA 6.4M	12.36	0.32	3
Antioxidation	RT	Sorb	17.59	2.14	4
		Sorb (AA)	14.68	1.07	2
	-20 °C	Sorb	10.66	2.11	3
		Xyl	5.08	0.76	3

‡ Denoting the condition. Antioxidation: Na₂S₂O₅, RT: room-temperature storage, -20 °C: -20 °C storage, -82 °C: preparation of the freeze-drying sample through

direct contact at -196 °C unless denotified by -82 °C, § Sorb: sorbitol, SA: sialic acid, Xyl: xylitol, Man: mannitol, Fru: fructose, GA: gluconic acid, AA: ascorbic

acid as antioxidant, Dual formula: sorbitol+sialic acid ranging from 2:1 to 7:1, sorbitol+xylitol ranging from 1:2 to 3:1 and sorbitol+mannitol ranging from 2:3 to

4:1. Mean: average of the data taken for samples with similar conditions, SEM: standard error of mean, N: number of samples involved for statistics.