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APPENDICES



APPENDIX A

Table A.1 Calibration curve of APTES in EtOH

[APTES] mM	Absorbance ($\lambda_{\max} = 238 \text{ nm}$)				SD
	1st	2nd	3rd	Average	
0.0000	0.000	0.000	0.000	0.00	0.0000
0.0003	0.102	0.101	0.102	0.102	0.0005
0.0013	0.212	0.211	0.213	0.212	0.0008
0.0025	0.420	0.421	0.419	0.420	0.0008
0.1000	0.846	0.845	0.845	0.845	0.0005
0.2000	1.690	1.688	1.685	1.688	0.0021

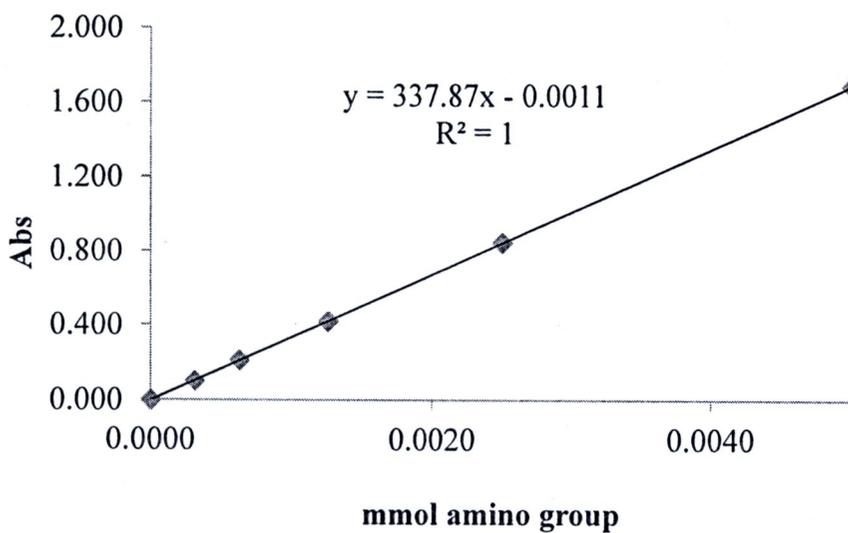


Figure A.1 Calibration curve of APTES in EtOH

Table A.2 The effect of APTES volume/ MNPs weight ratio to loading of amino group on aminopropyl magnetic silica surface

%APTES (%v/w)	Run	weight (mg)	A₅₇₀	Loading (mmol/g)	Average loading (mmol/g)
10	1	4.9	0.422	0.25	0.27
	2	4.8	0.470	0.29	
	3	5.0	0.473	0.28	
20	1	5.0	0.441	0.26	0.28
	2	5.0	0.455	0.27	
	3	4.8	0.501	0.31	
30	1	4.8	0.572	0.35	0.33
	2	4.8	0.453	0.28	
	3	5.1	0.641	0.37	
40	1	5.1	0.531	0.30	0.30
	2	4.9	0.502	0.30	
	3	4.8	0.510	0.31	
50	1	4.8	0.501	0.31	0.31
	2	4.9	0.533	0.32	
	3	4.9	0.535	0.32	

Table A.3 The effect of reaction times to loading of amino group on aminopropyl magnetic silica surface

Reaction (h)	Run	weight (mg)	A ₅₇₀	Loading (mmol/g)	Average loading (mmol/g)
0.5	1	13	0.212	0.0468	0.05
	2	13	0.213	0.0469	
	3	13	0.213	0.0470	
1	1	16	0.802	0.1458	0.15
	2	16	0.802	0.1457	
	3	16	0.802	0.1458	
2	1	13	0.744	0.1637	0.16
	2	13	0.743	0.1635	
	3	13	0.745	0.1638	
3	1	11	0.749	0.1975	0.20
	2	11	0.749	0.1974	
	3	11	0.749	0.1975	
5	1	9	1.013	0.3294	0.33
	2	9	1.012	0.3292	
	3	9	1.012	0.3291	
7	1	9	0.937	0.3115	0.31
	2	9	0.936	0.3112	
	3	9	0.937	0.3115	
9	1	8	0.931	0.3243	0.32
	2	8	0.931	0.3243	
	3	8	0.931	0.3244	

Table A.4 Calibration curve of NA in 6:1 (v/v) EtOH:H₂O

[NA] mM	Absorbance ($\lambda_{\max} = 262 \text{ nm}$)				SD
	1st	2nd	3rd	Average	
0.00	0.000	0.000	0.000	0.00	0.0000
0.10	0.297	0.296	0.296	0.296	0.0006
0.20	0.529	0.529	0.528	0.529	0.0006
0.40	1.009	1.007	1.01	1.009	0.0015
0.80	1.803	1.802	1.801	1.802	0.0010

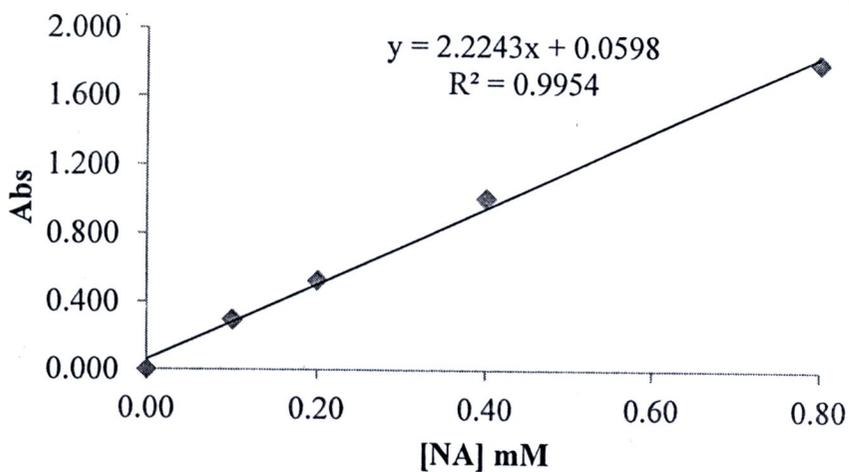
**Figure A.2** Calibration curve of NA in 6:1 (v/v) EtOH:H₂O

Table A.5 Calibration curve of NAM in 6:1 (v/v) EtOH:H₂O

[NAM] mM	Absorbance ($\lambda_{\max} = 262 \text{ nm}$)				SD
	1st	2nd	3rd	Average	
0.00	0.000	0.000	0.000	0.00	0.0000
0.10	0.364	0.365	0.366	0.365	0.0010
0.20	0.642	0.651	0.654	0.649	0.0062
0.40	1.188	1.187	1.186	1.187	0.0010
0.80	2.231	2.228	2.230	2.230	0.0015

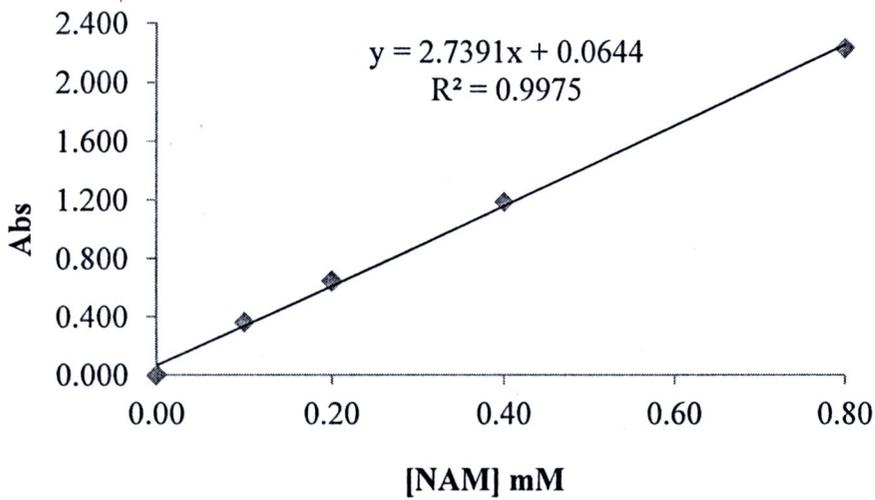
**Figure A.3** Calibration curve of NAM in 6:1 (v/v) EtOH:H₂O

Table A.6 Calibration curve of 3-APY in 6:1 (v/v) EtOH:H₂O

[3-APY] mM	Absorbance ($\lambda_{\max} = 238 \text{ nm}$)				SD
	1st	2nd	3rd	Average	
0.0000	0.000	0.000	0.000	0.00	0.0000
0.0125	0.145	0.144	0.145	0.145	0.0006
0.0250	0.245	0.244	0.243	0.244	0.0010
0.0500	0.466	0.465	0.465	0.465	0.0006
0.1000	0.919	0.948	0.944	0.937	0.0157
0.2000	1.806	1.809	1.809	1.807	0.0017

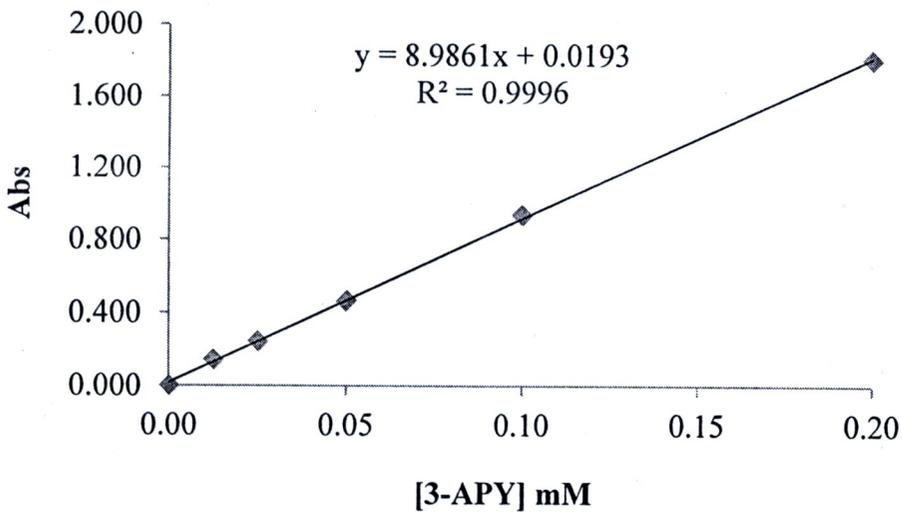
**Figure A.4** Calibration curve of 3-APY in 6:1 (v/v) EtOH:H₂O

Table A.7 Calibration curve of NA in 1:1 (v/v) EtOH/H₂O

[NA] mM	Absorbance ($\lambda_{\max} = 262 \text{ nm}$)				SD
	1st	2nd	3rd	Average	
0.00	0.000	0.000	0.000	0.00	0.0000
0.10	0.347	0.345	0.344	0.345	0.0015
0.20	0.688	0.692	0.694	0.691	0.0031
0.40	1.452	1.455	1.452	1.453	0.0017
0.60	2.072	2.082	2.079	2.078	0.0051
0.80	2.599	2.597	2.599	2.598	0.0012

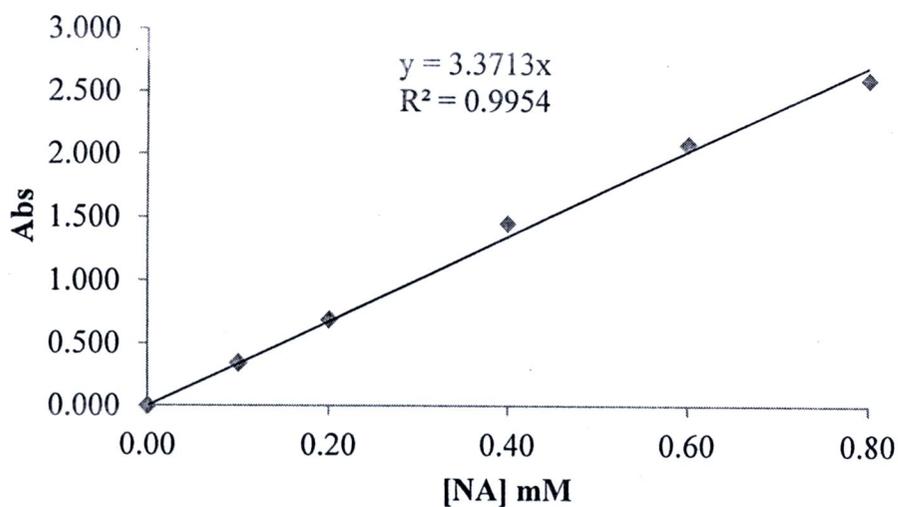
**Figure A.5** Calibration curve of NA in 1:1 (v/v) EtOH/H₂O

Table A.8 Calibration curve of NA in EtOH

[NA] mM	Absorbance ($\lambda_{\max} = 262 \text{ nm}$)				SD
	1st	2nd	3rd	Average	
0.00	0.000	0.000	0.000	0.00	0.0000
0.10	0.300	0.298	0.299	0.299	0.0010
0.20	0.592	0.592	0.593	0.592	0.0006
0.40	1.170	1.173	1.171	1.171	0.0015
0.60	1.678	1.680	1.678	1.679	0.0012
0.80	2.197	2.197	2.202	2.199	0.0029

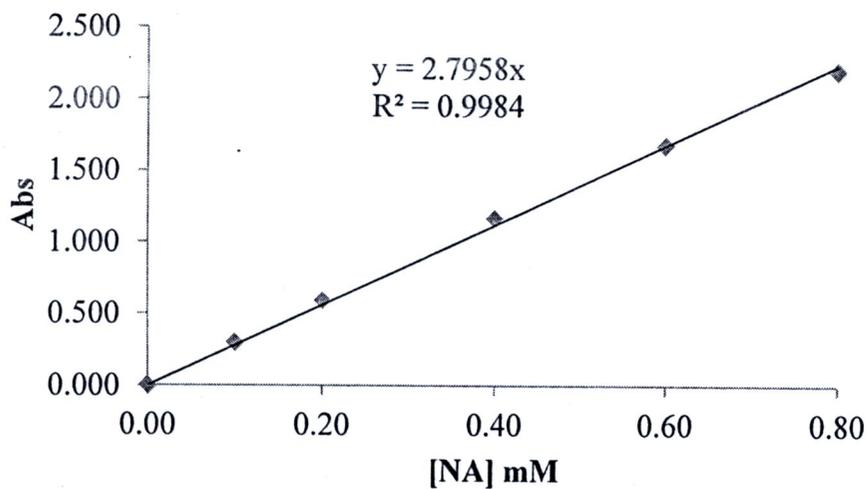
**Figure A.6** Calibration curve of NA in EtOH

Table A.9 Calibration curve of NA in MeOH

[NA] mM	Absorbance ($\lambda_{\max} = 262 \text{ nm}$)				SD
	1st	2nd	3rd	Average	
0.00	0.000	0.000	0.000	0.00	0.0000
0.10	0.324	0.325	0.324	0.324	0.0006
0.20	0.630	0.630	0.631	0.630	0.0006
0.40	1.165	1.167	1.170	1.167	0.0025
0.60	1.743	1.738	1.740	1.740	0.0025
0.80	2.181	2.185	2.191	2.186	0.0050

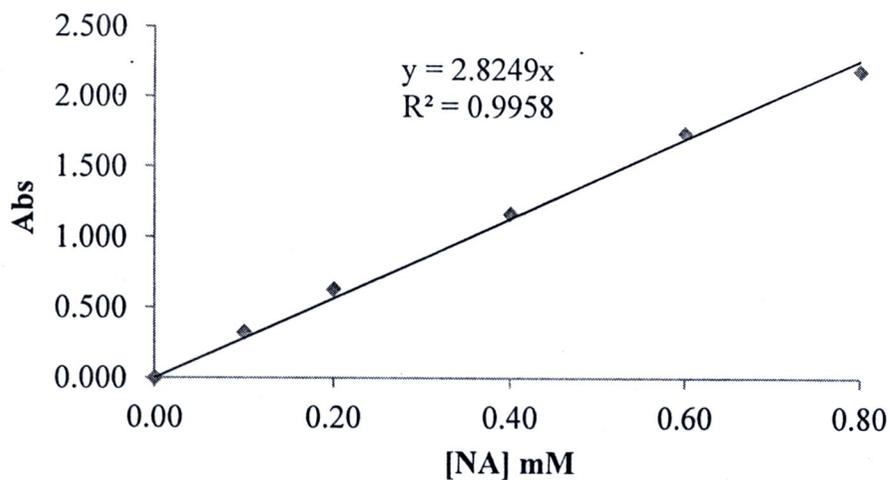
**Figure A.7** Calibration curve of NA in MeOH

Table A.10 Calibration curve of NA in MeCN

[NA] mM	Absorbance ($\lambda_{\max} = 262 \text{ nm}$)				SD
	1st	2nd	3rd	Average	
0.00	0.000	0.000	0.000	0.00	0.0000
0.10	0.244	0.244	0.243	0.244	0.0006
0.20	0.590	0.589	0.588	0.589	0.0010
0.40	1.015	1.013	1.012	1.013	0.0015
0.60	1.647	1.648	1.653	1.649	0.0032
0.80	2.062	2.061	2.061	2.062	0.0010

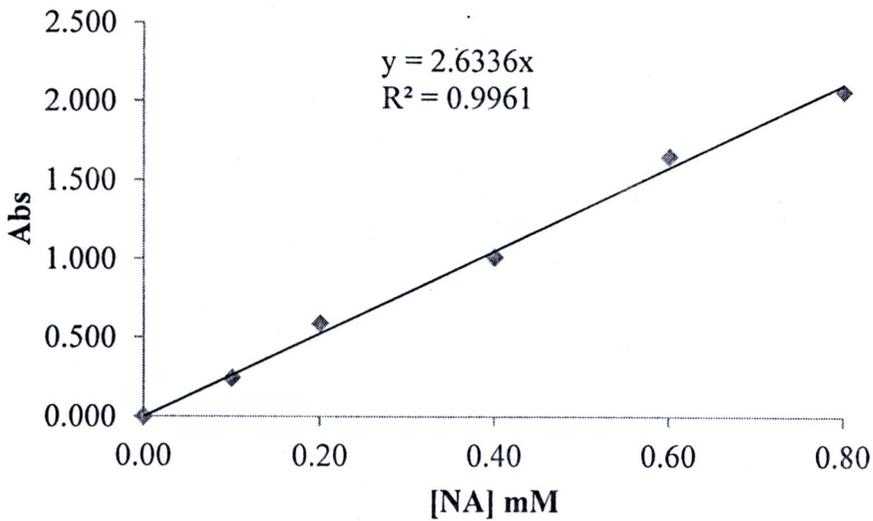
**Figure A.8** Calibration curve of NA in MeCN

Table A.11 Calibration curve of NA in 4:1 (v/v) MeCN

[NA] mM	Absorbance ($\lambda_{\max} = 262 \text{ nm}$)				SD
	1st	2nd	3rd	Average	
0.00	0.000	0.000	0.000	0.00	0.0000
0.10	0.314	0.313	0.314	0.314	0.0006
0.20	0.574	0.574	0.574	0.574	0.0000
0.40	1.234	1.234	1.234	1.234	0.0000
0.60	1.819	1.820	1.816	1.818	0.0021
0.80	2.242	2.244	2.243	2.243	0.0012

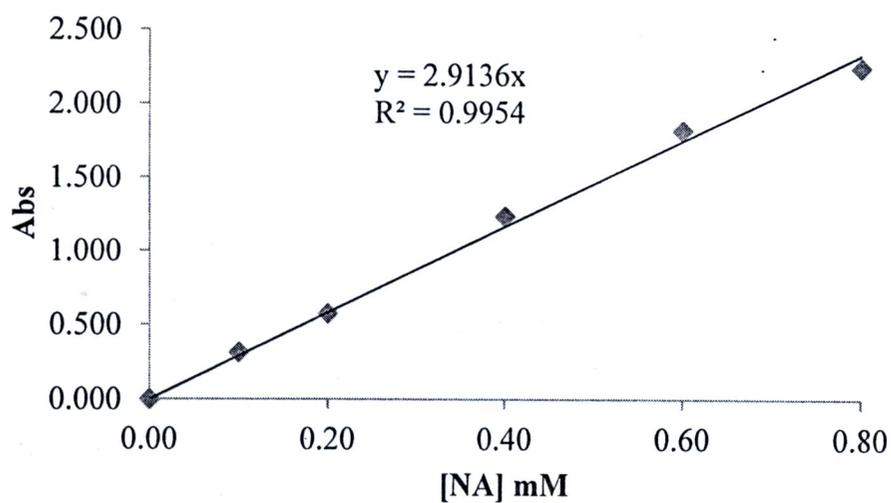
**Figure A.9** Calibration curve of NA in 4:1 (v/v) MeCN

Table A.12 Calibration curve of NAM in EtOH

[NAM] mM	Absorbance ($\lambda_{\max} = 262 \text{ nm}$)				SD
	1st	2nd	3rd	Average	
0.00	0.000	0.000	0.000	0.00	0.0000
0.10	0.314	0.315	0.315	0.315	0.0006
0.20	0.556	0.552	0.556	0.555	0.0023
0.40	1.171	1.172	1.74	1.172	0.0015
0.60	1.757	1.755	1.756	1.756	0.0010
0.80	2.150	2.150	2.152	2.151	0.0012

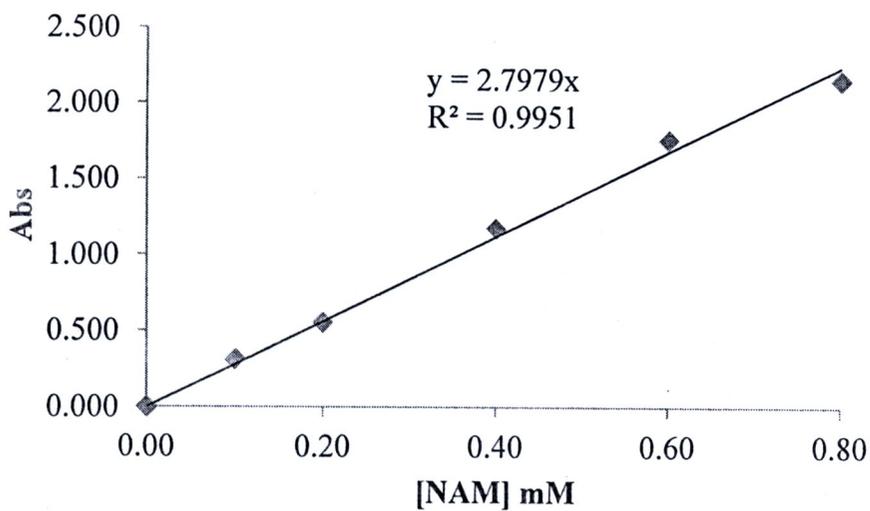
**Figure A.10** Calibration curve of NAM in EtOH

Table A.13 Calibration curve of NTA in EtOH

[NAM] mM	Absorbance ($\lambda_{\max} = 262 \text{ nm}$)				SD
	1st	2nd	3rd	Average	
0.00	0.000	0.000	0.000	0.00	0.0000
0.10	0.288	0.288	0.288	0.288	0.0000
0.20	0.556	0.552	0.556	0.555	0.0023
0.40	1.181	1.184	1.184	1.183	0.0017
0.60	1.847	1.849	1.846	1.847	0.0015
0.80	2.235	2.243	2.233	2.237	0.0053

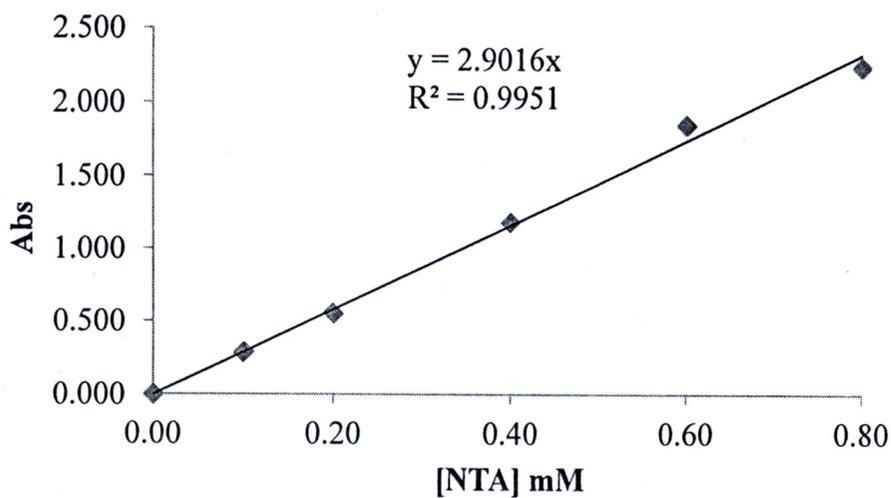
**Figure A.11** Calibration curve of NTA in EtOH



Table A.14 Calibration curve of C-NA in EtOH

[C-NA] mM	Absorbance ($\lambda_{\max} = 270 \text{ nm}$)				SD
	1st	2nd	3rd	Average	
0.00	0.000	0.000	0.000	0.00	0.0000
0.10	0.354	0.353	0.358	0.355	0.0026
0.20	0.672	0.669	0.674	0.672	0.0025
0.40	1.187	1.187	1.187	1.187	0.0000
0.60	1.811	1.814	1.809	1.811	0.0025
0.80	2.296	2.294	2.296	2.29	0.0012

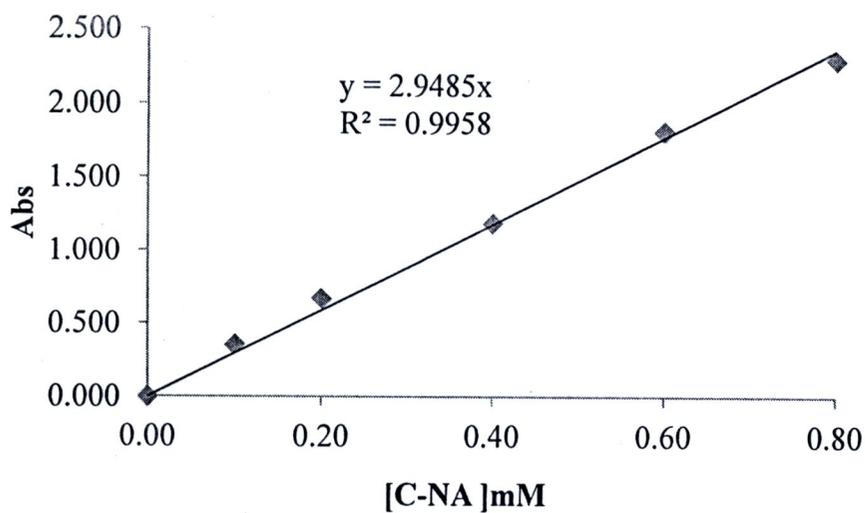


Figure A.12 Calibration curve of C-NA in EtOH

Table A.15 Calibration curve of BZA in EtOH

[BZA] mM	Absorbance ($\lambda_{\max} = 272 \text{ nm}$)				SD
	1st	2nd	3rd	Average	
0.00	0.000	0.000	0.000	0.00	0.0000
0.10	0.110	0.112	0.112	0.111	0.0012
0.20	0.184	0.185	0.183	0.184	0.0010
0.40	0.360	0.360	0.360	0.360	0.0000
0.60	0.520	0.521	0.522	0.521	0.0010
0.80	0.680	0.679	0.680	0.680	0.0006

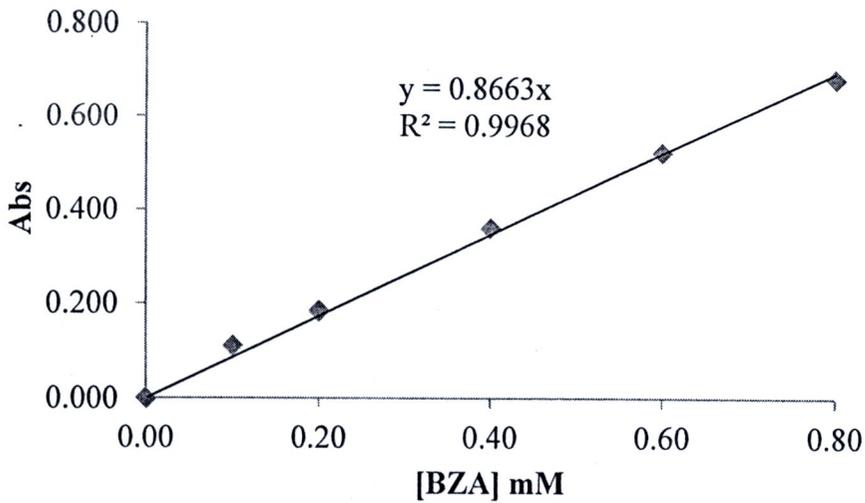
**Figure A.13** Calibration curve of BZA in EtOH

Table A.16 Calibration curve of 3-APY in EtOH

[3-APY] mM	Absorbance ($\lambda_{\max} = 240 \text{ nm}$)				SD
	1st	2nd	3rd	Average	
0.000	0.000	0.000	0.000	0.00	0.0000
0.025	0.271	0.272	0.271	0.271	0.0006
0.050	0.553	0.553	0.553	0.553	0.0000
0.100	1.009	1.009	1.008	1.009	0.0006
0.150	1.477	1.479	1.477	1.478	0.0012
0.200	1.873	1.869	1.878	1.873	0.0045

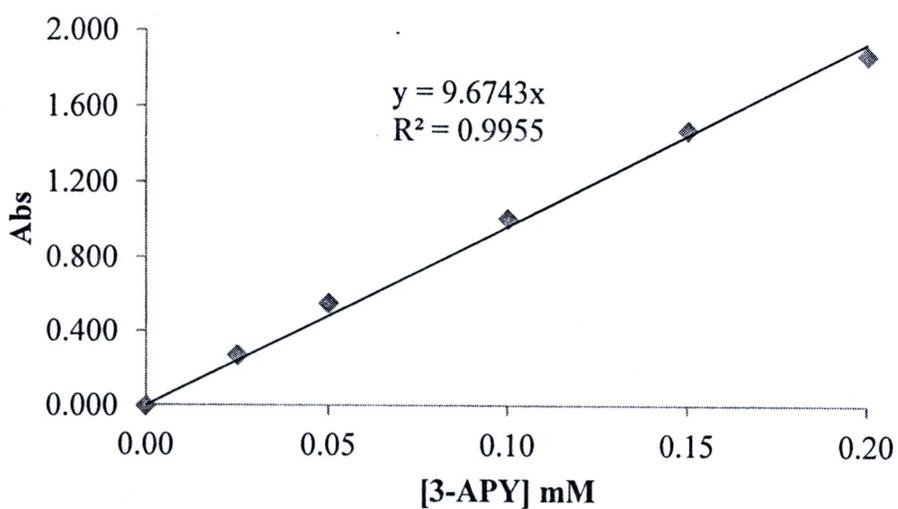
**Figure A.14** Calibration curve of 3-APY in EtOH

Table A.17 The binding study of 0.2 mM NA in 6:1 (v/v) EtOH:H₂O with 10 mg of M-MIS 1 and M-NIS.

Imprinted silicas	Absorbance ($\lambda_{\max} = 262 \text{ nm}$)				%Bound	SD	α
	1st	2nd	3rd	Average			
M-MIS 1	0.484	0.482	0.484	0.483	32.0525	0.1623	2.04
M-NIS	0.618	0.589	0.591	0.599	15.7451	2.2770	

Table A.18 The binding study of 0.2 mM NAM in 6:1 (v/v) EtOH:H₂O with 10 mg of M-MIS 2 and M-NIS.

Imprinted silicas	Absorbance ($\lambda_{\max} = 262 \text{ nm}$)				%Bound	SD	α
	1st	2nd	3rd	Average			
M-MIS 2	0.593	0.617	0.594	0.601	13.4772	1.9535	1.25
M-NIS	0.614	0.631	0.616	0.620	10.7434	1.3369	

Table A.19 The binding study of 0.2 mM 3-APY in 6:1 (v/v) EtOH:H₂O with 10 mg of M-MIS 3 and M-NIS.

Imprinted silicas	Absorbance ($\lambda_{\max} = 238 \text{ nm}$)				%Bound	SD	α
	1st	2nd	3rd	Average			
M-MIS 3	1.719	1.736	1.716	1.724	-4.6338	0.6547	nd
M-NIS	1.654	1.645	1.660	1.653	-0.3440	0.4583	

*nd = can not calculate because it is negative value

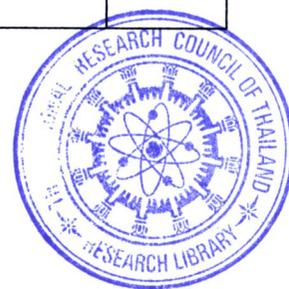


Table A.20 The binding study of 0.2 mM NA in 6:1 (v/v) EtOH:H₂O with 10 mg of M-MIS 1, M-MIS 2, M-MIS 3 and M-NIS.

Imprinted silicas	Absorbance ($\lambda_{\max} = 262 \text{ nm}$)				%Bound	SD	α
	1st	2nd	3rd	Average			
M-MIS1	0.484	0.482	0.484	0.483	32.0525	0.1623	2.04
M-MIS2	0.736	0.728	0.702	0.722	-1.4995	2.4990	nd
M-MIS3	0.634	0.612	0.605	0.617	13.2615	2.1274	0.84
M-NIS	0.618	0.589	0.591	0.599	15.7451	2.2770	-

*nd = can not calculate because it is negative value

Table A.21 The binding study of 0.2 mM NA in various media with 10 mg of M-MIS 1 and M-NIS.

Media	Absorbance ($\lambda_{\max} = 262 \text{ nm}$)				%Bound	SD	α
	1st	2nd	3rd	Average			
6:1 (v/v) EtOH/H ₂ O	0.484	0.482	0.484	0.483	32.0525	0.1623	2.04
1:1 (v/v) EtOH/H ₂ O	0.583	0.573	0.568	0.575	30.2589	0.9269	1.40
EtOH	0.213	0.205	0.214	0.211	67.0667	0.7493	1.99
MeOH	0.251	0.212	0.254	0.239	62.2035	3.7054	1.76
MeCN	0.030	0.056	0.056	0.047	91.6910	2.6351	1.06
4:1 (v/v) MeCN/ H ₂ O	0.520	0.518	0.515	0.518	18.4777	0.3963	4.35

Table A.22 %Bound of NA on to M-MIS 1 in various times

Time (min)	%Bound_{MIS}	SD_{MIS}	%Bound_{NIS}	SD_{NIS}
0	0.0000	0.0000	0.0000	0.0000
15	68.9042	1.2687	33.2760	3.9261
30	68.3249	2.4283	23.8310	1.4374
60	70.2966	2.6092	26.0632	5.1103
120	67.8120	0.0425	29.2299	1.0160
180	66.4880	1.3211	38.8321	4.4255
300	66.2163	0.3052	46.1024	0.2782

Table A.23 %Bound of NA on to M-MIS 1 in various concentrations

[NA] mM	Absorbance ($\lambda_{\max} = 262 \text{ nm}$)				%Bound	SD
	1st	2nd	3rd	Average		
0.10	0.208	0.208	0.206	0.207	31.2593	0.3930
0.20	0.213	0.214	0.205	0.211	67.0667	0.7493
0.30	0.252	0.357	0.315	0.308	67.8951	5.5691
0.40	0.400	0.398	0.402	0.400	66.6975	0.1679
0.50	0.521	0.447	0.433	0.467	69.3218	3.1305
1.00	0.114	0.125	0.115	0.118	67.5702	1.6294
1.50	0.174	0.172	0.177	0.174	69.8245	0.4950
2.00	0.255	0.246	0.255	0.252	62.5865	0.7468

Table A.24 %Bound of NA on to M-NIS in various concentrations

[NA] mM	Absorbance ($\lambda_{\max} = 262 \text{ nm}$)				%Bound	SD
	1st	2nd	3rd	Average		
0.10	0.216	0.213	0.228	0.219	27.4033	2.6311
0.20	0.434	0.419	0.417	0.423	33.7507	1.4541
0.30	0.459	0.509	0.545	0.504	47.3739	4.5067
0.40	0.641	0.637	0.642	0.640	46.6963	0.2204
0.50	0.809	0.819	0.829	0.819	46.2246	0.6566
1.00	0.221	0.221	0.220	0.221	39.4328	0.1585
1.50	0.368	0.354	0.355	0.359	37.6375	1.3567
2.00	0.528	0.528	0.519	0.525	21.9911	0.7721

Table A.25 Q_{Bound} of NA on to M-MIS 1 in various concentrations

[NA] mM	Q_{bound} ($\mu\text{mol/g}$)	
	M-MIS	M-NIS
0.10	2.5841	2.1668
0.20	12.4649	4.8582
0.30	18.9835	11.9610
0.40	25.6928	17.1085
0.50	33.2964	20.7061
1.00	57.7938	21.0721
1.50	87.6446	21.5931
2.00	109.8648	12.2183



Table A.26 Scatchard plot of M-MIS 1

Q_{bound} ($\mu\text{mol/g}$)	Q_{free}	$Q_{\text{bound}}/Q_{\text{free}}$
12.4649	0.0754	165.4241
18.9835	0.1102	172.3182
57.7938	0.4221	136.9322
87.6446	0.6236	140.5564
109.8648	0.9014	121.8889

Table A.27 Selectivity of M-MIS 1

Compounds	λ_{max} (nm)	Absorbance				%Bound	SD	α
		1st	2nd	3rd	Average			
NA	262	0.213	0.205	0.214	0.211	67.0667	0.7493	1.00
NAM	262	0.593	0.657	0.593	0.614	-2.0437	6.1423	-0.03
NTA	262	0.372	0.362	0.329	0.354	37.9655	3.9169	0.57
2-CNA	270	0.343	0.275	0.363	0.327	55.3888	6.2933	0.83
BZA	272	0.127	0.116	0.135	0.126	57.9319	3.1869	0.86
3-APY	240	1.991	1.986	1.984	1.987	-3.1854	0.1879	-0.05

Table A.28 Reusability of M-MIS 1

Cycles	Q_{bound} ($\mu\text{mol/g}$)	
	M-MIS	M-NIS
1	67.0667 \pm 0.7493	33.7507 \pm 1.4541
2	66.7570 \pm 0.6773	40.0759 \pm 0.3387
3	63.5644 \pm 0.7019	41.9802 \pm 1.2455
4	52.7919 \pm 1.9823	36.1252 \pm 0.8913
5	34.4079 \pm 0.4109	32.3026 \pm 0.3947
6	35.3665 \pm 1.2874	35.5805 \pm 0.4040

APPENDIX B

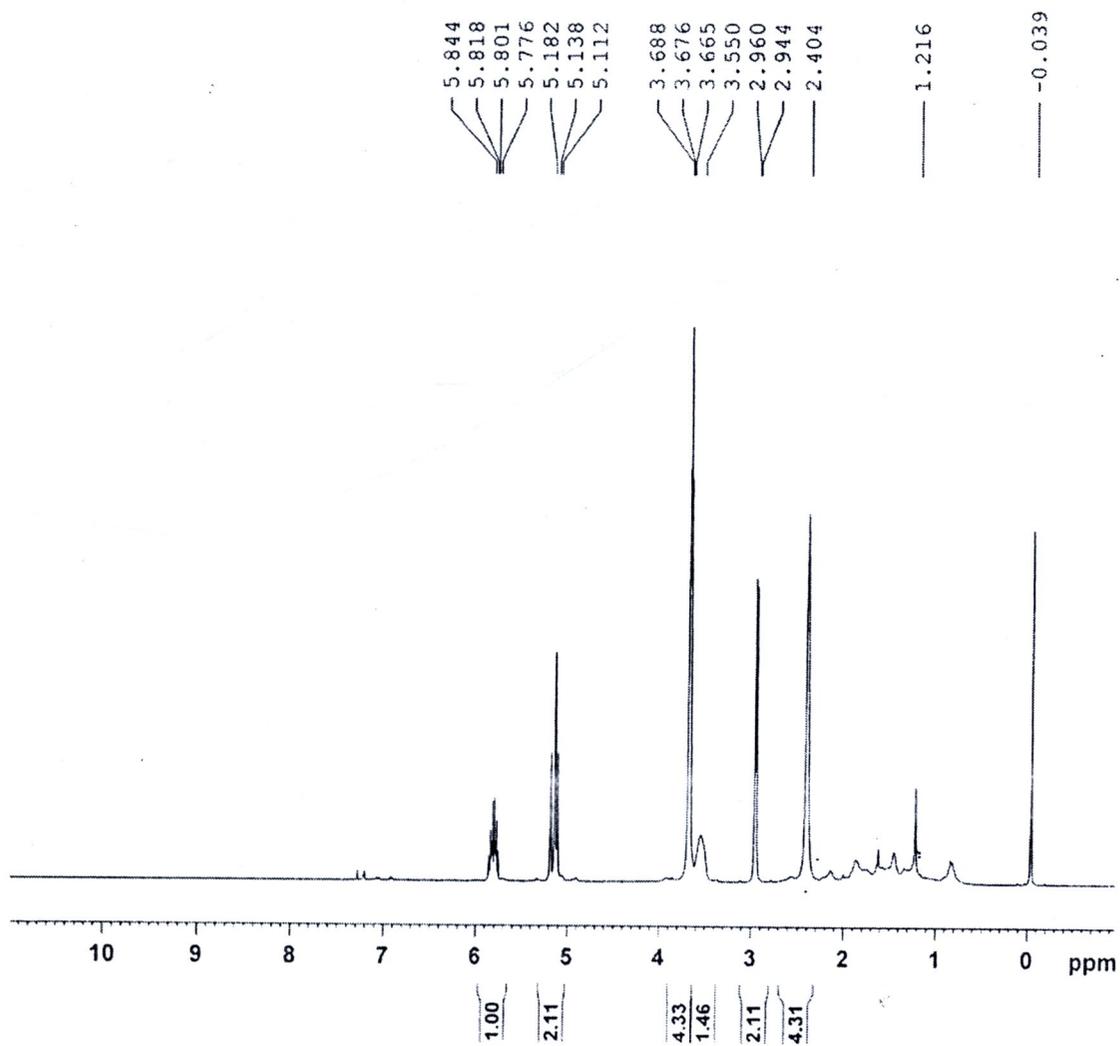


Figure B.1 The 400 MHz ^1H -NMR (CDCl_3) of allymorpholine

Table B.1 GC data for bromobenzene internal standard

Concentration	Peak area				SD
	1st	2nd	3rd	Average	
Conc. 1	1572.901	1603.869	1514.521	1563.764	45.36922
Conc. 2	1376.642	1392.294	1397.298	1388.745	10.77574
Conc. 3	1246.222	1236.933	1261.714	1248.29	12.51902
Conc. 4	1238.794	1306.674	1209.301	1251.589	49.93175
Conc. 5	1182.818	1199.311	1188.982	1190.371	8.333323

Table B.2 GC data for benzaldehyde

Concentration	Peak area				SD
	1st	2nd	3rd	Average	
Conc. 1	7738.537	7705.659	7964.146	7802.781	140.7104
Conc. 2	3480.865	3476.293	3472.31	3476.489	4.281212
Conc. 3	1269.472	1281.515	1271.515	1274.167	6.444557
Conc. 4	619.1576	594.098	594.9476	602.7344	14.22925
Conc. 5	107.5949	109.8382	110.715	109.3827	1.609156

Table B.3 GC calibration curve for benzaldehyde

Concentration	Mole ratio	Area ratio
Conc. 1	3.967742	4.989744
Conc. 2	1.983871	2.503332
Conc. 3	0.790323	1.020731
Conc. 4	0.395161	0.481575
Conc. 5	0.079032	0.09189

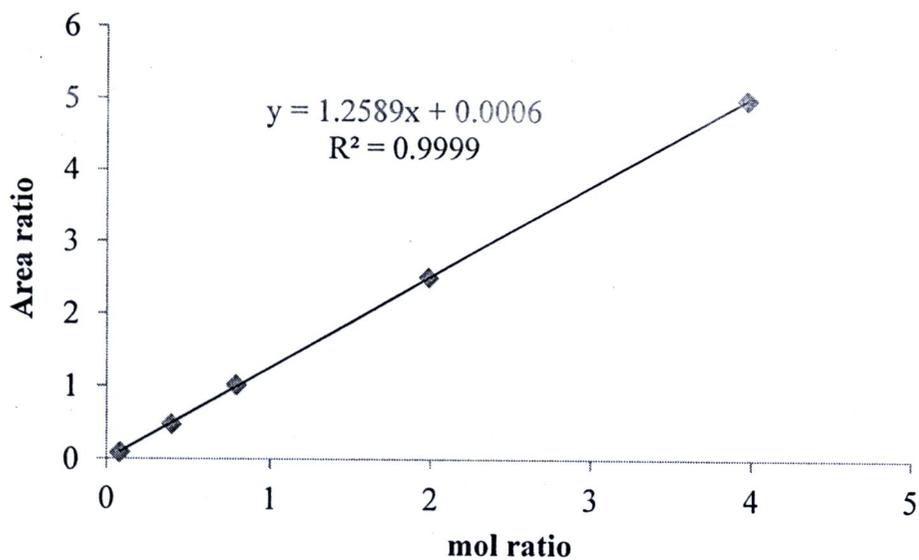
**Figure B.1** GC calibration curve for benzaldehyde

Table B.4 GC data for oxidation of benzyl chloride using M-NMO 1^a

Analytes	Run 1		Run 2		Run 3	
	Time	Peak area	Time	Peak area	Time	Peak area
Bromobenzene	5.301	754.5	5.301	750.7	5.303	732.8
Benzaldehyde	7.385	227.2	7.386	224.0	7.387	221.4
Benzylchloride	8.472	7,929.5	8.472	7,870.4	8.471	7,619.5

^aReaction condition : Benzylchloride (0.013 ml, 0.1 mmol), M-NMO 1 (300 mg), NaBr (cat. amount) and DMSO (0.7 ml), 25 °C, sonication times 20 min.

Table B.5 GC data for oxidation of benzyl chloride using M-NMO 2^a

Analytes	Run 1		Run 2		Run 3	
	Time	Peak area	Time	Peak area	Time	Peak area
Bromobenzene	5.310	563.5	5.309	571.0	5.309	565.8
Benzaldehyde	7.395	223.3	7.395	225.0	7.393	222.9
Benzylchloride	8.499	7,829.1	8.500	7,956.3	8.498	7,847.6

^aReaction condition : Benzylchloride (0.013 ml, 0.1 mmol), M-NMO 2 (300 mg), NaBr (cat. amount) and DMSO (0.7 ml), 25 °C, sonication times 20 min.

Table B.6 GC data for oxidation of benzyl chloride using M-NMO 3^a

Analytes	Run 1		Run 2		Run 3	
	Time	Peak area	Time	Peak area	Time	Peak area
Bromobenzene	5.309	563.5	5.309	571.0	5.309	565.8
Benzaldehyde	nd	nd	nd	nd	nd	nd
Benzylchloride	8.499	7,829.1	8.500	7,956.3	8.498	7,847.6

^aReaction condition : Benzylchloride (0.013 ml, 0.1 mmol), M-NMO 3 (300 mg), NaBr (cat. amount) and DMSO (0.7 ml), 25 °C, sonication times 20 min.

nd = not detected

Table B.7 GC data for oxidation of benzyl bromide using M-NMO 1^a

Analytes	Run 1		Run 2		Run 3	
	Time	Peak area	Time	Peak area	Time	Peak area
Bromobenzene	5.289	881.1	5.299	889.5	5.301	565.8
Benzaldehyde	7.359	130.6	7.36	117.5	7.361	110.4
Benzylbromide	8.494	17,653.0	8.495	17,572.2	8.490	17,082.0

^aReaction condition : Benzylbromide (0.017 ml, 0.1 mmol), M-NMO 1 (300 mg), NaBr (cat. amount) and DMSO (0.7 ml), 25 °C, sonication times 20 min.

Table B.8 GC data for oxidation of benzyl bromide using M-NMO 2^a

Analytes	Run 1		Run 2		Run 3	
	Time	Peak area	Time	Peak area	Time	Peak area
Bromobenzene	5.309	663.7	5.309	659.5	5.310	654.1
Benzaldehyde	7.393	313.6	7.392	312.0	7.394	308.0
Benzylbromide	8.501	7,789.1	8.499	7,887.6	8.501	7,879.4

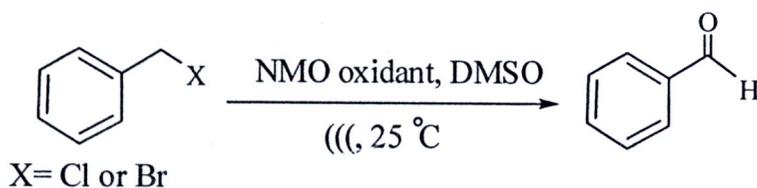
^aReaction condition : Benzylbromide (0.017 ml, 0.1 mmol), M-NMO 2 (300 mg), NaBr (cat. amount) and DMSO (0.7 ml), 25 °C, sonication times 20 min.

Table B.9 GC data for oxidation of benzyl bromide using M-NMO 3^a

Analytes	Run 1		Run 2		Run 3	
	Time	Peak area	Time	Peak area	Time	Peak area
Bromobenzene	5.303	585.5	5.303	641.0	5.306	614.2
Benzaldehyde	7.389	183.2	7.391	193.1	7.393	184.7
Benzylbromide	8.495	7,833.4	8.497	8,205	8.498	7,893.1

^aReaction condition : Benzylbromide (0.017 ml, 0.1 mmol), M-NMO 3 (300 mg), NaBr (cat. amount) and DMSO (0.7 ml), 25 °C, sonication times 20 min.

Table B.10 The percentage yield of the oxidation of benzyl halide to benzaldehyde using various NMO oxidants



Entry	Substrates	Reagent	Conditions	% Yield
1	BnBr	NMO	a	90.8 ^c
2	BnBr	M-NMO 1	b	4.1
3	BnBr	M-NMO 2	b	13.9
4	BnBr	M-NMO 3	b	9.0
5	BnBr	-	a	0.0 ^d
6	BnCl	NMO	a	92.2 ^c
7	BnCl	M-NMO 1	b	11.3
8	BnCl	M-NMO 2	b	15.1
9	BnCl	M-NMO 3	b	0.0
10	BnCl	-	a	0.0 ^d

^a sonicate, 10 min, 25 °C

^b sonicate, 20 min, 25 °C

^c Isolated yield

^dTLC

Table B.11 GC data for oxidation of benzyl chloride using M-NMO **2** in various solvents^a

Solvent	Analytes	Run 1		Run 2		Run 3	
		Time	Peak area	Time	Peak area	Time	Peak area
DMSO	Bromobenzene	5.310	563.5	5.309	571.0	5.309	565.8
	Benzaldehyde	7.395	223.3	7.395	225.0	7.393	222.9
DMF	Bromobenzene	5.271	813.4	5.270	784.6	5.274	844.3
	Benzaldehyde	7.362	319.8	7.370	302.4	7.366	333.5
MeCN	Bromobenzene	5.369	572.3	5.343	684.2	5.422	760.8
	Benzaldehyde	7.756	109.2	7.721	192.8	7.886	105.8
THF	Bromobenzene	5.426	426.7	5.149	352.9	5.521	317.4
	Benzaldehyde	7.843	168.4	7.838	90.2	8.139	89.4
MeOH	Bromobenzene	5.389	652.5	5.354	683.9	5.394	643.3
	Benzaldehyde	7.779	206.4	7.729	181.6	7.774	150.2
Toluene	Bromobenzene	5.329	577.0	5.337	597.1	5.336	425.7
	Benzaldehyde	7.693	198.0	7.713	229.6	7.707	64.4

^aReaction condition : Benzylchloride (0.013 ml, 0.1 mmol), M-NMO **2** (300 mg), NaBr (cat. amount) and solvent (0.7 ml), 25 °C, sonication times 20 min.

APPENDIX C

C. 1 Calculation loading of acrylamide-functionalized groups on magnetic silica nanoparticles

$$\text{Loading of aminopropyl magnetic silica beads} = 0.26 \text{ mmol/g}$$

$$\text{This revealed that beads 1 g contain aminopropyl groups} = 0.26 \text{ mmol}$$

$$\begin{aligned} \text{Weight of bead containing 1 mol of functional groups} &= \frac{1 \text{ mol} \times 1000}{0.26 \text{ mmol/g}} \\ &= 3,846.15 \text{ g} \end{aligned}$$

This calculated value indicated that molecular weight of bead is 3,846.15 g/mol

The aminopropyl magnetic silica beads were modified with acrylic acid to form acrylamide-functionalized magnetic silica nanoparticles

$$\text{Additive molecular weight of acrylic acid moiety} = 55.06 \text{ g/mol}$$

Therefore, molecular weight of acrylamide-functionalized magnetic silica beads can be calculated from

$$\begin{aligned} \text{Molecular weight of acrylamide-functionalized beads} &= 3,846.15 + 55.06 \text{ g/mol} \\ &= 3,901.21 \text{ g/mol} \end{aligned}$$

$$\begin{aligned} \text{So, loading of acrylamide-functionalized beads} &= \frac{1}{3,901.21 \text{ g/mol}} \\ &= 2.56 \times 10^{-4} \text{ mol/g} \\ &= 0.256 \text{ mmol/g} \end{aligned}$$

C. 2 Calculation theoretical loading of M-NMO

For example, M-NMM 1 synthesized from allymorpholine 13.66 mmol

In assumption;

If 100% yield, M-NMM should be contained of NMM = 13.66 mmol

From result, weight of M-NMM is 4.9074 g

Theoretical loading of M-NMM 1 = 13.66 mmol/4.9074 g

$$= 2.78 \text{ mmol/g}$$

This revealed that 1 g M-NMM 1 contain NMM groups = 2.78 mmol

Weight of M-NMM 1 containing 1 mol of NMM groups = $\frac{1 \text{ mol}}{2.78 \text{ mmol/g}}$

$$= 359.71 \text{ g}$$

This calculated value indicated that molecular weight of M-NMM 1 is 359.71 g/mol

Then, the M-NMM 1 was converted to M-NMO 1

Additive molecular weight of oxygen moiety = 16 g/mol

Therefore, molecular weight of M-NMO 1 can be calculated from

Molecular weight of 1 mol M-NMO 1 = 359.71 + 16 g/mol

$$= 375.71 \text{ g/mol}$$

So, theoretical loading of M-NMO 1 = $\frac{1}{375.71 \text{ g/mol}}$

$$= 2.66 \text{ mmol/g}$$

C. 3 Calculation of effective loading of M-NMO

For example, M-NMO 1 was used as an oxidant in benzyl chloride oxidation.

The effective loading of M-NMO 1 can be calculated from %yield of aldehyde product comparing to that of the reaction using standard NMO reagent.

If a certain mole of standard NMO reagent was used to convert benzyl chloride to benzaldehyde, the effective loading of M-NMO 1 can be achieved by using this followed equation

$$\text{Effective loading (mmol g}^{-1}\text{ of support)} = \frac{\left(\frac{\text{Mol}_{\text{NMO}}}{\% \text{Yield}_{\text{NMO}}}\right) \times \% \text{Yield}_{\text{support}}}{\text{Wt}_{\text{support}} \text{ (g)}}$$

In case of 4 mmol of standard NMO reagent was used and provided 92.17 %yield.

Whilst 0.30 g of M-NMO 1 provided 11.34 %yield

Therefore, the effective loading of M-NMO 1 can be calculated as;

$$\begin{aligned} \text{Effective loading (mmol g}^{-1}\text{ of M-NMO 1)} &= \frac{(4 \text{ mmol}) \times 11.34\%}{\frac{92.17\%}{0.30 \text{ (g)}}} \\ &= 1.64 \text{ mmol/g} \end{aligned}$$

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International Conferences

Bannarak Khumraksa and Mookda Pattarawarapan,
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