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APPENDICES

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APPENDIX A

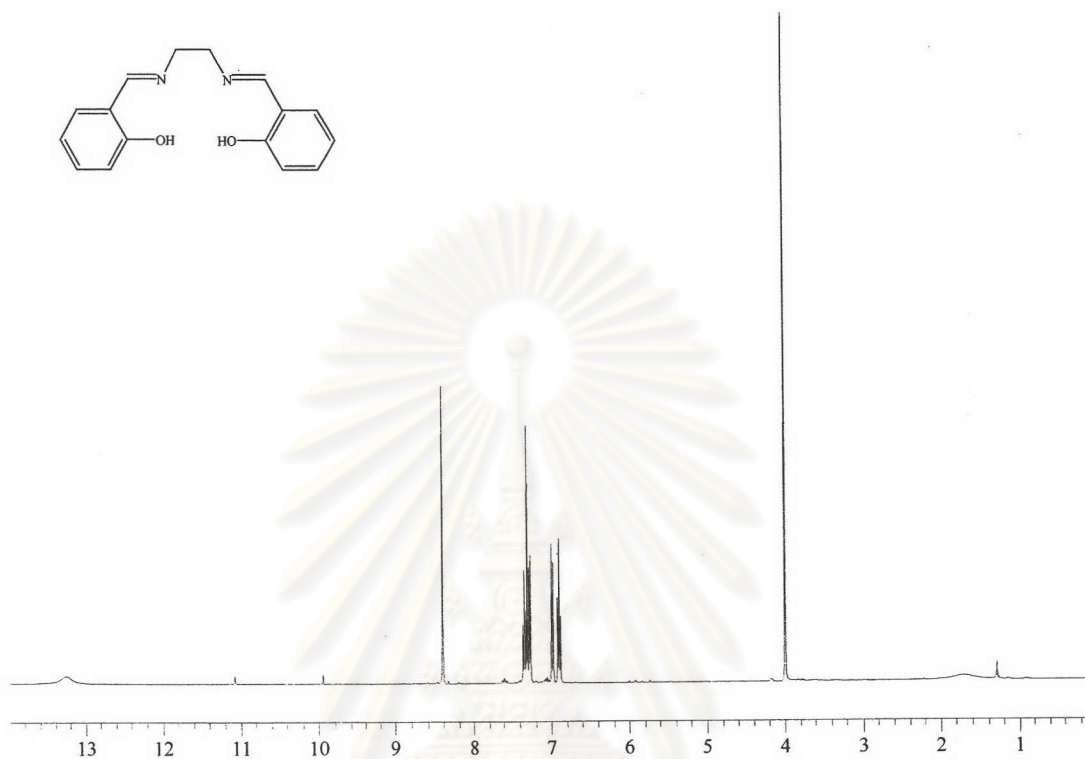
 $^1\text{H-NMR}$ spectrum of Schiff's base ligands

Figure A.1 $^1\text{H-NMR}$ spectrum of salen in CDCl_3 with 400 MHz

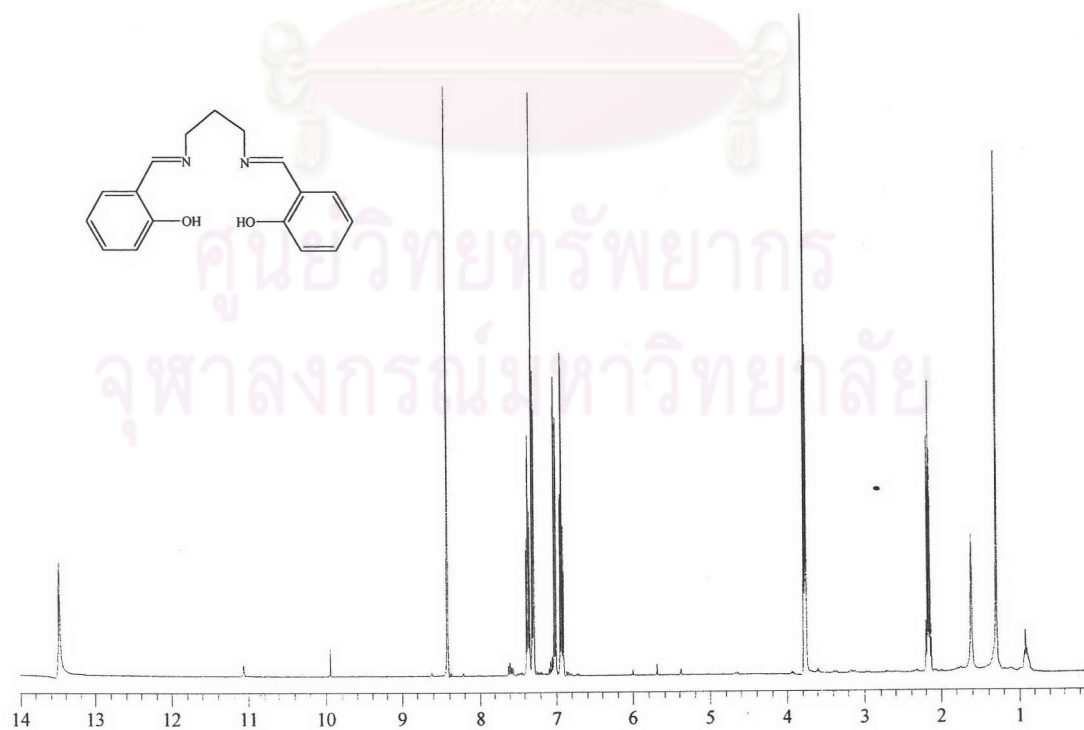


Figure A.2 $^1\text{H-NMR}$ spectrum of saltn in CDCl_3 with 400 MHz.

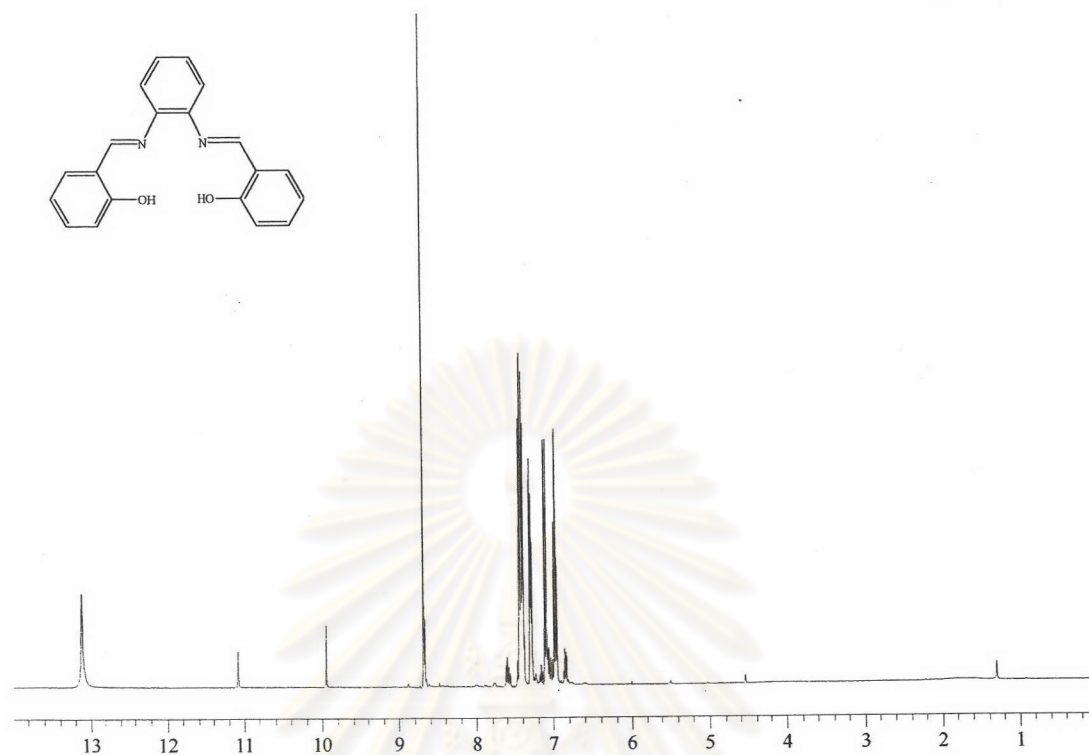


Figure A.3 ¹H-NMR spectrum of salophen in CDCl₃ with 400 MHz.

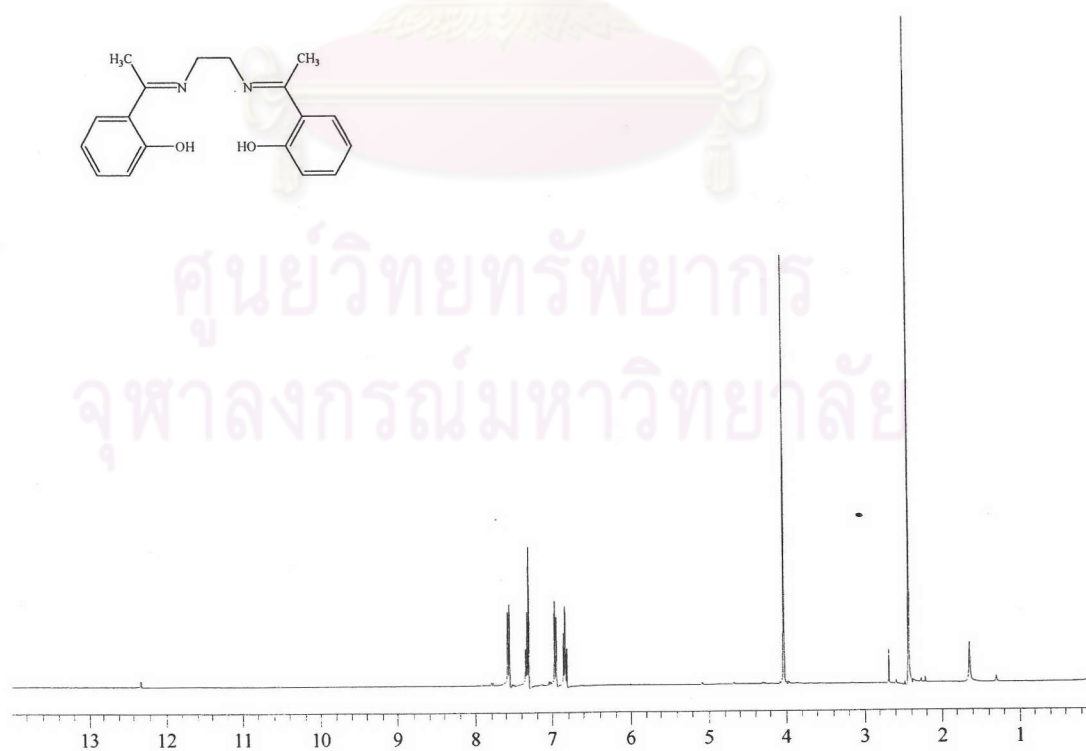


Figure A.4 ¹H-NMR spectrum of haen in CDCl₃ with 400 MHz.

APPENDIX B

**CALCULATION OF ORGANIC MATTER CONTENTS
IN MODIFIED MESOPOROUS SILICA**

The quantity of organic matters in the synthesized silica could be determined from the composition of starting materials shown in Table B.

Table B The amounts of starting materials used for the synthesis of various modified mesoporous silica.

Starting materials		Modified silica				
		Non-doped	Salen doped	Saltn doped	Salophen doped	Hean doped
TEOS	(g)	5.2086	5.2085	5.2087	5.2088	5.2072
	(mole)	0.0250	0.0250	0.0250	0.0250	0.0250
H ₂ O	(g)	63.05	62.97	63.01	63.00	63.01
	(mole)	3.5027	3.4983	3.5003	3.4999	3.5004
CTAB	(g)	1.6402	1.6402	1.6404	1.6403	1.6403
	(mole)	0.0045	0.0045	0.0045	0.0045	0.0045
EtOH	(g)	14.9984	14.9879	15.0312	14.9847	15.0077
	(mole)	0.3256	0.3253	0.3263	0.3253	0.3258
Schiff's bases	(mole)	-	2.26×10^{-3}	1.09×10^{-3}	1.17×10^{-3}	1.67×10^{-3}

From Table B the amount of organic matters in each modified mesoporous silica could be calculated as follows.

1. Non-doped mesoporous silica

$$\text{TEOS } 0.025 \text{ mole provided SiO}_2 \text{ } 0.025 \text{ mole} = 0.025 \times 60.0843 \text{ g} = 1.5021 \text{ g}$$

$$\text{CTAB } 1.6402 \text{ g provided CTA}^+ = (1.6402/364.46) \times 284.56 \text{ g} = 1.2806 \text{ g}$$

$$\text{Organic matters in non-doped mesoporous silica} = 1.2806 \text{ g}$$

$$\text{Mass of non-doped mesoporous silica} = 1.5021 + 1.2806 \text{ g} = 2.7827 \text{ g}$$

Therefore, the percentage of organic matters in non-doped mesoporous silica

$$= (1.2806/2.7827) \times 100 \% = 46.02 \%$$

2. Salen doped mesoporous silica

$$\text{TEOS } 0.025 \text{ mole provided SiO}_2 \text{ } 0.025 \text{ mole} = 0.025 \times 60.0843 \text{ g} = 1.5021 \text{ g}$$

$$\text{CTAB } 1.6402 \text{ g provided CTA}^+ = (1.6402/364.46) \times 284.56 \text{ g} = 1.2806 \text{ g}$$

$$\text{Amount of salen in silica} = 2.26 \times 10^{-3} \times 268.18 \text{ g} = 0.6061 \text{ g}$$

Organic matters in salen doped mesoporous silica

$$= 1.2806 + 0.6061 \text{ g} = 1.8867 \text{ g}$$

Mass of salen doped mesoporous silica

$$= 1.5021 + 1.2806 + 0.6061 \text{ g} = 3.3888 \text{ g}$$

Therefore, the percentage of organic matters in salen doped mesoporous silica

$$= (1.8867/3.3888) \times 100 \text{ g} = 55.68 \%$$

3. Saltn doped mesoporous silica

$$\text{TEOS } 0.025 \text{ mole provided SiO}_2 \text{ } 0.025 \text{ mole} = 0.025 \times 60.0843 \text{ g} = 1.5021 \text{ g}$$

$$\text{CTAB } 1.6404 \text{ g provided CTA}^+ = (1.6404/364.46) \times 284.56 \text{ g} = 1.2808 \text{ g}$$

$$\text{Amount of saltn in silica} = 1.09 \times 10^{-3} \times 282.19 \text{ g} = 0.3076 \text{ g}$$

Organic matters in saltn doped mesoporous silica

$$= 1.2808 + 0.3076 \text{ g} = 1.5884 \text{ g}$$

Mass of saltn doped mesoporous silica

$$= 1.5021 + 1.2808 + 0.3076 \text{ g} = 3.0905 \text{ g}$$

Therefore, the percentage of organic matters in saltn doped mesoporous silica

$$= (1.5884/3.0905) \times 100 \text{ g} = 51.40 \%$$

4. Salophen doped mesoporous silica

$$\text{TEOS } 0.025 \text{ mole provided SiO}_2 \text{ } 0.025 \text{ mole} = 0.025 \times 60.0843 \text{ g} = 1.5021 \text{ g}$$

$$\text{CTAB } 1.6403 \text{ g provided CTA}^+ = (1.6403/364.46) \times 284.56 \text{ g} = 1.2807 \text{ g}$$

$$\text{Amount of salophen in silica} = 1.17 \times 10^{-3} \times 316.22 \text{ g} = 0.3700 \text{ g}$$

Organic matters in salophen doped mesoporous silica

$$= 1.2807 + 0.3700 \text{ g} = 1.6507 \text{ g}$$

Mass of salophen doped mesoporous silica

$$= 1.5021 + 1.2807 + 0.3700 \text{ g} = 3.1528 \text{ g}$$

Therefore, the percentage of organic matters in salophen doped mesoporous silica

$$= (1.6507/3.1528) \times 100 \text{ g} = 52.35 \%$$

5. Haen doped mesoporous silica

$$\text{TEOS } 0.025 \text{ mole provided SiO}_2 \text{ } 0.025 \text{ mole} = 0.025 \times 60.0843 \text{ g} = 1.5021 \text{ g}$$

$$\text{CTAB } 1.6403 \text{ g provided CTA}^+ = (1.6403/364.46) \times 284.56 \text{ g} = 1.2807 \text{ g}$$

$$\text{Amount of haen in silica} = 1.67 \times 10^{-3} \times 296.20 \text{ g} = 0.4945 \text{ g}$$

Organic matters in haen doped mesoporous silica

$$= 1.2807 + 0.4946 \text{ g} = 1.7753 \text{ g}$$

Mass of haen doped mesoporous silica

$$= 1.5021 + 1.2807 + 0.4946 \text{ g} = 3.2774 \text{ g}$$

Therefore, the percentage of organic matters in haen doped mesoporous silica

$$= (1.7753/3.2774) \times 100 \text{ g} = 54.17 \%$$



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APPENDIX C

CALCULATION OF ELEMENTAL MASS

The percentage of elemental mass of all mesoporous silica was calculated using the data in Appendix B and described below.

1. Non-doped mesoporous silica

$$\text{CTAB 1.6402 g provided C} = 19 \times 12.01 \times 4.5 \times 10^{-3} \text{ g} = 1.027 \text{ g}$$

$$\text{Mass of non-doped mesoporous silica} = 2.783 \text{ g}$$

Therefore, the percentage of C in non-doped mesoporous silica

$$= (1.027/2.783) \times 100 \% = 36.90 \%$$

$$\text{CTAB 1.6402 g provided H} = 42 \times 1.00 \times 4.5 \times 10^{-3} \text{ g} = 0.1890 \text{ g}$$

$$\text{Mass of non-doped mesoporous silica} = 2.783 \text{ g}$$

Therefore, the percentage of H in non-doped mesoporous silica

$$= (0.1890/2.783) \times 100 \% = 6.791 \%$$

$$\text{CTAB 1.6402 g provided N} = 1 \times 14.01 \times 4.5 \times 10^{-3} \text{ g} = 0.0630 \text{ g}$$

$$\text{Mass of non-doped mesoporous silica} = 2.783 \text{ g}$$

Therefore, the percentage of N in non-doped mesoporous silica

$$= (0.0630/2.783) \times 100 \% = 2.264 \%$$

2. Salen doped mesoporous silica

$$\text{CTAB 1.6402 g provided C} = 19 \times 12.01 \times 4.5 \times 10^{-3} \text{ g} = 1.027 \text{ g}$$

$$\text{Salen 0.6061 g provided C} = 16 \times 12.01 \times 2.26 \times 10^{-3} \text{ g} = 0.4343 \text{ g}$$

$$\text{Mass of C in salen doped mesoporous silica} = 1.027 + 0.4343 \text{ g} = 1.461 \text{ g}$$

$$\text{Mass of salen doped mesoporous silica} = 3.389 \text{ g}$$

Therefore, the percentage of C in salen doped mesoporous silica

$$= (1.461/3.389) \times 100 \% = 43.12 \%$$

$$\text{CTAB 1.6402 g provided H} = 42 \times 1.00 \times 4.5 \times 10^{-3} \text{ g} = 0.1890 \text{ g}$$

$$\text{Salen 0.6061 g provided H} = 16 \times 1.00 \times 2.26 \times 10^{-3} \text{ g} = 0.0362 \text{ g}$$

$$\text{Mass of H in salen doped mesoporous silica} = 0.1890 + 0.0362 \text{ g} = 0.2252 \text{ g}$$

$$\text{Mass of salen doped mesoporous silica} = 3.389 \text{ g}$$

Therefore, the percentage of H in salen doped mesoporous silica

$$= (0.2252/3.389) \times 100 \% = 6.645 \%$$

$$\text{CTAB } 1.6402 \text{ g provided N} = 1 \times 14.01 \times 4.5 \times 10^{-3} \text{ g} = 0.0630 \text{ g}$$

$$\text{Salen } 0.6061 \text{ g provided N} = 2 \times 14.01 \times 2.26 \times 10^{-3} \text{ g} = 0.0633 \text{ g}$$

$$\text{Mass of N in salen doped mesoporous silica} = 0.0630 + 0.0633 \text{ g} = 0.1263 \text{ g}$$

$$\text{Mass of salen doped mesoporous silica} = 3.389 \text{ g}$$

Therefore, the percentage of N in salen doped mesoporous silica

$$= (0.1263/3.389) \times 100 \% = 3.727 \%$$

3. Saltn doped mesoporous silica

$$\text{CTAB } 1.6404 \text{ g provided C} = 19 \times 12.01 \times 4.5 \times 10^{-3} \text{ g} = 1.027 \text{ g}$$

$$\text{Saltn } 0.3076 \text{ g provided C} = 17 \times 12.01 \times 1.09 \times 10^{-3} \text{ g} = 0.2225 \text{ g}$$

$$\text{Mass of C in saltn doped mesoporous silica} = 1.027 + 0.2225 \text{ g} = 1.250 \text{ g}$$

$$\text{Mass of saltn doped mesoporous silica} = 3.091 \text{ g}$$

Therefore, the percentage of C in saltn doped mesoporous silica

$$= (1.250/3.091) \times 100 \% = 40.44 \%$$

$$\text{CTAB } 1.6404 \text{ g provided H} = 42 \times 1.00 \times 4.5 \times 10^{-3} \text{ g} = 0.1890 \text{ g}$$

$$\text{Saltn } 0.3076 \text{ g provided H} = 18 \times 1.00 \times 1.09 \times 10^{-3} \text{ g} = 0.0196 \text{ g}$$

$$\text{Mass of H in saltn doped mesoporous silica} = 0.1890 + 0.0196 \text{ g} = 0.2086 \text{ g}$$

$$\text{Mass of saltn doped mesoporous silica} = 3.091 \text{ g}$$

Therefore, the percentage of H in saltn doped mesoporous silica

$$= (0.2086/3.091) \times 100 \% = 6.749 \%$$

$$\text{CTAB } 1.6404 \text{ g provided N} = 1 \times 14.01 \times 4.5 \times 10^{-3} \text{ g} = 0.0630 \text{ g}$$

$$\text{Saltn } 0.3076 \text{ g provided N} = 2 \times 14.01 \times 1.09 \times 10^{-3} \text{ g} = 0.0305 \text{ g}$$

$$\text{Mass of N in saltn doped mesoporous silica} = 0.0630 + 0.0305 \text{ g} = 0.0935 \text{ g}$$

$$\text{Mass of saltn doped mesoporous silica} = 3.091 \text{ g}$$

Therefore, the percentage of N in saltn doped mesoporous silica

$$= (0.0935/3.091) \times 100 \% = 3.025 \%$$

4. Salophen doped mesoporous silica

$$\text{CTAB 1.6403 g provided C} = 19 \times 12.01 \times 4.5 \times 10^{-3} \text{ g} = 1.027 \text{ g}$$

$$\text{Salophen 0.3700 g provided C} = 20 \times 12.01 \times 1.17 \times 10^{-3} \text{ g} = 0.2810 \text{ g}$$

$$\text{Mass of C in salophen doped mesoporous silica} = 1.027 + 0.2810 \text{ g} = 1.308 \text{ g}$$

$$\text{Mass of salophen doped mesoporous silica} = 3.153 \text{ g}$$

Therefore, the percentage of C in salophen doped mesoporous silica

$$= (1.308/3.153) \times 100 \% = 41.48 \%$$

$$\text{CTAB 1.6403 g provided H} = 42 \times 1.00 \times 4.5 \times 10^{-3} \text{ g} = 0.1890 \text{ g}$$

$$\text{Salophen 0.3700 g provided H} = 16 \times 1.00 \times 1.17 \times 10^{-3} \text{ g} = 0.0187 \text{ g}$$

$$\text{Mass of H in salophen doped mesoporous silica} = 0.1890 + 0.0187 \text{ g} = 0.2077 \text{ g}$$

$$\text{Mass of salophen doped mesoporous silica} = 3.153 \text{ g}$$

Therefore, the percentage of H in salophen doped mesoporous silica

$$= (0.2077/3.153) \times 100 \% = 6.587 \%$$

$$\text{CTAB 1.6403 g provided N} = 1 \times 14.01 \times 4.5 \times 10^{-3} \text{ g} = 0.0630 \text{ g}$$

$$\text{Salophen 0.3700 g provided N} = 2 \times 14.01 \times 1.17 \times 10^{-3} \text{ g} = 0.0328 \text{ g}$$

$$\text{Mass of N in salophen doped mesoporous silica} = 0.0630 + 0.0328 \text{ g} = 0.0958 \text{ g}$$

$$\text{Mass of salophen doped mesoporous silica} = 3.153 \text{ g}$$

Therefore, the percentage of N in salophen doped mesoporous silica

$$= (0.0958/3.153) \times 100 \% = 3.038 \%$$

5. Haen doped mesoporous silica

$$\text{CTAB 1.6403 g provided C} = 19 \times 12.01 \times 4.5 \times 10^{-3} \text{ g} = 1.027 \text{ g}$$

$$\text{Haen 0.4945 g provided C} = 18 \times 12.01 \times 1.67 \times 10^{-3} \text{ g} = 0.3610 \text{ g}$$

$$\text{Mass of C in haen doped mesoporous silica} = 1.027 + 0.3610 \text{ g} = 1.388 \text{ g}$$

$$\text{Mass of haen doped mesoporous silica} = 3.277 \text{ g}$$

Therefore, the percentage of C in haen doped mesoporous silica

$$= (1.388/3.277) \times 100 \% = 42.36 \%$$

$$\text{CTAB 1.6403 g provided H} = 42 \times 1.00 \times 4.5 \times 10^{-3} \text{ g} = 0.1890 \text{ g}$$

$$\text{Haen 0.4945 g provided H} = 20 \times 1.00 \times 1.67 \times 10^{-3} \text{ g} = 0.0334 \text{ g}$$

$$\text{Mass of H in haen doped mesoporous silica} = 0.1890 + 0.0334 \text{ g} = 0.2224 \text{ g}$$

$$\text{Mass of haen doped mesoporous silica} = 3.277 \text{ g}$$

Therefore, the percentage of H in haen doped mesoporous silica

$$= (0.2224/3.277) \times 100 \% = 6.787 \%$$

$$\text{CTAB } 1.6403 \text{ g provided N} = 1 \times 14.01 \times 4.5 \times 10^{-3} \text{ g} = 0.0630 \text{ g}$$

$$\text{Haen } 0.4945 \text{ g provided N} = 2 \times 14.01 \times 1.67 \times 10^{-3} \text{ g} = 0.0468 \text{ g}$$

$$\text{Mass of N in haen doped mesoporous silica} = 0.0630 + 0.0468 \text{ g} = 0.1098 \text{ g}$$

$$\text{Mass of haen doped mesoporous silica} = 3.277 \text{ g}$$

Therefore, the percentage of N in haen doped mesoporous silica

$$= (0.1098/3.277) \times 100 \% = 3.351 \%$$



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VITAE

Pornmanee Khamloet was born on July 9th, 1980 in Suphanburi, Thailand. She received a B. Sc. degree in Chemistry from Chulalongkorn University in 2001. Since 2002, she has been a graduate student studying in Analytical Chemistry at Department of Chemistry, Faculty of Science, Chulalongkorn University, Bangkok, Thailand. During the course study, she obtained financial support from the Graduate School Chulalongkorn University. She had completed her study leading to a Master of Science Degree in Analytical Chemistry in 2005.



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