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APPENDICES

APPENDIX A

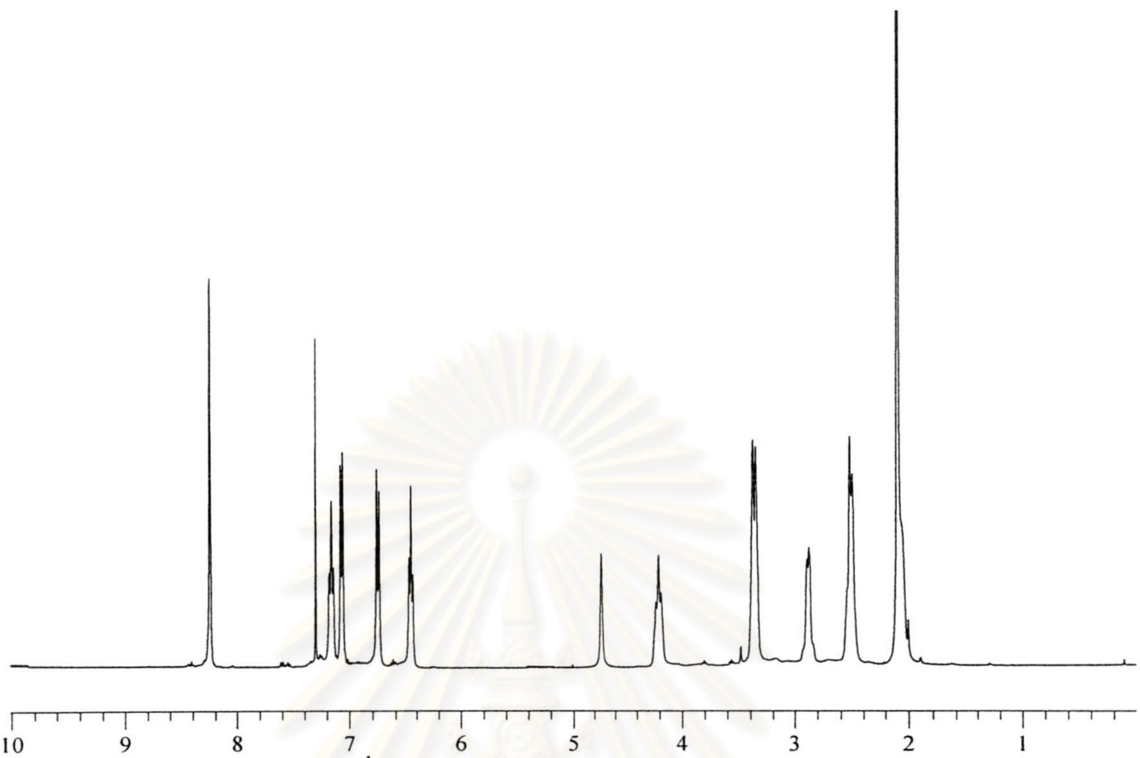


Figure A.1 ^{1}H-NMR spectrum of $Zn(Sal)_2$trien in $CDCl_3$

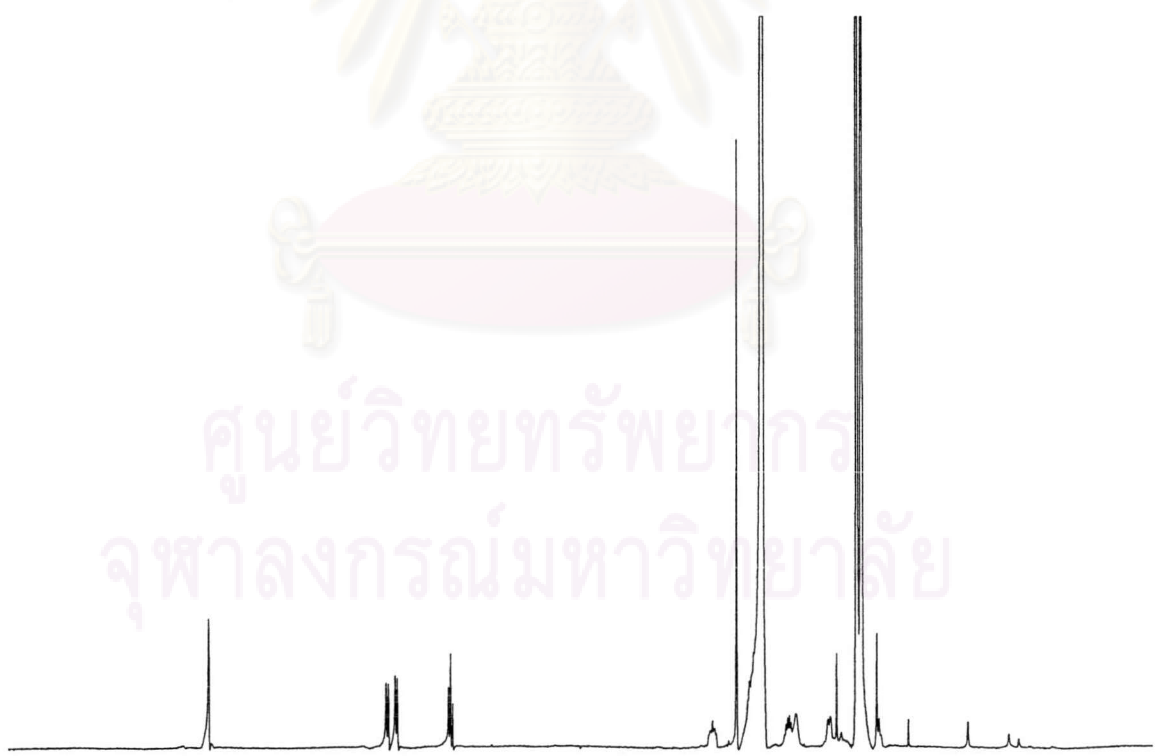


Figure A.2 ^{1}H-NMR spectrum of $Zn(MeOSal)_2$trien in DMSO-d_6

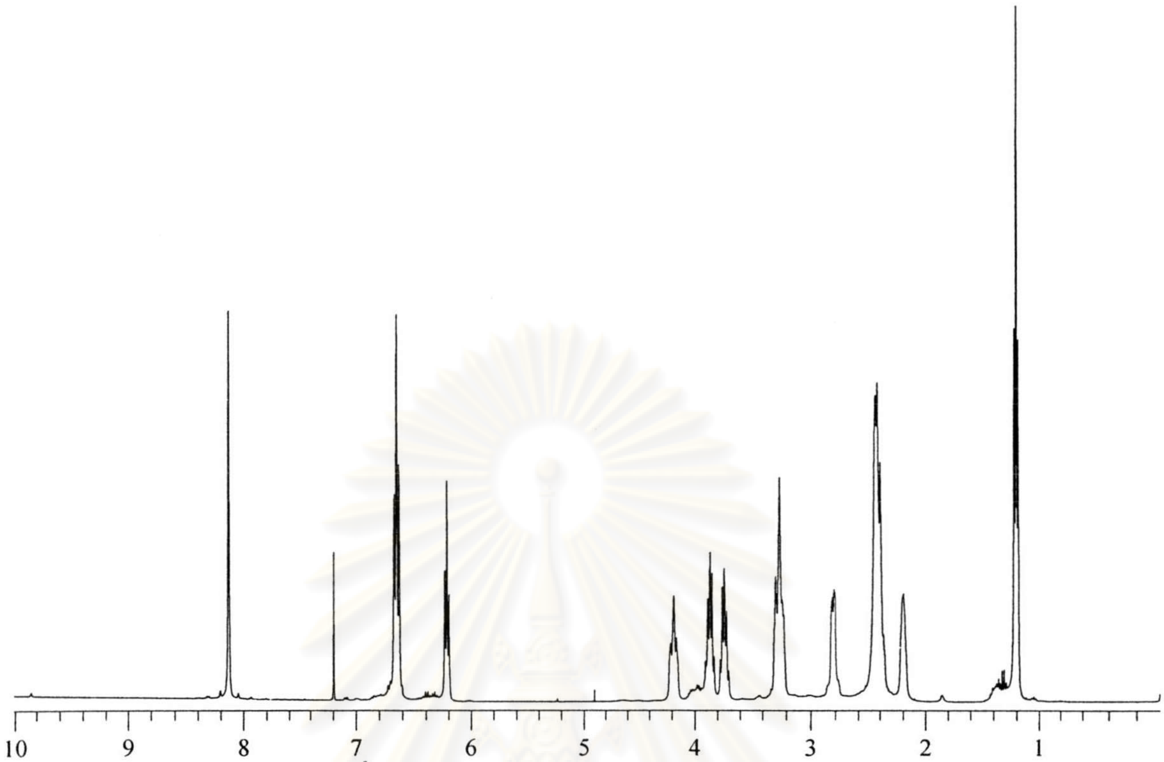


Figure A.3 ^{1}H-NMR spectrum of Zn(EtOSal)$_2$trien in $CDCl_3$

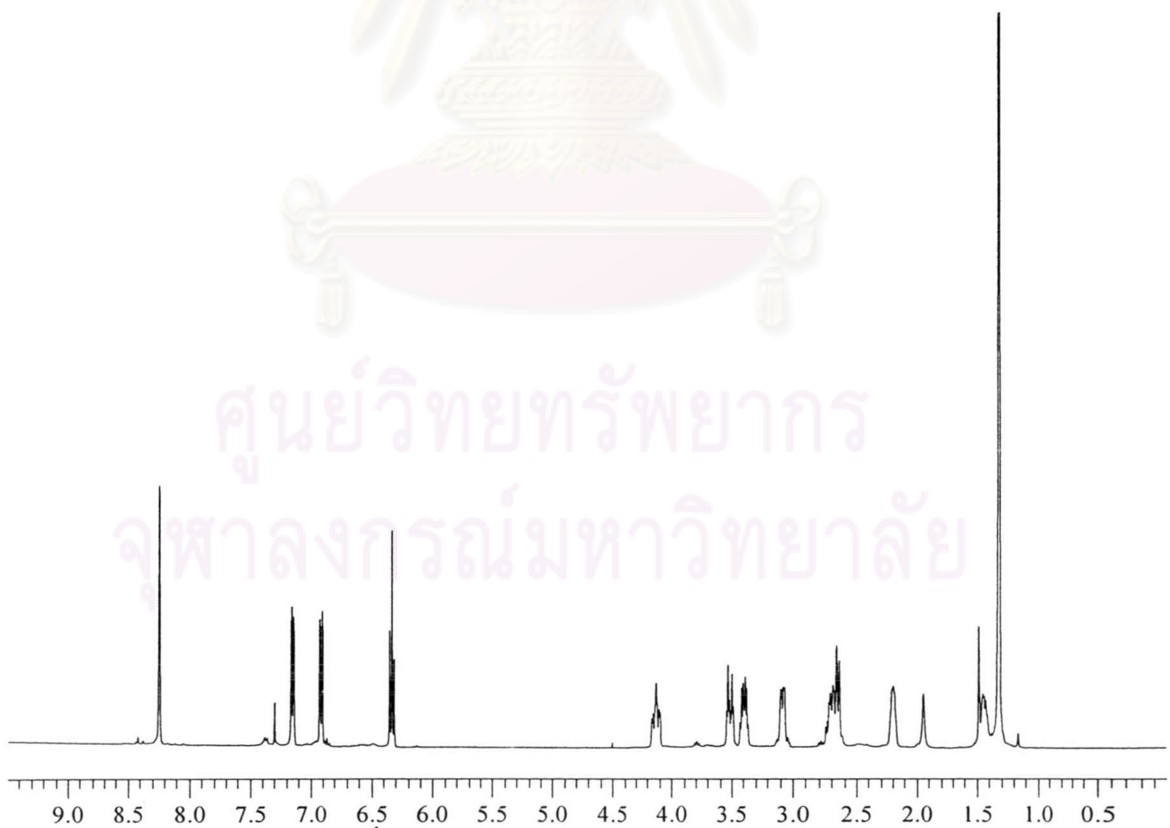


Figure A.4 ^{1}H-NMR spectrum of Zn(BuSal)$_2$trien in $CDCl_3$

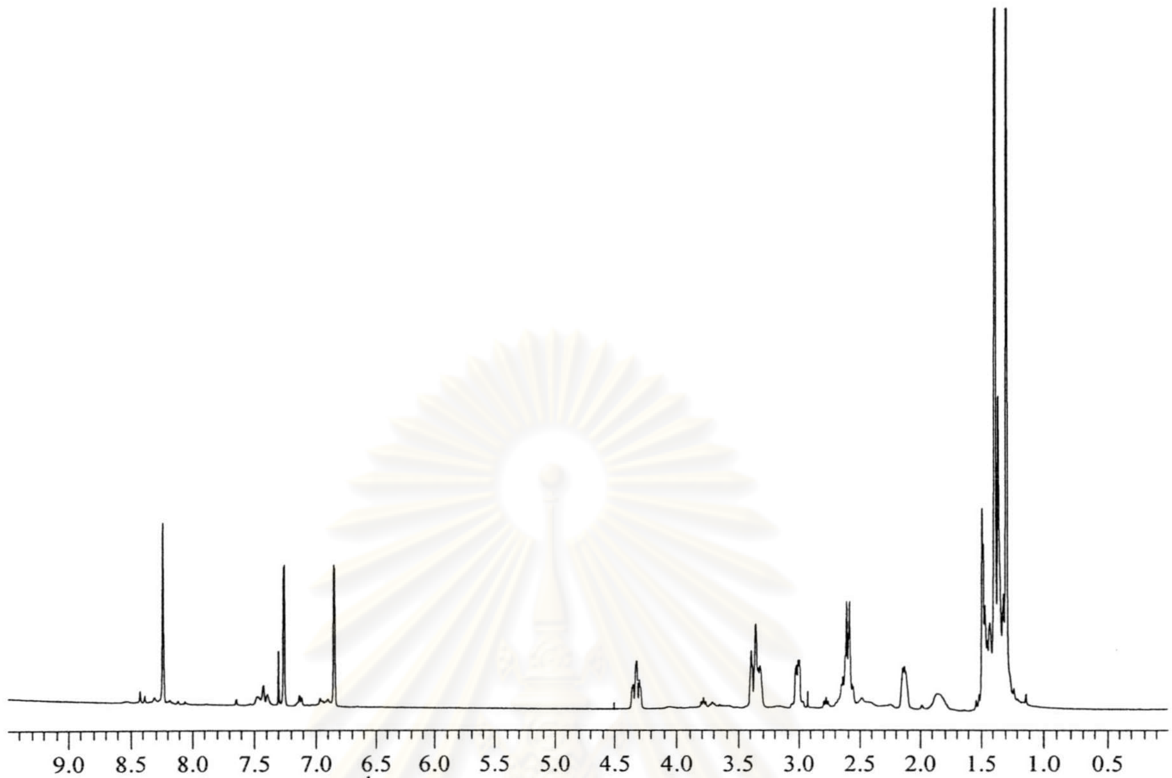


Figure A.5 ^{1}H-NMR spectrum of $Zn(diBuSal)_2trien$ in $CDCl_3$

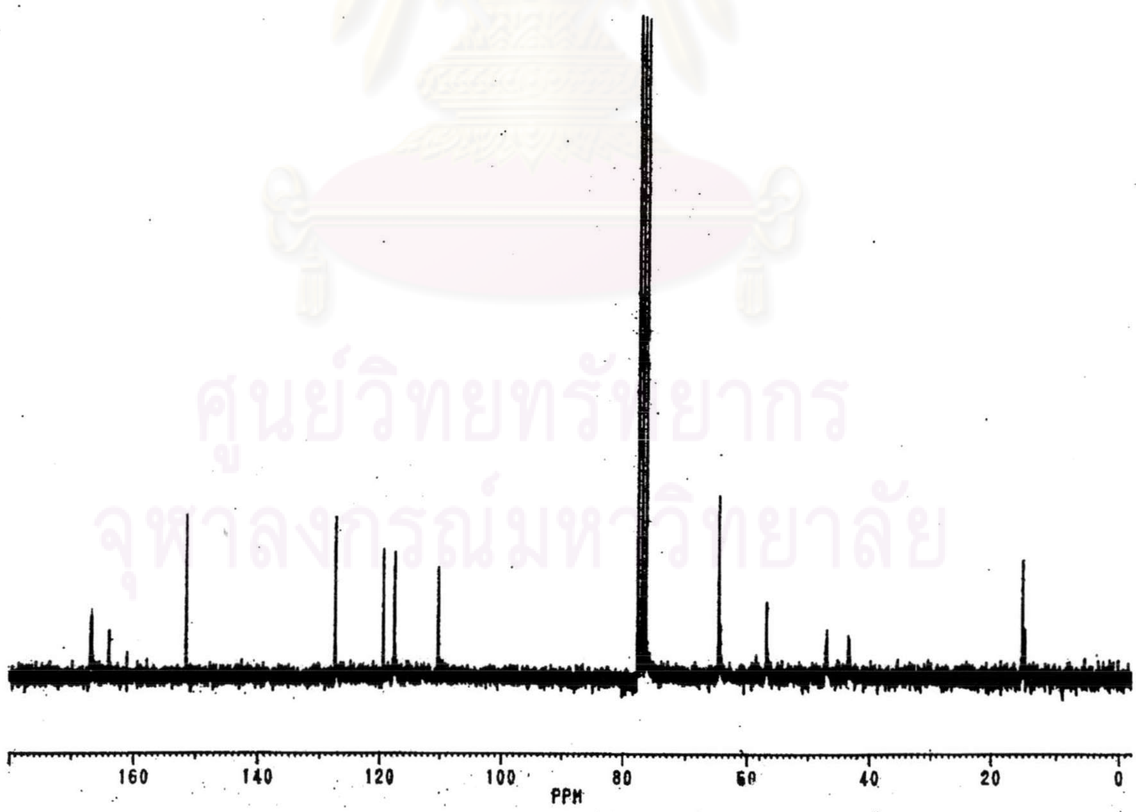


Figure A.6 ^{13}C-NMR spectrum of $Zn(EtOSal)_2trien$ in $CDCl_3$

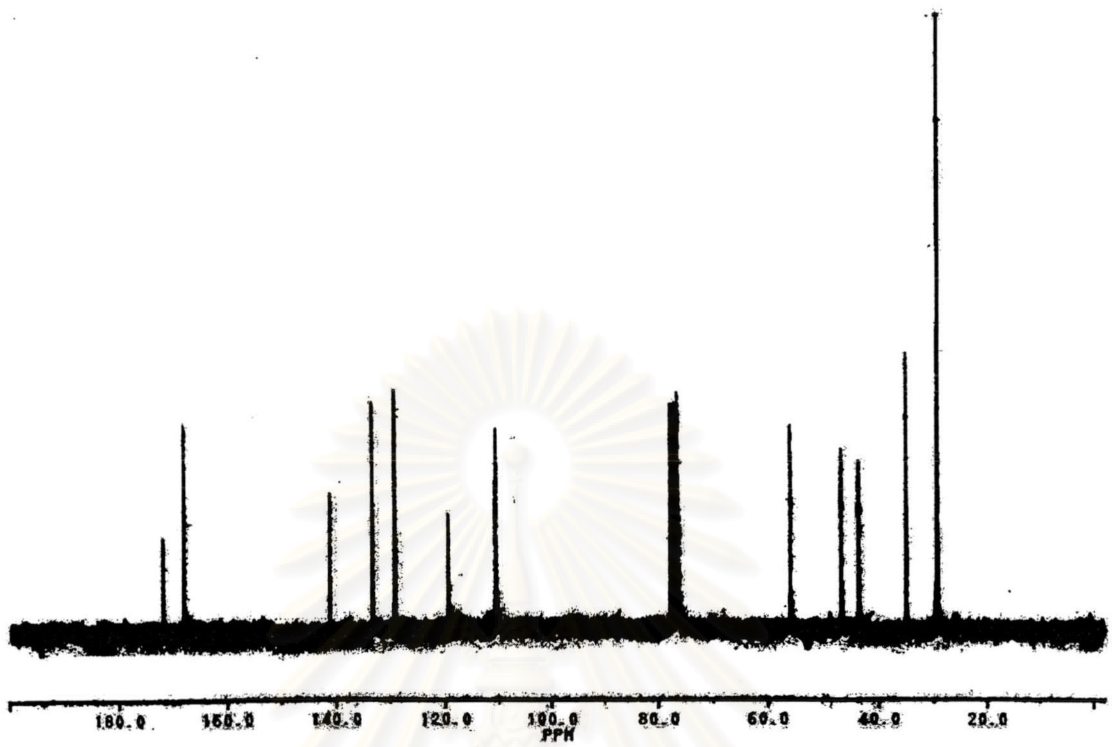


Figure A.7 ^{13}C-NMR spectrum of Zn(BuSal)$_2$trien in $CDCl_3$

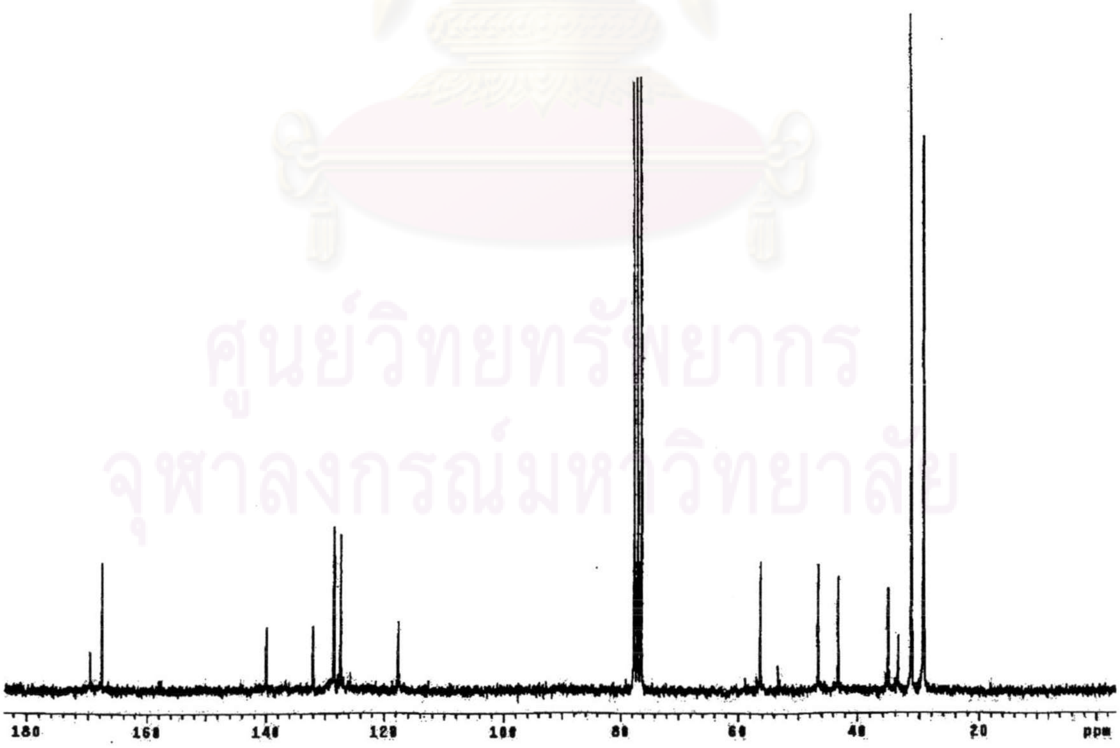


Figure A.8 ^{13}C-NMR spectrum of Zn(diBuSal)$_2$trien in $CDCl_3$

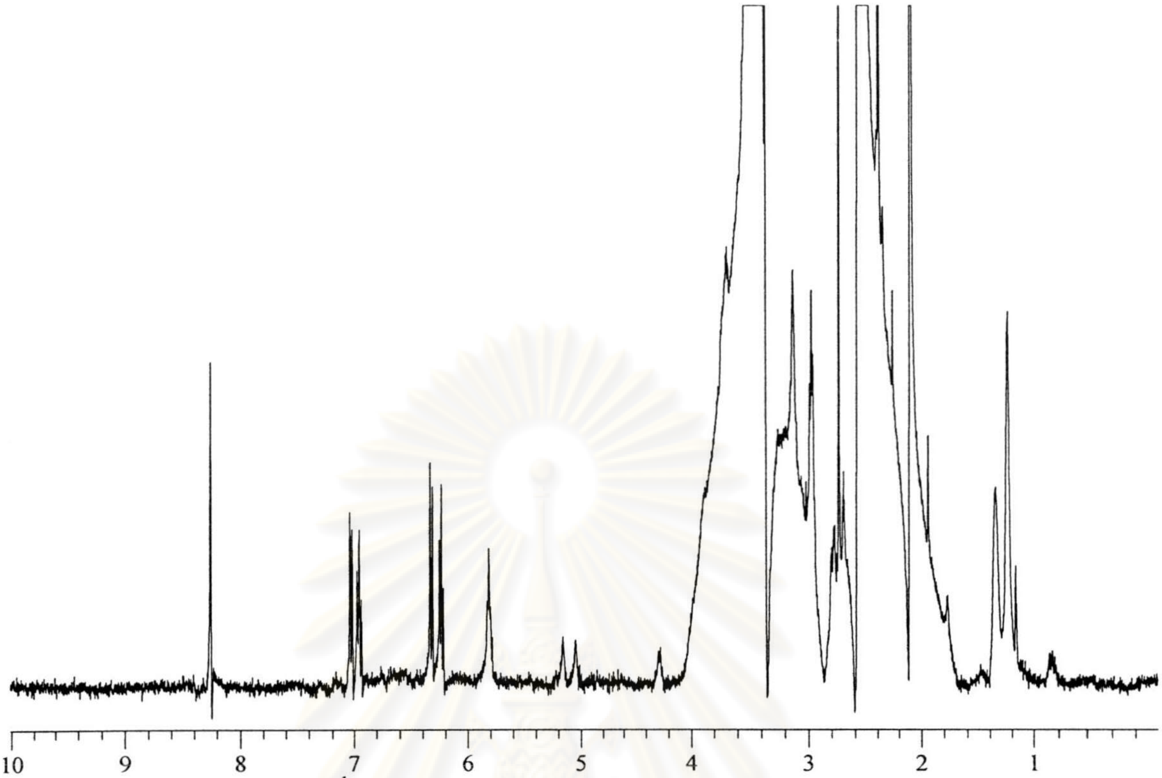


Figure A.9 ^{1}H-NMR spectrum of $ZnSal_2$trien-HDI in DMSO-d_6

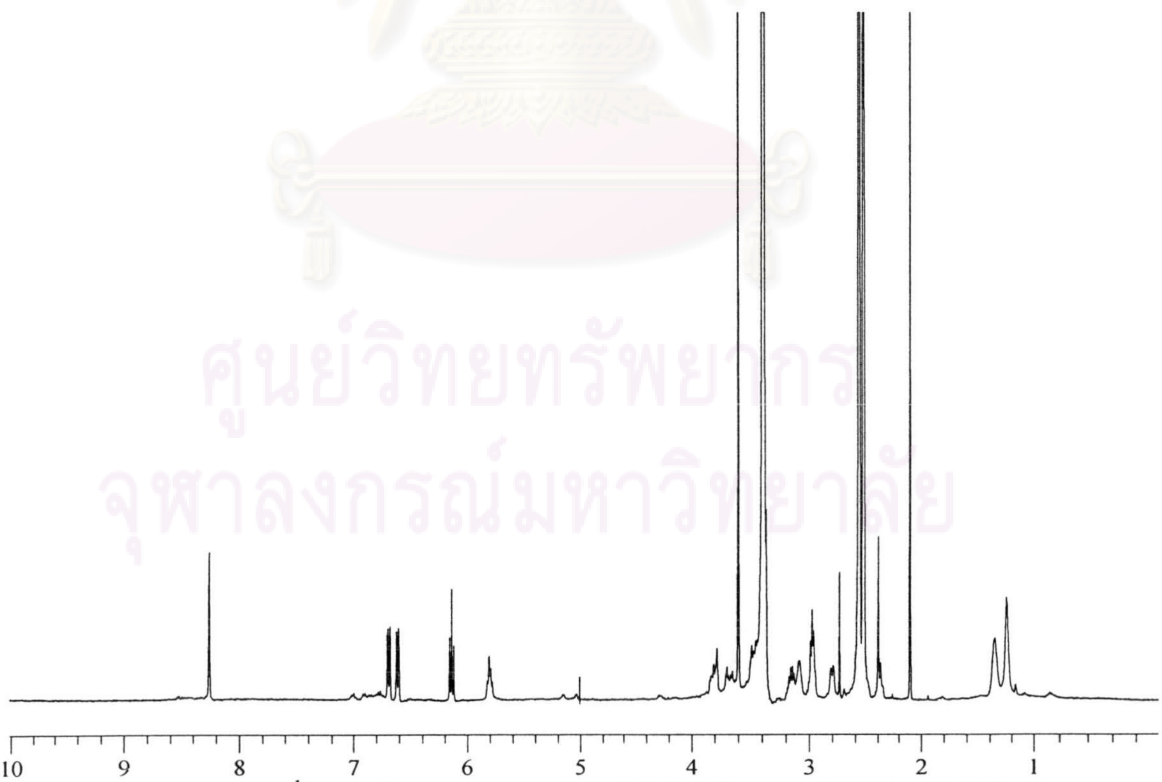


Figure A.10 ^{1}H-NMR spectrum of $Zn(MeOSal)_2$trien-HDI in DMSO-d_6

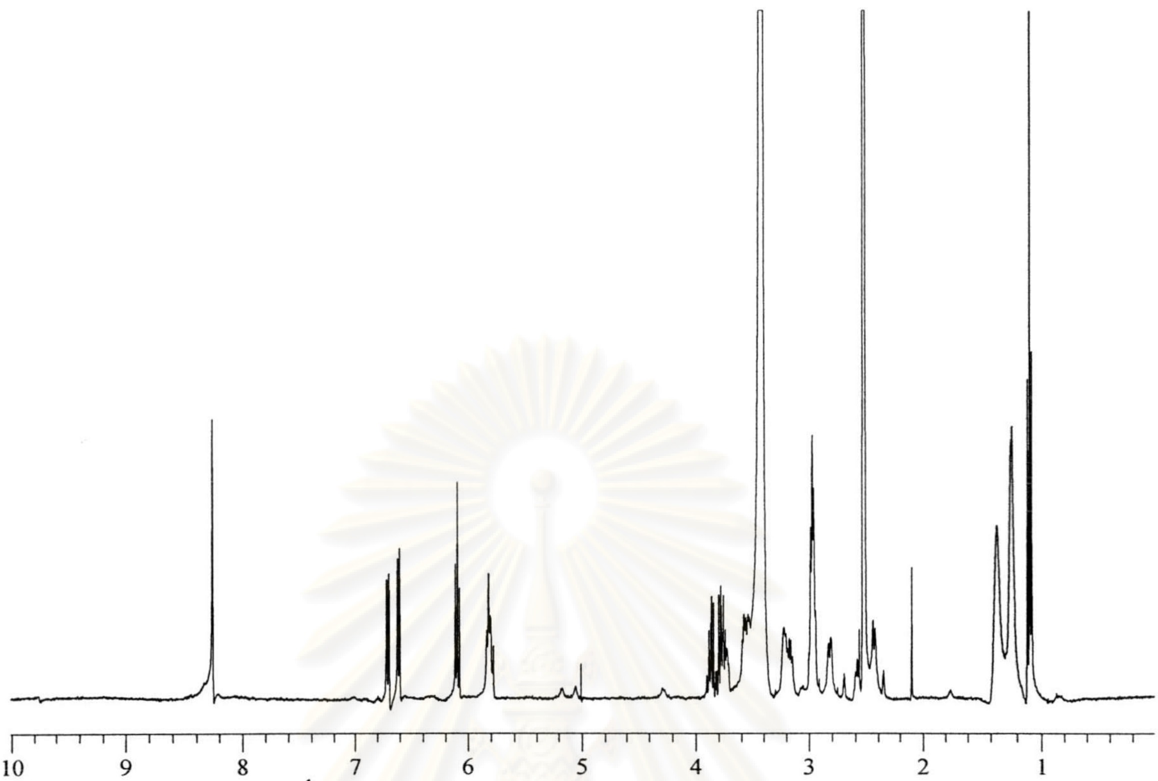


Figure A.11 ^{1}H-NMR spectrum of Zn(EtOSal)$_2$trien-HDI in DMSO-d_6

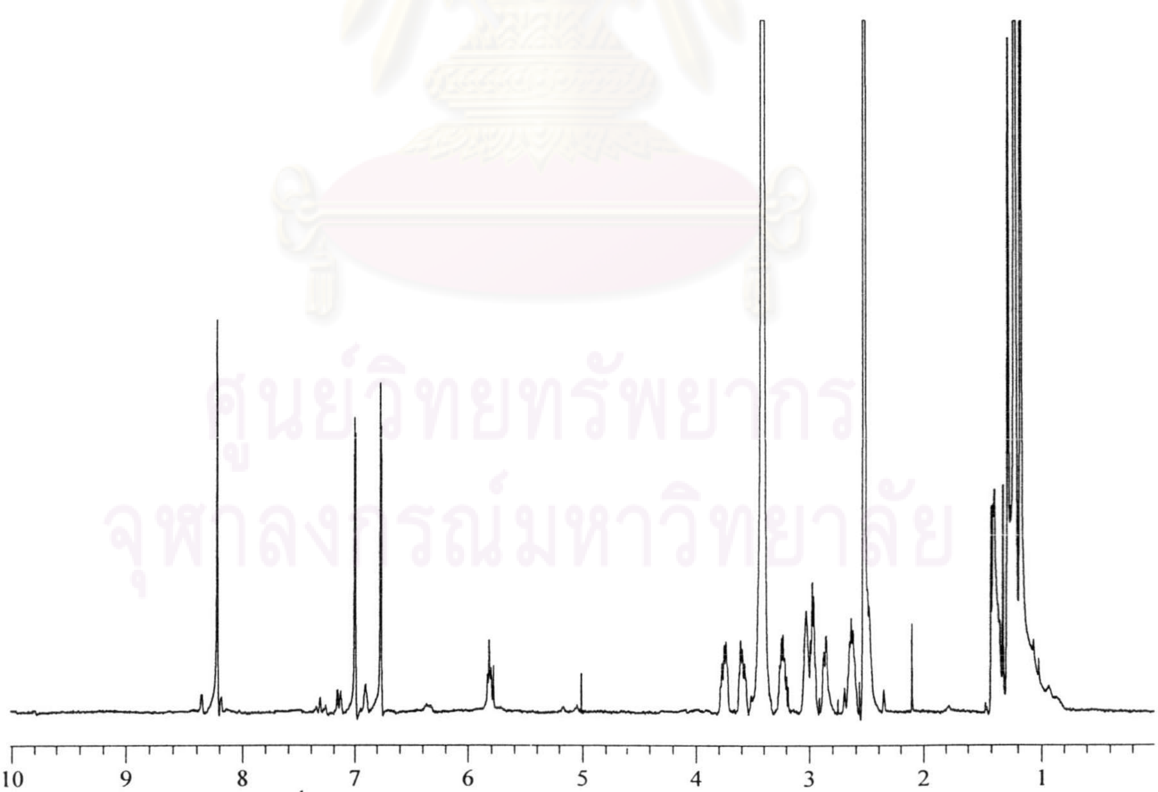


Figure A.12 ^{1}H-NMR spectrum of Zn(diBuSal)$_2$trien-HDI in DMSO-d_6

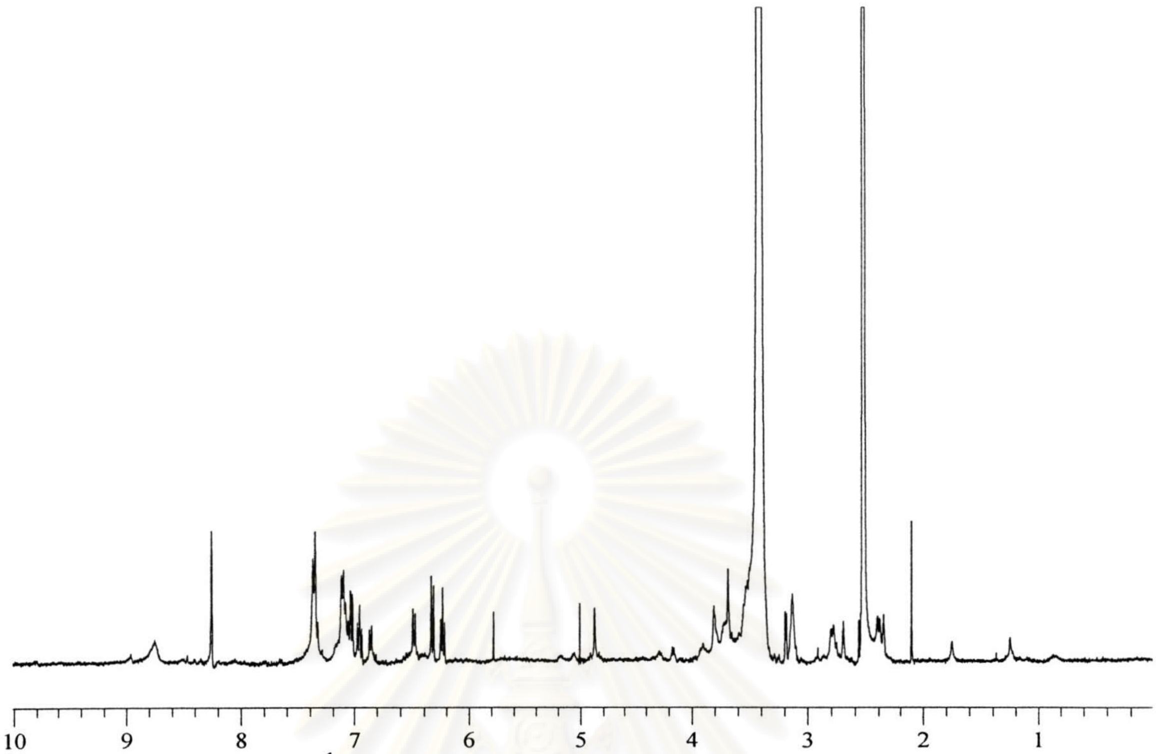


Figure A.13 ^{1}H-NMR spectrum of Zn(Sal)$_2$trien-MDI in DMSO-d_6

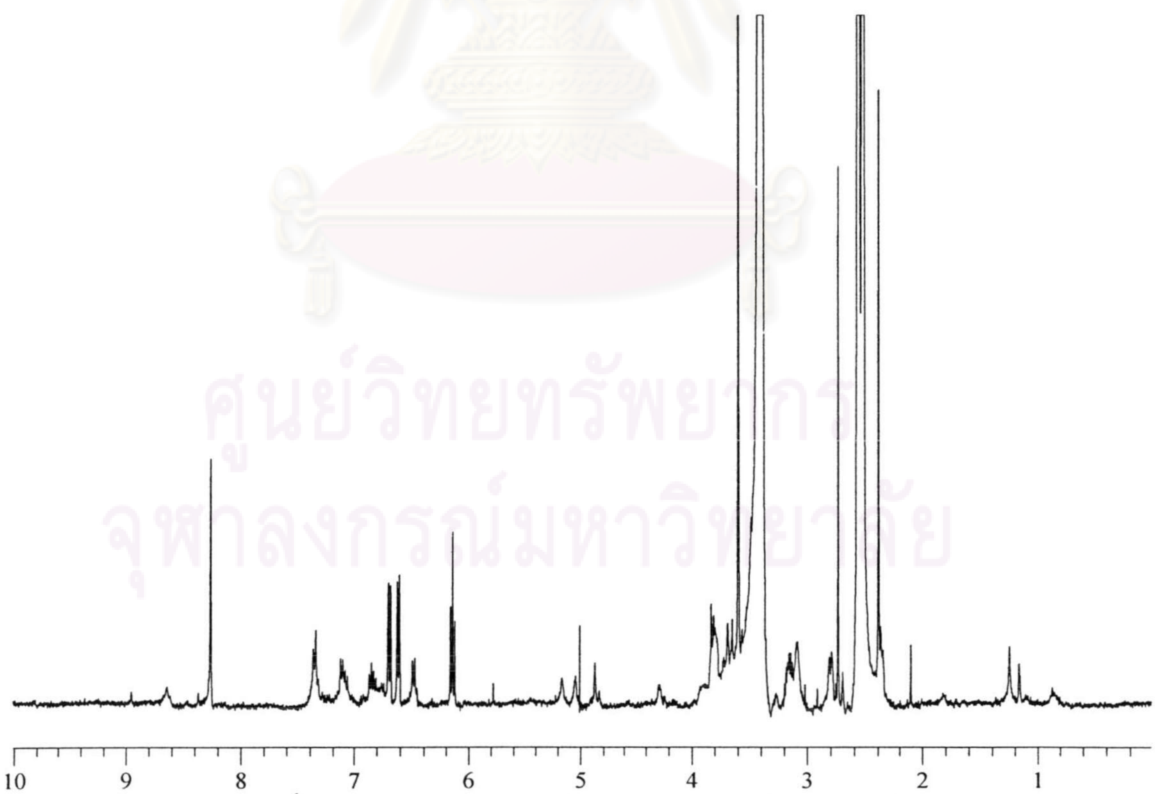


Figure A.14 ^{1}H-NMR spectrum of Zn(MeOSal)$_2$trien-MDI in DMSO-d_6

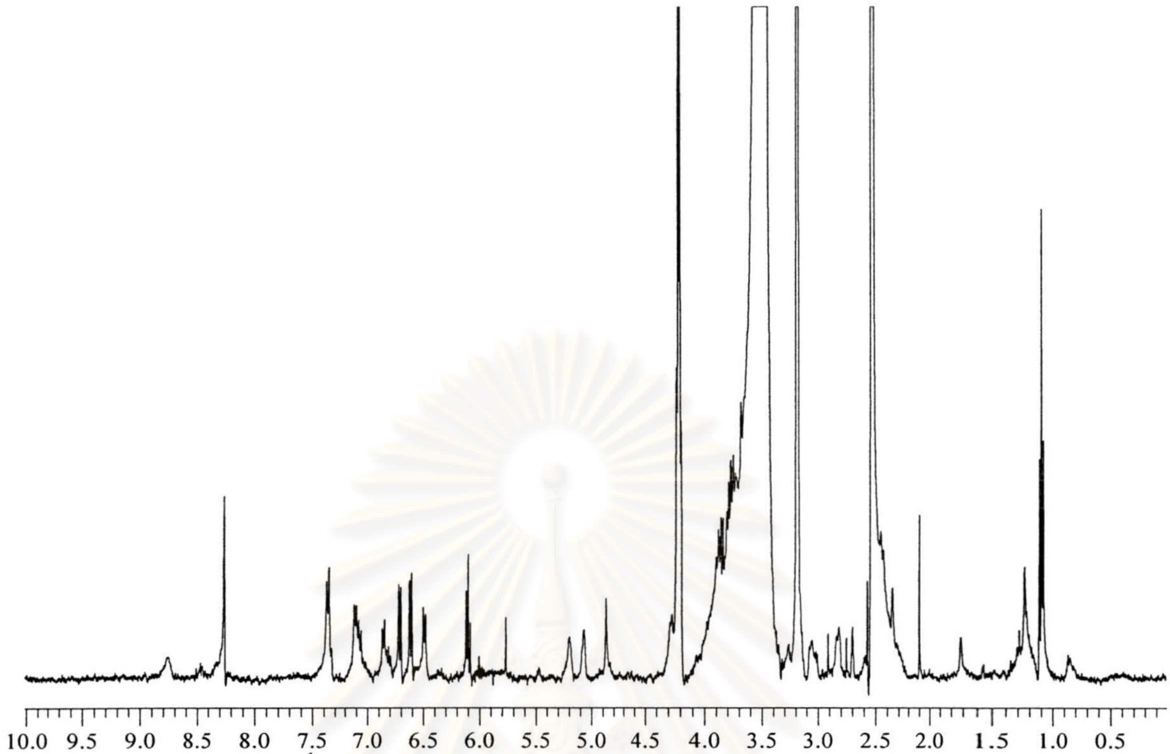


Figure A.15 ^{1}H-NMR spectrum of Zn(EtOSal)$_2$trien-MDI in DMSO-d_6

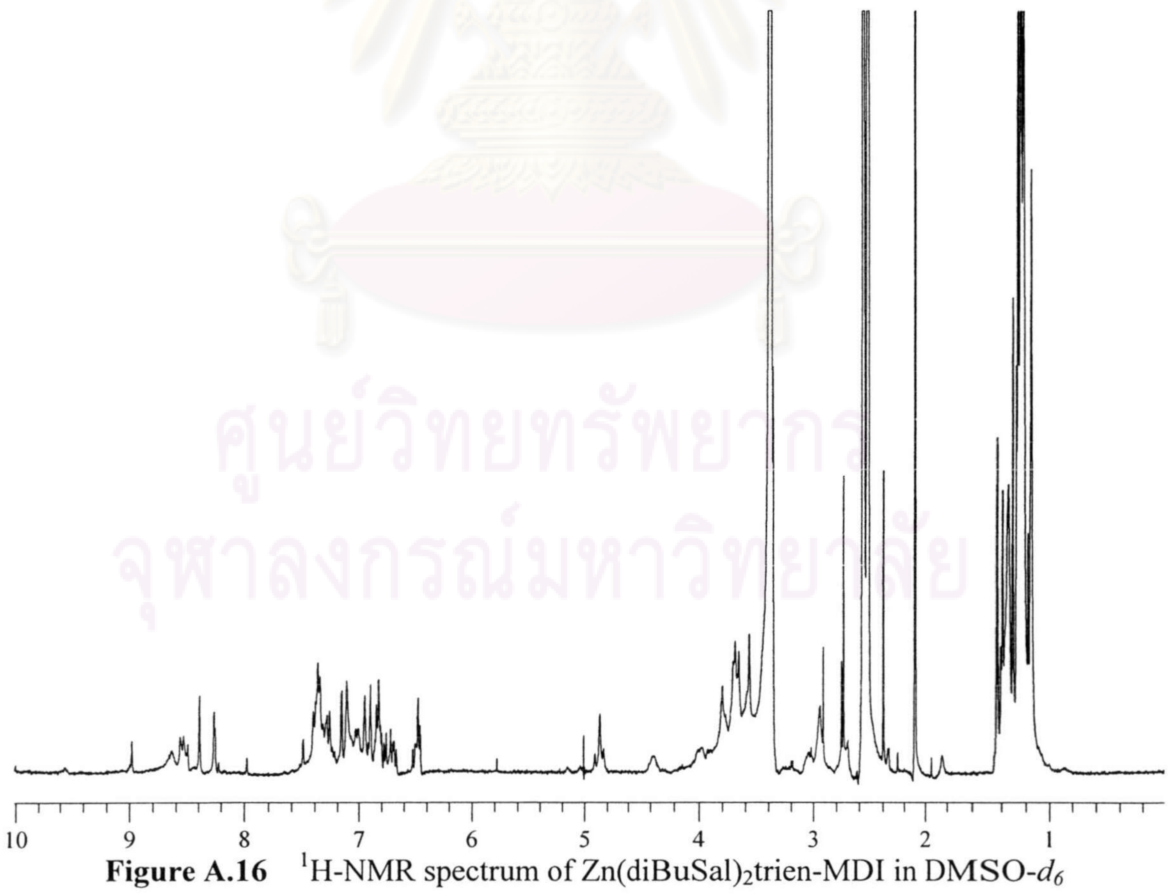


Figure A.16 ^{1}H-NMR spectrum of Zn(diBuSal)$_2$trien-MDI in DMSO-d_6

APPENDIX B

TableB.1 Crystal data and structure refinement for $Zn(MeOSal)_2trien$

	$Zn(MeOSal)_2trien$
Empirical formular	$C_{22}H_{28}N_4O_4Zn$
Color/shape	Pale yellow/prism
Formula weight	477.85
Space group	*Cc*
Temperature	293(2) K
Cell constants	a = 18.8355(3) A, b = 7.551(1), c = 15.387(1), β = 91.428(1)
Cell volume ($Å^3$)	2170.02(5)
Formula unit/unit cell	4
F(000)	1000
D_{calc}(Mg m^{-3})	1.463
μ_{calc}(mm^{-1})	1.168
Diffractometer/scan	Bruker SMART CCD
Radiation used, graphite monochromator	Mo Kα (λ=0.71073 Å)
Maximum crystal dimension (mm)	0.075x0.025x0.375
Reflections measured	7,755
Index range	$-23 \leq h \leq 25$, $-8 \leq k \leq 10$, $-21 \leq h \leq 21$
Data/parameters	4,910/380
GOF	1.018
R_1/wR_2 for observed reflection [$I>2\sigma(I)$]	0.0274/0.0641
$R1/Wr_2$ for all data	0.0370/0.0674
Largest resolution peak/hold (e $Å^3$)	0.220, -0.416

Table B.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for Zn(MeOSal)$_2$trien. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

Zn(MeOSal)$_2$trien	**x**	**y**	**z**	***B*(Å)**
Zn(1)	3470(1)	3175(1)	1414(1)	39(1)
N(1)	3185(1)	4266(1)	750(1)	50(1)
N(2)	3574(1)	2134(1)	2178(1)	46(1)
N(3)	4786(2)	3544(2)	1247(1)	58(1)
N(4)	3744(1)	2175(2)	639(1)	55(1)
O(1)	2120(1)	2998(1)	1154(1)	43(1)
O(2)	3513(1)	3836(1)	2450(1)	44(1)
C(1)	1587(1)	3603(2)	1221(1)	44(1)
C(2)	817(2)	3449(2)	1500(2)	68(1)
C(3)	217(2)	4053(3)	1527(3)	93(1)
C(4)	351(3)	4833(3)	1281(3)	108(2)
C(5)	1089(3)	5009(2)	1035(2)	83(1)
C(6)	1731(2)	4416(2)	1006(2)	50(1)
C(7)	2508(2)	4687(2)	764(2)	55(1)
C(8)	3934(2)	4656(2)	516(2)	71(1)
C(9)	4761(2)	4421(2)	1099(2)	74(1)
C(10)	4971(2)	3057(3)	582(2)	74(1)
C(11)	4685(2)	2190(2)	639(2)	70(1)
C(12)	3451(2)	1410(2)	939(2)	63(1)
C(13)	3764(2)	1357(5)	1831(2)	63(1)
C(14)	3180(2)	2105(2)	2750(2)	47(1)
C(15)	2893(2)	2796(2)	3142(1)	44(1)
C(16)	2420(2)	2624(2)	3726(2)	63(1)
C(17)	2090(2)	3224(2)	4123(2)	73(1)

Zn(MeOSal)$_2$trien	**x**	**y**	**z**	***B*(Å)**
C(18)	2225(2)	4024(2)	3941(2)	68(1)
C(19)	2693(2)	4226(2)	3386(2)	54(1)
C(20)	3050(1)	3623(1)	2961(1)	41(1)
O(1W)	1341(2)	1458(2)	1333(2)	82(1)
O(2W)	3534(2)	5583(2)	2261(2)	99(1)
O(3W)	449(2)	1589(2)	3533(3)	152(1)

Table B.3 Bond lengths [Å] for $Zn(MeOSal)_2trien$

$Zn(MeOSal)_2trien$	**Å**	**$Zn(MeOSal)_2trien$**	**Å**
Zn(1)-O(1)	2.063(4)	C(15)-C(20)	1.367(9)
Zn(1)-O(2)	2.055(4)	C(15)-C(16)	1.473(7)
Zn(1)-N(1)	2.147(5)	C(16)-C(17)	1.299(12)
Zn(1)-N(2)	2.146(5)	C(17)-C(18)	1.402(13)
Zn(1)-N(3)	2.295(5)	C(18)-C(19)	1.397(9)
Zn(1)-N(4)	2.228(6)	C(19)-O(3)	1.427(8)
C(1)-O(1)	1.308(6)	C(19)-C(20)	1.439(9)
C(1)-C(2)	1.455(9)	C(20)-O(2)	1.291(7)
C(1)-C(6)	1.492(8)	C(21)-O(4)	1.417(9)
C(2)-O(4)	1.340(8)	C(22)-O(3)	1.427(8)
C(2)-C(3)	1.388(8)		
C(3)-C(4)	1.420(12)		
C(4)-C(5)	1.419(11)		
C(5)-C(6)	1.371(9)		
C(6)-C(7)	1.441(8)		
C(7)-N(1)	1.290(7)		
C(8)-C(9)	1.501(10)		
C(8)-N(1)	1.503(7)		
C(9)-N(3)	1.485(9)		
C(10)-C(11)	1.511(4)		
C(10)-N(3)	1.519(9)		
C(11)-N(4)	1.458(9)		
C(12)-N(4)	1.464(10)		
C(12)-C(13)	1.566(9)		
C(13)-N(2)	1.433(8)		
C(14)-N(2)	1.288(7)		
C(14)-C(15)	1.458(9)		

Table B.4 Bond angle (°) for $Zn(MeOSal)_2trien$

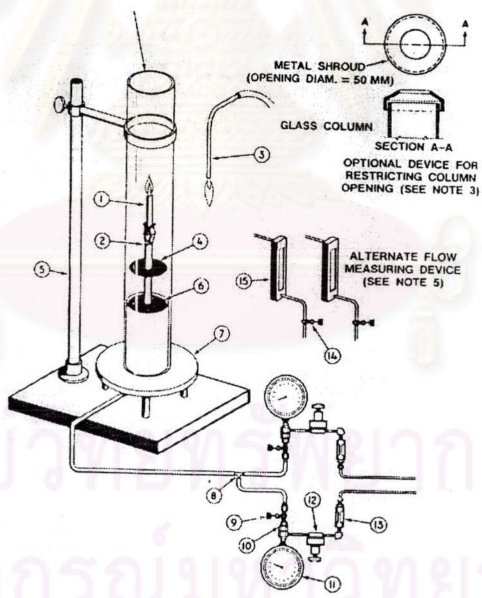
$Zn(MeOSal)_2trien$	**(°)**	**$Zn(MeOSal)_2trien$**	**(°)**
C(7)-C(6)-C(1)	122.8(5)	C(9)-N(3)-C(10)	116.7(5)
N(1)-C(7)-C(6)	126.8(5)	C(9)-N(3)-Zn(1)	104.3(4)
C(9)-C(8)-N(1)	111.1(5)	C(10)-N(3)-Zn(1)	104.4(45)
N(3)-C(9)-C(8)	110.7(6)	C(11)-N(4)-C(12)	115.4(5)
C(11)-C(10)-N(3)	110.0(6)	C(11)-N(4)-Zn(1)	111.7(5)
N(4)-C(11)-C(10)	105.6(6)	C(12)-N(4)-Zn(1)	106.6(4)
N(4)-C(12)-C(13)	108.7(5)	C(1)-O(1)-Zn(1)	129.7(4)
N(2)-C(13)-C(12)	108.8(5)	C(20)-O(2)-Zn(1)	128.4(4)
N(2)-C(14)-C(15)	127.1(6)	C(22)-O(3)-C(19)	117.2(6)
C(20)-C(15)-C(14)	123.2(5)	C(2)-O(4)-C(21)	116.4(6)
C(20)-C(15)-C(16)	121.8(6)	O(2)-Zn(1)-O(1)	108.21(5)
C(14)-C(15)-C(16)	114.9(6)	O(2)-Zn(1)-N(1)	91.11(18)
C(17)-C(16)-C(15)	119.8(7)	O(1)-Zn(1)-N(1)	86.21(18)
C(16)-C(17)-C(18)	121.5(6)	O(2)-Zn(1)-N(2)	86.04(17)
C(19)-C(18)-C(17)	118.9(7)	O(1)-Zn(1)-N(2)	90.92(17)
C(18)-C(19)-O(3)	123.0(7)	N(1)-Zn(1)-N(2)	175.12(6)
C(18)-C(19)-C(20)	122.1(7)	O(2)-Zn(1)-N(4)	154.33(17)
O(3)-C(19)-C(20)	114.8(5)	O(1)-Zn(1)-N(4)	90.9(2)
O(2)-C(20)-C(15)	126.5(5)	N(1)-Zn(1)-N(4)	107.52(19)
O(2)-C(20)-C(19)	118.0(6)	N(2)-Zn(1)-N(4)	76.43(18)
C(15)-C(20)-C(19)	115.4(5)	O(2)-Zn(1)-N(3)	89.54(18)
C(7)-N(1)-C(8)	119.2(5)	O(1)-Zn(1)-N(3)	156.63(16)
C(7)-N(1)-Zn(1)	126.1(4)	N(1)-Zn(1)-N(3)	78.17(19)
C(8)-N(1)-Zn(1)	114.1(4)	N(2)-Zn(1)-N(3)	105.74(18)
C(14)-N(2)-C(13)	118.1(6)	N(4)-Zn(1)-N(3)	77.59(8)
C(14)-N(2)-Zn(1)	123.5(4)		
C(13)-N(2)-Zn(1)	117.4(4)		

APPENDIX C

C-1 Limiting Oxygen Index (LOI)

ASTM D2863-70: the minimum concentration of oxygen, expressed as volume percent, in a mixture of oxygen and nitrogen that will just support flaming combustion of a material initially at room temperature. The LOI method used for self-supporting samples has been modified as described below to accommodate the viscous of the powdery samples. The measurement was carried out as follows. About 1 g. of the polymer sample was placed in a glass cup (diameter 20 mm, height 10 mm) fitted to the specimen holder. An external flame of 20 mm length was maintained in contact, for 10 s, with the polymer. The LOI value was taken as the minimum percentages of oxygen required in a nitrogen-oxygen atmosphere, surrounding the sample, to maintain its combustion for at least 30 s after ignition. The LOI value was taken as the average of five experiments each.

Apparatus



11. Burning Specimen
12. Clamp with Rod Support
13. Igniter
14. Wire Screen
15. Ring Stand

6. Glass Beads in a Bed
7. Brass Base
8. Tee
9. Cut-off valve
10. Orifice in Holder

1. Pressure Gage
2. Precision Pressure Regulator
3. Filter
4. Needle Valve
5. Rotameter

Figure C.1 LOI apparatus

Procedure

1. Calibrate the flow-measuring system using a water-sealed rotalin drummter in accordance with Method D 1071.
2. The test shall be conducted at room temperature condition in accordance with Practice D 618.
3. Clamp the specimen vertically in the approximate center of the column.
4. Select the desired initial concentration of oxygen. If the specimen burns rapidly, start at a concentration of about 18 %.
5. Set the flow valves so that desired initial concentration of oxygen in flowing through the column.
6. Allow the gas to flow for 30 s to purge the system.
7. Ignition the entire top of the specimen with the ignition flame so that the specimen is well lighted. Remove the ignition flame and start the timer.
8. Do not adjust the oxygen concentration after ignition the specimen.
9. The concentration of oxygen must be raised if the flaming of the specimen extinguishes before meeting.
10. Adjust the oxygen concentration, insert a new specimen.

C-2 DETERMINATION OF INHERENT VISCOSITY

Inherent viscosity [η_{inh}] ASTM D2270: Inherent viscosity is calculated from the dilute solution (1% or less) relative viscosity of the polymer. The inherent viscosity is calculated as:

The relative viscosity is given by:

$$\eta_{rel} = \frac{\text{solution flow time (t), sec}}{\text{solvent flow time } (t_0)\text{, sec}}$$

The inherent viscosity is calculated as:

$$\eta_{inh} = \frac{\eta_{rel}}{C}$$

where

C	=	concentration of the polymer in grams per 100 ml of solvent; usually, C = 0.5 g/100 mL
$\ln\eta_{rel}$	=	natural logarithm of the relative viscosity of the dilute polymer solution
K	=	0.01431, t_0 = 98.97 sec, Kt_0 = 1.4163 sec

Relative viscosity can be taken as the ratio of the flow times of a polymer solution and the pure solvent in the same viscometer and at the same temperature. Relative viscosity values generally are used for calculating the intrinsic or inherent viscosity of a polymer. The solvent to be used will depend on the polymer solubility. In general, the solvent should completely dissolve the sample in less than 30 minutes. It is desirable that the polymer be dissolved at room temperature although, heating is permissible if no degradation occurs. Select the viscometer through which the solvent will flow in not less than 100 seconds and not more than 200 seconds.

Table C.1 Inherent viscosity of metal-containing polyureas

Polymer	Time (sec)			$t_{average}$ (sec)	$Kt_{average}$ (sec)	η_{rel}	η_{inh}
	t_1	t_2	t_3				
$ZnSal_2$trien-HDI	108.28	107.48	108.02	107.93	1.5445	1.0904	0.1731
$ZnOMeSal_2$trien-HDI	108.70	108.40	108.18	108.43	1.5516	1.0954	0.1822
$ZnOEtSal_2$trien-HDI	110.33	110.11	110.26	110.23	1.5774	1.1136	0.2152
$ZnBuSal_2$trien-HDI	116.63	116.85	116.75	116.74	1.6705	1.1794	0.3299
$ZnSal_2$trien-MDI	109.18	108.84	107.20	108.35	1.5504	1.0945	0.1806
$ZnOMeSal_2$trien-MDI	111.29	111.81	112.19	111.76	1.5993	1.1291	0.2428
$ZnOEtSal_2$trien-MDI	113.63	113.70	113.67	113.67	1.6266	1.1483	0.2766
$ZnBuSal_2$trien-MDI	117.24	117.88	118.58	117.90	1.6871	1.1910	0.3497
$NiSal_2$trien-HDI	106.11	106.59	105.95	106.22	1.5200	1.0731	0.1410
$NiOMeSal_2$trien-HDI	105.03	105.11	105.37	105.17	1.5050	1.0625	0.1212
$NiOEtSal_2$trien-HDI	109.13	109.11	108.84	109.03	1.5602	1.1014	0.1932
$NiBuSal_2$trien-HDI	115.65	115.23	115.89	115.59	1.6541	1.1677	0.3101
$NiSal_2$trien-MDI	105.01	104.79	105.19	104.99	1.5024	1.0606	0.1177
$NiOMeSal_2$trien-MDI	110.57	110.61	110.27	110.48	1.5810	1.1161	0.2198
$NiOEtSal_2$trien-MDI	115.15	114.95	114.91	115.00	1.6457	1.1618	0.3000
$NiBuSal_2$trien-MDI	117.35	117.97	-	117.66	1.6837	1.1886	0.3456

VITAE

Miss Thussanee Mananunsap was born on July 9, 1980 in Bangkok, Thailand. She received the Bachelor Degree of Science and Technology in Chemistry from Thammasat University in 2001. Since then, she has been a graduate student studying in the field of Organic Chemistry at Chulalongkorn University and become a member of the Supramolecular Chemistry Research Unit under supervision of Associate Professor Dr. Nuanphun Chantarasiri. She graduated with a Master Degree of Science in Chemistry in 2004.