CHAPTER III

APPARATUS AND EXPERIMENTAL METHODS

3.1 Reagents and materials

3.1.1 Polymeric MDI

(Bayer Thai Co.Ltd.)

Desmodur 44p 90

Carbodiimide-modified diphenyl methane diisocyanate

3.1.2 Polyether polymer polyol

(Union Carbide Thailand Ltd.)

NIAX 31-28

Typical viscosity at 25°C 3,150 cp.

Apparent specific gravity 25/20°C 1.049

Average hydroxyl Number(mg KOH/g) 28.0

3.1.3 Catalyst

(Fluka Chemie AG.)

Dibutyltin dilaurate, $C_{32}H_{64}O_4Sn$

3.1.4 Surfactant

(Dow corning Australia PTY. Ltd.)

Silicone, Dow corning 200 fluid 100 Cs

Table 3-1 Physical properties of MDI and polymeric MDI

Property	MDI	Polymeric MDI composition
Physical state at room temperature	Solid	Liquid
Colour	White to pale yellow	Fawn to dark brown
Odour	None. Pungent at high temperatures	None to aromatic at room temperature
Molecular weight	250.26	Typically about 450
Specific gravity at 15°C	–	1.244
at 20°C	–	1.242
at 25°C	1.23 (solid)	1.239
at 40°C	–	1.224
at 50°C	1.19 (liquid)	–
Melting point (°)	38 to 43	0 (varies with composition)
Boiling point		
at 1 mm Hg. °C	170	–
at 760 mm Hg. °C	314	–
Heat of fusion (Cal/g)	24.3	–
Viscosity,		
(mPa s at 0°C)	–	6000 Typical values
(mPa s at 10°C)	–	1300 Typical values
(mPa s at 20°C)	–	400 Typical values
(mPa s at 25°C)	–	250 Typical values
(mPa s at 50°C)	4.7	–

3.1.5 Blowing agent

(Fluka Chemie AG.)

Trichloromonofluoromethane

3.1.6 Stannous chloride

(Carlo erba. Analyticals)

$SnCl_2.2\ H_2O$, 98%

3.1.7 Stannic chloride pentahydrate

(May and Baker Ltd.)

$SnCl_4 . 5H_2O$ 95%, iron (Fe) 0.004% max.

3.1.8 Zinc chloride, granular

(BDH laboratory)

Minimum assay 95%

Maximum limits of impurities:

Zinc oxide (ZnO)	4%
Iron (Fe)	0.01%
Lead (Pb)	0.07%

3.1.9 Fire retardants

(ITRI)

- Zinc stannate ($ZnSnO_3$)
- Zinc hydroxystannate ($ZnSn(OH)_6$)
- Hydroromarchite ($Sn_3O_2(OH)_2$)

Properties of zinc hydroxy stannate and zinc stannate

	ZHS	*ZS*
Sn	41%	51%
Zn	23%	28%
Cl	< 0.1%	< 0.1%
free H_2O	< 1%	< 1%
specific gravity	3.3	3.9
decomposition temp.(°C)	> 180	> 570
toxicity	very low	very low
specific surface area (m^2/g) Bet method	23.31	42.21

3.1.10 **Ammonium sulphate** , granular grade

(Siam science service Ltd.)

3.1.11 **Decabromodiphenyl ether**

(Fluka Chemie AG.)

$C_6Br_5OC_6Br_5$, M.W. 959.3

melting range 290-310 °C,

bromine content % 82-83

3.1.12 **Sodium hydroxide, pellets GR**

(E.Merck)

M = 40,00 g/Mol

Na_2CO_3	max. 1%
Cl	max. 0.005%

PO_4	max. 0.0005%
N	max. 0.003%
Pb	max. 0.0005%
Al	max. 0.0005%

3.1.13 Ammonia solution 28%

(May and Baker Austalia PTY Ltd.)

NH_3	17.3%

assay (as NH_3) 28.0 to 30.0% w/w

density @ 20 ° C about 0,89 g/ml

3.1.14 Ether

3.1.15 Ethanol

3.2 Apparatus

3.2.1 Flammability testing (Limit Oxygen Index (LOI) Test. ASTM D 2803-87)

3.2.2 Tensile testing (ASTM D 412)

machine model Lloyd 500

3.2.3 X-ray diffractometer

JEOL Model JDX - 8030

3.2.4 Fourior transform infrared spectrometer (FTIR)

Perkin-Elmer Model 1760 x

3.2.5 Differential Scanning Colorimetry (DSC)

Shimadzu Model DT-30

3.2.6 Inductive couple plasma spectroscopy (ICP)

Perkin-Elmer Model Plasma 1000

3.2.7 Bet apparatus

3.2.8 Mechanical stirrer

Ika- Laborteknik Model RW 20

3.3 Preparation of fire retardants

3.3.1 Sodium hydroxystannate ($Na_2Sn(OH)_6$)

White gelatinous precipitate of stannic hydroxide, $Sn(OH)_4$, was prepared by adding 1 N sodium hydroxide solution to acid solution of 20 gm. stannic chloride pentahydrate $SnCl_4 \cdot 5H_2O$. Sodium hydroxide solution was added with stirring continuously until the precipitate of stannic hydroxide disappeared and the clearsolution was produced (about 5 N of OH group). The mixture was left for three hours at room temperature and the white colloidal precipitate of sodium hydroxystannate was occured. The precipitate of sodium hydroxystannate was seperated from the solution by using flash evaporator. The insoluble residues was filtered off, and washed with 5N-sodium hydroxide, alcohol, and finally ether.The precipitate of sodium hydroxystannate was dried in desscicator at room temperature (18). This procedure gave 98% yield.

$$SnCl_4.5H_2O \; + \; 4\; NaOH \xrightarrow{HCl} Sn(OH)_4 \; + \; 4\; NaCl$$

2 NaOH

$Na_2Sn(OH)_6$

colloidal precipitate

98% yield

3.3.2 Zinc hydroxy stannate and zinc stannate

Zinc hydroxy stannate, $ZnSn(OH)_6$, was prepared in aqueous solution by a double decomposition reaction between the zinc nitrate or zinc chloride (1.5 gm.) and sodium hydroxy stannate (3 gm.). Zinc chloride was first dissolved in excess aqueous ammonia before addition to the $Na_2Sn(OH)_6$ and the pH of

$$Na_2Sn(OH)_6 \; + \; ZnCl_2 \longrightarrow ZnSn(OH)_6 \; + \; NaCl$$

the solution was kept above 12. The product was washed with distilled water and dried at 60 ^{o}C in air.

Zinc stannate (anhydrous stannate), $ZnSnO_3$, was prepared by thermal dehydration of the zinc hydroxy stannate at 100 °C in air.

$$ZnSn(OH)_6 \xrightarrow{100^{\circ}C} ZnSnO_3$$

Hydroromachite, $Sn_3O(OH)_2$, was prepared in the a similar manner, except stannous chloride was not dissolved in excess ammonia solution.

$$Na_2Sn(OH)_6 + SnCl_2 \longrightarrow Sn_3O_2(OH)_2 + NaCl$$

The identities of all products were confirmed by infrared spectrometer and X-ray diffraction patterns.

3.3.3 Char preparation

The polyurethane samples were burned completely to be char and then analyzed tin contents by ICP.

3.4 Sample preparation

3.4.1 Incorporation of tin compounds

In preparation of polyurethane foams, the amount of zinc stannate, zinc hydroxystannate and hydroromarchite required to impart flame retardancy can be calculated on the amount of total ingradients are employed, Preferably, from about 0.5 to about 10 part by weight of tin compound per 100 parts by weight of total composition.

The flexible foams are usually prepared at ambient conditions by adding the polyol, catalyst, surfactant, blowing agent and flame retardant, as one component, to the isocyanate and water, using conventional techniques.

In preparation rigid foams, water as a blowing agent, is usually eliminated as an ingradient to preclude the liberation of carbon dioxide.

3.4.2 The ingradient used of polyurethanes

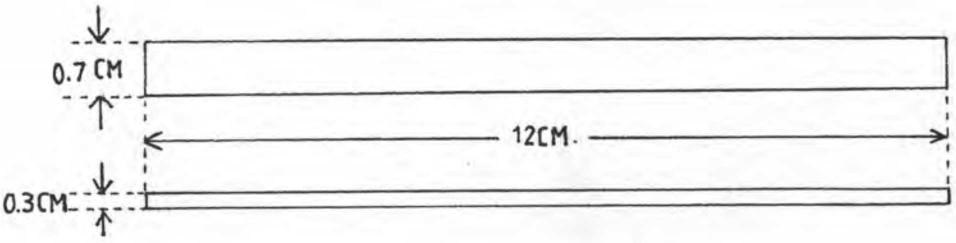
The samples were prepared by 3 main steps :

3.4.2 (a) ***Blending of polyurethane compound.*** The mechanical stirrer was used for blending. The raw materials were devided into 2 parts :

Part A :		Part B :	
	polyether polyol		polymeric MDI
	catalyst		water
	surfactant		
	blowing agent		
	fire retardant		

First, the raw materials in part A except fire retardant were mixed together at a motor speed of 500 rpm. at ambient temperature for 1 hours. Then fire retardant was mixed into the materials. The mixing time was 2 hours because fire retardant was absorbed by the polyol mixture (all of compound materials were liquids except fire retardant). After that, the materials in part B were added into part A and mixed at the same speed for 1 minute (the temperature slightly increased).

3.4.2 (b) ***Moulding.*** Pouring the polyurethane compound into the silicone rubber mould (the dimension used as the figure below). The reaction time for produced CO_2 bubbles and formed the polymer matrix was 3 hours (the temperature slightly increased).



width	**0.7**	**cm.**
length	**12.0**	**cm.**
thickness	**0.3**	**cm.**

3.4.2 (C) ***Curing.*** Polyurethanes were cured at 80 °C for 1 hour to form cross-linked structure.

3.5 Recipes of polyurethane compounding

The sample of polyurethanes were prepared in 3 recipes :

Recipe 1 **Polyurethane (no fire retardants)**

Table 3-2 Ingradient used in flexible and rigid polyurethane foams.

Ingradient	formulation, amt.,pbw.	
	Flex. PU	Rigid PU
A.		
Polyol	100	100
Catalyst	0.02	0.02
Surfactant	1.0	1.0
Blowing agent	0.2	0.2
B.		
Water	0.5	-
Polymeric MDI	30	30

Recipe 2 : Fire retardant polyurethane foams

Fire retardant used :

- Zinc stannate (ZS)
- Zinc hydroxy stannate (ZHS)
- Hydroromarchite (HDC)

Table 3-3 Ingradient used in fire retardant polyurethane foams.

Ingradient	formulation, amt., pbw.	
	Flex. PU	Rigid PU
Polyol	100	100
Catalyst	0.02	0.02
Surfactant	1.0	1.0
Blowing agent	0.2	0.2
Fire retardant :		
o.5 %	0.665	0.655
1.0 %	1.330	1.315
2.0 %	2.690	2.625
5.0 %	6.940	6.565
10.0 %	14.635	13.125
Water	0.5	-
Polymeric MDI	30	30

Recipe 3 : Fire retardant polyurethane containing halogen compound

Fire retardants used :
A. Zinc hydroxy stannate (ZHS)
Zinc stannate (ZS)
B. Dibromodiphynyl ether (DBDPE)
Ammonium sulphate (AS)

Table 3-4 (a) Ingradient used in flexible polyurethane foam containing ZHS and DBDPE compound.

Ingradient	formulation, amt., pbw.
Polyol	100
Catalyst	0.02
Surfactant	1.0
Blowing agent	0.2
Fire retardant :	
1% ZHS : 2% DBDPE	1.375 : 2.750
: 5% DBDPE	1.400 : 7.030
: 10% DBDPE	1.488 : 14.825
: 20% DBDPE	1.663 : 33.360
5% ZHS : 2% DBDPE	7.100 : 2.838
: 5% DBDPE	7.313 : 7.313
: 10% DBDPE	7.750 : 15.488
: 20% DBDPE	8.788 : 35.150
Polymeric MDI	30
Water	2.0

Table 3-4 (b) Ingradient used in flexible polyurethane foam containing ZS and DBDPE compound.

Ingradient	formulation, amt., pbw.
Polyol	100
Catalyst	0.02
Surfactant	1.0
Blowing agent	0.2
Fire retardant :	
1% ZS + 2% DBDPE	1.375 : 2.750
: 5% DBDPE	1.400 : 7.030
: 10% DBDPE	1.488 : 14.825
: 20% DBDPE	1.663 : 33.360
5% ZS : 2% DBDPE	7.100 : 2.838
: 5% DBDPE	7.313 : 7.313
: 10% DBDPE	7.750 : 15.488
: 20% DBDPE	8.788 : 35.150
Polymeric MDI	30
Water	2.0

Table 3-5 (a) **Ingradient used in fire retardant flexible polyurethane foams containing ZHS and AS.**

Ingradient	formulation, amt., pbw.
Polyol	100
Catalyst	0.02
Surfactant	1.0
Blowing agent	0.2
Fire retardant :	
1% ZHS + 2% AS	1.375 : 2.750
+ 5% AS	1.400 : 7.013
+ 10% AS	1.488 : 15.488
+ 15% AS	8.250 : 24.750
5% ZHS + 2% AS	7.100 : 2.838
+ 5% AS	7.313 : 7.313
+ 10% AS	7.750 : 15.488
+ 15% AS	8.25 : 24.750
Polymeric MDI	30
Water	2.0

Table 3-5 (b) Ingradient used in fire retardant flexible polyurethane foams containing ZS and AS.

Ingradient	formulation, amt., pbw.
Polyol	100
Catalyst	0.02
Surfactant	1.0
Blowing agent	0.2
Fire retardant :	
1% ZS + 2% AS	1.375 : 2.750
+ 5% AS	1.400 : 7.013
+ 10% AS	1.488 : 15.488
+ 15% AS	8.250 : 24.750
5% ZS + 2% AS	7.100 : 2.838
+ 5% AS	7.313 : 7.313
+ 10% AS	7.750 : 15.488
+ 15% AS	8.250 : 24.750
Polymeric MDI	30
Water	2.0

3.6 Measurement

3.6.1 Flammability test

Because most polyurethane foams are use in building construction, upholstery and others, it is important that most flammability testing is carried out. The objective of fire testing is both to avoid any increase in the hazards in the real fire and to guide progress toward reduction in the fire hazard whilst improving the cost and energy efficiency of the building.

Flammability tests are of three types :

- Small-scale laboratory bench tests for quality control, specification or sorting purposes.
- Intermediate-scale tests designed to classify a material or construction for use in buildings.
- Full-scale fire testing under controlled conditions.

Limit Oxygen Index (LOI) Test (ASTM D 2863-87)

Small-scale tests include flash point and oxygen index tests which measure properties of the polymer rather than that of the foam or of a particular foam composite structure. ASTM 2863-87 are oxygen index test. This test which give good reproductibility, describes a procedure of measuring the oxygen index- the minimum concentration of oxygen in the flowing mixture of oxygen and nitrogen that will just support flaming combustion. Such test cannot be used to predict the performance of a material in a real fire, but have often been used to rank the effect of novel additives on flammability.

The minimum concentration of oxygen in a mixture of oxygen and nitrogen flowing upward in a test column that will just support combustion is measured under equilibrium conditions of candle-like burning. The equilibrium is established by the relation between the heat loss to the surroundings as measured by one or the other of two arbitrary criteria, namely, a time of burning or a length of specimen burned. This point is approached from both sides of the critical of oxygen concentration in order to establish the oxygen index (16).

Apparatus of flammability test

- **Test column** consisting of a heat-resistant glass-tube of 105 mm. minimum inside diameter and 450 mm. minimum height. The bottom of the column or the base to which the tube is attached shall contain noncombustible material to mix and distribute evenly the gas mixture entering at this base. Glass beads 3 to 5 mm. in diameter in a bed 80 to 100 mm. deep have been found sutiable (an example is shown in Fig. 3-1)

- **Specimen Holder** - Any small holding device that will support the specimen at its base and hold it vertically in the center of the column is acceptable. For physically self-supporting specimens, a typical arrangement consists of a laboratory thermometer clamp inserted into the end of a glass tube held in place by glass beads .

- **Gas Supply** - Commercial grade (or better) of oxygen and nitrogen shall be used.

- **Flow measurement and control devices.** - Suitable flow measurement and control devices shall be available in each line that will allow monitoring. The volummetric flow of each gas flew into the column within the range 0-2 L/min.

- **Ignition source** - The igniter shall be a tube with a small orifice (1 to 3 mm.in diameter) leaving a LPG gas flame at the end that can inserted into the open end of the column the test specimen. A suitable flame may be form 6 to 25 mm.long.

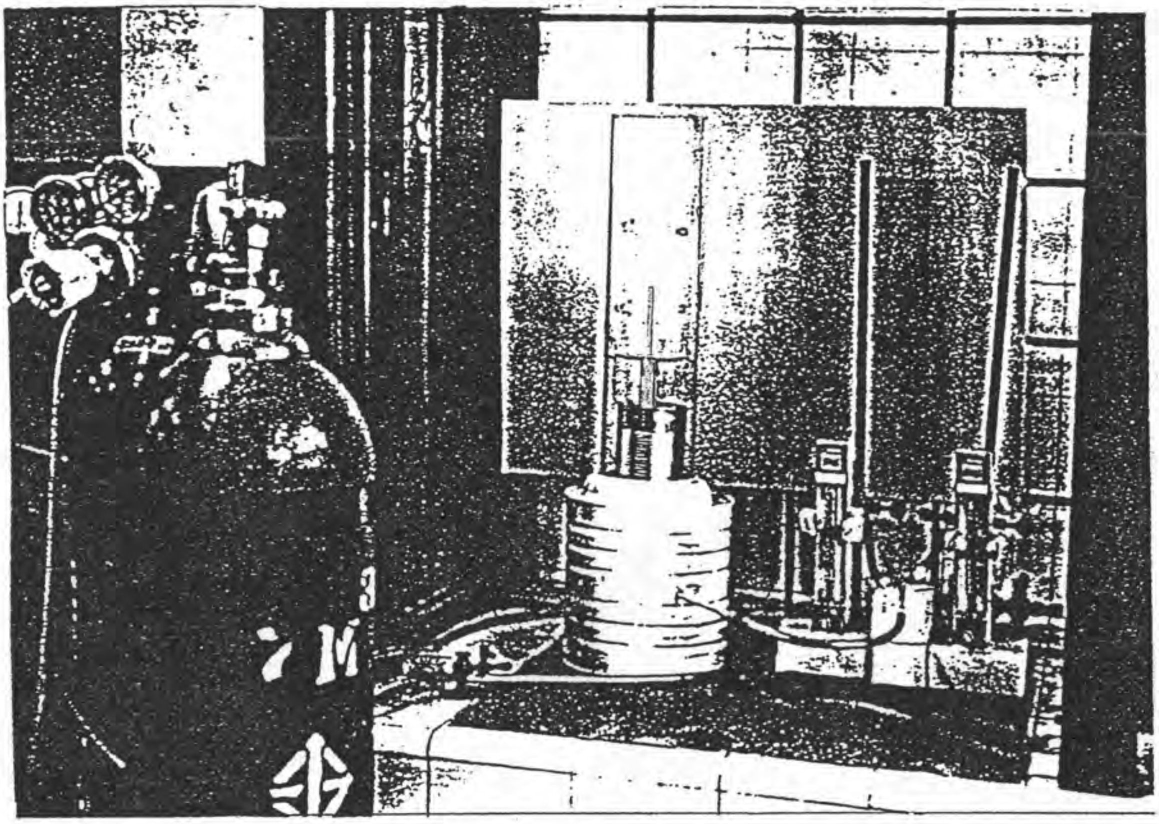
- **Test specimens** - Be cutted a sufficient of specimens (normally 5 to 10) from the material to be tested. Type B from Table 3-6 is the specimen dimensions used.

Table 3-6 The specimen dimension used (mm.)

Type	**Plastic form**	**Wdth**	**Thickness**	**Length**
A	Plastic self-supporting	6.5+0.5	3.0+0.5	70 to 150
B	Alternate for self-supporting flexible plastic	6.5+0.5	2.0+0.25	70 to 150
C	Cellular plastic	12.5+0.5	12.5+0.5	125 to 150

In system of polyurethanes, ZHS and ZS were insoluble in the precursors to the polyurethane foams, and, as the foam forms, these insoluble additives are came along within the heading of the rising foam, with the result that these additives were poorly distributed with in the resulting polymer. Cutting of the specimens from the polyurethane materials to be tested were not the real representatives (17).

Figure 3-2 LOI apparatus



Because lacking of the equipments to measure LOI values of polyurethane foams by means of ASTM.2863-87, the LOI equipment was built by following the procedure described by ASTM.2863-87. By that way, LOI values were determined by fixed the gas flow rate in the column at 4 + 1 mm/sec at STP and converted to the total flow of gas in mm/s by multiplied with area of the column.

Procedure

a. Correction for gas flow

The volume flow of air at standard conditions for each of the flowmeters could converted to the flow of oxygen and nitrogen gases at standard condition by means of equation below :

$$V_o' = V_o \left[\frac{0.00120}{\rho_o} \right]^{1/2}$$

where, V_o = volume of gas in ml/min. measured and flowing at standard condition

V_o = volume of air in ml/min. measured and flowing at standard from manual

ρ_o = density of gas in gms./ml at STP

Equation above was accurate when the viscosity of the gas was close to that of air. The test should be conducted at room temperature conditions, the volume of gas when flowing at other than standard conditions could be calculated from the volume at standard conditions by means of equation below:

$$\left[\frac{P\ V}{T}\right]_{\text{room temp}} = \left[\frac{P\ V}{T}\right]_{\text{STP}}$$

$T_{\text{room temp}}$ = 34 °C

$P_{\text{room temp}}$ = 1007.8 millibar

then,

$V_{\text{room temp}} = 1.13034 \times V_{\text{STP}}$

Then, the volume of gas flow at room temperature were determined,

$$\underset{\text{(mm/sec)}}{\text{flow rate of gas}} = \frac{\text{Total volummetric flow gases (mm}^3\text{/sec)}}{\text{area of the columm (mm}^2\text{)}}$$

total volummetric flow of gases = flow rate of gas x area

flow rate of gas = 4 mm/sec

diameter of glass column = 10.3 cm.

$$\text{total volummetric flow of gases} = \frac{4}{10} \times \pi \left[\frac{10.3)}{2}\right]^2$$

$$= 2 \quad \text{L/min}$$

By set LOI value, $[O_2]$ and $[N_2]$ are determined by the equation below :

$$\text{LOI (\%)} = \frac{[O_2]}{[O_2]+[N_2]} \times 100$$

$$[O_2] = \text{LOI (\%)} \times \frac{[O_2]+[N_2]}{100}$$

but $$[O_2]+[N_2] = 2 \quad \text{L/min}$$

$$[O_2] = \frac{\text{LOI (\%)} \times 2}{100} \quad \text{L/min}$$

example , if set LOI = 18 % , then

$$[O_2] = \frac{18 \times 2}{100} \quad \text{L/min}$$

$$= 0.36 \quad \text{L/min}$$

$$= 360 \quad \text{ml/min}$$

$$[N_2] = 2000-360 = 1640 \quad \text{ml/min}$$

Table 3-7 List of $[O_2]$ and $[N_2]$

LOI	$[O_2]$	$[N_2]$
18	360	1640
19	380	1620
20	400	1600
...	...	...
30	600	1400
31	620	1380
...	...	...

From the scale reading of flowmeter, the positions of glass ball of nitrogen and oxygen which had the volummetric flow rate as above :

Table 3-8 Glass ball positions which calculate from $[N_2]$ and $[O_2]$

LOI	$[O_2]$	$[N_2]$	ball position	
			O_2	N_2
18	360	1640	17.0	47.0
19	380	1620	17.8	46.5
20	400	1600	18.5	46.0
...	...	...	...	...
30	600	1400	25.5	41.9
31	620	1380	25.8	41.4

Set the glass ball positions so that the desired $[o_2]$ and $[N_2]$ were flowing through the column. Allow the gas flow for 1 min. to purge the system. Ignite the entire top of the specimen with ignition flame so that the specimen well lighted. Remove the ignition flame and start the timer. Adjust the $[O_2]$ and $[N_2]$, insert a new specimen, or if the previous specimen was long enough, turn it end for end or cut off the burned end, then purged and re-ignited. Continue repeating these steps until the critical oxygen concentration was determined.

Type	A and B	C
Criteria for burning	at least 3 min. or 50 mm.	at least 3 min. or 75 mm.

This is the lowest oxygen concentration that would meet the criterion of above table.

3.6.2 Mechanical test

The characteristic properties of material were obtained by using standard test methods on specimens of a standard size and shape under standard environmental conditions. The recommended tests for the basic properties were those defined by the standard test methods of the American Society for Testing and Materials (ASTM).

Polyurethane materials were characterised by their density and their basic mechanical properties.

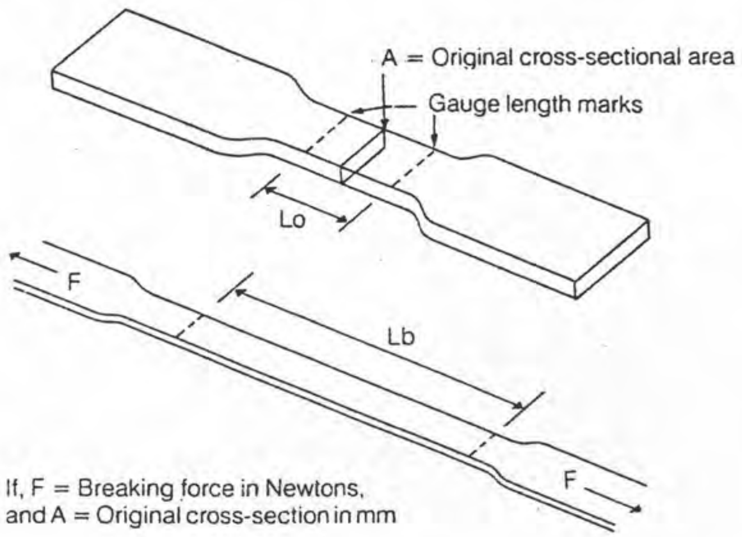
Tensile test (ASTM D412)

Tensile properties were important characteristics of the strength of polyurethane foams. The specimen was is stretched at a constant standard rate until it was broken. The **tensile strength** is the maximum stress the materials withstands before rupture.

The **elongation at break** or the **ultimate elongation** is the maximum extension of the specimen at the point of rupture. By plotting stress against strain (stress is usually expressed as the percentage deformation of the specimen) the same yield the same modulus of elasticity.

Specimens of polyurethanes for tensile testing are produced by moulding. The polyurethane mixture is poured in to silicone rubber mould. Figure 3-3 show the specimen dimension used for tensile testing. Perform the test three times.

Figure 3-3 Measuring tensile properties



If, F = Breaking force in Newtons,
and A = Original cross-section in mm

Then, Tensile strength (MPa or Kg/mm^2) = 1,000 F/A

and Elongation at break (0%) $\doteq \frac{(Lb - Lo) \times 100}{Lo}$

Specimen was type IV (19) as illustrated in figure 3.3 The cross head speed of universal testing machine at 100 mm/min was used. The test condition was at 27+2 ° C. An average of five specimens was considerd as representative value. The gage range was 2.50 cm. and the thickness of specimen was 2 mm.

Tensile stress = F/A

F = Tensile force (N)

N = Cross section area (m^2)

Tensile stress = N/m^2 (Pascal) or MPa

Tensile strength at break is tensile stress at which the specimen ruptured.

Modulus at 100% elongation = F/A

F = Tensile forced at 100% elongation (N)

3.6.3 X-ray diffraction measurement

X-ray diffraction spectrophotometer is used for studying elemental analysis, electronic structure and crystal structure. The structures of sodium hydroxystannate, zinc hydroxystannate and hydroromarchite were investigated by X-ray diffraction. X-ray diffraction patterns, line patterns of sodium hydroxystannate, zinc hydroxystannate and hydroromarchite are shown in Figure 4-12 to 4-14.

3.6.4 Fourier transform infrared spectrophotometer (FTIR)

Fourier transform infrared spectrophotometer is used for investigation of functional groups of chemical compounds. Sodium hydroxystannate, zinc hydroxystannate and hydroromarchite were prepared by KBr pellet method. The functional groups of these compounds were determined. The FTIR spectrograms are shown in Figure 4-15 to 4-17.

3.6.5 Differential scanning calorimetry (ASTM D 3417)

DSC is a technique for calorimetric measurements either by recording and integrating the temperature difference or recording the energy necessary to establish zero temperature difference between a substance and a reference material against either time or temperature as the two specimens are subjected to identical temperature regimes in an environment heated or cooled at a controlled rate. DSC is a thermal analysis technique that measures the quantity of energy absorbed or evolved (given off) by a sample in calories as its temperature is changed.

A polymer characterization with DSC technique is carried out by studying the melting behavior, crystalline melting point, degree of cure, percentage of additives in a polymer, oxidative stability, and degree of cross linking. DSC results are shown in Table 4-12.

3.6.6 Inductively coupled plasma spectroscopy (ICP)

ICP technique are similar in principle to flame emission spctroscopy but use much more energetic atomization-excitation processes. the energy available in these atomizers is sufficient to exite atoms to many upper lavels. In addition, a substantial number of ions are formed, which also become excited. As a result, the emission spectra are complex, containing dozens of line of each element. This complexity can be an advantage in identifying which elements are present in a sample. These high-energy atomizers also are more efficient than flame at breaking down the stable oxides formed by refractory elements.

The samples of polyurethane containing zinc stannate and zinc hydroxystannate and the corresponding ash were investigated tincontents. Such samples were digested with sulfuric acid and hydrochloric acid. Then the sample liquids were dissolved in 10% hydrochloric acid. The sample solutions were analyzed by ICP. ICP results were used to enhance the mode of action of metal stannates were in condense phase or vapor phase. These results are shown in Table 4-7 .

3.6.7 BET apparatus

The BET method is the modern technique for measuring particles by nitrogen absorption for determination of filler's particle size. The results are shown on page 68.

3.6.8 Smoke density apparatus

It has been reported that (23) that smoke emission can present a greater or more immediate danger than toxic gases since smoke itself is toxic and, more importantly, it reduces visibility and could prevent a rapid escape from the hazardous situation. It has been estimated that approximately 80% of the victims of fire are not harmed by flames, but die as a result of " smoke inhalation " (24). Hence, the emission of smoke and toxic gases represents one of the major hazards of fires. Unfortunately, many flame retardant additives cause and increase in the amount of smoke formed on burning.

Several test methods, all based on the use of a photoelectric cell to measure the optical density of the smoke, have been devised. A smoke chamber, marketed by Stanton Redcroft, is designed for operation with their Limiting Oxygen Index equipment although, in this chamber, a horizontal light beam passes through the smoke evolved. Smoke density determinations were obtained by Stanton-Redcroft FTB Unit. In each case, four samples were examined and the mean value of optical density was recorded in Figure 4-18 and 4-19.