CHAPTER III

EXPERIMENTAL SECTION

Materials

1. Polypropylene
   (Thai Petrochemical Industry Public CO., LTD.)
   "POLENE 1100 RC" , Injection Molding Grade.

2. Polypropylene
   (Thai Petrochemical Industry Public CO., LTD.)
   "POLENE 1196 RC" , Injection Molding Flame Retardant Grade.

3. Antimony trioxide
   (Cookson Minerals Limited)
   'Trimonox' Red Star Standard Grades

Sb_2O_3 content	99.6%
Arsenic (As)	0.2%
Iron (Fe)	0.003%
Lead (Pb)	0.08%
Acidity	0.02%

4. Decabromodiphenyl oxide

   (Dead Sea Bromine CO., LTD.)

   $C_{12}Br_{10}O$ M.W. 959.2

   melting range 300-305 °C

   bromine content 83%

   specific gravity (20°C) 3.0

5. Zinc Stannate

   (Alcan Chemicals Limited.)

   $ZnSnO_3$

6. Zinc Hydroxystannate

   (Alcan Chemicals Limited.)

   $ZnSn(OH)_6$

Table 3-1 Chemicals and Physical Properties of zinc stannate and zinc hydroxystannate

Properties	ZS	ZHS
Decomposition temperature (°C)	> 300	200
Median particle size (μ m)	1.7	< 2
Specific gravity (g/cm^3)	3.9	3.3
Free moisture (%)	< 1	< 1

Instruments

1. **Flammability Testing**

   Limit Oxygen Index (LOI) ASTM D 2863

   Polymer Laboratories (Epsom) Limited.,

   Model FTA II Serial Number 16000

2. **Tensile Testing**

   Tensile Properties of plastic ASTM D 638-58 T

   LLOYD Instruments Limited.,

   Model LR 30 K

3. **Impact Testing**

   Impact Resistance of plastics and Electrical Insulating Materials ASTM D 256-56

   Yasuda Impact Tester (Izod Type)

4. **Two-Rolls Mill**

   Lab Tech CO., LTD.

   Model LRM 150

5. **Thermogravimetric**

   NETZSCH STA 409 C

6. **Neutron Activation Analyzer**

   CANBERRA S100

Experimental

1. Sample Preparation

1.1 Method of Compounding

The first step in preparing of PP sample for testing was blending polypropylene with different materials into a homogeneous mass.

As stated earlier, blending was carried out typically on a Two-Roll Mills. The material was dropped directly onto a mill and allowed to preheat for 1-2 minutes prior to start the roll turning.

To aid mixing, the compound was crossed cut back and forth using a mill knife. A mixed batch should be carried for the minimum of 5 minutes after blending. Distance between the rolls (the roll nip) was adjusted to facilitate fluxing. Later, Strip cutters are used to cut ribbons of stock from the roll for feeding to cool down and ground into small pieces.

Front roll temperature : 175°C
Back roll temperature : 168°C

1.2 Molding

For molding flammability, tension and impact specimens of general purpose, the final blend polypropylene were molded on a injection-molding machine with the following processing conditions :

Time-programming.

melting time	:	24	seconds
cooling time	:	10	seconds
injection time	:	8	seconds
die close/open time	:	14	seconds

Pressure-programming

1st injection pressure	:	30	PSI
2nd injection pressure	:	30	PSI
3rd injection pressure	:	30	PSI

Temperature-programming

1st zone temperature	:	180°C
2nd zone temperature	:	190°C
3rd zone temperature	:	200°C

2. Recipes of Polypropylene Compounding

The sample of polypropylene were prepared as follows

Recipe 1 : Flame-retarding polypropylene.

Flame retardant used : Zinc stannate (ZS)
Zinc hydroxystannate (ZHS)
Antimony trioxide (Sb_2O_3)

Table 3-2 Ingredient used in flame-retarding polypropylene containing ZS, ZHS and Sb_2O_3.

Ingredient	Formulation (g.)
Polypropylene	100
Flame retardant	
2.5 %	2.56
5.0 %	5.26
7.5 %	8.10
10.0 %	11.11

Recipe 2 : Flame-retarding halogenated polypropylene.

Flame retardant used : Decabromodiphenyl oxide (DBDPO)

Table 3-3 Ingredient used in flame-retarding polypropylene containing DBDPO compound.

Ingredient	Formulation (g.)
Polypropylene	100
Flame retardant :	
5 % DBDPO	5.62
10 % DBDPO	11.11
20 % DBDPO	25.00
30 % DBDPO	42.85

Recipe 3 : Flame-retarding halogen-free polypropylene system.

Flame retardant used : Alumina trihydrate (ATH).

Table 3-4 Ingredient used in flame-retarding polypropylene containing ATH compound.

Ingredient	Formulation (g.)
Polypropylene	100
Flame retardant :	
10% ATH	11.11
20% ATH	25.00
30% ATH	42.85
40% ATH	66.66
50% ATH	100.00

Recipe 4 : Flame-retarding halogenated polypropylene system.

Flame retardant used : A Zinc stannate (ZS)

Zinc hydroxystannate (ZHS)

Antimony trioxide (Sb_2O_3)

B. Decabromodiphenyl oxide (DBDPO)

Table 3-5 (a) Ingredient used in flame-retarding polypropylene containing ZS and DBDPO compound.

Ingredient	Formulation (g.)
Polypropylene	100
Flame retardant :	
2.5% ZS : 5% DBDPO	2.70 : 5.40
10% DBDPO	2.85 : 11.42
20% DBDPO	3.22 : 25.80
30% DBDPO	3.07 : 44.44
5.0% ZS : 5% DBDPO	5.55 : 5.55
10% DBDPO	5.88 : 11.76
20% DBDPO	6.66 : 26.66
30% DBDPO	7.69 : 46.15
7.5% ZS : 5% DBDPO	8.57 : 5.71
10% DBDPO	9.09 : 12.12
20% DBDPO	10.34 : 27.58
30% DBDPO	12.50 : 48.00

Table 3-5 (b) Ingredient used in flame-retarding polypropylene containing ZHS and DBDPO compound.

Ingredient	Formulation (g.)
Polypropylene	100
Flame retardant :	
2.5% ZHS : 5 % DBDPO	2.70 : 5.40
10 % DBDPO	2.85 : 11.42
20 % DBDPO	3.22 : 25.80
30 % DBDPO	3.07 : 44.44
5.0% ZHS : 5 % DBDPO	5.55 : 5.55
10 % DBDPO	5.88 : 11.76
20 % DBDPO	6.66 : 26.66
30 % DBDPO	7.69 : 46.15
7.5% ZHS : 5 % DBDPO	8.57 : 5.71
10 % DBDPO	9.09 : 12.12
20 % DBDPO	10.34 : 27.58
30 % DBDPO	12.50 : 48.00

Table 3-5 (c) Ingredient used in flame-retarding polypropylene containing Sb_2O_3 and DBDPO compound.

Ingredient	Formulation (g.)
Polypropylene	100
Flame retardant :	
2.5% Sb_2O_3 : 5% DBDPO	2.70 : 5.40
10% DBDPO	2.85 : 11.42
20% DBDPO	3.22 : 25.80
30% DBDPO	3.07 : 44.44
5.0% Sb_2O_3 : 5% DBDPO	5.55 : 5.55
10% DBDPO	5.88 : 11.76
20% DBDPO	6.66 : 26.66
30% DBDPO	7.69 : 46.15
7.5% Sb_2O_3 : 5% DBDPO	8.57 : 5.71
10% DBDPO	9.09 : 12.12
20% DBDPO	10.34 : 27.58
30% DBDPO	12.50 : 48.00

3. Measurement

3.1 Flammability Testing

Limiting Oxygen Index (LOI) Testing : ASTM D 2863-91

The limiting oxygen index (LOI) of the specimens, in the form of thin strips of approximate dimensions of 70x7x3 mm., was determined according to ASTM D 2863-91, using a Stanton Redcroft FTA module.

At least five specimens were tested for each sample at room temperature conditions.

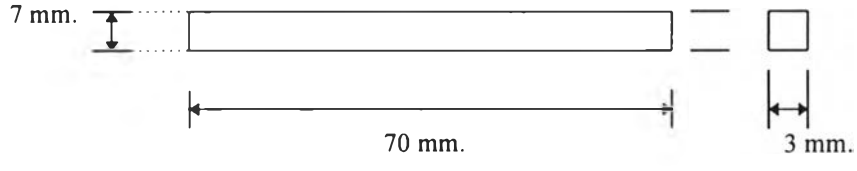


Figure 3-1 Specimen dimensions for measurement of limiting oxygen index.

Oxygen index which was defined as the minimum concentration of oxygen, expressed as volume percent, in the mixture of oxygen and nitrogen that would support flaming combustion of a material, was measured under equilibrium conditions of burning. The equilibrium was established by the relation between the heat generated from the combustion of the specimen and the heat lost to the surroundings as measured by one of these two criterias, namely, a time of burning or a length of specimen burned.

Test procedures are as following:

a) Ensure oxygen and nitrogen are connected and turn on.

b) Connect mains to instrument and switch the switch on the rear panel on . Leave to stabilize for 15 minutes.

c) With O_2 on/off value closed, open N_2 on/off valve. Adjust N_2 Needle valve until the flowmeter indicates a flow of 18 l/min.

d) Adjust zero potentiometer for a display of 0.00 % O_2.

e) Close N_2 on/off valve and open O_2 on/off valve. Adjust O_2 needle valve until the flowmeter indicates a flow of 18 l/min.

f) Adjust span potentiometer for a display of 99.7 % O_2 (or the %O_2 of the oxygen used).

g) Close the O_2 on/off valve.

h) Place specimen in the sample holder.

i) Remove chimney and place sample holder with loaded sample in position (See Figure 3-2).

j) Place chimney over sample holder and sample, and ensure it is clipped securely into retaining clips.

k) Light the burner and place in a safe position.

l) Open both O_2 and N_2 on/off valves and adjust both needle valve until the total flow is 18 l/min. and the concentration of O_2 is as required for the sample. Purge at this flow rate for 30 to 40 seconds to ensure stability.

(N.B. If the sample being tested is unknown, set the starting O_2 concentration to approximately 25% O_2)

m) Using the burner, light the sample following the precise protocol for the standard being followed.

n) Using the O_2 and N_2 needle valves, adjust the O_2 concentration, whilst maintaining the flow at 18 l/min., so that the sample burning front is just maintained.

o) Record the O_2 concentration as the oxygen index percent.

p) At the completion of the run, close the O_2 and N_2 on/off valve and the needle valves. Remove the chimney and sample holder and any remnants of the sample.

q) Unless further runs are to follow, turn off the instrument and the O_2 and N_2 supplies.

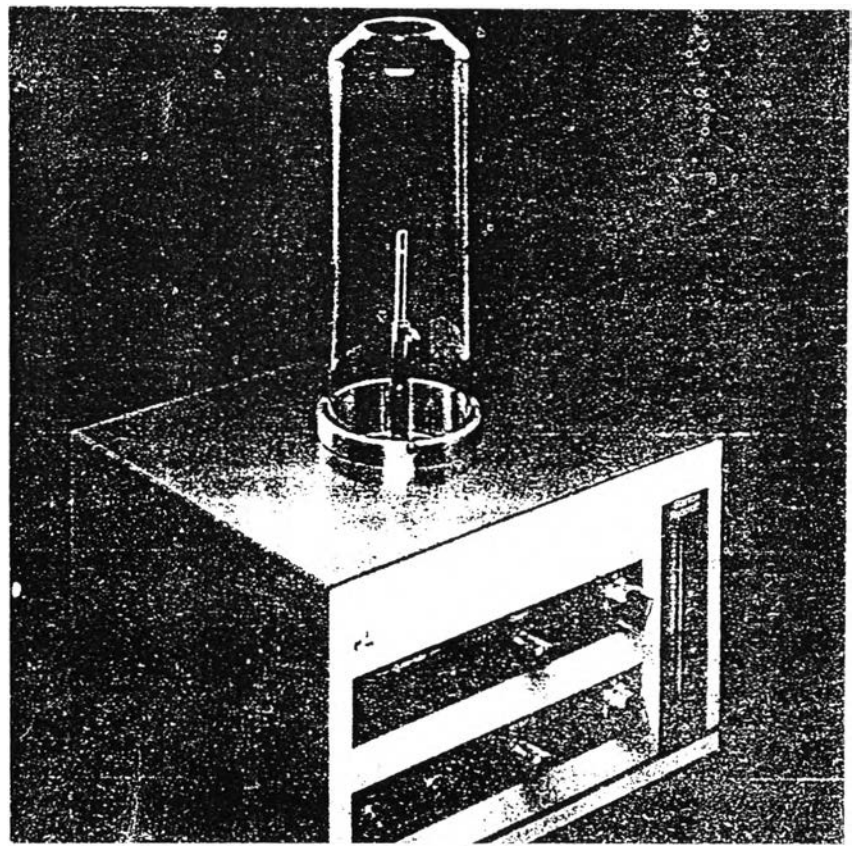


Figure 3-2 Oxygen index tester.

3.2 Physical Properties Testing.

3.2.1 Tensile Properties Testing (ASTM D 638-58T)

At least five specimens were tested for each sample in the case of isotropic materials. The test specimen was conformed to the dimension shown in Figure 3-3, conditioned and tested under the room temperature which was maintained at 23°C in accordance with procedure of the Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (ASTM designation : D 618)

The test specimen was placed in the grips of the testing machine, with the distance between grip set to 100 mm. And the speed of testing was 50 mm/min. The tensile strength and modulus of elasticity were obtained.

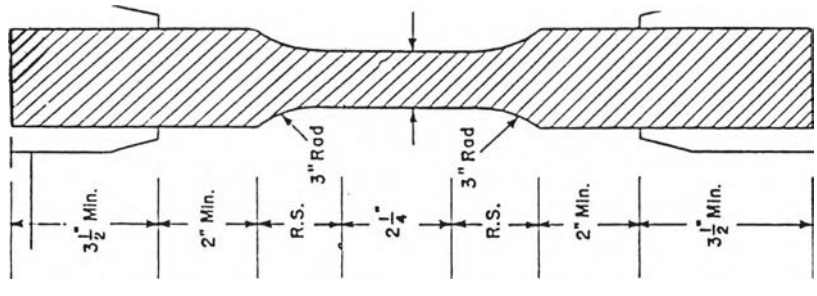


Figure 3-3 Tension test specimen dimensions for mold plastics.

This method was intended for determining the comparative tensile properties of plastics in the form of standard test specimens and when tested under refined conditions of pretreatment, temperature, and testing machine speed.

3.2.2 Impact Resistance Testing (ASTM D 256-56)

These testing methods were intended to determine the relative susceptibility to fracture by shock of plastic materials and electrical insulating materials as indicated by the energy expanded by a standard pendulum type impact machine in breaking in one blow a standard specimen.

In this research, the specimen was tested on Izod type test in which it was held as a cantilever beam and was broken by a blow delivered at fixed distance from the edge of the specimen clamp, as shown in figure 3-4. The test specimen was conformed to the dimensions shown in figure 3-5,

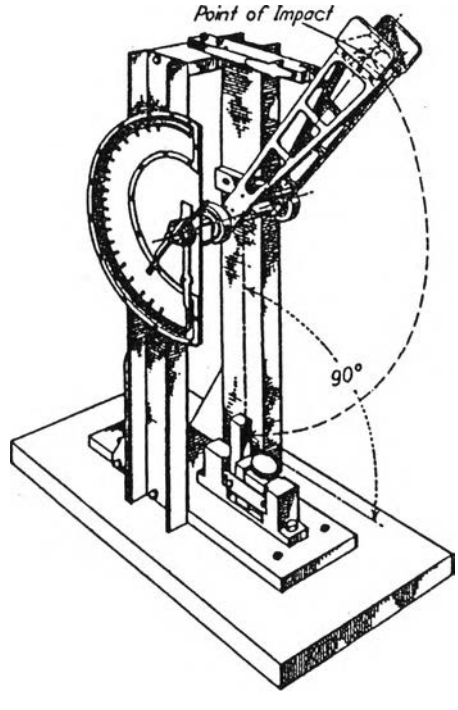


Figure 3-4 Cantilever beam (Izod type) impact machine.

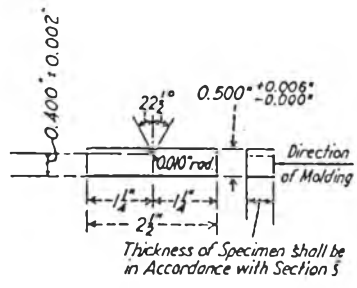


Figure 3-5 Cantilever beam (Izod type) impact test specimen dimension.

4. Neutron Activation Analysis

The neutron activation analysis test procedures were as following:

a) Balanced the weight of sample inside polyethylene package "PE vial" before heat sealed .

b) Balanced the calculated weight of elemental standard (that expected nearly to same amount elemental in sample) inside PE vial before heat sealed .

c) Packed both vials of sample and standard in to polyethylene rabbit.

d) Irradiated sample pack with neutrons from nuclear reactor (as shown in figure 3-6) by loaded the sample pack into load tube, loading time which were depended on each elemental, after that let it in shielding (protected irradiation) before counting activity rate.

Loading time and decay period before counting activity rate.

element	Br	loading time	1 hr.	decay period	24 hrs.
element	Sb	loading time	1 hr.	decay period	24 hrs.
element	Sn	loading time	5 min.	decay period	10 min.
element	Zn	loading time	1 week	decay period	30 days

e) Took all PE vials of sample and standard from rabbit and put each vial on the top of detector to determining the count rate of each sample. Counting system diagram as shown in figure 3-7

f) Calculated percentage of element in sample as shown in equation 2.20.

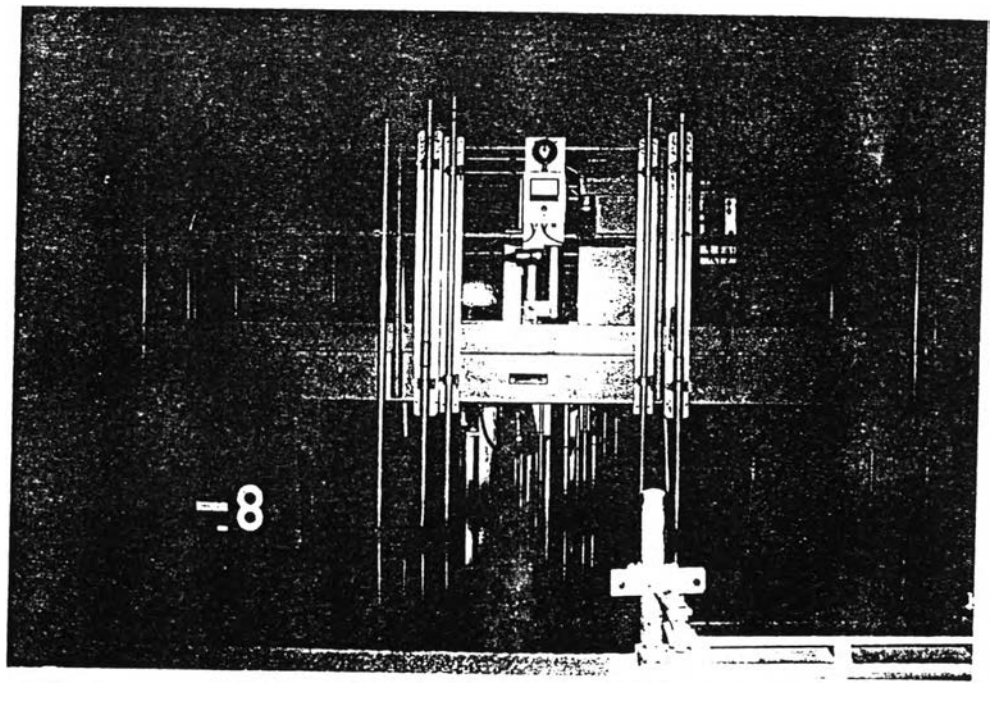
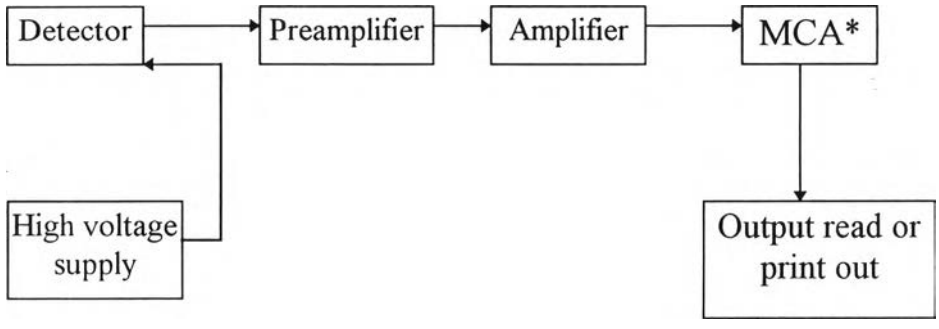


Figure 3-6 Nuclear reactor.



*MCA = Multichannal analyzer

Figure 3-7 Counting system diagram of neutron activation analysis

5. Thermal Analysis

To indicate the mechanism of flame retardation of additive on polypropylene, simultaneous thermogravimetry (TG), derivative thermogravi metry (DTG), of the flame-retarding polypropylene samples were carried out on a Netzsch STA 409 C instrument with a computer enhancement facility (CETA). The sample weight 50 mg, the heating rate was 10°C/min (from ambient temperature to 800°C) and the atmosphere was air, with a flow rate of 50 cm^3/min.