CHAPTER III

APPARATUS AND EXPERIMENTAL METHODS

Materials

1. Suspension PVC Resin

Suspension PVC Resin SG-610 K-Value 61

(Manufacturer : Thai Plastics and Chemical Co.,Ltd.)

2. Plasticizer

Dioctyl Phthalate ($C_{24}H_{38}O_4$)

Molecular Weight	390
Viscosity at 30°C	43 cps.
Specific Gravity at 20°C	0.986

(Manufacturer : TOA Plasticizer Industries Co.,Ltd.)

3. Heat Stabilizer

Ba-Cd-Zn Complex liquid stabilizer

Viscosity at 25°C	50 cps.
Specific Gravity at 20°C	1.03

(Manufacturer : Hacros Co.,Ltd.)

4. Co-stabilizer

Epoxydized Soyabean Oil

Viscosity at 20°C	400 cps.
Specific Gravity at 20°C	0.993

(Manufacturer : Akzo Chemical Co.,Ltd.)

5. Lubricant

Stearic acid

Molecular Weight	284.47
Melting Point	69.3 °C
Specific Gravity	0.847

(Manufacturer : Imperial Industrial Chemicals Co.,Ltd.)

6. Filler

Stearic acid coated $CaCO_3$

Bulk Density	1.20 g/cc.
Specific Gravity	2.70
Mean Particle Size	3.50 micron

(Manufacturer : Surin Omya Chemicals (Thailand) Co.,Ltd.)

7. Fire retardant

7.1 Zinc hydroxystannate , Flamtard H , $(ZnSn(OH)_6)$

7.2 Zinc stannate , Flamtard S , $(ZnSnO_3)$

(Manufacturer : Alcan Chemicals)

Table 3-1 Properties of zinc hydroxystannate and zinc stannate (3)

Properties	Flamtard H	Flamtard S
Decomposition temperature (20°C)	200	> 300
Median particle size (micron)	< 2	1.7
Density (g/cm^3)	3.3	3.9
Whiteness	85	87

7.3 Antimony trioxide (Sb_2O_3)

(Manufacturer : Cookson Ceramics and Antimony Limited)

Table 3-2 Properties of antimony trioxide (2)

Item	Value	Unit
Total antimony content	99.4	%
Arsenic (As)	0.3	%
Cooper (Cu)	0.01	%
Iron (Fe)	0.0002	%
Lead (Pb)	0.08	%
Nickel (Ni)	0.001	%
Acidity (H_2SO_4)	0.08	%
Average particle size	1.25	micron

Apparatus

1. **Tension Testing**

   Tensile strength and elongation testing (JIS K-6301)

   Tear strength testing (JIS K-6301)

   Suga Universal Tester model AGS-500A

2. **Two-Roll Mill**

   Lab Tech Co.,Ltd. model LRM 150

3. **Mechanical Mixing Machine**

   Lab Tech Co.,Ltd. model LMX 5

4. **Flammability Testing**

   Flammability (ISO 3795)

5. **Thermal Gravimetric Analysis (TGA)**

   Perkin-Elmer Co.,Ltd. model TGA7

6. **Neutron Activation Analysis (NAA)**

   Canberra Co.,Ltd. model S100

Sample Preparation

1. Blending PVC Ingredients

Blending is the first step in preparing a PVC compound for testing. The amount of ingredients required to blend can be calculated on part by weight of ingredient per 100 parts by weight of total PVC resin.

A plasticized formulation can be blended at ambient temperature using a mixing procedure as follow:

1.1 Lubricant and plasticizer were mixed in a beaker using gentle heating and agitation. The mixture was cooled to room temperature and any other liquid ingredients were added.

1.2 All solids (except the lubricant) were added to the mixing bowl.

1.3 Mixed at slow speed for 1 minute.

1.4 Plasticizer mixed from step 1.1 was added slowly to the bowl while continuing to mix at low speed.

1.5 Continued mixing for 1 minute after all the liquid from step (a) had been added.

1.6 Used a portion of the mix from step 1.5 to absorb the residual liquid from the beaker; then, added this back in the mixing bowl with the aid of a rubber spatula.

1.7 Cleaned the mixing bowl and the mixing blade with a rubber spatula in order to assure that all ingredients were incorporated into the mix.

1.8 Mixed at medium speed until a uniform mix was produced (typically 3-5 minutes).

2. Fluxing

Fluxing was carried out typically on a two-roll mill. The material was placed on the mill and allowed to preheat for 1-2 minutes prior to starting the roll turning. To aid mixing, the compound was cross cut back and forth using a mill knife. A batch should be mixed for minimum of 5 minutes after blending. Distance between the rolls (the roll nip) was adjusted to facilitate fluxing. Later, the nip was adjusted for removal of the batch at 0.20 mm. thickness of test sheets.

Recipes of Poly (vinyl chloride) Compounding

The samples of poly (vinyl cloride) were prepared as follows.

Table 3-3 Fire-retardance plasticized poly (vinyl chloride) film with and without fire retardant.

Ingredient	Formulation			
	NO FR.	Sb_2O_3 4 phc	ZHS 4 phc	ZS 4 phc
PVC K-61	100	100	100	100
Plasticizer	25	25	25	25
Co-stabilizer	2	2	2	2
Stabilizer	1	1	1	1
Lubricant	0.2	0.2	0.2	0.2
Filler	10	10	10	10
Fire retardant :				
Sb_2O_3	-	5.53	-	-
ZHS	-	-	5.53	-
ZS	-	-	-	5.53

NO FR. = Standard plasticized PVC film without fire retardant.

phc. = Part per hundred of compound.

Measurement

1. Mechanical properties testing (9)

Tensile strength and elongation testing

Each of the five test pieces conformed to the dimension shown in Figure 3-1 was taken from the test specimen in the longitudinal and lateral directions and will be provided on the center with two bench marks with a distance of 40 mm. The test piece was then mounted on tension tester as shown in Figure 3-2 and pulled at a speed of 200 mm/min. with the distance between tester jaws set to 90 mm. When the tester indicated a maximum load, the load applied and the distance between the bench marks should be obtained. The tensile strength was expressed by the load found in N/cm^2 when a maximum load was marked. The tensile strength and elongation at break were calculated from the following formula.

Tensile strength = F/A

Where, F = maximum load (Kgf)

A = cross-section area of test piece (cm^2)

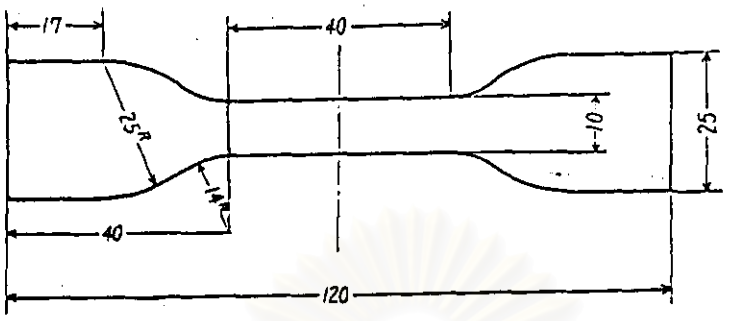
Elongation (%) = $[(L-L_o)/L_o] \times 100$

Where, L = distance between bench marks at the point of rupture (mm)

L_o = original distance between bench marks

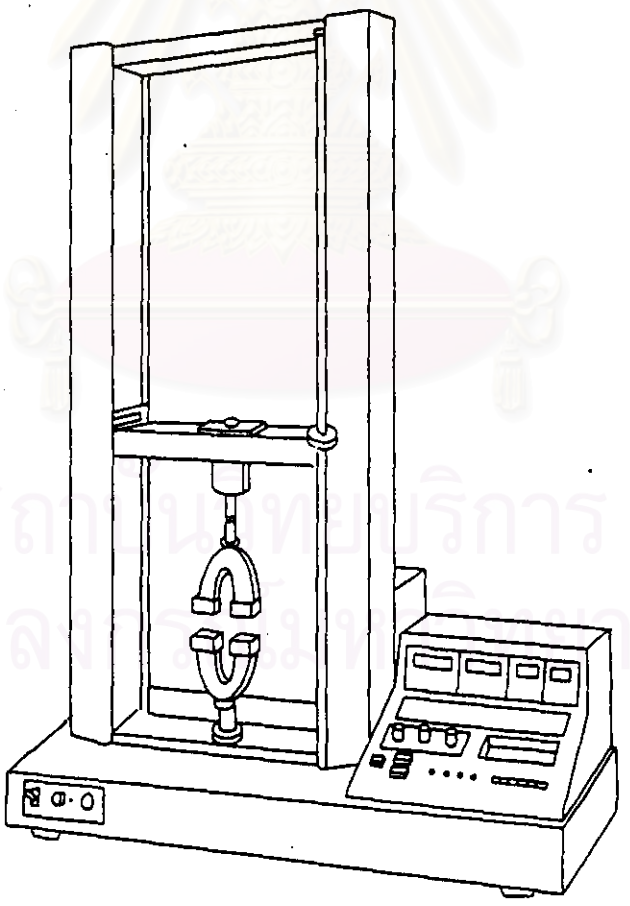
The test results were expressed in an average value of each five test pieces taken in the longitudinal and lateral directions. When the test piece was cut off at a point other than the area between the bench marks, the measured value should not be empolyed and another test piece should be prepared for retesting.

Figure 3-1 Tension test specimen dimension



Unit: mm

Figure 3-2 Universal tester load frame, set up for tensile testing of plastics

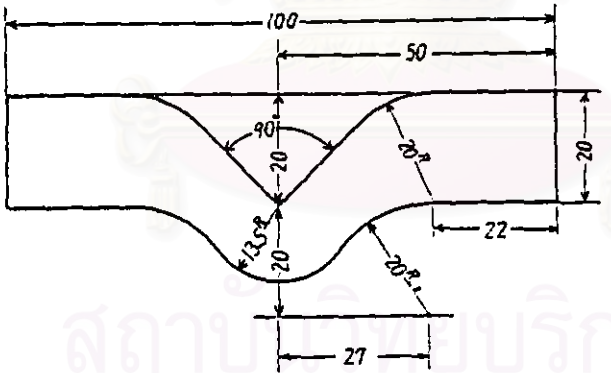


Tearing Strength Testing

Each five test pieces, dimension as shown in Figure 3-3, shall be taken in the longitudinal and lateral directions clamped by the tester jaws so that the test piece shall then be torn at a tension speed of 200 mm/min. The test result shall be expressed in an average value, by kgf(N), of each five test pieces in the longitudinal and lateral directions. When a constant speed elongation type tension tester with an automatic recorder is used, an average value of the maximum.

Figure 3-3 Dimension of tear test specimen

Unit: mm



2. Flammability Testing

Flammability Testing (8)

For the purpose of this test, test specimens should be conditioned by being left at a temperature of 23±2°C and at a relative humidity of 50±5% for a period not less than 24 hours and not more than 7 days. The tests were conducted in a metal box as shown in Figure 3-4 in a laboratory that temperature and relative humidity were kept at mentioned values.

The box was provided with an access window of a heat resistant glass plate in the front, and, a door that allow two U-shape metal plates (herein after referred to as U-shape Clamp) as shown in Figure 3-5 to enter the box, and a hole for passing a hose from a gas burner, on the side. The box also be provided with four slits 15 mm in width on the upper part of all sides and the bottom plate should have ten gores 19 mm in diameter. The box was fitted with legs and the bottom part should be raised by 10 mm.

A test piece 100 mm in width , 356 mm in length , and as shown in Figure 3-6 was chucked in the U-shape Clamp. Where the burning end of the test piece was softened and deflected, a 0.25 mm heat resistant wire was passed over the U-shape Clamp spaced at an interval of 25 mm to support the test piece.

The U-shape Clamp was held horizontal in the center of the box and a Bunsen Burner was placed that the center of the nozzle of the burner was positioned about 19 mm lower the center of the opening of the test

piece. The Bunsen Burner was 9.5 mm inside diameter and the total calorific value of the gas for testing should be about 9,080 kcal/m^3 (38 MJ/m^3). The burner was held vertical and adjusted so as to generate a flame 38 mm in length and the air vent was kept closed.

A flame should be allowed to strike the test piece for 15 seconds and time from the time when the flame reaches the first measuring point to the time when the flame reaches the second measuring point should be measured. The burning rate should be calculated from the following formula. On starting the test the burning apparatus and U-shape Clamp should be kept at a temperature of 30^0C or lower.

Burning rate (mm/min.) = 60 X (D/T)

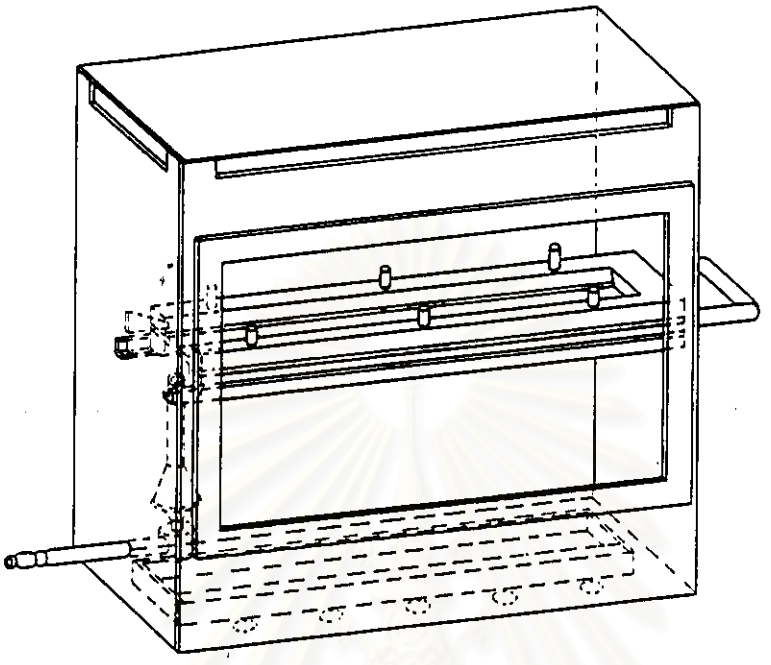
Where : D = burning distance (mm)

T = time required for burning over a distace of D (mm)

The test should be conducted under such conditions as to lead to the most disadvantageous results. If a flame goes out before reaching the second measuring point, time required for the flame to go out should be measured.

If a flame is started but it does not reach the first measuring point, the burning rate should be regarded as self-extinguished.

Figure 3-4 Example of combustion chamber



Dimensions in millimetres
Tolerances according to ISO 2768-1

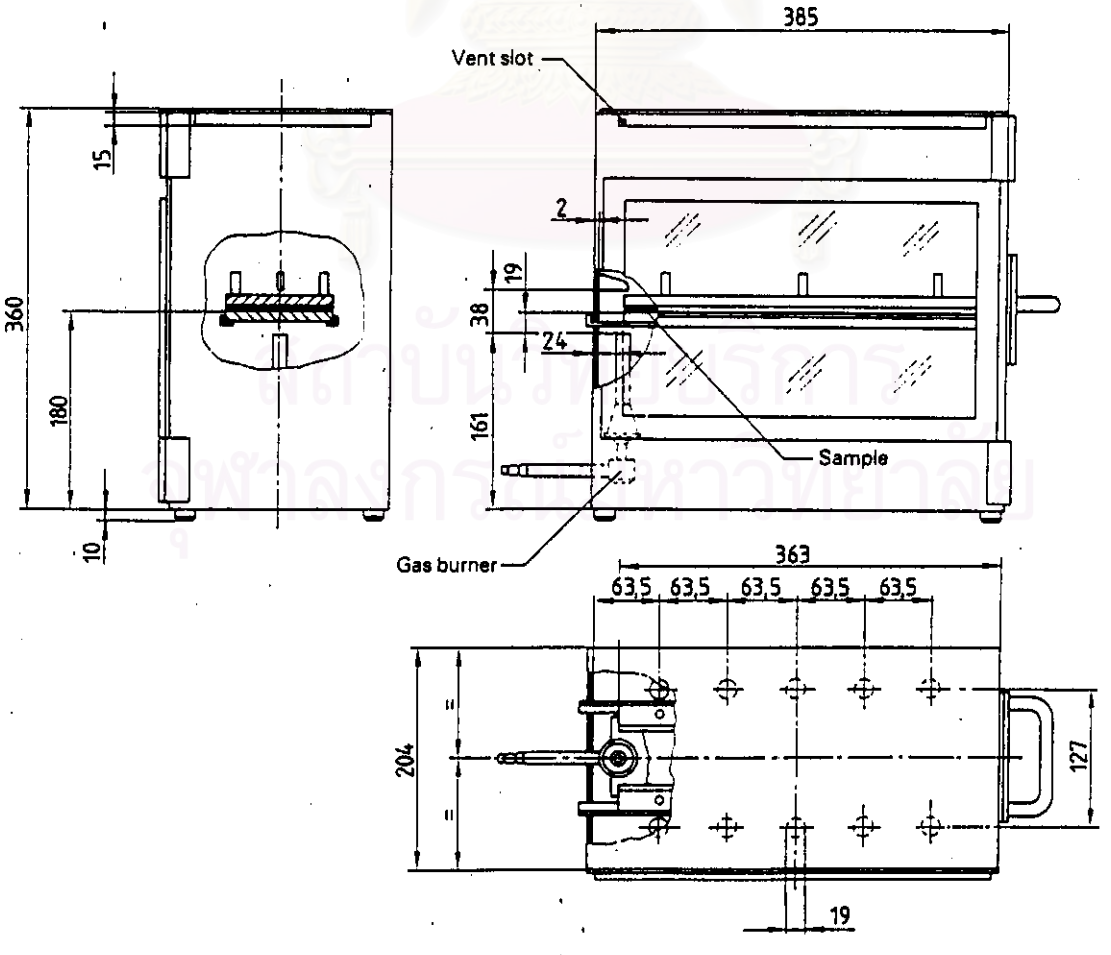


Figure 3-5 Example of U-shape Clamp

Dimensions in millimetres
Tolerances according to ISO 2768-1 if not otherwise indicated

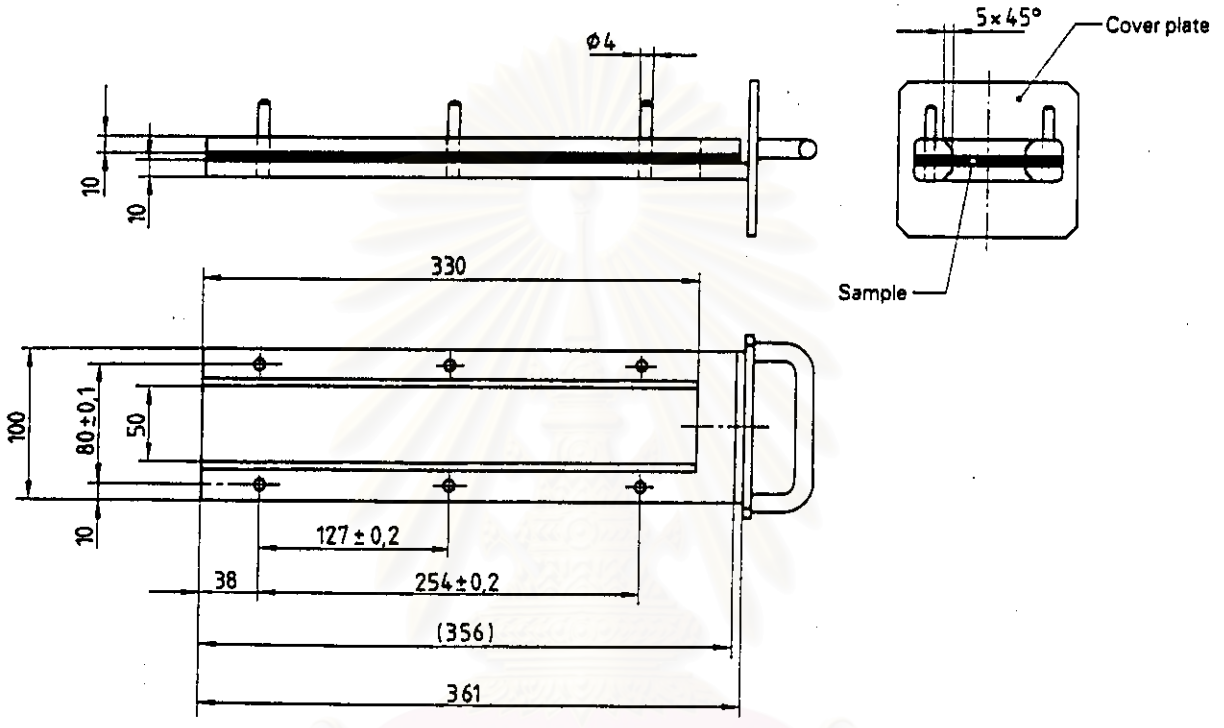


Figure 3-6 Test piece