CHAPTER IV

RESULTS AND DISCUSSIONS

1. Mechanical Properties Evaluation

The mechanical test of flexible PVC samples showed that; loading 4 phc of Sb_2O_3 , ZHS and ZS fire retardant had little or no significant effect on tensile strength, elongation at break and tear strength comparing to flexible PVC sample without fire retardant as shown in Table 4-1. Moreover, one can see the difference clearly when compare tensile strength on both mechanical direction (MD) and transverse direction (TD) of all samples in Fig. 4-1 and Fig. 4-2, elongation at break on both MD and TD direction in Fig. 4-3 and Fig. 4-4, and tear strength on both MD and TD direction in Fig. 4-5 and Fig. 4-6.

Table 4-1 Mechanical properties of flexible PVC samples.

Item		Unit	Sample			
			NO FR.	Sb_2O_3 4 phc	ZHS 4 phc	ZS 4 phc
Tensile Strength	MD	N/cm^2	300	280	290	280
	TD	N/cm^2	270	260	260	270
Elongation	MD	%	500	495	490	480
	TD	%	480	470	460	470
Tear Strength	MD	N	26.5	24.5	26.5	24.5
	TD	N	24.5	24.5	25.5	23.5

Fig. 4-1 Effect of 4 phc of Sb_2O_3 , ZHS and ZS on Tensile Strength in mechanical direction of samples.

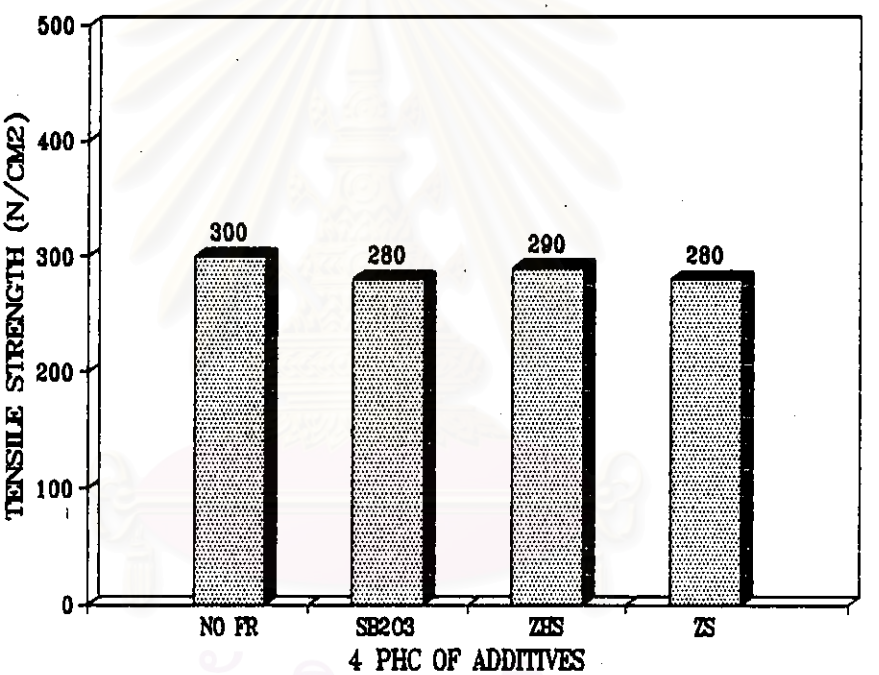


Fig. 4-2 Effect of 4 phc of Sb_2O_3 , ZHS and ZS on Tensile Strength in transverse direction of samples.

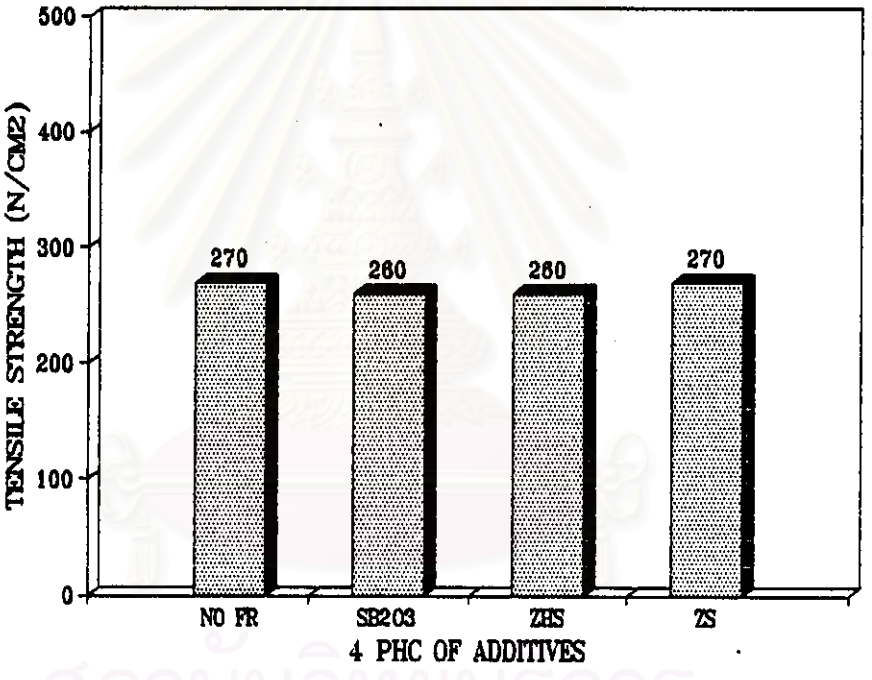


Fig. 4-3 Effect of 4 phc of Sb_2O_3 , ZHS and ZS on Elongation in mechanical direction of samples.

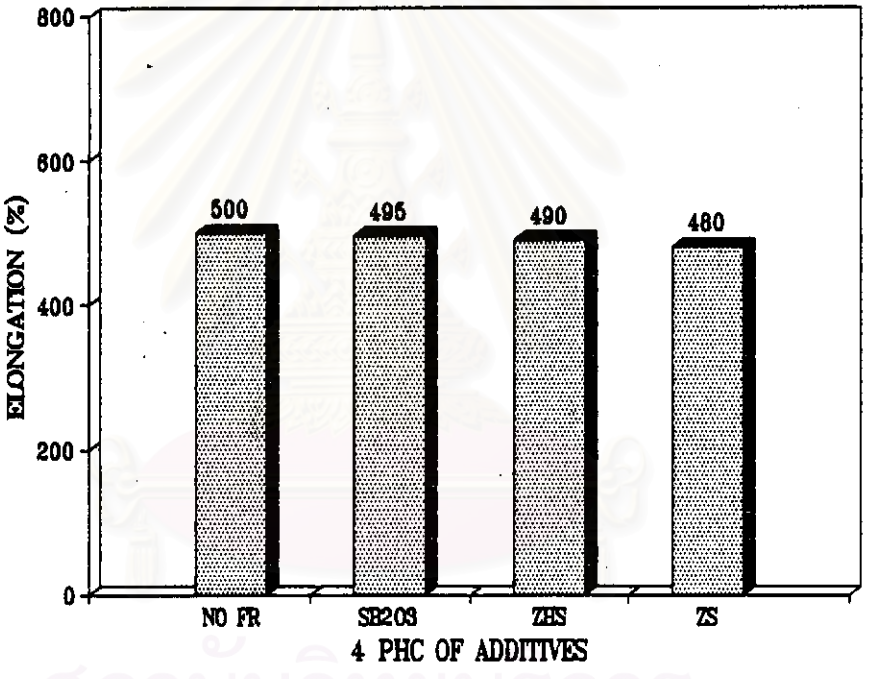


Fig. 4-4 Effect of 4 phc of Sb_2O_3 , ZHS and ZS on Elongation in transverse direction of samples.

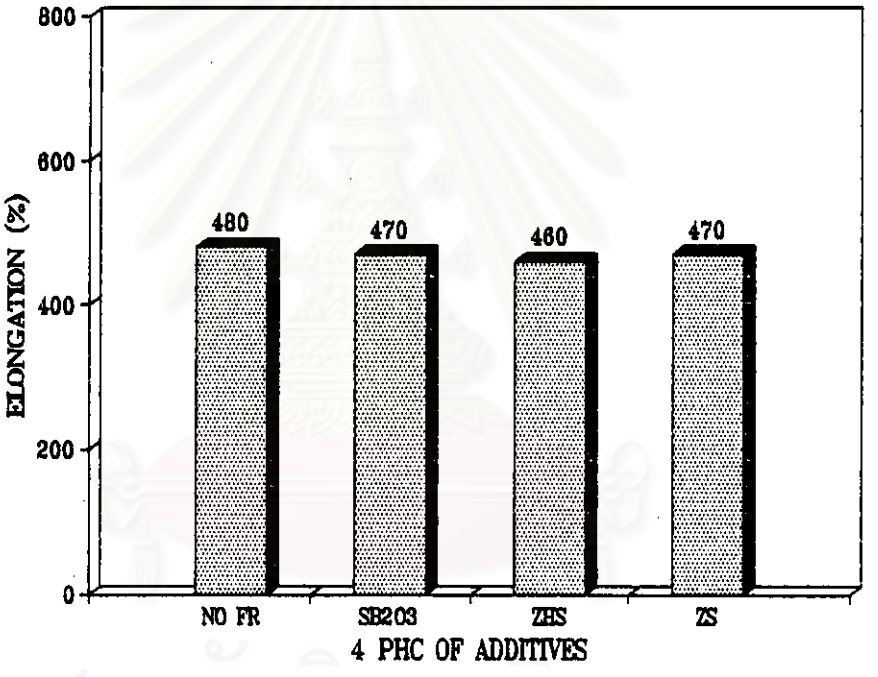


Fig. 4-5 Effect of 4 phc of Sb_2O_3 , ZHS and ZS on Tear Strength in mechanical direction of samples.

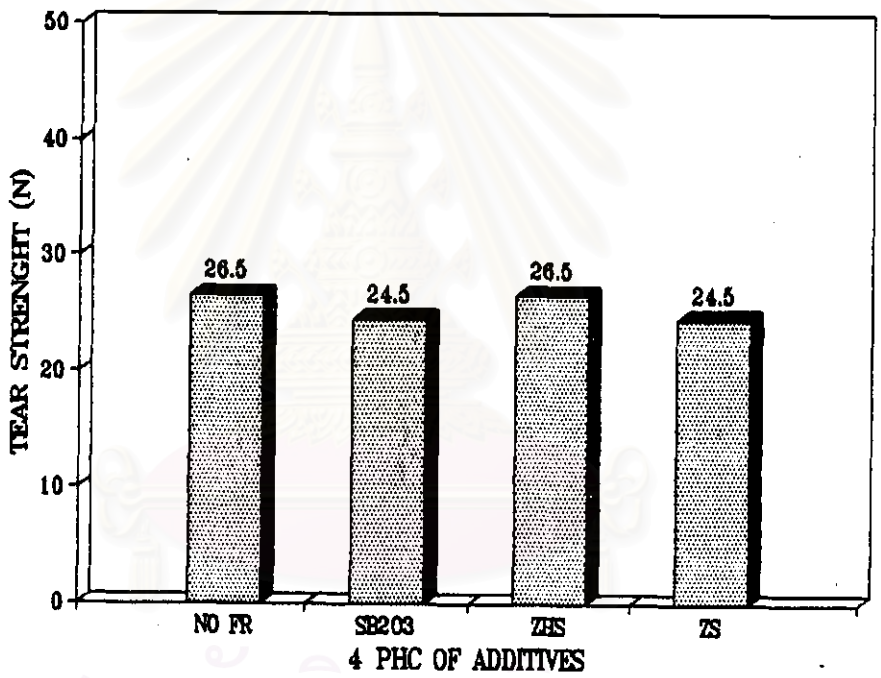
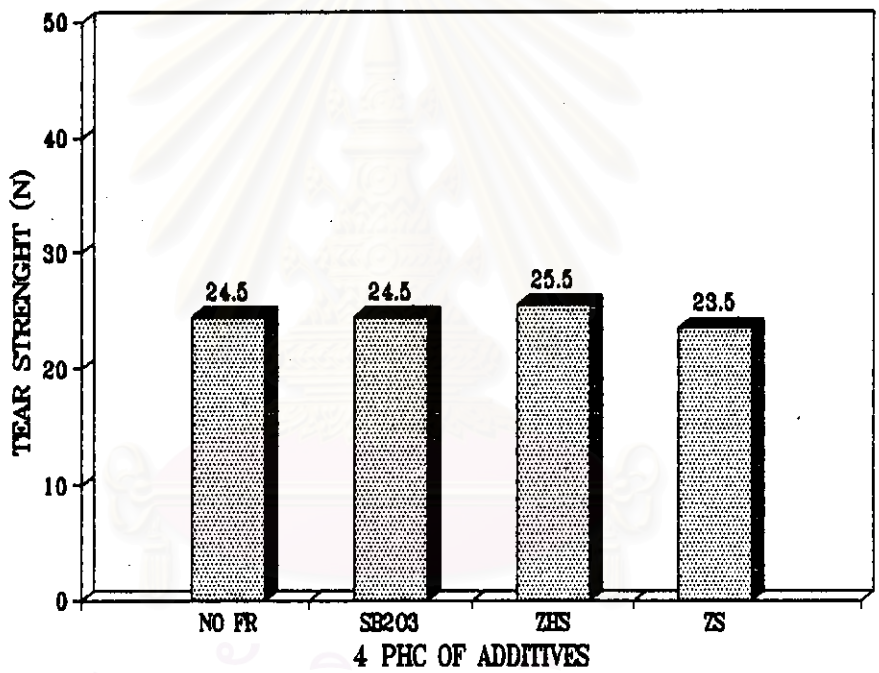


Fig. 4-6 Effect of 4 phc of Sb_2O_3 , ZHS and ZS on Tear Strength in transverse direction of samples.



2. Flammability Evaluation

The burning rate is defined as the horizontal burning rate of material used in occupant compartment of road vehicles, and of tractors and small machinery for agriculture and forestry, after exposure to a small flame in the fixed time. This testing can illustrate that adding 4 phc of Sb_2O_3, ZHS and ZS fire retardant in flexible PVC samples resulted in self-extinguished PVC. On the other hand, flexible PVC sample without fire retardant has the burning rate of 145 mm/min.

Table 4-2 Burning rate of flexible PVC samples

Sample	Burning Rate (mm./min.)
NO FR.	145
Sb_2O_3 4 phc	Selx
ZHS 4 phc	Selx
ZS 4 phc	Selx

Selx = Self-extinguished

3. Thermal Gravimetry Analysis (TGA)

Thermal gravimetric analysis diagrams of flexible PVC samples without fire retardant and with Sb_2O_3 , ZHS and ZS fire retardants 4 phc was illustrate in Fig. 4-7 to Fig. 4-10. As this trial set temperature range at 50°C - 900°C and from all thermal gravimetric analysis diagrams, it could be separate this temperature range into 2 stages. First stage was called initial degradation stage (100°C - 400°C) and second stage was called char oxidation stage (400°C - 900°C)

Flexible PVC sample without fire retardant and with Sb_2O_3 fire retardant 4 phc had nearly the same pattern for weight loss (%) and TGA max (°C) in both initial degradation stage and char oxidation stage. As we known the mechanism of Sb_2O_3 in flexible PVC was act in gas phase, so nearly all of Sb_2O_3 must lost from substrate between heat up 50°C - 900°C. While, flexible PVC samples adding with 4 phc of ZHS and ZS fire retardant also had diagram of TGA in the same pattern. The initial degradation stage occurring at a lower temperature than that of the untreated flexible PVC sample and treated with 4 phc of Sb_2O_3 , which may be indicative of promotion of chloride volatilisation by the ZHS and ZS. Furthermore, the amount of residue burnt off in the char oxidation step was increased in the tin-containing samples, and the temperature at which this process occured significantly higher and broader than in the base resin and incorporated with Sb_2O_3 fire retardant 4 phc as shown in Table 4-3. Same as the thermal gravimetric analysis result of brominated

polyester samples without fire retardant and with Sb_2O_3 , ZHS and ZS as fire retardant which have tested by Cusack and Killmeyer (11).

Fig. 4-7 Thermal gravimetric analysis diagram of flexible PVC sample without fire retardant.

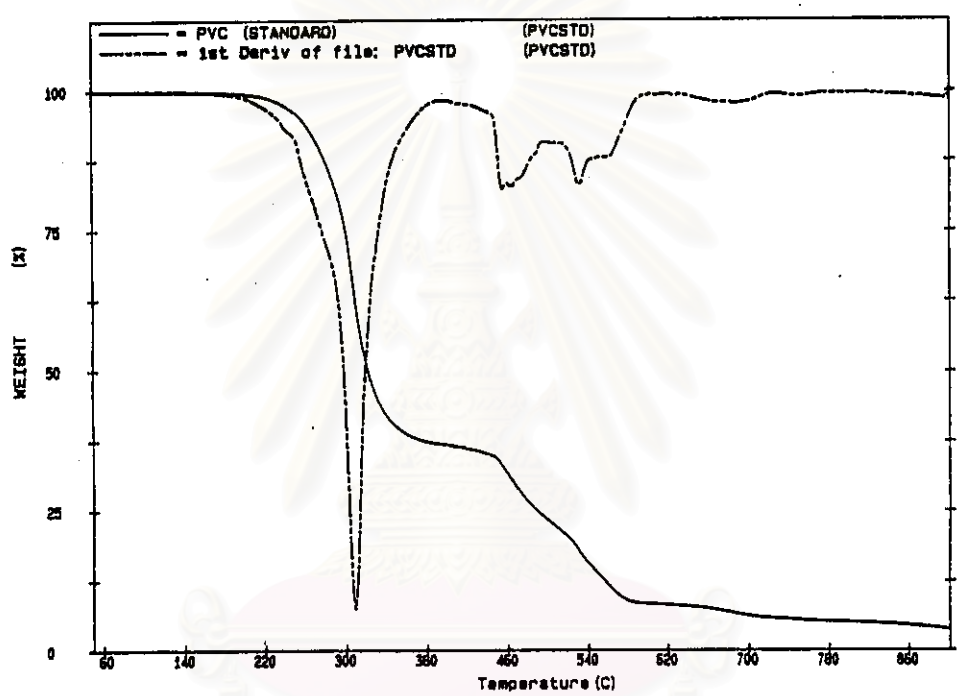


Fig. 4-8 Thermal gravimetric analysis diagram of flexible PVC sample with 4 phc Sb_2O_3 fire retardant.

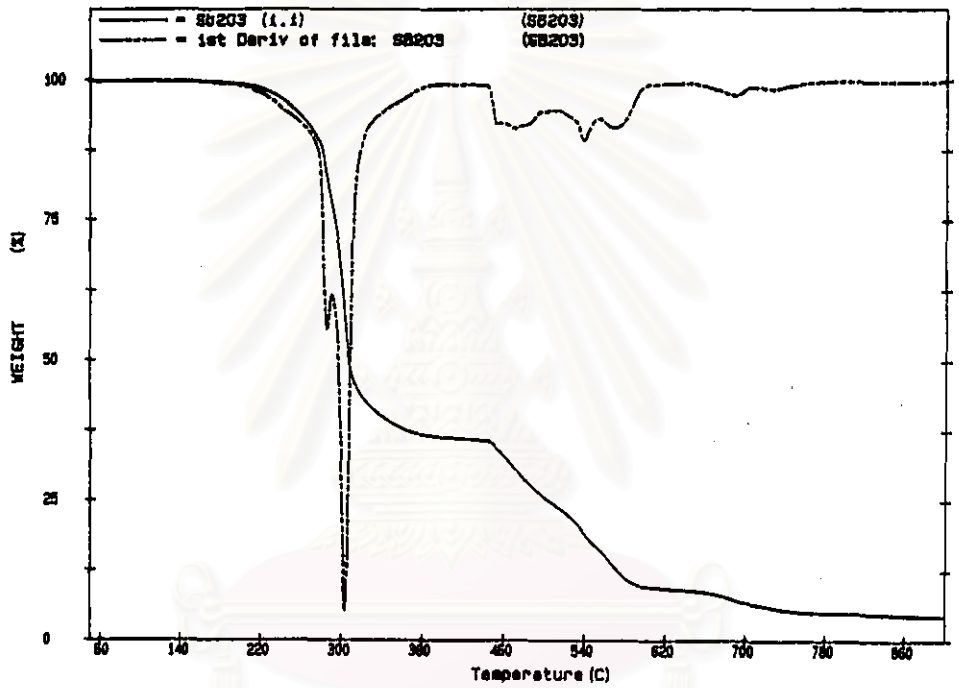


Fig. 4-9 Thermal gravimetric analysis diagram of flexible PVC sample with 4 phc ZHS fire retardant.

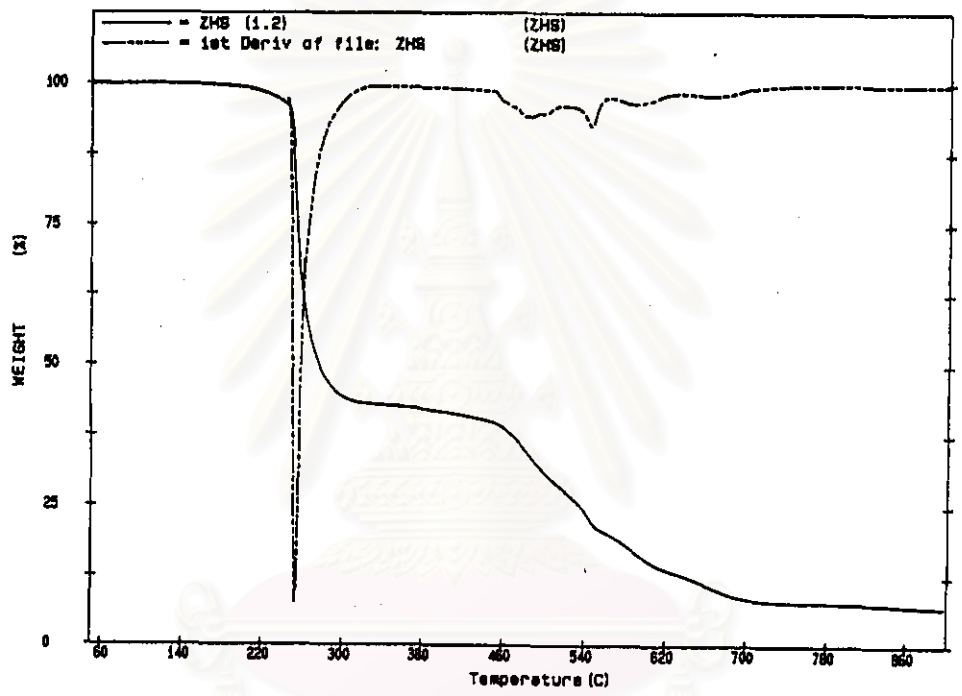


Fig. 4-10 Thermal gravimetric analysis diagram of flexible PVC sample with 4 phc ZS fire retardant.

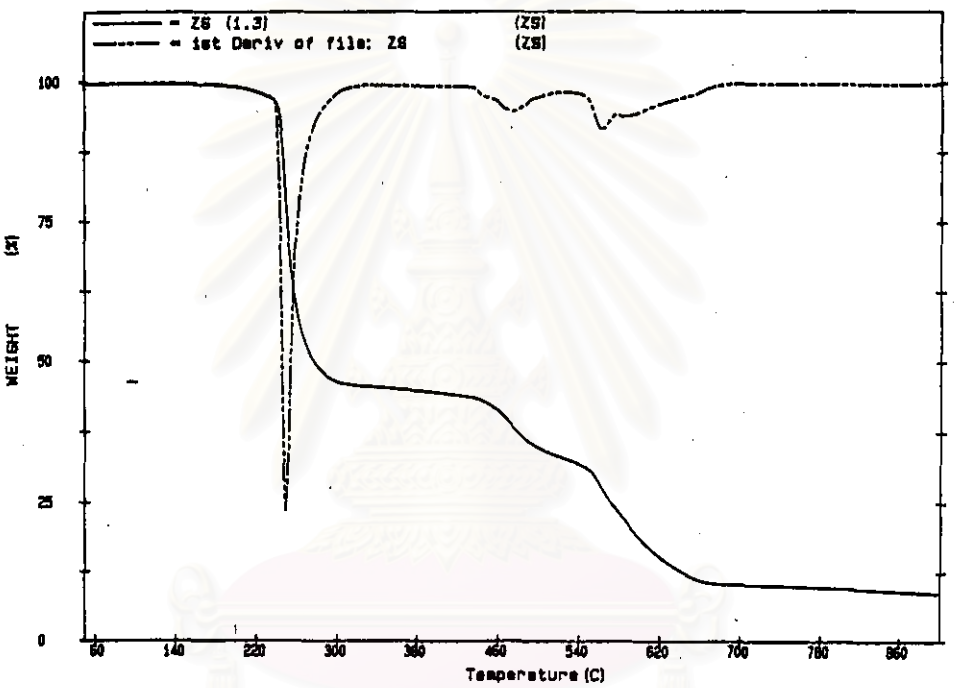


Table 4-3 Thermal gravimetric analysis data for flexible PVC samples.*

Sample	Initial Degradation+		Char Oxidation++	
	Weight Loss (%)	TGA Max. (°C)	Weight Loss (%)	TGA Max. range (°C)
NO FR.	63.1	310	32.7	460 - 580
Sb_2O_3 4 phc	63.4	310	31.9	460 - 590
ZHS 4 phc	57.3	255	35.6	460 - 665
ZS 4 phc	54.7	250	36.3	450 - 660

* Experimental condition : Atmosphere - air
Flow rate - 20 cm^3/min.
Heating rate - 10°C/min.

+ Temperature range : ca. 100 - 400°C

++ Temperature range : ca. 400 - 900°C

4. Evolved Neutron Activation Analysis (NAA)

Insight into the mode of action of the individual additives was provided by simple combustion experiments, carried out in air. This testing shown that the yield of involatile carbonaceous char, formed when flexible PVC was burned to completion, was nearly to doubled when adding with 4 phc of ZHS and ZS fire retardants, this observation being consistent with condensed phase behaviour. While flexible PVC with Sb_2O_3 fire retardant 4 phc has the yield of involatile carbonaceous char a little higher than base resin, this observation being consistent with vapor phase behaviour.

Neutron activation analysis (NAA) is a common analysis method to consider chemical elements in trace quantities analysis. From neutron activation analysis diagram as illustrate in Fig. 4-11 to Fig. 4-18 the elemental volatilisation (%) can be calculated by comparing the count rate of a standard with that of the sample and refer to residual char yield. Elemental analysis of the residues suggested that, a very significant proportion of both the zinc and the tin was volatilised from the zinc hydroxystannate and zinc stannate-containing polymer, which may be indicative of vapor phase action. Antimony trioxide, which undergoes almost complete volatilisation in the polymer, shown little char enchancing behaviour and operated primarily in the vapor phase. The apparent ability of zinc hydroxystannate and zinc stannate to act in both the condensed and

vapor phases. Residual char yields and extents of elemental volatilisation from flexible PVC samples during combustion in air as shown in Table 4-4 has result same style as Cusack and Killmeyer (11) tested in brominated polyester samples during combustion in air.

Fig. 4-11 Neutron Activation Analysis diagram of standard Sb.

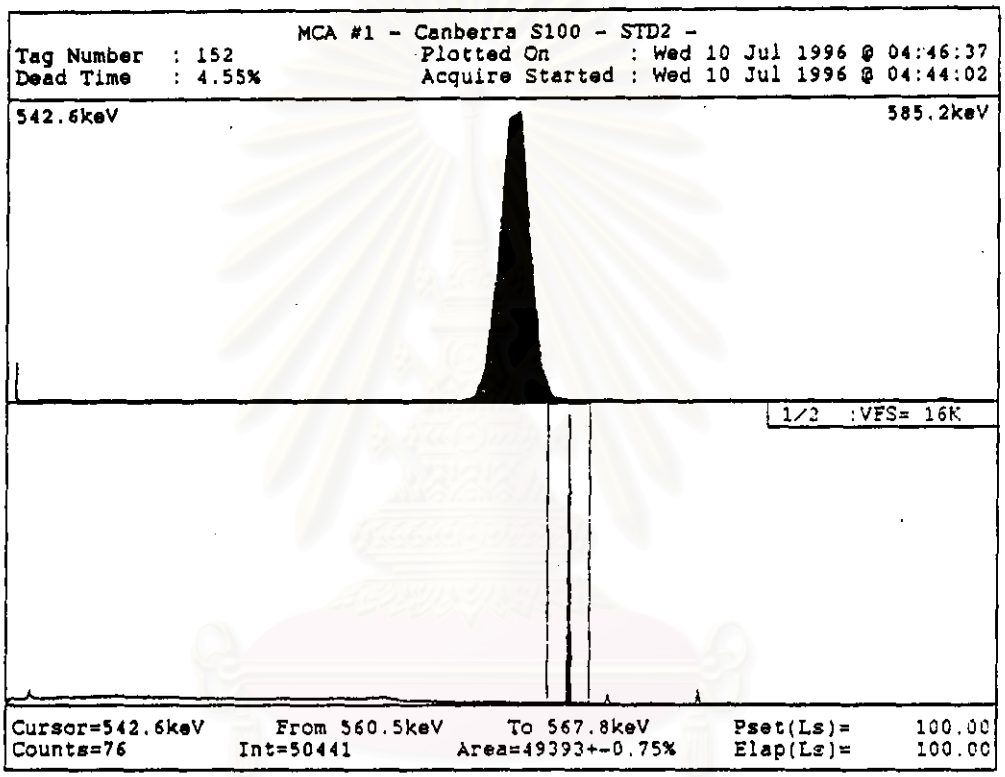


Fig. 4-12 Neutron Activation Analysis diagram of Sb from flexible PVC sample with 4 phc Sb_2O_3 fire retardant.

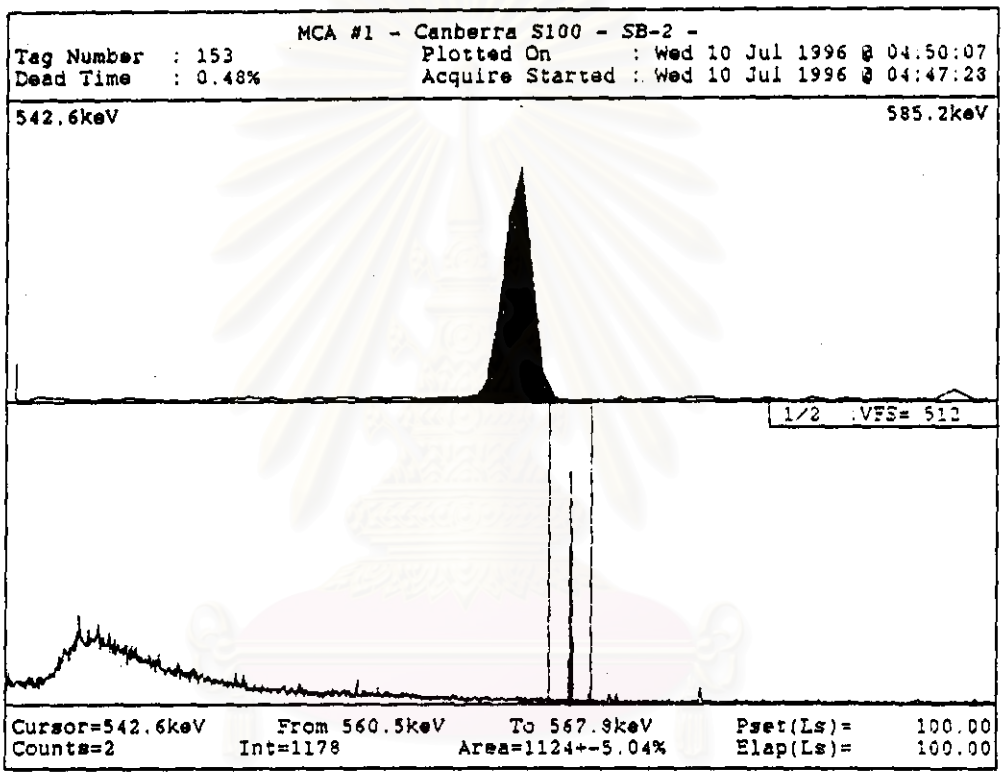


Fig. 4-13 Neutron Activation Analysis diagram of standard Sn.

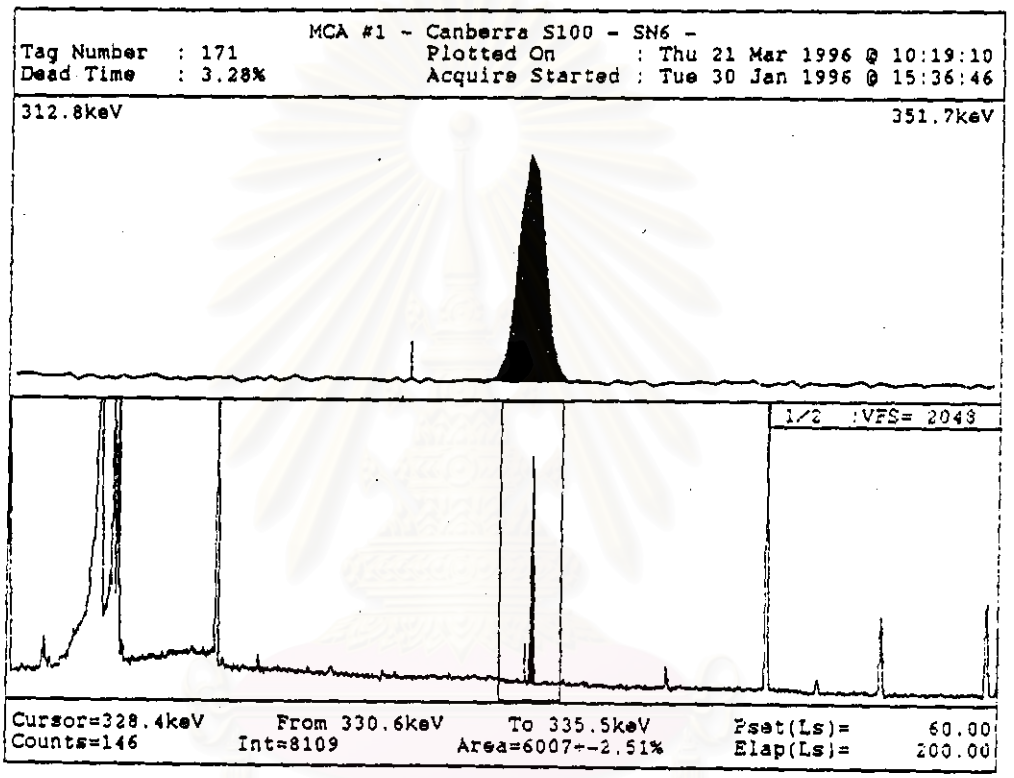


Fig. 4-14 Neutron Activation Analysis diagram of Sn from flexible PVC sample with 4 phc ZHS fire retardant.

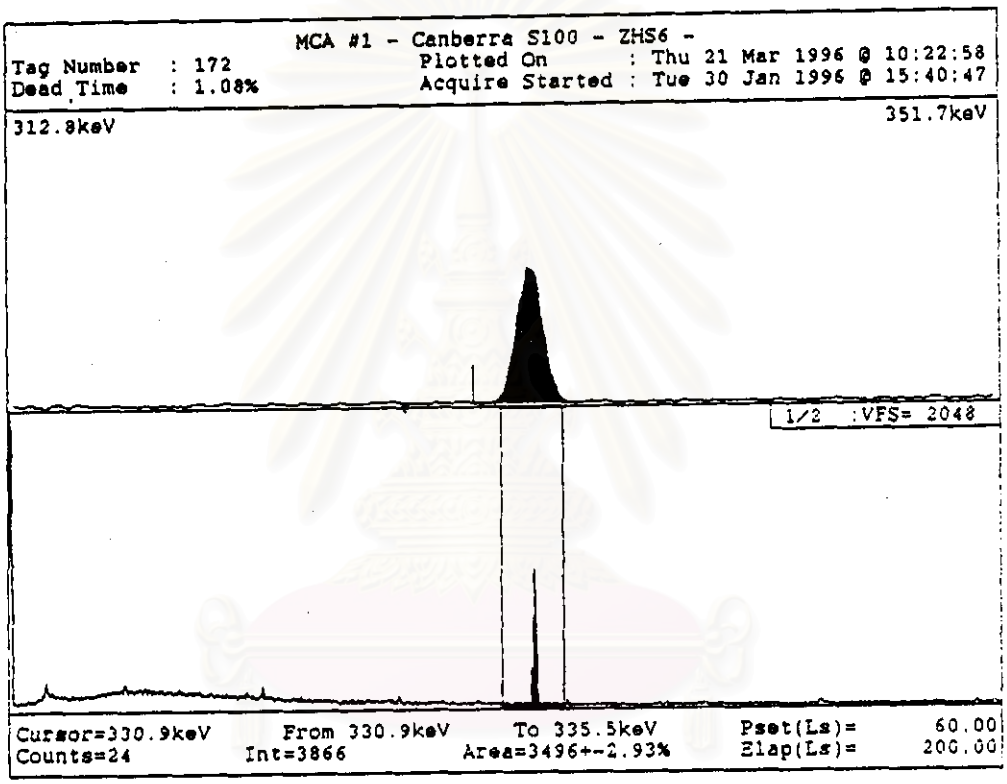


Fig. 4-15 Neutron Activation Analysis diagram of Sn from flexible PVC sample with 4 phc ZS fire retardant.

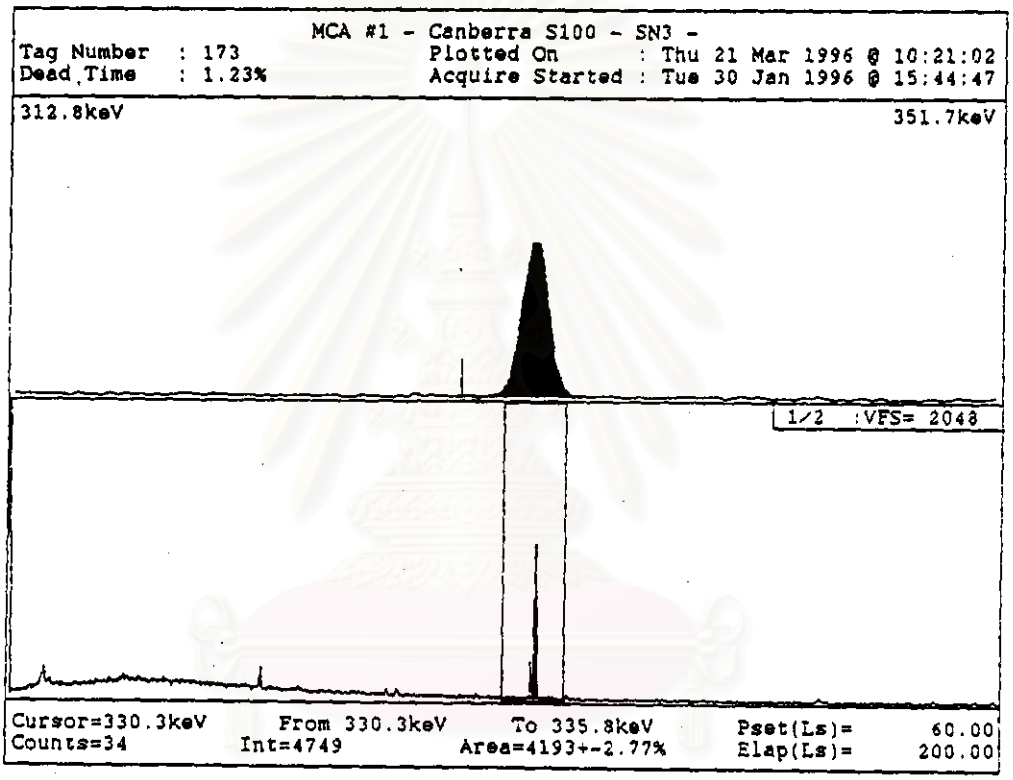


Fig. 4-16 Neutron Activation Analysis diagram of standard Zn .

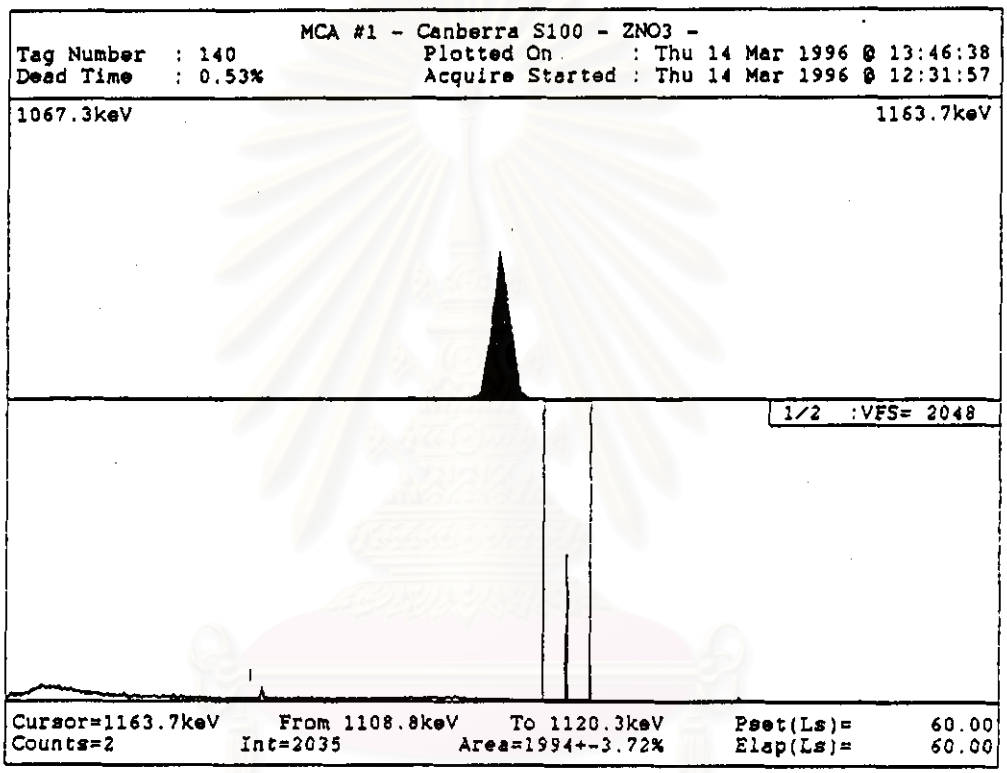


Fig. 4-17 Neutron Activation Analysis diagram of Zn from flexible PVC sample with 4 phc ZHS fire retardant.

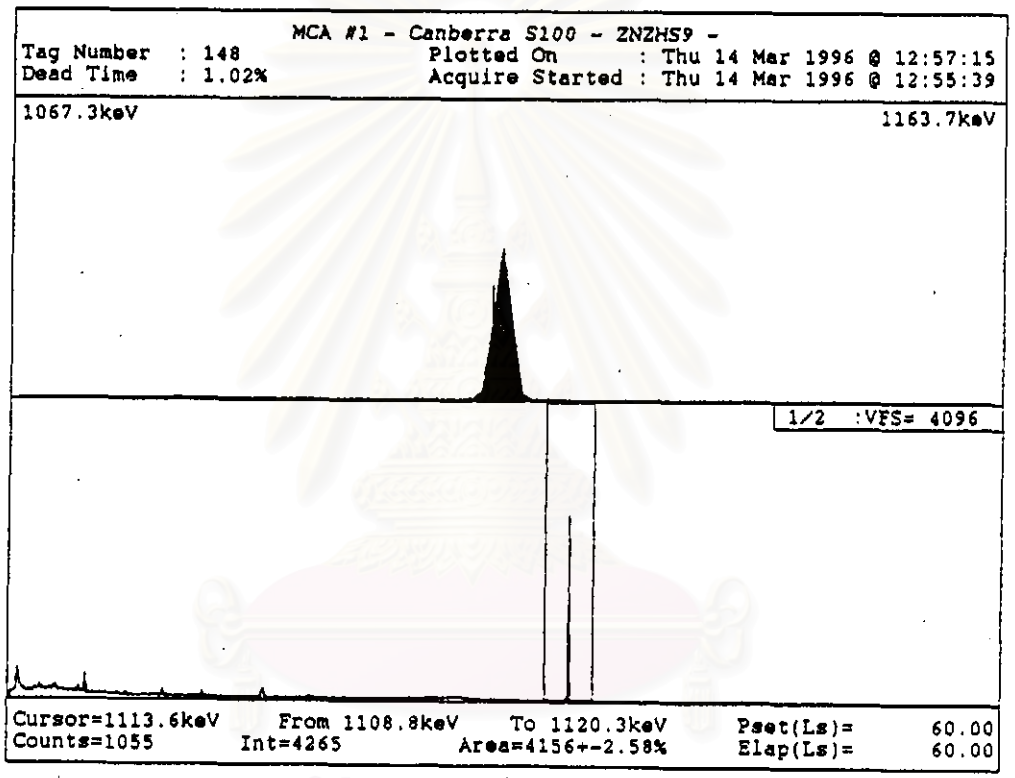


Fig. 4-18 Neutron Activation Analysis diagram of Zn from flexible PVC sample with 4 phc ZS fire retardant.

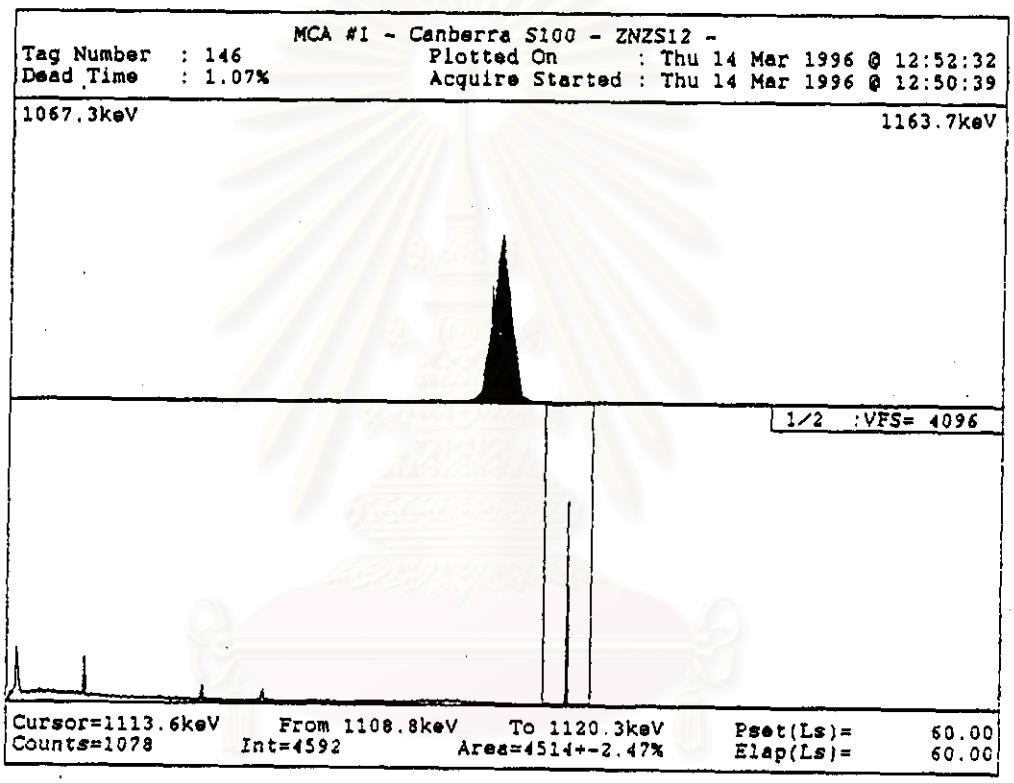


Table 4-4 Residual char yields and extents of elemental volatilisation from flexible PVC samples during combusion in air.

Sample	Char Yield (%)	Elemental Volatilisation (%)			Primary Phase of Action *
		Sn	Zn	Sb	
NO FR.	9.38	-	-	-	-
Sb_2O_3 4 phc	10.75	-	-	99.52	vapor
ZHS 4 phc	18.33	90.93	63.95	-	condensed+vapor
ZS 4 phc	20.65	85.86	64.45	-	condensed+vapor

* With regard to metallic element ; chlorine itself acts almost exclusively in the vapor phase.