CHAPTER IV

RESULTS

4.1 Limited Oxygen Index (LOI) measurements

Limited Oxygen Index (LOI) measurements were carried out using equipment constructed according to ASTM D 2863-87 standard method for flammability testing of plastics. In each case , ten samples were examined and the mean value of LOI was recorded.

The LOI results for the metal stannates incorporated into the polyurethane panels at levels of 0.5, 1.0, 2.0, 5.0 and 10.0 % pbw. were recorded in Table 4.2- Table 4.4. Table 4.1 indicated the amount of fire retardants which the polyurethanes were self-extinguished.

Significant relationships had been found between metal stannate and decabromodiphenyl ether (DBDPE). The combination of DBDPE with zinc stannate and zinc hydroxystannate at levels 1% and 5% pbw. showed an effect superior to the individual capacity of each, or synergism. Table 4-6 and Table 4-7 indicated the effect of synergism of metal stannate on polyurethanes containing DBDPE at level 2%, 5%, 10% and 20% pbw. respectively. For ammonium sulphate, the synergistic relationship was clearly observed at 2%, 5%, 10% and 15% pbw. of ammonium sulphate (AS) and metal

stannate at level 1% and 5% pbw.. The effect of synergism of ZHS and ZS were shown in Table 4-8 and Table 4-9.

Table 4-1 The amount of fire retardants that polyurethane were self-extinguished.

Fire retardant	Amount, % pbw.
Zinc hydroxy stannate	10
Zinc stannate	20
Ammonium sulphate (granular)	32
Ammonium sulphate (fine)	23
Melamine	23
Zinc Stannate :	2
Ammonium sulphate	10
Zinc hydroxy stannate :	2
Ammonium sulphate	10
Zinc hydroxy stannate :	2 **
Melamine	20
Ammonium sulphate (fine) :	11
Melamine	11

** could not self-extinguish

Table 4-2(a) Effect of ZHS, ZS and HDR as fire retardants in flexible polyurethane foams.

Fire retardant	LOI		
	ZHS	ZS	HDR
No additive	18	18	18
0.5% additive	20	19	18
1.0% additive	20	19	18
2.0% additive	20	19	18
5.0% additive	21	19	20
10.0% additive	23	20	20

Table 4-2(b) Effect of ZHS, ZS and HDR as fire retardants in rigid polyurethane foams.

Fire retardant	LOI		
	ZHS	ZS	HDR
No additive	18	18	18
0.5% additive	19	19	18
1.0% additive	19	20	19
2.0% additive	19	20	20
5.0% additive	19	20	20
10.0% additive	23	20	20

Table 4-3 LOI result of ammonium sulphate and DBDPE

Fire retardant	LOI
2% AS	18
5% AS	24
10% AS	27
15% AS	29
2% DBDPE	18
5% DBDPE	23
10% DBDPE	30
20% DBDPE	42

Table 4-4 Effect of ZHS and ZS as synergistic compounds of DBDPE additives.

Fire retardant	LOI	
	ZHS	ZS
No additives	18	18
1.0% additive + 2.0% DBDPE	18	18
5.0% DBDPE	22	22
10.0% DBDPE	28	28
20.0% DBDPE	42	42
5.0% additive + 2.0% DBDPE	23	20
5.0% DBDPE	25	26
10.0% DBDPE	31	30
20.0% DBDPE	48	47

Table 4-5 Effect of ZHS and ZS as synergistic compounds of AS additives.

Fire retardant	LOI	
	ZHS	ZS
No additives	18	18
1.0% additive + 2.0% AS	18	18
5.0% AS	22	22
10.0% AS	28	28
15.0% AS	42	42
5.0% additive + 2.0% AS	23	20
5.0% AS	25	26
10.0% AS	31	30
15.0% AS	48	47

Table 4-6 Char formation studied of ZHS, ZS and ZHS, ZS plus AS-containing polyurethane and DBDPE containing polyurethane

Additives	% char
20% DBDPE	35.11
15% AS	44.48
10% ZS	35.60
10% ZHS	38.09
1% ZS + 15% AS	57.26
5% ZS + 15% AS	30.91
1% ZHS + 15% AS	38.17
5% ZHS + 15% AS	41.19
1% ZS + 20% DBDPE	37.28
5% ZHS + 20% DBDPE	31.71
1% ZS + 20% DBDPE	26.40
1% ZHS + 20% DBDPE	30.73

Table 4-7 Ignition of polyurethane samples

Sample	original metal in ash (ppm.)	ash (ppm.)
2% ZHS	131.19	82.48
10% ZHS	2024.35	1022.50
2% ZS	207.2	207.9
10% ZS	1945.74	1591.29

4.2 Mechaical property measurement

The mechanical properties, among all the properties of plastic materials, are often the most important properties because virtually all service conditions and the majority of end-use aplications involve some degree of mechanical loading. The results of mechanical properties of fire retardant polyurethane were shown below.

Table 4-8 Mechanical properties on flexible polyurethane foams containing ZHS, ZS and HDR.

Fire Retardant	Stress at break (N/mm^2)	Strain at break (%)	Modulus (N/mm^2)
No additives	1.788	22.7	7.038
0.5 % ZHS	1.264	27.6	3.951
1.0 % ZHS	1.441	29.5	4.192
2.0 % ZHS	1.175	25.8	4.033
5.0 % ZHS	1.361	26.3	4.639
10.0 % ZHS	1.236	25.0	4.826
0.5 % ZS	1.856	29.0	6.180
1.0 % ZS	1.506	28.1	4.759
2.0 % ZS	1.629	29.6	5.007
5.0 % ZS	1.559	26.9	5.347
10.0 % ZS	1.533	23.5	6.065
0.5 % HDR	1.400	30.8	4.019
1.0 % HDR	1.375	30.9	4.181
2.0 % HDR	1.354	28.7	4.359
5.0 % HDR	1.428	24.6	5.275
10.0 % HDR	1.763	28.3	6.218

Table 4-9 Mechanical properties on rigid polyurethane foams containing ZHS, ZS and HDR.

Fire Retardant	Stress at break (N/mm^2)	Strain at break (%)	Modulus (N/mm^2)
No additives	1.391	29.3	4.442
0.5 % ZHS	1.396	31.1	4.028
1.0 % ZHS	1.409	31.0	3.908
2.0 % ZHS	1.508	31.3	4.243
5.0 % ZHS	1.631	28.6	5.338
10.0 % ZHS	1.731	27.1	5.865
0.5 % ZS	1.462	26.2	5.090
1.0 % ZS	1.478	28.9	4.847
2.0 % ZS	1.638	29.3	5.149
5.0 % ZS	1.583	27.9	5.596
10.0 % ZS	1.787	26.7	6.879
0.5 % HDR	1.884	23.1	7.531
1.0 % HDR	1.708	20.8	7.720
2.0 % HDR	1.969	23.2	8.350
5.0 % HDR	1.748	17.4	10.130
10.0 % HDR	1.785	33.1	7.649

Table 4-10 Result of stress at break, strain at break and modulus polyurethane containing ZS and DBDPE

Fire retardant	Stress at break (N/mm^2)	Strain at break (%)	Modulus (N/mm^2)
No additives	1.788	22.7	7.038
1% ZHS + 2% DBDPE	0.803	27.67	2.881
5% DBDPE	0.986	24.70	4.159
10% DBDPE	1.235	29.74	4.346
20% DBDPE	0.860	23.82	3.869
5% ZHS + 2% DBDPE	1.402	28.94	4.217
5% DBDPE	0.950	52.77	3.259
10% DBDPE	1.104	26.22	4.408
20% DBDPE	0.866	28.61	3.314
1% ZS + 2% DBDPE	0.867	32.815	2.744
5% DBDPE	0.976	29.93	3.356
10% DBDPE	0.901	28.49	3.192
20% DBDPE	0.962	21.13	4.579
5% ZS + 2% DBDPE	0.924	26.02	3.755
5% DBDPE	1.198	26.70	4.472
10% DBDPE	1.033	28.45	3.752
20% DBDPE	0.910	29.40	3.820

Table 4-11 Result of stress at break, strain at break and modulus on poyurethane containing ZHS, ZS and AS

Fire retardant	Stress at break (N/mm^2)	Strain at break (%)	Modulus (N/mm^2)
No additive	1.788	22.7	7.038
1% ZHS + 2% AS	0.904	37.33	2.512
5% AS	0.899	33.38	2.674
10% AS	0.817	28.52	2.683
20% AS	1.044	29.18	3.624
5% ZHS + 2% AS	0.933	32.52	2.861
5% AS	1.386	28.06	4.939
10% AS	1.593	24.07	7.829
20% AS	1.676	23.10	8.017
1% ZS + 2% AS	1.023	30.99	2.554
5% AS	0.765	28.67	2.600
10% AS	0.980	34.94	2.851
20% AS	0.849	31.95	2.796
5% ZS + 2% AS	0.930	32.30	2.926
5% AS	0.927	30.48	2.974
10% AS	1.033	28.80	3.843
15% AS	0.879	27.16	3.586

Figure 4-1 Effect of zinc hydroxy stannate, zinc stannate and hydroromarchite as fire retardants in flexible polyurethane foams.

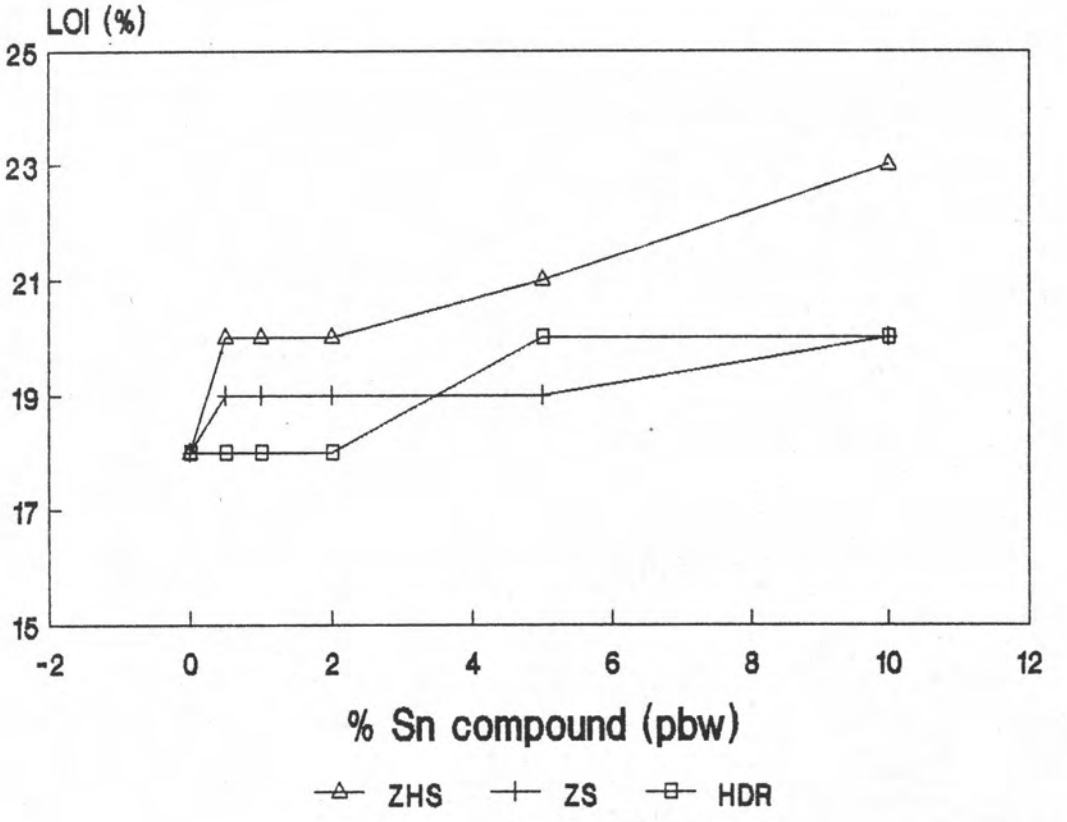

Figure 4-2 Effect of zinc hydroxy stannate, zinc stannate and hydroromarchite as fire retardant in rigid polyurethane foams.

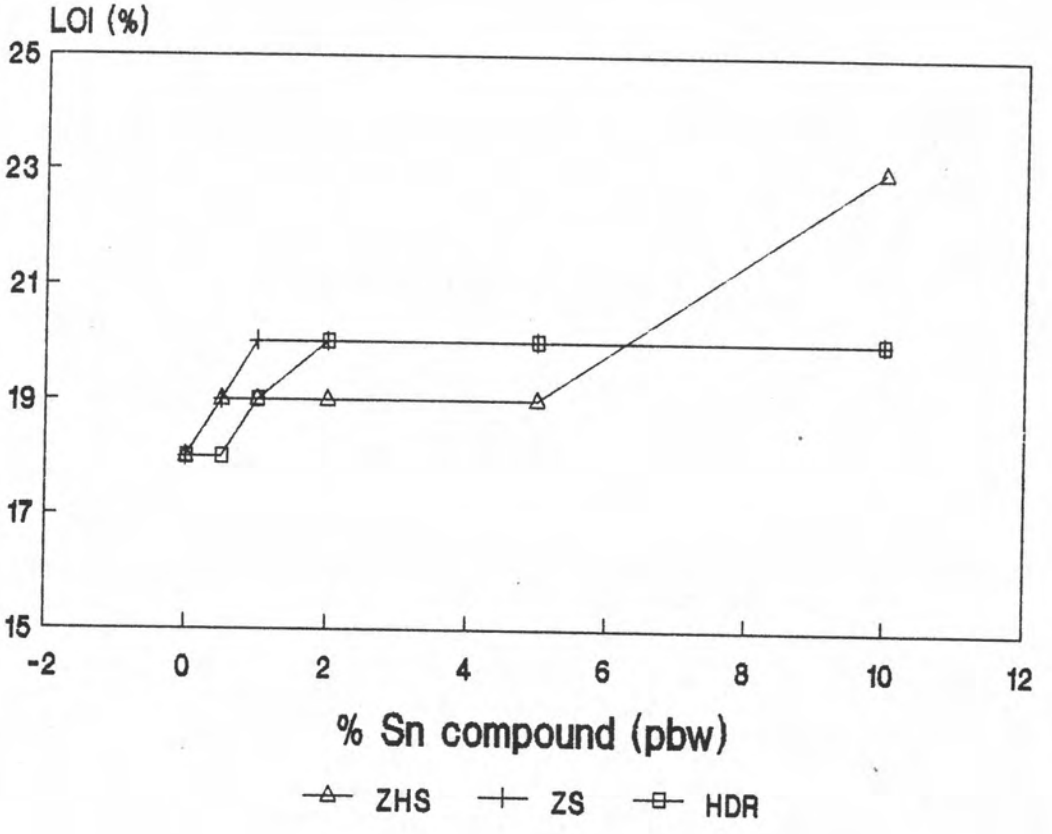

Figure 4-3 Effect of zinc hydroxystannate and zinc stannate on the flammability of flexible polyurethane 1% and 5% containing 2%, 5%, 10% and 20% DBDPE.

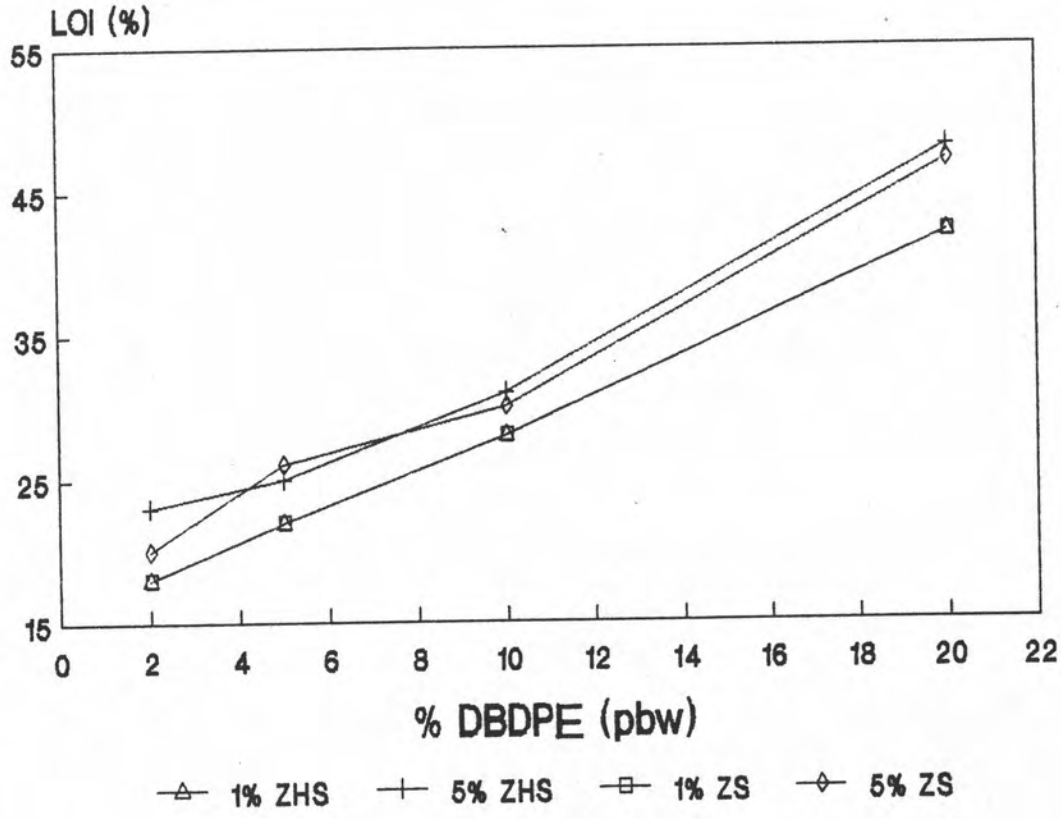

Figure 4-4 Effect of zinc hydroxystannate and zinc stannate at level 1% and 5% pbw.stannate on the flammability of flexible polyurethane containing 2%, 5%, 10% and 15% pbw. ammonium sulphate.

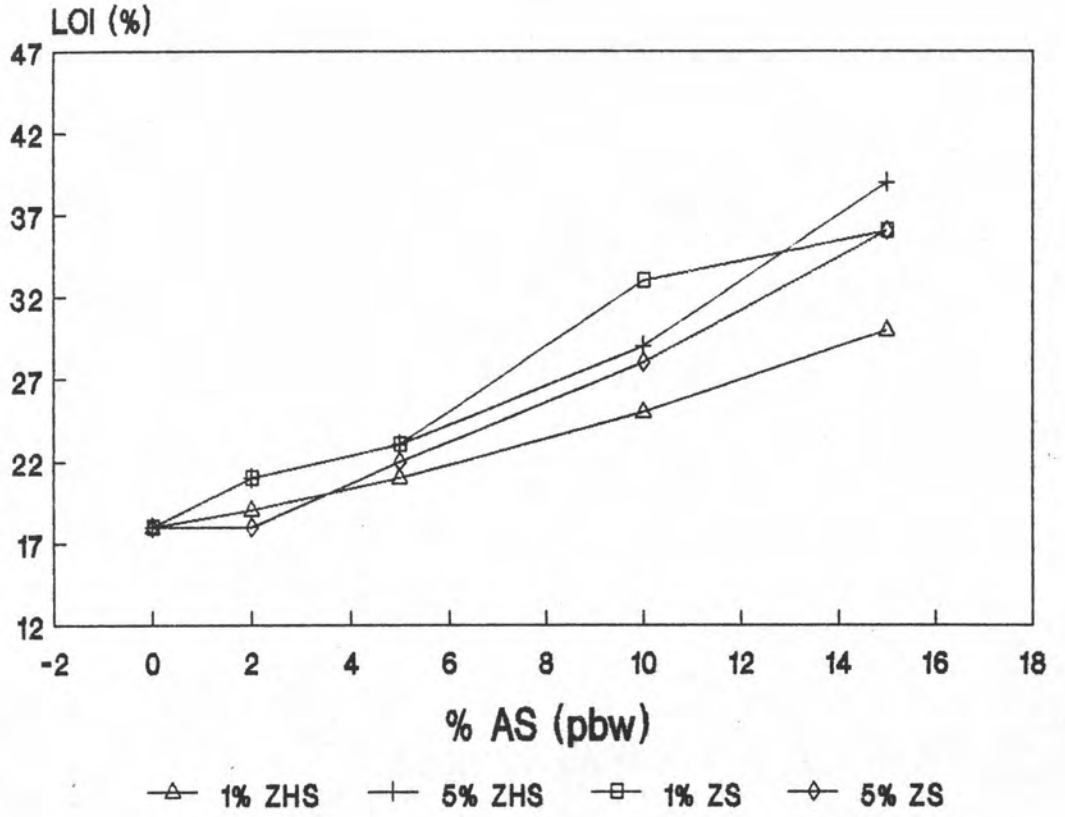

Figure 4-5 Synergistic effect of ZHS and ZS on flammability of polyurethane containing DBDPE

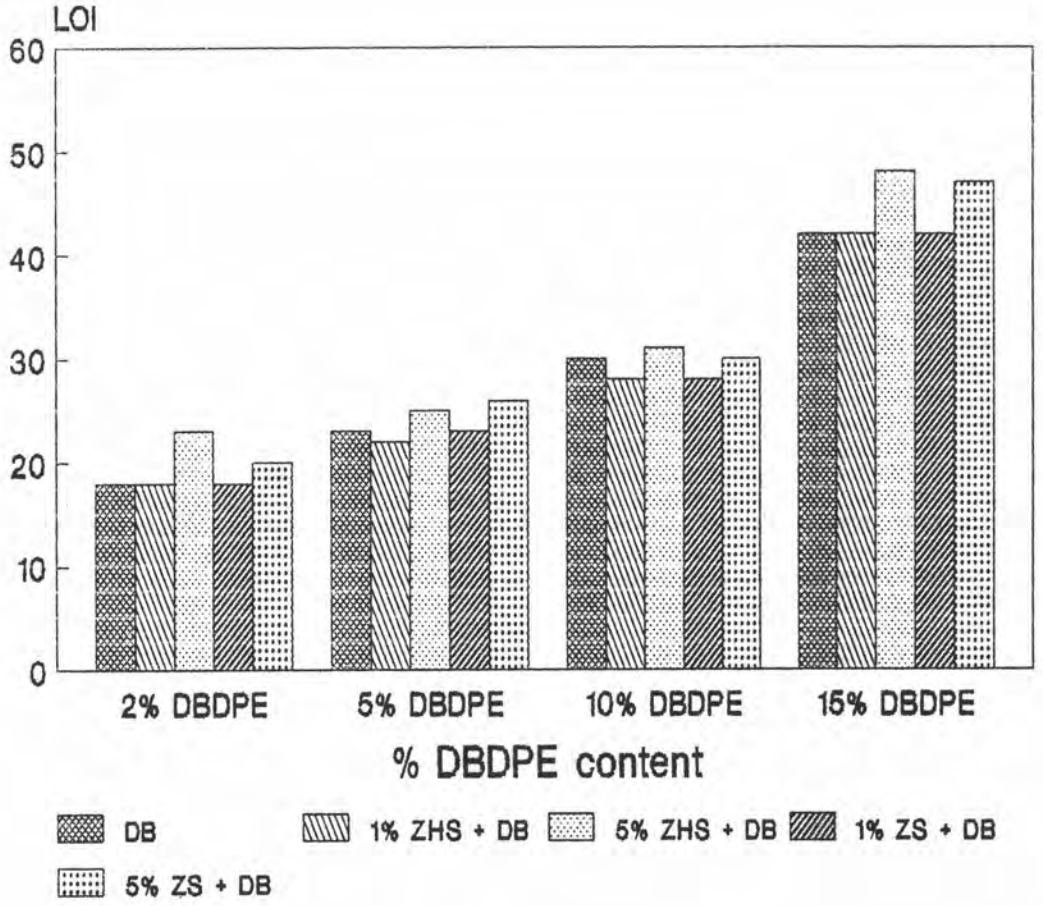

Figure 4-6 Synergitic effect of ZHS and ZS on flammability of polyurethane containing AS

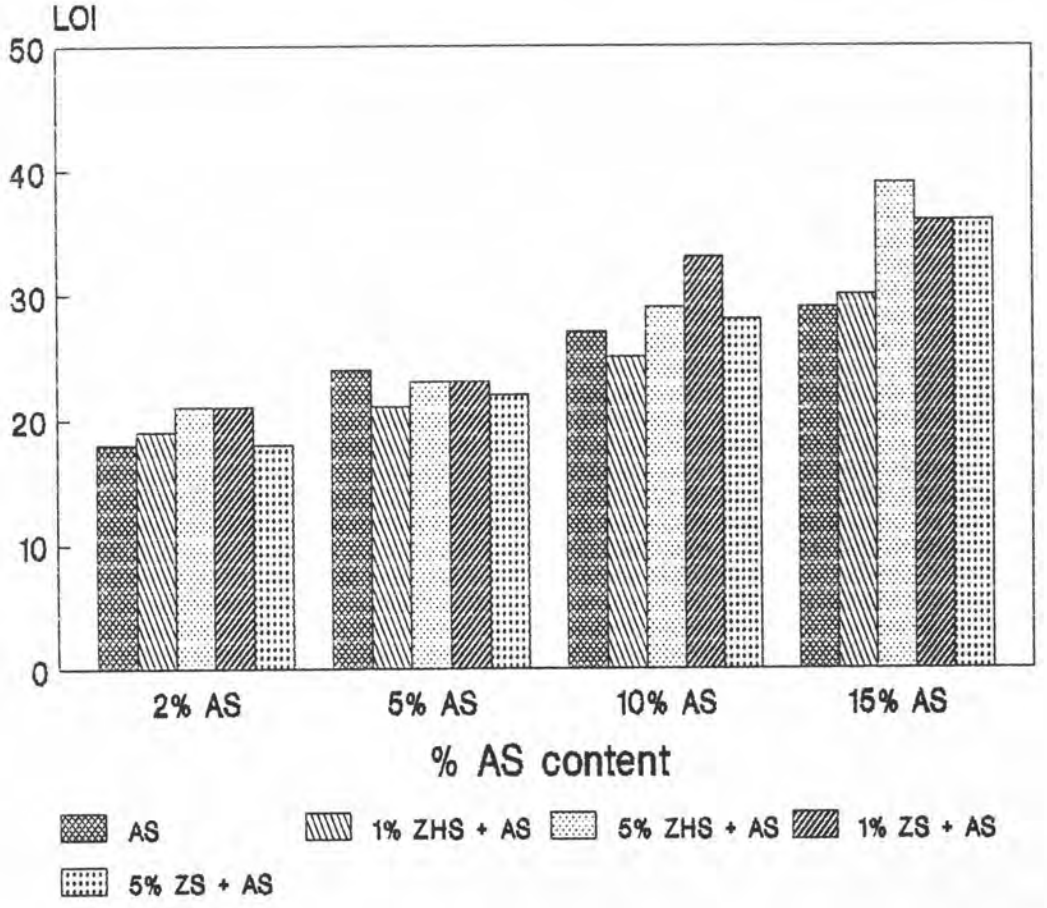

Figure 4-7 (a) Effect of ZHS, ZS and HDR on stress at break in flexible polyurethane

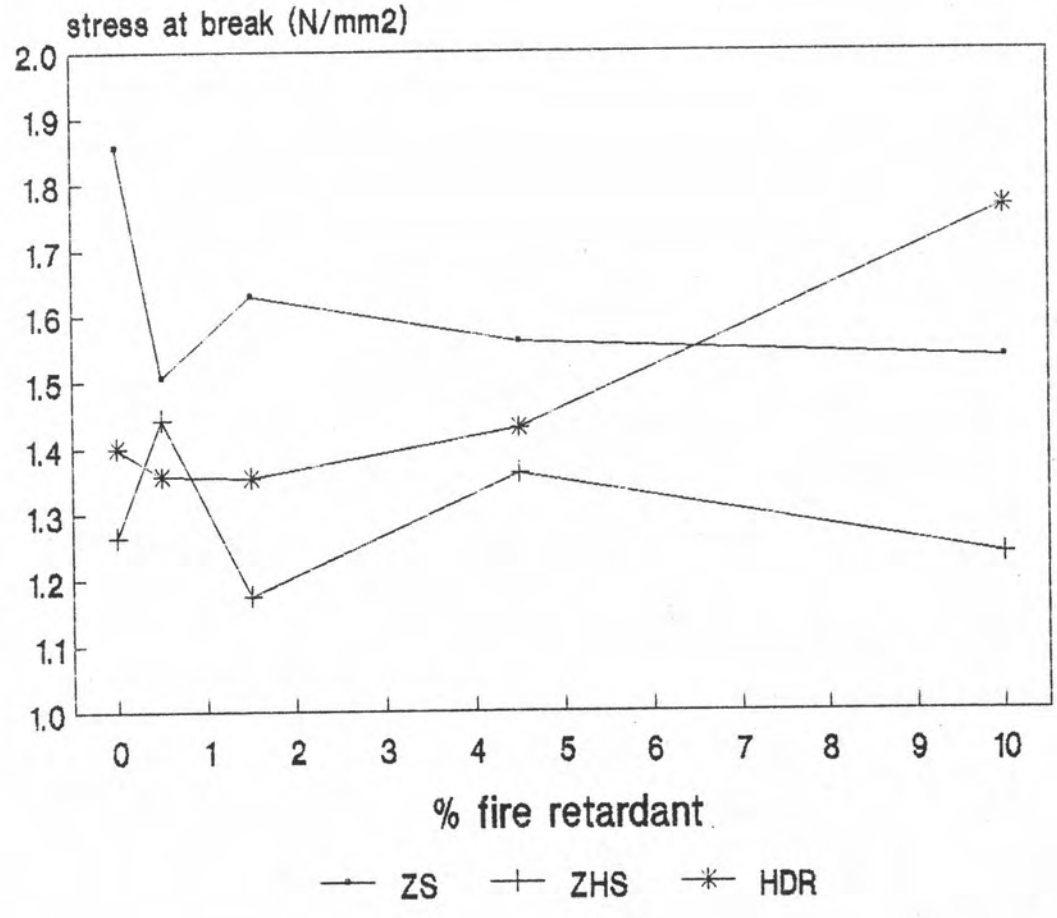

Figure 4-7(b) Effect of ZHS, ZS and HDR on stress at break in rigid polyurethane

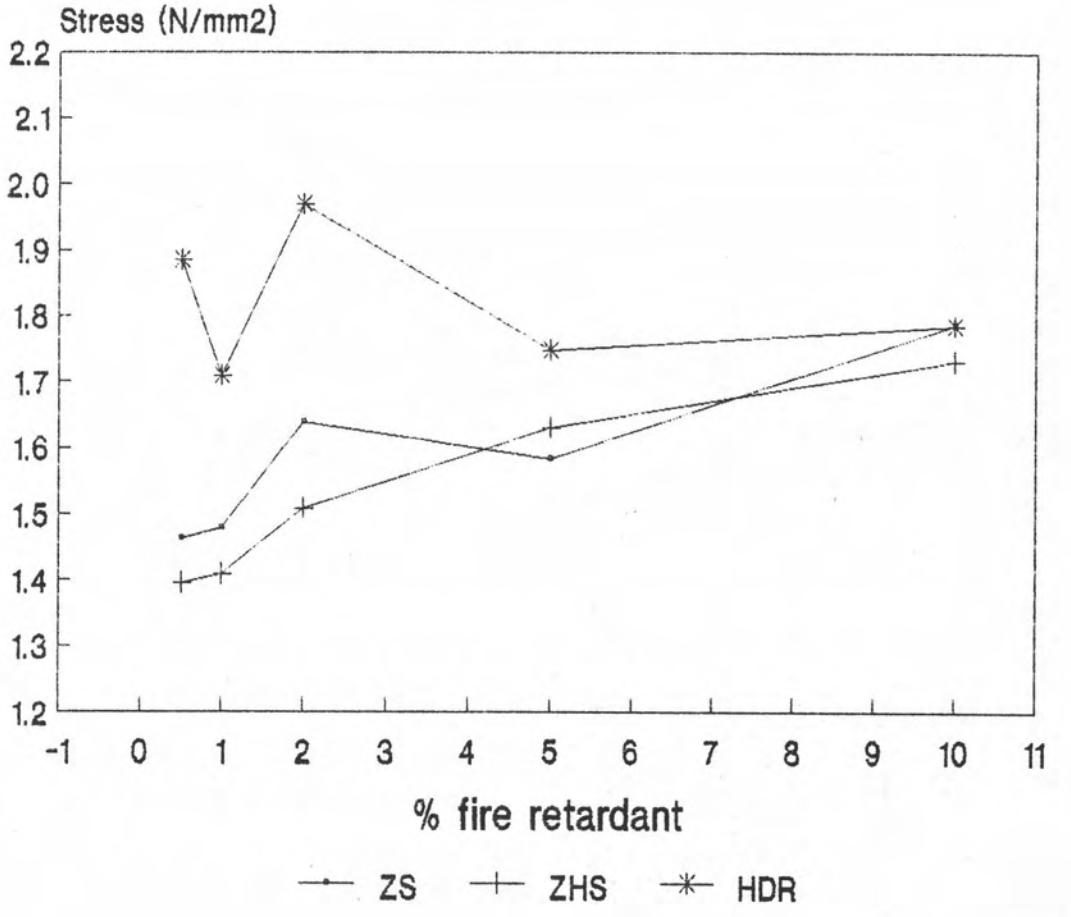

Figure 4-8(a) Effect of ZHS, ZS and HDR on strain at break in flexible polyurethane

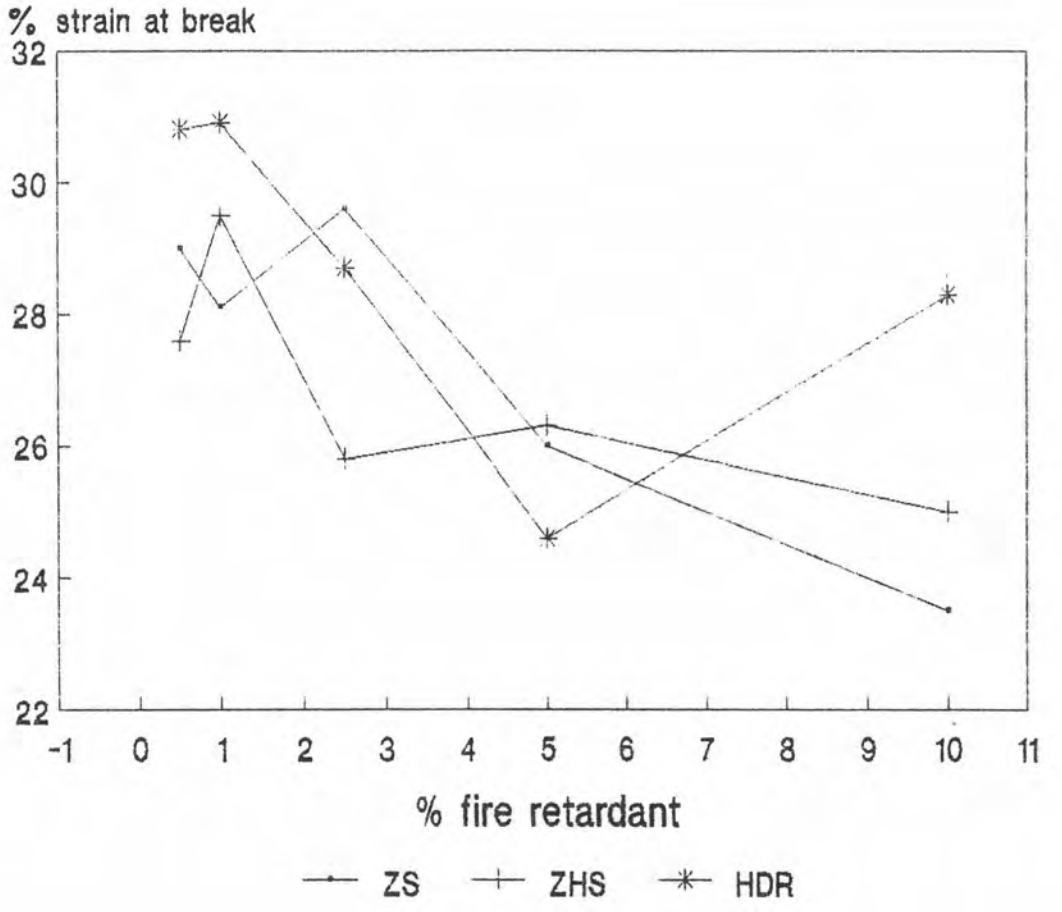

Figure 4-8(b) Effect of ZHS, ZS and HDR on strain at break in rigid polyurethane

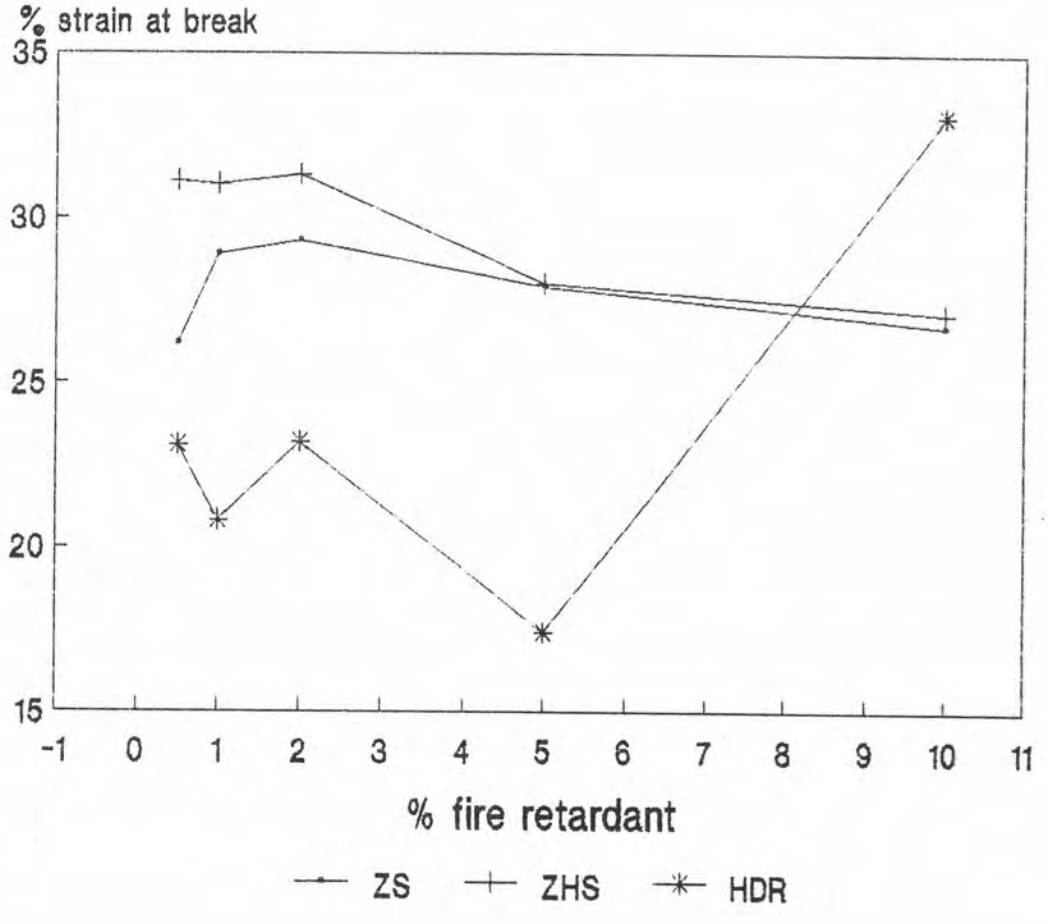

Figure 4-9(a) Effect of ZHS, ZS and HDR on 100% modulus in flexible polyurethane

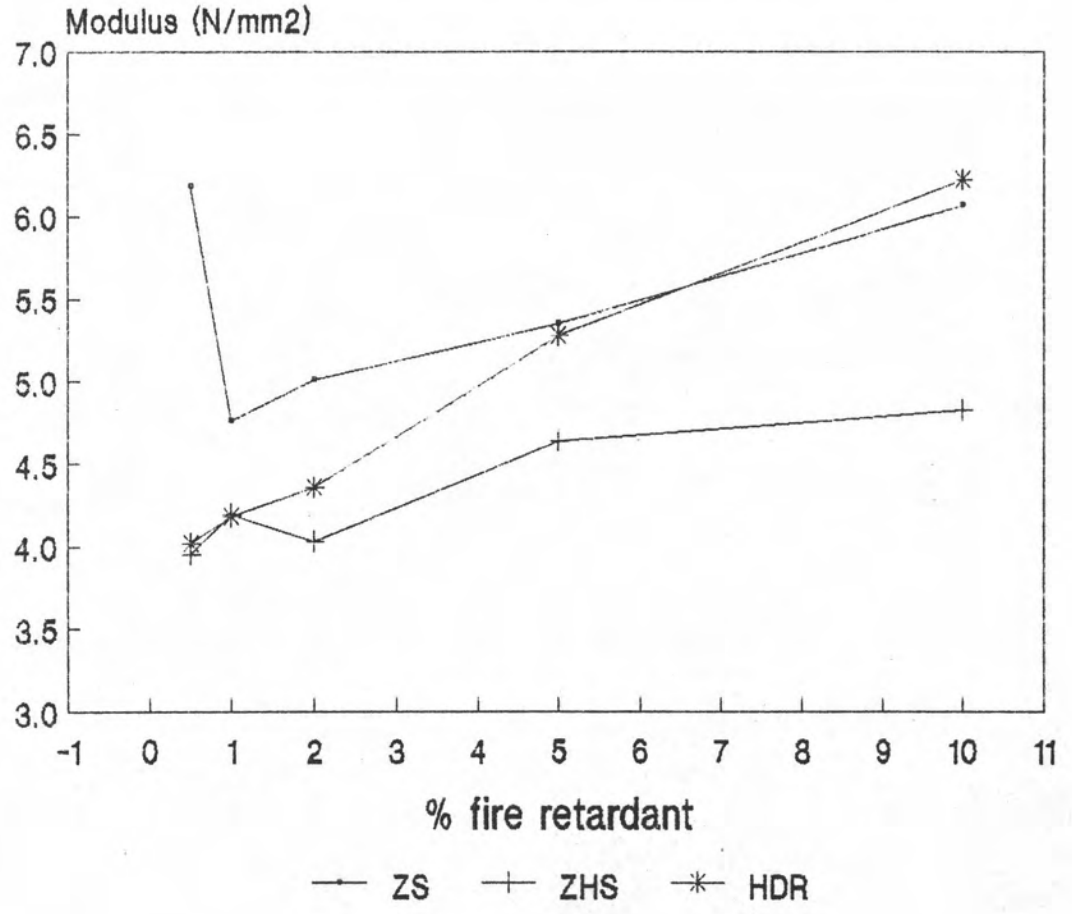

Figure 4-9(b) Effect of ZHS, ZS and HDR on 100% modulus in rigid polyurethane

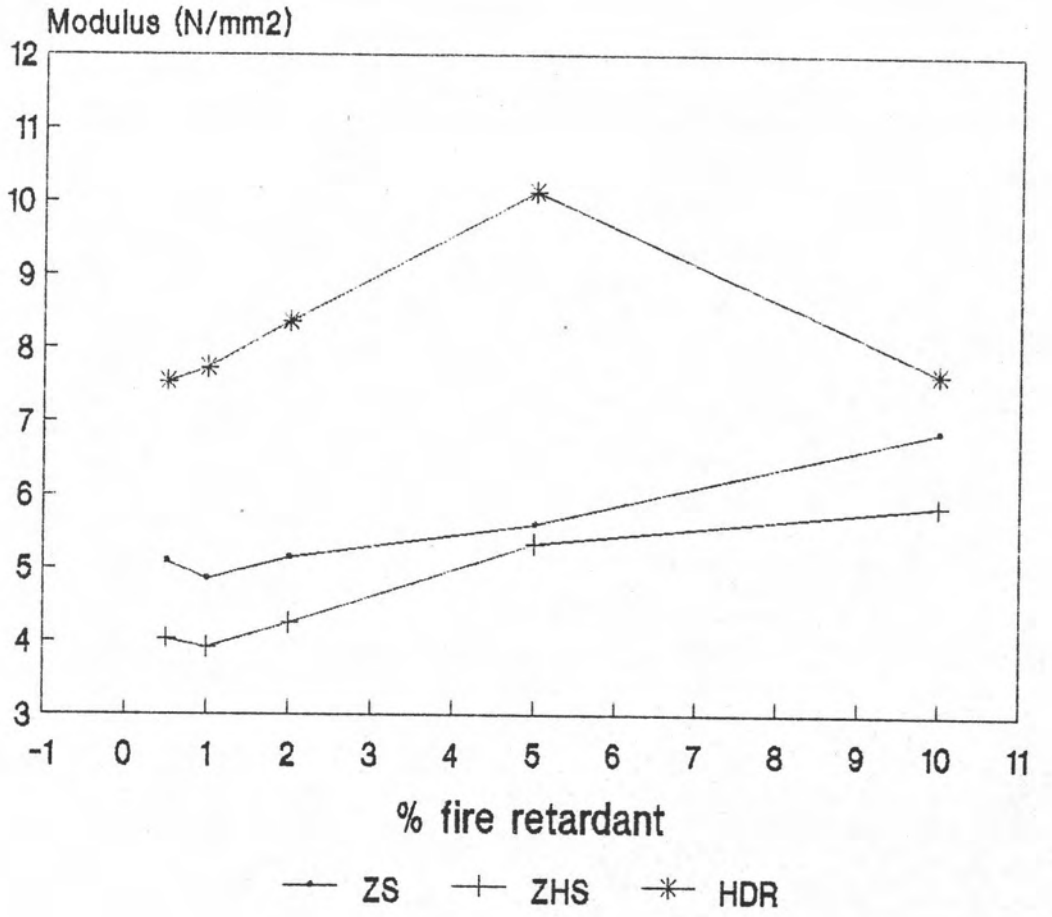

Figure 4-10(a) Effect of ZHS, ZS and DCDBE on stress at break in flexible polyurethane

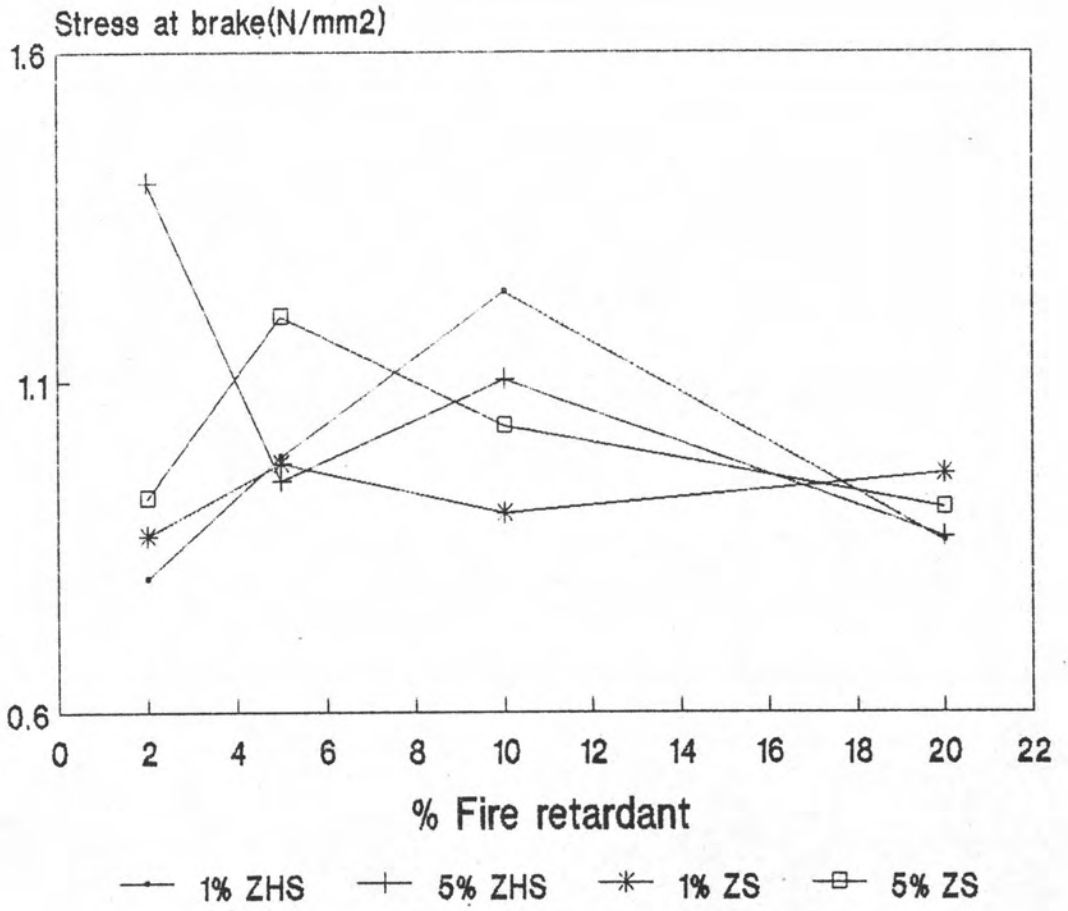

Figure 4-10(b) Effect of ZHS, ZS and DCBDE on strain at break in flexible polyurethane

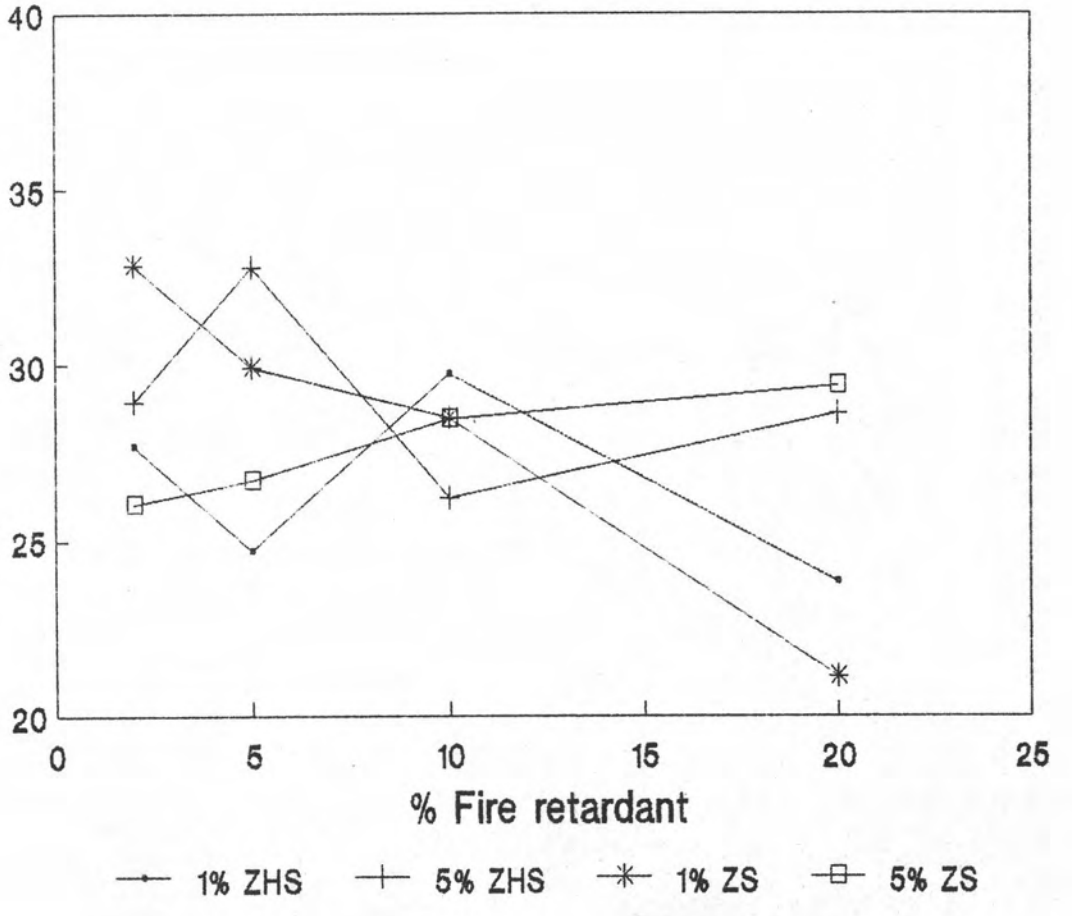

Figure 4-10(c) Effect of ZHS, ZS and DCBDE on 100% modulus in flexible polyurethane

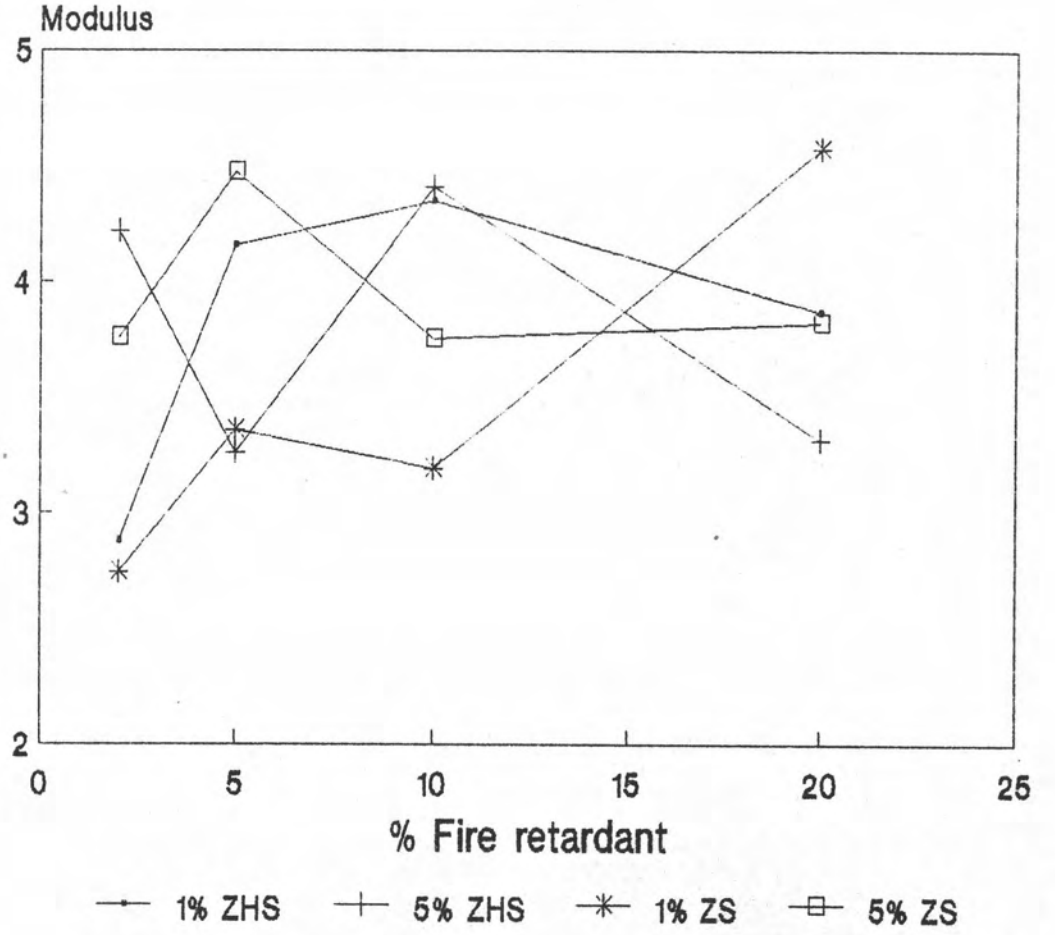

Figure 4-11(a) Effect of ZHS, ZS and AS on stress at break in flexible polyurethane

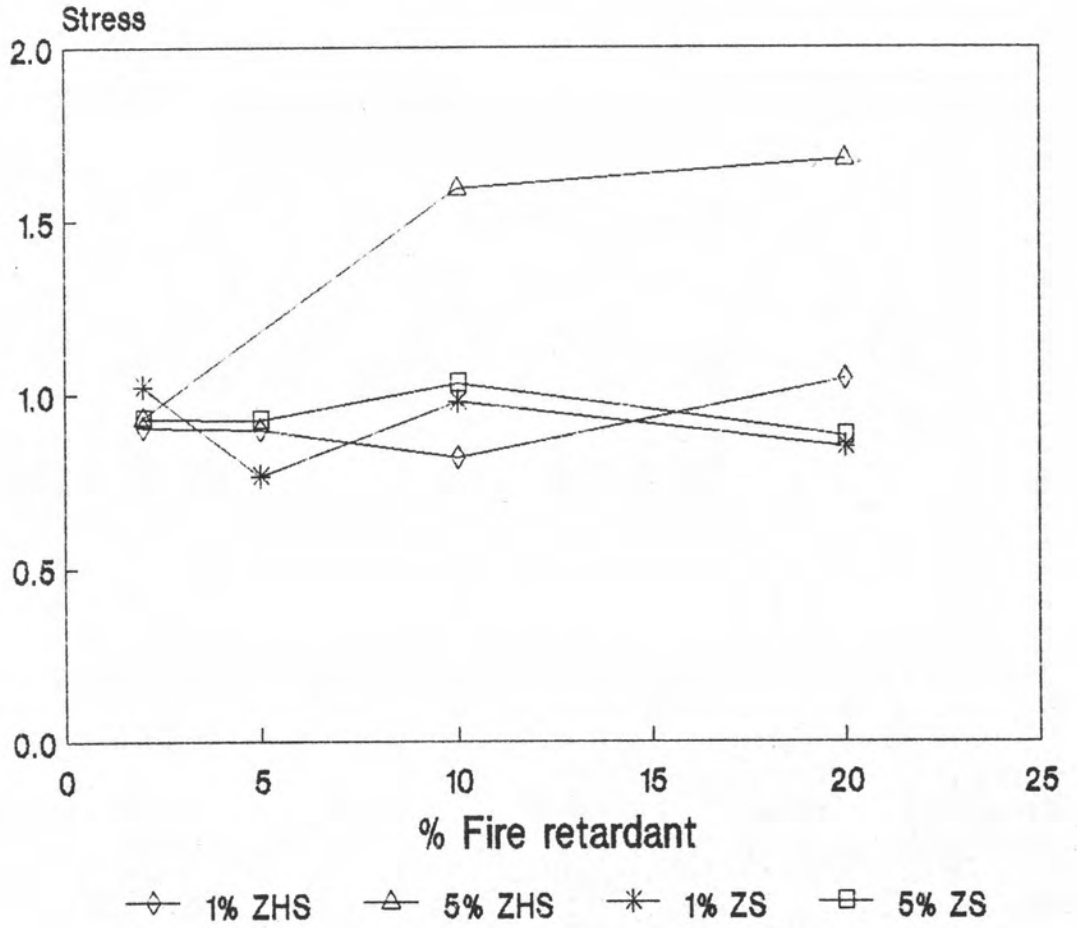

Figure 4-11(b) Effect of ZHS, ZS and AS on strain at break in flexible polyurethane

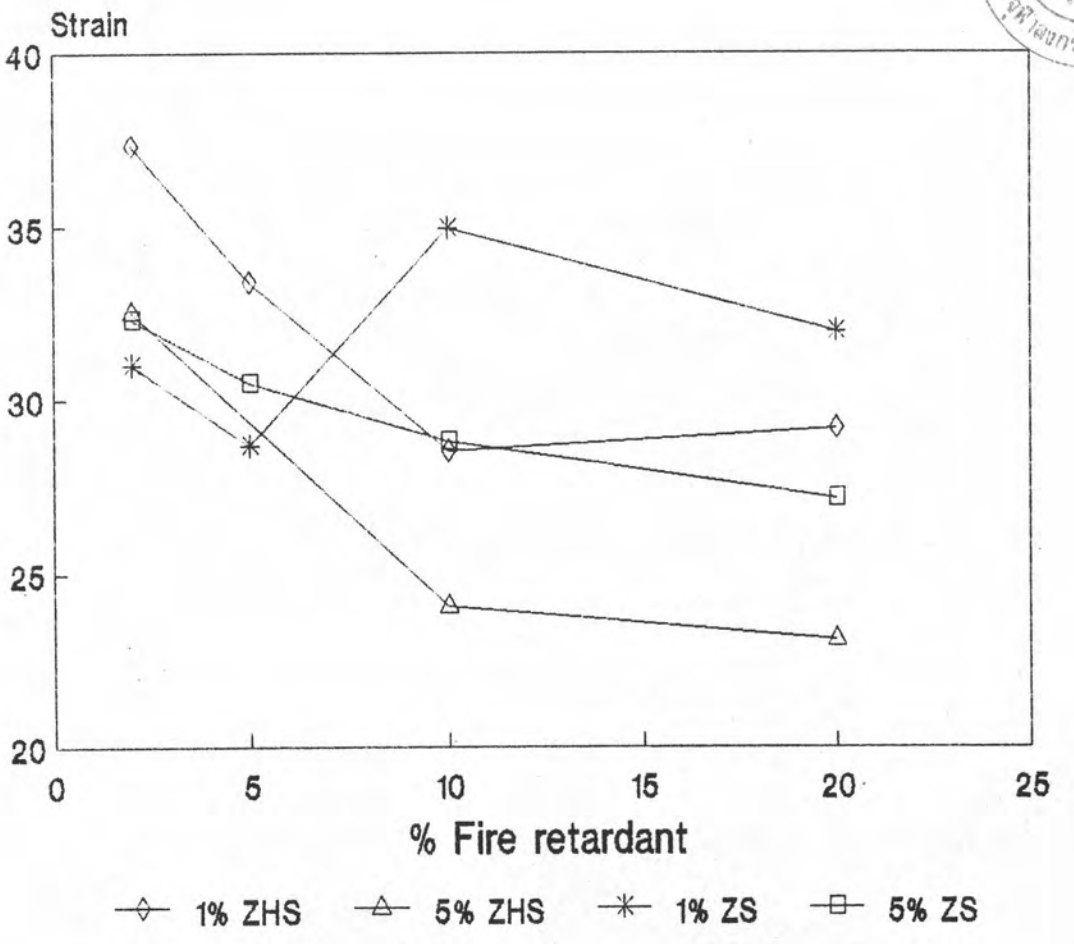

Figure 4-11(c) Effect of ZHS, ZS and AS on modulus in flexible polyurethane

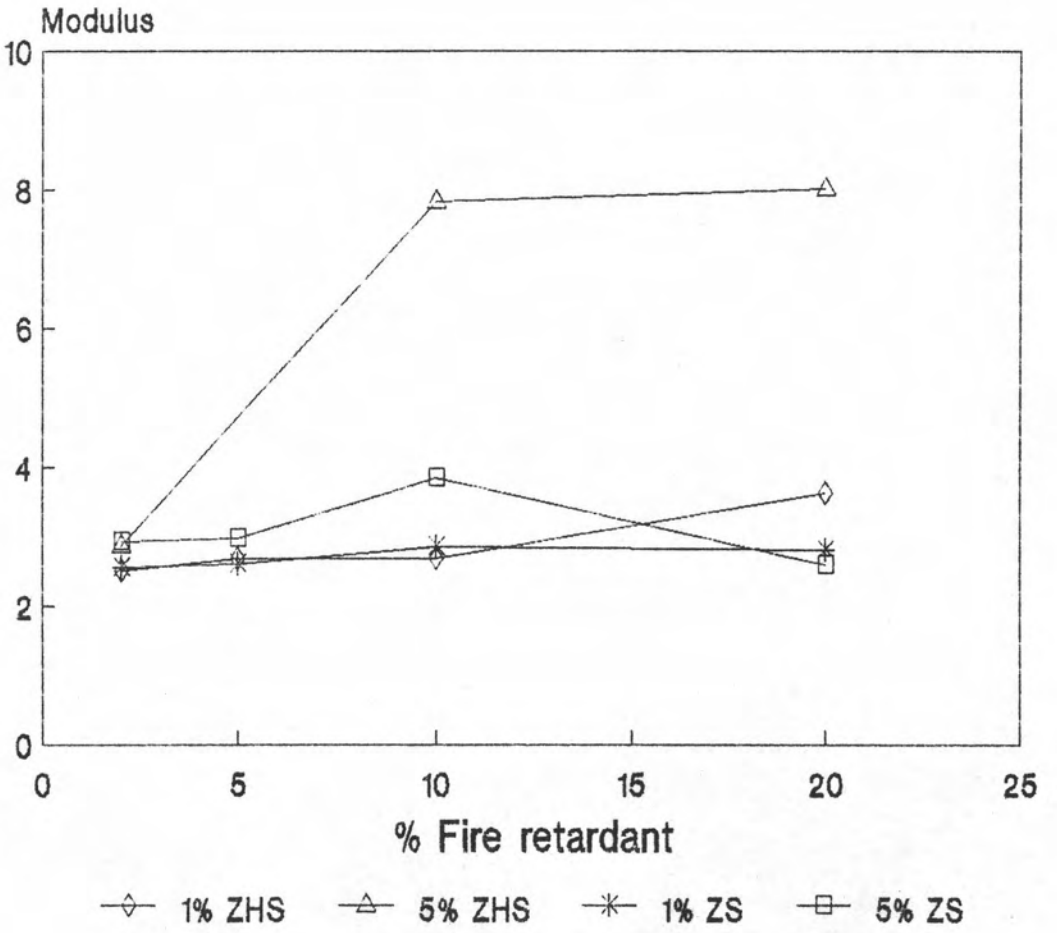

Table 4-12 DSC data for flexible polyurethane containing ZHS and ZS

% fire retardant	Endothermic Temperature (°C)	
No additive	268	290
0.5% ZHS	267	306
1.0% ZHS	267	286
2.0% ZHS	267	286
5.0% ZHS	209	287
10.0% ZHS	219	264
0.5% ZS	267	290
1.0% ZS	267	358
2.0% ZS	263	327
5.0% ZS	265	318
10.0% ZS	283	314

Figure 4-12 X-ray diffraction pattern and line pattern of synthetic sodium stannate

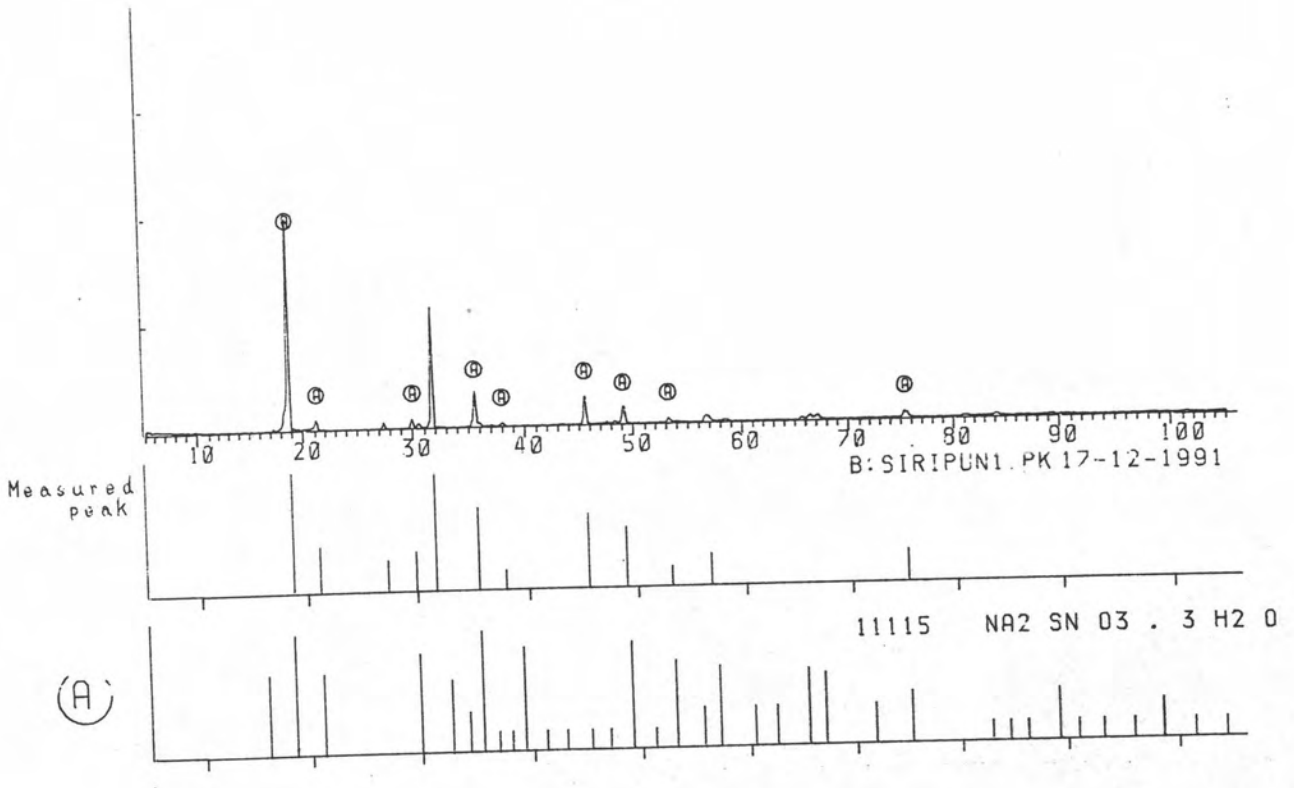

Figure 4-13 X-ray diffraction pattern and line pattern of synthetic zinc hydroxystannate.

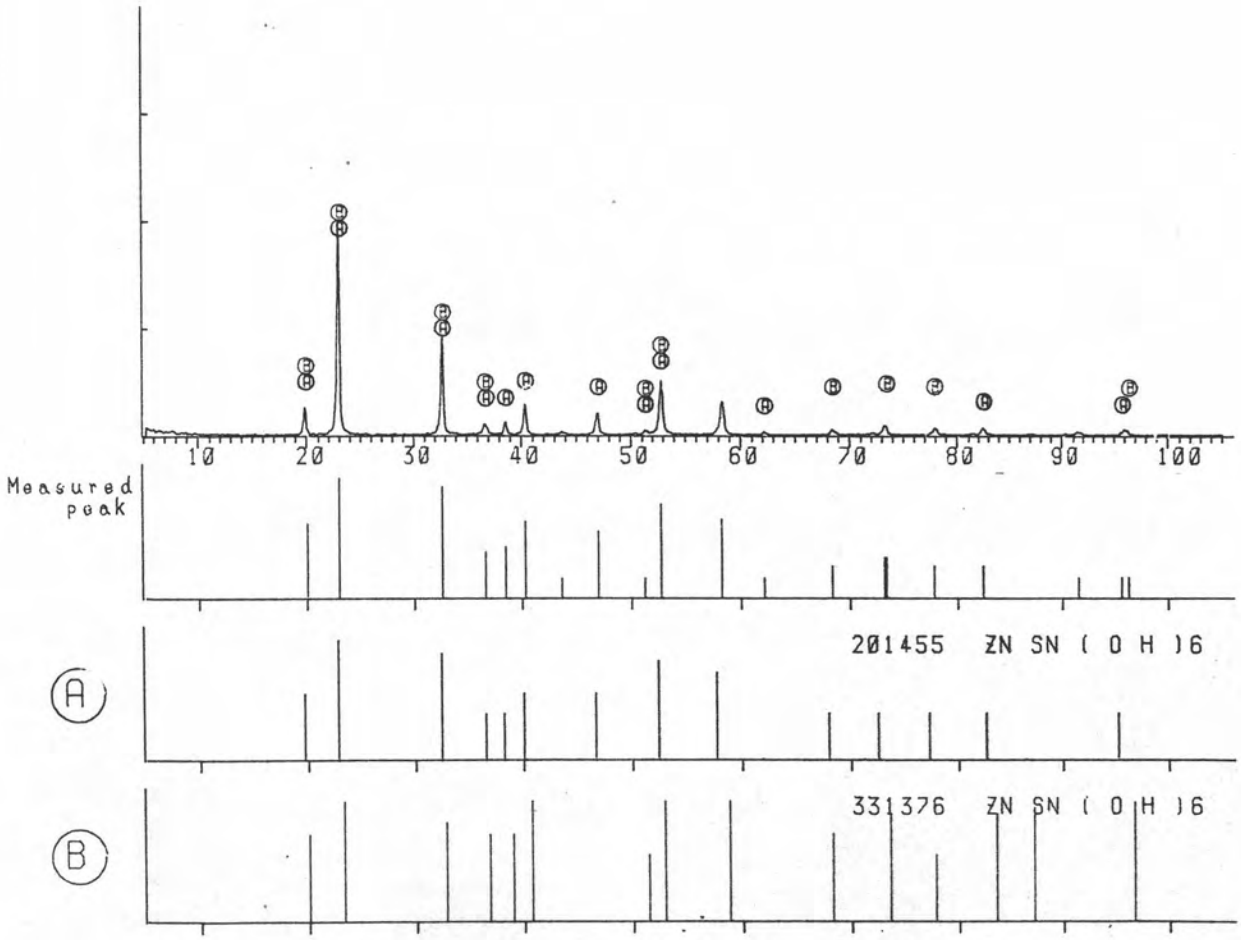

Figure 4-14 X-ray diffraction pattern and line pattern of synthetic hydroromarchite.

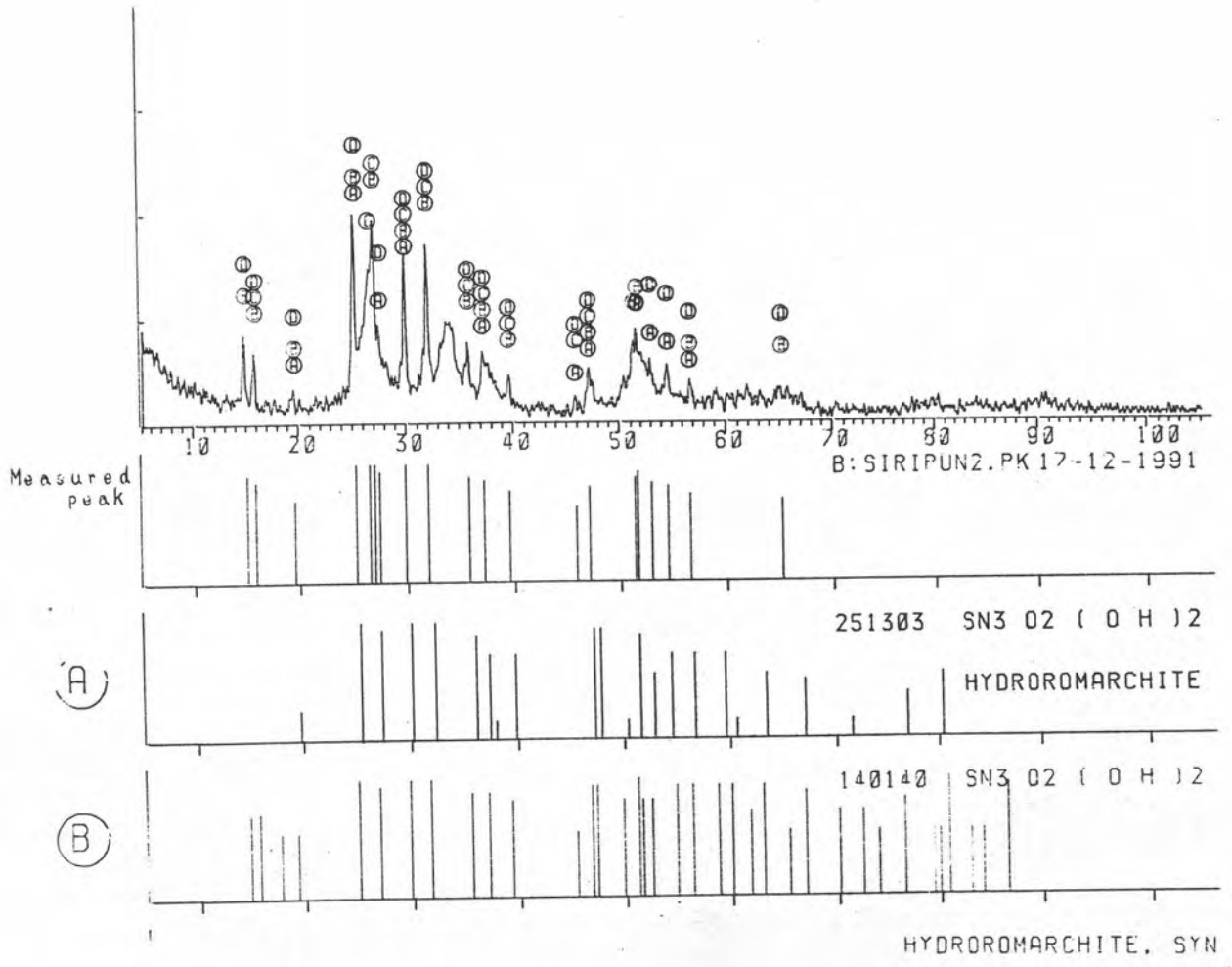

Figure 4-15 FTIR result from the synthetic sodium hydroxystannate

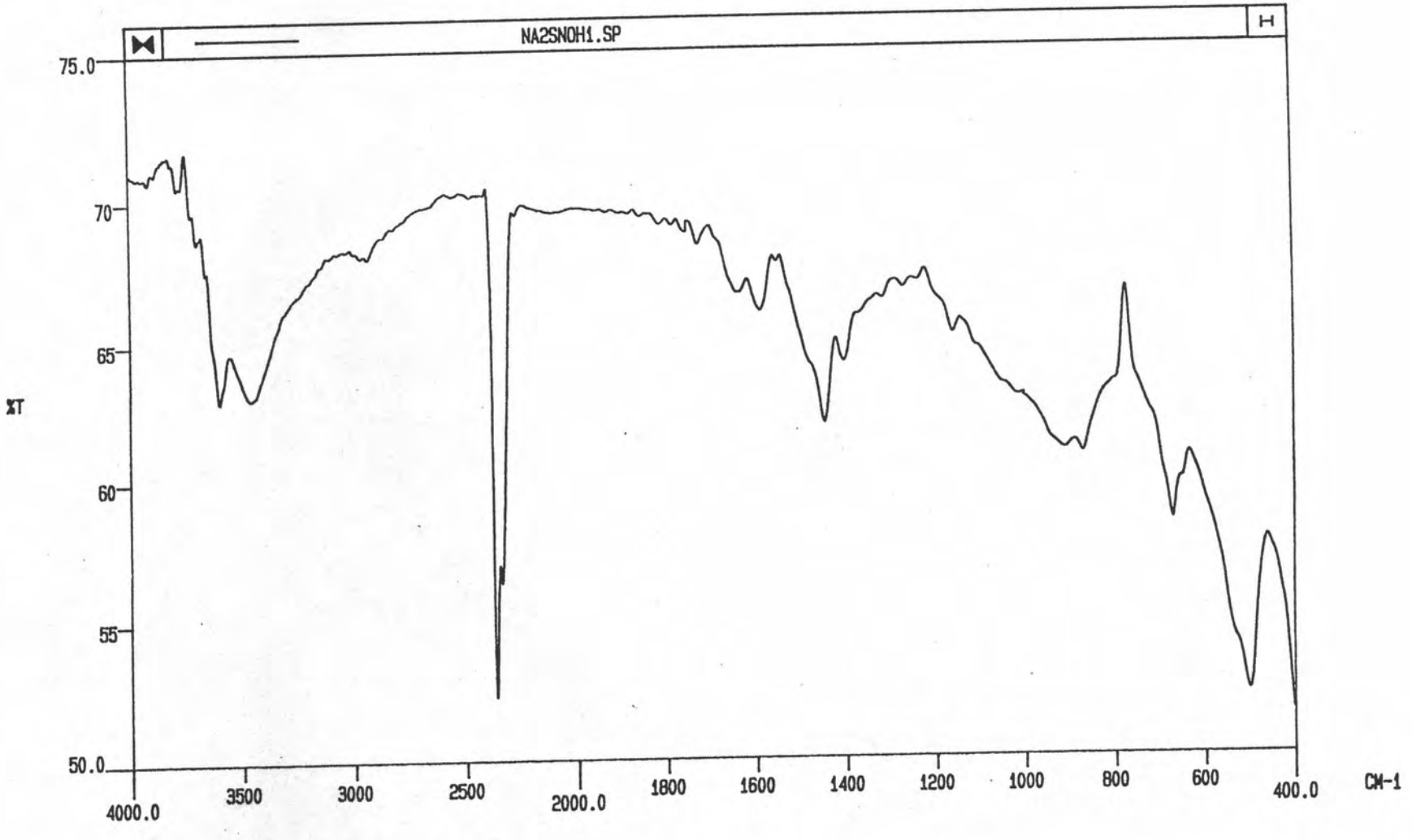

Figure 4-16 FTIR result from synthetic zinc hydroxystannate

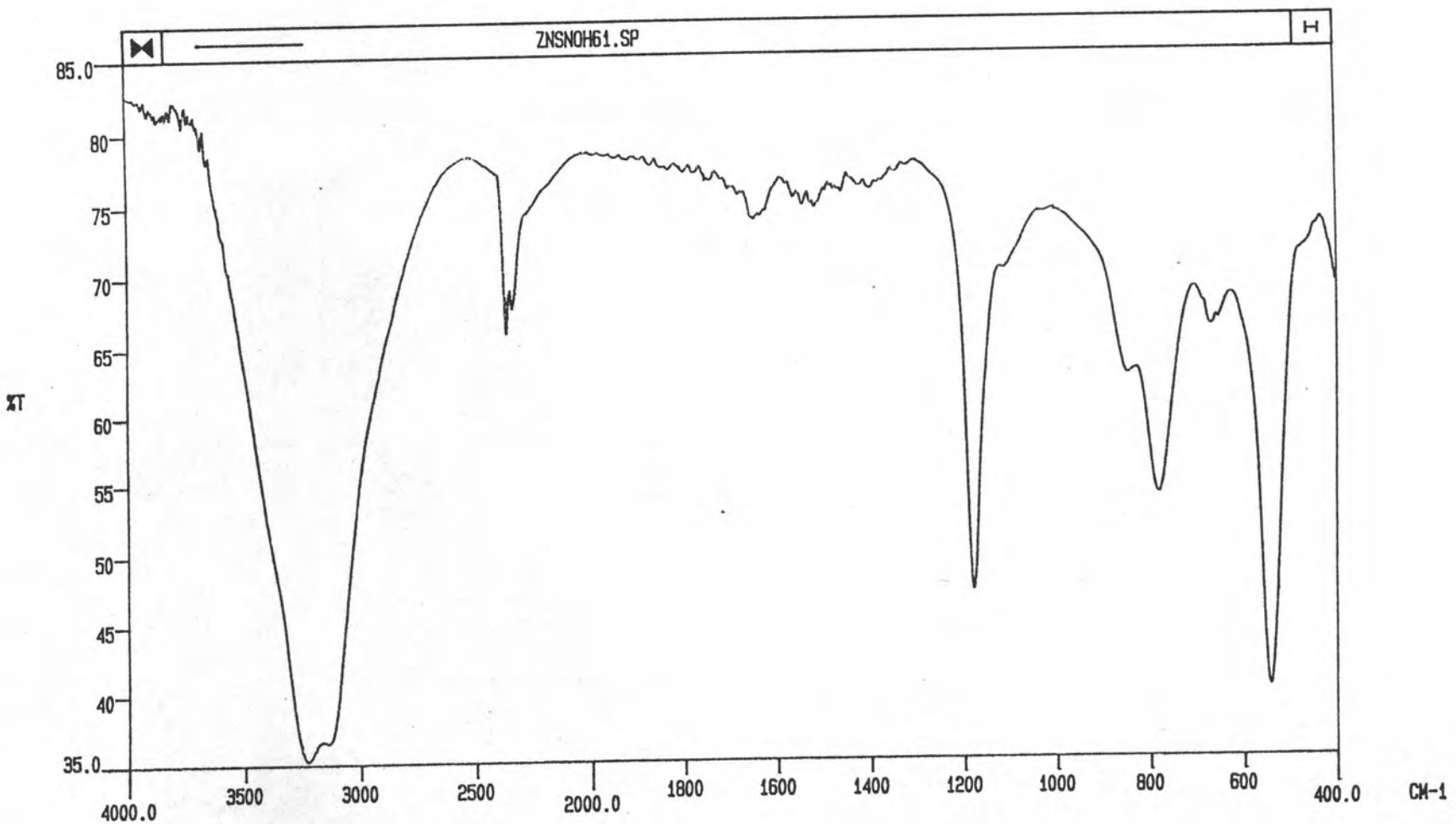

Figure 4-17 FTIR result from synthetic zinc stannate

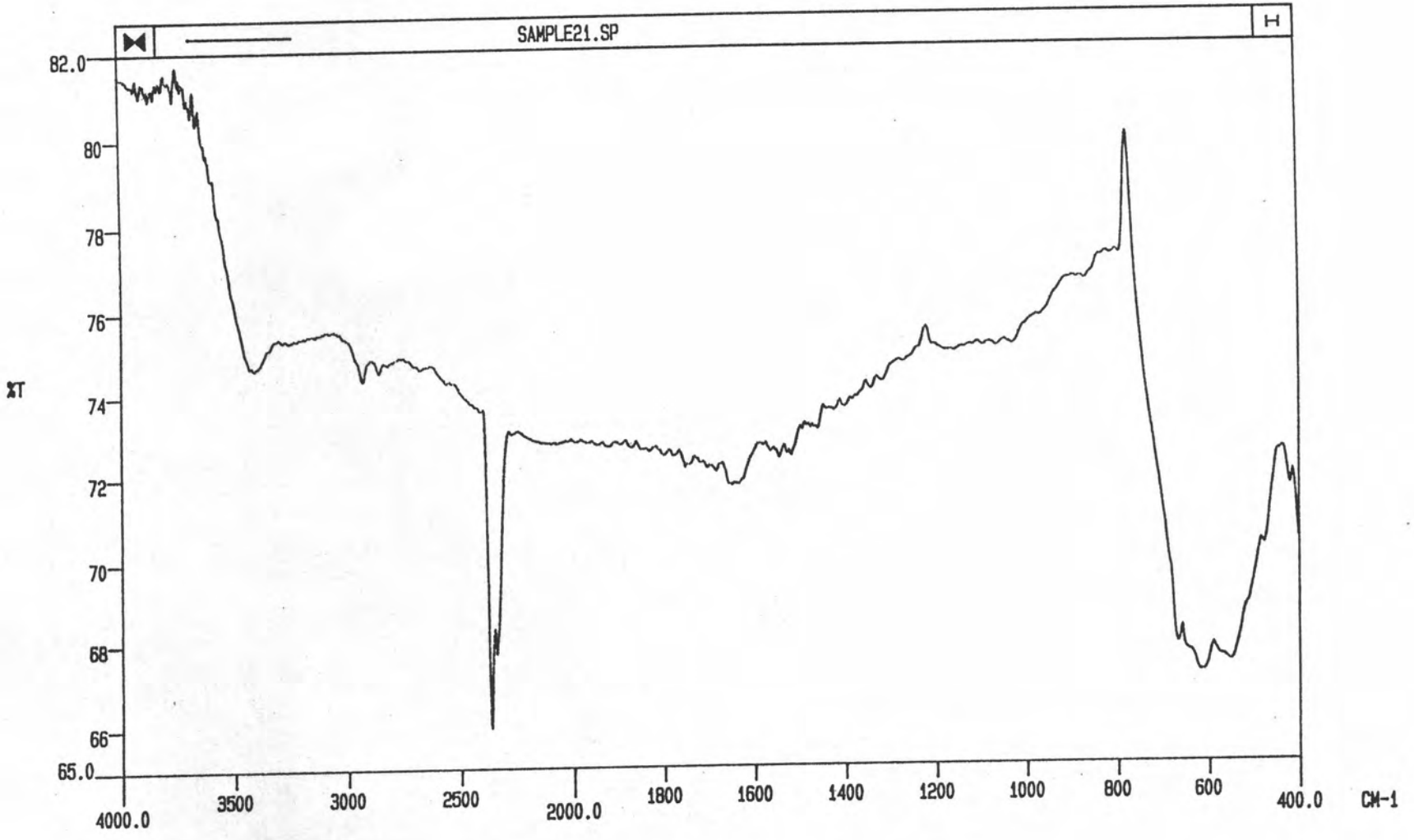

Figure 4-18 Average smoke density (Ds/g) values of polyurethanes containing ZHS

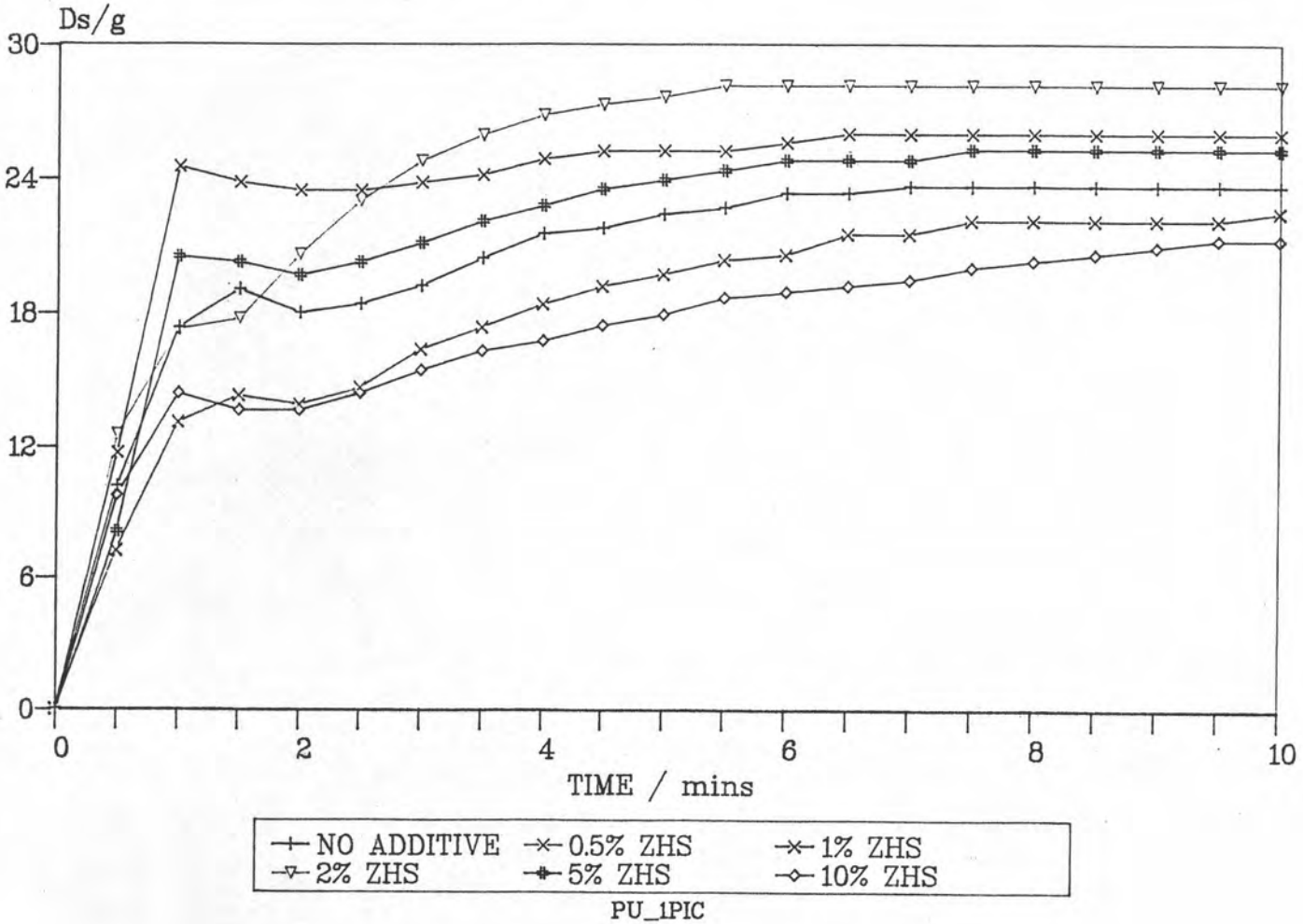

Figure 4-19 Average smoke density (Ds/g) values of polyurethanes containing ZS