

CHAPTER 4

RESULTS

The LOI and smoke evolution measurements for polyurethane treated DMMP or tin or halogenated compound were reported in table 4.1 to 4.2. The LOI data for polyurethane treated tin in combination with halogenated compound, binary system, were shown in table 4.3, whereas smoke data were presented in table 4.4 and figure 4.1 to 4.6.

For the binary system, significant relationships between tin compound and chlorowax 70 (C70) or between tin compound and decabromodiphenyl oxide (DBDPO) was found. Hence this study concentrated on the effect of synergism properties of those compounds at level 10 g halogenated compound with 1, 3 and 7 g of tin additive. The results were shown in table 4-5 to 4-6 and figure 4-7-4-14.

Ultimately, the mechanisms of flame retardant additives in binary system were studied by using TGA/DTA/DTG apparatus. The results were demonstrated in table 4-7 to 4-8 and plotted in figure 4-15 to 4-28.

Table 4-1 The LOI and Dmc/g data for the polyurethane containing tin additives.

Material	LOI	LOI ^a	Dmc/g	% smoke reduction ^b
No additive	20.5	-	34.7	55.3
DMMP	23.6	3.1	77.7	-
MBTO	21.4	0.9	-	-
MOTO	21.1	0.6	-	-
DBTA	22.7	2.2	84.8	-9.1
DBTM	22.2	1.7	80.6	-3.7
DBTO	20.9	0.4	61.8	20.5
DOTO	20.3	-0.2	-	-
ZHS	20.9	0.4	70.0	9.9

a : Each LOI value was compared to that of no additive

b : Each Dmc/g value was compared to that of DMMP.

Table 4-2 The LOI and Dmc/g data for the polyurethane containing halogenated additives.

Material	LOI	LOI ^a	Dmc/g	%smoke reduction ^b
No additive	20.5	-	34.7	55.3
DMMP	23.6	3.1	77.7	-
C70	22.6	2.1	46.3	40.4
DBNPG	22.5	2.0	82.0	-5.5
DBDPO	23.1	2.6	88.9	-14.4
DECH	20.8	0.3	47.7	38.6

a : Each LOI value was compared to that of no additive

b : Each Dmc/g value was compared to that of DMMP.

Table 4-3 The LOI data for the polyurethane containing tin and halogenated additives.

Material	C70	DBDPO	DBNPG	DECH
No additive	22.6	23.1	22.5	20.8
DMMP	25.7	26.4	25.7	24.0
MBTO	23.5	22.3	22.7	21.3
MOTO	23.4	21.7	22.4	21.1
DBTA	24.1	24.3	24.3	22.9
DBTM	23.9	23.9	22.7	22.0
DBTO	24.8	23.1	22.6	22.2
DOTO	23.5	d	21.7	22.0
ZHS	24.7	22.6	22.6	21.3

d = foam collapsed.

Table 4-4 The Dmc/g data for the polyurethane foams containing tin and halogenated additives.

Material	C70	DBDPO	DBNPG	DECH
No additive	46.3	88.9	82.0	47.7
DMMP	90.0	117.0	103.8	89.3
MBTO	-	-	-	-
MOTO	-	-	-	-
DBTA	54.4	106.4	88.1	71.2
DBTM	47.8	114.5	-	-
DBTO	55.5	85.8	-	-
DOTO	-	d	-	-
ZHS	61.5	57.9	-	-

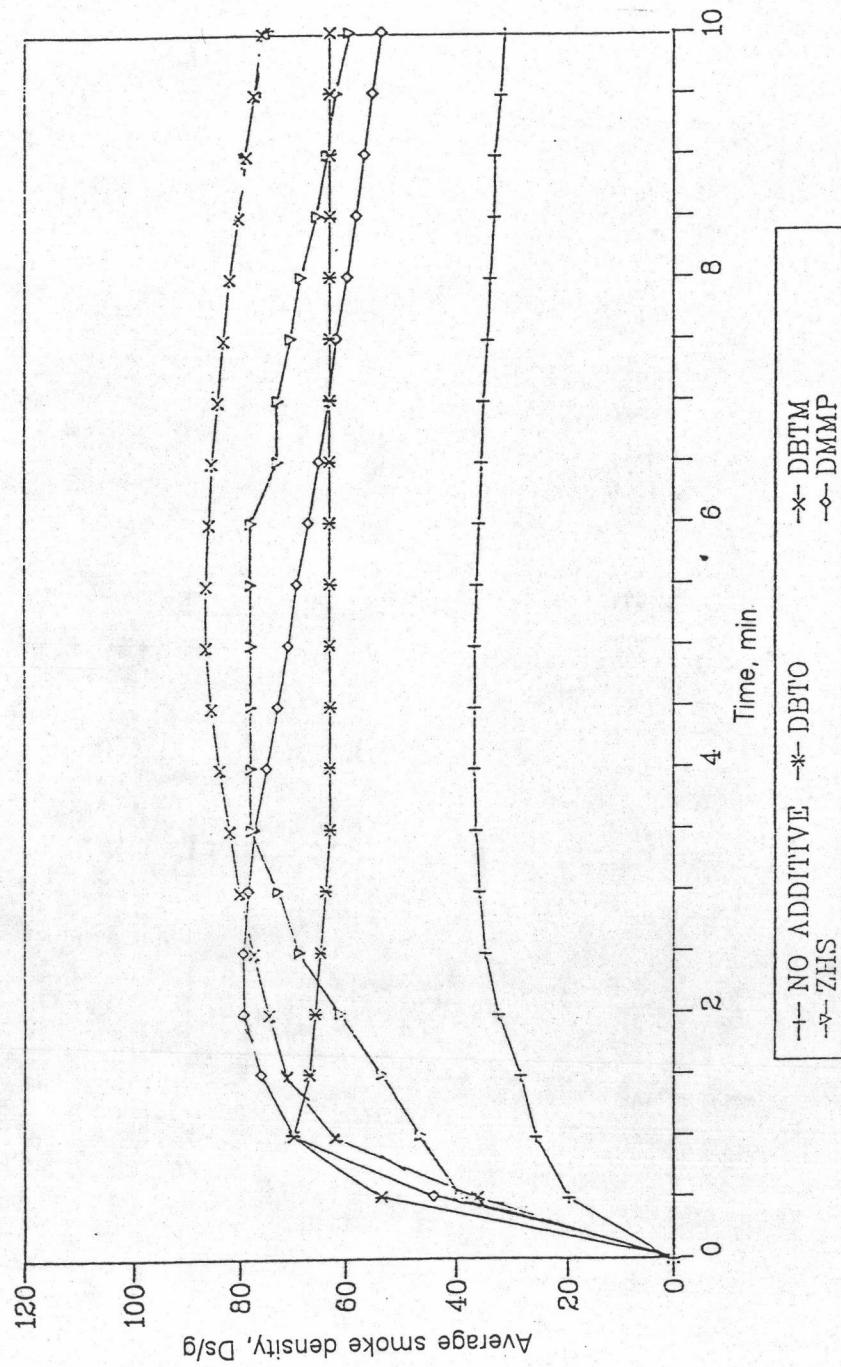


Figure 4-1 Average smoke density values of polyurethanes containing 7g tin compounds

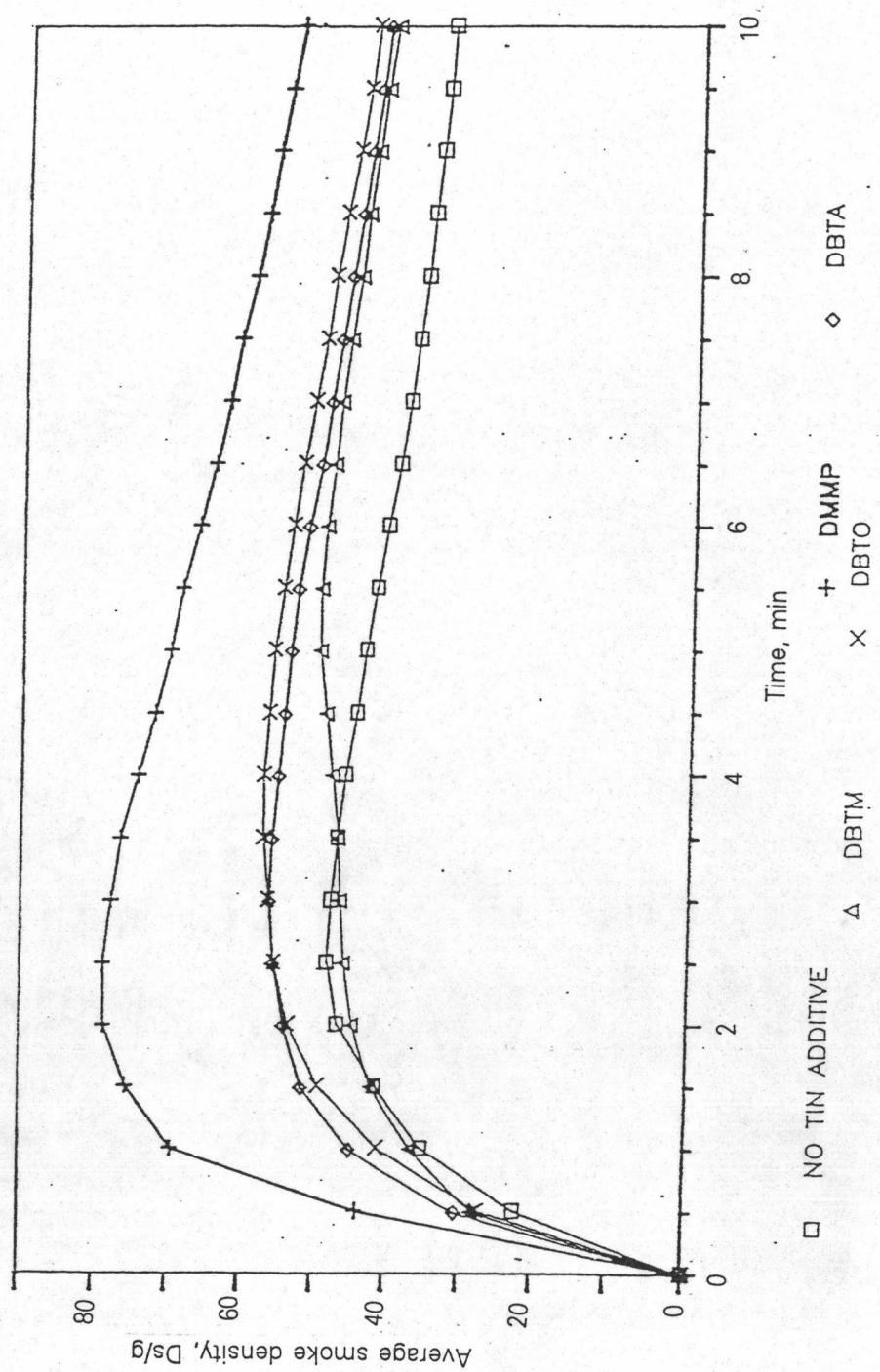


Figure 4-2 Average smoke density values of polyurethanes containing 10g C70 and 7g organotin compounds

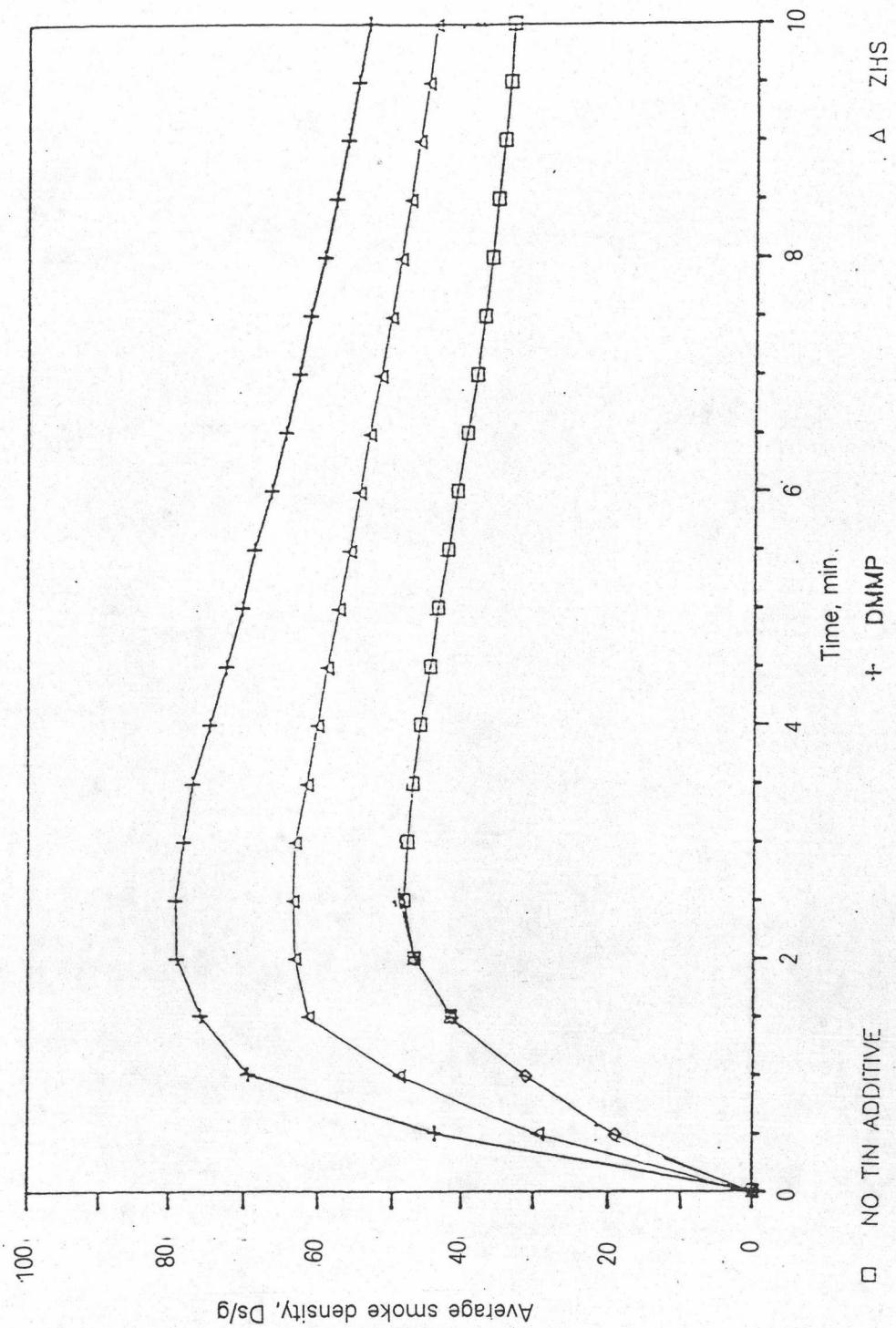


Figure 4-3 Average smoke density values of polyurethanes containing 10g C70 and 7g inorganic tin compounds

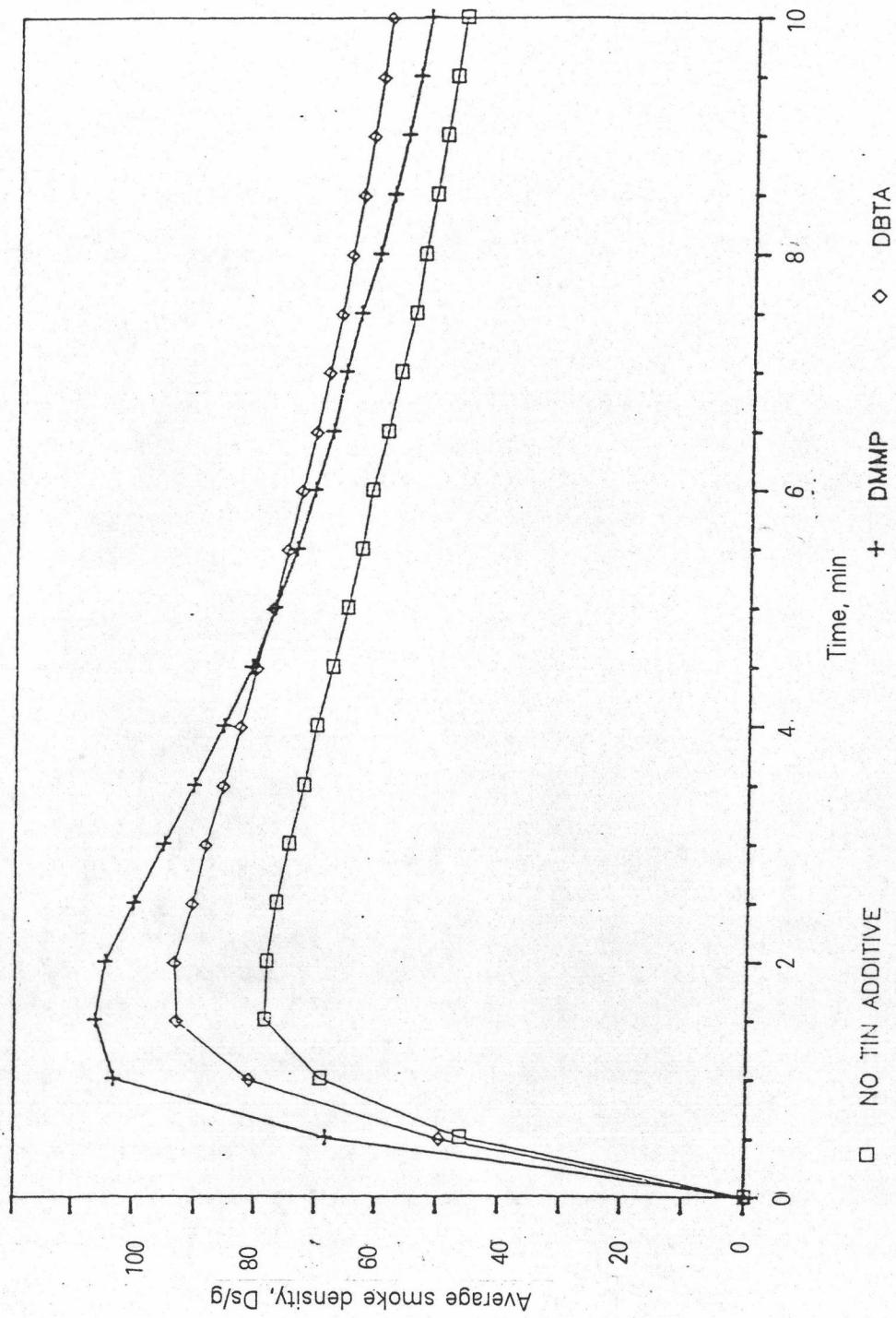


Figure 4-4 Average smoke density values of polyurethanes containing 10g DBNPG and 7g tin compounds

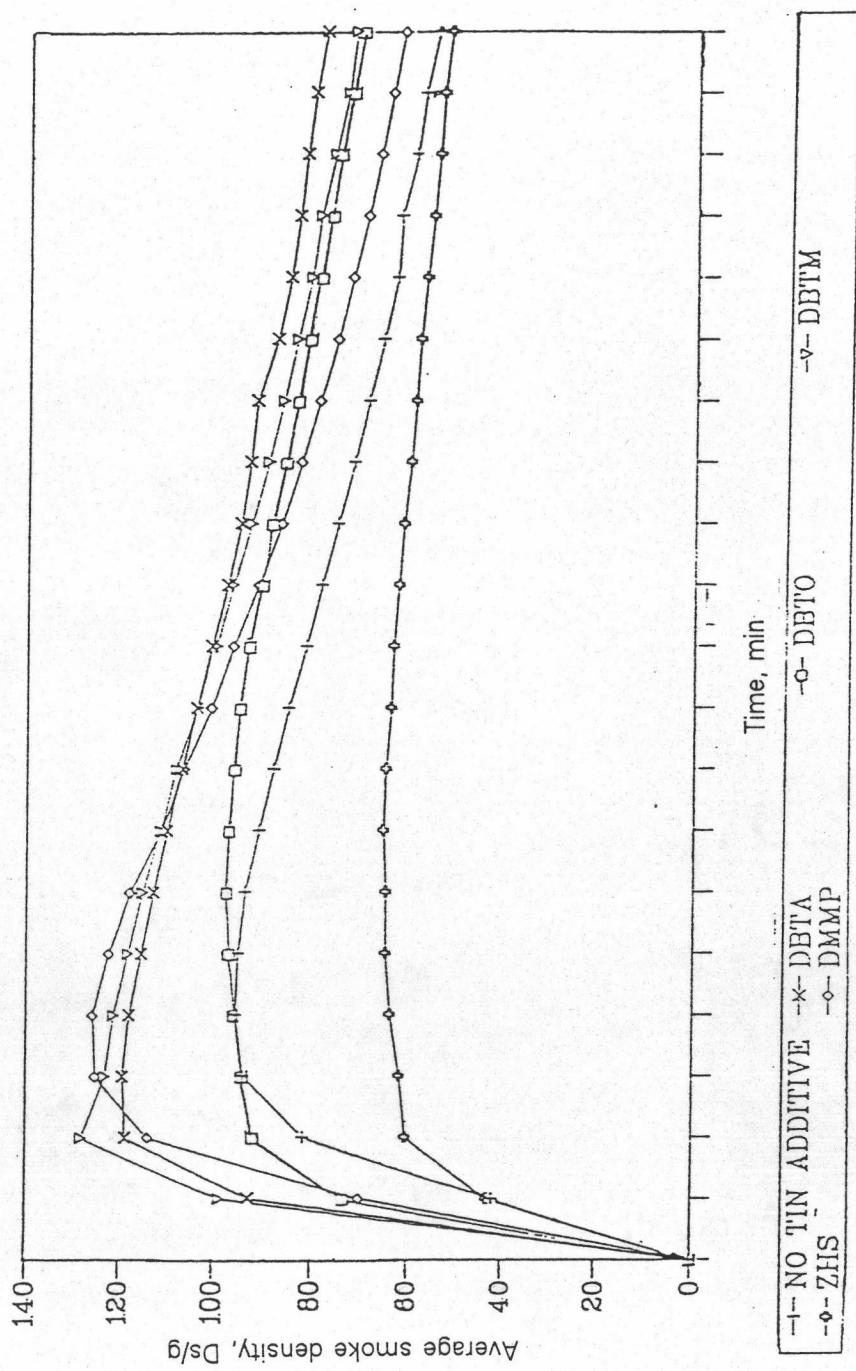


Figure 4-5 Average smoke density values of polyurethanes containing 10g DBDPO and 7g tin compounds

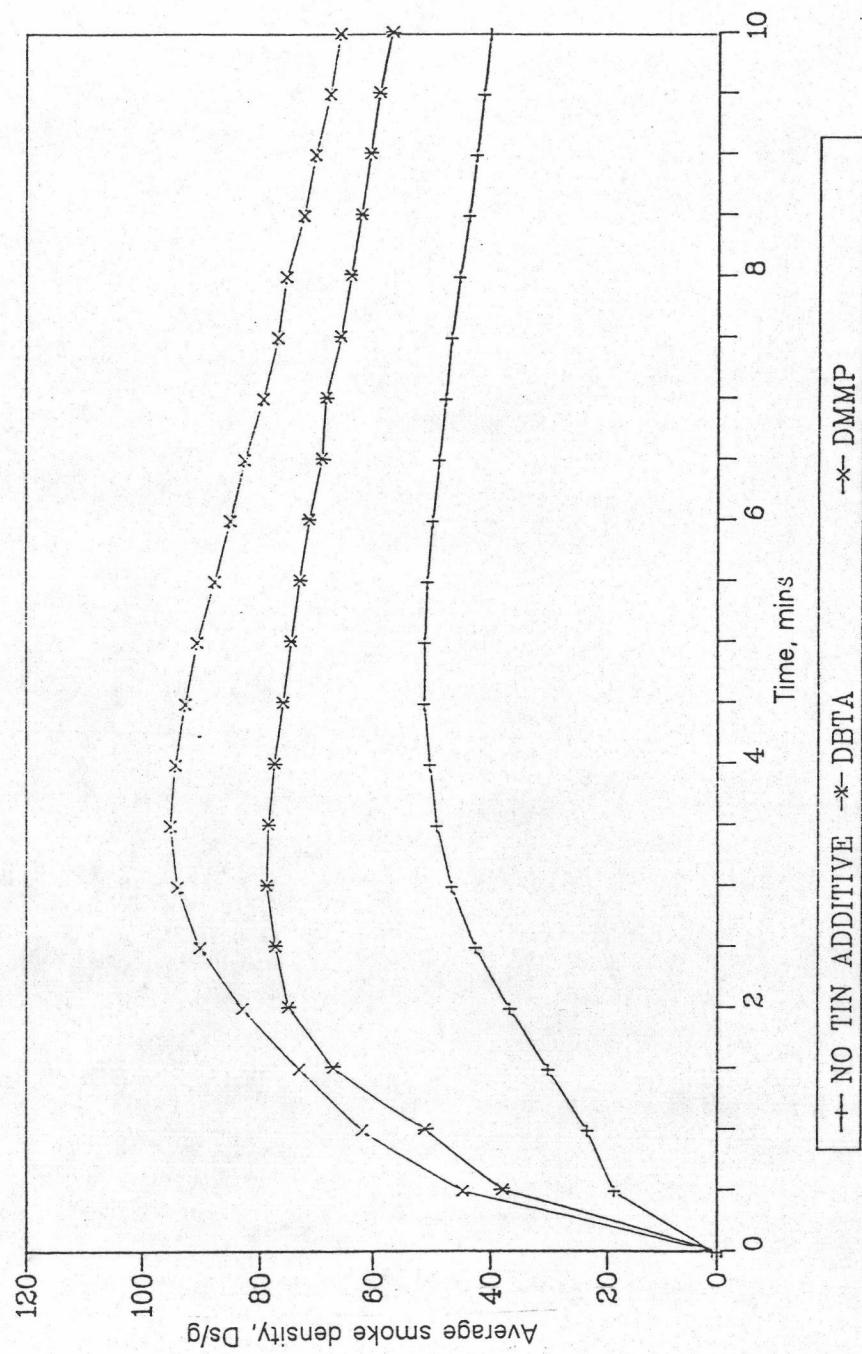


Figure 4-6 Average smoke density values of polyurethanes containing 10g Dech and 7g tin compounds

Table 4-5 The LOI and Dmc/g data for the polyurethane containing tin and C70 additives.

Material	LOI	LOI ^a	Dmc/g	% smoke reduction ^b
No additive	20.5	-	34.7	55.3
DMMP	23.6	3.1	77.7	-
C70	22.8	2.3	46.3	40.4
DMMP/C70	25.7	5.2	90.0	-15.8
1g DBTA/C70	23.1	2.6	41.6	46.5
3g DBTA/C70	23.8	3.3	44.0	43.4
7g DBTA/C70	24.1	3.6	54.4	30.0
1g DBTO/C70	23.3	2.8	59.1	23.9
3g DBTO/C70	24.3	3.8	69.4	10.7
7g DBTO/C70	24.8	4.3	55.5	28.6
1g ZHS/C70	22.4	1.9	72.8	6.3
3g ZHS/C70	22.9	2.4	83.4	-7.3
7g ZHS/C70	24.7	4.2	61.5	20.8
1g ZS/C70	22.9	2.4	76.0	-15.6
3g ZS/C70	23.1	2.6	62.8	30.2
7g ZS/C70	22.9	2.4	61.2	32.0

a : Each LOI value was compared to that of no additive

b : Each Dmc/g value was compared to that of DMMP.

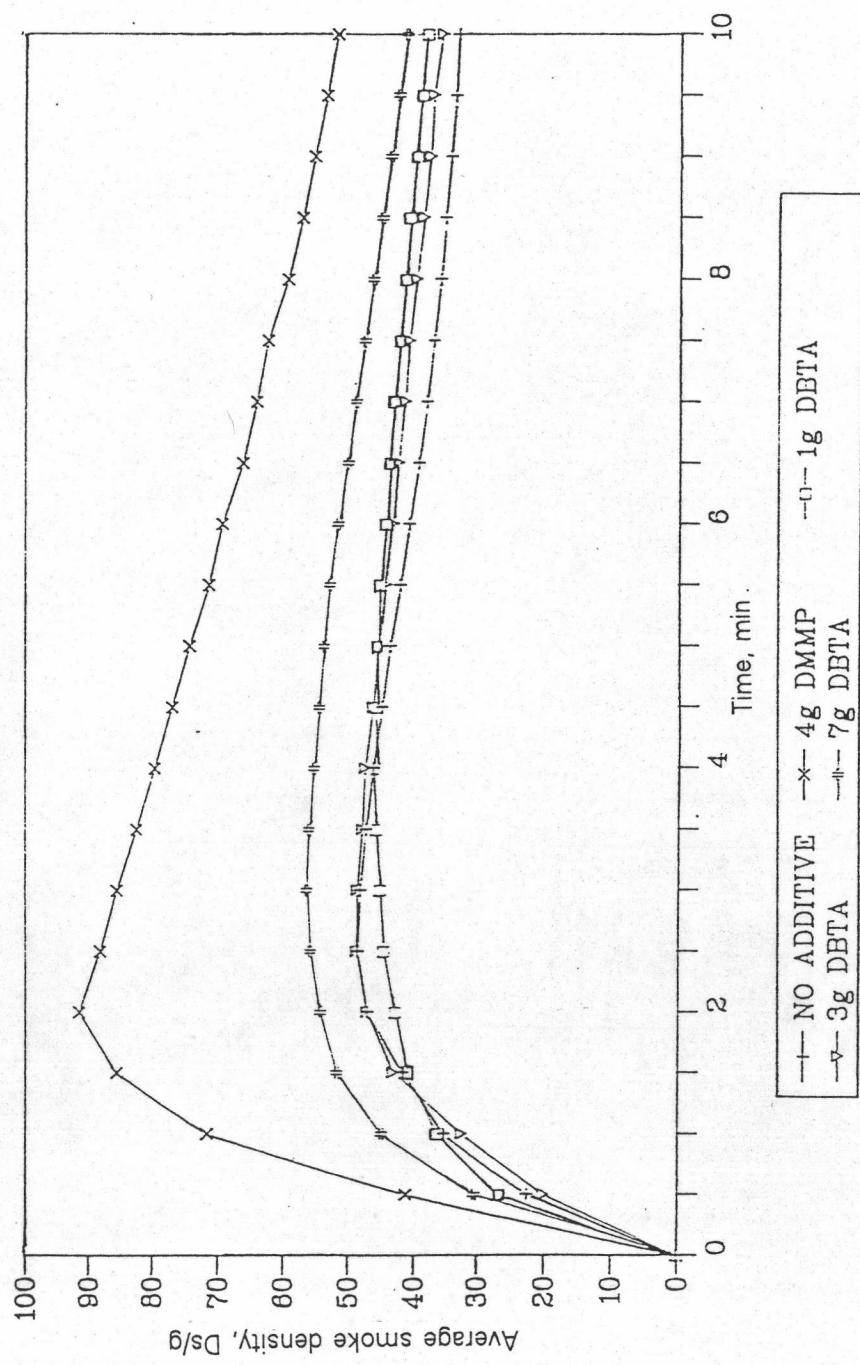


Figure 4-7 Average smoke density values of polyurethanes containing 10g C70 and 1, 3 or 7g DBTA compounds

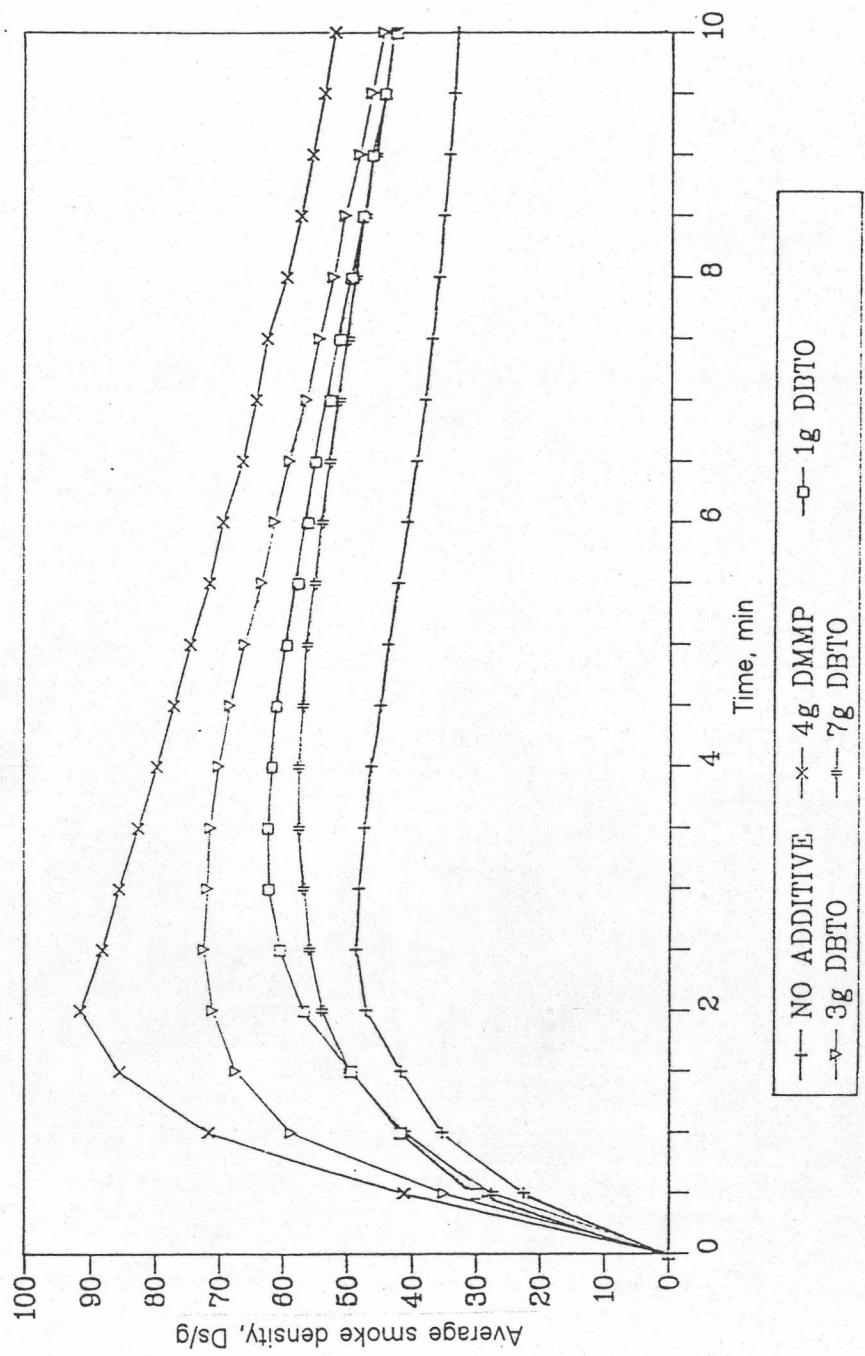


Figure 4-8 Average smoke density values of polyurethanes containing 10g C70 and 1, 3 or 7g DBTO compounds

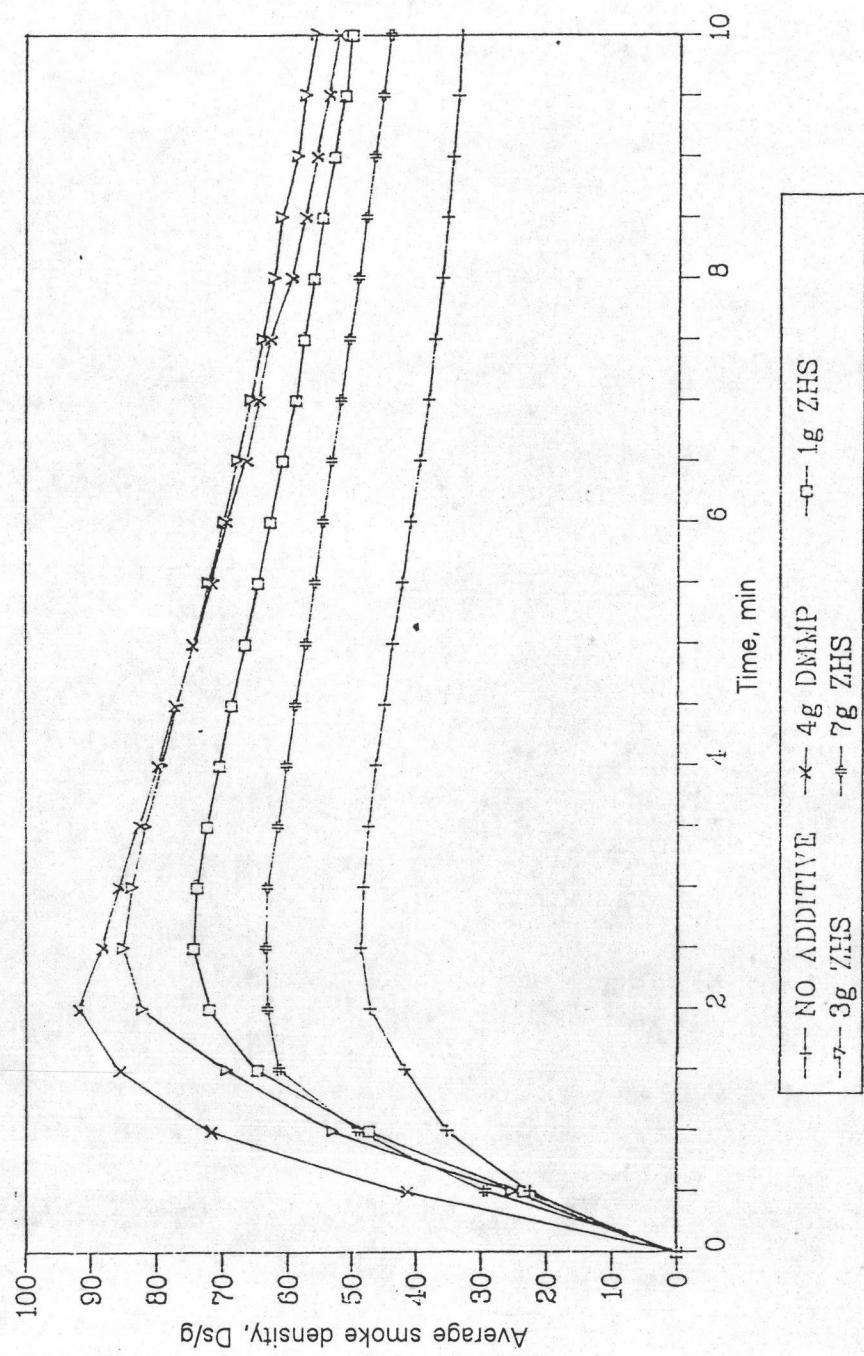


Figure 4-9 Average smoke density values of polyurethanes containing 10g C70 and 1, 3 or 7g ZHS compounds

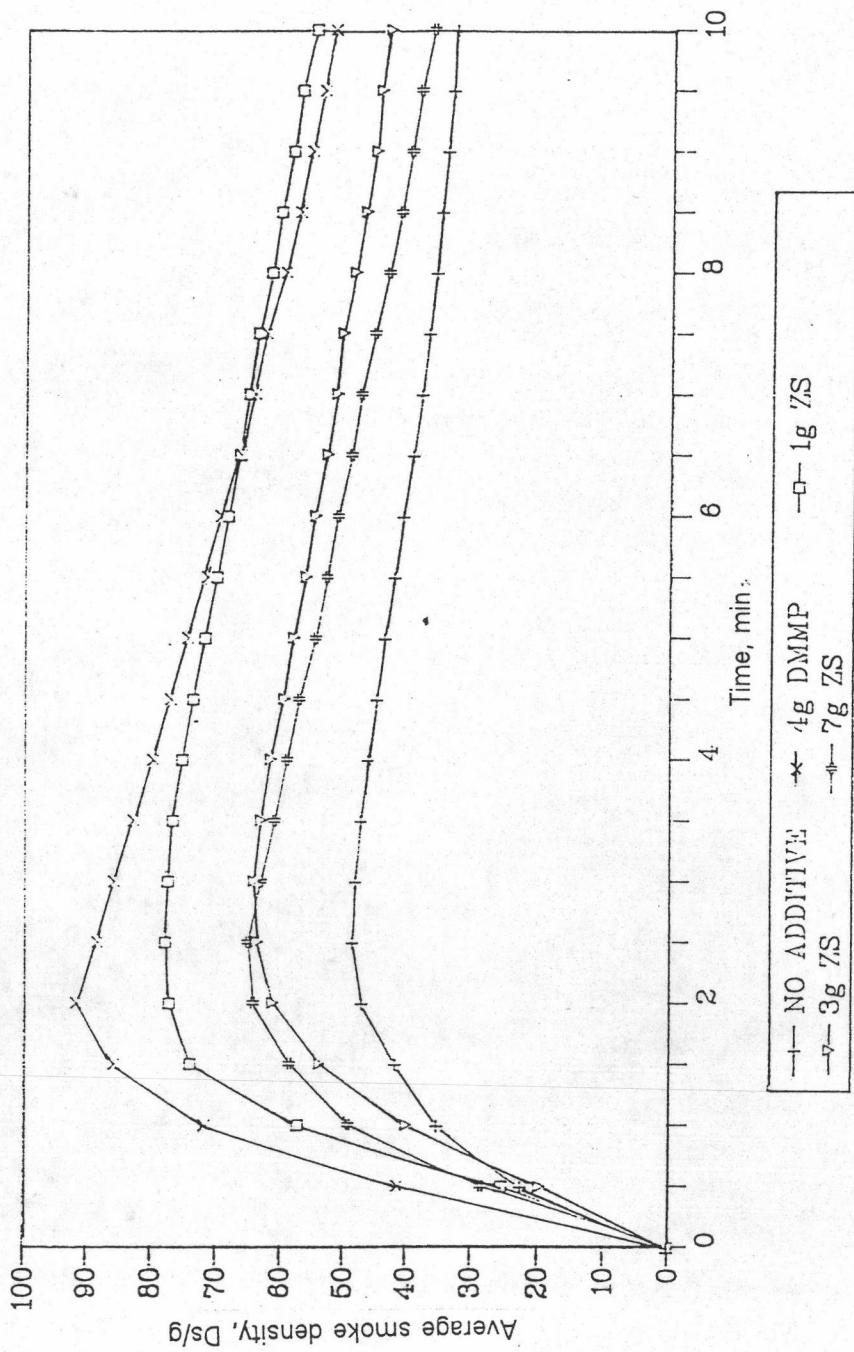


Figure 4-10 Average smoke density values of polyurethanes containing 10g C70 and 1, 3 or 7g ZS compounds

Table 4-6 The LOI and Dmc/g for the polyurethane containing tin and DBDPO additives.

Material	LOI	LOI ^a	Dmc/g	% smoke reduction ^b
No additive	20.5	-	34.7	55.3
DMMP	23.6	3.1	77.7	-
DBDPO	23.6	3.1	88.9	-14.4
DMMP/DBDPO	26.4	5.9	117.0	-50.6
1g DBTA/DBDPO	23.3	2.8	66.4	14.5
3g DBTA/DBDPO	24.2	3.7	87.5	-12.6
7g DBTA/DBDPO	24.3	3.8	106.4	-36.9
1g DBTO/DBDPO	22.9	2.4	99.9	-28.6
3g DBTO/DBDPO	22.3	1.8	88.5	-13.9
7g DBTO/DBDPO	23.1	2.6	85.8	-10.4
1g ZHS/DBDPO	21.8	1.3	64.0	17.6
3g ZHS/DBDPO	22.7	2.2	65.3	16.0
7g ZHS/DBDPO	22.6	2.1	57.8	25.6
1g ZS/DBDPO	22.0	1.5	70.3	9.5.6
3g ZS/DBDPO	22.9	2.4	67.3	13.4
7g ZS/DBDPO	22.9	2.4	47.9	38.4

a : Each LOI value was compared to that of no additive

b : Each Dmc/g value was compared to that of DMMP.

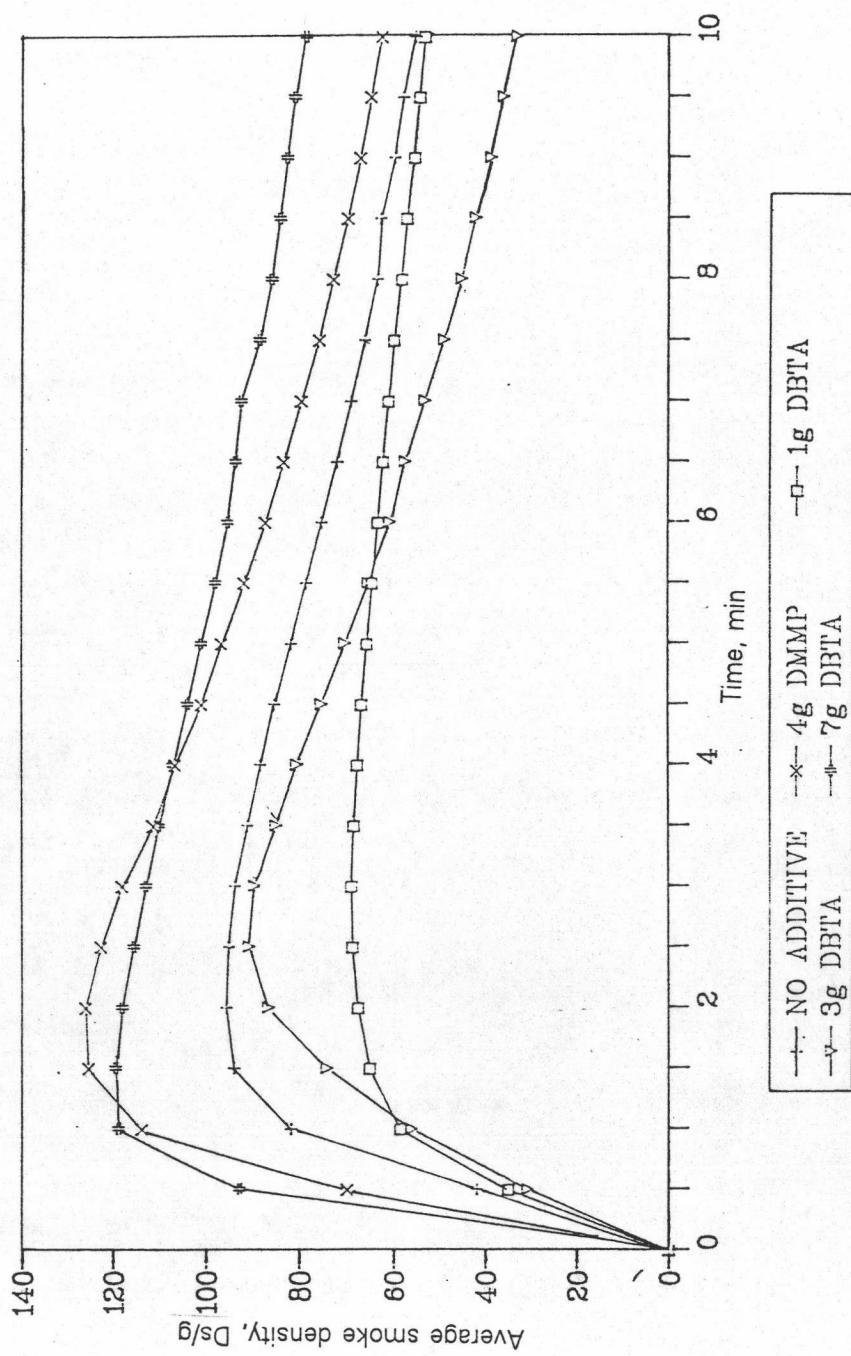


Figure 4-11 Average smoke density values of polyurethanes containing 10g DBDPO and 1, 3 or 7g DBTA compounds

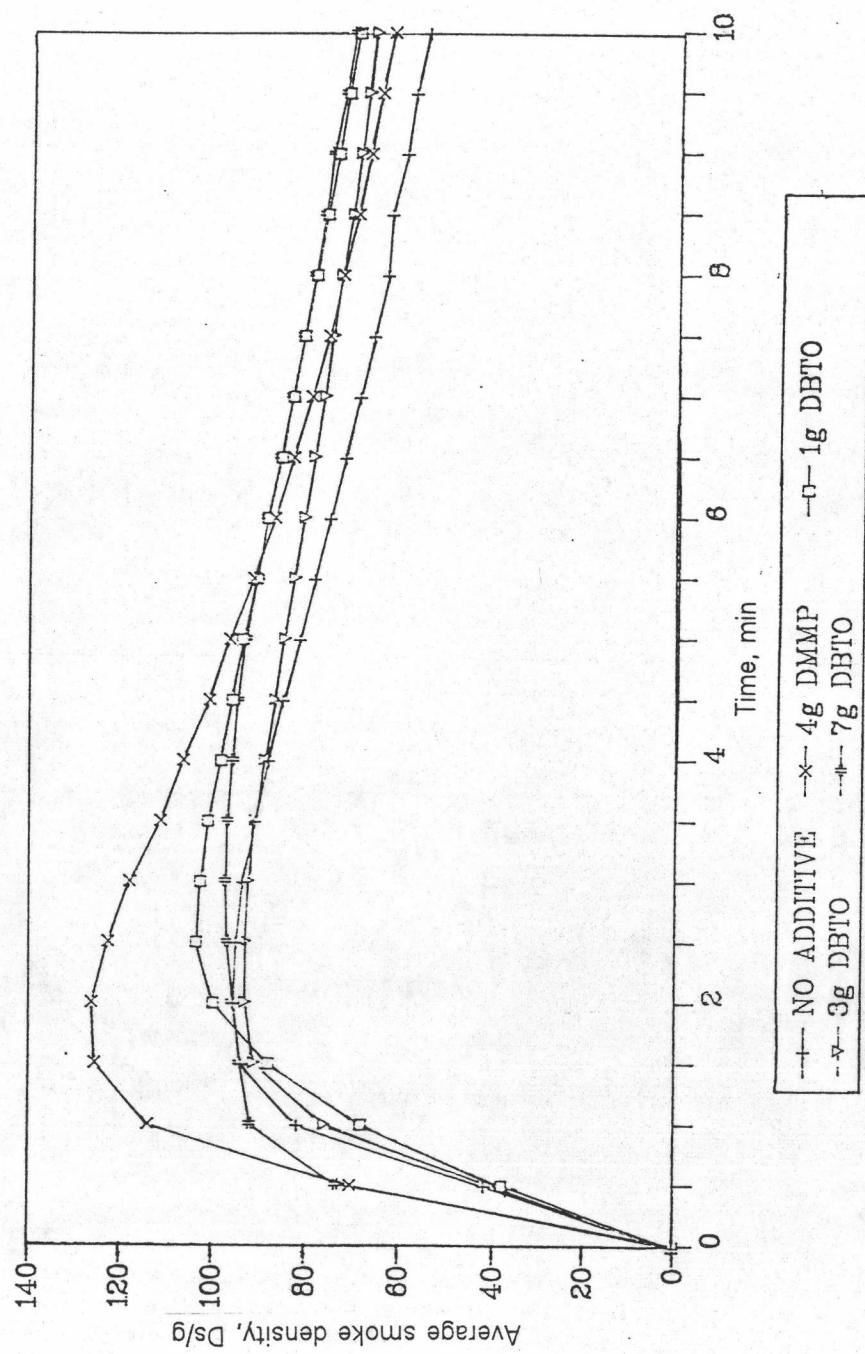


Figure 4-12 Average smoke density values of polyurethanes containing 10g DBDPO and 1, 3 or 7g DBTO compounds

Table 4-7 Thermal degradation of polyurethane containing tin and C70 additives.

Additive	Initial Degradation		Char Oxidation		%Residue at 1000°C
	%wt. loss	DTG _{max}	%wt. loss	DTG _{max}	
No additive	44.95	288.02	53.85	531.86	0.88
DMMP	43.30	289.58	55.56	537.27	1.14
C70	42.45	247.20	55.37	539.24	1.20
DMMP/C70	8.83 23.29 12.85	123.65 237.55 307.48	42.64	532.27	10.10
1g DBTA/C70	5.67 23.18 11.16 40.01	165.75 243.41 307.48	45.20	529.35	10.60
3g DBTA/C70	12.75 35.92 48.67	201.44 231.36	41.89	527.45	4.10
7g DBTA/C70	6.94 10.63 20.49 38.06	160.45 207.90 227.35	45.56	522.03	14.40
1g DBTO/C70	23.11 13.29 36.40	240.16 303.02	54.83	539.63	0.00
3g DBTO/C70	17.47 19.38 36.85	242.63 299.41	55.32	523.42	7.75

Table 4-7 Thermal degradation of polyurethane containing tin and C70 additives(cont).

Additive	Initial Degradation		Char Oxidation		%Residue at 1000°C
	%wt. loss	DTG _{max}	%wt. loss	DTG _{max}	
7g DBTO/C70	38.46	245.08	48.60	523.67	2.20
	12.36	308.51			
	50.82				
1g ZHS/C70	6.59	175.40	56.27	529.92	1.15
	35.78	246.08			
	42.49				
3g ZHS/C70	37.75	242.99	55.61	521.53	1.20
7g ZHS/C70	4.69	169.81	40.26	576.20	13.20
	19.65	243.98	8.20	576.27	
	12.42	292.74	48.46		
	36.76				
1g ZS/C70	36.82	243.63	51.59	521.63	8.00
3g ZS/C70	33.36	249.49	51.63	516.63	10.50
7g ZS/C70	32.74	246.08	42.26	521.63	9.33
			10.67	563.85	
			52.93		

Table 4-8 Thermal degradation of polyurethane containing tin and DBDPO additives.

Additive	Initial Degradation		Char Oxidation		%Residue at 1000°C
	%wt. loss	DTG _{max}	%wt. loss	DTG _{max}	
No additive	44.95	288.02	53.85	531.86	0.88
DMMP	43.30	289.58	55.56	537.27	1.14
DBDPO	48.57	296.71	47.13	515.31	2.78
DMMP/DBDPO	40.47	292.73	51.95	523.32	3.20
1g DBTA/DBDPO	3.96 11.87 25.82 41.65	164.27 235.39 304.59	52.92	525.74	0.52
3g DBTA/DBDPO	28.74 13.30 42.04	242.67 312.25	51.82	523.16	1.63
7g DBTA/DBDPO	1.01 9.70 34.38 45.09	176.74 215.70 248.21	45.26	5235.70	0.40
1g DBTO/DBDPO	34.29	294.43	50.59	528.23	10.67
3g DBTO/DBDPO	19.68 31.17 50.85	246.18 303.80	46.00	527.09	11.62
7g DBTO/DBDPO	25.06 14.45 39.51	246.48 303.80	48.89	524.86	16.47

Table 4-8 Thermal degradation of polyurethane containing tin and DBDPO additives(cont).

Additive	Initial Degradation		Char Oxidation		%Residue at 1000°C
	%wt. loss	DTG _{max}	%wt. loss	DTG _{max}	
1g ZHS/DBDPO	52.45	284.92	45.56	521.85	1.99
3g ZHS/DBDPO	34.54	300.35	42.37	519.62	9.90
			7.01	613.34	
			49.38		
7g ZHS/DBDPO	37.92	301.44	39.31	521.28	8.74
			10.56	590.83	
			49.87		
1g ZS/DBDPO	37.39	295.83	48.63	522.87	2.60
			4.56	598.44	
			53.19		
3g ZS/DBDPO	34.10	300.07	40.90	529.24	6.18
			7.10	600.01	
			48.00		
7g ZS/DBDPO	33.72	301.78	37.64	520.88	15.70
			9.49	592.53	
			43.13		

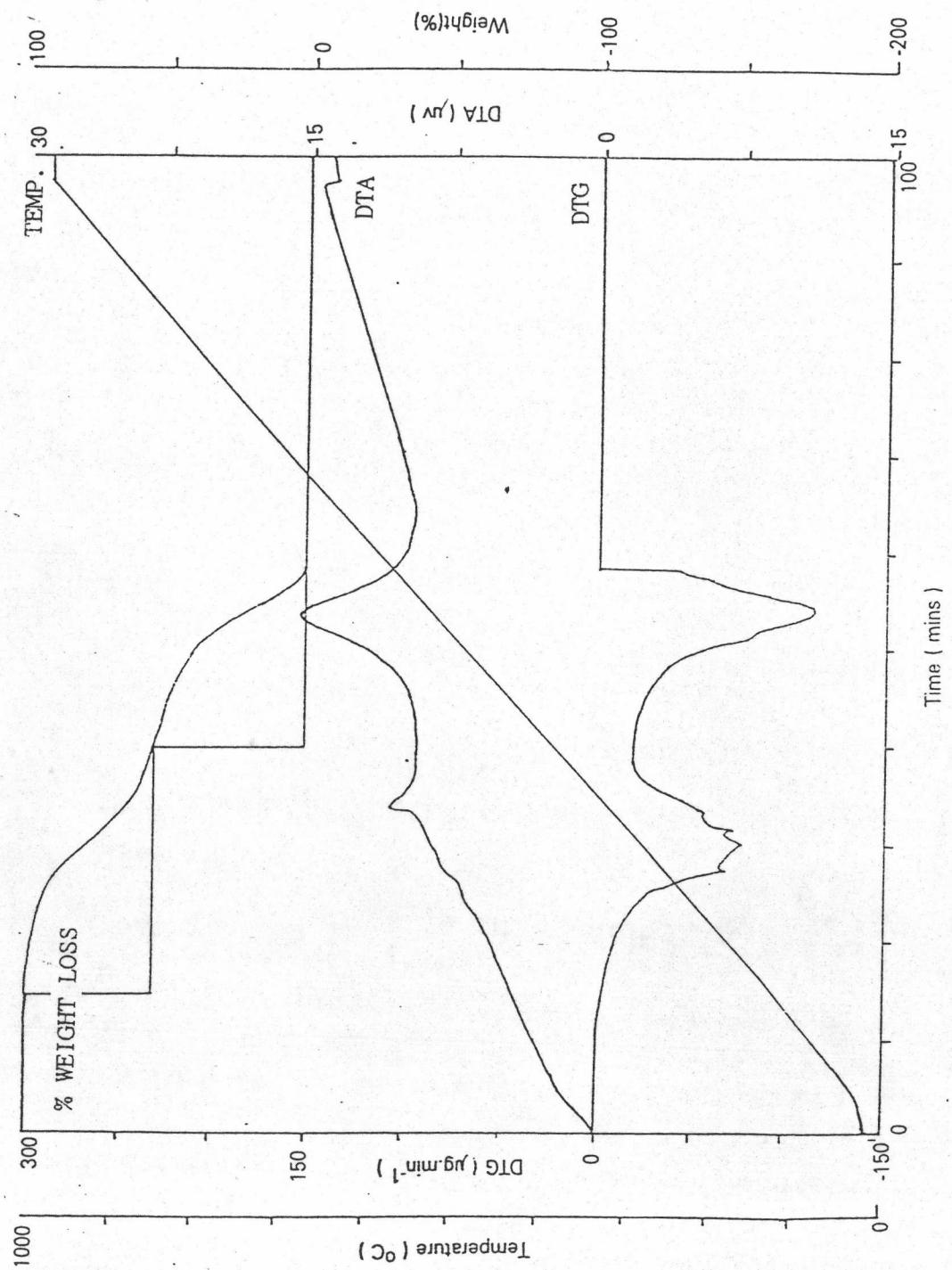


Figure 4-15 TGA and DTA curves for polyurethane

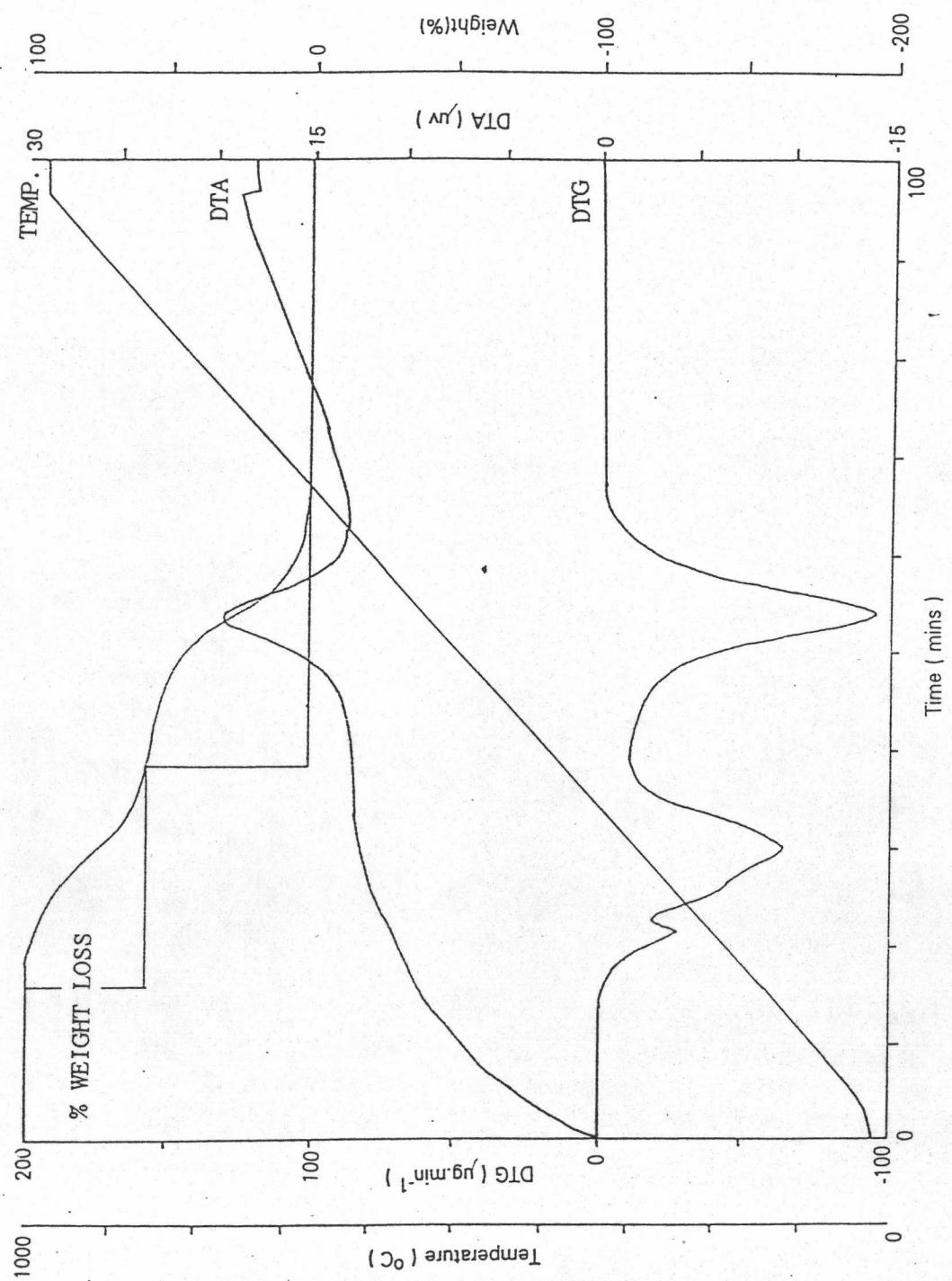


Figure 4-16 TGA and DTA curves for polyurethane containing DMMP

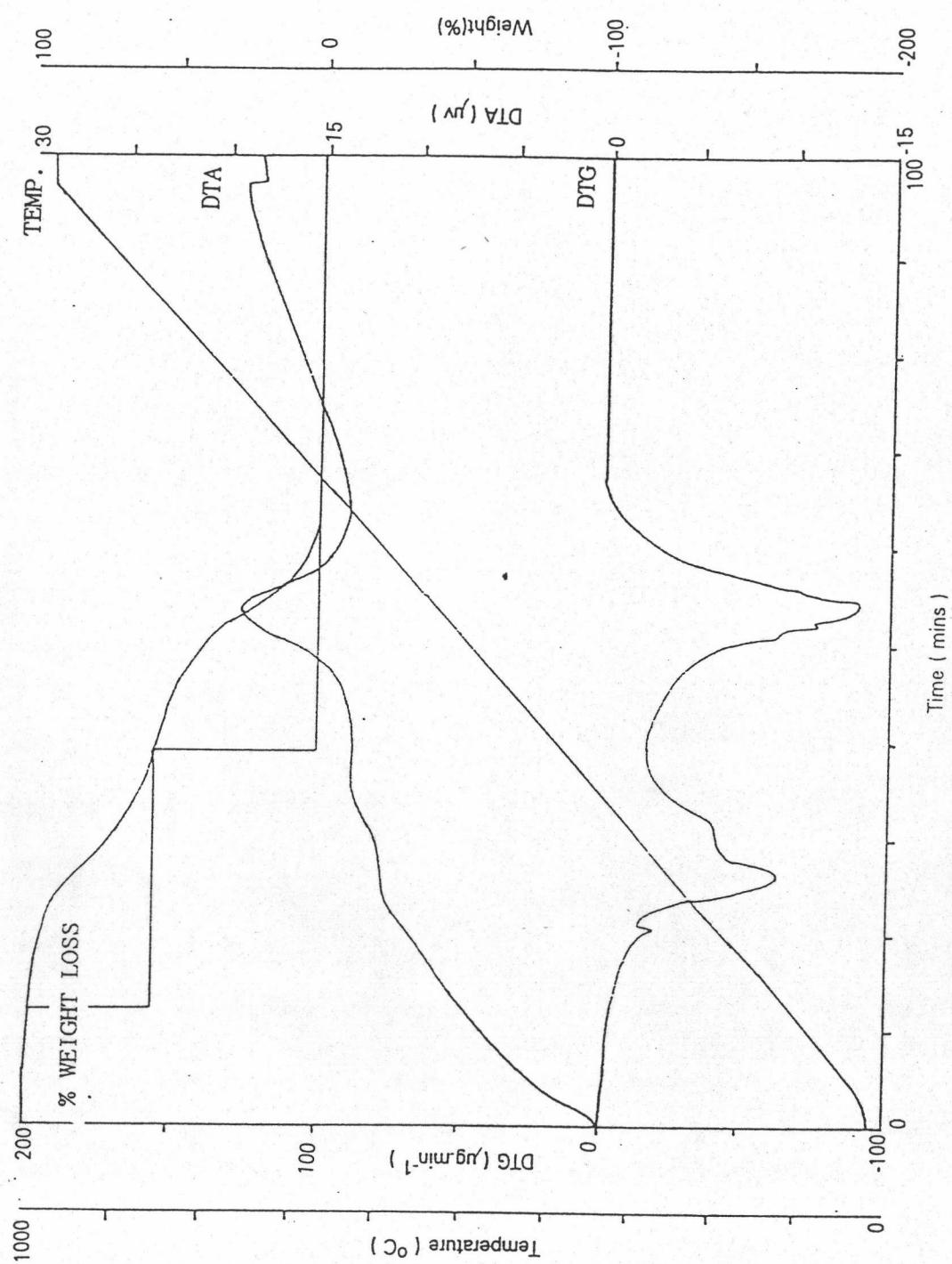


Figure 4-17 TGA and DTA curves for polyurethane containing C70

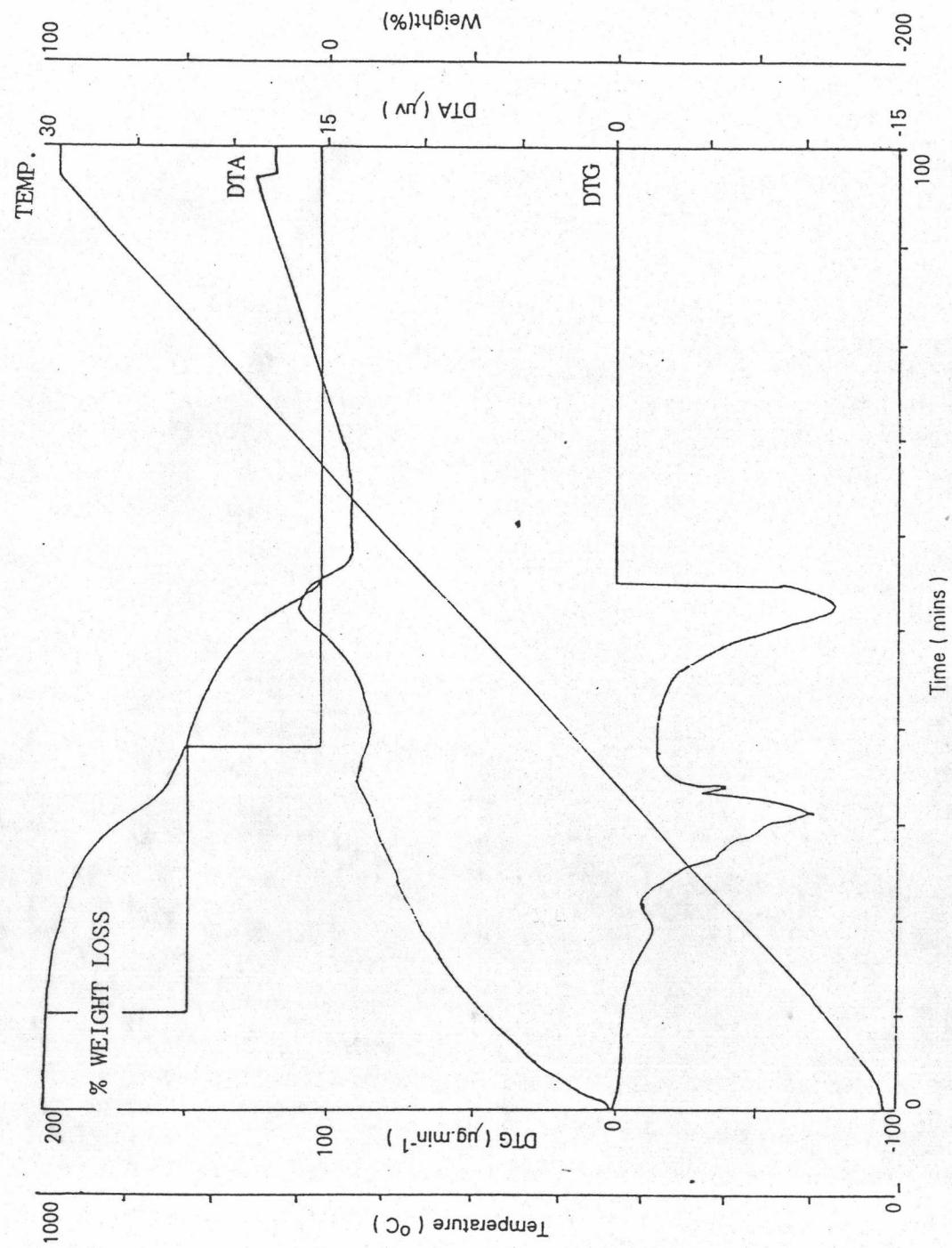


Figure 4-18 TGA and DTA curves for polyurethane containing DBDPO

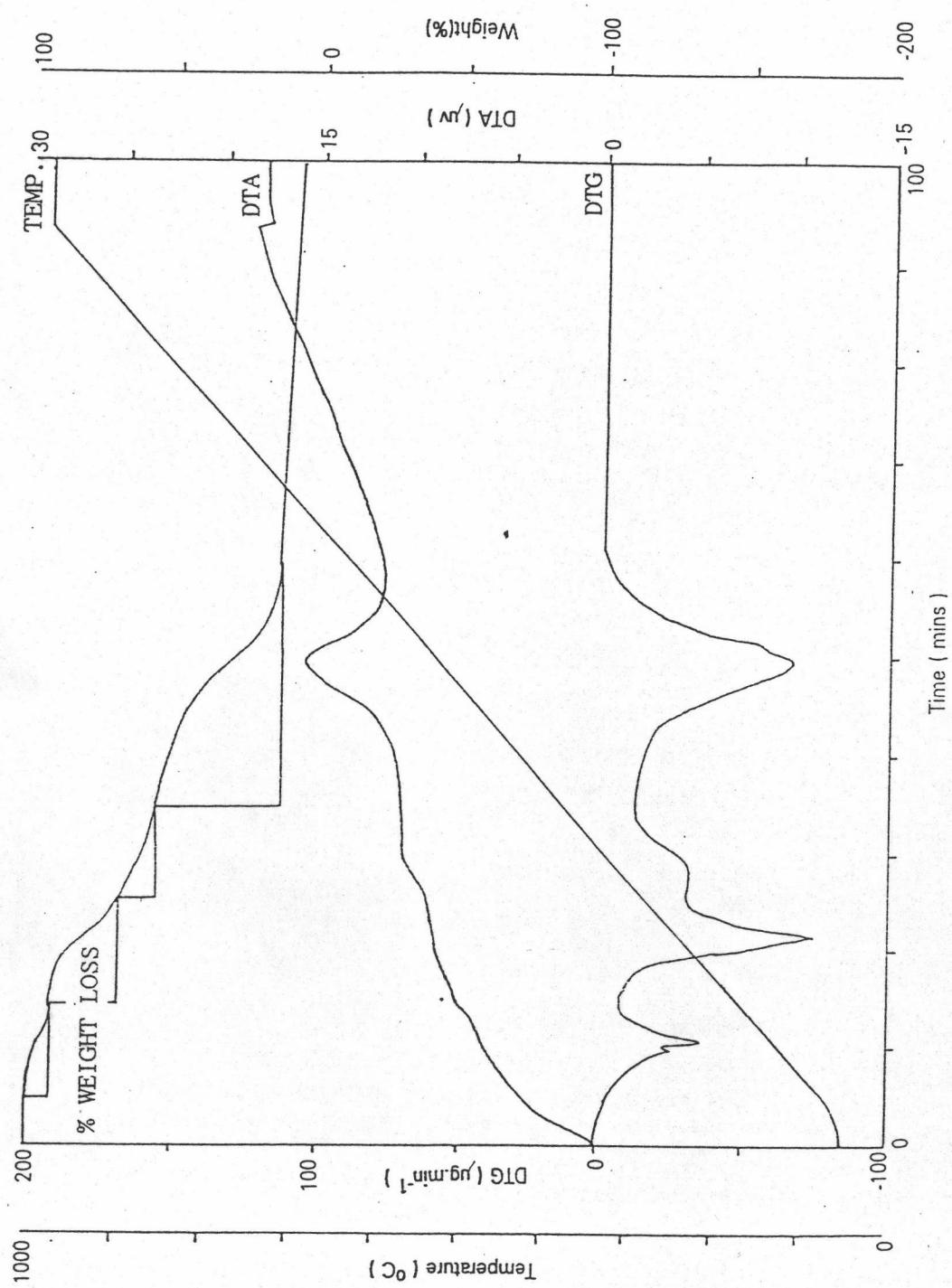


Figure 4-19 TGA and DTA curves for polyurethane containing DMMP/C70

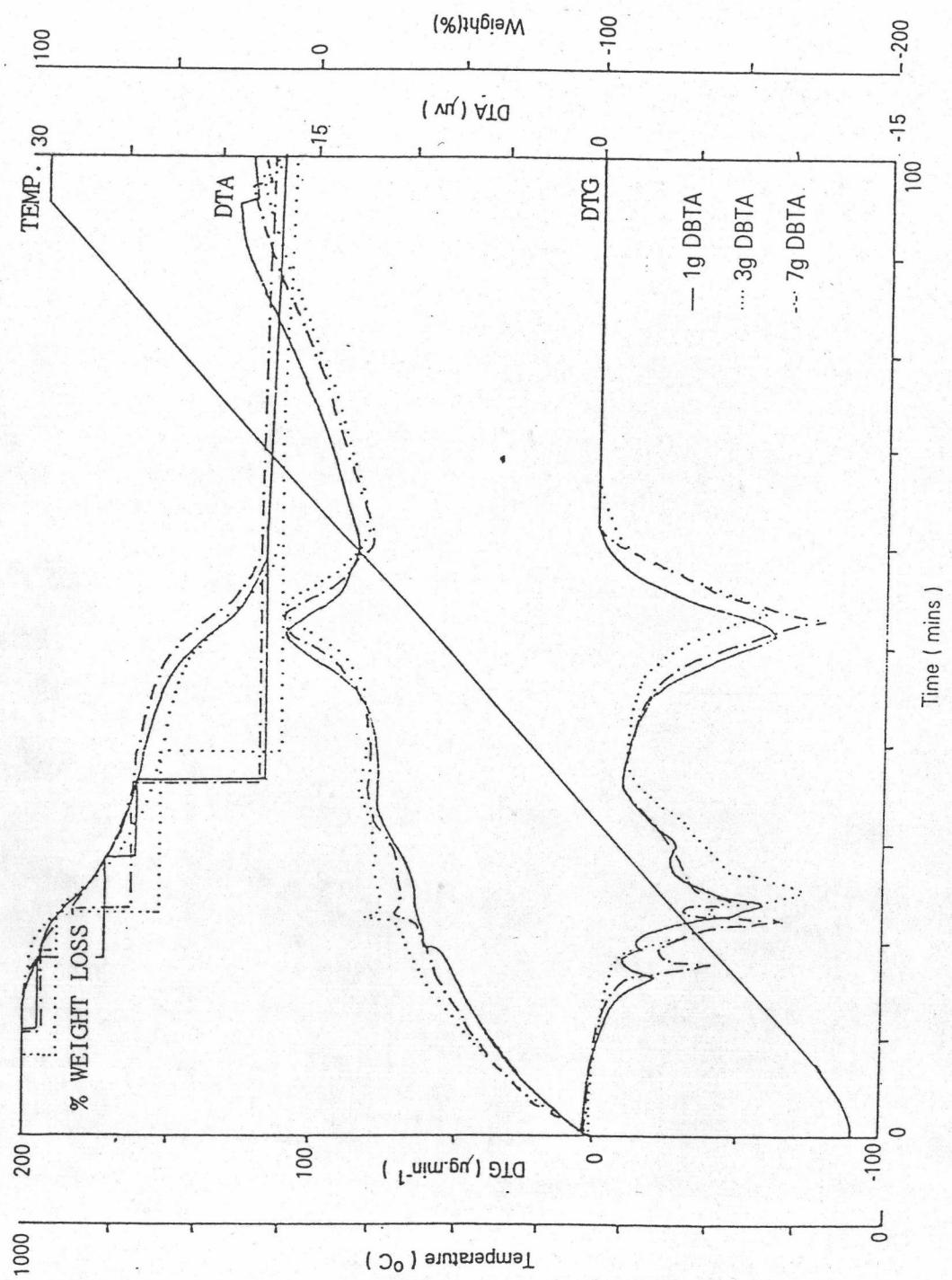


Figure 4-20 TGA and DTA curves for polyurethane containing DBTA/C70

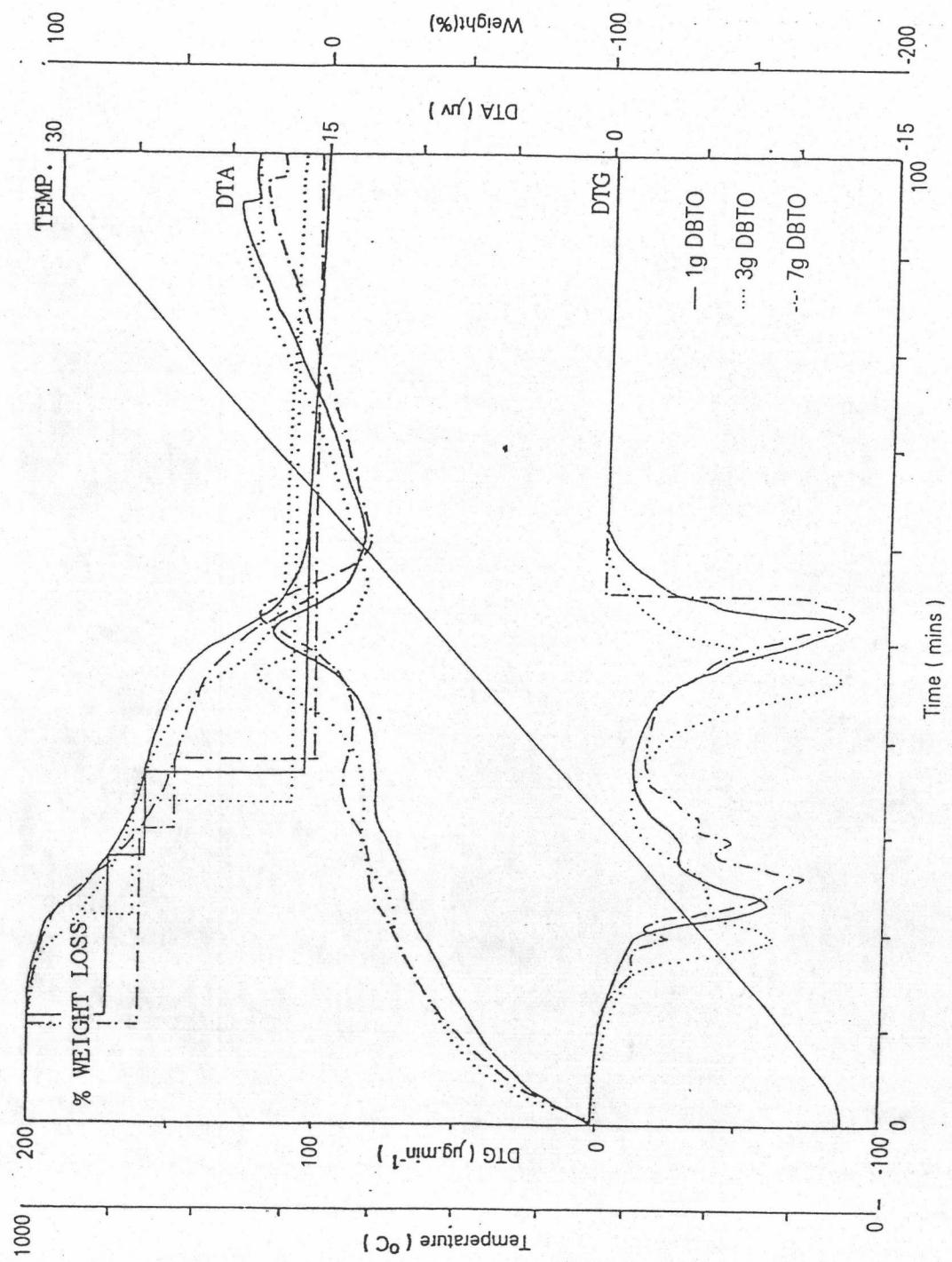


Figure 4-21 TGA and DTA curves for polyurethane containing DBTO/C70

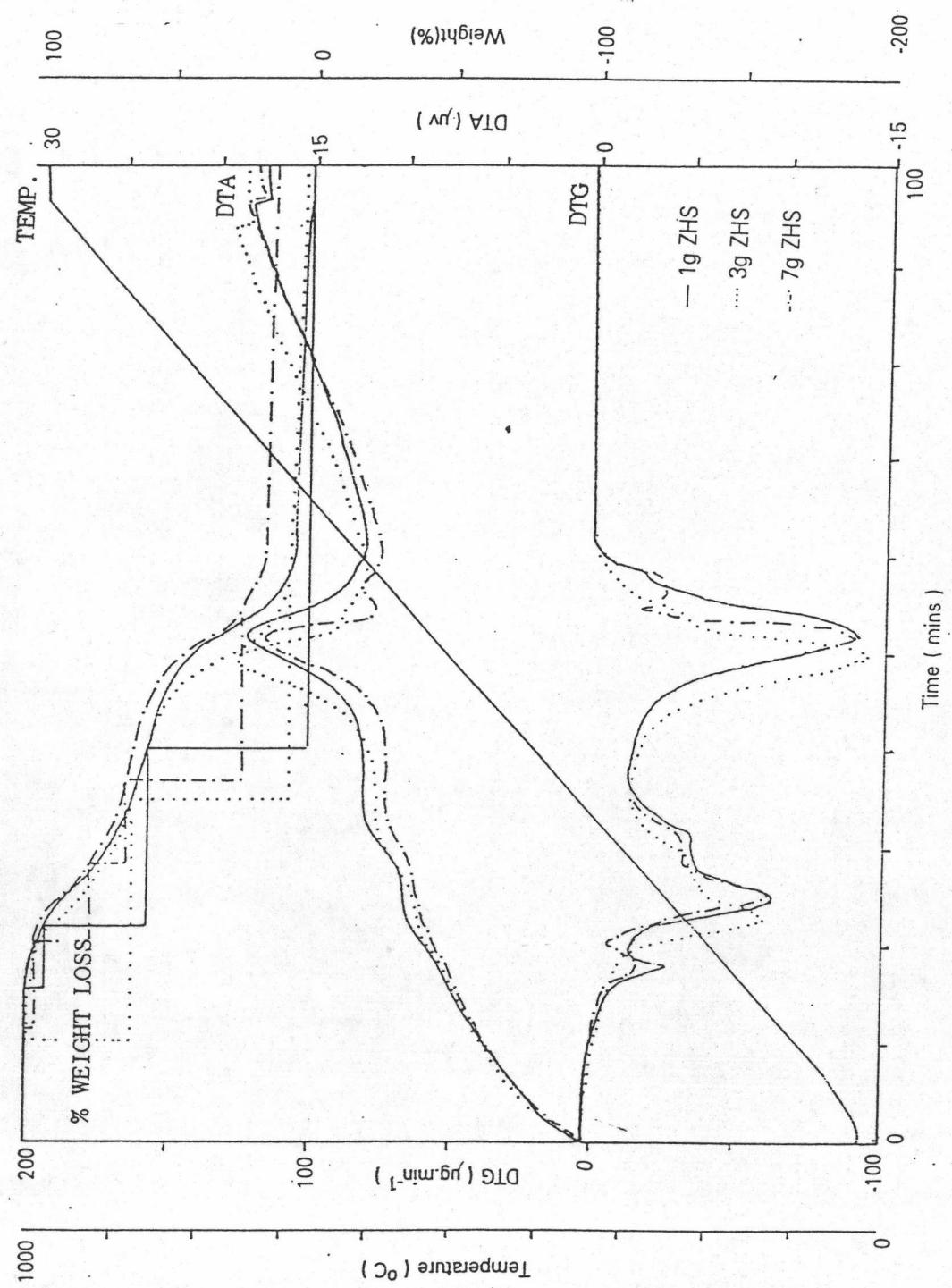


Figure 4-22 TGA and DTA curves for polyurethane containing ZHS/C70

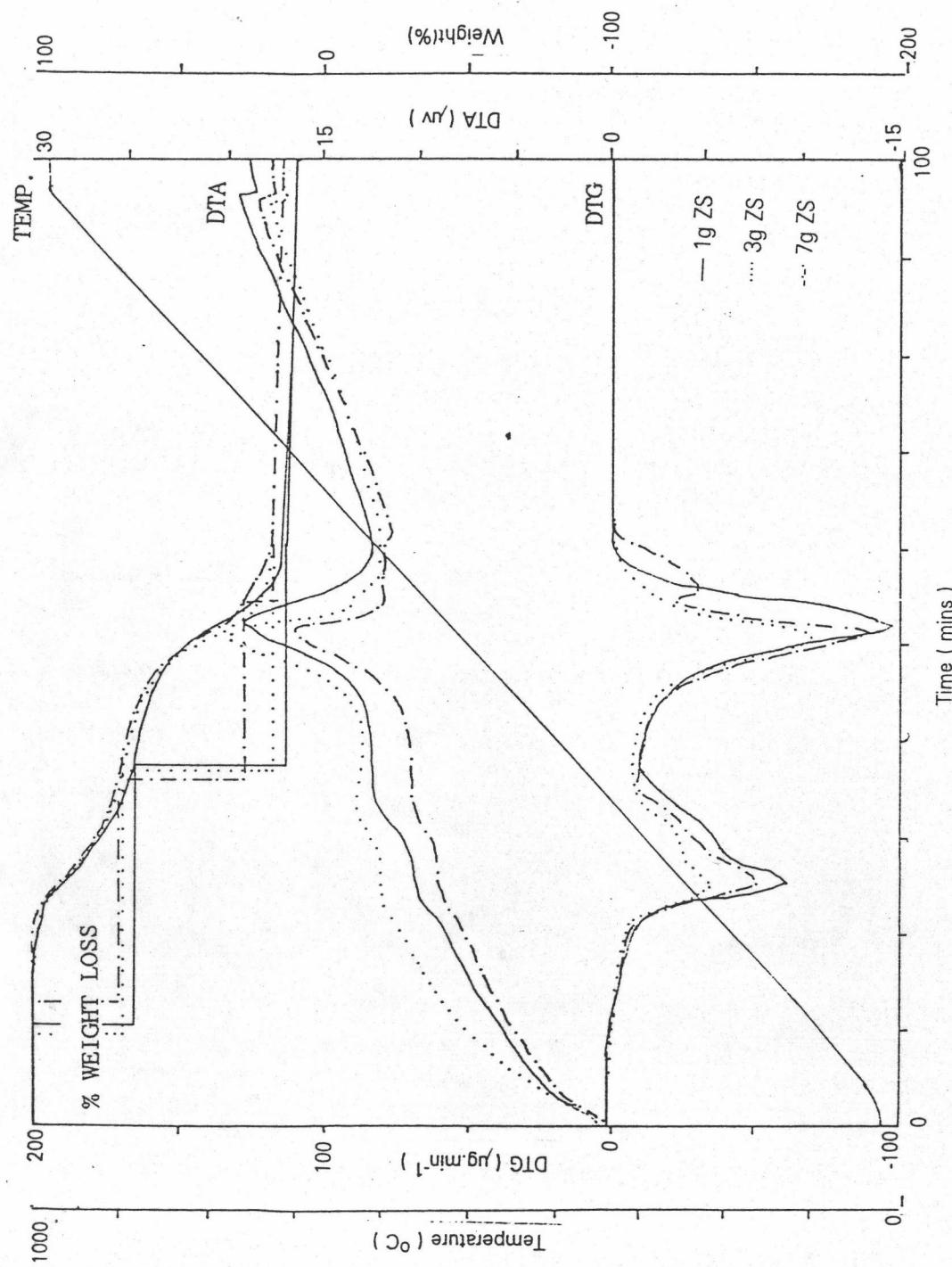


Figure 4-23 TGA and DTA curves for polyurethane containing ZS/C70

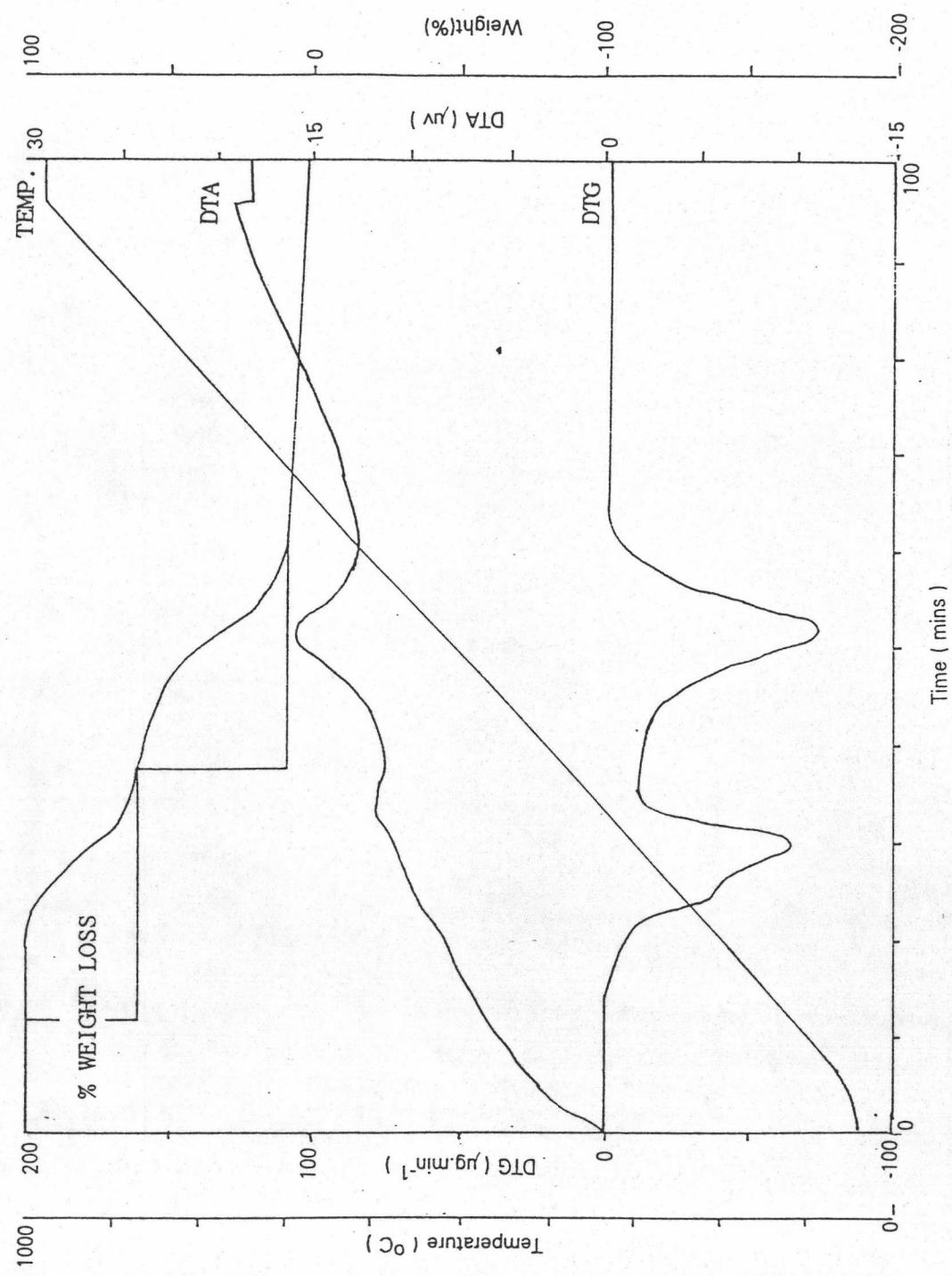


Figure 4-24 TGA and DTA curves for polyurethane containing DMMP/DBDPO

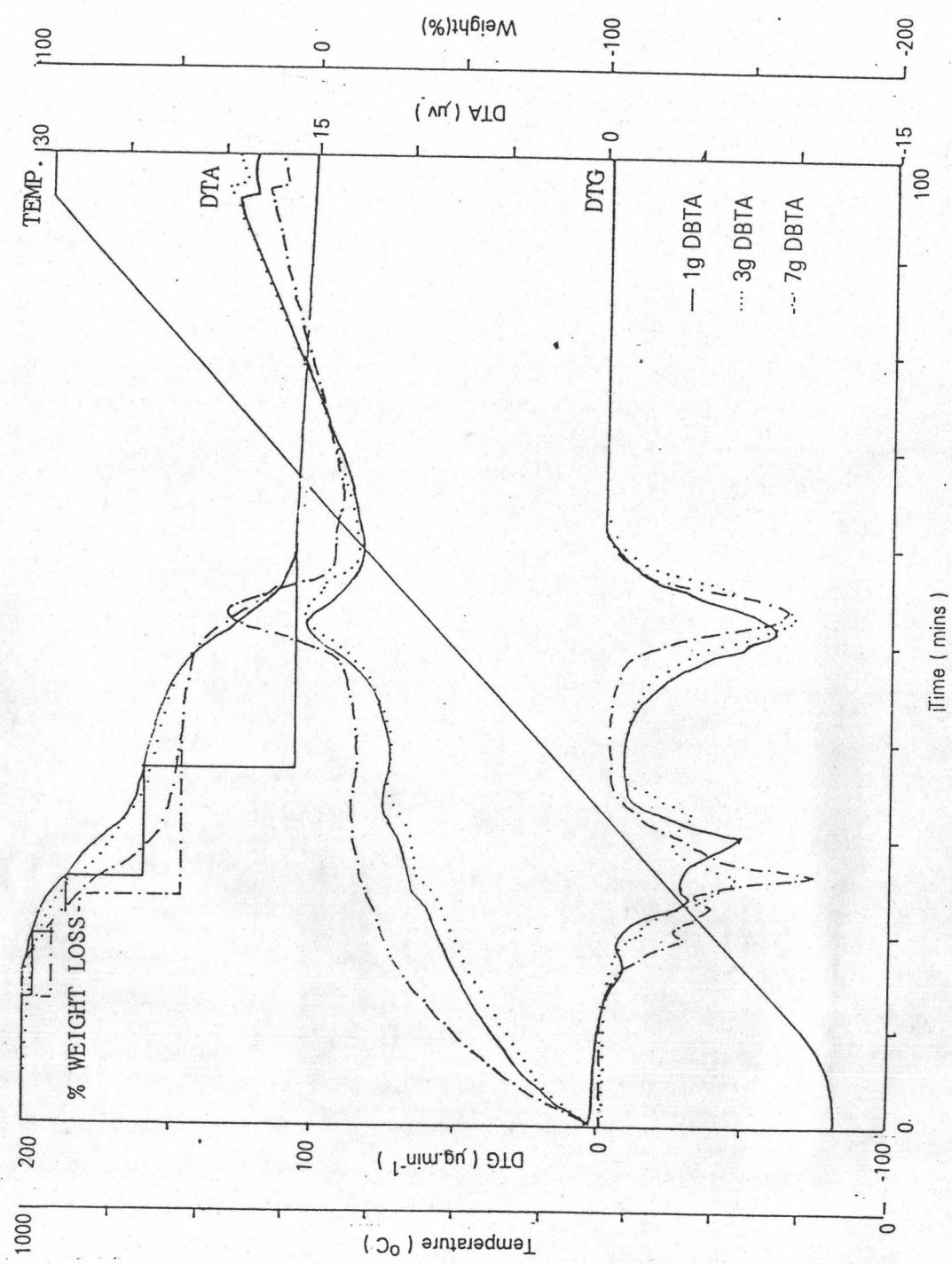


Figure 4-25 TGA and DTA curves for polyurethane containing DBTA/DBDPO

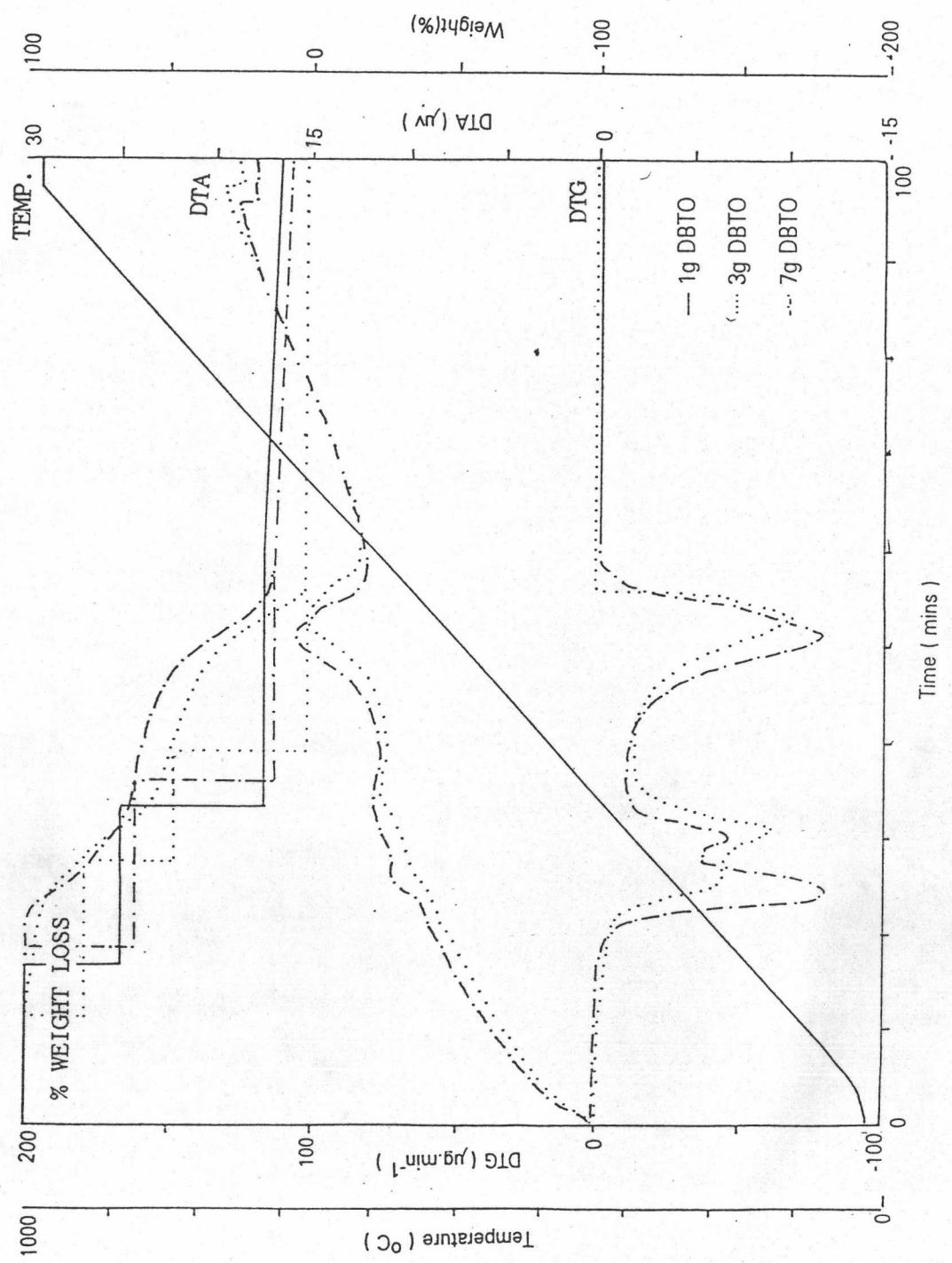


Figure 4-26 TGA and DTA curves for polyurethane containing DBTO/DBDPO

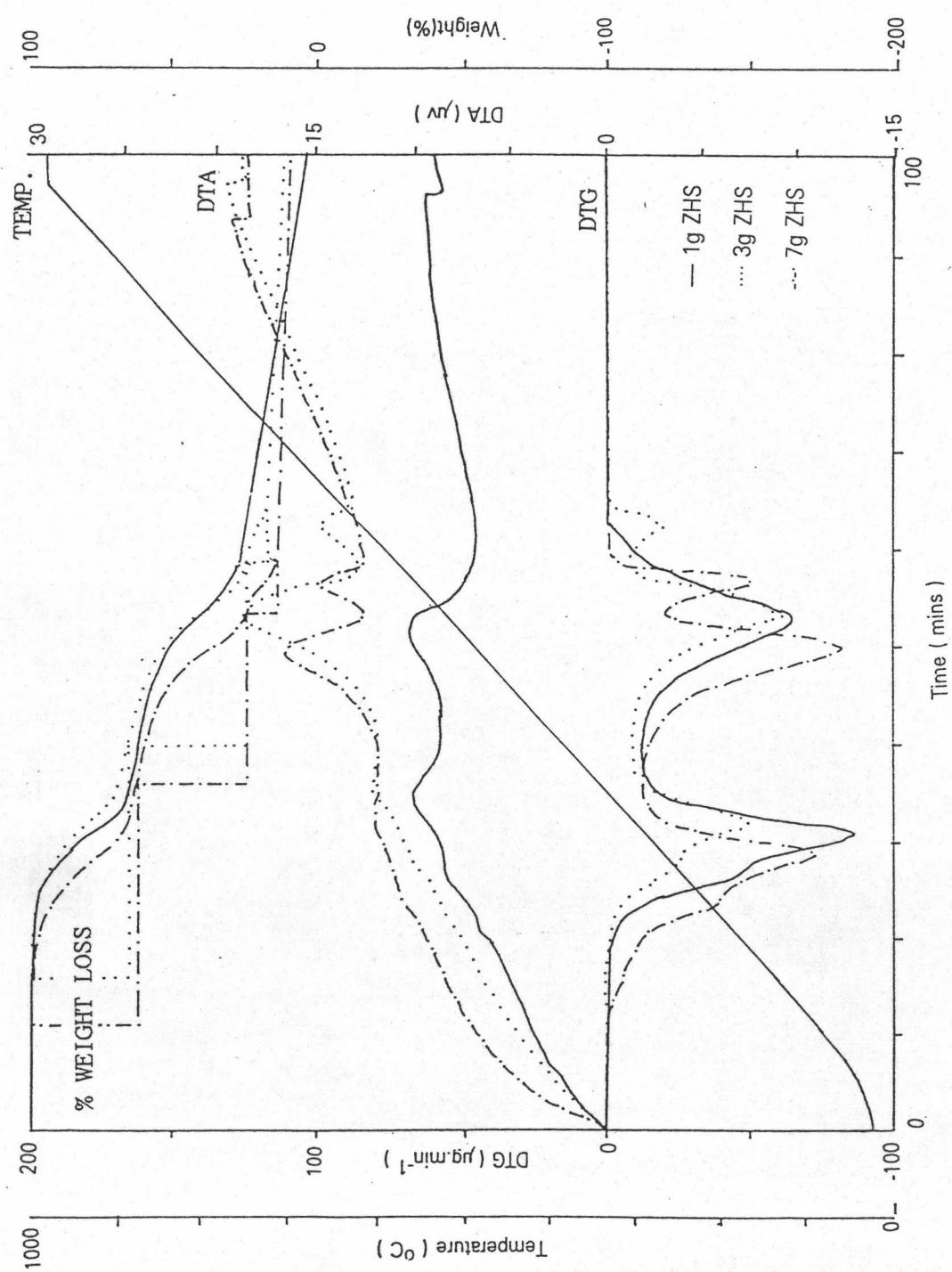


Figure 4-27 TGA and DTA curves for polyurethane containing ZHS/DBDPO

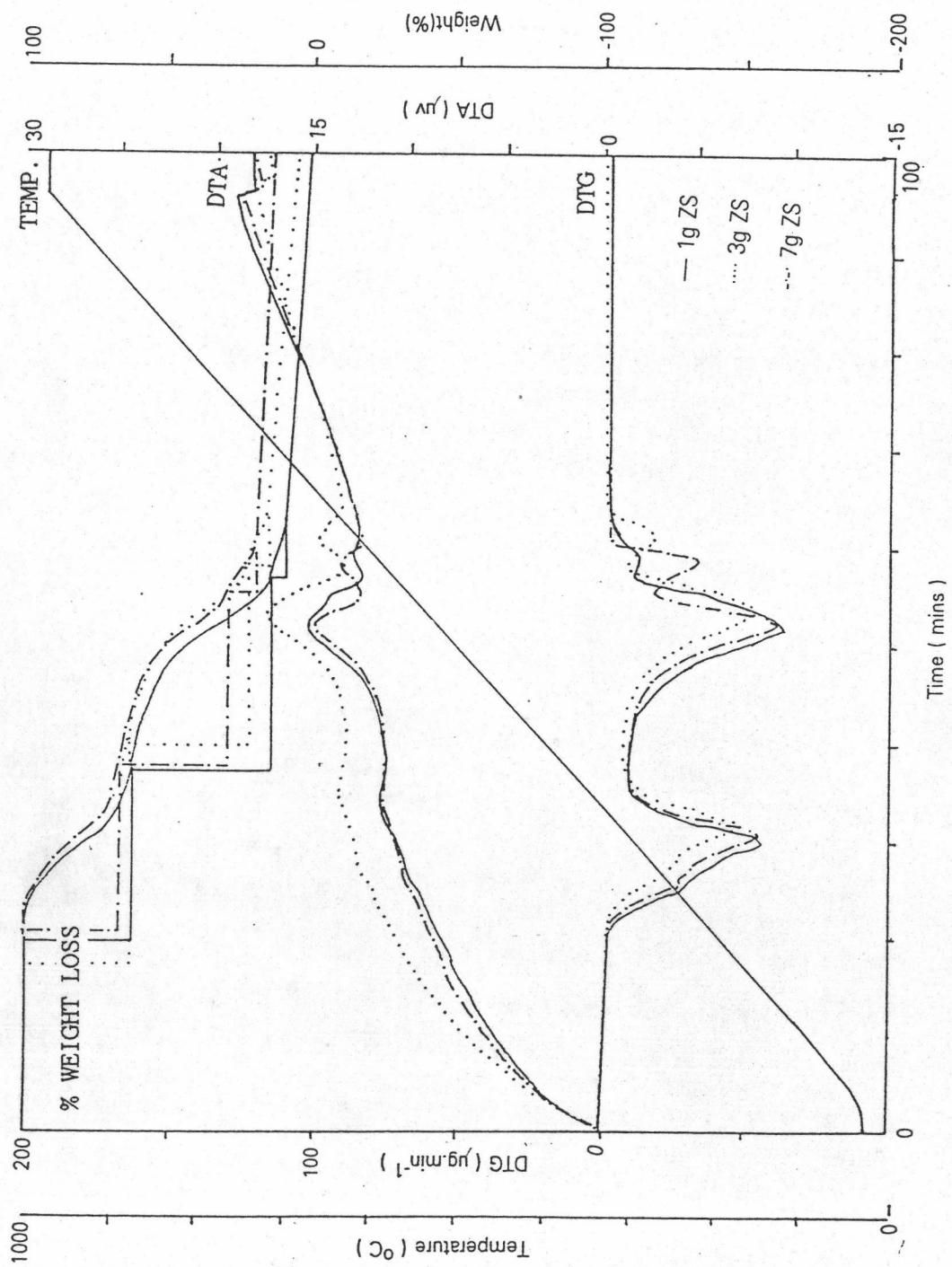


Figure 4-28 TGA and DTA curves for polyurethane containing ZS/DBDPO