

# Contents

PREFACE .....	5
CONTENTS .....	6
<b>1 INTRODUCTION TO THERMAL ANALYSIS .....</b>	<b>10</b>
1.1 DEFINITIONS.....	10
1.2 A BRIEF EXPLANATION OF IMPORTANT THERMAL ANALYSIS TECHNIQUES.....	11
1.3 APPLICATION OVERVIEW.....	13
1.4 THE TEMPERATURE PROGRAM.....	14
REFERENCES AND FURTHER READING.....	15
<b>2 A BRIEF HISTORY OF THERMAL ANALYSIS .....</b>	<b>16</b>
2.1 THERMAL ANALYSIS AT METTLER TOLEDO .....	17
REFERENCES AND FURTHER READING.....	18
<b>3 POLYMERS.....</b>	<b>19</b>
3.1 INTRODUCTION .....	19
3.2 SYNTHESIS OF POLYMERS.....	20
3.3 THERMOPLASTICS .....	22
3.4 THERMOSETS .....	24
3.5 ELASTOMERS.....	24
3.6 POLYMER ADDITIVES .....	26
3.7 USE OF THERMAL ANALYSIS TO CHARACTERIZE POLYMERS .....	26
REFERENCES AND FURTHER READING.....	27
<b>4 BASIC MEASUREMENT TECHNOLOGY.....</b>	<b>28</b>
4.1 DEFINITION .....	28
4.2 SENSITIVITY.....	28
4.3 NOISE .....	28
4.4 DETECTION LIMIT.....	29
4.5 DRIFT .....	29
4.6 TIME CONSTANT, LIMITING FREQUENCY.....	30
4.7 DIGITAL RESOLUTION AND SAMPLING INTERVAL .....	31
4.8 CALIBRATION AND ADJUSTMENT OF SENSORS.....	31
4.9 THE MOST IMPORTANT ELECTRICAL TEMPERATURE SENSORS .....	33
4.10 TEMPERATURES IN THERMAL ANALYSIS .....	34
<b>5 GENERAL THERMAL ANALYSIS EVALUATIONS.....</b>	<b>36</b>
5.1 THE OPTIMUM COORDINATE SYSTEM.....	36
5.2 EDITING DIAGRAMS.....	36
5.3 DISPLAYING INFORMATION FROM THE DATABASE .....	37
5.4 OPTIMIZING THE PRESENTATION OF A DIAGRAM .....	38
5.5 NORMALIZING MEASUREMENT CURVES TO SAMPLE MASS.....	38
5.6 DISPLAYING CURVES WITH RESPECT TO TIME, REFERENCE TEMPERATURE OR SAMPLE TEMPERATURE .....	39
5.7 SAMPLE TEMPERATURE AS A FUNCTION OF TIME.....	40
5.8 CURVE CORRECTION USING A BASELINE SEGMENT.....	40
5.9 MATHEMATICAL EVALUATIONS.....	41
5.10 CURVE COMPARISON .....	43

5.11	NUMERICAL EVALUATIONS .....	47
<b>6</b>	<b>GENERAL MEASUREMENT METHODOLOGY .....</b>	<b>51</b>
6.1	USUAL COORDINATE SYSTEMS OF DIAGRAMS .....	51
6.2	THE ATMOSPHERE IN THE MEASURING CELL .....	53
6.3	CRUCIBLES IN THERMAL ANALYSIS .....	57
6.4	OVERVIEW OF THERMAL EFFECTS .....	59
6.5	CALIBRATION AND ADJUSTMENT .....	61
	REFERENCES AND FURTHER READING .....	65
<b>7</b>	<b>DIFFERENTIAL SCANNING CALORIMETRY .....</b>	<b>66</b>
7.1	INTRODUCTION .....	67
7.2	DESIGN AND DSC MEASUREMENT PRINCIPLE .....	68
7.3	SAMPLE PREPARATION .....	75
7.4	PERFORMING MEASUREMENTS .....	77
7.5	INTERPRETATION OF DSC CURVES .....	79
7.6	DSC EVALUATIONS .....	92
7.7	SOME SPECIAL DSC MEASUREMENTS .....	128
7.8	DSC APPLICATION OVERVIEW .....	134
7.9	CALIBRATION AND ADJUSTMENT .....	135
7.10	APPENDIX: ASSESSING THE PERFORMANCE OF A DSC MEASURING CELL USING SIMPLE MEASUREMENTS .....	138
	REFERENCES AND FURTHER READING .....	142
<b>8</b>	<b>FAST SCANNING CALORIMETRY .....</b>	<b>144</b>
8.1	INTRODUCTION .....	144
8.2	DESIGN AND MEASUREMENT PRINCIPLE .....	145
8.3	SAMPLE PREPARATION .....	149
8.4	PERFORMING MEASUREMENTS .....	151
8.5	A TYPICAL APPLICATION .....	154
8.6	APPLICATION OVERVIEW .....	156
8.7	TEMPERATURE CALIBRATION .....	156
	REFERENCES AND FURTHER READING .....	157
<b>9</b>	<b>DIFFERENTIAL THERMAL ANALYSIS .....</b>	<b>158</b>
9.1	THE DTA MEASUREMENT PRINCIPLE .....	158
9.2	TYPICAL DTA CURVES .....	159
9.3	THE CALCULATION OF THE DSC CURVE FROM SDTA .....	160
9.4	THE SDTA EVALUATIONS IN THE STAR <sup>E</sup> SOFTWARE .....	161
	REFERENCES AND FURTHER READING .....	161
<b>10</b>	<b>THERMOGRAVIMETRIC ANALYSIS .....</b>	<b>162</b>
10.1	INTRODUCTION .....	162
10.2	DESIGN AND MEASURING PRINCIPLE .....	163
10.3	SAMPLE PREPARATION .....	166
10.4	PERFORMING MEASUREMENTS .....	167
10.5	INTERPRETING TGA CURVES .....	172
10.6	TGA EVALUATIONS .....	177
10.7	TYPICAL APPLICATION: RUBBER ANALYSIS .....	183
10.8	APPLICATION OVERVIEW .....	185

10.9	STOICHIOMETRIC CONSIDERATIONS.....	185
10.10	CALIBRATION AND ADJUSTMENT.....	185
	REFERENCES AND FURTHER READING.....	186
<b>11</b>	<b>THERMOMECHANICAL ANALYSIS.....</b>	<b>187</b>
11.1	INTRODUCTION.....	187
11.2	THE DESIGN AND MEASUREMENT PRINCIPLES OF A TMA.....	188
11.3	SAMPLE PREPARATION.....	192
11.4	TEMPERATURE PROGRAM.....	193
11.5	INTERPRETATION OF TMA CURVES.....	194
11.6	TMA EVALUATIONS.....	199
11.7	APPLICATION OVERVIEW FOR TMA.....	207
11.8	CALIBRATION AND ADJUSTMENT OF A TMA/SDTA.....	208
	REFERENCES AND FURTHER READING.....	209
<b>12</b>	<b>DYNAMIC MECHANICAL ANALYSIS.....</b>	<b>210</b>
12.1	INTRODUCTION.....	210
12.2	MEASUREMENT PRINCIPLE AND DESIGN.....	214
12.3	SAMPLE PREPARATION.....	220
12.4	PERFORMING MEASUREMENTS.....	221
12.5	INTERPRETATION OF DMA CURVES.....	223
12.6	DMA EVALUATIONS.....	235
12.7	DMA APPLICATION OVERVIEW.....	238
12.8	CALIBRATION OF THE DMA/SDTA.....	239
	REFERENCES AND FURTHER READING.....	239
<b>13</b>	<b>THE GLASS TRANSITION.....</b>	<b>241</b>
13.1	GLASSES AND THE GLASS TRANSITION.....	241
13.2	MEASUREMENT OF THE GLASS TRANSITION BY DSC.....	244
13.3	DETERMINATION OF THE DSC GLASS TRANSITION TEMPERATURE.....	247
13.4	PHYSICAL AGING AND ENTHALPY RELAXATION.....	249
13.5	THE GLASS TRANSITION FOR MATERIALS CHARACTERIZATION.....	250
13.6	OTHER THERMAL ANALYSIS TECHNIQUES FOR MEASURING THE GLASS TRANSITION.....	262
	REFERENCES AND FURTHER READING.....	267
<b>14</b>	<b>BINARY PHASE DIAGRAMS AND PURITY DETERMINATION.....</b>	<b>268</b>
14.1	INTRODUCTION.....	268
14.2	THE MOST IMPORTANT BINARY PHASE DIAGRAMS.....	269
14.3	THE USE OF THE TIE-LINE TO PREDICT DSC CURVES.....	272
14.4	CONSTRUCTING PHASE DIAGRAMS FROM DSC MEASUREMENTS.....	274
14.5	DSC PURITY DETERMINATION.....	276
	REFERENCES AND FURTHER READING.....	282
<b>15</b>	<b>POLYMORPHISM.....</b>	<b>283</b>
15.1	INTRODUCTION AND TERMS.....	283
15.2	DETECTION OF POLYMORPHISM.....	284
15.3	THE DSC INVESTIGATION OF THE POLYMORPHISM OF SULFAPYRIDINE.....	286
	REFERENCES AND FURTHER READING.....	286
<b>16</b>	<b>TEMPERATURE-MODULATED DSC.....</b>	<b>287</b>

16.1	INTRODUCTION.....	287
16.2	ISOSTEP® .....	287
16.3	ALTERNATING DSC.....	290
16.4	TOPEM® .....	294
	REFERENCES AND FURTHER READING .....	298
<b>17</b>	<b>EVOLVED GAS ANALYSIS .....</b>	<b>299</b>
17.1	BRIEF INTRODUCTION TO MASS SPECTROMETRY .....	300
17.2	BRIEF INTRODUCTION TO FOURIER TRANSFORM INFRARED SPECTROMETRY .....	300
17.3	BRIEF INTRODUCTION TO GAS CHROMATOGRAPHY .....	301
17.4	COUPLING THE TGA TO A GAS ANALYZER .....	301
17.5	EXAMPLES.....	303
	REFERENCES AND FURTHER READING .....	307
<b>18</b>	<b>TGA SORPTION ANALYSIS.....</b>	<b>308</b>
18.1	BRIEF INTRODUCTION TO TGA SORPTION ANALYSIS.....	308
18.2	EXAMPLES.....	309
18.3	CALIBRATION.....	312
18.4	TYPICAL APPLICATION AREAS .....	313
	REFERENCES AND FURTHER READING .....	313
<b>19</b>	<b>THERMOPTOMETRY.....</b>	<b>314</b>
19.1	INTRODUCTION.....	314
19.2	THERMOMICROSCOPY .....	314
19.3	CHEMILUMINESCENCE IN THERMAL ANALYSIS .....	318
19.4	CONCLUSIONS.....	322
	REFERENCES AND FURTHER READING .....	323
<b>20</b>	<b>METHOD DEVELOPMENT .....</b>	<b>324</b>
20.1	INTRODUCTION.....	324
20.2	STEP 1: CHOOSING THE RIGHT MEASUREMENT TECHNIQUE .....	326
20.3	STEP 2: SAMPLING AND PREPARATION OF THE TEST SPECIMEN .....	328
20.4	STEP 3: CHOOSING THE CRUCIBLE (DSC AND TGA).....	330
20.5	STEP 4: CHOOSING THE TEMPERATURE PROGRAM .....	330
20.6	STEP 5: CHOOSING THE ATMOSPHERE .....	332
20.7	STEP 6: EXAMINING THE TEST SPECIMEN AFTER MEASUREMENT .....	333
20.8	STEP 7: EVALUATION .....	333
20.9	STEP 8: VALIDATION .....	334
20.10	CONCLUSIONS.....	334
	REFERENCES AND FURTHER READING .....	335
<b>21</b>	<b>OVERVIEW OF STANDARD METHODS FOR THERMAL ANALYSIS .....</b>	<b>336</b>
<b>22</b>	<b>INDEX.....</b>	<b>347</b>