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DATA EVALUATION IN THERMAL ANALYSIS THE ON-LINE-SYSTEM METTLER TA3000

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INTRODUCTION

The automatic data evaluation of thermal analysis data has been an important objective of Mettler for quite a long time. The first instruments have been connected with Hewlett Packard table calculators of early generations /HP9810 and 9815/ as far back as 1973. These first programs underwent many improvements ; new programs were added, and today a large program library is incorporated in the TA Processor TC10A the control unit of the TA3000 System.

The TA3000 Thermal analysis system

The system consists of the TA Processor TC10A to which a DSC, TMA, or TG me suring module is added. The Print Swiss Martix Printer/Plotter connected to the TC10A serves as data output device. An IBM PC either Standard, XT or AT can be added and serves as data memory.

The measuring modules for DSC may be operated in the temperature range from ambient to 600 C /DSC20/, or from - 170 to 600 C /DSC30/. A new low noise signal amplifier together with the well-proven Au/Ni-Thermopile as DSC-sensor give an excellent signal to noise ratio that allow to record DSC curves with 0,1 mW f.s.d.

The TMA40 module for constant and dynamic load TMA can be operated in the range from-100 to 1000 C. Built-in magnet force compensation for precise load programming and controlling and a high resolution linear transformer for length measurements with detection limits /resolution/ of 4,5 nm are but some of the unique features of this module.

The TG50 module uses a Mettler microbalance M3-03 for thermogravimetry. An ingenious built-in gas-flow system enables analysis under inert or oxidising atmospheres are possible. The maximum temperature of the TG50 is 1000 C.

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The Software Package

The software of the TA3000 can be subdivided into the following sections :

1. General operating software
2. Module operating software
3. Evaluation software

The General Operating Software assures the dialogue between man and machine. It includes such possibilities as method storage and deletion. It standardizes signal outputs for easy connection of computers and the Matrix printer/plotter.

The Module Operating Software is used for the programming of the experiment. All relevant experimental parameters and module configuration are contained therein.

The Evaluation Software uses the measured and stored data to calculate results and plot curves and derivative curves. Table 1 shows the software package.

Examples

1. Peak Integration

Fig. 1 shows the melting of a Polyester blend. In the present example both peaks were integrated, using the special Integral Baseline /type 8/ that follows the baseline slope due to change of specific heat. The baseline follows exactly the extent of the reaction. Enthalpy change and peak top temperatures are calculated. The minimum sampling rate is one second, but the operator has the choice of using all or a limited number of data. The maximum storage capacity being 6000 data points divided over the time of the experiment.

2. Calculation of Kinetic Reaction

Based on the curing curve of an epoxy premix, Fig. 2 shows the program that calculates the order of reaction /n/, the activation energy /EA/, and the Frequency factor /ko/, using a multiple linear regression calculation. The MLR calculation also generates the 95% confidence limits of the calculated results. The Applied Kinetic program produces either a table of curing times, power produced, and the lnk as a function of different isothermal temperatures, or plots a diagram with three curves of the extent of curing at different isothermal temperatures. Also the adiabatic temperature increase may be calculated.

3. Step Program for Thermogravimetry

Fig. 3 shows a TG curve of a rubber decomposition with autometic gas exchange of nitrogen to air at 600 C. The step program recognizes automatically all weight steps, calculates weight the change in mg and percent, plots weight curve and derivative curve and prints steps and their temperatures. The computation of the first derivative allows an excellent filtering without curve distortion following an algorithmi described by Savitzky Golay.

CONCLUSION

The use of automatic data evaluation of thermal analysis data has proved extremely helpful. The increasing popularity of thermal analysis in the industry is largly due to easy data handling and in digitizing thermal information. But also for scientific work the high flexibility of the evaluation software has proved very handy.

REFERENCES

1 H. Wyden and G. Widmann, Experientis Supplementum, Vol. 37, /E. Marti et al, ed./ Birkhauser, Basel, 1979, p. 284.
Mettler Collected Applications on Plastics and Thermosets

General:	- Autoscaling of curves
	- First derivative
	- Autocalibration
	- Blank experiments
DSC:	- Peak integration
	- Purity calculation with ASTM plot
	- Content calculations (e.g. crystallinity)
	- Alpha / Liquid Fraction / Partial Area
	- Glass Transition temperatures
	- Specific Heat
	- Kinetic evaluations and applied kinetics
	- Onset temperatures (e.g. oxidation induction)
TMA:	- Coefficient of linear expansion
	- Glass Transition temperatures
	- Step calculations in percent and absolute
TG:	- Step calculations in percent and absolute
	- Kinetic evaluations and applied kinetics

Table 1

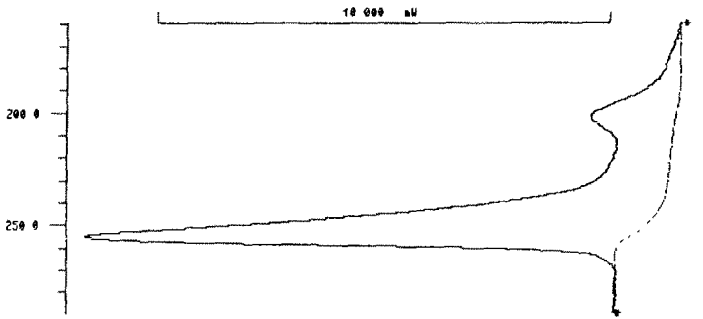
P E A K I N T E G R A T I O N

17 JAN 85 13:54

15 JAN 85 12:52 **

PEAK INTEGRATION	
DYN/ISO 1/2	1
AUTOLIMIT 0/1	0
START	160
END	290
START B. LINE	160
END B. LINE	290
BASELINE TYPE	8
PLOT CM	8
PLOT MODE	101
FILE NO.	00024.001
IDENT. NO.	6354
RATE K/MIN.	20
WEIGHT mG	13.52

TEMPERATURE °C HEAT FLOW
EXOTHERMAL-->



ΔH ENDO	mJ	765.60
ΔH	J/G	56.627
PEAK TEMP.	°C	252.9

********** METTLER TA3000 SYSTEM **********

Fig. 1

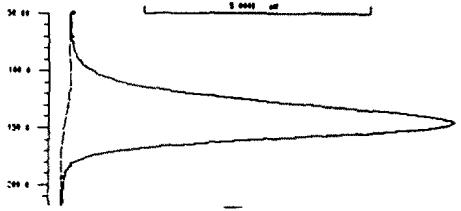
K I N E T I C A N A L Y S I S

17-JAN-95 15:36

15-JAN-95 12:52 **

KINETIC ANALYSIS	
DYN/ISO 1/2	1
START	36
END	229
BASELINE TYPE	8
ALPHA START	0.1
ALPHA END	0.8
PLOT CM	18
PLOT MODE	101
SINGLE ALPHA	0
APPLIED KINETICS	
ISO/AOI 1/2	1
PLOT CM	91
ΔT TABLE K	10
START TEMP °C	100
END TEMP. °C	200
ALPHA END	0.9
FILE NO.	00030.001
IDENT NO.	917
RATE K/MIN.	2
WEIGHT mG	24.9
END SCREEN °C	219.0

TEMPERATURE °C HEAT FLOW EXOTHERMAL-->



ΔH EXO	mJ	6455.0
ΔH	J/G	259.24
PEAK TEMP. °C		143.9
REACT ORDER		1.20
CONF. LIMIT		0.02
E A KJ/MOL		104.02
CONF. LIMIT		0.93
LN Ko		24.94
CONF. LIMIT		0.29

T °C		LN K	P W/KG	% MEM.
100.0	-	9.33	22.1	343.64
110.0	-	8.43	54.4	222.79
120.0	-	7.58	131.9	98.376
130.0	-	6.78	295.7	48.548
140.0	-	6.01	637.4	19.788
150.0	-	5.28	1288.0	9.4932
160.0	-	4.58	2443.1	4.7834
170.0	-	3.91	3186.3	2.4854
180.0	-	3.27	9807.5	1.2856
190.0	-	2.64	EEEE.E	.69716
200.0	-	2.05	EEEE.E	.38884

********** METTLER TA3000 SYSTEM **********

Fig. 2

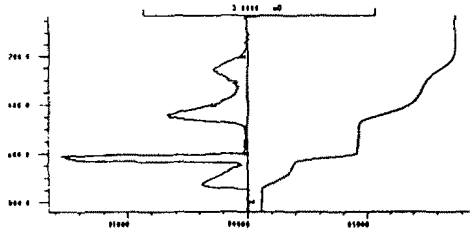
S T E P A N A L Y S I S

17-JAN-95 12:59

22-JUN-94 13:46 **

STEP ANALYSIS	
DYN/ISO 1/2	1
AUTOLIMIT 0/1	1
START	30
END	650
BASELINE TYPE	11
PLOT CM	8
PLOT MODE	2034
MOL MASS GAS	0
MOL MASS IMIT	0
FILE NO	00511.001
IDENT NO	22
RATE K/MIN	20
WEIGHT mG	5.4818
END SCREEN °C	845.3

TEMPERATURE °C WEIGHT GAIN----->



DTG mG/S

STEP			
START TEMP. °C		145.0	
PEAK TEMP. °C		354.0	
END TEMP. °C		519.0	
ΔH	mG	-1.44813	
ΔH	+/G	-11.9229	
STEP			
START TEMP °C		329.0	
PEAK TEMP °C		449.0	
END TEMP. °C		491.0	
ΔH	mG	-1.4284	
ΔH	+/G	-25.349	
ΔH	mG		-.81641
ΔH	+/G		-1.29388
STEP			
START TEMP. °C		340.0	
PEAK TEMP. °C		412.0	
END TEMP. °C		449.0	
ΔH	mG	-1.2991	
ΔH	+/G	-24.828	
STEP			
START TEMP. °C		447.0	
PEAK TEMP °C		738.0	
END TEMP °C		758.0	
ΔH	mG	-1.47587	
ΔH	+/G	-12.419	
ΔH	mG		-.87745
ΔH	+/G		-1.20444
RESIDUE			
TEMPERATURE °C		845.3	
ΔH	mG	1.3449	
ΔH	+/G	24.388	

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Fig. 3