Thermochimica Acta, 92 (1985) 359-362
Elsevier Science Publishers B.V., Amsterdam

NEWLY DEVELOPED THERMAL ANALYZER FOR THE SIMULTANEOUS CONTROL OF FOUR DETECTORS

M. Uchiike, K. Ito and M. Maruta
R/D Engineering Dept., Analytical Instruments Div.,
Shimadzu Corporation
1 Nishinokyo-Kuwabaracho, Nakagyo-Ku, Kyoto 604, Japan

INTRODUCTION

Thermal analysis is widely used in many fields, such as polymers, medicines, foods, electronic materials, ceramics and so on. But as it is a quite time-consuming technique, time and labour savings have been greatly demanded. Responding to the demands, we have developed the microprocessor controlled thermal analyzer, which can control 4 detectors and 4 flow controllers simultaneously and/or sequentially under independent measuring conditions. For example, parallel control of 4 detectors saves time and gives multifaceted data at once. Furthermore, this system has sufficient functions for thermal analysis, and can be used in various ways.

APPARATUS

Figure 1 shows the appearance of this system. The system of figure 1 consists of a system controller, detection modules such as the differential thermal analyzer, a recorder, and a flow controller. While 4 recorders can be connected with each amplifier to record all data in solid lines, a recorder with pen-lift capability can be used to record signals of all 4 detectors by way of time-sharing in the form of dotted lines. Further, all signals can be sent in real time to other computers through RS-232-C interface.

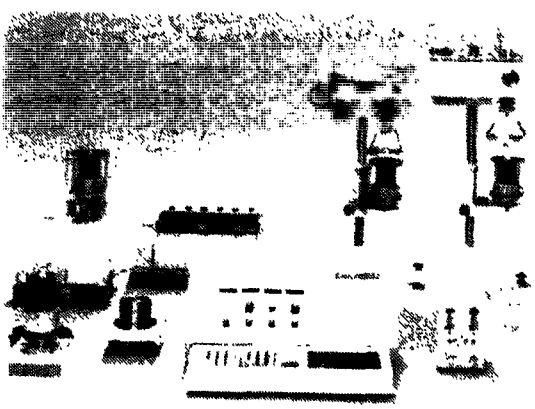

Fig. 1

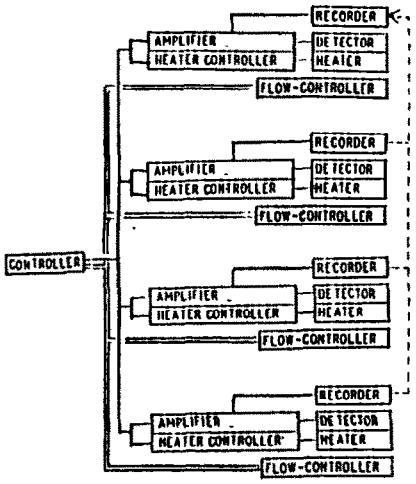

Fig. 2

Proceedings of ICTA 85, Bratislava

4-CHANNEL CONTROL

At present, five kinds of detector modules, namely DTA, DSC, TG, TMA and DTA-TG simultaneous measuring apparatus, are provided as detection modules for this system. One controller can control four of the above detector modules individually and simultaneously.

Figure 3 shows an example of simultaneous control data on epoxy resins, using 8-pen recorder.

TGA : pyrolysis of epoxy resin
TMA : expansion and glass transition of epoxy resin
DSC : polymerization of epoxy resin
DTA : glass transition of epoxy resin

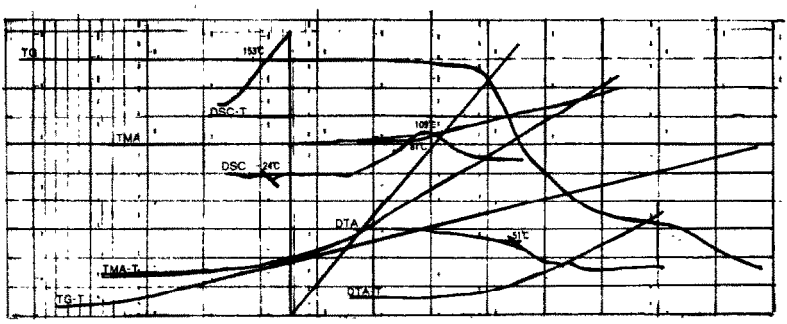

Fig. 3

The analytical conditions are shown in Table 1. In this case TG analysis was started first, followed by the TMA, DSC and DTA analyses. The succession is shown in Fig. 3. The four modules were heated according to their temperature programs, and the measurements ended when their temperatures reached the set points. Gathering those data using conventional thermal analyzers is a very time-consuming process, however, with this new system, the analysis time is drastically reduced.

	TG	TMA	DSC	DTA
SAMPLE	EPOXY RESIN	EPOXY RESIN	EPOXY RESIN	EPOXY RESIN
	14.89mg	10.47mm	25.82mg	40.22mg
ATMOSPHERE	AIR 80mℓ/min	AIR 50mℓ/min	N_2 50mℓ/min	AIR 50mℓ/min
HEATING RATE	10°C/min	5°C/min	10°C/min	10°C/min
AMP. RANGE	20mg	50μm	100mj/sec	500μV
TEMP. RANGE	1000°C	200°C	200°C	500°C
CHART SPEED	10mm/min	10mm/min	10mm/min	10mm/min

TABLE 1

Fig. 4 shows two sets of data in which dehydration of absorbed and crystalline water bonded to quinine sulfate was measured and recorded on chart paper using the dotted line time-sharing mode. The measurement conditions are shown in Table 2. Figure 4 clearly shows that sample weight drastically affects the behaviour of the dehydration process.

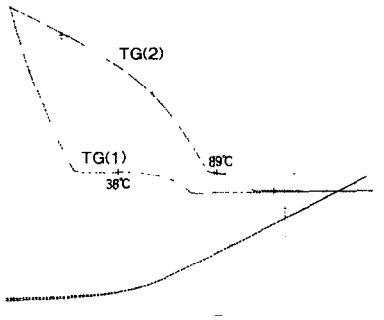

Fig. 4

As four of the same-type of detector can be controlled simultaneously, it is possible to execute efficiently measurements by changing conditions, such as sample weight, heating rate, atmospheric gas, and so on.

This system has another function, that is, sequential control of 4 detector modules. The sequential control means that the modules can start automatically in the order of the sequence selected by the operator. This function is suitable for overnight non-stop measurements.

	TG(1)	TG(2)
SAMPLE	QUININE SULFAS	QUININE SULFAS
	4.17mg	39.3mg
ATMOSPHERE	AIR 50m /min	AIR 50m /min
HEATING RATE	5℃/min	5℃/min
AMP. RANGE	2mg	20mg
TEMP. RANGE	500℃	500℃
CHART SPEED	5mm/min	5mm/min

TABLE 2

STORAGE OF ANALYTICAL PARAMETERS

In thermal analysis, the measurement conditions are important and generally complex, and they often require a long time to be determined. This system described preserves all conditions in IC memory and the previously set parameters can be recalled automatically when the power switch is turned on. Furthermore there are 10 files available for the storage of analytical conditions.

RS-232-C

This system has the ability of sending the all data of thermal analyses to other computer via the RS-232-C interface, this interface can also be automatically controlled and lends itself very well to the complete automation of this thermal analysis system. In addition, the analytical conditions can be as well as the output from 4 different detectors.

SAFETY DEVICES

This system has several bluit-in safety devices which constantly monitor maximum temperatures, over-currents in the furnaces, temperature control fluctuations and even a seismic detector. Other functions exist to ensure safety should be the microprocessor fail during measurement, so that if any of the problems described above occurs, the system will stop the temperature control, cut off power to the heaters as well as its own power source to prevent an accident.

CONCLUSION

This new thermal analysis equipment has many epoch-mak ing features as mentioned above, and it responds to the needs that thermal analysis measurements must be excuted efficinetly. Through the four channel control and RS-232-C interface, this system achieves ease in laboratory automation.