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Automation and control of the Perkin–Elmer TGS-2 thermogravimetric analyzer

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Abstract

The Perkin–Elmer [1] TGS-2 thermogravimetric analyzer (TGA) has been interfaced to a Hewlett–Packard Vectra Model ES/12 personal computer. Using several developmental software packages and TURBO–PASCAL [2], an efficient, user-friendly software package has been developed. A versatile software package called SPECTRA–CALC® was used for data analysis and representation.

INTRODUCTION

Many older instruments that are still being used in laboratories can be rejuvenated by interfacing them with an IBM PC/AT or compatible computer [3,4]. The rejuvenation of older instruments is a rather inexpensive alternative to buying a new instrument. The Perkin–Elmer TGS-2 TGA is normally controlled manually or with the use of a TADS interface module and a Model 3600 data station. The Model 3600 data station is a closed environment with limited connectivity to other hardware (e.g. LAN) and higher resolution graphics packages. The TADS software, for data analysis, is written in BASIC and lacks the speed and graphics capabilities of the higher level programming languages (e.g. TURBO–PASCAL). There was a need in our laboratory to upgrade our current system to a PC to allow us accessibility to more sophisticated software packages for data analysis.

One such software package which was of interest was SPECTRA–CALC® from Galactic Industries [5]. Although this software package was written for spectral analysis, the graphics and ARRAY BASIC® [5] utilities seemed easily adaptable to the handling of thermal analysis data.

AUTOMATION

The Model 3600 data station is a dedicated computer with 16K RAM, Motorola 6800 CPU and dual RS232 serial ports. The data station communicates with the TGS-2 via the TADS interface using normal ASCII string

COMMAND	FUNCTION
55,###	STARTING TEMPERATURE
58,###	FINAL TEMPERATURE
47,###	HEATING RATE
48,###	COOLING RATE
60,###	DWELL TIME

Fig. 1. Examples of ASCII command strings for the Perkin–Elmer TGS-2 TGA. (Note: All commands must be followed by a carriage return.)

commands. The TADS interface module is an A/D converter with built-in memory and BCD capabilities.

Before the HP Vectra could be interfaced to the TADS interface module, the communications protocol and the ASCII command strings used to run the instrument were needed. This was accomplished by connecting a Hewlett–Packard Protocol Analyser (Model 4951C) between the 3600 Data Station and the interface module. Using the analyzer the communications protocol was established and by performing several runs under different conditions, many of the command strings were determined. Some are shown in Fig. 1.

SOFTWARE

The main program was written in TURBO–PASCAL [2] but several commercially available software development packages were used which minimized the amount of custom programming. POWER SCREEN [6] was used to develop a user interface with real-time error checking. The user interface consists of pull-down menus with dialog boxes. TURBO ASYNCH PLUS [6] was used to handle all the serial communications without the need of writing ones' own serial interface drivers. TP GRAPHIX TOOLBOX [2] was then used to provide real-time high-resolution color graphics without the need for custom programming of scaling factors, axis-drawing routines, etc. The program starts with a main screen which consists of a menu bar across the top. Using the mouse, arrow keys or the hot keys which are highlighted for each selection, the user can select a particular task. The menu bar list several options which are the primary tasks needed by the user to run the instrument and to analyze the data, i.e. Conditions, Start, Save, SPECTRA–CALC® and Quit.

CONDITIONS MENU

The conditions menu appears below the main menu bar and has the following fields: High Temperature Setting; Low Temperature Setting; Heating Rate; Cooling Rate; Delay Time; Dwell Time; Smoothing.

The entry fields for the heating and cooling rates can have limits attached if desired to restrict input. The end condition and smoothing fields have pop-up screens which direct the user on which values are acceptable to enter. The delay-time entry field allows the user to enter a time (minutes) to wait before starting the heating or cooling ramp. This may be used when investigating hygroscopicity of a particular sample. The dwell time entry field sets the number of minutes after a heat/cool ramp. If any value other than zero is entered into either field, the real-time screen will display the x-axis scale in minutes. The end condition field directs whether the instrument will cool, hold isothermally or heat at the end of the current run. The smoothing field controls the smoothing factor used for acquiring data.

ZERO, SCAN AND SAVE SELECTIONS

The zero selection allows the user to view the current balance reading and make any necessary adjustments to the balance control unit.

The scan selection sends the operating conditions to the instrument and plots the data in real-time as it is received from the instrument. The user can abort the run at any time using the escape key. The data is stored as ASCII XY data pairs (temperature versus milligrams) in a temporary file on the hard disk. Therefore if the scan is aborted by the user, the data received up to that point is available for processing.

The save selection takes the current temporary file and using a macro written in SPECTRA–CALC® automatically imports the file and stores the data file, under the name given by the user, to a floppy disk. The macro is listed below:

"This program is designed to automatically import TGA files into SPECTRA–CALC from outside."

Macro "[F2]EDC:\[EN]"
Macro "[F2]FITEMPFILE.PRN[EN][EN]R"
Macro "[F2]EDA:\[EN]"
Macro "[F2]QY[EN]

SPECTRA–CALC®

SPECTRA–CALC® is a data analysis and graphics package which also allows the user via ARRAY BASIC® to write their own data analysis programs. The software comes with a group of programs which can handle various data

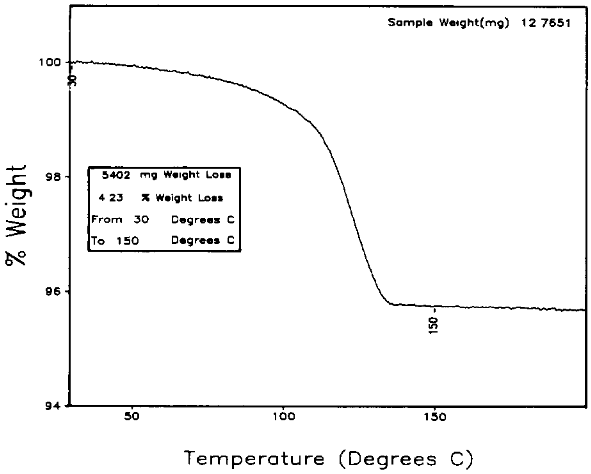

Fig. 2. Example of typical results obtained from data analysis using an ARRAY BASIC® program written within SPECTRA–CALC®. The output can be easily changed for individual needs.

manipulations such as deconvolution, smoothing, baseline correction, first and second derivatives, etc. Using ARRAY BASIC®, a program has been written for the TG percentage to determine the % weight loss and the milligram weight loss of a sample. The program allows the user to enter the temperature points manually or to use the mouse (or cursor keys) when determining the percentage weight loss. The user can also display the first derivative of the scan to see the inflection point. The results (Fig. 2) are placed in a drawing template which can be sent to a plotter or graphics printer or a disk file (HPGL format).

CONCLUSION

Many older instruments have been limited not by their capabilities but rather by the limitations of their accompanying computer systems. The Perkin–Elmer TGS-2 thermogravimetric analyzer is one such case. Using available software development tools, a user-friendly software package was assembled to control the TGA with an IBM AT compatible PC. The use of a PC for control and data analysis allows for greater versatility in the management and presentation of data.

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