Journal of Chromatography A, 659 (1994) 205–208
Elsevier Science B.V., Amsterdam

CHROM. 25 752

Short Communication

Fan-induced heating

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(First received September 20th, 1993; revised manuscript received November 19th, 1993)

ABSTRACT

A method is described in which the temperature of a chromatograph oven can be controlled over a range of 20°C above ambient temperature by controlling the power input to the fan.

INTRODUCTION

Temperature control of chromatographs just above ambient temperature poses special problems. The quantity of heat that must be supplied to balance losses is relatively small and yet must be supplied by a heating and control system that is designed to supply enough power for the chromatograph to heat up quickly and to operate up to, say, 500°C. In the early days of chromatography, control systems were designed that supplied power for part of each mains electricity cycle. For example, Ashbury *et al.* [1] used a modified Brown amplifier and a large thyratron to do just this. The famous Pye Model 104 chromatograph of the 1960s used solid-state circuitry to accomplish phase-angle control (Fig. 1a). Nowadays phase-angle control is frowned upon because it can introduce disturbances into the electricity mains and adversely affect the performance of digital electronic equipment.

This problem can be overcome by using circuitry that switches current on or off at the zero-volts part of the electrical cycle. A control circuit decides what proportion of the time the power shall be on. A thyristor stack can then be used to switch the power on and off. Two ways of doing this are illustrated in Fig. 1b and c. At low temperature the heater is only on for a small proportion of the time. It is easy to detect this happening. Fig. 2 shows pulses of power being supplied to the heater of a modern chromatograph to maintain its temperature at 40°C. Current flowing to the heater was detected by an inductive power meter. A potentiometric chart recorder was connected across a resistor connected across the terminals of the power meter. Although the electrical noise problem is solved, the power input is very uneven.

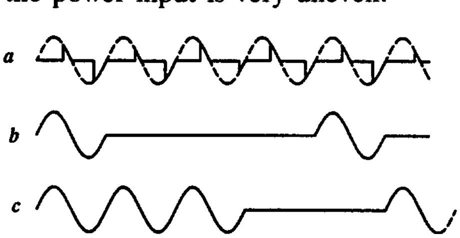

Fig. 1. Three ways of regulating power input: (a) phase-angle control; (b) single cycle; (c) fast cycle.

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SSDI 0021-9673(93)E1233-P

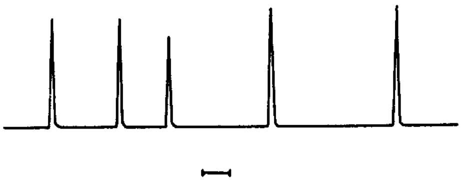

Fig. 2. Pulses of power being supplied to the heater of a modern chromatograph when operated at a relatively low temperature (40°C).

An approach used in the design of some modern chromatographs to aid operation at temperatures not much above ambient is to exchange air between the oven and its surroundings. This can be done via a motor-driven flap whose other purpose is to allow rapid cooling.

We have developed qualitative [2] and absolute [3] gas chromatographic methods based on measuring the small changes in flow rate that are caused by sorption and desorption, *i.e.* the "sorption effect". In these methods a pressure difference is measured with a sensitive pressure transducer. Thermal fluctuations at *any* point in the column cause pressure fluctuations that are sensed by the transducer as "thermal noise". This is in stark contrast to conventional gas chromatography where such fluctuations may cause band spreading but do not usually reveal themselves as signal noise. In capillary chromatography they can sometimes be seen on the chromatogram [4]. Flow-rate methods are worse affected by thermal noise than are conventional chromatographic measurements. Tests of several modern chromatographs proved none of them to perform as well for our purposes as the obsolete Pye Model 104. This machine has phase-angle control, no deliberate exchange of air with the surroundings and is insulated with substantial Marinite (asbestos cement) blocks.

Thermal noise can be reduced [5] by preventing exchange of air between the oven and its surroundings and by designing [6] the oven so that there is no temperature difference between the heater and the circulating air. This eliminates hot eddies. As part of this latter investigation, two methods of supplying power to the heater were devised that do not use phase-angle control; they can easily be implemented in the laboratory and adapted for most chromatographs.

There remains the problem of operating at just above atmospheric temperature. In this range the power input of the air-circulating fan in the chromatograph is sufficient to raise the temperature about 20°C. In this paper, we show that the temperature can be controlled satisfactorily just above ambient by controlling the power input to the fan. Our interest is in making sorption-effect measurements near ambient conditions, but the method will work just as well for conventional chromatography.

FAN HEATING

Baseline noise in sorption-effect chromatography has proved [5,6] to be a sensitive test of oven performance. We have demonstrated [5] the effect on noise of changing the fan speed by changing the motor voltage. We have also described [6] the use of a variable autotransformer (Variac) with negative-feedback temperature control to vary the power input to the *heater* in a chromatograph. These elements provide the basis for a method of controlling the oven temperature by varying the power dissipated by the fan.

The Pye Model 104 chromatograph has a two-speed fan. The speed is changed by switching between tappings on a transformer. The blades of the fan increase the temperature of the air inside the oven because of friction. The Pye Model 104 oven will attain a temperature about 17°C above ambient with the fan at full speed and about 12°C above on the low-speed setting. There is a range of fan speed above the speed at which the fan stalls for which the oven performance is satisfactory, so it is possible to control the temperature by varying the fan speed.

In the work reported here, a Variac adjusts the power supplied to the *fan* to maintain the oven temperature. A Eurotherm 821 three-term electronic controller is used to adjust the rotor position of the Variac by means of a servo motor and gear box. The temperature in the oven is sensed by a platinum resistance thermometer. The controller compares the measured value with its set point and adjusts the Variac to eliminate the error. The Variac supplies power to the fan motor and thus adjusts the fan speed to keep the temperature constant. Almost all the mechanical energy produced by the fan motor is dissipated as heat in the oven. Full details of the servo-system are reported elsewhere [6]. Depending on the design of the motor, there are many ways of controlling fan speed. It was convenient to use the servo-driven Variac "because it was there" but also because the motor was suitable for voltage speed control. The chromatograph could still be used in the usual way by connecting the thermometer to the chromatograph's own controller and setting the fan speed manually. The limits of this system are that the fan will stop if the voltage is too low and if the fan speed is too fast there are problems with random temperature fluctuations caused by the air turbulence. The fan motor voltage at zero controller input was adjusted to the minimum at which the fan kept moving by moving the Variac shaft gear relative to the gearbox output-shaft gear. The maximum speed could be set by a controller adjustment that limits the maximum output.

The three-term (PID, proportional, integral, derivative) Eurotherm 821 controller has 12-bit resolution (4096 divisions) over the configured temperature range. This was altered from 0–500°C to 0–100°C by reconfiguring the controller in set-up mode to give 40 divisions per degree instead of 8 divisions per degree. Two resistors, 1 kΩ and 1.5 kΩ, were connected in series across the controller output terminals to act as a potential divider and the servo control-system input was connected across the 1 kΩ resistor. This meant that instead of the controller output being 0 (0%)–5 V (100%) and giving the corresponding Variac arm movement (162° ≈ 130 V a.c.) via the servo control unit, the controller output becomes 0 (0%)–2 V (100%) thereby reducing the maximum arc through which the arm can move to approximately 65° (≈50 V a.c.). The change in the output of the Variac to a 1% change in the control signal is 0.5 V a.c. with this modification compared to 1.3 V a.c. with the previous [6] configuration.

There are several recognised ways [7,8] of finding initial controller settings to be used as the starting point and improved by trial and error. The methods involve simple tests whose object is to give an idea of the system dynamics. One such is to measure "the process reaction curve" obtained by making a step change in the voltage applied to the fan motor. The reaction curve (Fig. 3) looks like the sum of two exponential rises to limits, one being a rapid but modest rise the other being a slow but much larger rise. What is probably happening is that the power supply to the circulating air starts to change the air temperature rapidly but that heat then starts to be used to heat the heavy oven walls thus slowing the process. In these circumstances (no dead time) it would be expected [7,8] that proportional (or proportional-plus-integral) control could be used with high gain (and high reset rate if integral action is employed). The absence of any effective dead time was confirmed by increasing the gain. In the presence of dead time, this would make the temperature oscillate. It was not possible to cause oscillations. With the controller set to the most favourable control settings found, the temperature is only controllable within half a degree of setpoint but the drift is slow and little thermal noise is seen in sorption-effect measurements. The setpoint may be only a few degrees above ambient temperature (most commercial ovens have a minimum operating temperature that is ten degrees or so above ambient). Unless the oven application requires a very accurately set temperature this is not a problem. The advantage of this system for sorption-effect chromatography is that the temperature changes are smooth and slow and do not

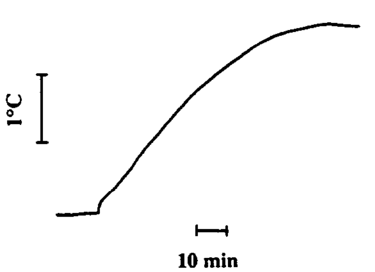

Fig. 3. A process reaction curve showing the temperature rise caused by a step change in the voltage applied to the heater fan of a Pye Model 104 chromatograph. There is no dead time.

disturb the flow as would the power pulses shown in Fig. 2. The temperature change while a chromatogram is recorded is very small. The system could perhaps be used in conjunction with a conventional heater for operation at higher temperatures. A constant voltage could be applied to the conventional heating element to set the temperature roughly. The fan speed could then be used to fine tune the temperature. It would also be possible to programme the temperature by programming the controller set point.

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