DESIGN, CALIBRATION, AND USE OF A CONVECTIVE AIR FLOW DY-NAMIC CALORIMETER*

BERNARD MILLER, J. RONALD MARTIN AND CHARLES H. MEISER, JR. Textile Research Institute, Princeton, N. J. 08540 (U.S.A.)

ABSTRACT

A new type of calorimeter has been developed that continuously monitors the rate of heat emission from a heat source by following the rate of convective air flow induced by the emitted heat. For this purpose a thermistor version of a hot-wire anemometer connected to a strip chart recorder is positioned to measure the flow of incoming air. Details are given for the construction, calibration, and operation of a form of the calorimeter designed for studying the upward burning of unrestrained fabric samples under conditions closely approximating natural burning.

INTRODUCTION

Techniques for measuring the heat emitted from a solid or liquid substance as the result of chemical or physical processes have almost invariably depended on the transfer of this heat (or a portion of it) to some predetermined target or heat sink. In most cases, heat transfer is monitored by following the change in temperature of such a target, one exception to this being the classic phase-change calorimeter of Lavoisier and Laplace¹. When the process of interest is combustion, heat transfer methods are complicated by the generation of products other than heat (i.e., gases, smoke) which are likely to interfere with the heat transfer measurement in an uncontrollable manner.

Another limitation of conventional combustion calorimetry is that the investigator seeking accuracy in determining the total amount of heat produced must do so at the expense of restrictions on the choice of combusion conditions and on response time. Total heat evolution is usually measured by monitoring the temperature rise of a relatively large heat sink surrounding the combustible sample. This is the basic principle of the oxygen bomb and other non-isothermal types of calorimeters². In both cases, the response of the heat sink (i.e., the temperature rise) is too slow to produce useful rate data, and conditions of combustion, such as sample

^{*} Presented at the 6th North American Thermal Analysis Society Meeting, Princeton, N.J., on June 20-23, 1976. The majority of the papers of this meeting have been published in *Thermochim. Acta*, Vol. 18, 1977.

size, mounting, and ventilation, are limited by the necessity of having to locate the sample within the heat sink³.

The alternative to this approach is to arrange for a fluid (gas or liquid) to pass in contact with the sample, and to monitor the rise in temperature of this fluid at some point downstream of the sample⁴. Such flow calorimeters can be designed to have short time constants if the temperature detectors used are of small mass and the fluid flow-rate is sufficiently high. Of course, this flow-rate must be controlled and known in order for the temperature rise data to be translatable into heat transfer quantities. The primary response, temperature rise as a function of time, can be integrated to give the total heat transferred, but such integration does not give results as accurate as bomb-type calorimetry.

For combustion studies, the obvious fluid for a flow calorimeter would be air or oxygen. This type of experiment has been used to study the flaming combustion behavior of organic solids (i.e., polymers)⁵, but the limitations caused by the coincident generation of combustion products has inhibited general usage, and the requirement that the air flow must be regulated makes the results unrelatable to what happens during natural burning under unrestricted convective air flow.

The pressing need for a realistic, sensitive, and accurate laboratory method for monitoring the burning behavior of fabrics has led to the development of a new type of dynamic calorimeter which appears to have certain advantages over other approaches to the problem. This instrument can measure, directly and continuously, the rate of heat emission from a burning material over the entire period of combustion. It does not depend on any direct measurement of temperature rise and can be carried out without imposing artificial environmental conditions during the combustion process. This paper describes the principles of the method, and the design, calibration, and use of an apparatus suitable for burning fabric samples. The concept, however, is general and can be scaled up or down for studies on any system that produces heat, that is, it is not restricted to flaming combustion studies.

MEASUREMENT PRINCIPLE

The technique depends on the phenomenon of induced convective air flow, which occurs whenever a localized heat source is present in any ventilated environment. The gases in the immediate vicinity of the heat source become heated above ambient temperature and rise, forcing cooler air to move in from below and producing a continuous upward flow. If the heat source is placed in a vertically oriented enclosure (e.g., a chimney) and there is an opening at the base of this enclosure—that is, below the location of the heat source—the convective flow will be enhanced, and its rate will respond directly to the rate at which heat is emitted. Thus, if one can arrange to measure this convective air flow-rate, it becomes possible, with appropriate calibration, to monitor the rate of heat emission from the source.

APPARATUS

Top and side views of the TRI convective air flow dynamic calorimeter used for fabric burning studies are sketched in Fig. 1. The dimensions of the apparatus have been chosen, on the basis of an extensive series of trials, as the best compromise between demands of sensitivity and practicality for fabric samples up to 0.2 m in width and 0.3 m in height. Convective air flow is directly dependent on the vertical distance from the heat source to the top of the "chimney"⁶; therefore one could build a more sensitive version by making the burning chamber taller. Another way to increase sensitivity would be to reduce the diameter of the air entry chamber at the point where the air flow-rate measurement is made (T in Fig. 1). Since this would impose an artificial throttling effect, it would not be desirable for fabric burning



CONVECTIVE AIR FLOW DYNAMIC CALORIMETER



Fig. 1. Top and side views of convective air flow dynamic calorimeter. S, fabric sample suspended within burning chamber; s, screens fitted into air entry chamber; T, thermistor; d_1 and d_2 , access doors.

studies; however, such a change would not be detrimental if air were not involved in the heat generating process.

The burning and air entry chambers are constructed from cylindrical sections of standard, galvanized heating duct units obtained from a local hardware supply. These units are not permanently attached to each other, making it convenient to disassemble them for occasional cleaning. During operation the joints between these sections are sealed with tape.

Three sections, each 0.6 m in length, make up the vertical burning chamber. In the lowest section, the upper door d₁ supplies access for mounting the sample which is suspended from a horizontal wire by small hooks. The lower door d, is used for recovering unburned residues. The two upper sections are water-cooled by means of 6.4-mm (1/4-in.) O.D. copper tubing per section; not shown in Fig. 1. Water flow during measurements is maintained at about 0.02 l s⁻¹. Water-cooling the burning chamber serves two purposes. It eliminates time dependence of the air flow response, thus greatly simplifying calibration of the apparatus. It also aids in the achievement of one of the major objectives of this work, the simulation of natural burning as opposed to burning in a confined space with arbitrary restrictions. One aspect of natural burning is that any heat escaping the immediate vicinity of the fabric is no longer available to maintain the burning process. However, if burning is carried out in a chamber whose walls can get hot and redirect heat back toward the fabric, an artificially enhanced burning will occur. Water-cooling the walls keeps them from becoming significantly hotter than ambient temperature, and the result is a close simulation of burning as it would happen if there were no walls at all. The instantaneous response of the air flow detector is not altered by the water-cooling, indicating that the induced convection is the direct result of heat transfer to the gas phase and does not depend on re-radiation of heat from the chamber wall.

The air entry chamber is curved both for compactness and to prevent flame radiance from disturbing the air flow detector at T. This chamber is horizontally oriented since it itself contains a heat source, namely, the thermistor, T; if the entry chamber were vertical, an extraneous air flow counter to the main flow would be produced. Upstream from the air flow detector are two coarse, wire mesh screens which serve to smooth out the air flow and to suppress the effects of room drafts.

The incoming air flow-rate is monitored by a version of a hot-wire anemometer, which detects the rate of air flow by the extent to which it is cooled below its static operating temperature. The unit uses a thermistor, T, for this purpose, mounted in the center of the air entry tube as shown in Fig. I. A second thermistor is located outside of the chamber to compensate for any changes in ambient temperature and humidity".

The two thermistor units are part of the bridge circuit described in Fig. 2. An applied bridge potential of 6.0 V serves to supply current to heat the thermistor

^{*} Matched thermistor pairs similar to the units used can be obtained from Allied Electronics Corp. (Stock No. 791-0446; p. 20 of 1976 purchasing guide).



Fig. 2. Bridge circuit for convective air flow dynamic calorimeter.

units. The bridge output is fed to a strip chart recorder with a full-scale range of 1 V.

CALIBRATION

A heat source with a known, adjustable rate of heat emission is required to convert observed air flow-rates to heat fluxes. A methane gas flame has been used for this purpose. A T-shaped gas burner (see Fig. 3), the same width as the fabric sample to be studied, is mounted in the burning chamber at a point corresponding



Fig. 3. Methane burner for calibration of convective air flow dynamic calorimeter.

to the vertical midpoint of the sample. Methane is metered to this burner at rates ranging from 0.008 to 0.4 l s⁻¹, which corresponds to a heat flux range from about 0.3 to 14 kJ s⁻¹. This range is sufficient to cover the heat emission behavior of nearly all ignitable textile materials. After a specific methane flow-rate has been established, the gas is ignited and the air flow-rate recorded for about 1 min. A typical calibration run is illustrated in Fig. 4. The apparent "noise" is in fact a true record of the velocity of the air passing the detector and is typical of turbulent gas flow. The average voltage value can be simply obtained by eye, or by any other signal averaging method. A calibration curve relating the anemometer response to heat flux is shown in Fig. 5. This curve can be used directly to convert anemometer output to an instantaneous rate of heat emission for any time during burning.



Fig. 4. Response for methane burning at a constant fuel feed rate.



Fig. 5. Calibration curve; anemometer response to known heat fluxes, dq/dt.



Fig. 6. Typical anemometer response for a burning fabric.

When a fabric burns, it produces an air flow trace such as that shown in Fig. 6. The peak value corresponds to the maximum rate of heat emission achieved by the sample. The total amount of heat emitted by a sample can be obtained by integrating the rates of heat emission over the time of burning (that is, converting the area under the curve to heat). Numerical integration can be simplified by converting the calibration curve in Fig. 5 to an analytical expression, which can be programmed into a computer so that successively entered values of anemometer output can be conveniently converted to rates of heat emission and integrated over the time of burning.

An analytical expression for the calibration curve in Fig. 5 might consist of two independent parts: a linear portion describing the calibration at low heat emission levels (for anemometer outputs less than 0.2 V in this case), and a parabolic portion for higher levels. The latter can be fitted by parabolic regression to an equation of the form

$$dq/dt = AV^2 + BV + C \tag{1}$$

where A, B, and C are constants, and where V is the anemometer output. The linear portion of the curve is fitted by eye (to pass through the origin and be continuous with the curve of eqn (1)) to an equation of the form

$$dq/dt = KV \tag{2}$$

where K is a constant. The total heat given off during burning is obtained by computing the rates of heat emission for small time intervals (using whichever of the above equations applies) and numerically integrating as follows:

$$q = \int \frac{\mathrm{d}q}{\mathrm{d}t} \,\mathrm{d}t = \sum 1/2 \left[\frac{\mathrm{d}q}{\mathrm{d}t_{\mathrm{s}}} \div \frac{\mathrm{d}q}{\mathrm{d}t_{\mathrm{s}+1}} \right] (\Delta t) \tag{3}$$

EXPERIMENTAL PROCEDURE

A fabric sample $(0.30 \times 0.15 \text{ m})$ is placed in the burning chamber with its long dimension in the vertical direction. A sheet of aluminum foil is placed in the bottom of the chamber to catch any unburned residue that falls. The voltage supply to the bridge is brought up from its minimal value to 6.0 V, and the bridge is balanced to produce a zero voltage output on the recorder. The sample is ignited in whatever manner desired (e.g., with small pilot flame), and the door is closed. Within a few seconds the aneniometer response indicates the progress of burning and produces a curve similar to that in Fig. 6, from which the following values may be obtained:

(a) The maximum heat emission rate $(kJ s^{-1})$ (from the peak anemometer response)

(b) The time to reach maximum rate

(c) The average burning acceleration (kJ s⁻²) (a/b)

(d) The total heat emitted per gram of sample (kJ g^{-1}) (determined by integrating the overall response curve)

(c) The total heat emitted per gram of volatilized sample (kJg^{-1}) (calculated on the basis of the weight of residue, including whatever fell on the aluminum foil).

Subsequent runs can be made as soon as the anemometer response has returned to zero.

Any configuration of fabric can be studied with this procedure. If a frame or any other mounting accessory with appreciable mass is to be included in the heated zone, a new calibration should be carried out to account for its thermal and gas flow obstruction contributions.

CONCLUSIONS

The convective air flow dynamic calorimeter appears to have certain advantages when compared with other available methods for monitoring burning behavior:

Heat emission during flaming combustion can be studied under conditions that closely approximate natural burning.

The only transducer is located where it will not be affected by the products of combustion.

The signal produced responds quickly to any change in burning rate and returns to zero almost immediately after flaming stops.

Variations in sample size, shape, configuration, orientation, or degree of restraint can be imposed without changes in the basic experimental arrangement.

The apparatus has proven to be simple to construct, operate, and maintain in working order.

REFERENCES

- 1 W. J. Moore, Physical Chemistry, Prentice-Hall, Englewood Cliffs, N.J., 3rd ed., 1962, p. 53.
- 2 J. M. Sturtevant, in A. Weisberger (Ed.), Physical Methods of Organic Chemistry, Part 1, Interscience, New York, 3rd ed., 1959.
- 3 M. M. Birkey and K.-N. Yeh, J. Appl. Polym. Sci., 17 (1973) 239.
- 4 W. J. Parker and M. E. Long, Ignition, Heat Release, and Noncombustibility of Materials, ASTM STP 502, American Society for Testing and Materials, 1972, p. 135.
- 5 E. E. Smith, Fire Technol., 8 (1972) 237.
- 6 J. H. Perry, Chemical Engineers' Handbook, McGraw-Hill, New York, 4th ed., 1963, pp. 9-43.