Calorimetric Study of Flammable Fabrics.   I. Instrumentation and Measurements*

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Synopsis

A calorimeter has been designed, calibrated, and tested to measure the total amount of heat released and the rate of heat released from the combustion of fabrics in air. Calibration of the calorimeter gave a reproducibility of ±3% for total heat measurements and ±5% on rate measurements. Consideration of systematic errors gives an expected accuracy of ±7% for total heat and ±10% for combustion rate measurements. Measurements on cotton show that 90% of the standard heat of combustion is released when cotton is burned in air. The rate of heat release for cotton is independent of fabric weight. The constancy of rate of heat release as determined calorimetrically confirmed the result implied by the 45° test measurements on flame spread rate. The rate and amount of heat release of other commercial fabrics and blends were also measured.

INTRODUCTION

The hazard associated with flammable fabrics is dependent on such parameters as (1) ease of ignition, (2) rate of heat release, (3) total amount of heat released, (4) rate of flame propagation, and (5) heat transfer mechanism. This is not necessarily a complete list, nor are the parameters independent of one another.

A calorimeter was designed to measure the rate of heat release and the total amount of heat released when fabrics are burned in air. The total amount of heat obtained from combustion of fabric in air is expected to be significantly less than the standard heat of combustion obtained with the oxygen bomb calorimeter; in the oxygen bomb, complete oxidation of the samples takes place, which represents the maximum amount of heat.

Extensive studies[1-4] on the physiology of burns show that the mechanism by which burns are produced is dependent on the time rate of heat exposure and not simply on the duration of exposure. Stoll[4] has shown that the energy required to produce a second-degree burn, defined as dosage (= heat flux × time), does not obey the "law of reciprocity." For example, a large amount of energy delivered over an extended period of time may produce little or no injury while the same amount of energy delivered in-

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stantaneously may produce extensive damage. This information (on the physiology of burns) suggests that the hazard due to flammable fabrics is closely related to the rate of heat release and that the rate of flame spread measurements, as used in the past as a means of evaluating the hazard of flammable fabrics, gives an incomplete picture of a complex phenomenon.

The rate of heat transfer from burning vertical cotton panels has been measured,[5-6] and in one case[6] the rate (of heat release) data were combined with the threshold curve for second-degree burns in an effort to develop criteria for determining the degree of hazard associated with flammable fabrics.

In the past, the relative hazard of flammable fabrics has been judged by flame spread rate as measured in a 45° angle standardized test apparatus (CS 191-53). Although this widely used test for the measurement of the flammability of textiles has produced extensive data, it is uncertain how these data relate to the wide variety of hazardous fabric situations.

The data obtained from the 45° test in various laboratories show that the flame spread rate is inversely proportional to the fabric weight per unit area for a given fabric. This suggests that the rate of heat release from a given type of fabric, for example, cotton, is independent of the fabric weight. However, the total amount of heat released per unit area for any given fabric will be dependent on fabric weight, and it is not surprising that measurements made by Webster, Wraight, and Thomas[4] show that the total heat transferred to a vertical surface from a burning fabric is proportional to the fabric weight. These authors concluded that all fabrics of any practical value released sufficient energy, if ignited, to produce second-degree burns.

The calorimeter for measuring the rate of heat release and the total heat released from a burning fabric was designed to operate in a constant-temperature environment. Calorimeters of this type are called isoperibol calorimeters. A complete measurement theory for total energy measurements has been worked out for this calorimeter.[8] However, the mathematical theory for analyzing rate data from an isoperibol calorimeter was not available in the literature. Consequently, the rate data in this paper were obtained by extensive calibration curves. Since the start of this work, the theory has been developed and will be the subject of another paper.

MEASUREMENT THEORY

The principle of operation of the calorimeter is to burn the sample in air inside the calorimeter and draw the gaseous combustion products through a heat exchanger that forms the walls of the calorimeter. The temperature rise of the calorimeter is measured with a thermopile referenced to a shield placed around the calorimeter.

The theory of the isoperibol calorimeter has been worked out in considerable detail and used extensively in thermochemistry.[8-10] The mea-

surement theory based on the first law and describing heat flow in the calorimeter is summarized by West and Churney's[8] eq. (20) and is included here as eq. (1):

$$W = E\left[T_2 - T_1 + b_1 \int_{t_1}^{t_2} (T - T_\infty)dt \right] \qquad (1)$$

where W is the work done on the calorimeter by the burning fabric or by the gas burner used for calibration to determine the "energy equivalent" E of the calorimeter; T is the observed temperature at time t, T_1 and T_2 are the observed temperatures at time t_1 (usually zero or time of ignition) and t_2, respectively, which occur during rating periods; b_1 is the cooling constant; and T_∞ is the convergence temperature (steady-state temperature reached by the system after infinite time when $W = 0$).

The right-hand side of eq. (1) is simply an energy equivalent, E, determined in a calibration experiment, multiplied by a "corrected temperature rise" ΔT_c. This temperature rise consists of the difference between the final and initial temperatures, T_2 and T_1, which must be observed when the temperature is a single exponential function of time, plus the product of the "cooling constant" b_1 and the integral of the temperature–time curve.

When $W = 0$ (heat source has been removed) or when W is independent of time, the solution to the heat flow problem in the calorimeter leads to an expression for the temperature T of the calorimeter that involves a series of exponential functions of time. Following a suitable waiting period after the heat source has been removed from the calorimeter, it is observed that the temperature becomes a single exponential function of time. At this time, the calorimeter temperature is given by eq. (2):

$$T - T_\infty = a_1\Phi_1 \exp(-b_1 t) \qquad (2)$$

where a_1 is determined from initial conditions and Φ_1 is a geometric factor which is dependent on the position of the thermometer or temperature-sensing device.

When eq. (2) defines the temperature, the calorimeter is said to be in a rating period, and the change in temperature is given by eq. (3):

$$(dT/dt)_t = -b_1(T - T_\infty). \qquad (3)$$

A measurement begins with the calorimeter in an initial rating period and in our case this initial temperature gradient is zero $[(dT/dt)_t = 0]$, although the zero gradient is not a requirement. Similarly, the measurement ends when the calorimeter temperature decay is given by a single exponential function of time, i.e., the final rating period. The final temperature T_2 can be obtained at any time during this period.

CALORIMETER

Design requirements to obtain the total heat and the rate of heat release are difficult to meet with a single instrument since each measurement
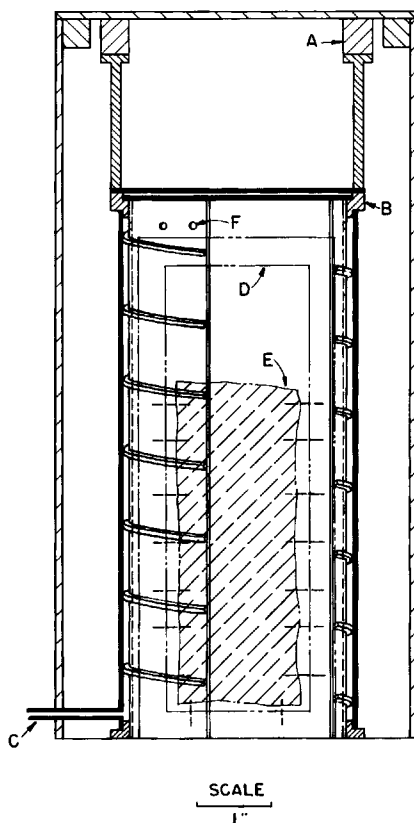
Fig. 1. Cross section of calorimeter. Points A and B are thermocouple positions, C is air exhaust port, D is fabric holder frame, E is fabric, and F is inlet port for hot gases to heat exchanger.

requires a calorimeter with different and frequently conflicting requirements. For rate measurements, the calorimeter should be low in mass and have a rapid time response; while for precise total heat measurements, a relatively high mass is desirable such that the temperature rise of the calorimeter is small. Consequently, to make a calorimeter for both measurements, a compromise must be made. Also, to obtain the rate of heat release, the sample must be sufficiently long for steady-state burning to take place over a significant time period or over a significant fraction of the sample. The time constant, or response time, for the calorimeter can be approximated by the response of a semiinfinite slab and is proportional to the length squared.[11] This simply means that the calorimeter should be kept as short as possible, subject to the minimum length to obtain steady-state burning data.

With these design requirements, the first calorimeter was 15 cm long and 6 cm in diameter and was designed to burn a sample 5.1 cm wide by 12.7 cm long. This length of sample was considered a minimum for obtaining
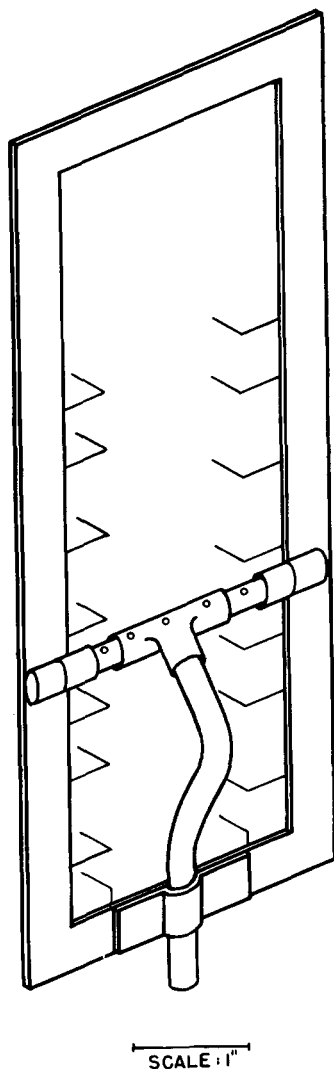
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Fig. 2. Fabric holder which is placed inside the cylinder in Fig. 1.

rate data. Also, the calorimeter was made of oxygen-free high-conductivity copper to increase the rate of response.

When cotton samples were burned in this calorimeter, the fraction of the standard heat of combustion obtained was a function of the sample length: the shorter the sample, the higher the percentage of ΔH_c obtained. The most obvious explanation appeared to be quenching of the flames by the cold calorimeter walls. This was not surprising since it was one of the early concerns in the design.

To overcome the quenching problem, a larger isoperibol calorimeter (Fig. 1) was designed based on measurements of the smaller version. The

samples burned in the calorimeter were maintained at 5.1 cm × 12.7 cm. However, the dimensions of the calorimeter were increased to 7.5 cm in diameter and 19 cm in length. Air was pulled through the calorimeter at 65 liters/min. This flow rate was chosen to provide maximum combustion in air. The calorimeter was constructed with a double wall between which the hot gases are exhausted. A baffle was placed between the two concentric cylinders in the form of a helix, forcing the hot gases to spiral around the calorimeter. This design was adopted to facilitate rapid transfer of the heat from the air to the calorimeter.

A two-junction thermopile was used to measure the temperature rise of the calorimeter. The cold junctions were fastened to the top of the container used to enclose the calorimeter (A in Fig. 1). This container provided
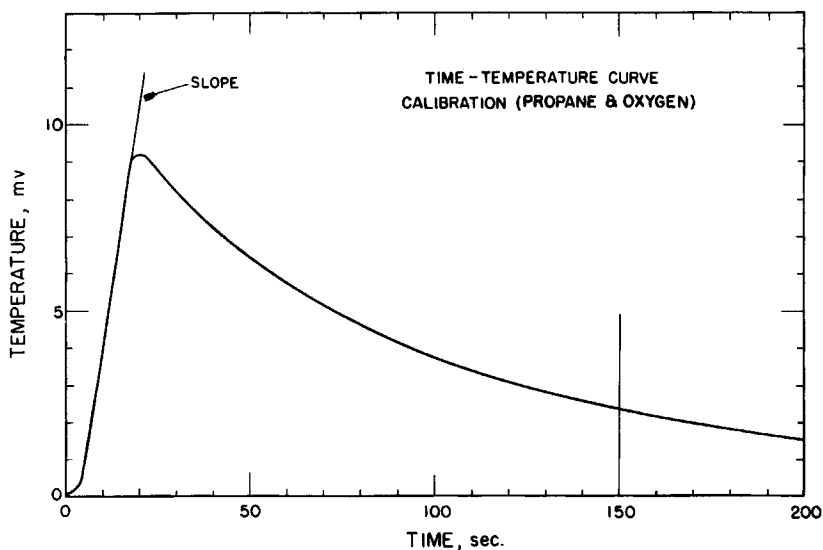

Fig. 3. Typical temperature–time response of calorimeter during calibration.

the necessary heat sink for the reference junction of the temperature sensor and also provided the isothermal environment for the calorimeter. The hot junctions are attached to ring B in Figure 1.

The calorimeter was calibrated with a premixed oxygen–propane burner. The burner was equipped with a Tesla coil that ignited the gas mixture simultaneously with the opening of the solenoid valve. The gas burner, used for calibration, was mounted on the back of the fabric holder, as shown in Figure 2.

A typical calibration time–temperature curve for the calorimeter is shown in Figure 3. Figure 4 is a typical response curve when a fabric is burned in the calorimeter. The total heat released was calculated using eqs. (1) and (3). Equation (3) is used to determine b_1 and T_∞ from the rating period by taking the slope (dT/dt) of the experimental curve for two
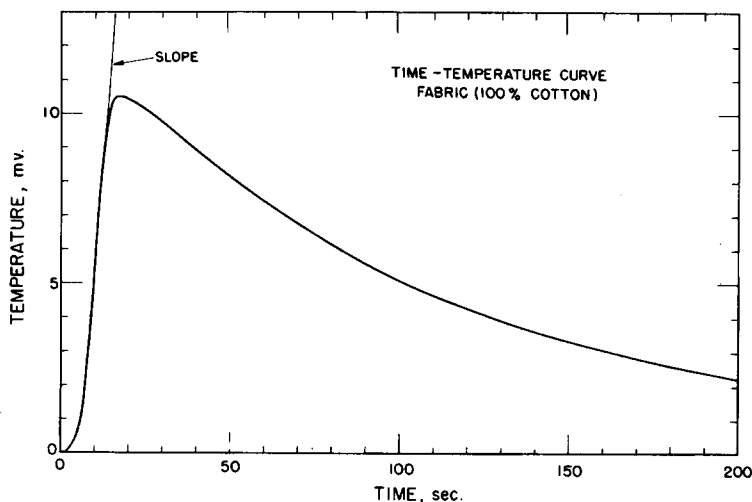
Fig. 4. Typical temperature–time response of calorimeter during burning of cotton.

different times. The corrected temperature rise of the calorimeter is calculated from eq. (4):

$$\Delta T_c = T_{150} - T_0 + b_1 \int_0^{150} (T - T_\infty)\, dt. \tag{4}$$

Equation (4) is the bracketed quantity in eq. (1), where $T_2 = T_{150}$, $T_1 = T_0 = 0$, $t_1 = 0$, and $t_2 = 150$ sec. Experimentally, it was determined that the calorimeter entered a rating period 150 sec after ignition of the sample. Consequently, the integral is evaluated from 0 to 150 sec. After the calorimeter has entered a rating period, the same corrected temperature rise (ΔT_c) will be obtained by evaluation of eq. (4) at 180 sec or any later time. The constancy of the corrected temperature rise is a convenient check to determine whether the calorimeter has entered a rating period.

In calibration, a known amount of propane, measured with a flowmeter, was combusted in the calorimeter. The standard heat of combustion (ΔH_c) of propane was corrected for the heat of vaporization of water, giving a net heat of combustion. The calorimeter was calibrated at various heat inputs and rates of heat input. The results of the calibration are shown in Table I.

From the calibration data in Table I, the energy equivalent E of the calorimeter is 210.4 cal/mV, with a standard deviation of 6.6 cal/mV. This gives a precision of about $\pm3\%$. However, it is estimated that the accuracy of the calorimeter is about $\pm7\%$ because of various systematic errors. For example, radiant energy is lost from the bottom of the calorimeter. Another possible source of error is the heat carried away by incompletely cooled exhaust gases. Both of these possible sources of errors are compensated to a large degree by the calibration.

TABLE I
Calibration Data

Rate, cal/sec	Total heat input, cal	Energy equivalent E, cal/mV
61	594	202
	1218	206
	1818	202
	2727	210
	3927	202
96	923	201
	971	201
	1461	200
	2864	203
	2883	210
	4296	211
134	642	212
	1378	211
	1994	212
	1378	216
	1994	210
	1378	218
	2020	220
169	897	216
	1710	220
	1556	216
	3336	221
240	2428	209
	3318	210
	4207	220
	Ave.	210.4
	S.D.	6.6

The temperature of the exhaust gases was monitored with a thermo-couple at the exhaust port C in Figure 1. The amount of heat transferred out of the calorimeter by the exhaust gases is given by

$$h = F \int_0^t \Delta T(t) C_p(t) dt \qquad (5)$$

where C_p is the heat capacity of the gases, F is the flow rate, and ΔT is the temperature rise of the gases. The calorimeter was calibrated over the same range of total heat input and rate of input as given off by the burning fabric, so that ΔT, t, and F are approximately the same in both the cali-bration and fabric experiments. The actual heat capacities of the com-bustion products for a calibration experiment will not be exactly the same. However, this is true only for the first few seconds of the experiment, i.e., only when combustion is taking place, and the resulting error in the energy measurement is expected to be small. However, this contributes to the overall uncertainty.
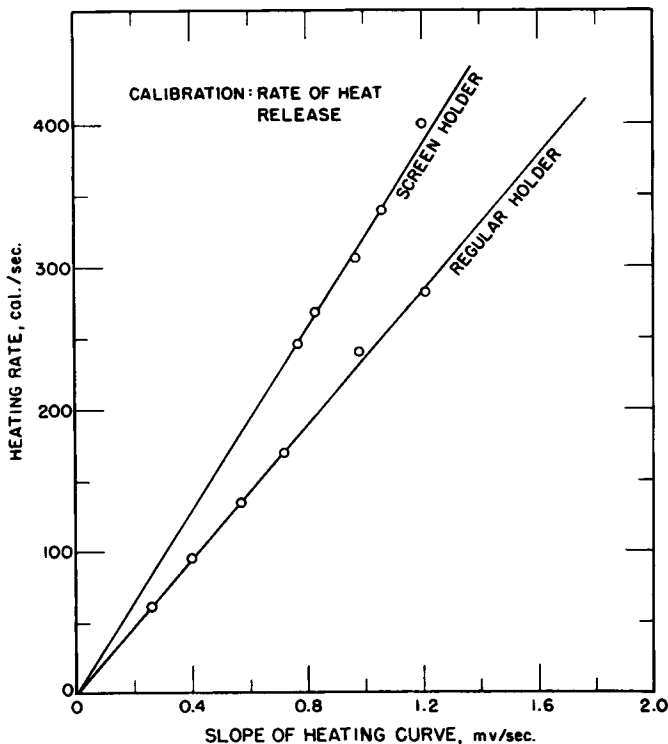
Fig. 5. Calibration curve for rate of heat release from propane.

Within the calibration range, a linear relationship was found between the rate of temperature rise of the calorimeter (slope of Fig. 3) and the known rate of heat release from the combustion of propane at a given flow rate. The results of measurements of rate of heat release, in cal/sec, and the rate of temperature rise, in mV/sec, of the calorimeter with the regular holder are shown graphically in Figure 5. A least-squares regression of the data points gives eq. (6):

$$\text{rate} = \dot{Q} = 236.6S \tag{6}$$

where S is the slope, in mV/sec, of the experimental curve as shown in Figures 3 and 4. The standard deviation of the slope is equivalent to 5%, and the estimated accuracy is 10%.

RESULTS

The calorimeter was used to measure the rate and amount of heat released from 15 fabrics. All fabric samples were 5.1 cm wide and 12.7 cm long and conditioned at 20°C and 65% relative humidity. The samples were weighed before burning, and the residue was collected and weighed. The fabric weights ranged from light-weight print cloth and tricot to heavy-weight twill. The results of these measurements are shown in Table II.

TABLE II
Rate and Total Heat Release Data

Fabric	Fabric wt, oz/yd²	Residue, %	Heat release rate,[a] cal/sec cm	Total heat release −ΔH,[b] cal/gm	Calorimetric net heat of combustion −(ΔH₀),[c] cal/g	Net heat, %
Cotton	3.0	1.5	78.7 ± 1.0	3324	3688	90.1
	3.3	1.5	78.3 ± 1.0	3315	3688	89.9
	3.6	1.5	78.3 ± 2.0	3316	3688	89.9
	4.8	1.5	75.0 ± 3.0	3236	3688	87.7
	5.9	1.5	78.7 ± 1.0	3240	3688	87.9
	8.5	1.5	73.6 ± 4.7	3245	3688	88.0
Regenerated cellulose	4.4	1.5	70.9 ± 1.0	3177	3516	90.4
	6.1	1.5	73.6 ± 1.6	3142	3516	89.4
Cellulose diacetate	2.6	11.5 ± 1.2	42.5 ± 3.7	3289	4100	80.2
Cellulose triacetate	3.9	13.3 ± 0.9	38.8 ± 4.3	3084	4200	73.4
90/10 Cellulose triacetate/nylon	6.2	11.3 ± 0.1	36.7 ± 1.2	3502	4473[d]	78.3
Acrylic	9.5	22.1 ± 1.0	43.7 ± 1.8	4118	7020	58.7
Poly (1,4-cyclohexylene-dimethylene terephthalate)	4.3	28.9 ± 2.9	65.4 ± 2.8	3363	6496	51.8
Polyethylene terephthalate[e]	4.0	41.9 ± 4.9	17.5 ± 3.0	2188 ± 143	5255	41.6
Nylon 66[e,f]	2.8	37.4 ± 7.6	13.2 ± 1.2	3715 ± 135	6926	53.6

a Rate obtained from sample 5.1 cm wide and 12.7 cm long.
b Standard deviation = ±3% (estimated from calibration).
c Oxygen bomb data with the correction of the heat of vaporization of water.
d Calculated from the net heats of combustion of pure components in the blend.
e Reinforced with three metal wire stitches.
f Cotton gauze underlay as ignition.

Column 4 of Table II lists the rate of heat released from the fabrics. The rate of heat release was obtained from the rate of temperature rise of the calorimeter and the calibration curve shown in Figure 4. The rate obtained from Figure 4 was divided by the fabric width (5.1 cm) to get the rate of heat release per unit fabric width. Within the experimental uncertainty, the rate of heat release from cotton per centimeter of fabric width is independent of fabric weight.

For calculating the rate of heat release data in Table II, it was assumed that steady-state combustion of the sample took place. With a sample length of 12.7 cm, such an assumption may not be entirely valid. As discussed earlier, it was necessary to make compromises in the calorimeter design, and a calorimeter that is long enough to contain a sample to give true steady-state combustion would have been unduly massive with an unreasonable time constant. However, the data on rates certainly have relative significance. In addition, the constancy of the rate of heat release for cotton over the fabric weights measured is not expected to change with sample length.

In the last column of Table II, the amount of heat released in air (column 5) is compared to the net heats of combustion listed in column 6 of Table II. The net heats of combustion were calculated from the standard heats of combustion measured by Yeh and Barker.[12]

To obtain the amount of heat released from the synthetics that melt and drip, a fine wire screen was attached to the fabric holder to catch the melt. The calorimeter was recalibrated with the new holder since the addition of the metal screen changes the energy equivalent of the calorimeter.

DISCUSSION

The total heat release from most of these fabrics has been measured previously[12] using an indirect method. A comparison of the values obtained by the two techniques is shown in Table III. The values of ΔH using the indirect method[12] were obtained by burning the fabric in air, then putting the residue in an oxygen bomb calorimeter to obtain the residual heat of combustion of the char. The value so obtained was subtracted from the standard heat of combustion of the fabric to obtain the heat release during combustion in air. The value obtained by this technique represents a maximum amount of heat released because it is assumed that the fraction of the sample burned is combusted completely to CO_2 and H_2O.

The large differences in the residue yield from cellulose diacetate, cellulose triacetate, and acrylic shown in Table III is attributed to the different techniques used to collect the melt as it drips from the burning fabric. In the present work, the mesh was fine enough so that the melt continued to burn for a short period after collection on the screen. In the work by Yeh and Barker,[12] the melt was quenched in a porcelain collection dish. Quenching resulted in a larger residue yield.

TABLE III
Comparison of Total Heat Release

Material	Residue, %		Total heat release, cal/g	
	Present work	Y & B[12]	Present work	Y & B[12]
Cotton	1.5	1.5	3324	3688
Regenerated cellulose	1.5	1.5	3160	3516
Cellulose diacetate	11.5	29.9	3289	2896
				(3821)[a]
Cellulose triacetate	13.3	52.5	3084	2025
				(3868)[a]
Acrylic	22.1	62.3	4118	2740
				(5659)[a]
Poly (1,4-cyclohexylene- dimethylene terephthalate)	28.9	26.8	3363	4692

[a] Recalculated value, using residue yield of present work.

The last column in Table II shows that polyester gave the lowest percentage (41.6%) of the total possible heat.

As mentioned earlier, the rate of flame spread over cotton as determined from the 45° test is inversely proportional to the fabric weight over a broad range of fabric weights.[13] A plot of flame spread rate as a function of fabric weight for measurements made on cotton in which a 6-in. strip of cotton is burned at an angle of 45°, as defined in ASTM procedure D 1230-60, is shown in Figure 6; it gives a slope of 27 mg/cm sec. This relationship between flame spread rate and fabric weight can be expressed by eq. 7:

$$dx/dt = K_1/\delta \tag{7}$$

where dx/dt is the rate of flame spread and δ is the fabric weight. Lawson, Webster, and Gregstin[14] obtained a similar relationship for the rate of flame spread for cotton fabric suspended vertically and ignited at the bottom. The flame spread rate was calculated from continuous weight loss measurements, in this case, $K = 80$ mg/cm sec. Neilsen and Richards[15] also measured the flame spread rate over a 25-in. specimen in the vertical position. A plot of their flame spread rate versus reciprocal of the fabric weight gives a slope of 58 mg/cm sec.

From these measurements on flame spread rates, one can deduce that the rate of mass transfer from the burning cotton fabric is given by

$$dm/dt = \dot{M} = K\delta y dx/dt \tag{8}$$

where δ is the fabric weights per unit area, y is the width of the fabric, and dx/dt is the flame spread rate, which is inversely proportional to δ. If it is assumed that the width of burning fabric is a constant determined by the experiment, the mass transfer rate is a constant.
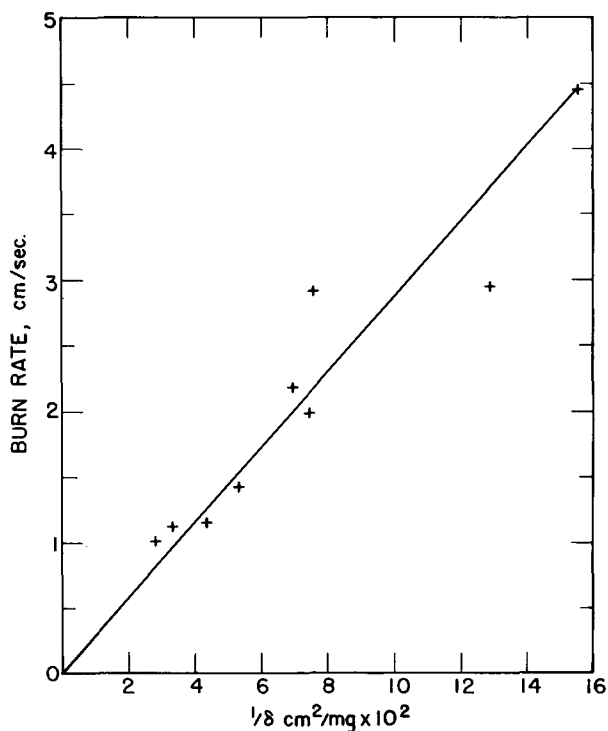
Fig. 6. Relationship between 45° flame spread rate and fabric weight for cotton fabrics.

The total amount of heat released by the fabric during the test is given by

$$Q = \Delta H \delta y x \tag{9}$$

where ΔH is the heat released per unit mass of the fabric (cal/g). The rate of heat release is simply the differential of eq. (9):

$$\dot{Q} = \Delta H \delta y (dx/dt) \tag{10}$$

or, using eq. (7),

$$\dot{Q} = \Delta H K_1 y. \tag{11}$$

According to eq. (11), the rate of heat release for a constant fabric width from a given fabric is independent of fabric weight. However, it may be that the rate of heat release is dependent on construction.

As indicated in column 4 of Table II, eq. (11) was verified by determining the rate of heat release for a number of cotton fabrics of different weights; and, as mentioned earlier, the rate of heat release was found to be independent of fabric weight within the experimental uncertainty. The quantity $\dot{Q}/y$ in eq. (11) is the rate tabulated in Table II, column 4; and for cotton, it is 78.7 cal/sec cm. Substitution of $\dot{Q}/y$ and ΔH for cotton

from Table II into eq. (11) gives $K_1 = 24$ mg/cm sec, in close agreement to 27 mg/cm sec obtained from the 45° test data (Fig. 6).

It is also clear from column 4 of Table II that, of the fabrics burned in the calorimeter, cotton has the largest rate of heat release. One should not interpret this to mean that cotton is the most hazardous of the fabrics listed in Table II, since the hazard must include ease of ignition and total amount of heat released once ignition occurs. The high rate of heat release would, however, indicate that once ignition occurs, the individual has less time to take evasive action before a second-degree burn results. That is to say, the hazard of a flammable fabric must take into account ignition properties, flame spread rate, rate of heat release, and total heat release. For this reason, the heat-transfer data obtained by Brown and Vassalo[6] were interpreted in terms of the time required to produce second-degree burns once ignition occurred.

The unpredictable behavior of the fabric blends is of considerable concern and significance. As shown in Table II, the total heat released by a 90/10 cellulose triacetate/nylon blend is much higher than that expected from the values for total heat released from pure cellulose triacetate and pure nylon. If one assumes that the heats of release are additive, one would expect a value of 3148 cal/g for the 90/10 cellulose triacetate/nylon blend. However, the value obtained was greater by 350 cal/g. The residue yield indicates an increased combustion of either, or probably both, components of the blend. Within the experimental error, the rate of heat release of this blend is the same as that of pure cellulose triacetate, although the rate of heat release of the pure nylon is only about one third that of pure cellulose triacetate.

More studies of blends are currently under way that may offer added information about the behavior of blends in fires.

SUMMARY

A calorimeter was designed to measure the rate and total heat evolved when fabrics are burned in air. Measurements were made on a total of 15 different fabrics. The rate of heat release was determined for cotton of six different fabric weights. The rate of heat release for steady-state burning at an angle of 45° for a 5.1-cm-wide specimen was 78.8 cal/sec cm of flame front, independent of fabric weight.

Approximately 90% of the standard heat of combustion of cellulose is released by cotton during combustion in air. Of the nine different types of fabrics tested, poly(ethylene terephthalate) (polyester) released the lowest percentage (41.6%) of its standard heat of combustion.

The total amount of heat released from the 90/10 cellulose triacetate/nylon blend was considerably higher than that expected from assuming that the heats of the pure components are additive. The residue yield from the blend suggests that one or both of the components for the blend undergo more complete combustion in the blend.

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