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AN EVALUATION OF THE SETCHKIN METHOD FOR DETERMINING THE IGNITION PROPERTIES OF PLASTICS

by

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PREFACE

The examination of a test procedure by what is now commonly known as a round-robin test program is a well-established practice. Attention is usually focussed on a particular problem area, and the exchange of experience between laboratories is almost always useful and sometimes stimulating. The work carried out by the Fire Research Section of the Division in the course of a round-robin program on the measurement of certain properties of plastics is now reported. The author is a physical chemist and a research officer in the Fire Research Section having a special interest in the chemical changes in materials produced by heating such as may occur in fires.

Ottawa
June 1960

N. B. Hutcheon
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1. INTRODUCTION

In 1955, Committee D-20 on plastics of the American Society for Testing Materials recognized the need for a suitable method of determining the ignition properties of plastic materials. Its subcommittee on thermal properties was therefore charged with the responsibility of studying the problem and of developing a testing procedure which could later be considered for adoption as a standard.

A survey was first carried out of the literature on existing methods which might find application in the field of plastics. Of the two papers found to be of particular significance, the one published in 1949 by Setchkin (1) deserved special consideration. Setchkin's work was later used by the subcommittee as the basis in preparing a tentative testing procedure by which flash- and self-ignition temperatures could be determined using a constant temperature method.

Various laboratories, through their representatives on the subcommittee, were then asked to participate in a round-robin series of tests aimed at evaluating the suggested procedure. The Division of Building Research, National Research Council of Canada, is represented on the subcommittee by Mr. J.E. Hanna of the Canadian Government Specifications Board. Since the Fire Research Section of this Division had recently acquired a Setchkin Ignition Apparatus to study the ignition characteristics of solid materials

in general, the Division was pleased to participate in the round robin along with six American organizations:

1. E.I. duPont de Nemours and Co., Inc., Wilmington, Delaware.
2. Rohm and Haas Co., Bristol, Pennsylvania.
3. The Dow Chemical Co., Midland, Michigan
4. Monsanto Chemical Co., St. Louis, Missouri.
5. International Business Machines Corporation, Endicott, New York.
6. National Bureau of Standards, Washington, D.C.

2. SCOPE

It was the intention in this series of tests to use two main ignition characteristics of plastics for the comparative evaluation of the proposed testing procedure. The first is the flash point or flash-ignition temperature, that is, the lowest temperature at which the specimen develops combustible gas at a sufficient rate to form an ignitable mixture with ambient air. The other is the self-ignition temperature or the lowest temperature at which, in the absence of an igniting source, self-heating of the specimen will proceed at a sufficient rate to carry the ignition process to visible ignition (flame or glow).

For the round-robin tests, five plastic materials were selected by the subcommittee, and appropriate quantities of each were distributed to the seven participating laboratories with instruction that they be tested according to the suggested method.

The materials selected were:

1. A polyamide: Zytel 101, HC-10, lot 4217B
2. A polyester-fiberglass: sample No. RP-D856C
3. A polystyrene: Styron 666 M27 Clear 71, lot PB73
4. A polyethylene: Polyethylene 610M Nat. No. 1 (M.I.=50), batch 705723
5. An ethyl cellulose: Ethocel 880-1 27 Nat. 2, lot 6084, P-10.

All of the materials were supplied in pellet form except for the polyester-fiberglass which consisted of a 1/16-inch thick sheet that had to be cut in 3/4-inch square pieces for the test.

3. EXPERIMENTAL DETAILS

3.1 Apparatus

The apparatus described in the method of test is essentially that developed by Setchkin (1) except for minor modifications in the method of suspension of the specimen. It is shown with other required instrumentation in Fig. 1. This same apparatus is also used by the American Society for Testing Materials in the determination of the non-combustible character of elementary materials (2) and is now available commercially as the "Setchkin Self-Ignition Apparatus for Solids" (3).

3.1.1 Furnace

The furnace (Fig. 2) consists primarily of two 10-inch-long refractory tubes of different diameter arranged coaxially with axes vertical. The inner tube, with a 3-inch bore, forms the combustion chamber proper. The outer tube, with a 4-inch bore, serves as furnace tube and is heated electrically by means of an electric coil of 16-gauge Brown and Sharpe nickel-chrome wire

wound around the exterior of the tube. The coil is protected with alundum cement; asbestos wool provides the required insulation.

The space between the two tubes is used for circulation and heating of the combustion air which is admitted tangentially at a controlled rate near the top of the annular space. To provide access of this pre-heated air to the combustion chamber, the inner tube rests on spacer blocks at the bottom of the furnace.

The cover, made of transite, has a circular opening (1 1/8 inch in diameter) for the passage of smoke and gases, and is provided with a pilot flame burner for flash-point determinations. An inspection plug at the bottom of the furnace can be removed for the cleaning out of residues that may accumulate during tests.

3.1.2. Thermocouples

Three chromel-alumel (0.020 inch) thermocouples are used for temperature measurements: thermocouple T_1 at the centre of the sample to detect any self-heating within the sample; thermocouple T_2 below the sample to record the temperature of the pre-heated air; and thermocouple T_3 on the heating coil.

In this series of tests, all three thermocouples were connected to a multipoint recorder. Temperatures at thermocouple T_1 were recorded at 8-second intervals; temperatures at the other two thermocouples at 16-second intervals.

3.1.3. Sample container

The sample container consists of the bottom half of a 1/2-ounce metal salve container, style 100. It is held in a ring of 1/16-inch stainless steel welding rod welded to a length of same rod extending through the cover of the furnace (Fig. 3).

3.1.4. Flowmeter

The rate of air flow through the furnace is controlled by means of a Brooks Sho-rate 150 rotameter equipped with a constant differential relay to maintain a constant flow rate irrespective of line pressure variations. The "Tru-taper" tube (size 6-15-2) of the rotameter is calibrated directly in units of 0.02 cubic foot per minute over a range extending from 0.06 to 0.80 cubic foot per minute. Air flows can thus be adjusted to the closest 0.01 cubic foot.

3.2. Procedure

It has been established by C.R. Brown (4) that when operating under dynamic conditions for the determination of ignition temperatures, the rate of air flow past the sample can appreciably affect the end results. Consequently, where minimum ignition temperature values are desired, optimum air flow conditions have to be predetermined. It is usually found more expeditious, particularly when a constant temperature method of test is used, to carry out these preliminary determinations by running quick tests under rising temperature conditions. In the test method, it is therefore necessary to proceed in a step-by-step manner whereby:

(i) first, the optimum flow of air is determined by running tests at various air flows under rising temperature conditions using a rate of air temperature (T_2) increase of approximately 1100°F (or 600°C) per hour;

(ii) secondly, approximation of the ignition temperature is obtained by running tests at the optimum rate of air flow under rising temperature conditions using a rate of air temperature (T_2) increase of approximately 600°F (or 300°C) per hour, and

(iii) finally, the minimum ignition temperature is determined under constant temperature conditions.

3.2.1. Determination of optimum rate of air flow

3.2.1.1. Flash-ignition temperature.- Approximately 3 grams ± 0.5 grams of the plastic material is placed in the furnace and the air flow is set to give a linear air velocity of 5 feet per minute past the sample. The variable transformer controlling the current through the heating coil is then adjusted to provide a rate of rise in temperature at thermocouple T_2 of approximately 1100°F (or 600°C) per hour and the pilot flame is lit.

The recorded air temperature (thermocouple T_2) at which the temperature at the centre of the sample (thermocouple T_1) starts to rise rapidly is reported as the approximated flash-ignition temperature at this particular rate of air flow.

The procedure is repeated at linear velocities of flow of 15 and 20 feet per minute and in each case the flash points are noted. The rate of air flow at which the lowest flash-ignition temperature is obtained is referred to as the optimum flow rate.

3.2.1.2. Self-ignition temperature. - The same procedure as given in 3.2.1.1. is repeated without the pilot flame and the optimum flow for determining self-ignition temperature is obtained. There are cases, however, where it is not possible during the rising temperature run to reach ignition because some plastics, e.g. polystyrene, boil away before self-ignition can take place.

3.2.2. Approximation of ignition temperature

3.2.2.1. Flash-ignition temperature. - With the sample of material in the furnace and the air flow set at the optimum flow found under 3.2.1.1., the electric current is adjusted to provide a rate of rise in temperature at thermocouple T_2 of approximately 600°F (or 300°C) per hour and the pilot flame is lit. The temperature T_2

at which the temperature at the centre of the sample (thermocouple T_1) starts to rise rapidly is reported as the approximated flash-ignition temperature.

3.2.2.2. Self-ignition temperature. - The same procedure as in 3.2.2.1. is repeated in the absence of an igniting source and the approximated self-ignition temperature is obtained.

3.2.3. Determination of minimum ignition temperature

3.2.3.1. Flash-ignition temperature. - With air flow rate set at the optimum flow the furnace temperature (T_2) is stabilized within 20°F (or 10°C) below the approximate flash temperature found in 3.2.2.1. The sample is then introduced in the furnace and the pilot flame lit.

If ignition occurs, the same procedure is repeated using a new sample at a temperature 20°F lower and so on until no ignition is detected over a minimum period of 30 minutes. When a temperature T_2 is thus reached at which no ignition occurs, a second run is conducted at the same temperature to ascertain that this is truly below the self-ignition temperature.

The lowest air temperature T_2 at which ignition occurs is reported as the minimum flash-ignition temperature.

3.2.3.2. Self-ignition temperature. - With air flow rate set at the optimum flow, constant temperature runs are conducted as in 3.2.3.1. without the pilot flame, starting at a temperature T_2 20°F (or 10°C) lower than the approximate self-ignition temperature found in 3.2.2.2.

The lowest air temperature (T_2) at which ignition occurs is reported as the minimum self-ignition temperature.

Where ignition could not be obtained during rising temperature runs (see 3.2.1.2.), it is wise to start the constant temperature runs at a temperature approximately 200°F (or 100°C) higher than the flash-ignition temperature previously determined (3.2.3.2.), using an air flow rate equal to that used in the flash-ignition temperature determination.

4. RESULTS

Because of limitations in the maximum current that could be passed through part of the testing equipment used when these tests were started, the first step of the procedure was by-passed and optimum flow rates were determined at a rate of temperature rise of 600°F instead of the 1100°F rate specified. This could not have any effect on the outcome of the tests but merely meant that a few additional hours would be required to complete the series.

The flash- and self-ignition temperatures obtained at the three specified air flows during these rising temperature runs are listed in Table I. Except for the polyester-fiberglass and the polystyrene samples, for which no self-ignition point could be obtained, optimum flow for self-ignition temperature determinations was similar to that found in flash-ignition tests, and corresponded in every case to a linear air velocity past the sample of 5 feet per minute.

In the cases of the polyamide and ethyl cellulose, however, there was at least one alternate optimum flow at which the flash-ignition point could have been determined, but since the

value of 5 feet per minute fitted every case, it was adopted as the optimum flow even when an alternative existed.

Tables II and III summarize results obtained during constant temperature runs aimed at determining minimum flash- and self-ignition temperatures respectively. These minimum ignition temperatures have been listed separately in Table IV for each of the five plastic materials tested.

5. COMMENTS

Results from four of the participating laboratories are still unavailable and it would therefore be presumptuous to express at this time an opinion on the merits of the Setchkin method in the determination of the ignition properties of plastic materials. Complete results from three laboratories indicate, however, that good correlation between the participating agencies can be expected.

As a result of this laboratory's participation in the tests, it has been possible to appreciate some of the merits of the proposed test method, e.g. elaborate but well detailed procedure, and simple design and low cost of testing equipment. There are, however, slight modifications either in procedure or equipment that would probably improve further the over-all applicability of the method. These are discussed below.

5.1. Temperature scale: Fahrenheit versus Celsius

In the suggested procedure supplied to the participating laboratories, all temperatures are expressed in Celsius degrees only. At this stage in the development of the testing procedure this may be unimportant, but since some laboratories are equipped with temperature recording instruments calibrated in Fahrenheit

degrees, it is suggested that both temperature values (Fahrenheit and Celsius) be given. This would be in accord with what seems to be a general policy adopted by the ASTM in the publication of its standards.

5.2. Optimum flow-rate determinations

According to the suggested procedure, the optimum flow rate is determined by conducting series of tests at a rate of heating of 1100°F per hour using successive air flows corresponding to linear air velocities past the sample of 5, 15, and 20 feet per minute. Although, as mentioned previously, these determinations had to be performed in this laboratory at a rate of temperature rise of 600°F, it should be remarked that, for all the materials tested under these conditions, the optimum flow rate was found to be the lowest of the three specified, that is, that corresponding to a linear velocity of 5 feet per minute. No lower flow rates were experimented upon, but it is questionable whether lower ignition temperature values would have been obtained had lower flows been used.

One item in the procedure that is not understood in this laboratory is the reason for omitting the linear velocity of 10 feet per minute.

5.3. Modification of equipment

For the constant temperature runs, the furnace has to be preheated to a predetermined constant temperature before the sample can be introduced. It has been found in this laboratory that during the preheating period a spare cover for the furnace helped greatly in manipulating the sample, especially its introduction in to the furnace.

The procedure adopted in the DBR laboratory was as follows: during the preheating period, the spare cover was set on the furnace and the cover used normally during the tests was held upright on a laboratory support close to the furnace. When the furnace temperature reached steady conditions, the sample container with the required quantity of plastic material was set in position on its holder, the pilot flame lit where specififed, and the normal cover was rapidly substituted for the spare cover. The changeover operation could be done in a matter of a few seconds. Blank runs had shown that the substitution of a cold cover for the hot one did not increase the temperature drop normally experienced during the introduction of the sample following the specified procedure.

REFERENCES

1. Setchkin, N.P. A method and apparatus for determining the ignition characteristics of plastics. Journal of Research of the National Bureau of Standards, Vol. 43, 1949, p.591. (Research Paper RP2052)

2. Method of test for defining non-combustibility of building materials. American Society for Testing Materials, Standard E 136-58T.

3. Setchkin Self-Ignition Apparatus for Solids, Model CS88 Custom Scientific Instruments, Inc., Kearny, N.J.

4. Brown, C.R. The determination of the ignition temperatures of solid materials. Fuel, Vol. 14, 1935, p.14, 56, 80, 112, 149.

TABLE I

DETERMINATION OF OPTIMUM FLOW RATE

Plastic Material	Rate of Temperature Rise (°C/Hour)	Air Velocity Past Sample (Ft/Min)	Flash-Ignition Temperature (°F)	Self-Ignition Temperature (°F)
Polyamide	600	5	830	830
	600	15	835	940
	600	20	830	940
Polyester-Fiberglass	600	5	740	No ignition
	600	15	790	
	600	20	820	
Polystyrene	600	5	700	No ignition
	600	15	760	
	600	20	800	
Polyethylene	600	5	680	670
	600	15	710	675
	600	20	720	720
Ethyl Cellulose	600	5	600	580
	600	15	600	615
	600	20	600	610

TABLE II

DETERMINATION OF FLASH-IGNITION TEMPERATURE

Plastic Material	Temperature at Thermocouple T_2 (°F)	Air Velocity Past Sample (Ft/Min)	Observations
Polyamide	795	5	Flashed at 6 minutes
	775	5	Flashed at 9½ minutes
	755	5	No ignition
	755	5	No ignition
Polyester-Fiberglass	730	5	Flashed at 10½ minutes
	710	5	No ignition
	710	5	No ignition
Polystyrene	680	5	Flashed at 11 minutes
	660	5	No ignition
	660	5	No ignition
Polyethylene	670	5	Flashed at 5½ minutes
	645	5	Flashed at 10½ minutes
	625	5	No ignition
	625	5	No ignition
Ethyl Cellulose	550	5	Flashed at 5 minutes
	530	5	No ignition
	530	5	No ignition

TABLE III

DETERMINATION OF SELF-IGNITION TEMPERATURE

Plastic Material	Temperature at Thermocouple T_2 (°F)	Air Velocity Past Sample (Ft/Min)	Observations
Polyamide	790	5	Flashed at 8 minutes
	770	5	No ignition
	770	5	No ignition
Polyester-Fiberglass	845	5	Flashed at 3 minutes
	830	5	No ignition
	830	5	No ignition
Polystyrene	900	5	Flashed at 1 minute
	885	5	Volatilized
	880	5	Volatilized
Polyethylene	670	5	Flashed at 6 minutes
	655	5	No ignition
	655	5	No ignition
Ethyl Cellulose	570	5	Flashed at 5 minutes
	550	5	No ignition
	550	5	No ignition

TABLE IV

MINIMUM FLASH- AND SELF-IGNITION TEMPERATURES

Plastic Material	Flash-Ignition Temperature			Self-Ignition Temperature		
	Optimum Air Velocity (Ft/Min)	Temperature (°F)	(°C)	Optimum Air Velocity (Ft/Min)	Temperature (°F)	(°C)
Polyamide	5	775	413	5	790	421
Polyester-Fiberglass	5	730	388	5	845	452
Polystyrene	5	680	360	5	900	482
Polyethylene	5	645	341	5	670	354
Ethyl Cellulose	5	550	288	5	570	299

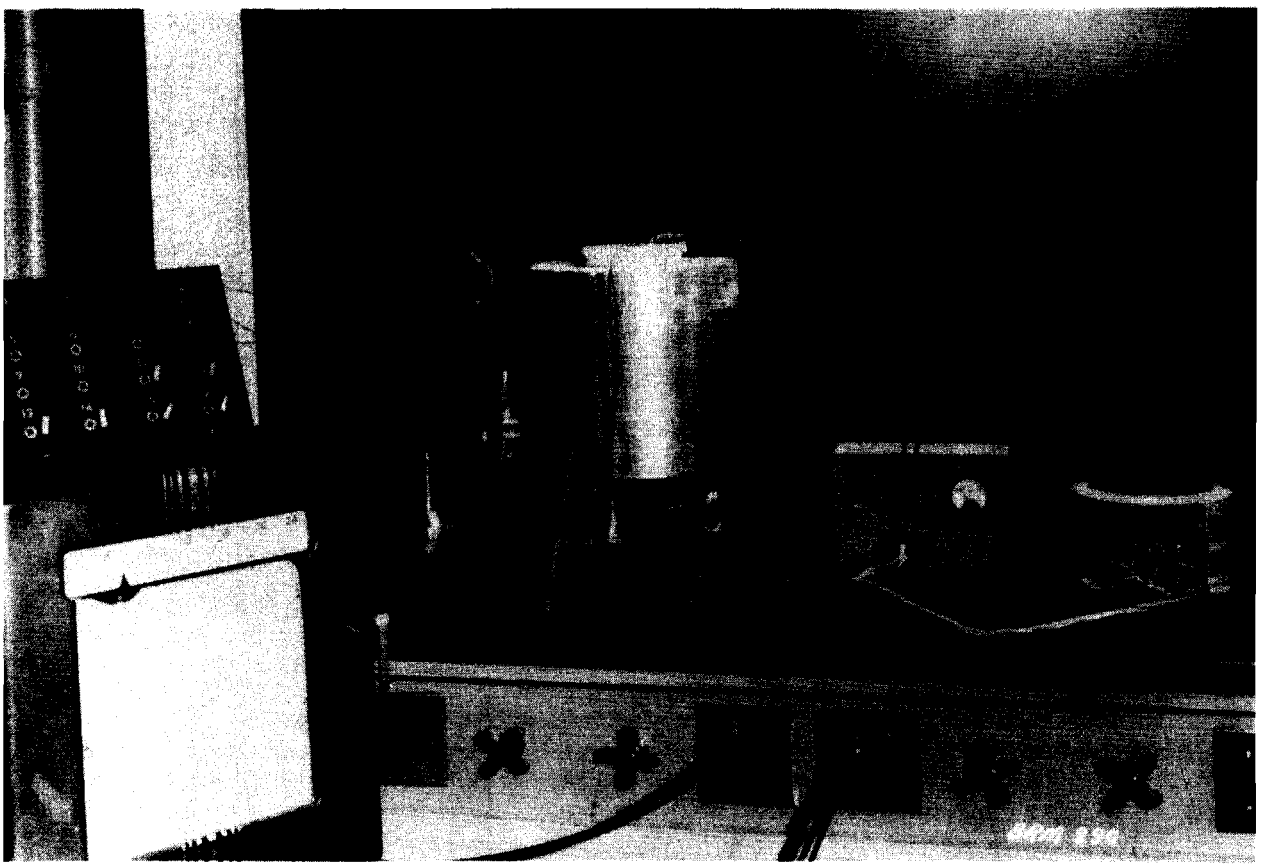

FIGURE I - INSTRUMENTATION USED FOR THE TESTS.

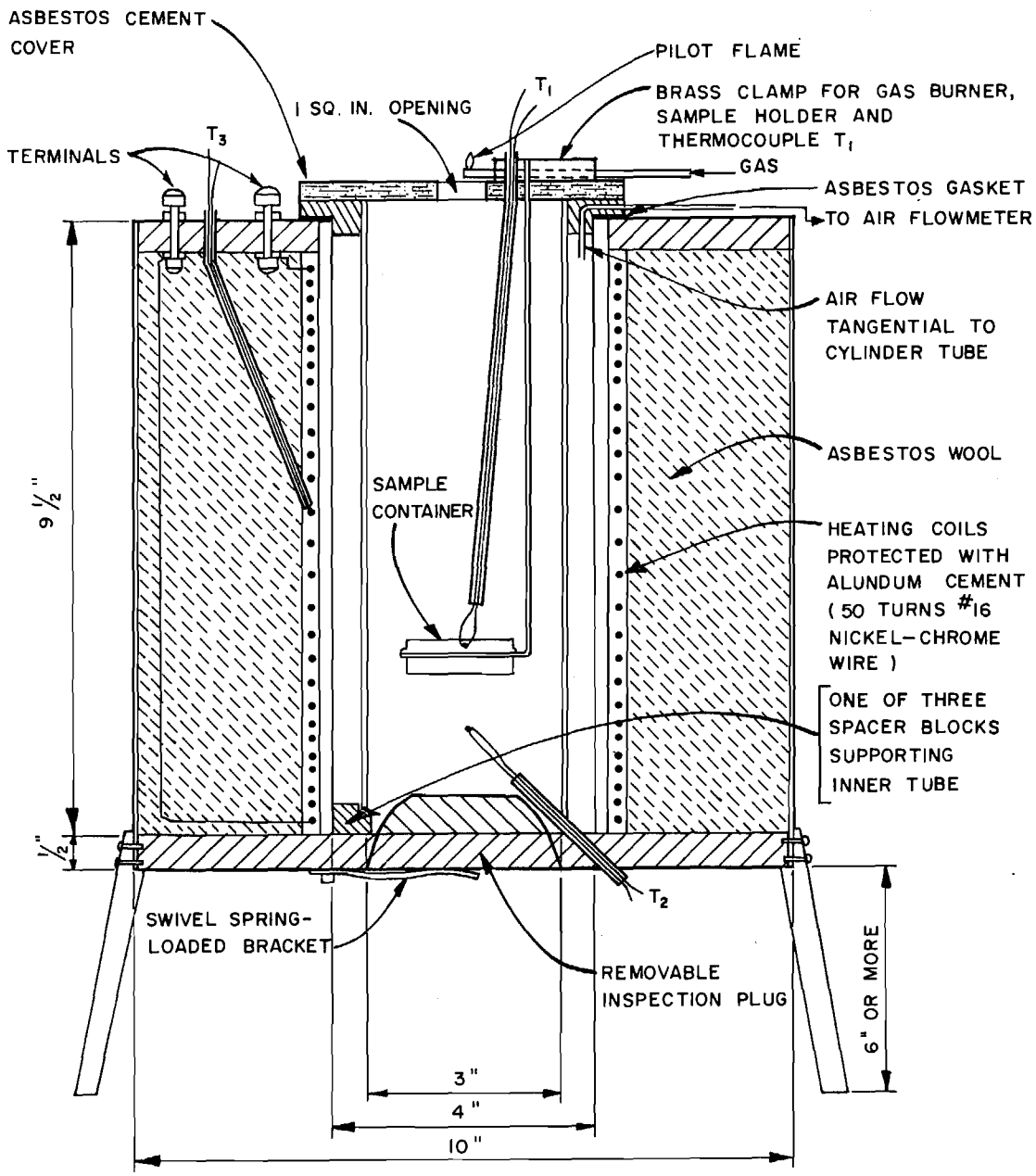

FIGURE 2 SETCHKIN SELF-IGNITION APPARATUS

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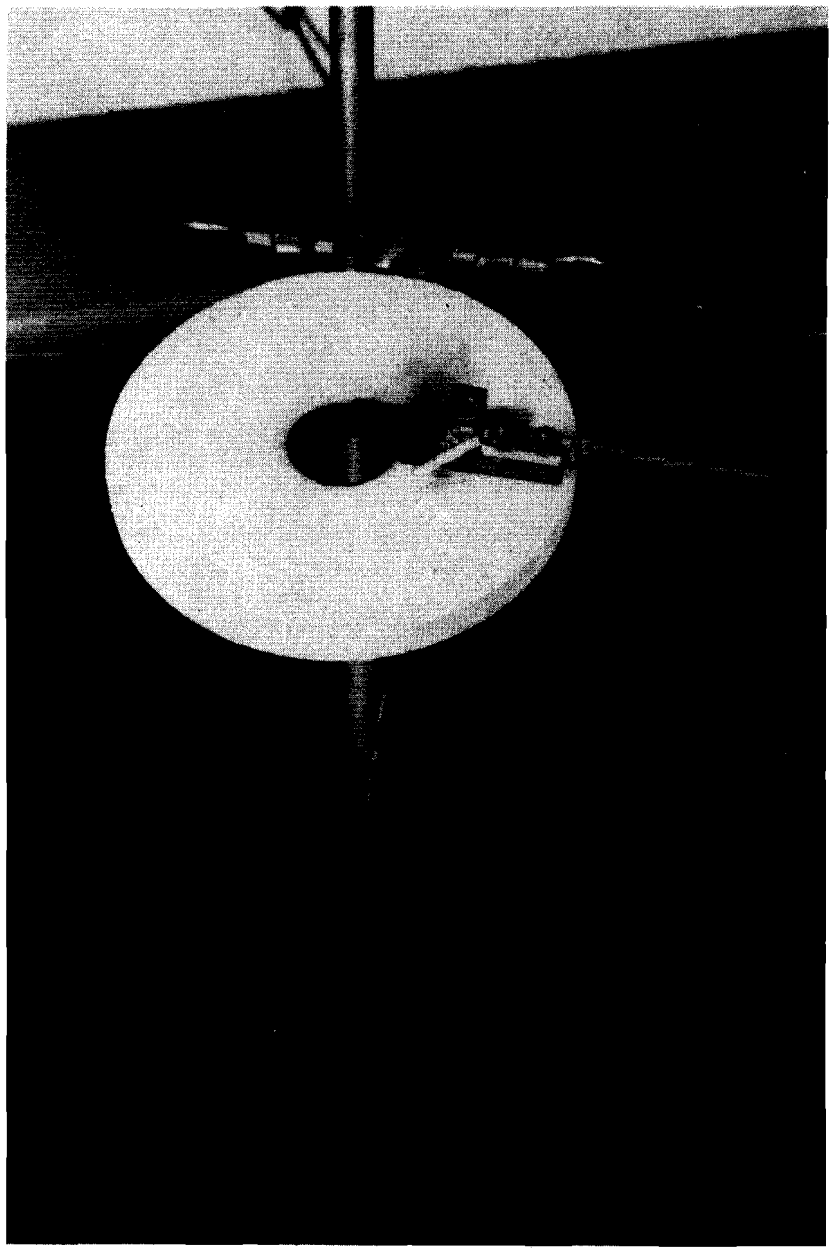

FIGURE 3 - COVER OF FURNACE AND SAMPLE HOLDER. (BRM 274)