AN ABSTRACT OF THE THESIS OF <u>Charles William Harrison</u> for <u>M.S.</u> in <u>M.E.</u> (Name) (Degree) (Major) Date Thesis presented <u>July 19, 1946</u> TITLE <u>AN APPARATUS FOR DETERMINING THE IGNITION</u> <u>TEMPERATURES OF ORGANIC SUBSTANCES.</u> Abstract Approved:

In summary of this paper, the work has covered a study of the different methods and equipment for determing the ignition temperatures of various substances. The results of the survey may be condensed by stating that approximately thirty-eight terms are used in designating the ignition temperature with approximately an equal number of definitions.

(Major. TTOTOBOAT)

A study of equipment previously used resulted in the design and building of newer, and it is believed, improved the accurate determination of ignitiom temperatures.

Continuation of the study and further experimental determinations, together with the establishment of a simple criterion defining the ignition phenomena should yield information which will contribute to public safety.

AN APPARATUS FOR DETERMINING THE IGNITION TEMPERATURES OF ORGANIC SUBSTANCES

by

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TABLE OF CONTENTS

Page

INTRODUCTION	1
EQUIPMENT	4
Furnace Process Controller Recording Potentiometer Rotometer Variac Relays	4 4 9 16 16
OPERATION OF EQUIPMENT	18
PRELIMINARY TESTS	25
CONCLUSIONS	32
BIBLIOGRAPHY	34

ILLUSTRATIONS

Figui	99	Page
1.	Furnace assembly	5
2.	A.C. wiring diagram	10
3.	Thermocouple circuits	11
4.	Schematic diagram of Brown continuous balance system	12
5.	Calibration curve with aluminum float	15
6.	Interior of furnace showing sample	21
7.	Rotameter correction factor nomograph for gases	23
8.	Furnace time-temperature curves with fixed circuit transformer settings as the parameter	24
9.	The apparatus used in determining ignition temperatures	17
10.	The recorded ignition temperature of above test sample	26
11.	Typical ignition temperature determination	27
12.	Effect of heating rate upon ignition temperature with constant rate of air flow	29
13	Cam designs	31

AN APPARATUS FOR DETERMINING THE IGNITION TEMPERATURES OF ORGANIC SUBSTANCES

INTRODUCTION

A considerable number of terms have been used to designate the initial burning temperature of a substance. One of the principal causes of the confusion is the lack of a clear concept of ignition phenomena. Unless this process is more completely understood and an agreement reached as to what factors constitute the process no single or simple definition is possible, and the published data are of little practical value. The need has been of an apparatus that is capable of simulating practical conditions and capable of yielding information that will contribute to the public safety.

Such an apparatus must be elastic as to the experimental conditions so that it may be adapted to any of the numerous problems in the storage of materials in hazardous locations. In the studies of case histories of fires it was found that sufficient information for sound conclusions was seldom available to the men writing the reports and some of the more or less intuitive conclusions arrived at seem to be physically impossible. A survey and analysis of the existing literature on ignition temperature, indicates that there are three principal reasons for the diversity of results.

First, the various definitions which have been given for ignition temperature are not on the same basis with respect to the phenomena of ignition. The process has long been known to man and it is rather difficult to define; to quote some authors:

Beyersdorfer (1)+ Ignition is an occurrence which produces a visible combustion. Bunsen (2). The lowest temperature at which the constituents of a gas mixture combine. Nernst (3), That temperature to which a point of the system must be heated to cause combustion. Plenz (4), The temperature at which fuel, in contact with air at the same temperature, undergoes oxidation at such a rate that a marked temperature rise and production of combustion products result. Van't Hoff (5), Gibbs (6), Schultes (7), The temperature at which the rate of generation of heat becomes greater than its rate of dissipation. Brown (8), The temperature in the combustible at which the rate of heat developed by the reactions inducing ignition just exceeds the rate +Numbers in parentheses refer to references listed in appendix.

at which heat is dissipated by all causes, under the given conditions.

Secondly, the experiments for the determination of ignition temperature vary widely in the equipment used, procedure, and applied experimental conditions. Continued improvement of experimental procedure and equipment should add materially to the solution of the problem.

Finally the materials themselves vary in both physical and chemical states.

Because of the lack of reliable and consistent ignition data on such materials as paper, wood fiber etc aparatus has been designed and built to determine the ignition temperatures under systematic variations of time rate to temperature increase, volume of air flow, size and kind of specimen and humidity. In the preliminary tests here reported air at atmospheric pressure and humidity was used; however, by minor modifications the equipment may be adapted to tests under varying conditions of atmosphere composition, pressure and moisture content.

DESCRIPTION OF EQUIPMENT

1. Furnace:

The furnace is constructed of two rectangular boxes, one within the other with vermiculite insulation between. (See Figure 1). The legs, which are electrically welded to the bottom plate, support the total system above the table and allow a circulation of air under the furnace. The electrical outlets are made through transite bolted to the back of the furnace.

The inner tank has six 500 watt and four 350 watt Chromalox electric strip heaters bolted to the outer surface to provide the heat input when energized. The packing glands through which the pyrex combustion tube passes are welded in the ends of the inner tank four inches below the top and in the center of the tank. These glands are made of two inch standard pipe $5\frac{1}{2}$ inches long with a packing stop welded inside 1.75 in from the outside threaded end of the gland. A standard two inch pipe cap is bored to accommodate the pyrex tube and used to compress the asbestos packing around the tube.

2. Process Controller.

The Process Controller consists of six elements, namely:

- 1. Synchronous motor clock (24 hour)
- 2. Gear arrangement
- 3. Cam

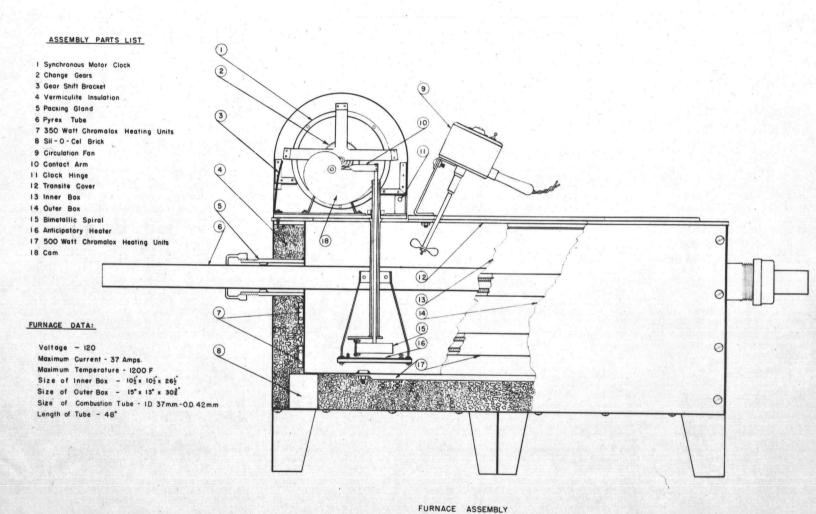


Figure 1.

СЛ

- 4. Contact arm
- 5. Bimetallic element
- 6. Anticipatory heater

The synchronous clock is the power and timing element of the cam drive. Upon the shaft of the clock is mounted a stub shaft with four gears of different sizes to provide a change gear ratio for the cam drive (See Figure 1). The cam thus far used provides a process of linearly increasing temperature with time, but cams for other schedules may readily be designed and used. The outline of the cam is determined by the desired process.

The contact arm, (See Figure 1) is made of spring steel and when in contact with the cam excites a Mercoid relay and closes the heating circuit. The edge of the arm in contact with the cam is ground to a hypocycloid curve along the contact edge in order to maintain a volute form cam for linear movement of arm with respect to temperature.

The bimetallic element (See Figure 1), consists of two metals of unlike compositions and different rates of thermal expansion. The metals are bonded together throughout their contact surfaces, and in this equipment for mechanical convenience the bimetallic strip has been wound in plane spiral form in preference to using a flat strip. The metal operating range is from room temperature to 1000 F. The bimetallic element rotates the contact arm away from the cam as the temperature increases. The element was designed in this manner to operate in conjunction with normally open relays. The manufacturers data sheets for spiral bimetallic elements give an empirical equation for angular rotation as:

 $\Theta = K (T_1 - T_2) L$

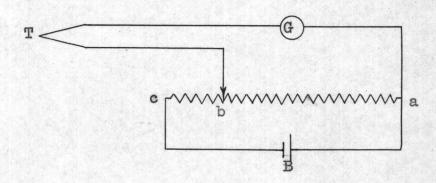
 θ = Angular rotation of central shaft K = Constant of metal and dimensions T_1 = Final temperature T_2 = Initial temperatures L = Length of strip

In this element the length is variable and affords a variable magnification of the temperature-time relation in any desired process (or cam layout).

The anticipator (See Figure 1) is a small heating element of high resistance to predict or anticipate the amount of energy required for the contact arm to follow the cam outline. The associative energy relations in mass and temperature between the anticipatory heater tend to control the off and on frequency of the current in the furnace coils. This auxiliary heater has been fabricated with a low mass so the temperature oscillations will be damped within the inner box air chamber. This element has also been designed to vary the sensitivity of the control circuit by moving it up or down the shaft housing. High sensitivity may be obtained by placing the predictor close to the bimetallic element.

3. The Recording Potentiometer

The most accurate method for measuring the electromotive force of a thermocouple is by use of a potentiometer. The fundamental principles of this instrument is illustrated by the diagram below.



A constant current from the battery B flows through the slide wire resistance, abc. One wire of the couple T is connected to the movable contact b and the other wire, in series with a sensitive galvanometer, is connected to a. The contact b is moved until the galvanometer reads zero thus showing that no current is flowing through the thermocouple circuit. When this balance of zero setting is made, the true electromotive force is equal to the potential drops across ab. This follows from Ohm's law, e = ir, where i is the current flowing through the resistance r = ab. (9). As the emf is always adjusted to a definite value the slidewire may be graduated to read millivolts or temperature directly.

The recording potentiometer (See Figure 3 and 4) used in this project is fundamentally of the same circuit except that the instrument recognizes the direction and magnitude of the unbalance in the potentiometer and in turn is balanced by a servo-motor mechanism, thus eliminating manual balancing. The recording mechanism is designed with an integral selector switch capable of serially switching four different thermocouples into the potentiometer circuit and printing on the chart for each couple a numeral designating the couple and a cross indicating the temperature of the thermocouple.

4. The Rotameter

The Rotameter is a flow measuring instrument constructed with a float in a calibrated tube. The inner bore of the tube is ground on a modified taper and the action of the float in this tube provides a variable annular orifice at different rates of flow. The tube is etched in terms of metric measurements and calibrated in rate of flow in English measure.

Since the float is in equilibrium under a constant rate of flow the summation of forces acting upon the float is equal to zero. The downward force acting

LIST OF APPARATUS SHOWN ON DIAGRAM

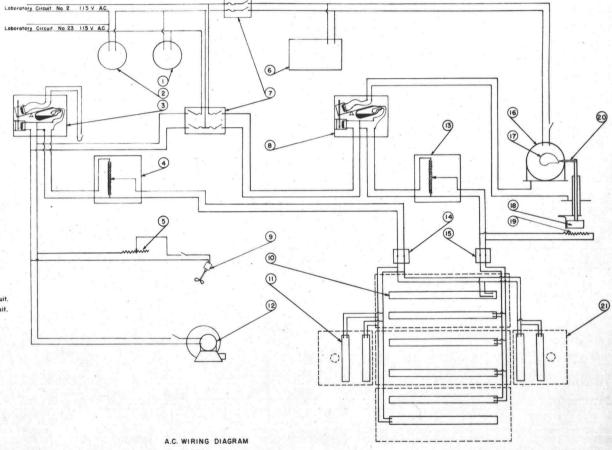
- Voltmeter
- 2 Ammeter

i.

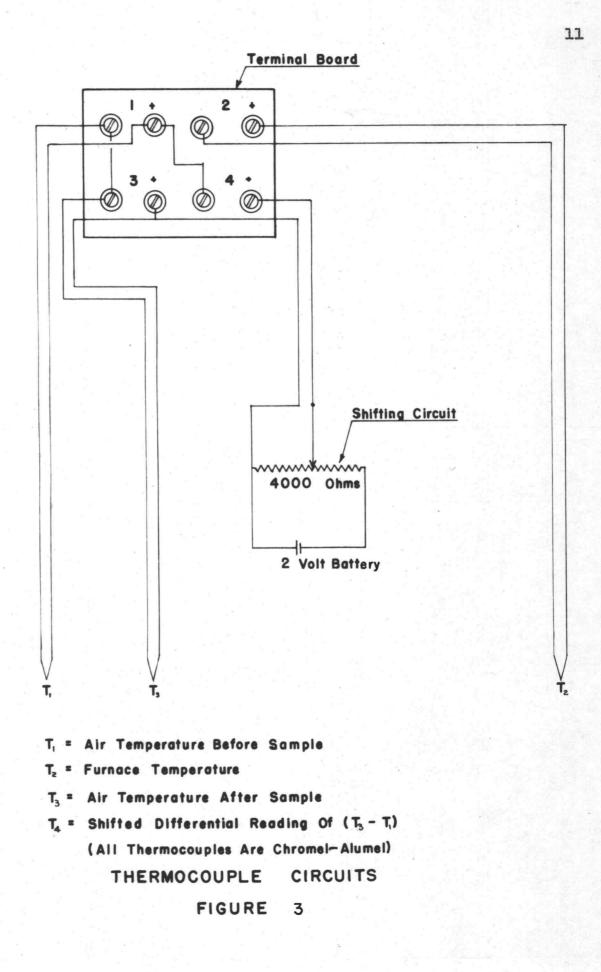
- 3 Mercoid Relay (Circuit No. 2)
- 4 Variac (Circuit No. 2)
- 5 Variable Resistance
- 6 Brown Recording Pyrometer
- 7 Switch and Fuse Board
- 8 Mercoid Relay (Circuit No. 1)
- 9 Circulating Fan Motor
- 10 500 Watt Chromalox Heating Unit
- II 350 Watt Chromalox Heating Unit
- 12 Blower Motor
- 13 Variac (Circuit No. 1)
- 14 Terminal Board (Circuit No. 1)
- 15 Terminal Board (Circuit No. 2)
- 16 Synchronous Motor Clock
- 17 Cam
- 18 Bimetallic Spiral
- 19 Anticipatory Heater
- 20 Contact Arm
- 21 Furnace Outline

NOTE

Circuit No. 1 used with reference to control circuit. Circuit No. 2 used with reference to fixed circuit.

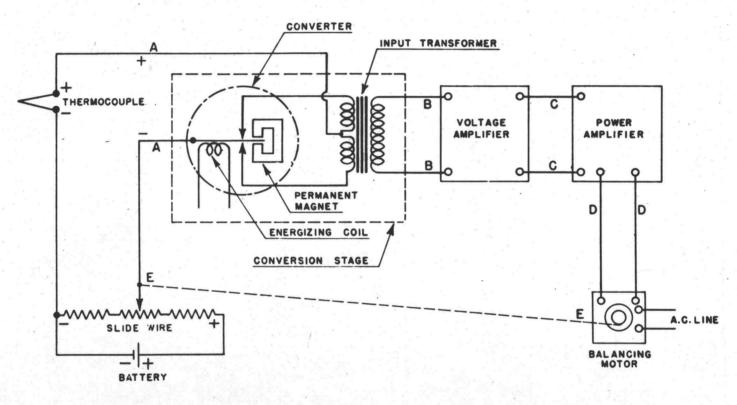






INSTRUMENT DATA:

Model No. 153X65P4 - X-2 Serial No. 328321 Chart No. 552 Range OF - 2000 F Voltage 110 - 125 Amperage .55



SCHEMATIC DIAGRAM OF BROWN CONTINUOUS BALANCE SYSTEM

Figure 4

upon the float is equal to the weight of the float minus the weight of the fluid displaced. Expressed in an equation of densities and volume.

$$F = V_{f} (\ell_{f} - \ell_{m})$$

$$F = Force$$

$$V_{f} = Volume of float$$

$$\ell_{f} = Density of float$$

$$\ell_{m} = Density of fluid medjum$$

The upward force acting on the float is equal to the product of pressure difference and projected axial of the float.

$$F = A_{f} (P_{1} - P_{2})$$

$$F = Force$$

$$A_{f} = Area of float$$

$$P_{1} = Pressure before float$$

$$P_{2} = Pressure after float$$

Converting to units of head of fluid across the floats the previous equations may be equated when equilibrium exists, the summation of forces being zero.

$$V_{f}(e_{f} - e_{m}) = A (P_{1} - P_{2})$$

Convert to units of measurements of head loss (h)

$$h = \frac{P_1 - P_2}{\ell_m}$$

$$P_t - P_2 = \frac{V_f(\ell_f - \ell_m)}{A_f}$$

$$h = V - \frac{(\ell_f - \ell_m)}{A_f}$$

From the ordinary flow equations

$$Q = A v$$
$$v = \sqrt{2gh}$$

Substituting terms (A in annular area around float)

$$Q = A\sqrt{2 \text{ gh}}$$

$$Q = A\left[\frac{2 \text{ g } V_{f}(e_{f} - e_{m})}{A_{f} e_{m}}\right]^{\frac{1}{2}}$$

Weight is product of density and volume.

$$W = Q \ell_m$$

$$W = A \left[2 g V_f (\ell_i - \ell_m) \ell_m \right]^{\frac{1}{2}}$$

$$A_f$$

These equations are the basic Rotameter equations and have assumed the orifice as 100% efficient. A coefficient of performance, C may be included.

Then

$$Q = C A \left[2 g V_{f} \left(\frac{\ell_{f}}{\ell_{f}} - \frac{\ell_{m}}{\ell_{m}} \right) \right]^{\frac{1}{2}}$$

and

$$V = C A \left[2 g V_{f} \frac{(l_{f} - l_{m})}{A_{f}} \right]^{\frac{1}{2}}$$

These equations reduce to $Q = K_{1}A$ and $W = K_{2}A$, which are straight lines, with a tube so ground that the area varies directly with the height a straight calibration curve results. The practical calibration curve (See Figure 5) deviates slightly from a straight line indicating friction effects at low flows or slight imperfections in grinding.

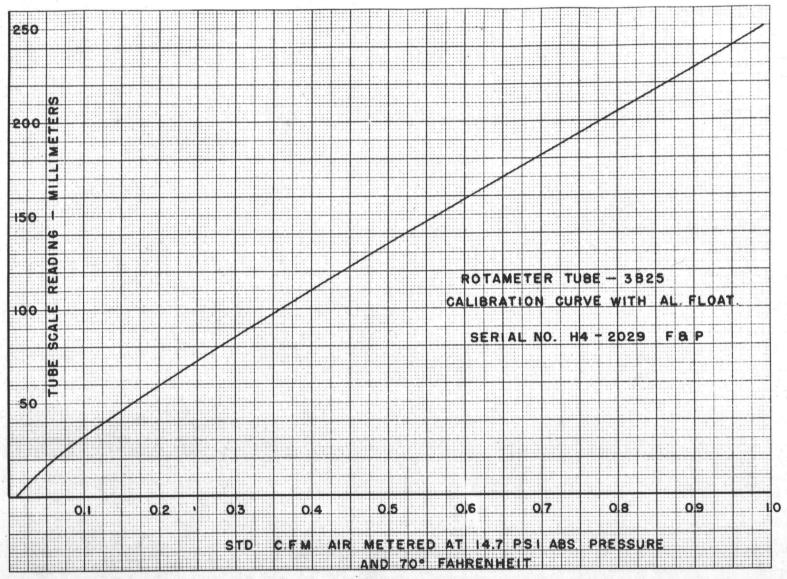


Figure 5

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5. The Variac

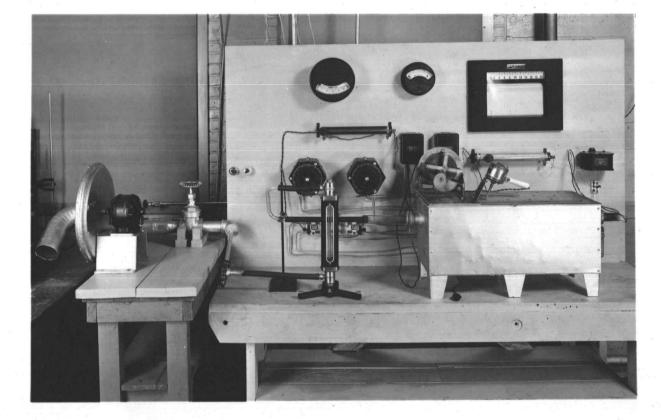
The variac may be classified as an auto-transformer and has only a single winding. (See Figure 2) A portion of this winding is used as both a primary and secondary winding. The output is taken off by two lines; one is common to the input and the other is connected to a brush that rotates over and in contact with the coil. Thus it is adjustable to any output voltage desired within the limits of the device.

6. Relays

The apparatus to control the electrical heating load used in the furnace consists essentially of two mutually inductive coils and a mercury contact cell.

The mercury cell and one coil are suspended on a fulcrum (See Figure 2). When the contact arm touches the cam the generation of current in the secondary coil creates a force which will tip the mercury cell and close one leg of the main circuit. This device makes possible the control of large amount of current by the use of a small amount. In this equipment the use of 10 watts controls 2400 watts.

The small amounts of current used in the miscellaneous motors such as the synchronous motor clock, induction motor on the blower drive, and the universal motor drive on the circulation fan are controlled by the use of single pole switches. (See Figure 2) The universal fan motor has a variable resistance in series with it to provide for speed control.



THE APPARATUS USED IN DETERMINING IGNITION TEMPERATURES

Figure 9

OPERATION OF EQUIPMENT

The accuracy and sensitivity of the measuring instruments and their simplicity of operation contribute to the value of the data in this problem.

The Rotameter is the atmosphere metering device, and from manufacturers data, the calibration of the device has been made by a number of individual tests by different men using the same procedure. By a comparison of the data the calibrations were found to vary no more than one percent, which is partially attributed to the error of reading the calibrations.

The Brown recording pyrometer has been compared to USBS Standards and has been found to meet the manufacturers standards of accuracy. In the temperature range of 0 F to 180 F the pyrometer is accurate to 0.6% or maximum deviation of 1.08 F. From 180 to 2000 F the accuracy of the instrument is within 0.06% of scale deflection. The accuracy of the temperature recording instrument and the present range of ignition determination limit the error of recordings to 0.6 F.

The change of flow of air through the system and the concurrent recordings may be used to check the sensitivity of the system.

The flow diagram for this apparatus involves the introduction of a metered supply of air (or modified atmosphere) into the pyrex tube. Heat passes from the heated tube into the air stream as it travels to the sample which is located at a fixed distance from the exhaust end of the tube. If the rate of air flow through the tube is changed in either direction the recorded temperature will change due to the difference in heat transfer.

In order to visualize the sensitivity of the equipment calculations of energy change resulting from a known change of air flow and the concurrent recorded temperature difference. The change of energy may be calculated from the formula:

$$\mathcal{Q} = \mathcal{C} (\mathbf{T}_1 - \mathbf{T}_2) W.$$

Q = Heat energy

T₁ = Temperature before change of flow rate
T₂ = Temperature after change of flow rate
W = Weight of air

The rate change of energy would be the product of a change in weight, the corresponding change in temperature and specific heat.

 $Q_1 - Q_2 = C (\Delta T) (\Delta W)$

From a known change in air supply of 0.025 efm changes the recorded temperature two degrees. Weight of air is the product of volume (V) divided by specific volume (\overline{V}) at the metering temperature.

$$\frac{V}{V} = \frac{0.025}{14} = 0.0017$$
 lb per min.

The Brown pyrometer records every five seconds, therefore; use the weight change during the recording period.

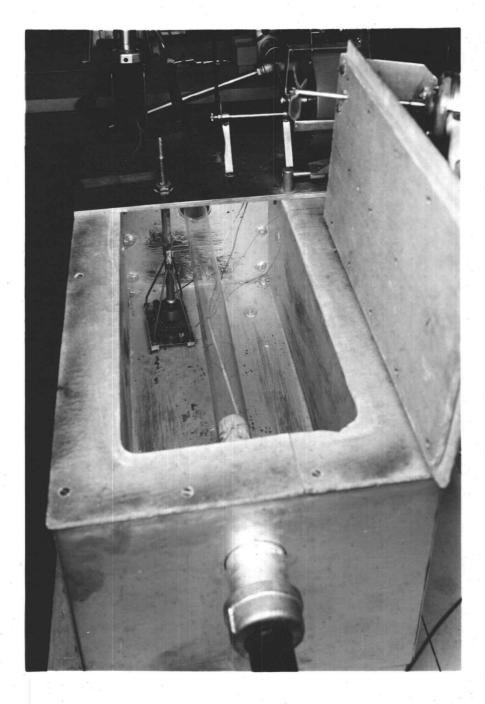
 $\frac{0.0017 \text{ lb per min}}{60 \text{ sec per min.}} \times 5 \text{ sec per reading} = 0.00014 \text{ lb per recording}$ Energy change per recording.

 $(Q_1-Q_2) = 0.24$ (2) (.00014) = 0.0000672 Btu per recording Convert to metric units

0.0000672 Btu per 5 sec x 252 Cal per Btu = 0.017 calorie per 5 sec

A recording that will indicate a change of 0.017 calorie every five seconds in the atmosphere surrounding the sample is well within results that should be of practical value.

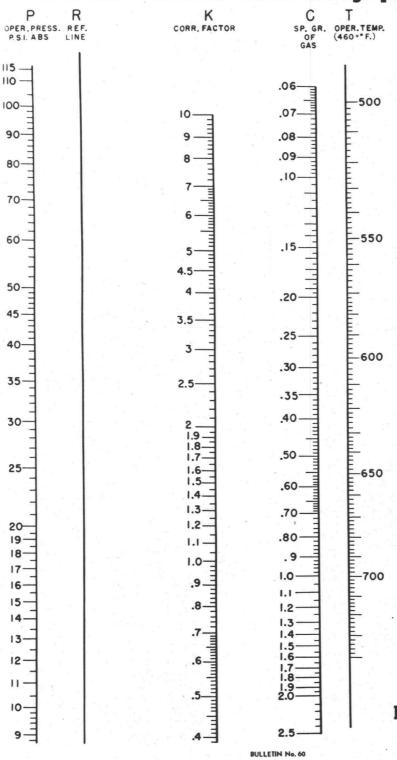
The simplicity of operation may be explained by describing the process of an ignition determination. The sample of paper is chosen and folded, rolled, or cut to a desirable size to slide in the tube, Thermocouples No. 1 and No. 3 are placed before and after the sample respectively. The sample and thermocouple are slid into the tube. Thermocouple No. 4 in effect is a differential couple recording a difference in temperature between 1 and 3. Thermocouple No. 2 is the furnace temperature and has a fixed location within the inner box. (See Figures 3 and 6)



INTERIOR OF FURNACE SHOWING SAMPLE Figure 6

To avoid any damage to the rotameter the blower exhaust must be closed and the fine adjustment valve opened before starting the blower motor. When the blower motor has attained its full speed the two inch gate valve may be opened to obtain the desired supply of air. Finer adjustments in the air-flow rate may be accomplished by use of the by-pass valve. For changes in the specific gravity and temperature of the air supply see correction nomograph Figure 7.

The control circuit and fixed circuits furnish the heat to execute any cam process chosen in the ignition determination. If the load circuits are correctly proportioned the control circuit will maintain the continuous progression, however; if the fixed circuit is adjusted in excess of the requirements of the desired furnace cycle, the control circuit will add no energy until the cycle has progressed to a point where the furnace demands energy from the control circuit. The setting of the fixed circuit (See Figure 2 for wiring diagram) determines the constant amount of energy added to the system. The rate change of temperature from this constant supply of energy must be less than the desired rate in order for the control circuit to be effective, since the latter can only add energy. Figure 8 is a plot of experimentally determined settings which have been found suitable for various rates of heating.



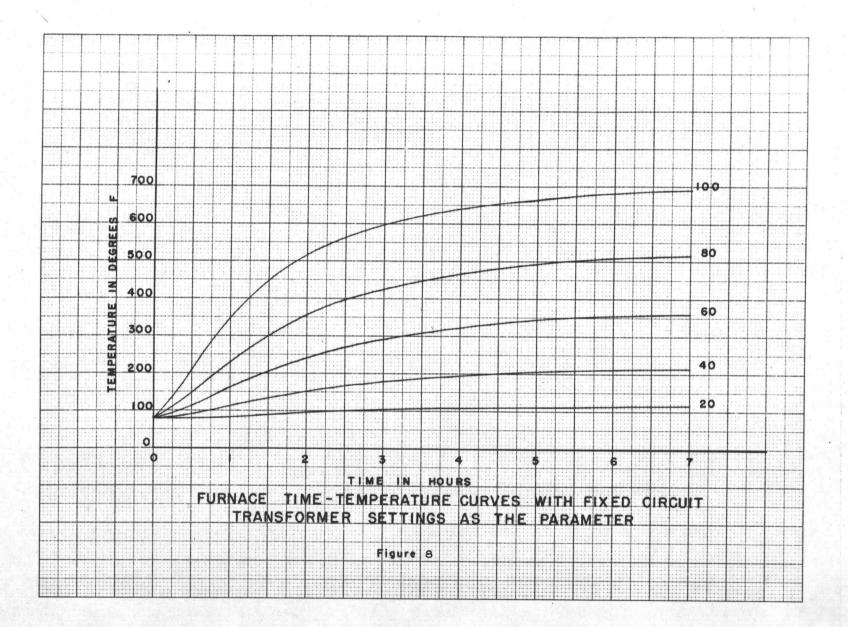
Rotameter Correction Factor Nomograph for Gases

This nomograph is used (1) to determine capacities of rotameters when handling gases at pressures, temperatures and specific gravities different than air, the capacity for air at 70° F. and 14.7#/[" pressure being known; (2) to determine capacity of rotanieters when handling air at 70° F. and 14.7#/[]" if capacities at some different temperature, pressure or specific gravity are, known: (3) to determine capacity of rotameters when handling gases at temperatures, pressures or specific gravities different than air at 70° F. and 14.7#/[" if capacities are available for some other gas at a temperature, pressure or specific gravity different than air at 70° F. and 14.7#/□".

To solve a problem falling in the first of these classifications connect absolute temperature Tto absolute pressure P. Connect point or intersection of reference line with specific gravity G (G is always the specific gravity of the gas at 70° F. and 14.7#/[" referred to air under the same conditions) and read correction factor where line crosses K. Multiply capacity readings taken at 70° F. and 14.7 \sharp/\Box'' by K to obtain readings under the desired operating conditions. For problems as per case two proceed in identical manner but, after the correction factor K is determined, divide the known capacity by K to obtain the capacities for air at 70° F. and 14.7#/[". To solve problems as per case 3, combine the methods for one and two. Use the method of case 2 first so as to obtain air capacity at 70° F. and 14.7#/[]". Then use the method for case 1 to change from air to the other gas.

FISCHER & PORTER CO. HATBORO, PA.

Figure 7



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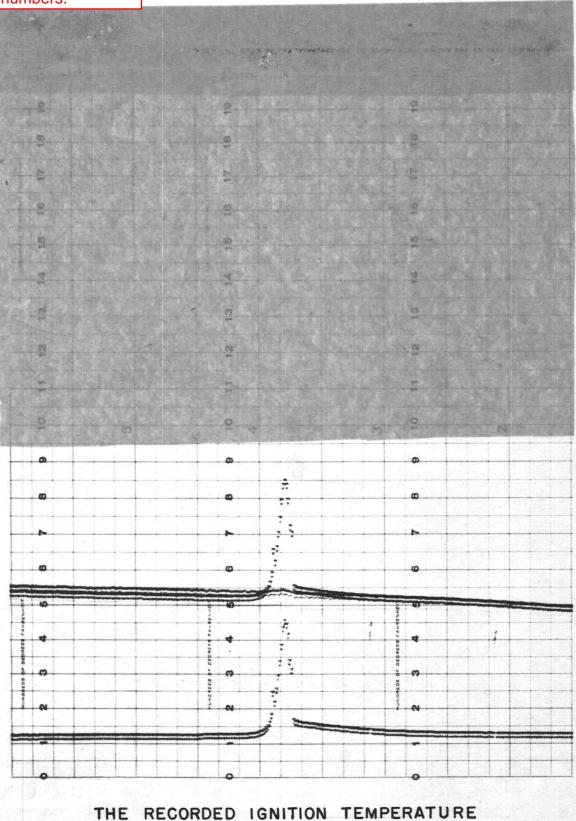
PRELIMINARY TESTS

The initial experiments were made on a paper chosen at random. It was used as a packing paper between aluminum sheets shipped from Alcoa plant of the Aluminum Company of America. The paper appears to have a wax glaze (See Figure 10) and is somewhat on the order of tissue paper. From external appearances it apparently was manufactured from wood pulp. Aside from this information nothing more is known of the manufacturing history.

The first time-temperature curves had an extremely high rate of temperature increase (12 degrees per minute) and the sample fired at 650 F. In the tests following various rates of temperature change were employed and were observed to effect the ignition temperature considerably. For the eight hour, straight line cycle the ignition temperature dropped to 540 F. (See sketch of typical ignition determination, Figure 11).

The primary change in the advanced oxidation rate may be the danger point in the storage of such material since the only noticeable effect is the discoloration of the sample and the emission of a faint blue smoke. Within the autogeneous oxidation range and near the ignition temperature the smoke becomes white with only a bluish tinge. There is a faint explosion, the smoke clears instantly and the remaining material burns.

Original has tissue paper glued over numbers.



OF THE ABOVE TEST SAMPLE

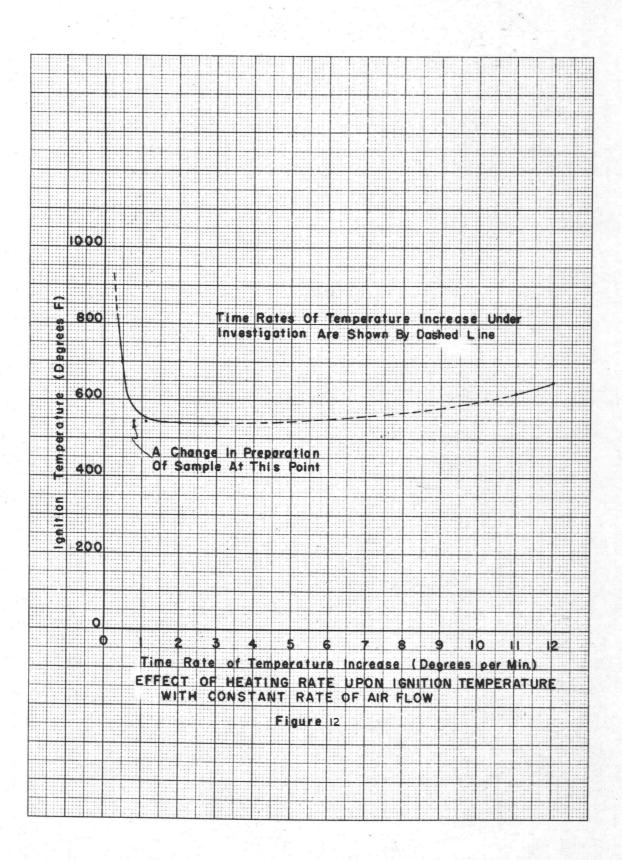
Figure 10

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In the lower rates of temperature increase (one-half degree per minute) smoke is never visible although the odors from the exhaust of the tube, manging from 400 F upward, proved that some chemicals were evaporating. At 500 F the sample began to shrink and at 670 F the sample was examined and found to have shrunk to one-third the original size. Ignition of this sample occurred at 700 F.

The manner in which the sample is prepared has some effect upon the evaporation of the chemicals from the specimen, for instance, if the sample is rolled tightly and closed at each end in order to retain as many of the elements as possible from evaporating into the surrounding atmosphere, the ignition temperature again is 540 F even with a low rate of temperature increase. (See Figure 12 for effect of time rate of temperature increase on ignition).

The explosion at the ignition temperature indicates that the analysis of the gases may prove beneficial in the study of ignition phenomena, that is, in determining the ignition temperature of a gaseous mixture in or near the 540 F it is possible this gas contains combustible elements evaporated from the paper specimen. With a few minor additions of tubing and instruments this equipment may be adapted to analyze the exhaust gases of some representative specimen at different temperature levels in a low rate process.

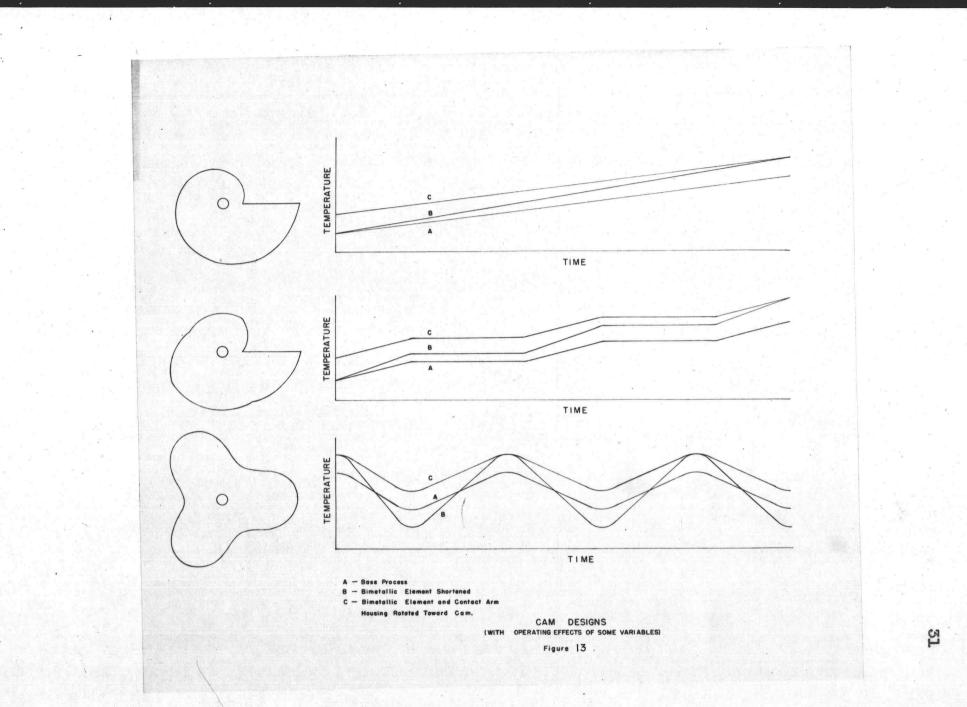


If the assumption that volatile substances evaporate from the paper is correct, the velocity of air around the sample will affect the ignition temperature by reducing the concentration of volatile gases. The concentration could be reduced by using high velocity atmosphere, thereby; increasing the ignition temperature.

The apparatus may be easily adapted to ignition temperature testing in any combination of the following variables:

- Processes and rates of temperature change.
 (See Figure 13)
- 2. Quantity and velocity of atmosphere around specimen.
- 3. Type of atmosphere surrounding specimen.
- 4. Small pressure variations similar to atmospheric changes.

In addition to the above information the manufacturing history, the chemical analysis and analysis of the gases evaporating from the specimen during a heating process will aid in establishing the basic knowledge necessary to solve the many problems.



CONCLUSIONS

In the initial tests of ignition temperature the results have brought out new factors to be considered. No definite conclusions can be drawn from the initial results, however; the results show that ignition involves a time element in some substances if not all.

Many authors state that ignition is only an event in time and does not contain time as one of the controlling variables. In this equipment the timetemperature increase rate may be closely controlled and through this operating characteristic it has been possible to show that a given sample of paper may vary 540 F to 800 F.

From the heating and ignition curve of a substance an increased oxidation rate is evident in the differential thermocouple reading and the thermocouple temperature after the sample. This advancing rate of oxidation, however, does not necessarily fix the ignition temperature, since even though it may not ignite, the sample is liberating energy to the surrounding air.

In the lower time rates of temperature increase the beginning and the end point in the oxidation reaction are less pronounced, evidently the oxidation process also contains time as factor.

Summarizing the discussion of the ignition temperature of a substance the inherent factors in determining the experimental procedure might be stated to be:

- 1. A proper criterion of ignition temperature
- 2. Type of process to be simulated.
- 3. Accuracy of measurements.
- 4. Standardization of equipment components.
- 5. Rate of temperature change and time elapse during process of determination
- 6. Quantity and rate of air flow.
- 7. Composition of atmosphere surrounding specimen.

Equipment has been designed and built and a test procedure outlined believed to be capable of yielding results of practical significance. Time limitations have not permitted an extensive program of testing which might well form the subject matter for an additional thesis; which would be the study of the ignition temperatures of papers, fibers, wood products, and other organic materials subject to storage under conditions where they may become heated from external sources.

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