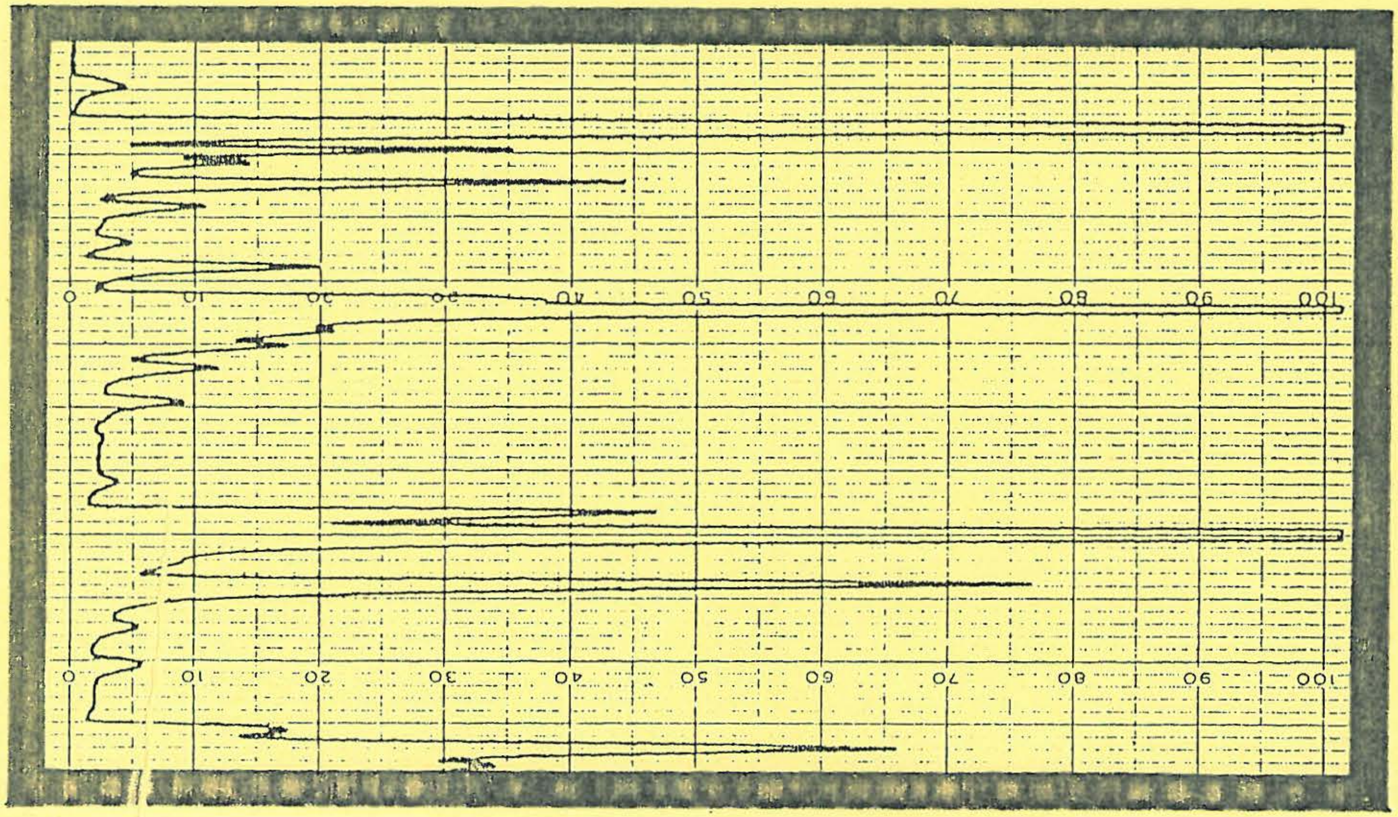
# Arson Analysis Newsletter



MAY 1979
Volume 3
Number 2

# ARSON ANALYSIS NEWSLETTER (AAN)

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Attention: R.N. Thaman

The AAN solicits contributions from forensic scientists, arson investigators, and interested parties which have some unique or routine analysis which helps in the identification of flammable liquid or explosive residues.

May 1979

# ARSON ANALYSIS NEWSLETTER
Volume 3, Number 2

# THE IMPLICATIONS OF REFINING OPERATIONS TO THE CHARACTERIZATION AND ANALYSIS OF ARSON ACCELERANTS

## Part I. Physical Separation

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## INTRODUCTION

Petroleum materials utilized as arson accelerants are composed of only two elements, carbon and hydrogen. What distinguishes one petroleum material from another is how those two elements are combined, and additionally, how the various materials are separated or converted from crude oil. It is the position of the authors that the interpretation of arson accelerant evidence, i.e., the characterization or identification of accelerant residues, requires a comprehensive understanding of refining operations, a knowledge of the chemistry of crude oil and the products that can be manufactured from it, and the implications of refining operations to the analytical data obtained from the gas chromatograph.

There are two major processes for the manufacturing of petroleum products from crude oil. The first is a physical separation process in which the hydrocarbons comprising the crude oil are categorized according to their naturally occurring formation. An example of this type of process would be the separation of straight chain hydrocarbons from cyclic hydrocarbons. The second major process is a chemical conversion to rearrange or break down complex hydrocarbons to a more simple form or into a form which provides a more marketable product. Part I of this work will describe physical separation processes. Part II will discuss chemical conversions, treating processes, and subsidiary processes. Part II will focus specifically on the forensic implications of these processes.

## CRUDE OIL MANUFACTURING PROCESSES

A large number of products are processed from crude oil, the type and proportion of the products depending principally on their marketability. The essential function of a refinery operation is to produce these products, such as gasoline, kerosines, oil lubricants, greases, fuel oils, gas oils, wax and asphaltic bitumen. There are two major processes by which these petroleum products are formed, and several ancillary processes.

The first major process is a physical separation process. In this operation the hydrocarbon molecules in the crude oil are separated in a manner in which no major change in their molecular structure occurs and no new compounds are formed. There are five processes by which crude

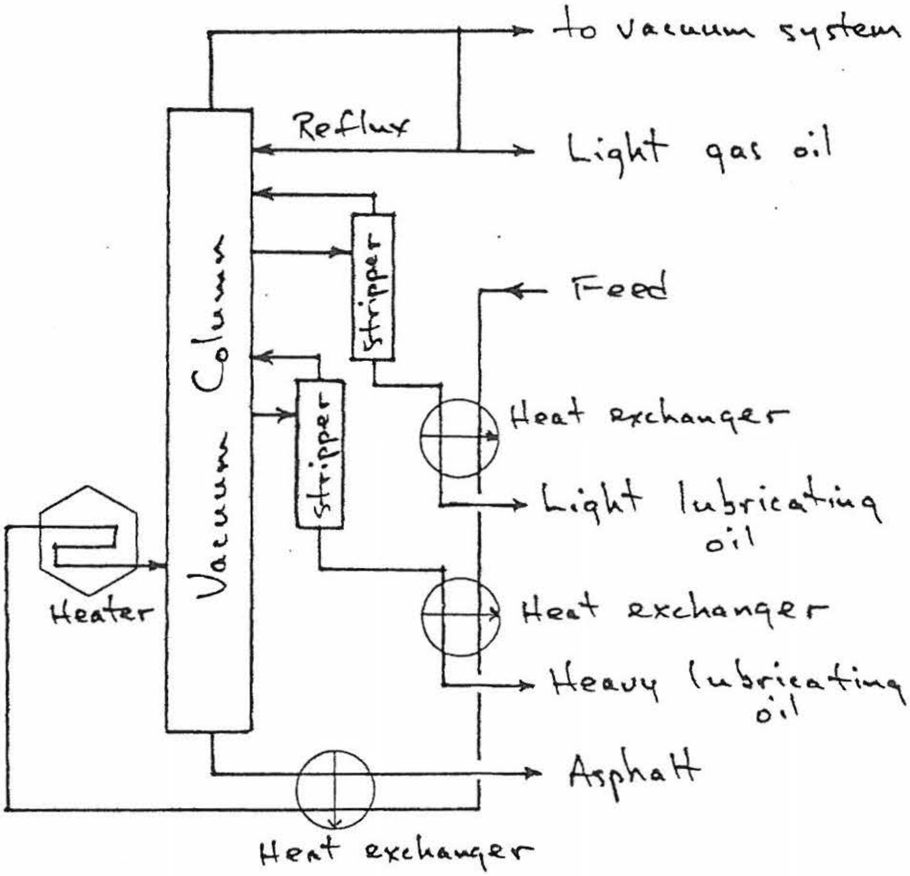

Figure 1. Vacuum distillation process

oil is physically separated: distillation, crystallization, solvent extraction, adsorption, and absorption. Distillation is a process in which the compounds in the crude oil are separated according to their molecular size depending on their boiling point. Crystallization separates according to size and type depending on the difference in melting point and solubility. The solvent extraction process utilizes the differences in adhesion to a porous material in gas-liquid or liquid-solid systems. Absorption separates according to size and type but utilizes differences in solubility in a liquid. These processes will be discussed in greater detail below.

The second major process that is involved in refinery operations is a chemical conversion process. This conversion process results in a change in the size of a hydrocarbon molecule, a change in the structure of the molecule, or a change in both size and structure. There are three major chemical conversion processes. The first are breakdown processes by thermal and catalytic reactions, often referred to as "cracking." The second are buildup processes by polymerization and alkylation, and the third are type-change processes such as dehydrogenation, isomerization, cyclization, and reforming. These processes may occur separately or simultaneously in the manufacturing process.

Following separation and conversion, petroleum products are generally run through a treatment process. In this process the products are treated by physical and chemical means to meet a specific marketable specification, such as color, odor, stability, etc. The treating process does not necessarily occur at the terminal end of the physical and chemical conversion, and in some instances intermediate products are treated to remove impurities which may interfere with subsequent processing steps.

From the three processes mentioned, physical separation, chemical conversion and the treating processes, there are a number of products obtained from crude oil. Some of these products are suitable for market once they have undergone these three processes. Usually, however, the products will undergo still another subsidiary process in which the products are blended with other compounds to form a higher quality or more suitable product. For example, gasolines with a desired ignition quality are produced by blending various gasoline fractions with lead compounds and other chemicals; lubricants for various automotive, marine and industrial applications are prepared by blending to the required viscosity characteristics, with or without additives; and bitumen is blended with kerosine to form "cutbacks."

## DISTILLATION

The initial step in the manufacturing of petroleum products is the separation of the various fractions within crude oil by their molecular size and their difference in boiling points. Distillation is one of the most important steps in the refinery process, not only for its general separation abilities, but also for refining the products obtained for marketing specifications.

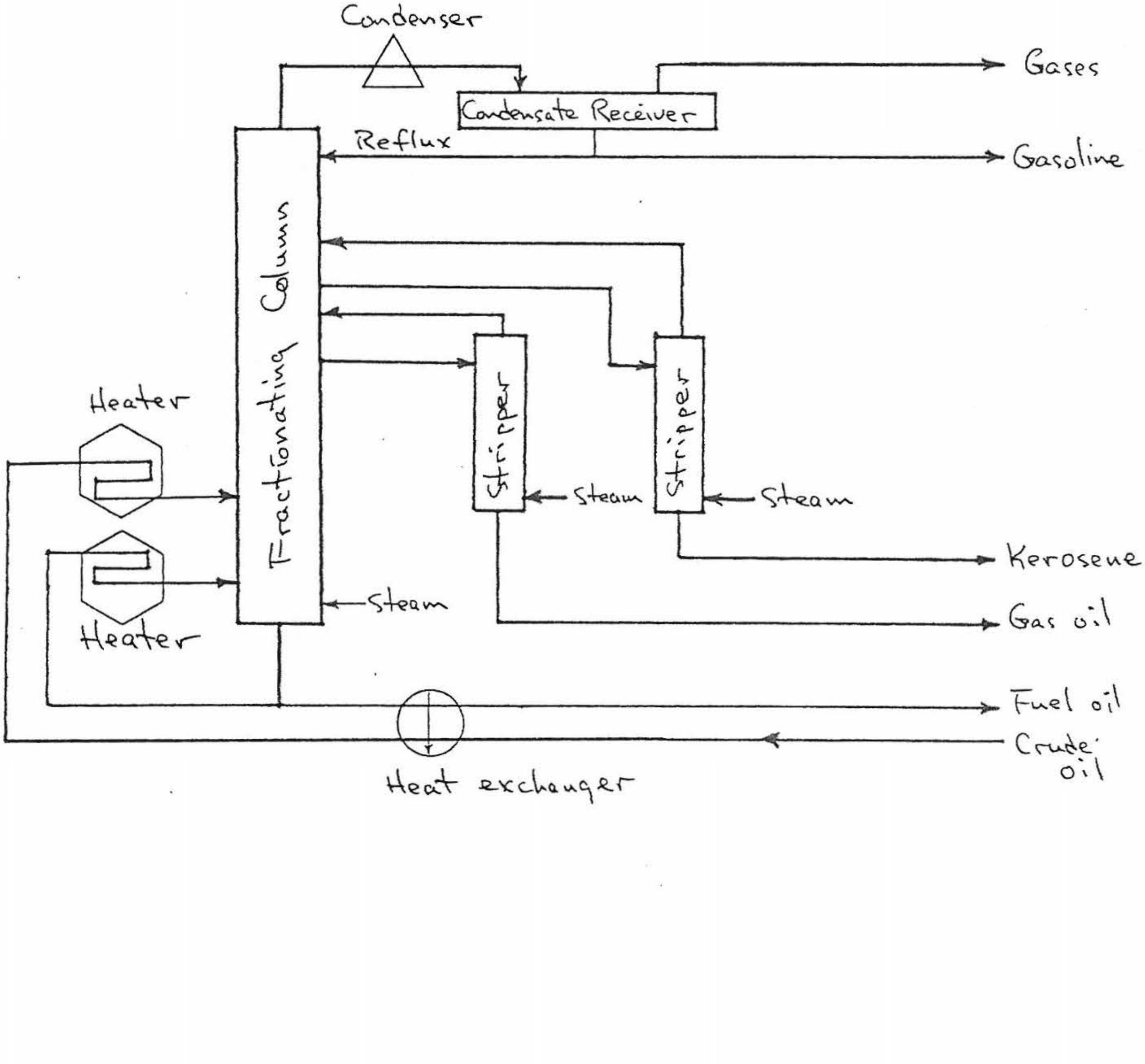

Figure 2. Simple distillation process

The major feature of this process of separation is the ability of the various compounents in crude oil to vaporize, i.e, their volatility. The volatility of a product depends on its molecular size; the larger the molecule, the lower the volatility. For example, at a normal temperature and pressure, gasoline vaporizes very readily from a liquid state. Kerosine and other fuel oils, on the other hand, require a higher temperature to vaporize. Still other products, such as paraffin wax, are in a solid state under normal conditions and require a relatively high temperature to liquify and a still higher temperature to vaporize.

The volatility of a substance is directly related to its boiling point. A liquid product which has a low boiling point is more volatile, whereas one with a higher boiling point is less volatile. The pressure along the surface of the liquid also contributes to the characteristic boiling point. When a liquid is heated, the energy of its molecules increases and more molecules are able to pass through the surface into the vapor state. The pressure above the surface of a liquid, even in the normal atmospheric state, tends to restrict the formation of the vapor. However, the temperature of the liquid determines the number of molecules leaving the surface of the liquid and in turn determines the vapor pressure of the liquid at that temperature.

Vapor formation occurs when the vapor pressure above the surface of the liquid equals or is slightly higher than the atmospheric pressure. The temperature of a pure liquid has a specific and characteristic boiling point and remains constant until all of the liquid is evaporated. The boiling point varies with the pressure; the higher the pressure, the higher the boiling point. Thus the temperature can be controlled either by increasing the pressure or reducing it as in a vacuum.

The heat that is transferred to the molecules in the process of boiling is also retained in the vapor. When this heat is removed, the vapor condenses back to the liquid state. The heat is given off as the heat of condensation when the vapor returns to its liquid form.

The distillation process of crude oils follows the general process as in a fractional distillation apparatus. In a single distillation step a mixture is boiled and a component with a characteristic boiling point is condensed into another container. Boiling starts at a temperature which lies in the range of the boiling points of all the components of the mixture. The initial boiling point is defined as the temperature at which the first distillate distills over. The temperature increases during the distillation as more and more volatile components distill over. The mixture thus becomes richer in the higher boiling point components until the final component distills over. Steam is used to assist in the separation of the volatile components. When the steam is blown onto the hot oil it contributes its own partial pressure thus reducing the partial pressure of the oil so that it boils at a lower temperature. This method of steam distillation is used to prevent the chemical decomposition of higher boiling point components. Figure 1 illustrates a vacuum, steam distillation process.

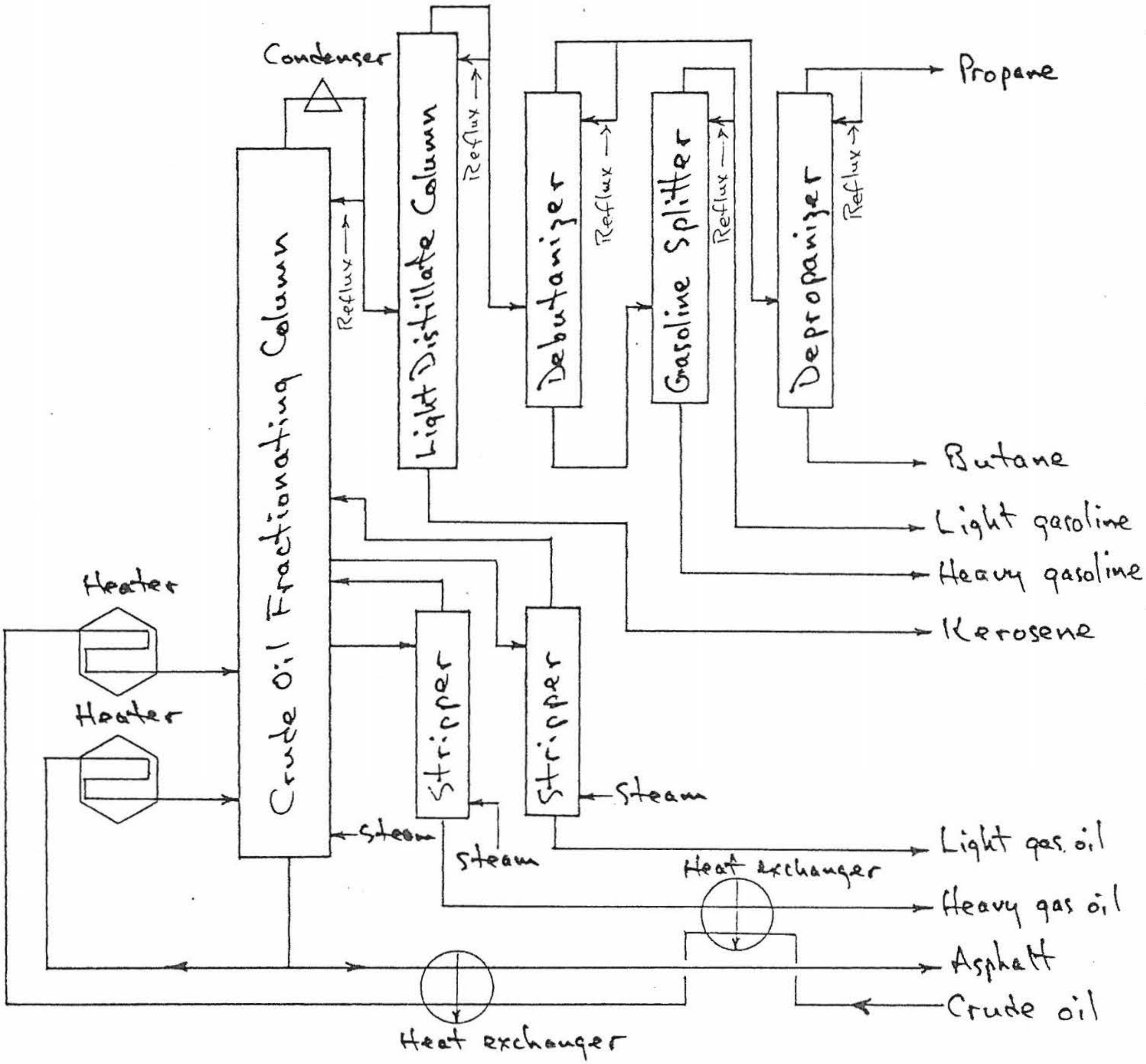

Figure 3. General scheme of crude oil distillation

However, lighter products with very volatile characteristics such as propane and butane cannot condense at an atmospheric pressure and normal temperatures because of their low boiling points (propane -42°C and butane -0.5°C). Therefore, to separate them from a crude oil mixture, distillation is carried out under increased pressure which increases their boiling points and thus their temperature of condensation.

Figure 2 illustrates the principle of a simple distilling unit. The crude oil first passes through a heat exchanger where the outgoing fuel oil is cooled by the incoming crude. The preheated crude oil then enters a furnace where the temperature is raised to that needed to vaporize all the volatile components. The hot vapor liquid mixture is then injected as a mist into the fractionating column with the vapor passing upward and the liquid downward. The process of condensation and re-evaporation takes place in the "rectifying" section. The hot oils then pass through a stripping section, which is another distillation column where the light products are removed by steam.

Products are removed along various points of the column as shown in figure 2. In this illustration two side streams are for kerosine and gas oil. The distillate products are removed into a side stripper which further purifies the product. Steam is injected into the side stripper so that the more volatile components are evaporated from the desired product and returned to the main column. The remaining product at the bottom of the column is cooled and stored.

Efficient fractionation is essential in the distillation process. When overlapping occurs, such as if a gasoline fraction is contaminated or mixed with some higher boiling point fraction such as kerosine, the final boiling point of the gasoline would be raised and its performance affected as an engine fuel. Consequently, the oil fractions from the main column are directed through another distillation column to ensure complete separation.

The products separated in the crude oil distilling unit of a modern refinery may be classified in order of decreasing volatility into (1) gases, (2) light distillates, (3) middle distillates and (4) residue. Figure 3 illustrates a general scheme of crude oil distillation and these basic distillates.

The gases and lighter distillate products are comprised mostly of methane, ethane, propane and butane. Propane and butane may be liquified at this step of the process and used as petroleum gas.

The light distillates comprise fractions of the light gasoline, heavy gasoline and kerosine products. The heavier, higher boiling point fractions, such as kerosine, are used for lighting, heating and jet fuel oils. The heavy gasoline fractions are often referred to as "naphthas" and are used as additives for other products.

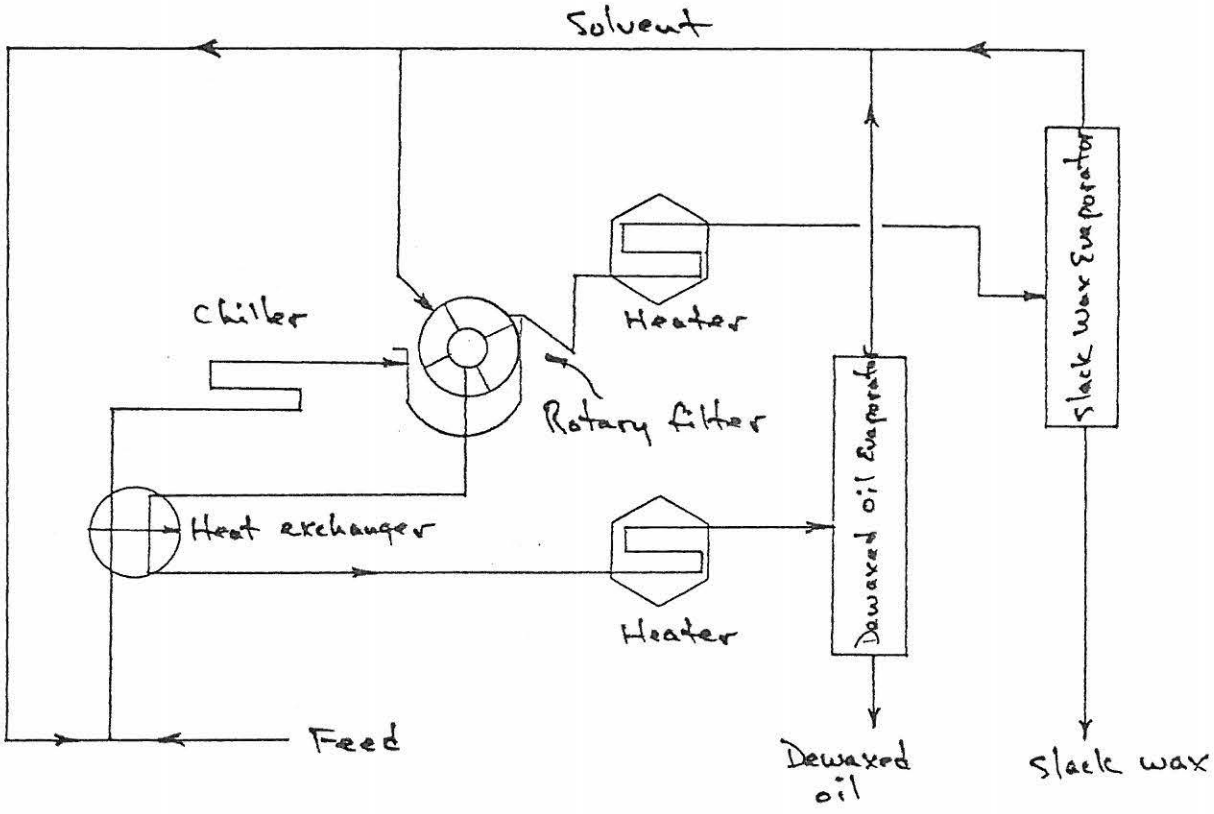

Figure 4. Solvent dewaxing process

The middle distillates are used as light and heavy gas oils. These are diesel fuels and oils used in the blending process with residual products to yield furnace fuel as the final product. The middle distillate products are also used as feedstocks in the cracking process.

The last components consist of residues which are not susceptible to distillation. They are treated in other manufacturing processes to form products such as lubricating oils, wax and bitumen.

## CRYSTALLIZATION

The next step in the physical separation process of crude oil is crystallization. Paraffin wax occurs in those crude oils having a high paraffin content. The waxes must be removed in order to avoid poor performance and fluidity of oils at low temperatures. Paraffin wax consists essentially of paraffinic hydrocarbons of high molecular weight. They readily separate from an oil fraction by crystallizing out of solution when the oil is cooled.

Solvent dewaxing is the major process for the elimination of paraffin waxes from crude oils. This method utilizes a variety of solvents in which a filtrate of low viscosity is obtained at lowered temperatures. The most widely used solvents are toluene and methyl ethyl ketone (MEK). The toluene dissolves the oil and maintains the fluidity at low temperatures whereas the MEK acts as a precipitating agent. Other occasionally used solvents are propane and chlorinated hydrocarbons such as dichloromethane.

There are three stages which the dewaxing process follows (see Figure 4). First the oil is mixed with the solvents and chilled to a low temperature, usually -20°C. The mixture then flows into a rotary filter where the paraffin wax is removed. Finally, the dewaxed oil and wax are distilled by separate columns and either stored or receive further processing treatments.

The dewaxed oil and solvent mixture are separated by distillation. The solvent is recycled and the dewaxed oil is stored. The wax cake is also separated from any remaining solvents by distillation and the slack wax stored or treated in the sweating process.

The sweating process is a treatment whereby the various components of the slack wax are separated. The wax in its solid state is laid down on a tray with a raised wire gauze bottom. As the temperature is increased the lower boiling point oils or "sweat oils" drip through the gauze into a collection tank. After these oils are obtained the remaining wax, composed of the higher boiling point oils, is melted by steam. This sweating process is used to obtain purified wax products with specific boiling point characteristics.

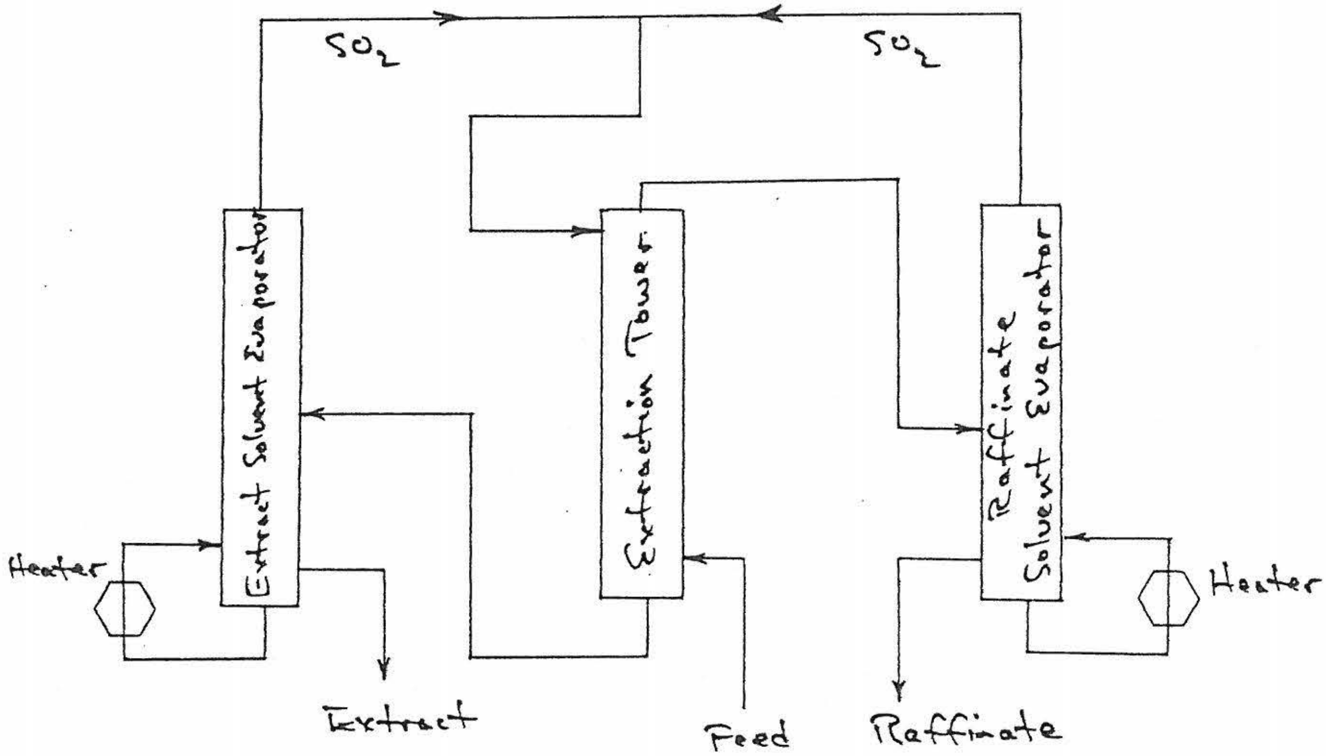

Figure 5. Edeleanu extraction process

## SOLVENT EXTRACTION

Solvent extraction is a process developed to remove aromatic compounds from the various oil fractions after distillation and crystallization. Distillation does not differentiate between compounds with the same boiling point characteristics. Therefore, one oil fraction may contain paraffins, aromatics and naphthenes. Aromatic compounds must be removed from oil fractions such as kerosine, lubricating oils and fuel oils because they cause undesirable effects in these products.

The general process involves a solvent in which the aromatic compound preferentially dissolves. When the mixture is allowed to settle, two phases will form. One phase contains the aromatics and solvent, and the other contains the oil free from aromatics. Usually the lower extract phase will contain the aromatics and the upper or raffinate phase contains the less soluble components. Both the compounds in the extract and raffinate are unchanged chemically, as the process involves only a physical separation. The solvent remaining in either phase is removed by distillation and recycled into the system.

Figure 5 illustrates the Edeleanu process which is the basic solvent extraction process for kerosine. The solvent used in this process is sulfur dioxide. Instead of mixing and allowing the mixture to settle into their two phases, the oil is fed into a "packed" column. The "packed" column is filled with ceramic cylinders to break up the flow of liquid. The solvent is added at the top and the oil feed at the bottom. As the oil vapor rises, the aromatics are caught in the solvent slowly flowing down. The raffinate is removed from the top as a vapor and the extract from the bottom of the column. The separation of the raffinate and extract are controlled by the temperature of the column.

The solvent extraction used in lubricating oils utilizes the Furfural extraction process. The solvents used in this process must have a higher boiling point than the feed oil; otherwise both will volatilize. The solvents furfural and phenol, which have boiling points of 162°C and 181°C, respectively, are used in this process.

The Furfural process is very similar to the Edeleanu process except that a rotating disc device is used instead of a "packed" column (see figure 6). The column has a central shaft which connects the many compartments within the column. A disc attached to the shaft rotates in each compartment mixing the solvent and oil feed. The mixing is controlled by the rotation of the shaft.

Figure 7 illustrates the processes in which pure aromatics such as benzene, toluene and xylenes are recovered. An oil fraction rich in these aromatics is introduced into a distillation column. A solvent with a very high boiling point compared to the volatility of the aromatics is introduced at the top of the column. The solvents used are usually phenol or acetonitrile. The aromatic compounds are highly soluble in the solvent

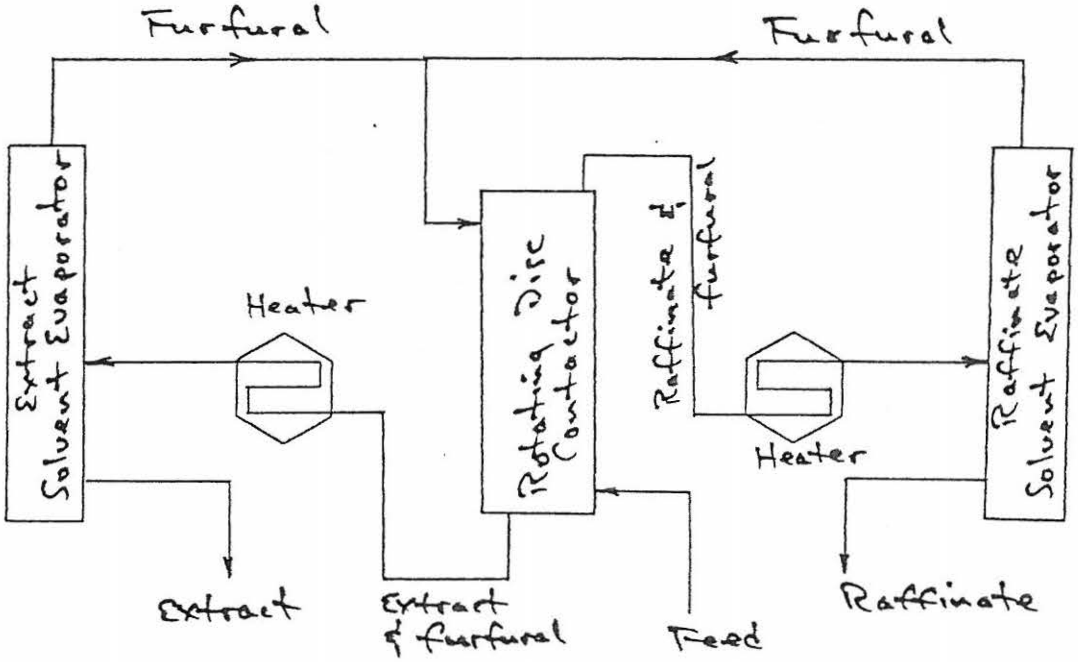

Figure 6. Furfural extraction process

and are extracted from the bottom of the column. The solvent is separated from the pure aromatic compound by simple distillation and recycled into the system.

## ADSORPTION

Adsorption is the process in which certain hydrocarbons in gas have a characteristic feature of adhering to certain highly porous materials. In the refinery process a stream of gas is passed through the porous adsorbent material; heavier hydrocarbons condense on the surface of the material. Not all gases can be treated in this manner. For example, if hydrogen sulfide is present in high quantities in the gas it will have a greater affinity for the adsorbent and will saturate the material before the other hydrocarbons can adhere to it.

The materials used as an adsorbent are activated charcoal, silica gel, and alumina. The adsorbent is usually in a number of large vessels in which the gas mixture is passed. The vessels are changed as the material becomes saturated with the adsorbent hydrocarbons. The adsorbed component is removed by steam and recondensed into a collector. The adsorbent is treated with hot gas and cooled to be recycled.

## ABSORPTION

Absorption is similar to adsorption except that it is a solution process to purify light gases such as methane, ethane, butane and propane. These low boiling point, highly volatile hydrocarbons may liquify under varying conditions. For example, under normal temperatures and increased pressure methane and ehtane will become liquids. It is in the liquid state that these compounds can be purified.

Figure 8 illustrates one of two absorption processes, the rectifying absorption process. It is a more efficient process in terms of better control and quality of product recovered. In this process the absorption and stripping are combined in the same column.

This process utilizes two feeding lines, one a liquid and the other a vapor. The absorption oil is introduced from the top of the column. As the oil flows down the column it first encounters the rising vapor and collects the less volatile components. The lighter hydrocarbons such as methane and ethane pass out from the top of the column as dry gas. Meanwhile, the absorption oil encounters the liquid feed and an additional absorbing effect occurs. The heat released by the condensation of the vapor is removed by a cooler, which maintains a constant temperature in this section.

The absorption oil now contains more absorbing components than desired. The excess components are removed from the oil as it flows past the vapor inlet. This part of the rectifying absorption column is called the stripper section. Here the excess is removed by a heater or reboiler.

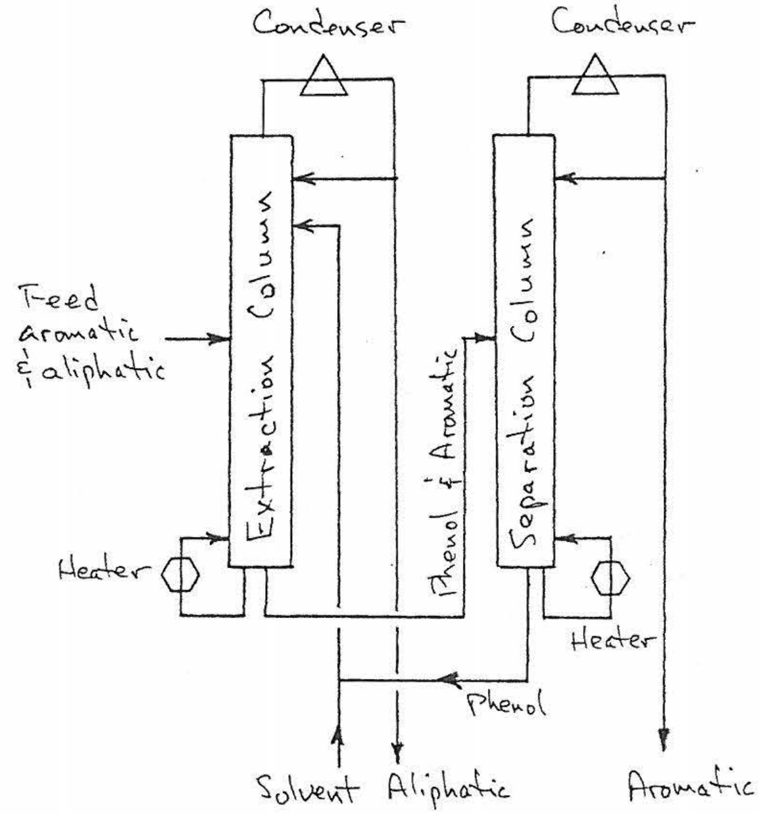

Figure 7. Extractive distillation process

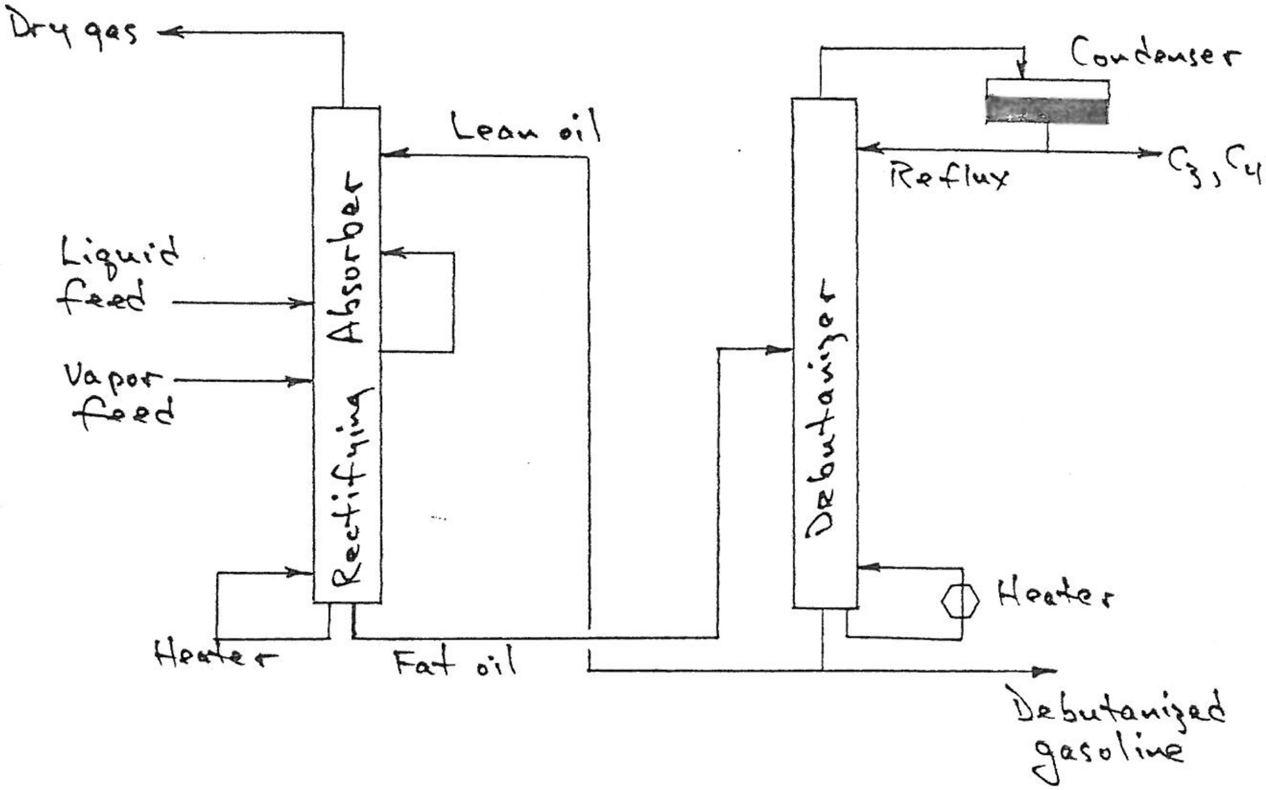

Figure 8. Rectifying absorption process

The absorption oil containing the $C_3$ and $C_4$ hydrocarbons are removed from the bottom of the column and separated in a debutanizer. The $C_3$ and $C_4$ hydrocarbons are removed under pressure. The residue from the debutanizer is gasoline and can be recycled back into the system as the absorption oil.

[Part II will discuss chemical conversion (thermal and catalytic cracking, reforming, polymerization and alkylation), treating processes and subsidiary processes.]

PART TWO WILL APPEAR IN THE

NEXT ISSUE OF THE AAN

# MECHANICAL MODULII FOR TEMPERATURE DETERMINATION

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# Mechanical Modulii for Temperature Determination

## Abstract

This paper presents an investigation of the temperature effects on springs. Application is directly related to fire investigations as a tool for origin determination. Under high temperature conditions, springs undergo load losses which in most instances are due to a decrease in the torsional elastic limit of the material. The functional relationship between temperature and slow plastic deformation resulting in a permanent set is investigated. The amount of heat intensity, working stress, and time duration were all examined during controlled laboratory tests. With the preliminary results, yet another tool for aid in fire investigation has been postulated.

## Introduction

For several years there has been a continuing research program at the Ohio State University and Systems Engineering Associates aimed at illuminating the use of Mechanical Modulii for Temperature Determination.$^{1-4}$ The objective of this paper is to present an experimental investigation of this technique as yet another tool for use in fire causation.

## Preliminaries

The occurrence of springs in mechanical systems is very common and they often remain after a fire. Thus, it was postulated that springs might record, for example, the attained temperature of a fire and could be used as a tool for fire investigators. When a spring is heated to a high temperature and then cooled, certain physical properties of the spring change. Specifically, the first objective was to determine if the spring was in tension or in a relaxed state when heated during the fire. Secondly, since the modulus of rigidity has been shown to change for various spring materials (5), it was speculated that a relationship may exist between the amount of change in the spring constant (6) and the temperature to which the spring was heated. Finally, it would be desirable to relate the functional expression(s) such as

$$f = F[T(^{o}C), t(hr), k(N/m), \Delta L(cm)]$$

Knowledge of such relationships could be very useful in the total investigation of a fire.

A literature search of engineering and fire related research provided no information on a temperature-spring constant relationship or deflection temperature dependence. One text even mentioned that there was very little information available on temperature effects of springs (7). Indeed, there has not been much, if any, testing of springs which have been brought to temperatures greater than the eutectoid temperature and then cooled to see if the spring constant has changed. Also, no theoretical model was found to exist in the literature.

The objectives of this research program were based on two different theories. The first part of the experiment was designed to find out if the heating of springs above the eutectoid temperature and then cooling would change the spring constant. The testing program consisted of over 80 springs heated from 1350°F (733°C) to 2000°F (1094°C) by 50°F (27.8°C) increments. The second phase was to determine if a relationship existed between a spring heated in tension versus one heated in a relaxed state.

## Part I: Temperature vs. Spring Constant

### The Experiment

The over 80 springs tested in this study had the following geometry: Wire size 12 gauge, free length of 6.35 cm (2.5 in), O.D. of 2.54 cm (1 in), with ten coils and square ground ends, see Figure 1. This size was selected for two reasons. First, these springs fit an existing test device (see Figure 2) and second, preliminary testing illustrated that softer springs gave better deflections than stiffer springs with hard to measure deflections. Out of the 84 springs tested, 35 of them were randomly selected for calibration to give a good average value for the spring constant before heating. Out of the 35 springs calibrated before heating at least 2 were placed in each of the groups of springs tested at each temperature. This was to make sure that a good distribution of random samples throughout the entire population occurred. These 35 springs randomly selected were calibrated from zero to twenty pound load (88.9N) in 2 pound (8.88N) increments. The upper plate and rod assembly placed an initial load of about 2 pounds (8.88N) on the springs. With this load present the scale was zeroed and the weights then applied in 2 pound increments up to 20 pounds. At each increase of 2 pounds the scale reading was recorded.

The testing device used was made of two circular flat plates about 6 inches in diameter between which the springs to be tested were placed. The upper and lower plates were steel. The upper plate has a half inch diameter (2.27 cm) steel rod welded at its center perpendicular to the face of the plate. The lower plate has a one half inch (1.27 cm) diameter nominal rod at its center aligned with the 6 inch (15.24 cm) long steel bearing welded to the underside of the plate, again about a half inch (1.27 cm) outside diameter. The rod attached to the top plate passes through the lower plate bearing assembly. The whole assembly is supported by a circular flanged unattached base made of aluminum. A scale was clamped to the lower plate and aligned so that the spring deflections were read by the displacement of the upper plate as each weight was applied to the top of the upper plate. A six inch long one fiftieth inch scale was used to measure the deflections. The weights used were one, two, and five pound standard weights calibrated prior to the experiment.

The thirteen groups of six springs were then heated in an oven for one hour at each desired temperature. They were removed from the oven and allowed to air cool to room temperature.

## Experimental Results

Several springs were tested as preliminary testing in order to establish what type of spring would best suit this experiment. All of the preliminary springs were calibrated before heating and then recalibrated after being heated to the desired level for the desired time. Some of these springs were water quenched but upon recalibration failed when loaded with the calibrated weights. From this point on the rest of the springs were cooled in air.

A time test was performed while holding the temperature constant at 1500$^{0}$F (816$^{0}$C). Only one spring was tested at each different time length but the results from each of these springs showed no appreciable change in spring constant for the different time lengths (see Table 1).

Plotting the spring constant versus temperature shows that as temperature increases, in general the spring constant decreases for the spring size tested in this report, see graph 1 and graph 2. In Table 2, the springs are listed with their respective spring constants, temperatures, time and percentage change in the spring constant.

## Error Discussion

Of the major sources of error, deformation of the springs due to excessive temperature was probably one of the largest causes. As the temperature was increased, the springs became more deformed, bent and had large amounts of variation from one spring to the next as to how it behaved. Indeed, some would fail earlier than others, and some would give data that was out of line with the rest. As the temperature increased, so did the problems. Error was also due to the irregularities in the oven temperature and hot spots in the oven. Springs near the door were not quite as hot as the ones in the back and the oven temperature fluctuated about its temperature setting. However, the overall error obtained using standard statistical analysis, indicates that all experimental points fall within error limits thereby showing the data as reliable.

## Experimental Conclusions

The temperature spring constant relationship does exist for the spring size tested herewith. It probably can be generalized to most springs and thus be a very useful concept. As the temperature to which a spring is raised increases, then air cooled, the spring constant decreases from its original value. This relationship appears to have no time dependence as long as the spring being heated remains unloaded, otherwise relaxation could occur. If enough different size springs are tested it seems probable that an expression can be developed from experimental data. This is further verified by the linear relationship herewith presented. This mathematical expression is empirically derived and only valid for the springs tested.

$$K = -0.03T + 70.6 \quad lb_f/in$$

where $K$ = spring constant ($lb_f/in$)

$T$ = Temperature ($^oF$)

## Practical Applications

The significance of this fundamental research is directly applicable to fire investigators. As in most suspected arson car fires, see Figure 3, the interior of the vehicle is totally consumed. Thus, it would be the seat springs (Figures 4 & 5) that would be removed and tested in order to establish the temperature attained by the fire. Knowledge of this temperature would aid the investigator in determining probable causes of the fire, such as accelerants or smoldering cigarettes. And, since accelerants would have a higher attained heat value than a smoldering fire, a scientific verification of probable cause can be made.

Presently at Systems Engineering Associates, Arson Investigators are collecting spring samples from incendiary fires. In some preliminary cases (a positive accelerant was identified by the Chemistry Lab) a definite correlation has shown to exist with the spring constant of the tested springs. However, before any usable results can be verified, a vast standardization of spring constant must be recorded as a basis for factual judgment.

Furthermore, the greater the percentage change in the spring constant, the higher the attained temperature in the area of the sample spring (see table 2). Thus, although lacking a complete heated spring data curve for a particular size, a few data points can illustrate a low, medium, or elevated attained temperature of a given sample spring.

## Part II: Mechanical Properties of Springs After Heating Under Preload

### The Experiment

Over one hundred (100) springs were tested in this portion of the study. The springs were made of spring steel wire, wire diameter 0.476 cm (3/16 in.), O.D. 5.08 cm (2 in.), free length 30.48 cm (12 in.) and having 50 coils with looped ends. This size spring was choosen for its similarity to garage door and attic trap door springs used in the commercial building industry.

After each spring was identified, it was placed in a special bracket as shown in Figure 6. This bracket held four springs each at four different temperatures ranging from $800^{o}$F ($427^{o}$C) to $1800^{o}$F ($982^{o}$C). Also, the bracket placed each spring in a different preload condition (0, 2, 4 and 6 inches of stretch), and allowed for "free sag" during heating. After heating in the oven, the springs were allowed to cool. They were then measured for their free length after heat condition as well as their spring rate as determined from the Instron Testing machines.

## Background

It is speculated that heating a spring with preload will change the free length. This change will also cause incremental changes in other dimensions of the spring, especially mean coil radius and outside wire diameter again affecting the spring rate. It has been shown that the spring rate can be related to wire diameter and mean coil radius as follows:

$$k = \frac{Gd^4}{64R^3N}$$

where $k \triangleq$ spring rate
$G \triangleq$ modulus of rigidity
$d \triangleq$ wire diameter
$R \triangleq$ mean coil radius
$N \triangleq$ number of active coils

Assuming that G and N do not change, k can be written as:

$$k = C\,\frac{d^4}{R^3}$$

where $C = \text{constant} = \frac{G}{64N}$

If both R and d are changed by an incremental value $\Delta$, the effect on spring rate will be:

$$k' = C\,\frac{(d+\Delta d)^4}{(R+\Delta R)^3}$$

Let $R' = R+\Delta R$ and $d' = d+\Delta d$.

If d is decreased by one percent, and R is decreased by one percent, then k'/k will be:

$$\frac{k'}{k} = \frac{C \dfrac{.99d^4}{.99R^3}}{C \dfrac{d^4}{R^3}} = 0.99$$

Thus a small change in d and R will only make a small change in k.

The assumption that G remains constant can be considered valid. It has been shown that G varies with temperature, but the temperature is constant during experimental measuring of the spring rate of the spring.

By stretching the spring when it is heated, some degree of relaxation is introduced. This relaxation causes a change in the free length of the spring, which changes R and d of the spring.

The amount of relaxation will be a function of the temperature to which it is heated and preload on the spring rate should be a function of temperature and preload: which can be expressed as

$$k = f(d,R)$$

$$d,R = f(\text{RELAXATION})$$

$$\text{RELAXATION} = f(T,\text{PRELOAD})$$

$$k = f(T,\text{PRELOAD})$$

## Experimental Results

The data obtained in testing the cooled springs is shown in Tables 3, 4, and 5.

As noted, the spring holding bracket was constructed so that the springs would be allowed to "droop" due to their own weight. At higher temperatures, however, the springs at the top of the bracket were held up by the ones on the bottom and were not allowed to deflect as much as possible. Unfortunately, this led to irregularities in the test groups. Also due to heating, a scale was built up on the surface of the springs and when these springs were tested the scale caused the force-deflection curves to become irregular. This made the calculation of the spring constant difficult. Springs at higher temperatures were stretched out so much that they would not fit in the test apparatus. To test these springs, the middle of the spring was put in the test apparatus and the spring constant for that section was adjusted for the whole spring. This was done assuming that the relationship

$$k = \frac{Gd^4}{64R^3N}$$

holds for this case and G, d, and r were assumed to remain constant.

## Error Analysis

There were many sources of error in this portion of the test program. Specifically, the springs were not uniform and the free lengths varied from 12 to 12.5 inches. Fortunately, this affected the final results very little, as it changed the spring rates a small amount, because all the springs had the same number of active coils. The scale readabilities of the instruments introduced some error. The free lengths were measured to the nearest 0.1 inch and the temperature on the oven could only be set to ±5 °F of the desired temperature.

The major error introduced, however, was due to the curvature in the springs after heating, and having to measure only a portion of the spring to obtain the spring rate. The curved springs did not have uniform stresses in them, and by making special end preparations for pulling the elongated springs, error in spring rate was introduced. Finally, the slope of the force vs. deflection curve recorded by the Instron was approximated, as only the most linear portion of the resulting graph was used.

## Experimental Conclusions

As indicated by the slopes of the normalized spring rate vs. deflection curves (see Figures 7 - 10) there is a trend that the spring rate decreases with increasing preload. This is verified at all temperatures except 1600°F and 1800°F. At these two temperatures, there was a very heavy oxide scale on the springs. Indeed, the more a spring was stretched, the more it was exposed and then the more scale there was on it. Because the scale was very hard and brittle, its effect was to stiffen the springs. Thus the greater the deflection, the more scale, the stiffer the spring, and the higher the spring rate. This is verified by the positive slopes of the normalized spring rate vs. deflection lines.

Although a general trend has been recognized, it would not be accurate to try to develop an empirical equation to define this trend, because the data is so spread out.

The slopes of the normalized spring rate vs. deflection lines were obtained using a least squares linear regression. This method also produced a correlation factor, which, if close to one, would indicate high correlation. The data produced correlations in the neighborhood of 0.240 to 0.5, which indicates a wide spread in data and lowers the chances for the data to be accurately predicted by a function.

The student t-test was also performed on the data, in pairs of adjacent temperatures, i.e. the data from 800$^{0}$F was compared to 1000$^{0}$F etc. This test was used to check if the difference in the trends of the data is due to normal error from a Gaussian distribution. The test gives a number P, which if greater than 1.5, indicates the differences were due to normal Gaussian distribution. However, the data produced P values from 0.02 to 0.156, indicating more than normal error is present.

Because of these two factors, it would not be correct to try to determine a function to define the data; but only report and recognize the trend.

The math model predicted the spring rate to be a function of preload and temperature. It was verified to be a function of preload, as previously discussed, and the graph of spring rate vs. temperature shows it to be a function of temperature also; thus the math model was accurate in its predictions.

To determine the exact trend, it would be necessary to use a greater number of springs at many more preloads.

## Practical Applications

From the results of this second phase of the program, it appears that the deflection characteristic of a spring can also be used by fire investigators. This has direct applicability in determining if a spring was in tension or relaxed at the time of the fire (i.e. preloaded). Thus, it could be stated that, for example, the garage door was open (springs in tension) at the time of the fire.

## Conclusions

The results presented in this paper illustrate the potential of the use of mechanical properties for fire/arson investigation. Granted, the results are for only specific springs, but the method can be directly applied to all springs. This is presently being accomplished at Ohio State University and Systems Engineering Associates. However, a great deal of continued research is needed in the area of temperature determination from mechanical properties. Specifically, data curves for all types of springs, methods of spring selection for fire/arson investigators, theoretical models to predict attained temperatures and much more should be investigated.

It is evident that there are still many problems to be examined with questions to be answered.

1. Guenther, D. A., et al., "Fire Analyses from Mechanical Properties", Fire Technology, Vol. 12 (1976) pp. 173-185.

2. Guenther, D. A., et al., "Temperature Determination of a Fire from Mechanical Properties", Fire Technology, Vol. 14 (1978) pp. 61-66.

3. Kelley, K. M., "Mechanical Properties of Springs After Heating", Internal Report, The Ohio State University, Department of Mechanical Engineering, 1979.

4. Zwick, D. F., and Hampton, P., "Personnel Communication," Systems Engineering Associates, Atlanta, Georgia, 1978.

5. Kauligan, G. H., and Houseman, M. R., "Temperature Coefficient of the Moduli of Metal and Alloys Used as Elastic Elements", Journal of Research, National Bureau of Standards, Vol. 10 (1933), p. 345.

6. Wahl, A. M., "Mechanical Springs" (1949), p. 79.

7. Spotts, M. F., Design of Machine Elements, fourth edition (Prentice-Hall, Englewood Cliffs, New Jersey, 1971), p. 217.

| SPRING NUMBER | K, lbf/in (BEFORE HEATING) | K, lbf/in (AFTER HEATING) | TEMPERATURE °F | TIME HOUR | % ΔK |
|---|---|---|---|---|---|
| 20a | 52.88 | 49.94 | 1500 | 1/2 | 5.56% |
| 21a | 53.92 | 50.91 | 1500 | 1 | 5.58% |
| 22a | 53.79 | 51.64 | 1500 | 1 1/2 | 4.0 % |
| 23a | 56.63 | 53.28 | 1500 | 2 | 5.92% |
| 24a | 51.61 | 48.81 | 1500 | 2 1/2 | 5.43% |

TABLE 1. TIME TESTED SPRINGS

| SPRINGS | TEMPERATURE °F | TIME HOUR | K, lbf/in | % ΔK |
|---|---|---|---|---|
| Before Heating | - | - | K standard 31.99 | - |
| 1-6 | 1350 | 1 | 31.5 | 1.53 |
| 7-12 | 1400 | 1 | 29.01 | 9.32 |
| 13-18 | 1450 | 1 | 28.71 | 10.25 |
| 19-24 | 1500 | 1 | 26.00 | 18.72 |
| 25-30 | 1550 | 1 | 25.99 | 18.76 |
| 31-36 | 1600 | 1 | 23.47 | 26.63 |
| 37-42 | 1650 | 1 | 22.07 | 31.01 |
| 43-48 | 1700 | 1 | 20.23 | 36.76 |
| 49-54 | 1750 | 1 | 20.37 | 36.32 |
| 55-60 | 1800 | 1 | 18.00 | 43.73 |
| 61-66 | 1850 | 1 | 18.65 | 41.70 |
| 67-72 | 1900 | 1 | 11.90 | 62.80 |
| 73-78 | 1950 | 1 | 14.50 | 54.64 |
| 79-84 | 2000 | 1 | UNABLE TO BE TESTED | |

TABLE 2. TEMPERATURE TESTED SPRINGS

## TABLE 3

| TEMPERATURE, $^{o}F$ | SLOPE OF NORMALIZED SPRINGS RATE VS. DEFLECTION LINE |
|---|---|
| 800 | - 0.0092 |
| 1000 | - 0.035 |
| 1200 | - 0.0956 |
| 1400 | - 0.092 |
| 1600 | 0.1417 |
| 1800 | 0.0960 |

## TABLE 4

| TEMPERATURE, $^{o}F$ | P VALUE FROM STUDENT t-TEST |
|---|---|
| 800 - 1000 | 0.06218 |
| 1000 - 1200 | 0.0848 |
| 1200 - 1400 | 0.0536 |
| 1400 - 1600 | 0.01979 |
| 1600 - 1800 | 0.156 |

# TABLE 5

| 1200°F | SPRING NO. | FREE LENGTH (INCHES) | NUMBER OF COILS TESTED | SPRING RATE MEASURED ($\frac{LB}{IN}$) | TRUE SPRING RATE CALCULATED ($\frac{LB}{IN}$) |
|---|---|---|---|---|---|
| 0" | 48 | 13.5 | 50 | 6.0 | 6.0 |
| - | 89 | 14.0 | 50 | 6.0 | 6.0 |
| - | 95 | 17.5 | 37 | 7.5 | 5.5 |
| - | 29 | 18.5 | 30 | 8.25 | 4.95 |
| 2" | 22 | 17.0 | 40 | 7.0 | 5.6 |
| AMOUNT OF - | 86 | 18.5 | 35 | 8.0 | 5.6 |
| DEFLECTION - | 30 | 17.5 | 35 | 8.25 | 5.8 |
| - | 85 | 19.0 | 36 | 8.0 | 5.76 |
| 4" | 82 | 20.0 | 35 | 8.25 | 5.78 |
| - | 98 | 21.0 | 34 | 8.0 | 5.44 |
| - | 91 | 20.5 | 34 | 8.0 | 5.44 |
| - | 87 | 19.5 | 37 | 7.5 | 5.55 |
| 6" | 30 | 23.5 | 33 | 9.25 | 6.11 |
| - | 14 | 21.0 | 32 | 9.0 | 5.76 |
| - | 2 | 22.0 | 34 | 8.5 | 5.78 |
| - | 26 | 28.0 | 27 | 11.0 | 5.94 |
| 1400°F | | | | | |
| 0" | 23 | 14.0 | 49 | 5.25 | 5.145 |
| - | 25 | 14.0 | 42 | 6.0 | 5.04 |
| - | 81 | 15.0 | 45 | 6.0 | 5.40 |
| - | 27 | 26.5 | 26 | 9.5 | 4.94 |
| 2" | 19 | 18.0 | 36 | 7.0 | 5.04 |
| - | 24 | 18.0 | 40 | 6.0 | 4.8 |
| AMOUNT OF - | 9 | 17.5 | 41 | 7.0 | 5.74 |
| DEFLECTION - | 92 | 21.5 | 38 | 6.5 | 4.94 |
| 4" | 84 | 20.0 | 33 | 7.75 | 5.12 |
| - | 20 | 25.5 | 33 | 8.0 | 5.28 |
| - | 47 | 21.5 | 37 | 6.75 | 4.99 |
| - | 93 | 19.0 | 39 | 7.0 | 5.46 |
| 6" | 16 | 22.0 | 35 | 7.0 | 4.9 |
| - | 73 | 27.0 | 27 | 8.75 | 4.73 |
| - | 58 | 22.0 | 36 | 7.0 | 5.04 |
| - | 44 | 27.0 | 30 | 7.5 | 4.5 |

TABLE

| 1600°F | SPRING NO. | FREE LENGTH (INCHES) | NUMBER OF COILS TESTED | SPRING RATE MEASURED $(\frac{LB}{IN})$ | TRUE SPRING RATE CALCULATED $(\frac{LB}{IN})$ |
|---|---|---|---|---|---|
| 0" | 42 | 21.0 | 39 | 4.5 | 3.51 |
| - | 38 | 18.0 | 40 | 5.0 | 4.0 |
| - | 32 | 18.0 | 39 | 5.0 | 3.9 |
| - | 76 | 13.0 | 51 | 5.0 | 5.1 |
| 2" | 48 | 19.0 | 41 | 4.75 | 3.9 |
| AMOUNT OF - | 72 | 26.5 | 31 | 6.0 | 3.7 |
| DEFLECTION - | 43 | 19.0 | 40 | 5.0 | 4.0 |
| - | 54 | 22.0 | 38 | 5.0 | 3.8 |
| 4" | 46 | 19.0 | 40 | 5.5 | 4.4 |
| - | 50 | 20.25 | 32 | 5.0 | 3.2 |
| - | 62 | 20.75 | 28 | 6.5 | 3.6 |
| - | 56 | 21.0 | 31 | 6.75 | 4.2 |
| 6" | 40 | 22.2 | 33 | 7.0 | 4.6 |
| - | 60 | 31.0 | 30 | 7.75 | 4.6 |
| - | 51 | 27.0 | 28 | 7.25 | 4.1 |
| - | 106 | 20.0 | 35 | 6.5 | 4.6 |
| 1800°F | | | | | |
| 0" | 55 | 19.5 | 28 | 6.5 | 3.6 |
| - | 61 | 20.0 | 28 | 6.0 | 3.4 |
| - | 45 | 13.5 | 15 | 4.0 | 1.2 |
| - | 74 | 14.0 | 37 | 4.5 | 3.3 |
| 2" | 53 | 17.5 | 38 | 5.25 | 4.0 |
| - | 77 | 15.0 | 43 | 3.75 | 3.2 |
| AMOUNT OF - | 104 | 17.0 | 35 | 6.0 | 4.2 |
| DELFECTION - | 105 | 18.0 | 35 | 4.5 | 3.2 |
| 4" | 110 | 20.2 | 33 | 6.5 | 4.3 |
| - | 107 | 19.5 | 34 | 6.0 | 4.1 |
| - | 101 | 19.5 | 33 | 6.25 | 4.1 |
| - | 13 | 20.0 | 33 | 5.0 | 3.3 |
| 6" | 103 | 19.5 | 29 | 7.0 | 4.1 |
| - | 67 | 22.2 | 29 | 6.75 | 3.9 |
| - | 70 | 27.0 | 30 | 6.5 | 3.9 |
| - | 5 | 31.75 | 22 | 9.0 | 4.0 |

## TABLE 5

| 800°F | SPRING NO. | FREE LENGTH (INCHES) | NUMBER OF COILS TESTED | SPRING RATE MEASURED $(\frac{LB}{IN})$ | TRUE SPRING RATE CALCULATED $(\frac{LB}{IN})$ |
|---|---|---|---|---|---|
| 0" | 3 | 12.25 | 50 | 6.25 | 6.25 |
| - | 69 | 12.38 | " | 6.0 | 6.0 |
| - | 28 | 12.25 | " | 6.0 | 6.0 |
| - | 99 | 12.0 | " | 6.25 | 6.25 |
| 2" | 34 | 12.88 | " | 6.0 | 6.0 |
| AMOUNT OF - | 8 | 13.0 | " | 6.0 | 6.0 |
| DEFLECTION - | 64 | 13.25 | " | 6.25 | 6.25 |
| - | 36 | 13.5 | " | 6.25 | 6.25 |
| 4" | 52 | 15.13 | " | 6.0 | 6.0 |
| - | 35 | 15.0 | " | 6.0 | 6.0 |
| - | 59 | 15.0 | " | 6.0 | 6.0 |
| - | 78 | 15.25 | " | 6.25 | 6.25 |
| 6" | 4 | 17.0 | " | 6.0 | 6.0 |
| - | 18 | 17.0 | " | 6.0 | 6.0 |
| - | 12 | 17.0 | " | 6.25 | 6.25 |
| - | 11 | 17.0 | 36 | 8.5 | 6.12 |
| 1000°F | | | | | |
| 0" | 97 | 12.75 | 50 | 6.0 | 6.0 |
| - | 94 | 13.0 | " | 6.5 | 6.5 |
| - | 88 | 12.88 | " | 6.0 | 6.0 |
| - | 66 | 12.5 | " | 6.0 | 6.0 |
| 2" | 21 | 15.38 | " | 5.75 | 5.75 |
| - | 96 | 16.0 | " | 5.5 | 5.5 |
| AMOUNT OF - | 6 | 15.88 | " | 5.75 | 5.75 |
| DEFLECTION - | 100 | 15.75 | " | 6.25 | 6.25 |
| 4" | 1 | 17.5 | " | 5.0 | 5.0 |
| - | 83 | 18.0 | 36 | 8.5 | 6.1 |
| - | 7 | 18.5 | 37 | 8.0 | 5.9 |
| - | 79 | 18.25 | 35 | 9.0 | 6.3 |
| 6" | 39 | 19.0 | 28 | 9.25 | 5.2 |
| - | 41 | 20.0 | 26 | 11.0 | 5.7 |
| - | 15 | 19.5 | 33 | 9.5 | 6.3 |
| - | 10 | 20.0 | 33 | 9.25 | 6.11 |

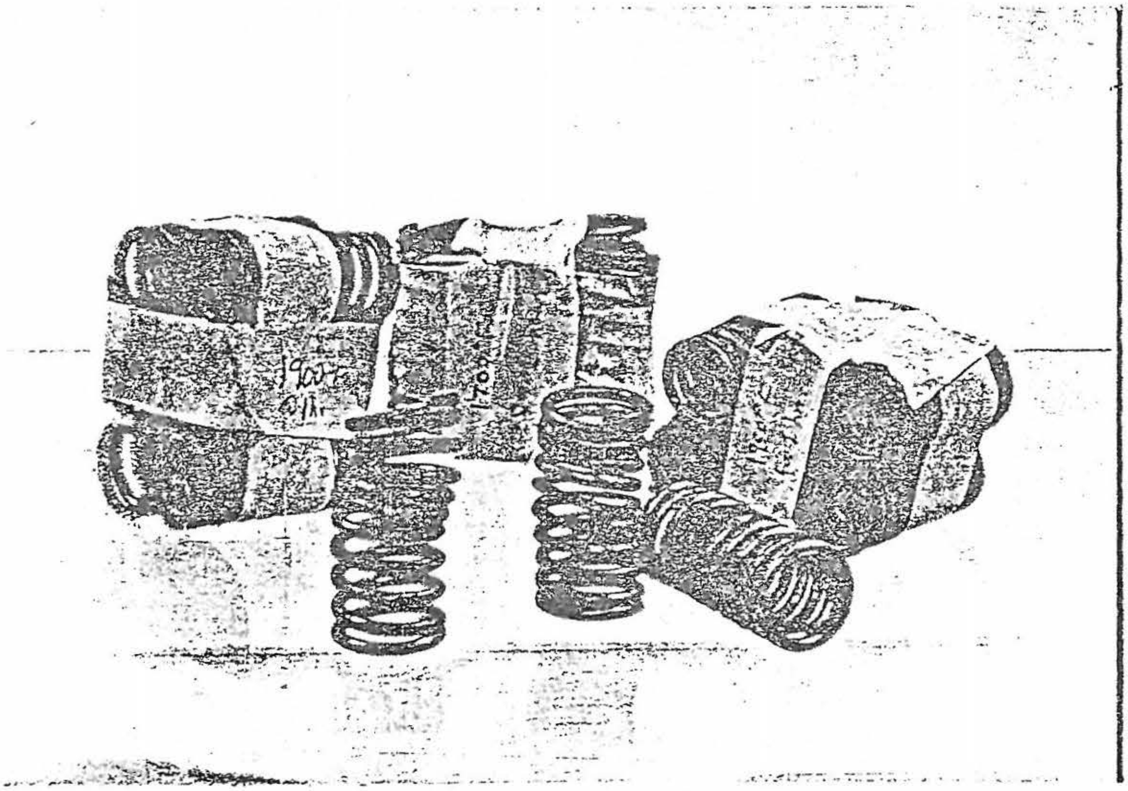


Figure No. 1

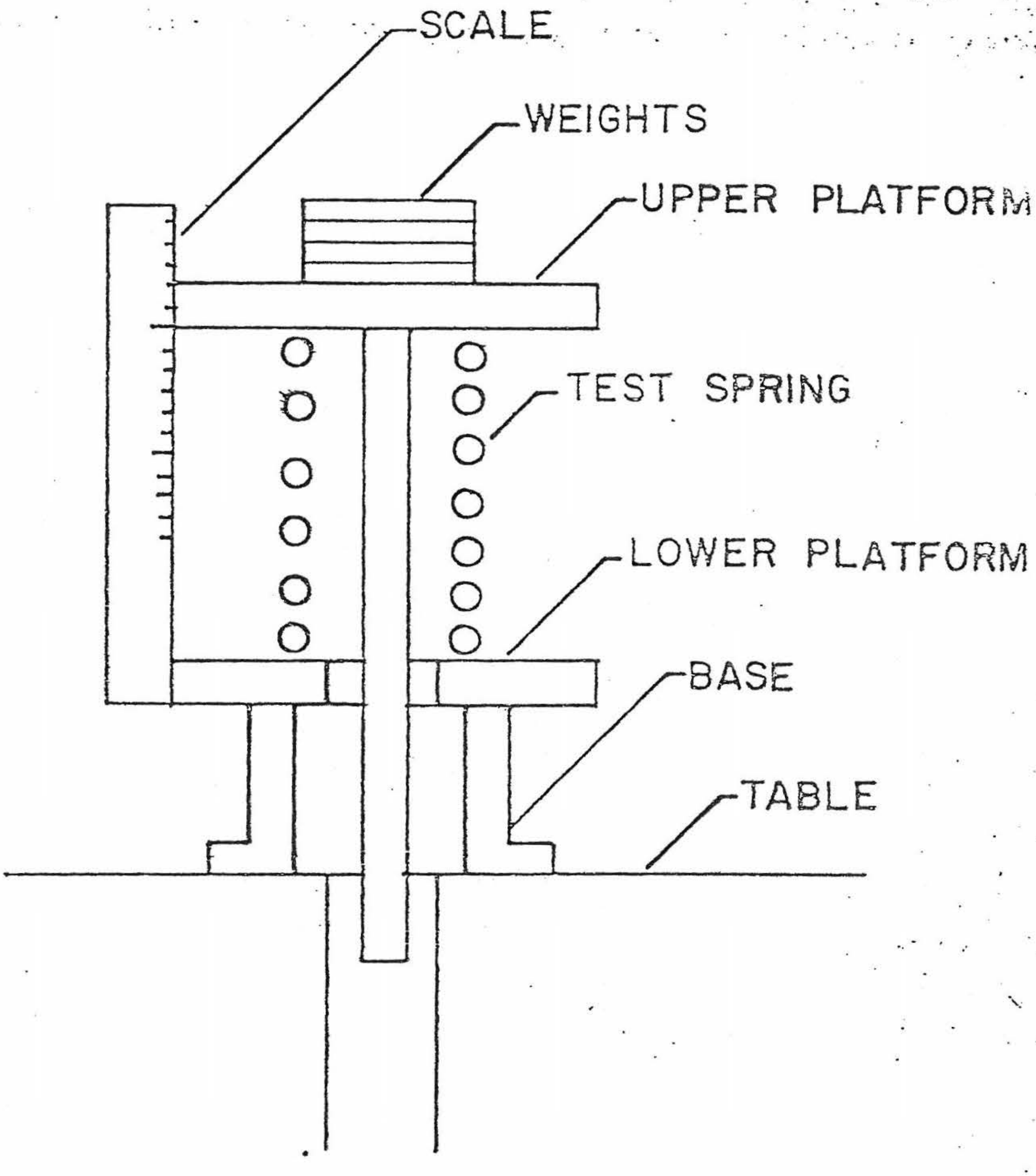

SCALE
WEIGHTS
UPPER PLATFORM
TEST SPRING
LOWER PLATFORM
BASE
TABLE

Figure No. 2

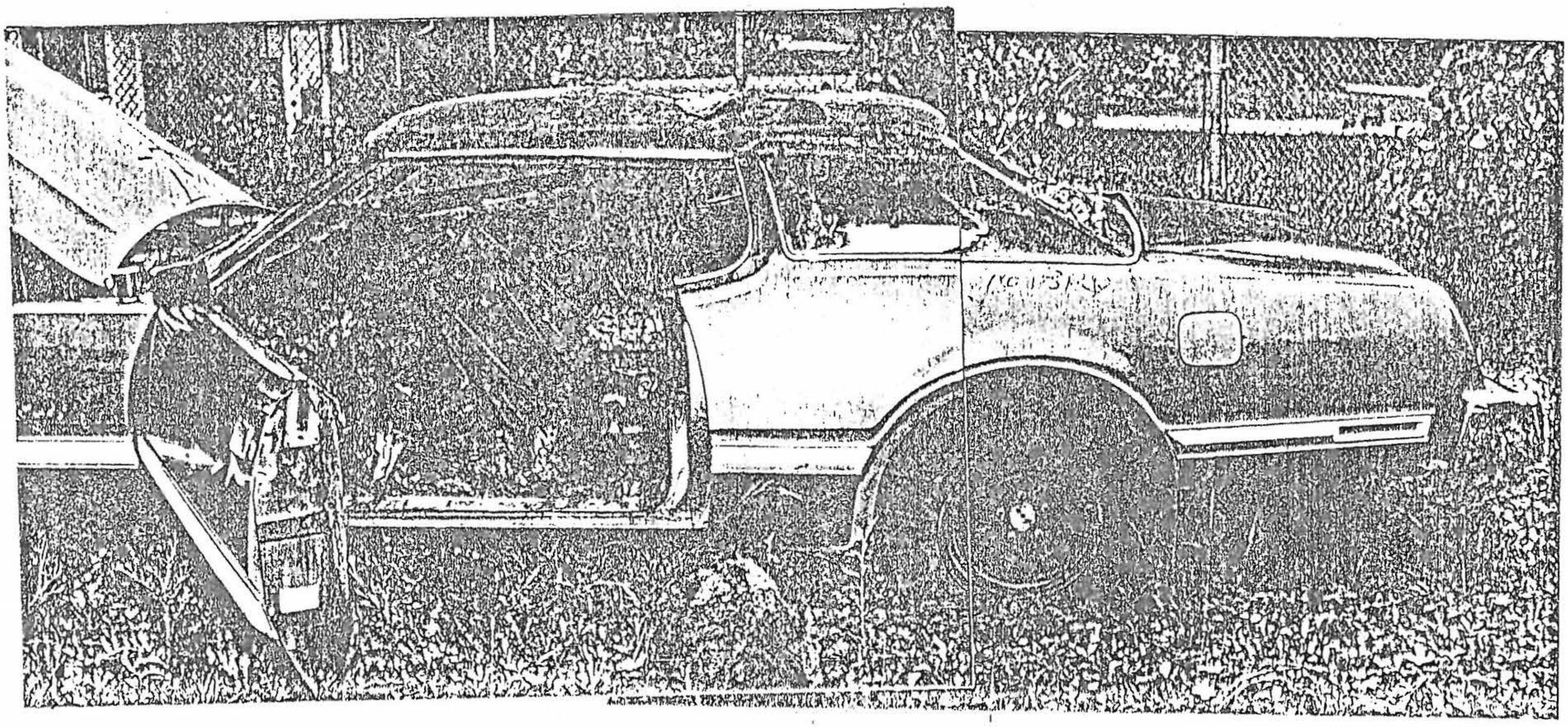


Figure No. 3

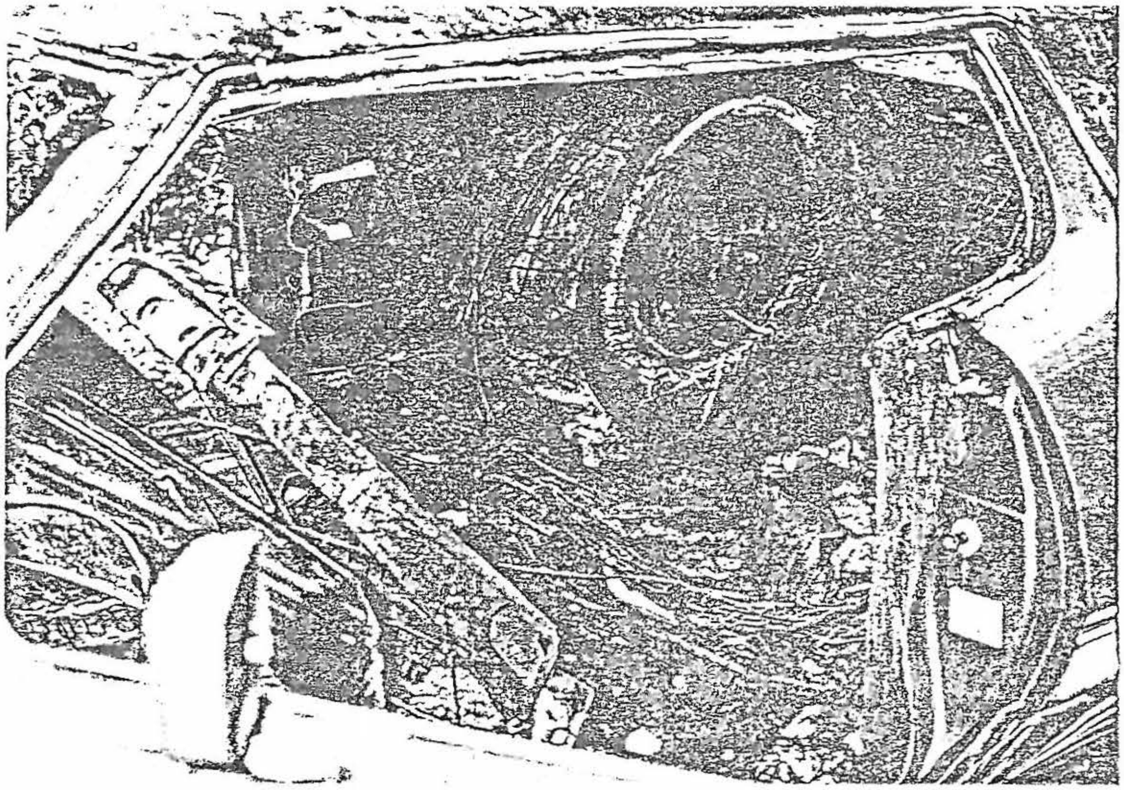


Figure No. 4

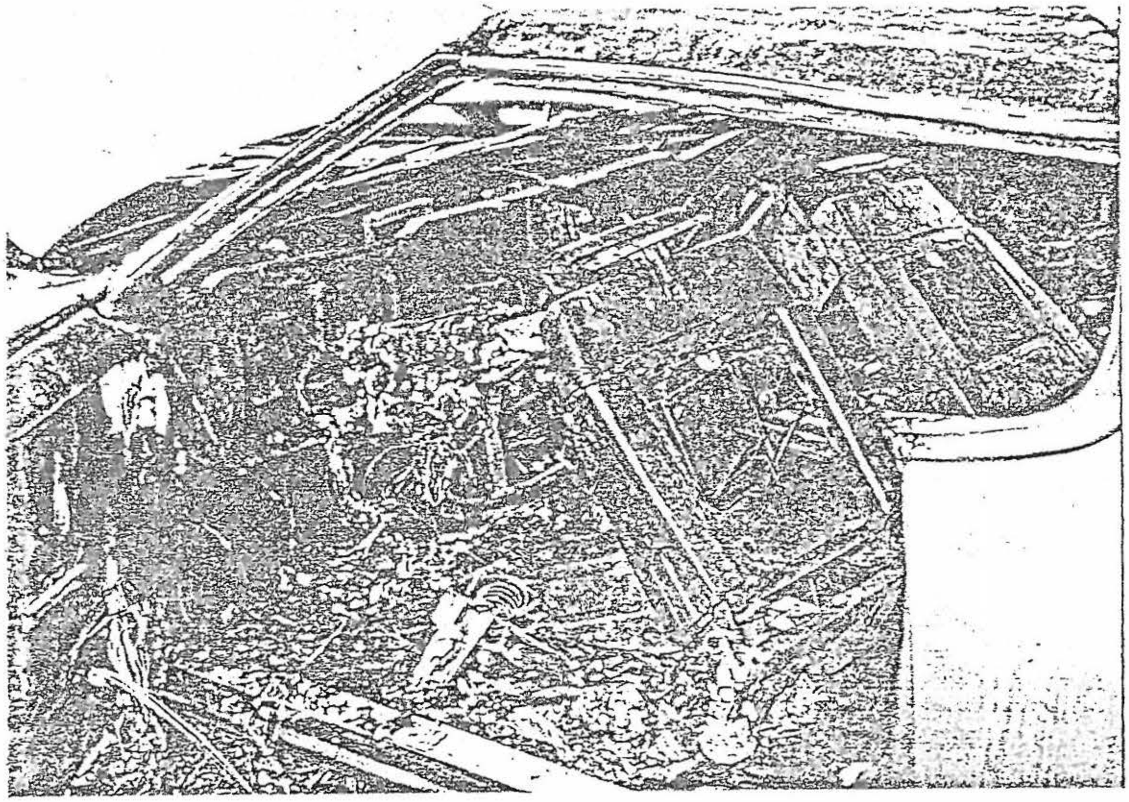
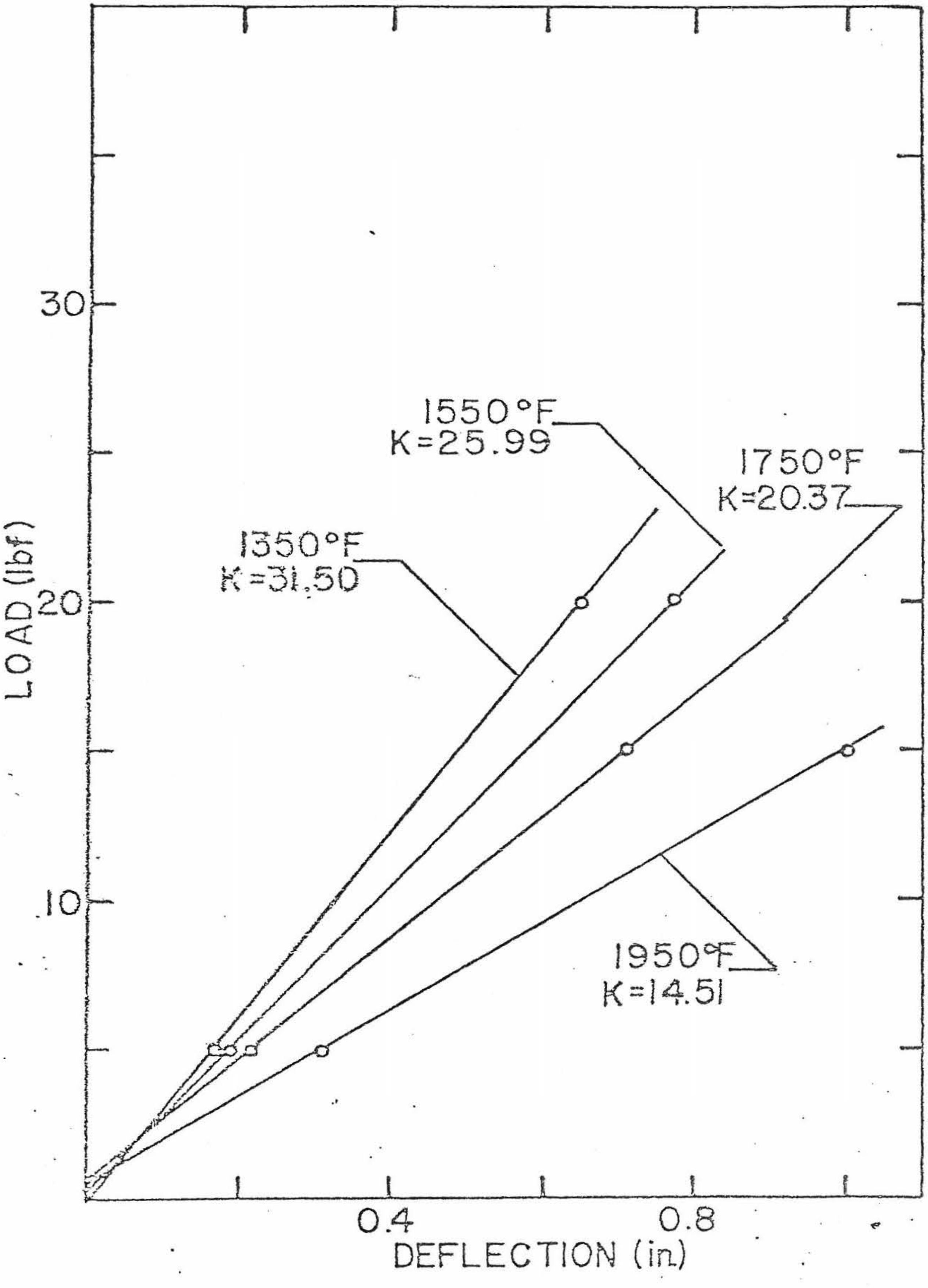
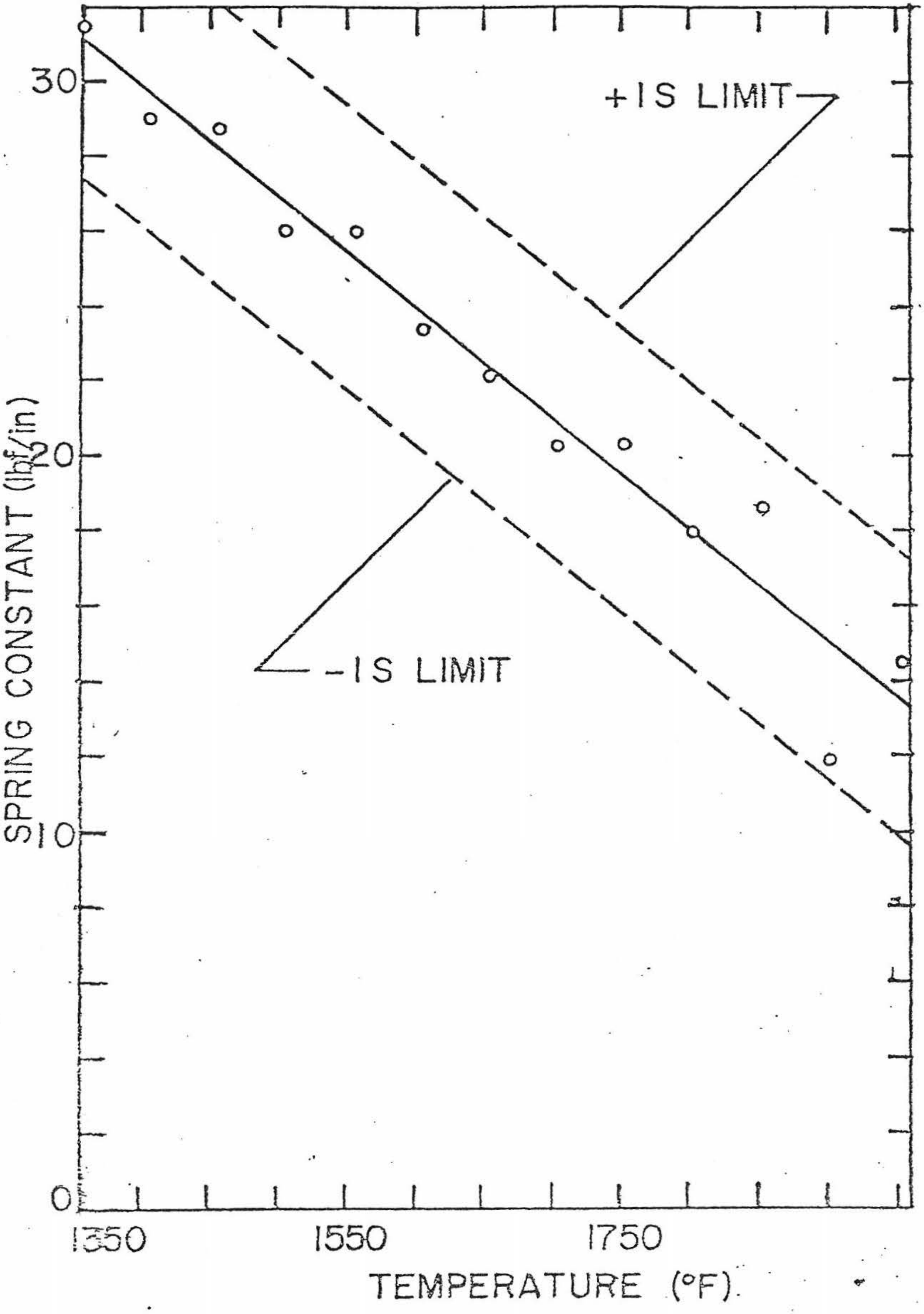


Figure No. 5


30
20
10
LOAD (lbf)
0.4
0.8
DEFLECTION (in)
1550°F
K=25.99
1750°F
K=20.37
1350°F
K=31.50
1950°F
K=14.51

Graph No. 1


30
20
10
0
1350
1550
1750
SPRING CONSTANT (lbf/in)
TEMPERATURE (°F)
+1S LIMIT
-1S LIMIT

Graph No. 2

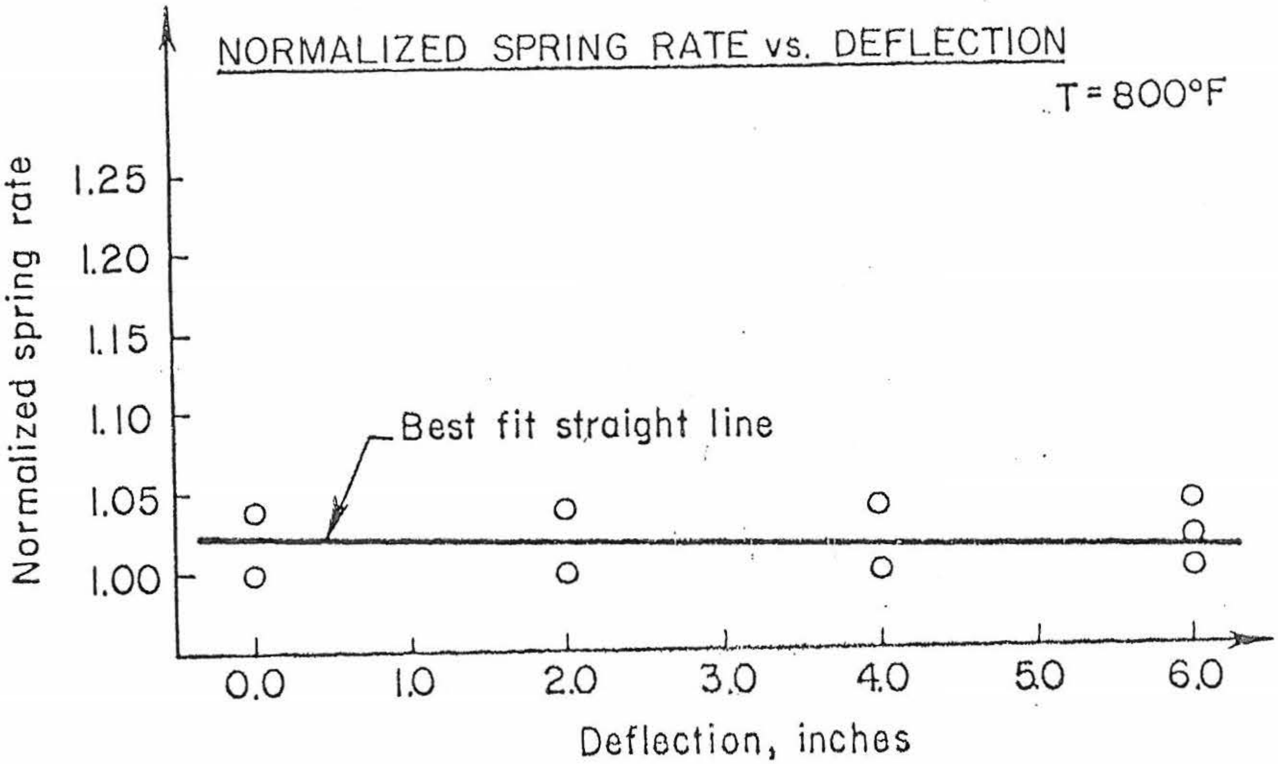

NORMALIZED SPRING RATE vs. DEFLECTION
T = 800°F
Normalized spring rate
1.25
1.20
1.15
1.10
1.05
1.00
Best fit straight line
0.0
1.0
2.0
3.0
4.0
5.0
6.0
Deflection, inches

Figure No. 7

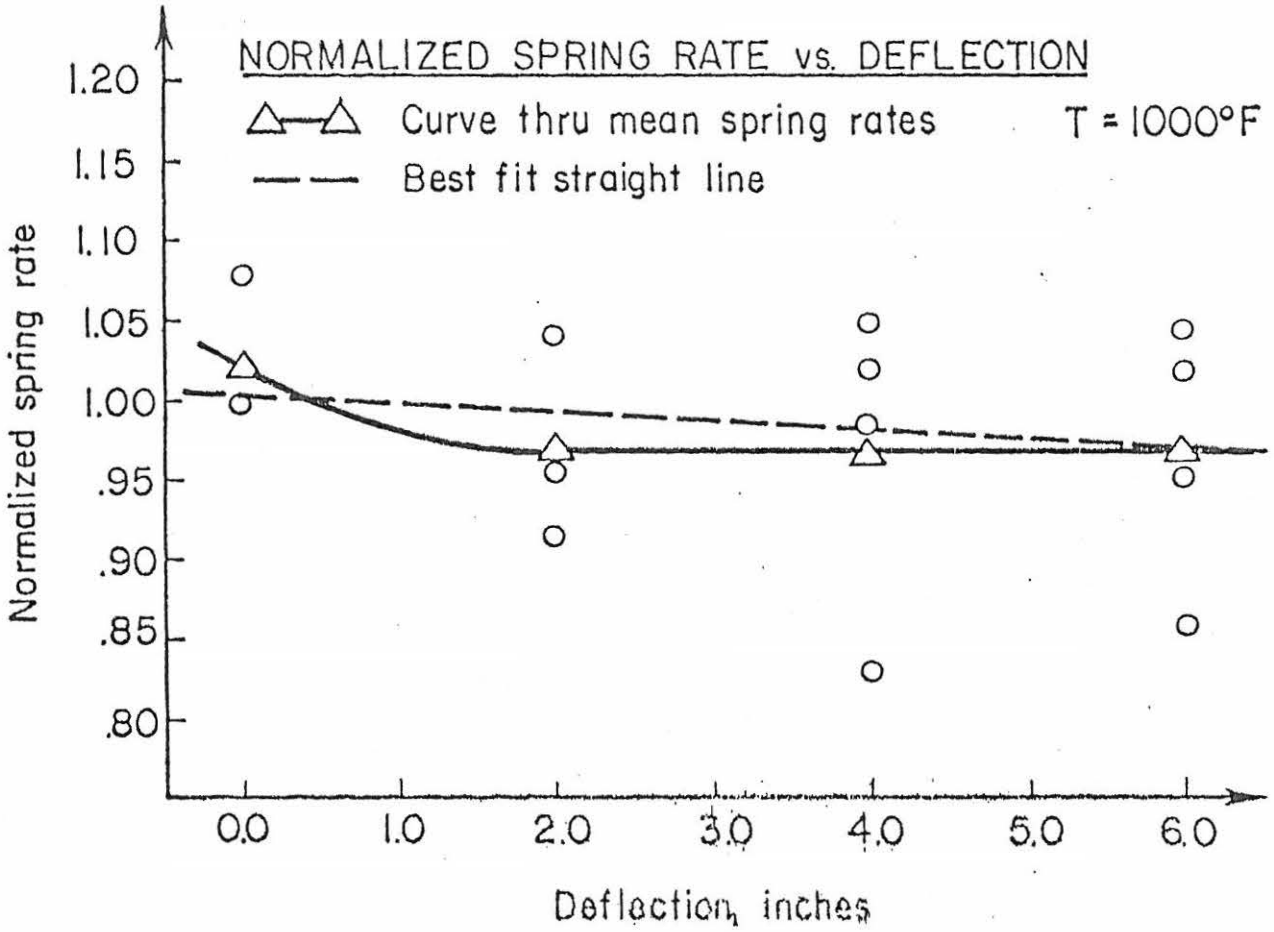

NORMALIZED SPRING RATE vs. DEFLECTION
△—△ Curve thru mean spring rates
T = 1000°F
— — Best fit straight line
Normalized spring rate
1.20
1.15
1.10
1.05
1.00
.95
.90
.85
.80
0.0
1.0
2.0
3.0
4.0
5.0
6.0
Deflection, inches

Figure No. 8

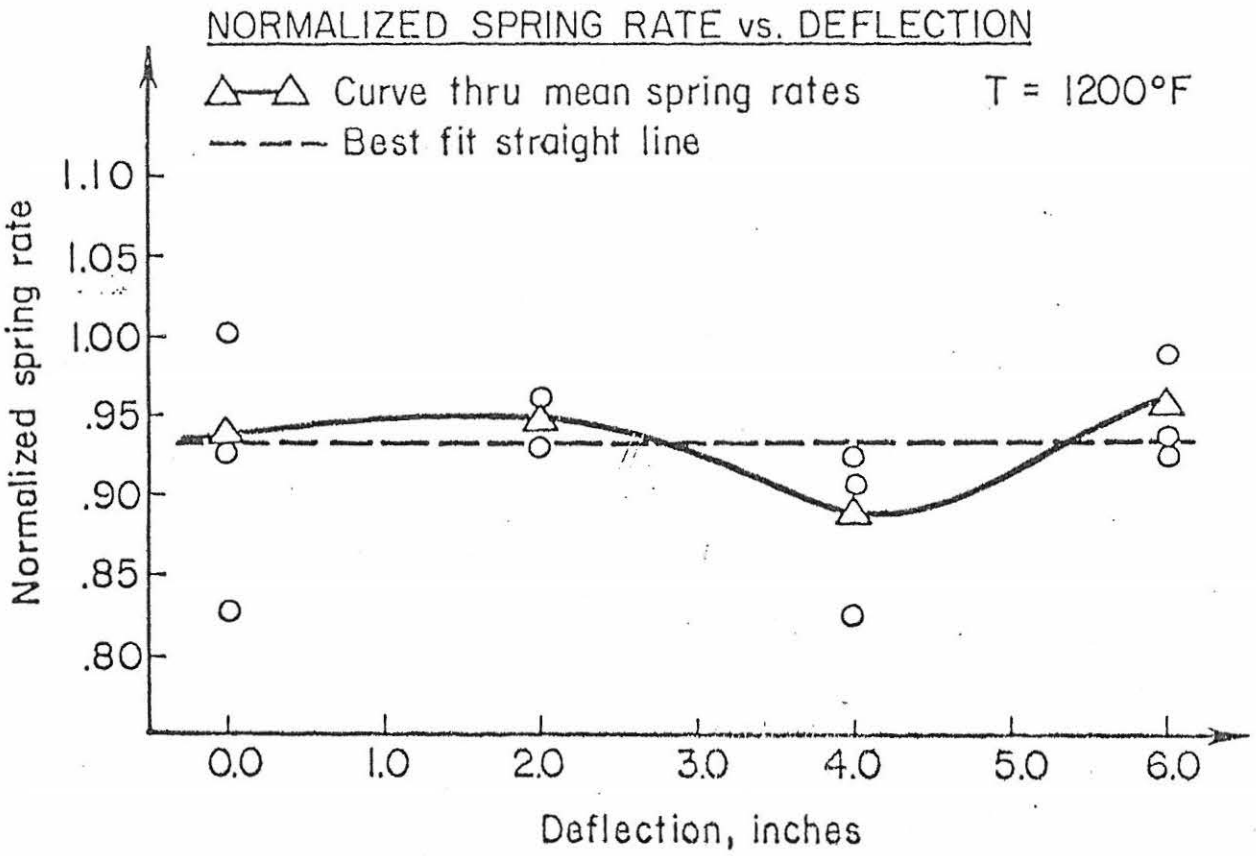

NORMALIZED SPRING RATE vs. DEFLECTION
△—△ Curve thru mean spring rates
T = 1200°F
— — — Best fit straight line
Normalized spring rate
1.10
1.05
1.00
.95
.90
.85
.80
0.0
1.0
2.0
3.0
4.0
5.0
6.0
Deflection, inches

Figure No. 9

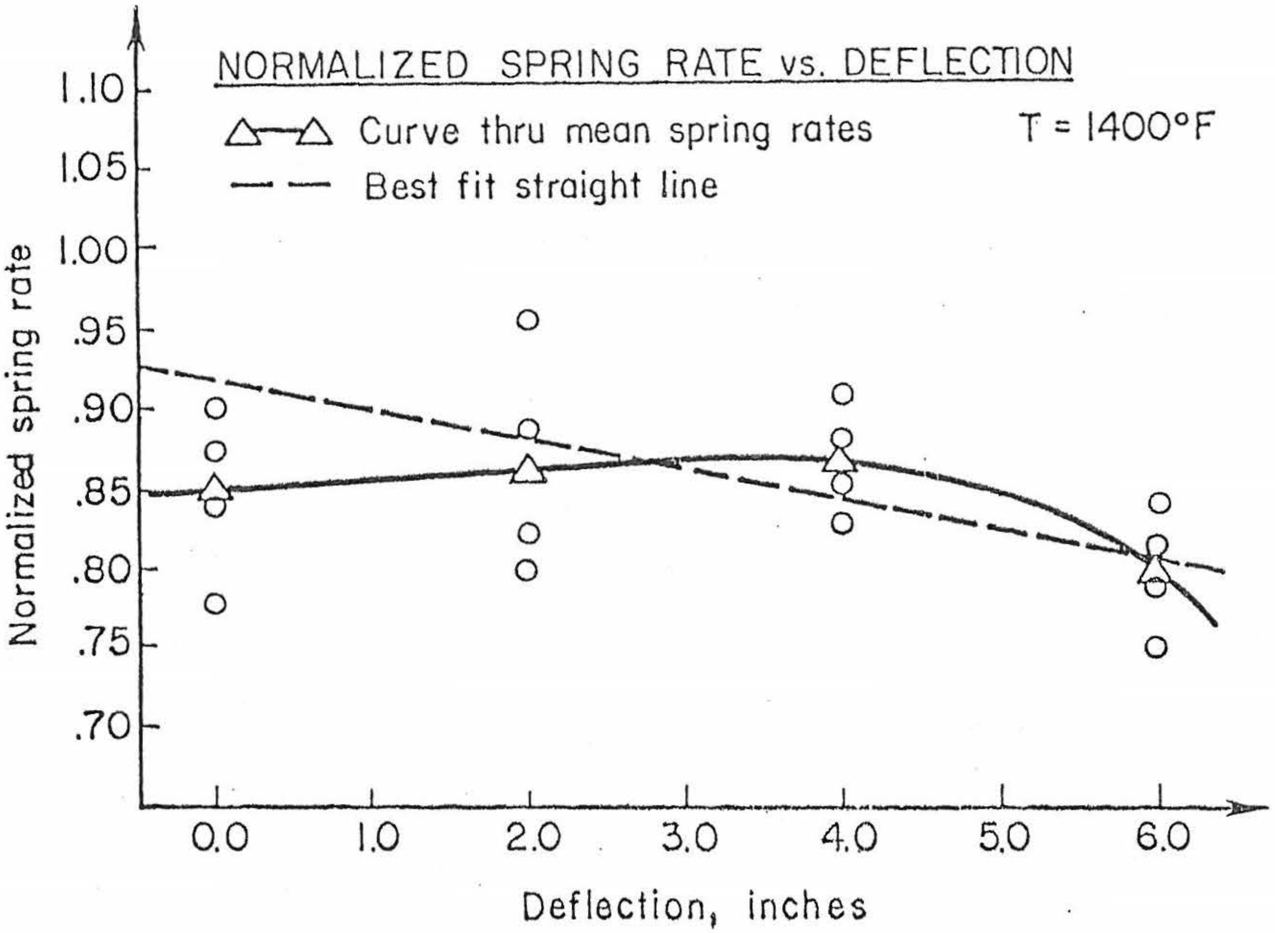

NORMALIZED SPRING RATE vs. DEFLECTION
Curve thru mean spring rates
Best fit straight line
T = 1400°F
Normalized spring rate
1.10
1.05
1.00
.95
.90
.85
.80
.75
.70
0.0
1.0
2.0
3.0
4.0
5.0
6.0
Deflection, inches

Figure No. 10

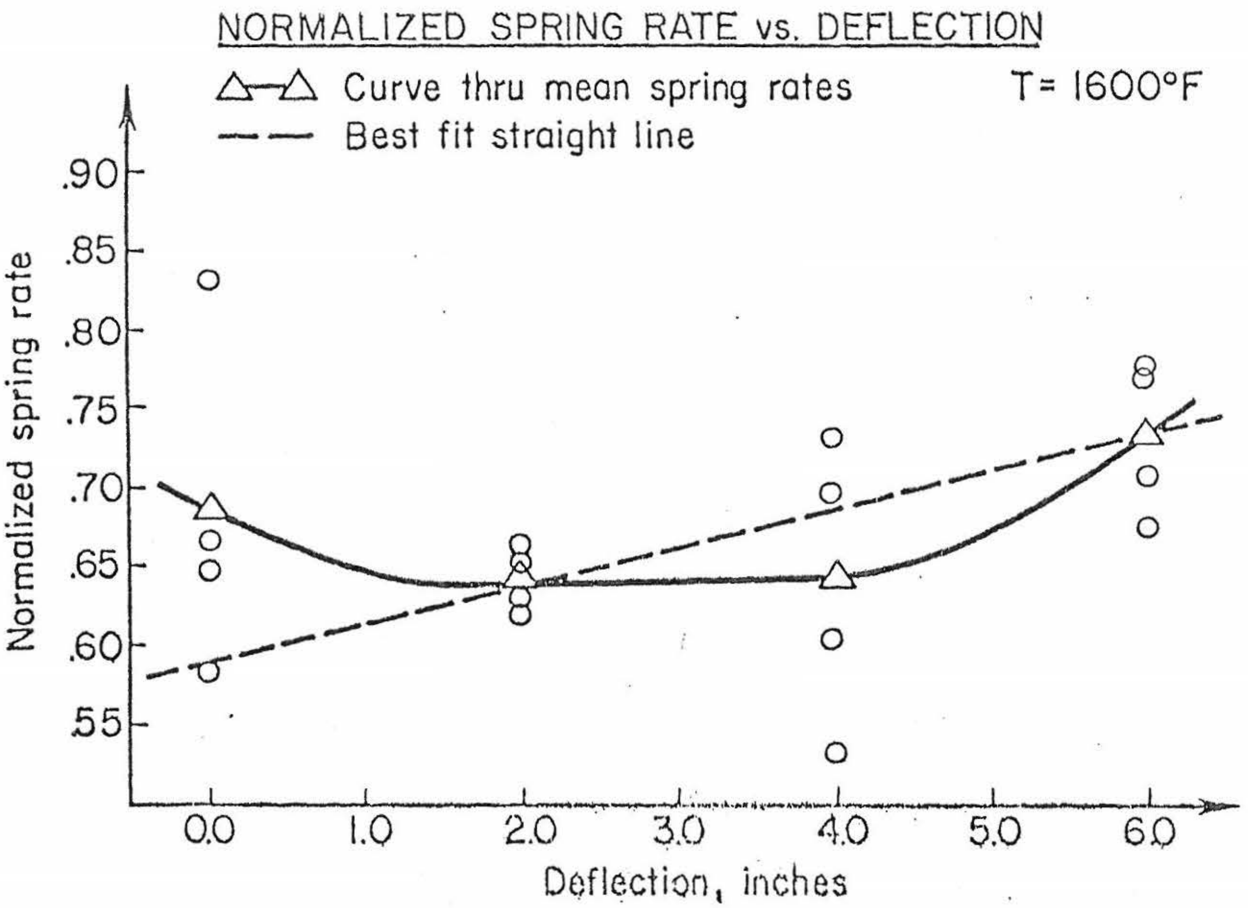

NORMALIZED SPRING RATE vs. DEFLECTION
△—△ Curve thru mean spring rates
T= 1600°F
— — Best fit straight line
Normalized spring rate
.90
.85
.80
.75
.70
.65
.60
.55
0.0
1.0
2.0
3.0
4.0
5.0
6.0
Deflection, inches

Figure No. 11

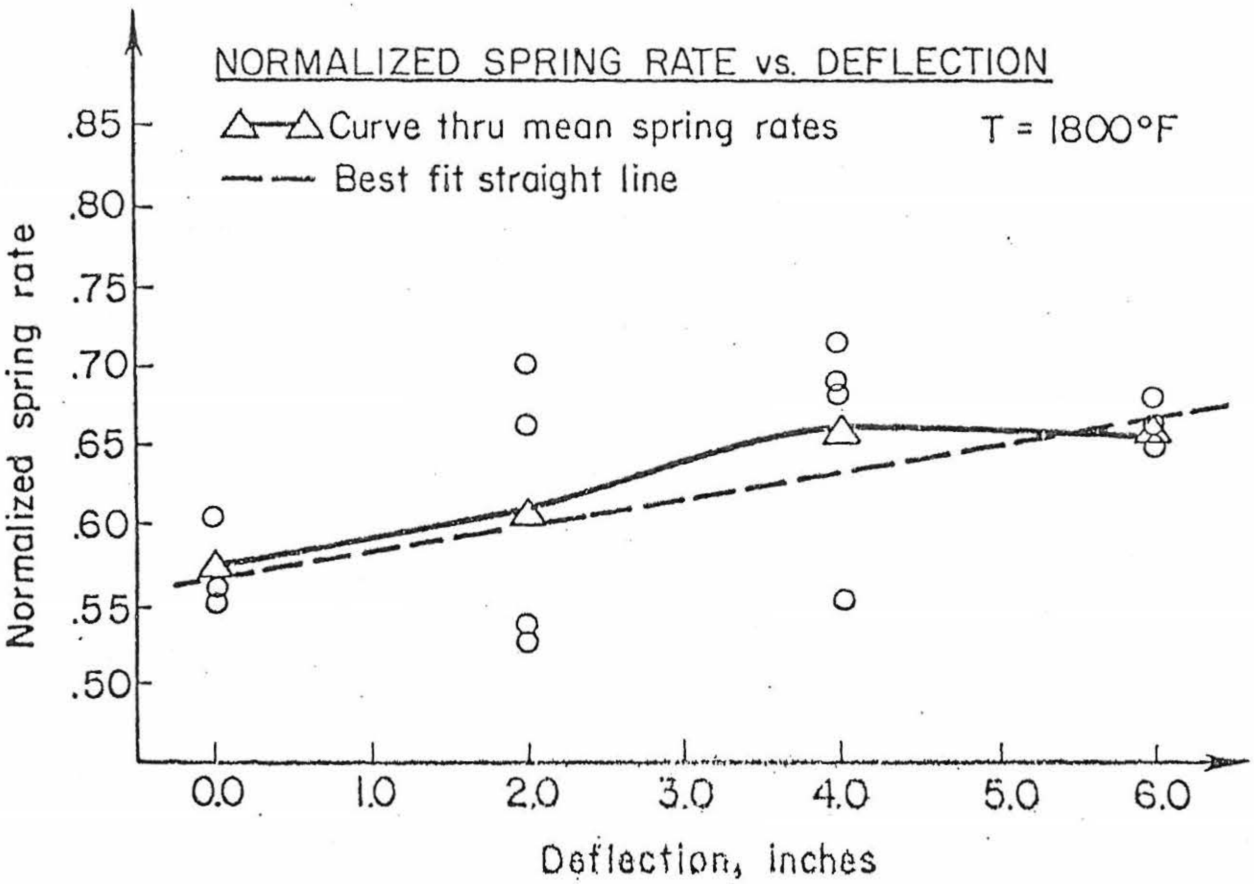

NORMALIZED SPRING RATE vs. DEFLECTION
△—△ Curve thru mean spring rates
— — Best fit straight line
T = 1800°F
Normalized spring rate
.85
.80
.75
.70
.65
.60
.55
.50
0.0
1.0
2.0
3.0
4.0
5.0
6.0
Deflection, inches

Figure No. 12

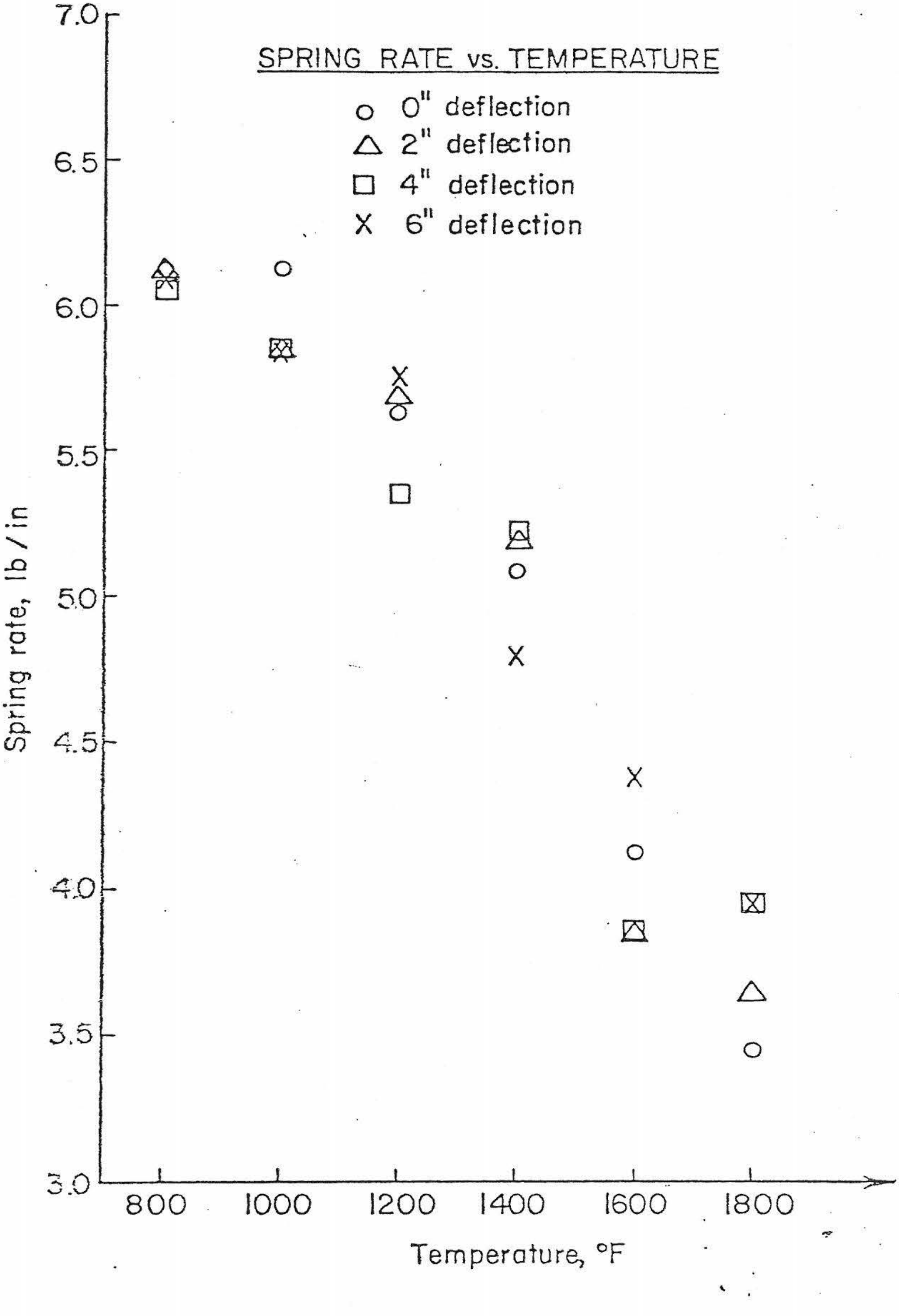

SPRING RATE vs. TEMPERATURE
○ 0" deflection
△ 2" deflection
□ 4" deflection
X 6" deflection
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
Spring rate, lb / in
800
1000
1200
1400
1600
1800
Temperature, °F

Figure No. 13

# COPPER BASED INCENDIARY

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95813

The Washoe County Sheriff's Department Crime Lab in Reno, Nevada has reported that within the last six months it had encountered four cases of arson or attempted arson in which an unusual incendiary or its residue had been encountered. Fortunately, in the first case examined, one of the incendiary devices had not ignited. This device consisted of a partial beer can containing approximately 80 grams of grey powdery material and a cigarette match delayed ignition mechanism. The grey powder was found to be a copper oxide-based "Thermite" mixture. Two companies were found that manufacture such a mixture, the first being Erico Products Incorporated of Solon, Ohio whose brand name is "Cadweld" and the second being Continental Industries Incorporated of Tulsa, Oklahoma, with their brand, "Thermoweld". These materials are available commercially and are used for welding copper buss bars together in large-scale electrical applications. The material can be ignited at approximately 800°F by the use of an ordinary match. The resulting exothermic reaction produces temperatures in excess of 4000°F. This product is marked in individual containers of up to one and one-half pound size.

Based upon laboratory tests of extremely small quantities of this material (which shattered porcelain crucibles and then welded them together) the effect of one of these large containers must be quite impressive. Erico Products reported the composition of their product to be as follows:

Copper mill scales (essentially copper oxide) 70-80%
Aluminum/copper alloy 14-18%
Fluorspar (calcium fluoride) 1-3%
Calcium-Silicon alloy 1-3%
Aluminum-vanadium alloy 0-2%
Tin 0-8%
Iron mill scale (essentially $FE_3O_4$) 0-1%

Information from Continental Industries indicates that their Thermoweld Product is very similar in composition and burn characteristics. Unfortunately, a detailed list of components was not available to confirm this.

Tests in the Sacramento DOJ Lab reveal that, upon ignition, a molten slag of quite low viscosity is produced almost immediately. When unconfined, this slag produces a radial splatter pattern of molten droplets. A residue upon cooling is found to consist of a number of small (1/2-2 mm diameter) beads of high purity copper, a black amorphous slag containing aluminum oxide and copper oxides, and masses of brown or blackened "gold"-colored metallic copper. Elemental analysis of the small beads of material reveal the presence of copper, aluminum, tin, silicon, calcium, and iron with traces of manganese and titanium.

Small, single-use quantities of Cadweld are packaged in press-top polyethylene vials, with the name "Cadweld" cast onto the top and bottom of each vial. This product, due to its ready availability and extremely high energy reactions, could be used in a wide variety of incendiary devices. In the Washoe County Sheriff's Department cased, the mixture had been used in conjunction with gasoline in a variety of containers, thus resulting in very efficient fires. The ease of ignition of this material lends itself to its use as an igniter in multiple sets of this type. Background case information from Reno indicates that devices of this type have been used in a variety of union disputes involving building sites. For further information, feel free to contact David Atkinson of the Crime Laboratory, Washoe County Sheriff's Department, P.O. Box 2915, Reno Nevada 89505, or John DeHaan at the Sacramento Lab., Department of Justice, P.O. Box 13337, Sacramento, California 95813.

January, 1979

O K L A H O M A

STATE BUREAU OF INVESTIGATION

NORTHEAST 36th AT EASTERN
P.O. BOX 11497
PHONE (405) 427-5421
OKLAHOMA CITY, OKLAHOMA 73136

Submitted by
Ronald W. Clodfelter

The Oklahoma State Bureau of Investigation Laboratory in Oklahoma City, Oklahoma is responsible for the examination of suspect fire debris submitted by law enforcement personnel from seventy-seven Oklahoma counties.

The goal of our laboratory is to provide investigative information to the submitting officer as rapidly as possible. (Normally 1-3 days after the evidence is submitted to the lab.) To accomplish this objective, we have assigned the responsibility of arson examination to three analysts. The use of multi-trained personnel versus the traditional approach of a single analyst offers at least a two-fold advantage:

1. One or more of the analysts are always available to receive and process the evidence the day it is submitted. This offers the additional opportunity for the analyst to discuss the evidence with the investigator and when necessary, request additional evidence (unburnt controls, etc.).
2. The use of three analysts trained in a particular discipline, allows for a more productive exchange of ideas and procedures.

The present methodology includes:

1. Steam distilling the suspect debris. (We currently distill from one-gallon paint cans fitted with metal lids which are modified to accept the 24/40 male end of a distilling receiver). The distillation time is normally 30 minutes to one hour; however, preliminary investigation indicates that the distillation time should be extended until a major portion of the accelerant is isolated. We have noted that the first few drops of distillate may contain only the most volatile portions of the accelerant with the less volatile components being left behind in the distillation container.
2. Recording the volume, color, odor and density (greater than or less than 1) of the distillate (the naturally occuring degradation products normally have a density less than 1, a pungent odor and are goldish in color. The pungent odor from the degradation products can easily mask the odor of most accelerants.)
3. Injecting ½-1 microliter of the distillate into a gas chromatograph equipped with a 1/8 inch 12 foot 15% Apiezon L column; programmed from 50 - 280$^\circ$C at 20$^\circ$C/min. with a one minute initial hold and a 7-20 minute final hold. All accelerants (gasoline, paint thinner, diesel, etc.) and experimental obtained degradation products have been examined at these parameters. This not only allows for a direct comparison of an unknown distillate

With many different accelerants and possible degradation products, but also allows us to categorize various liquids by relative retention times. When necessary, the parameters are changed to allow for better resolution of the various components.

4. Examining the controlled material. Occasionally we encounter extraneous peaks due to degradation products. When this occurs, we burn, then distill the control material and examine it chromatographically.
5. Flammability testing.
6. When appropriate, TLC examination of the gasoline dyes.
7. Storing the organic distillate in inverted $H_2O$ filled glass vials.

I would welcome any comments, experience, or literary references from other analysts concerning:

1. Selection of G.C. columns.
2. Application of EDX, IR, G.C./M.S. to arson evidence.
3. Analysis of additives in petroleum products.
4. Studies concerning degradation products, etc.

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# AA Notes

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* FROM THE JOURNALS:

Identification of Accelerants in Fire Residues by Capillary Gas Chromatography. Andrew T. Armstrong, Ph.D., Robert Wittkower, B.S., Department of Chemistry, University of Texas at Arlington, Texas. (paper D-45).

GC - Mass Spectroscopy: Its Use in Determining Gasoline Lead Alkyl Ratios and Their Use in Forensic Examinations. Leslie T. Lytle, Ph.D., Larry C. Ford, B.S., North Carolina State Bureau of Investigation, Raleigh N.C., Phillip T. Williamson, BS., Finnigan Instruments, Sunnyvale, CA. (paper B-46).

Use of "Pattern Recognition" in Arson Cases: A Progress Report. Bruce R. Kowalski, Ph.D., Department of Chemistry, University of Washington, Seattle Washington, and William R. Gresharm Ph.D., Western Washington State Crime Laboratory, Seattle Washington. (paper B-47).

The Use of Glass Capillary Columns in Arson Analysis. Kent A. Oakes Ph.D., Wisconsin Regional Crime Laboratory, Department of Justice, New Berlin, Wisconsin. (paper B-48)

A Systemwide Method of Identification of Petroleum Distillation in Arson Cases. Thomas J. Hopen, B.S., Richard A. Roper, Ph.D., Department of Toxicology and Criminal Investigation, Montgomery Regional Laboratory, Montgomery, Alabama. (paper B-49).

A Comparison of Head-Space and Solvent Extraction Techniques in the Analysis for Accelerant Residues in Arson Cases. Richard W. Henderson, Ph.D., Department of Chemistry and Physics, Frances Marion College, Florence, SC. (paper B-50).

The above papers were presented at the 31st Annual Meeting of the American Acedemy of Forensic Sciences, Feburary 12-17, 1979 in Atlanta, GA. A word of thanks to Dr. Jimmy Chao for the articles.

The November 1978 (vol.2, no.1) issue of Column Newsletter (Hewlett Packard) has a short article on the characterization of flammable liquids and explosives by chemical ionization mass spectroscopy. Ion chromatograms as well as mass spectrum of methyl acrylate, 2,4-dinitrotoluene, 2,4,6,-trinitrotoluene, nitroglycerin, 2,6,-dinitrotoluene, methyl centralite and butyl phthalate esters.

# AA Notes

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* Enclosed with this issue of the AAN, an invoice is supplied concerning most subscribers to the AAN. A prompt response to this invoice will allow for smoother operation of the AAN. Due to increased mailing costs the cost of the AAN to United State subscribers will be $6.00 and $20.00 for subscribers outside of the United States.

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