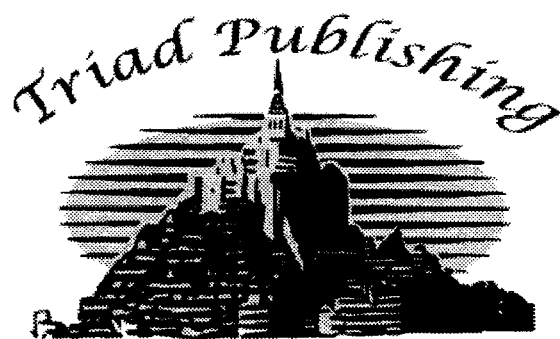


THE
THERMAL DECOMPOSITION
OF
METAL ACETATES
Historical and Chemical Aspects

by

Robert Allen Bartlett



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The original foreword from September 1997:

Dear Reader,

Over a decade ago, The Philosophers of Nature received the rights to a paper entitled "The Thermal Decomposition of Metal Acetates, Historical and Chemical Aspects" from the former Alchemed Foundation of Salt Lake City, Utah.

In January 1983, the Alchemed Foundation awarded Robert Allen Bartlett a financial grant to conduct research on the decomposition of acetates and to develop a paper on his findings. The subsequent dissolution of that foundation has prevented any publication before now.

The occasion of a practical workshop by The Philosophers of Nature on the subject of this paper seems an appropriate occasion to unveil this interesting work. We are indebted to the hard work and vision of Robert Bartlett and to the foresight of the administration of Alchemed. We hope that our own efforts will be added to theirs, so that those who come after us will benefit from the combined lights of their fellow seekers.

Ora et Labora!

Russ House

President, The Philosophers of Nature

TABULA SMARAGDINA HERMETIS

VERBA SECRETORUM HERMETIS

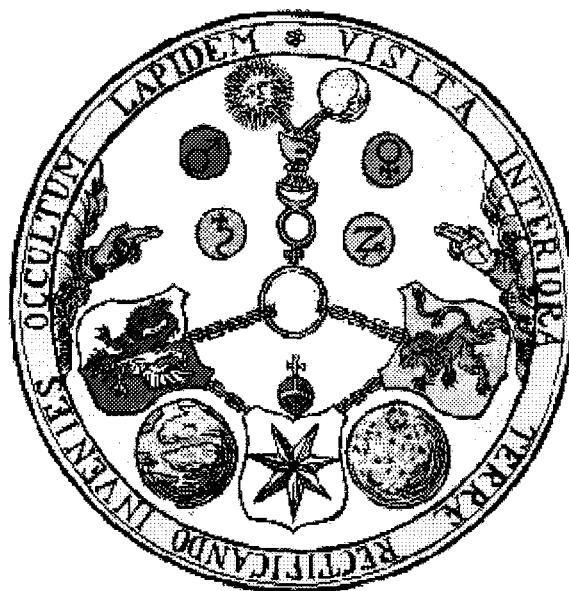
It is true, certain, and without falsehood, that whatever is below is like that which is above; and that which is above is like that which is below; to accomplish the one wonderful work. As all things are derived from the One Only Thing, by the will and by the word of the One Only One who created it in His Mind, so all things owe their existence to this Unity by the order of Nature, and can be improved by adaptation to that Mind.

Its Father is the Sea; its Mother is the Moon; the Wind carries it in its womb, and its nurse is the Earth. This Thing is the Father of all perfect things in the world. Its power is most perfect when it has been changed into Earth. Separate the Earth from the Fire, the subtle from the gross, but carefully and with great judgement and skill.

It ascends from earth to heaven, and descends again, new born, to the earth, taking unto itself thereby the power of the Above and the Below. Thus the splendor of the whole world will be thine, and all darkness shall flee from thee.

This is the strongest of all powers, the Force of all forces, for it overcometh all subtle things and can penetrate all that is solid. For thus was the world created and rare combinations, and wonders of many kinds are wrought.

Hence I am called HERMES TRISMEGISTUS, having mastered the three parts of the wisdom of the whole world. What I have to say about the masterpiece of the alchemical art, the Solar Work, is now ended.



INTRODUCTION

The main intent of this study has been to investigate a particular alchemical operation in the light of modern chemistry and briefly examine the ancient source from which it arose, alchemy.

The chemical operation has been termed ketogenic pyrolysis; wherein various metal salts (in this study, metal acetates) are decomposed by heat and the products of this decomposition captured for various industrial and medical applications. In more recent times, the greatest utility of this process has been in the production of acetone. However, with the rise of petroleum technology this process has largely been phased out in favor of methods that are more efficient in terms of energy, time, and yield. As a result, the process has become so neglected and obscure that relatively few individuals are familiar with it and even fewer who are familiar with its fascinating history. The operation has been known for centuries and in this paper we look at some of the historical, philosophical, and practical implications associated with it. The first mention of the process is in the writings of the early alchemists and it is here that we begin our search.

ALCHEMY

Today, alchemy is thought of as an obscure and primitive chemistry, the forerunner of modern chemistry. In order to gain a better appreciation of the development of the process involving decomposition of metal acetates, we must come to a more clarified picture of just what the ancient alchemists were seeking to accomplish. Of course, a detailed discussion of the art and science of alchemy would require many hundreds of volumes, if indeed it could be written down at all, for it is a very profound philosophy. It will be sufficient here to indicate some of the underlying currents of alchemical thought and their relation to the chemical operation under study.

*"The sacred, secret, ancient, and profound science of alchemy, the royal or sacerdotal art, also called the hermetic philosophy, conceals in esoteric texts and enigmatic emblems, the means of penetrating the very secrets of nature, life and death, of unity, eternity and infinity."*¹-- S. K. De Rolla

There was a time when alchemy was considered the highest and most sublime of all science and philosophy but later fell into disrepute in the hands of quacks and charlatans preying upon the credulous public. In some countries, during the Middle Ages, the practice of alchemy was punishable by death. The alchemical teachings were then largely hidden in obscure symbolism and mystical stories so that today, researchers are met with great problems in deciphering and interpreting its doctrine.

Many interpreters class alchemy as a system of spiritual allegory, using the symbolism of chemistry to conceal psychological and spiritual truths. Others classify it purely as a primitive chemical science. In the writings of the alchemists themselves it is a universal science applicable to all levels of being, physical as well as spiritual. The noted psychologist C. J. Jung recognized something of this when he wrote: *"it would be an unpardonable depreciation of*

value if we were to accept the current view, and reduce the spiritual striving of the alchemists to the level of the retort and the smelting furnace. Certainly this aspect belonged to it; it represented the tentative beginnings of exact chemistry. But it also had a spiritual side which has never yet been given its true value, and which from the psychological standpoint must not be underestimated.”²

To begin then, we must realize that alchemy is not a dead science. Though obscure and rarely heard of these days, progress in understanding the ways and means of earlier alchemists is being made. In this introductory material we must also realize that only the most brief and superficial views of the science can be given in a paper of this size because of the vastness of the subject both theoretical and practical. This would of course be the same with any other “officially recognized” science but this is particularly the case with alchemy where we are dealing not only with physical laws and materials, but also with underlying spiritual concepts.

Finally, we must realize the necessity for the extensive use of analogy and correspondence in discussing alchemical theory. This is because many of the concepts deal with abstractions such as soul, spirit, perfection, et cetera, and require an intuitional apprehension. In such cases, language can be a great barrier limiting the full impact of a concept to a narrowness evoked by the choice of words.

“Existence is beyond the power of words to define: terms may be used but are none of them absolute. In the beginning of heaven and earth there were no words, words came out of the womb of matter; and whether a man dispassionately sees to the core of life or passionately sees the surface, the core and the surface are essentially the same, words making them seem different only to express appearance. If name be needed, wonder names them both: from wonder into wonder existence opens.” -- Lao Tzu

HERMETIC MYSTERIES

According to alchemical theory, all that is, was, or ever will be, is derived from one ultimate source. Call it the All, the Absolute, Deity by any one of countless names, peoples of all nations and times have recognized the ultimate unity of all creation. Even today modern science teaches the essential equivalency of matter and energy. This is no different than what the ancients intuitively perceived as the duality inherent in the unity. Matter and energy, spirit and substance, yin and yang -- the two contending forces marking the extremes of expression or poles of one unified whole. The interaction or friction between these two poles was believed to give rise to a third form of expression, that of awareness or consciousness. So we could think of this as a vibration between the spirit aspect and the matter aspect inherent in the unity generating a “fire by friction.” This fire being the light of consciousness or the soul aspect. In more modern terms we could think of Einstein's $E=Mc^2$ where energy (spirit) is related to mass (matter) through a time/space relationship (consciousness). These constituted the so-called “three essentials” of the alchemist, the body, soul, and spirit or the “trinity manifest as one.”

The soul or consciousness was considered to be the mediator between the two aspects of

spirit and matter and thus, being in the place of balance, could partake of “the best of both worlds.” The development of consciousness and its ability to express (spirit) through more and more refined vehicles (matter) is the way of evolution. This is the sacred middle way or path to enlightenment of both Eastern and Western philosophies.

By understanding and applying natural laws, the alchemists believed that it was possible to increase the vibratory rate or interaction between spirit and matter thus “fanning the fire” of consciousness and expanding it until one was literally enlightened. The profound depths of alchemical teachings concerning these natural laws are astounding and complex beyond the scope of this paper. The Emerald Table of Hermes given as a frontispiece to this paper outlines many of these alchemical concepts.

This short piece of alchemical literature, the origin of which is lost in history, is one of the most widely quoted texts dealing with alchemical thought, and is worthy of prolonged contemplation.

Here we limit our discussion to some of the alchemical ideas as they relate to the laboratory operation of metal acetate decomposition.

LABORATORY ALCHEMY

*“Now, if you do not understand the use of the cabalists and the old astronomers, you are not born by God for the spagyric art, or chosen by nature for the work of Vulcan, or created to open your mouth concerning alchemical arts.”*³ -- Paracelsus

The goal of the alchemist was to assist natural laws in evolving a more perfect vehicle for the expression of the life force and because of the essential unity of all, this ultimately meant “the divine made manifest.” From this arises much of the controversy as to whether alchemy is a spiritual doctrine or a chemical operation. To the alchemist there is no distinction, the laws governing the spirit have their reflections in material laws and by studying one realm we could better understand the other.

Alchemical operations in the laboratory, then, served many purposes. As demonstrations of natural laws, chemical operations such as distillation were considered to have “inner” level correspondences. In other words, the manual operation at the physical was a reflection of changes occurring at mental and spiritual levels of being. Thus the substances manipulated by the alchemist in his laboratory were considered to be vehicles of higher principles.

*“Everything which is generated and produced of its elements is divided into three, namely, into salt, sulfur, mercury... learn the form which is peculiar to these three. One is liquor, and this is the form of mercury; one is oilness which is the form of sulfur; one is alcali, and this is from salt.”*⁴ -- Paracelsus

These three essentials we mentioned earlier were thought to assume material vehicles peculiar to their character. The spiritual essence or so-called mercury assumed the form of a volatile liquid. The soul qualities or character assumed the form of an oil and was called the

sulfur. The body or salt assumed the solid form.

Perhaps a simple example will help clarify these ideas. Let's consider a plant growing in the wild. It does not matter which plant; this example applies to the vegetable world in general. To the alchemist, the plant is a living, evolving entity possessing a body, a soul, and a spirit. We now take the plant into the alchemist's laboratory where we are able to assist nature's operations in a controlled manner. First we can obtain an essential oil from the plant by steam, or pressure, or extraction. This is the alchemical sulfur or soul of the plant. This oil was considered to be the vehicle of consciousness within the plant and as expressing the plant's character. The plant residue is left to putrefy and ferment, analogous to its death wherein the spirit is released from the body. This spirit is distilled from the fermented mixture and represented the vehicle of the alchemical mercury or life force. Today we call this volatile liquid alcohol though it is still widely known as "spirit". The soul and spirit removed from the plant, there now remains only the dead and impure body.

By the various operations of calcination, sublimation, and distillation, the three separated "essentials" were purified. The key to this purification was held to be the proper regulation of fire. Thus the volatile spirit and the oil or sulfur could be purified by redistillation and the body of the plant reduced to a pure white mineral salt by calcination. We must recall that as these operations occur and act upon these outer material agents, analogous separations and purifications were said to be taking place on inner spiritual levels by reflection.

Now, in a kind of resurrection, the purified spirit and soul were reunited in the purified body to create a substance which expressed the true essence and character of the original plant in a highly augmented and exalted form. Substances such as this constituted the various elixirs and medicines used by the alchemist to assist his own development as a channel of the divine life force.

*"Our second way of preparing this our vegetable elixir is by a right manipulation of a plant of the noblest degree, standing by itself, or supported by others: after the preparation of which and its putrefaction, reduction into an oil, separation of the three principles, with their purification, union, and spiritualization, the whole is to be turned into a spiritual ever living fountain, renewing every plant that shall be plunged in it."*⁵ -- Baro Urbigerus

Following similar lines of operation, the alchemists describe powerful medicines derived from the mineral realm. As in the case of plants, metals were considered to be living entities and those metals which were obtained from native sources were more highly esteemed than smelted metals. In the process of smelting, the volatile spirit and oil were mostly lost because of the excessive heat. This left the metal in a depleted state and thus not as useful from the alchemical standpoint.

*"Because in the exaltation of the double vapor, the commotion has been so great and searching, that the spirit, or agent, has evaporated, as occurs, with very close similarity, in the fusion of metals."*⁶

Thus a mineral's body, soul, and spirit counterparts or reflections in matter could be manipulated and as in our plant example, separated, purified and brought into the service of

the alchemist on exalted as well as physical levels.

*“The body of metals are domiciles of their spirits... when their terrestrial substance is by degrees made thin, extended, and purified... the life and fire hitherto lying dormant is excited and made to appear. For the life which dwells in the metals is laid, as it were, asleep... nor can it exert its power or show itself unless the bodies be first dissolved and turned into their radical source. Being brought to this degree at length by abundance of their internal light they communicate their tingeing properties to other imperfect bodies...”*⁷

One such method for preparing mineral and metallic derivatives, and the subject of study in this paper, involved the thermal decomposition of metal acetates.

INTRODUCTION TO THE EXPERIMENTAL

The origin of the method for metal acetate decomposition is lost to historians but the process appears fairly well known at the beginning of the seventeenth century.^{8,9}

Though it is often referred to in veiled terms, the process involving dry distillation of metal acetates is one of the better-documented operations in alchemical literature. In reviewing and comparing various alchemical tracts dealing with acetate distillation, Dr. C. A. Becker in 1867 gives a clear account saying:

*“Different descriptions in that regard combined, provide the following information: The secret material for the philosopher’s stone which has been hidden behind many names is calcined and dissolved in distilled wine vinegar. The solution is evaporated until it takes a thickness of a gum. From that, first you distill a tasteless water with gentle temperature; when white vapors appear another recipient is attached and the aqua ardens is obtained. This water has an extremely strong taste and at times a stinking smell, therefore it is called aqua foetens, or menstruum foetens. If the distillation continues at higher temperatures, a red vapor and finally red drops appear. You let the temperature gradually die down and keep the distillate in a tightly closed glass so that the volatile spirit may not disappear.”*¹⁰

Similarly, at the latter part of the sixteenth century the alchemist, J. Holland in his Work of Saturn, wrote:

*“The lead sugar (lead acetate), completely purified, is distilled first with gentle and later with stronger fire until the material passes over red as blood and thick as oil and sweet like sugar with a heavenly smell.”*¹¹

In each case, the product of this distillation or decomposition was highly esteemed by the alchemists. Sir George Ripley (1415-1490)¹² called it the blessed liquor or *menstruum foetens* and described it as containing three substances, the *aqua ardens* which burns like wine spirits, a thick white water called *lac virginum*, and a blood red oil called *sanguis leonis* or blood of the green lion. He further claims that the key to all chemistry lies hidden in this *menstruum foetens*.

¹³

J. Holland (about 1600) speaking of the distillate from lead acetate, which he calls the water of paradise, writes: "...there is in it a red and a yellow sulfur. In it is mercury, as may be seen, for mercury is extracted out of Saturn (lead) in a short time and with little labor." ¹⁴

We must recall that the terms sulfur and mercury refer to philosophical concepts and their related physical forms discussed earlier.

In 1685, Johannes Seger Weidenfeld, a student of the works of earlier alchemists, published a work dealing largely with the distillates of metal acetates, which he calls The Secret Wine Spirit of the Adepts. Concerning this he is quoted to say: "*The spiritus vini philosophici, or spiritus vini Lulliani is the basis, the beginning and the end of all solvents in the secret chemistry.*"¹⁵ With the rapid progress of chemical science during the 17th and 18th centuries, many alchemical concepts faded into obscurity in favor of "more sophisticated" ideas.

*"There was so little experimentation on the distillation of acetates and acetone in the eighteenth century that some chemists of the early nineteenth century were more or less unaware of previously obtained information. As a matter of fact, as late as 1840 Becker was urging physicians and pharmacists to regain possession of this extremely effective medicine."*¹⁶

The most volatile fraction of the metal acetate distillate (and incidentally one of the major components) received the greatest attention by chemists. This clear, volatile liquid was known to the alchemists as the spirit of the metal and by the mid-1800's its chemical formula and structure had been determined. Now called acetone, this solvent was beginning to find a wide industrial application and in 1888 the first large scale production in the United States was started.¹⁷ From that time to the present, various studies have been conducted in order to discover possibilities for increasing acetone yield,¹⁸ proposing reaction mechanisms^{19, 20, 21} and preparing specialty compounds by similar procedures applied to different starting materials.²² Knowledge of the alchemical roots of the process has practically disappeared. Modern petroleum technology has made acetone production much more efficient and the process of acetate distillation obsolete.

The oily fraction of the distillate seems very neglected in relatively recent literature, indeed it is often not mentioned at all. When it is mentioned, it is usually referred to as acetone oils, tarry oils, or just oil residue, and considered an undesirable by-product of the reaction. It has been reported as late as 1961: "*There is virtually no literature devoted to a scrupulous study of composition and yield of reaction products obtained from the dry distillation of calcium or barium carboxylates.*"²³

One of the major objectives of this study was to shed more light on the nature and composition of these oils. What are they chemically? Are they the same or do different metal acetates produce different oils? What use are they? These are some of the questions which prompted the study and to which the main emphasis of the experimental section has been devoted.

EXPERIMENTAL

Isolation of the metals

Five metal acetates were chosen for investigation. These are listed in Table 5-1. As indicated previously, native ores were preferred as starting materials. Native ores nearly always have associated with them other minerals so the first steps of the alchemical process, those of separation and purification, had to be taken. This required some analytical knowledge of the ores which was accomplished mainly by wet-chemical techniques and atomic absorption spectroscopy.

In the case of iron acetate, the raw material was pyrite, a sulfide of iron. Associated with the iron, however, were silica, copper, manganese, lead, and several metals in trace amounts. The pyrite was fairly weathered and it was found that simply by washing the powdered ore with distilled water, filtering and saving the liquid which was then evaporated, left large crystals of ferrous sulfate, which in turn were purified by recrystallizing several times in fresh water. Care in handling the ferrous sulfate was required throughout because of its instability in air. Limiting its exposure to air and heat as much as possible kept decomposition to a minimum. In this way, a fairly pure source of iron was obtained using little more than pure water.

For lead acetate, the starting material was galena which is a sulfide of lead. Analysis of the ore showed alumina, silica, copper, silver, and bismuth as impurities. Roasting the ore proved to be long and tedious, the object being to drive off the sulfur and to produce lead oxide which is easily converted to the acetate. Many alchemists avoided the use of acids or corrosives in various operations, others considered their use highly valuable especially as a means of "calcination with water". In the present case, it was decided to speed things up and dissolve the galena in nitric acid. The resulting solution then crystallized by evaporation and the lead nitrate so produced was recrystallized several times in water. This resulted in a fairly pure and easily handled lead source for later conversion to the acetate.

Sodium bicarbonate, the starting material chosen for preparation of sodium acetate, was produced by the Solvay Process (as is most commercially available sodium bicarbonate). It was quite pure and being readily soluble in acetic acid, offered no problems in preparation.

The native source of potassium chosen for this work was the crude tartar which is a by-product of the wine making industry. This is a relatively pure material being mainly potassium bitartrate and tartaric acid with various organic inclusions in the crystalline crusts which form in barrels of wine. This material was repeatedly calcined over a medium heat, dissolved in water, crystallized, and again calcined. After about six cycles of this process, a very clean, white, potassium carbonate was obtained.

Finally, the sulfide ore of zinc called sphalerite was obtained as a zinc source. This ore was found to be relatively pure, the major impurity being iron. By dissolving the powdered ore in hydrochloric acid, (still contaminated with iron, which, as will be seen, was removed later) the product was easily handled.

In each case the final conversion to an acetate was made by first producing the metal

The Thermal Decomposition of Metal Acetates

carbonate and subsequently dissolving this in acetic acid and recrystallizing. Apart from being perhaps the easiest reaction pathway from a chemical standpoint, we could think of this as an interesting analogy to the alchemical process. In this, the strictly mineral form (sulfide, oxide, silicate, etc.) is in a sense “evolved” stepwise by first gaining experience as a metal carbonate, a sort of transition vehicle between inorganic and organic, then making the jump into the world of carbon compounds by conversion to an organometallic compound, the acetate. This follows along the lines of successive refinement of the body or vehicle discussed earlier.

The ferrous sulfate once isolated was dissolved in water, then a concentrated solution of sodium carbonate was added in order to precipitate the iron as a carbonate. This was then washed very well with water and dissolved in acetic acid; on evaporating the liquid, a fine red-brown basic iron acetate remained.

In a similar manner, the lead nitrate obtained was dissolved in water then precipitated with sodium carbonate solution as white lead carbonate. After thoroughly washing with fresh water, this was dissolved in acetic acid and recrystallized several times. This resulted in large, brilliant crystals of lead acetate, which were protected from the air until later use.

Both sodium and potassium were already isolated as purified carbonates so it was only necessary to dissolve them in acetic acid and recrystallize several times in order to obtain these as acetates.

The zinc chloride solution was precipitated as the carbonate, again using the sodium carbonate solution. This was washed in water then dissolved in acetic acid. The iron impurity had largely followed through to this point and it was necessary to recrystallize using ethanol. Because of the wide difference of solubility in ethanol between zinc acetate and iron acetate, the iron impurity was almost totally removed leaving long clear crystals of zinc acetate.

At this point, the five metal acetates, being isolated and relatively pure, were ready for the actual decomposition process by dry distillation.

Table 5-1

Compound Name	Chemical Formula	Source
Iron Acetate	$\text{Fe}(\text{OH})(\text{CH}_3\text{COO})_2$	Pyrite (FeS)
Lead Acetate	$\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$	Galena (PbS)
Sodium Acetate	$\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$	Sodium Bicarbonate (NaHCO_3)
Potassium Acetate	$\text{CH}_3\text{COOK} \cdot \text{H}_2\text{O}$	Crude Tartar ($\text{KHC}_4\text{H}_4\text{O}_6$)
Zinc Acetate	$\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$	Sphalerite (ZnS)

DISTILLATION OF THE ACETATES

It is during this stage that the actual decomposition of the prepared metal acetates takes place. Today of course we have a great technological advantage over the early alchemists as far as temperature control (heating and extreme cooling), choice of vessel material, and ability to seal the apparatus are concerned. However; we must constantly bear in mind that from the alchemical viewpoint, the "outer" operation was only one part of the process which had analogous operations occurring at all levels of being. In other words, changes occurring in the physical, material had corresponding changes reflected in its spiritual counterpart, and vice versa. "As above, so below..."

The distillation apparatus used in this study was designed after an illustration in French's, *The Art of Distillation*²⁴ (see Figure 5-1). The vessel labeled *A* is called the still pot and contains the metal acetate to be distilled. As can be seen by its place in the fire, this is the actual scene of the thermal decomposition. The vessel labeled *B*, is here termed the first receiver. This catches the less volatile distillates. Vessel *C* consists of a barrel of ice water which surrounds a second receiver in the distillation train. This vessel collects the more volatile distillates and is called the cold trap.

The corresponding labels can be seen on the apparatus used here (see Figure 5-2).

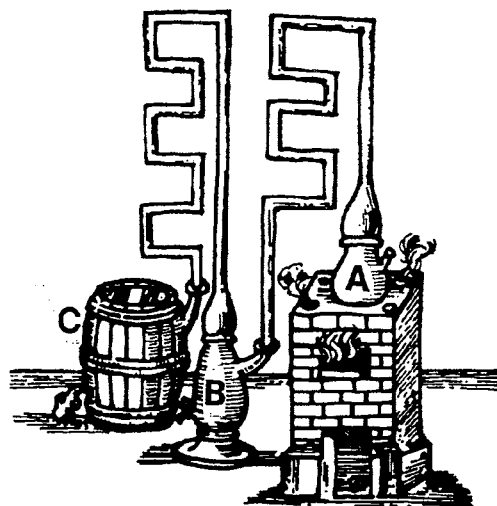
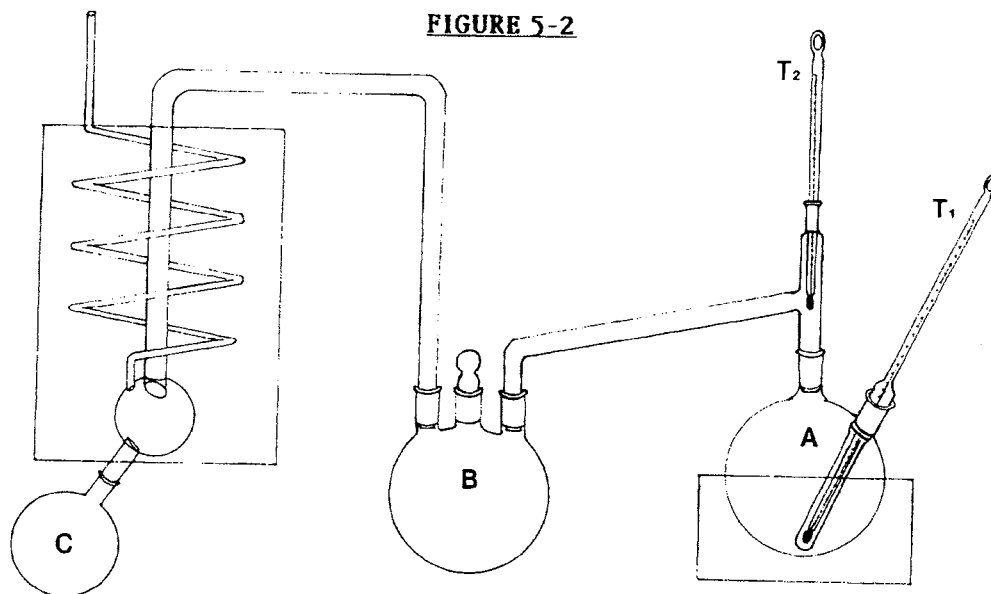
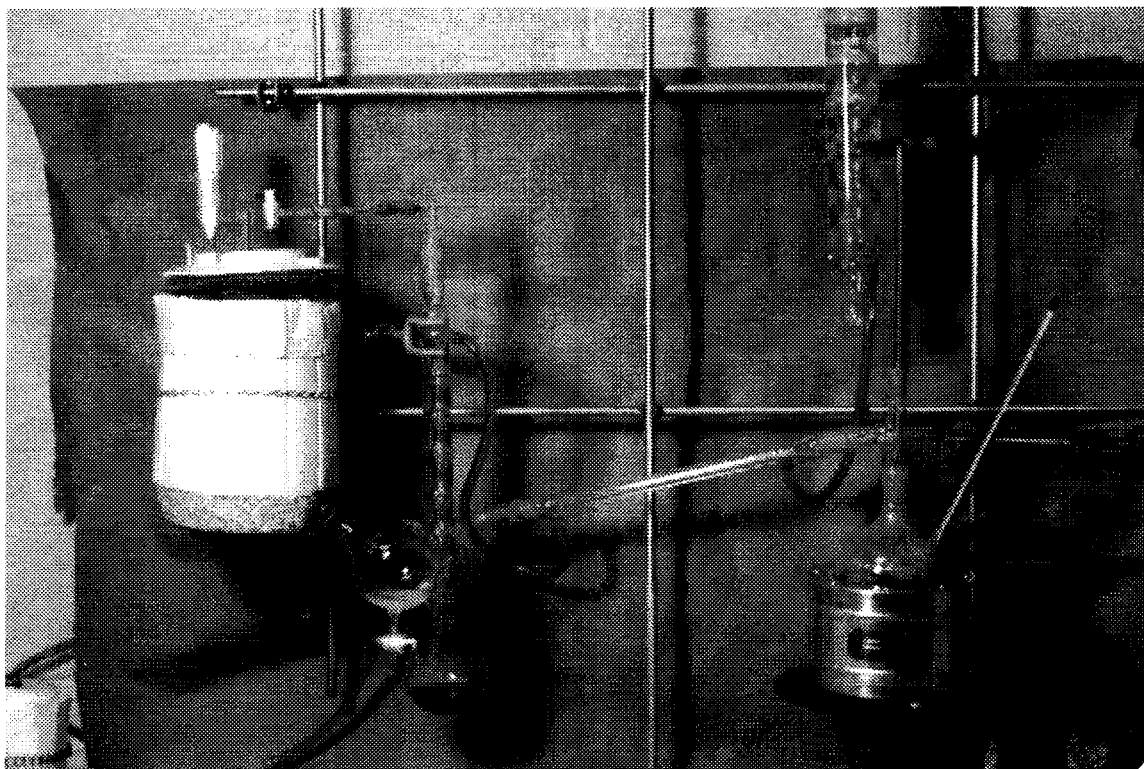


Figure 5 - 1



Heavy Pyrex glass was used throughout. The heating mantle was a Glas-Col series STM mantle with a 650° C maximum. The Still Pot (A) was equipped with a thermometer well (labeled T1) and a distillation head thermometer (T2) allowing temperature observation at these two points. The cold trap cooling mixture consisted of dry ice and acetone. This effectively maintained a temperature of almost -80° C. All of the 24/40 joints not directly exposed to the heat were assembled (without lubricant) then sealed externally by brushing on several coats of molten beeswax.



In a typical distillation run, 1/4 to 1/3 of the still pot was filled with the granulated metal acetate (about 20 mesh). After sealing the apparatus with beeswax, the heat was gradually increased while temperature vs. time measurements and observations were made (see pages 13 to 17). As the temperature increased, the residual water, acetic acid, and some crystal water distilled over first. This first portion of distillate was called “the phlegm” by alchemists. When this stopped distilling over, there was a sharp increase in temperature and it was at this point the alchemist would change the receiver with a fresh one. Soon, the decomposition temperature is reached and the apparatus fills with a thick, heavy, white vapor while an oily liquid distills over. Alchemically, this is the point where the metal “gives up the ghost” and releases the vehicles of soul and spirit from the gross body. In the cold trap collects a clear volatile liquid, “the spirit”, the new seat or focus of the metal's life essence. The oil collects in the first receiver and embodies the soul or character of the metal. In the still pot remains the dead and impure body, the gross physical vehicle. Table 5-2 indicates some typical yields of the various distillation products.

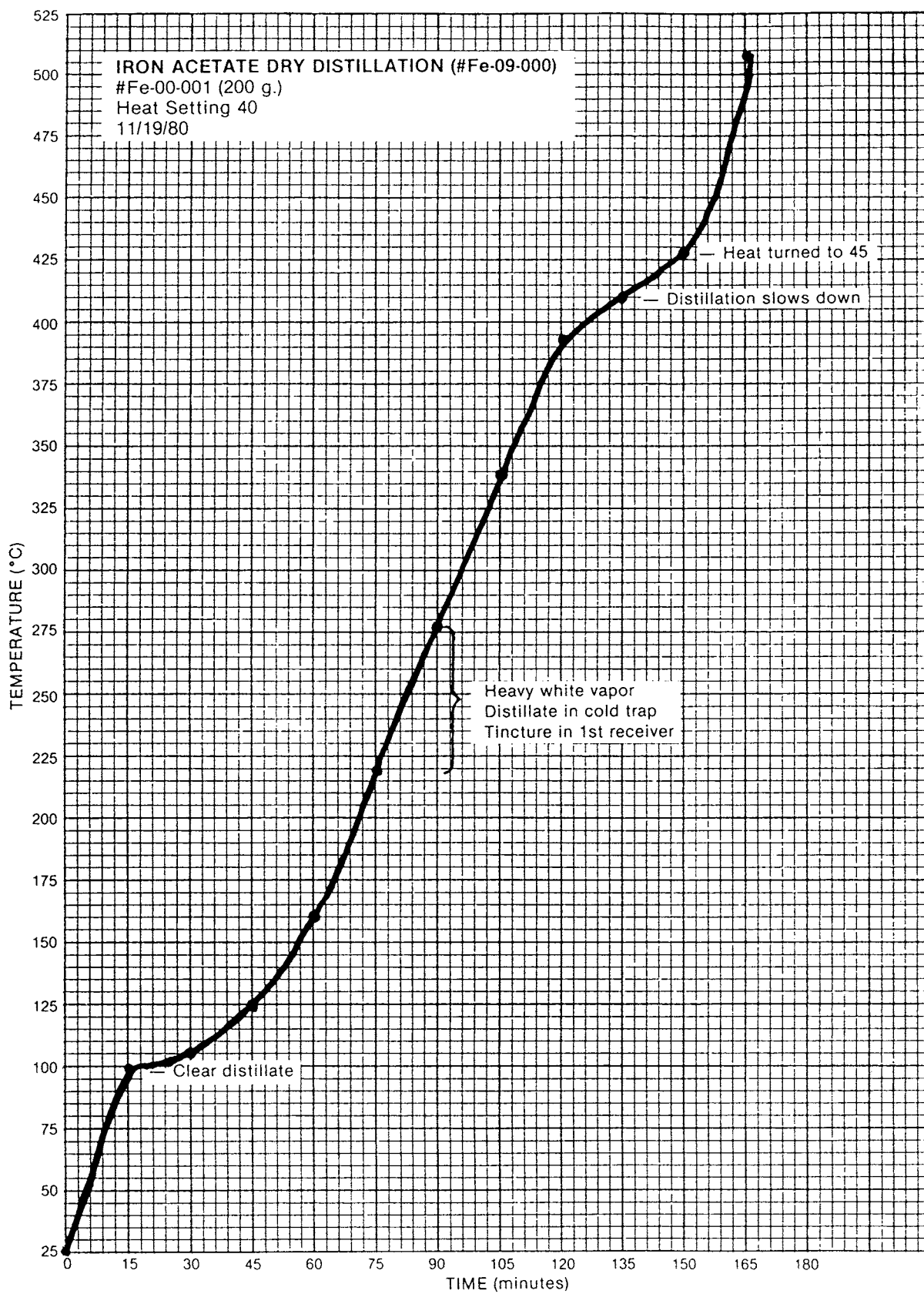
TABLE 5-2²⁵

Metal Acetate Distilled	K	Pb	Na
Residue			
• metal	---	97.26	---
• metal oxide	0.75	1.53	---
• metal carbonate	95.52	1.09	99.62
• carbon	3.73	0.12	0.39
Distillate			
• acetone	39.0	90.0	53.0
• acetic acid	0.16	2.68	0.12
• water	8.84	7.32	9.88
• oil	52.0	---	37.0
Gaseous Products			
• carbon dioxide	15.4	98.4	52.3
• Sk. W.	7.8	---	11.1
• carbon monoxide	28.2	---	7.6
• hydrogen	16.1	1.6	8.6
• methane	32.5	---	44.9

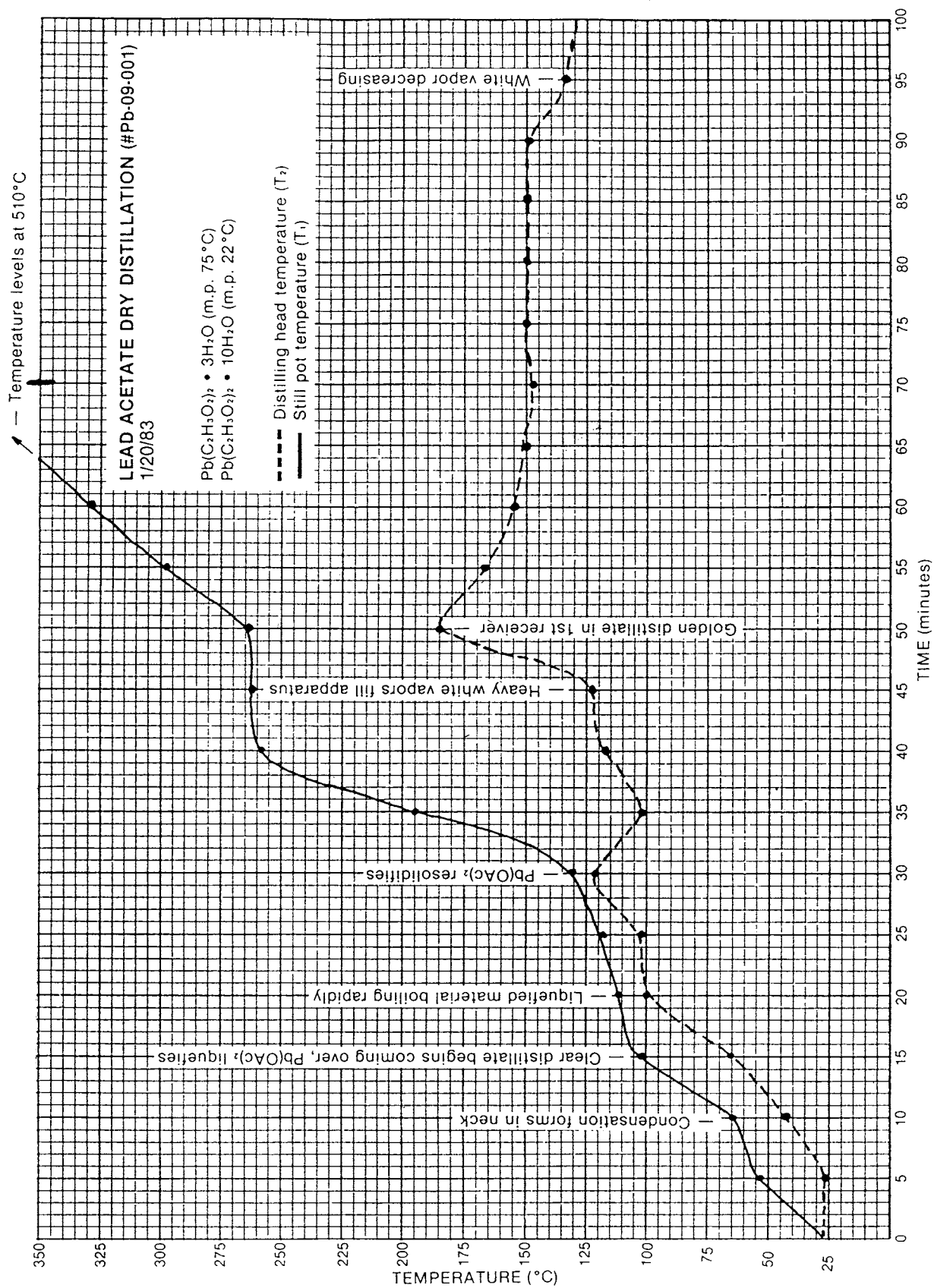
NOTE: all values are in percent by weight of product.

Each metal acetate distilled seemed to have its own characteristics as can largely be seen in the time vs. temperature curves. Some additional observations may be usefully inserted here. The "sweet fragrance", noted in the introductory comments, was noted to be characteristic of each run regardless of which metal acetate was used.

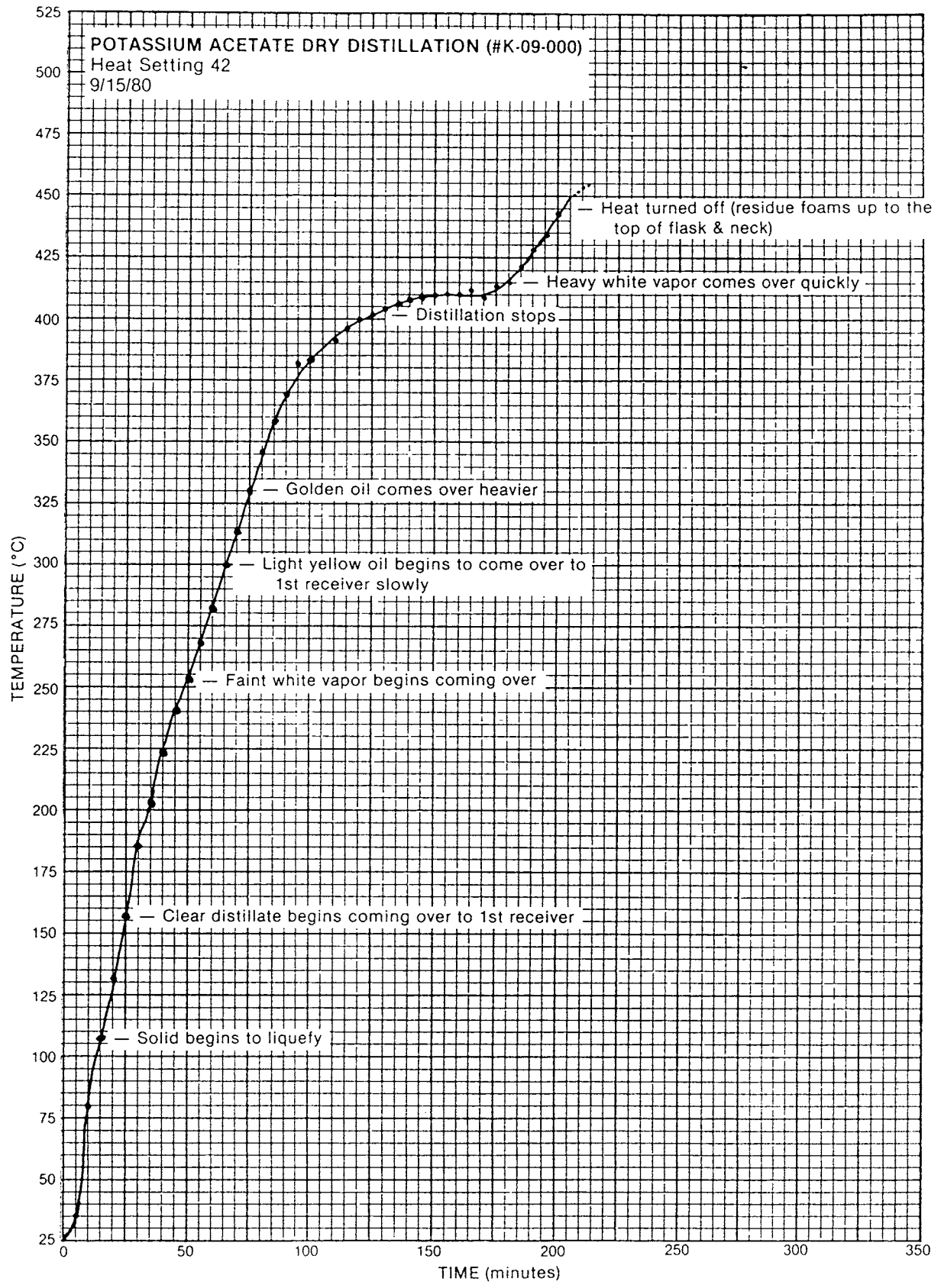
The Thermal Decomposition of Metal Acetates



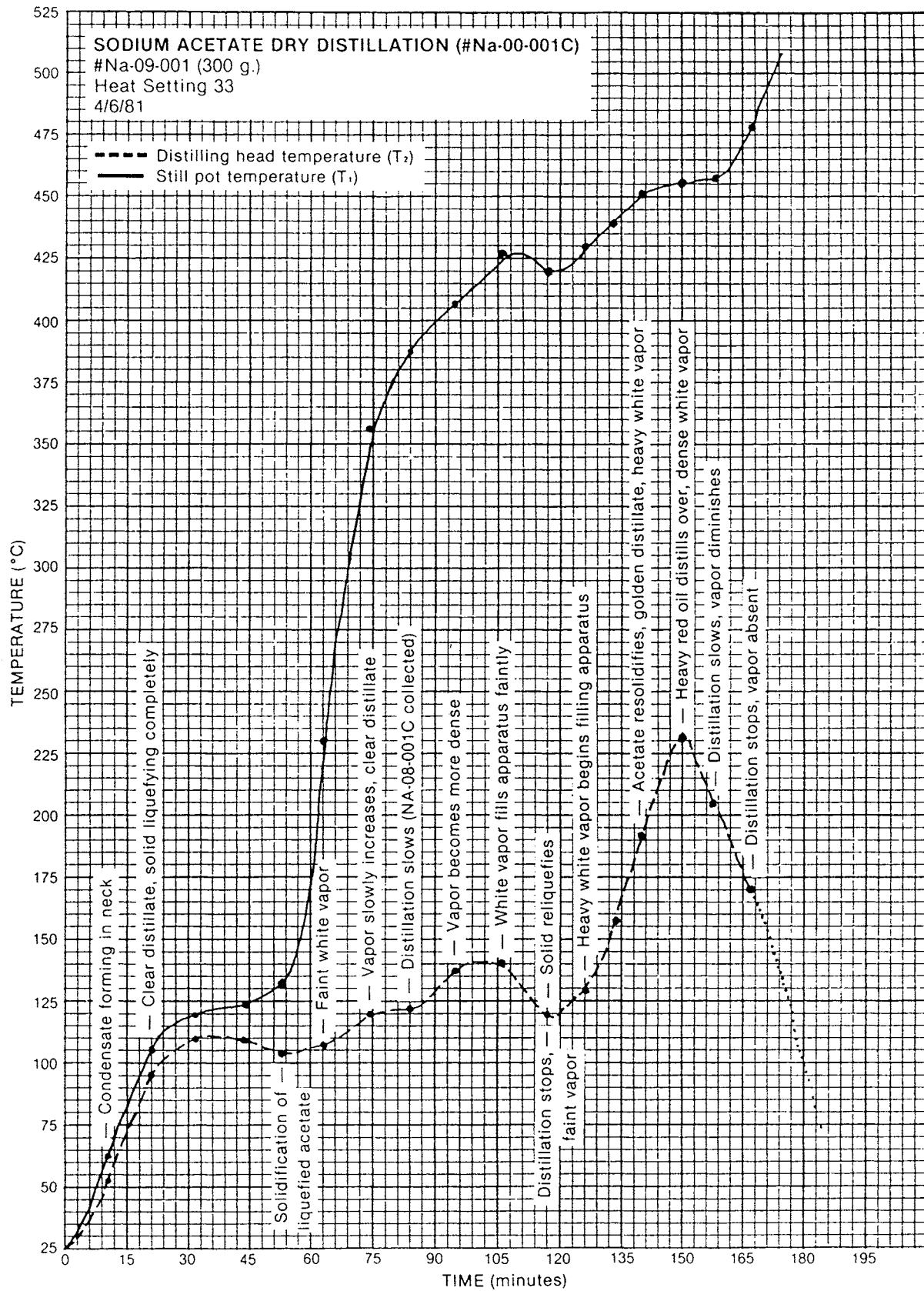
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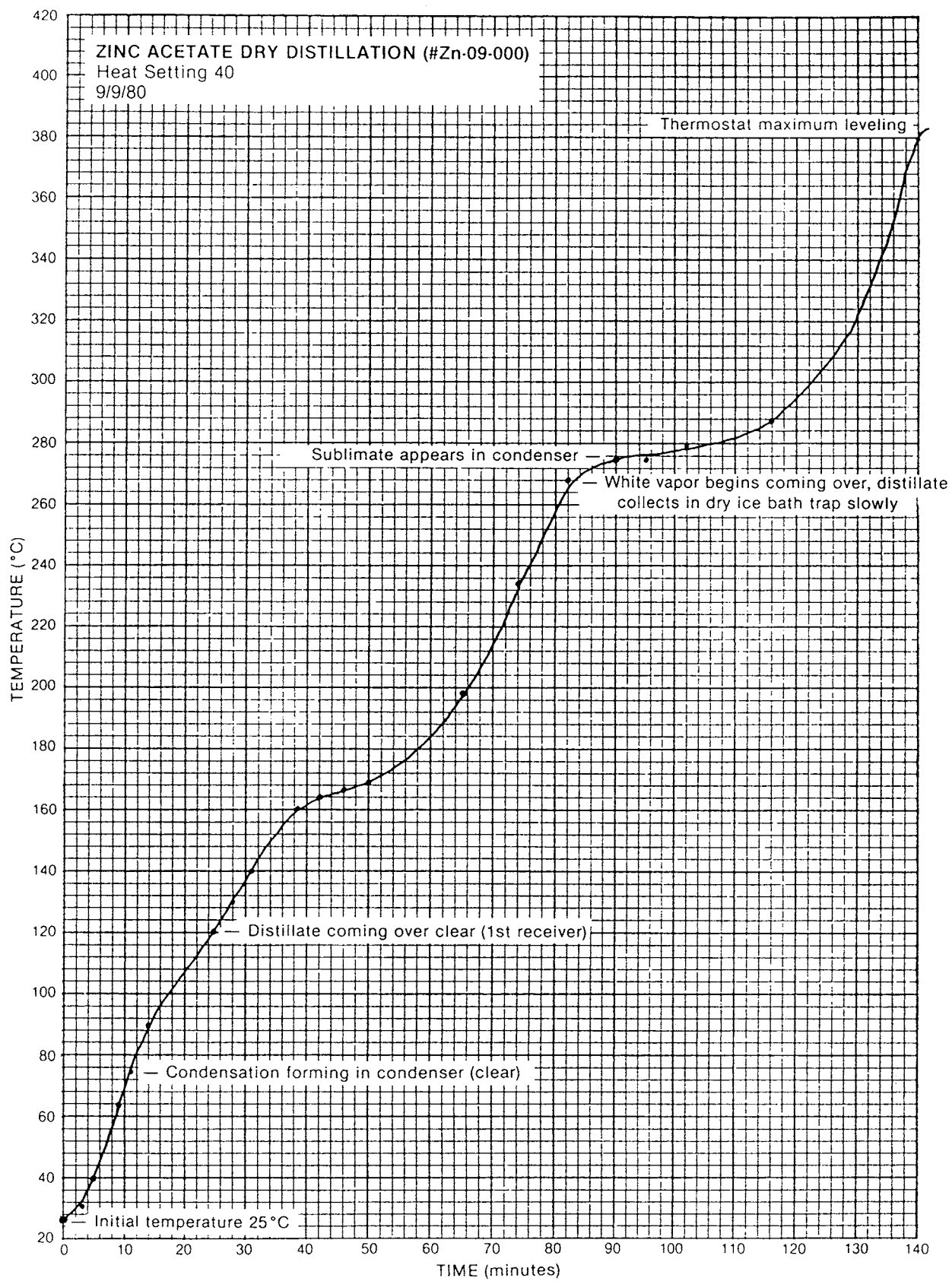
The Thermal Decomposition of Metal Acetates



The Thermal Decomposition of Metal Acetates



The Thermal Decomposition of Metal Acetates



During the course of distillation many of the acetates would melt then later resolidify as decomposition began or states of hydration changed. In the case of potassium acetate a stage was reached where a great deal of foaming occurred finally necessitating the termination of the distillation to avoid its foaming over. In order to avoid this in future experiments, we may take advantage of a distillation method developed by the early chemists. This consisted of saturating pieces of brick or broken pottery with the acetate solution, drying, then distilling from the brick.

Finally, the distillation residue from lead acetate is composed mainly of lead metal reduced during the reaction. This lead is so finely dispersed that on removal from the reaction flask, it quickly oxidizes and can become red-hot.

PRELIMINARY SEPARATIONS

In describing the products obtained from the distillation, we mentioned four. First the so-called “phlegm” (residual moisture and acetic acid, etc., then the first receiver distillate, the cold trap distillate, and of course the still-pot residue. These formed very convenient divisions in the subsequent analysis. There are apparatus parameters, heating rates, etc., that can alter the exact division and a whole study on optimum conditions similar to the work of Ardagh et al ²⁶ could lead to a future project.

Table 5-3 indicates some values obtained in the distillation of sodium acetate. The still-pot residues were found to contain the metal oxide, carbonate, or in the case of lead, the reduced metal itself, along with hard carbon inclusions. The “phlegm” consisted mainly of water, and acetic acid, with a trace of acetone and phenol (indicated by qualitative analysis and acid/base titration). As the decomposition begins, the apparatus fills with heavy white vapor. The first receiver slowly fills with a clear red liquid like deep burgundy wine. The cold trap fills slowly with a clear or faintly tinted liquid, which seems to be very volatile. Because of the apparatus design, these two liquids (i.e. first receiver distillate and cold trap distillate) are obtained separately. The early alchemists generally collected them as one liquid by employing very large vessels sealed to prevent any gases from escaping.

TABLE 5-3: Preliminary separation of sodium acetate distillates.

Weight Distilled (grams)	Total Distillate (ml)	1 st Receiver “phlegm” (ml)	“oil” (ml)	Cold Trap (ml)
300	168	124	40	4
300	175	120	50	5
400	219	162	50	7
400	231	158	65	8
400	229	148	74	7
400	225	156	61	8
500	229	152	68	9

The combined distillate is what Becker termed the *menstruum foetens*.²⁷ The most volatile part of this distillate, (that collected in the cold trap) proved to be mainly acetone containing traces of moisture and volatiles from the first receiver. The apparent high volatility when this "spirit" is first obtained can be explained by the out-gassing of the methane, ethane, and ketene dissolved in the condensed acetone.²⁸ By far the most complex portion of distillate was that obtained in the first receiver. This wine-red liquid had a penetrating acetous odor and acetone odor. Preliminary investigation using thin layer chromatography indicated these liquids contained up to ten different components. In each case, the first receiver distillate was re-distilled using a fractionating column to effect separation. The first fraction to be removed was acetone and constituted as much as 60% of the volume. After this was collected, a small amount of water and acetic acid were detected. The remaining thick, blood-red oil was then dissolved in petroleum ether. This caused a separation of two fractions, a red oil which was totally soluble in the petroleum ether, and a residual oil which was totally soluble in acetone.

Thin layer chromatography showed these oils to contain many of the same compounds, but also indicated that different metal acetates produced compounds peculiar to themselves. The relatively small yield of these oils made further separation by fractional distillation, solvent extraction, or thin layer chromatography very difficult and tedious. Thus it was decided to take advantage of higher technology and have the various oils analyzed by gas chromatography and mass spectrometry.

GAS CHROMATOGRAPHY AND MASS SPECTROMETRY

The gas chromatography and mass spectrometry analyses were accomplished by Western Analytical Laboratories of Salt Lake City, Utah.

Table 5-4 lists the various samples submitted for analysis and their code designations. Briefly, in this analysis a small portion of the oil sample was injected into a long column being swept by an inert gas and heated on a computer-programmed schedule. As the sample passed through the column, a separation of its component substances occurred based on volatility and chemical affinities with the column packing. Each fraction of the oil thus emerged from the column in a relatively pure state and was then passed into a chamber where its molecules were electrically ionized. These charged fragments were then accelerated in a known electrical field and then subjected to a known magnetic field, which altered the trajectories of the various accelerated fragments according to their mass. The time integrated sums of the various mass fragments, combined with the retention time and boiling data from the gas chromatography were compared by computers with the National Bureau of Standards Compound Library in order to arrive at the most probable identity of the compound.

In the data presented then, each sample covers one page. First a graphical representation of the oil's make-up showing the number of peaks (and thus number of components) and their relative proportions. Below the graph is a tabulation of the data obtained. Beginning on the left and reading across, the headings indicate: peak number, its graphical position (start,

middle, and end), the area of the peak and its height (indicating its relative proportion), the entry number in the NBS Library for the proposed compound identity, how well it fits the NBS data (1000 = perfect fit) the purity of the fraction, how well the mirror image or reverse fit of the curve fits the NRS data, compound molecular weight and boiling temperature.

TABLE 5-4: Sample designations for G. C./Mass Spec. analysis

acetate source	petroleum ether soluble fraction	acetone soluble fraction	code number
Iron	x		Fe-08-000B
Iron		x	Fe-08-000D
Lead	x		Pb-08-001C
Lead		x	Pb-08-001D
Sodium	x		Na-08-001C
Sodium		x	Na-08-001E
Sodium	"the phlegm"		Na-09-002A
Sodium	<i>Menstruum foetens</i>		Na-09-002B
Potassium	x (total solubility)		K-08-000A1
Zinc	x		Zn-08-000A2
Zinc		x	Zn-08-000A2

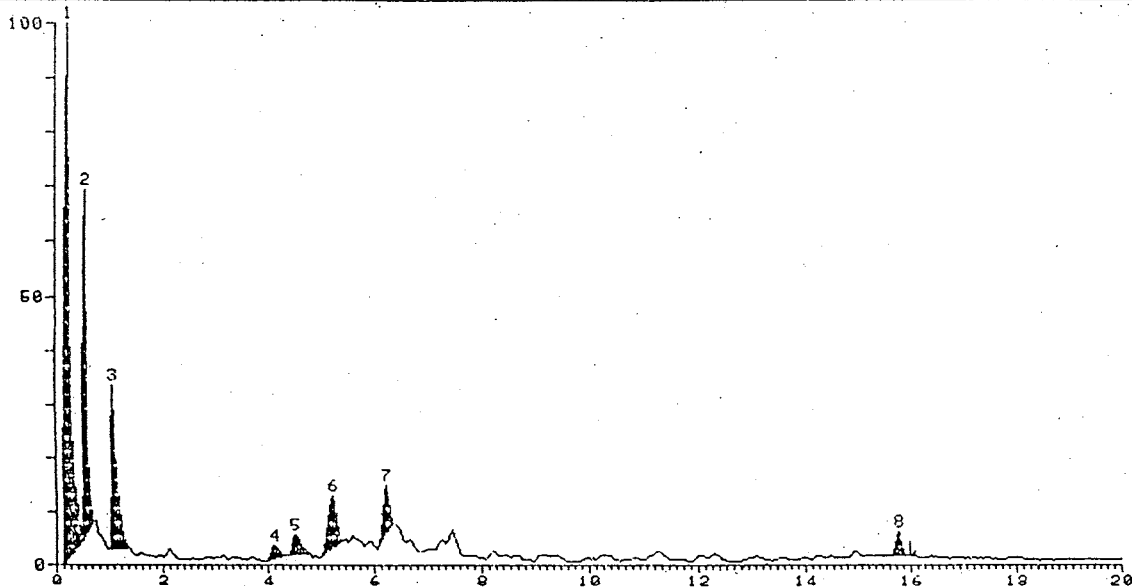
The Thermal Decomposition of Metal Acetates

IRON — petroleum ether soluble fraction — Fe-08-000B
 TABULATION OF RESULTS FROM THE NBS LIBRARY

JJ129 **47096, FE-08-000B
 BASE 1,321,902 ZERO 0
 TOTAL ION CHROMATOGRAM

SCALE 1.00

** MAR 8, 1983 9:07PM

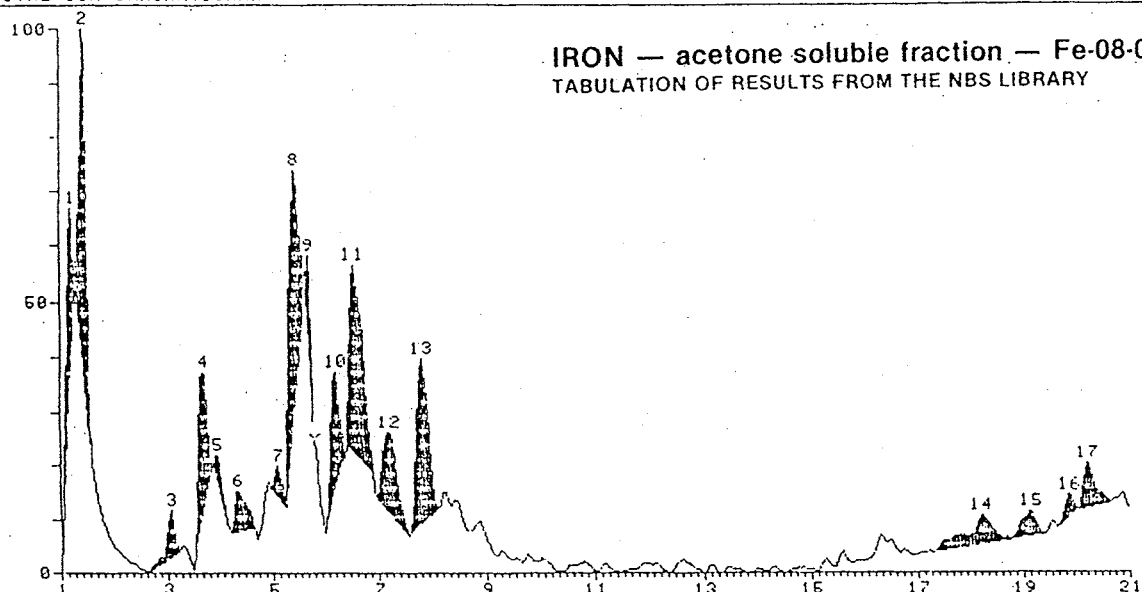


PEAK	START	TOP	END	PEAK AREA	PEAK HEIGHT	ENTRY	FIT	PUR	RFIT	MOL. WEIGHT	TEMP (°C)
1	.14	.24	.48	6844336	1303444	2505	886	687	694	118	57
COMPOUND: Ethanol, 1-methoxy-, acetate											
2	.46	.58	.70	2677312	828864	5759	791	754	780	146	64
COMPOUND: Ketone, methyl 2-methyl-1,3-oxothiolan-2-yl											
3	1.00	1.07	1.35	2081425	404398	1060	899	867	904	98	71
COMPOUND: Cyclopropane, 1,1,2,2-tetramethyl-											
4	3.97	4.15	4.35	272200	33817	4009	957	603	617	132	109
COMPOUND: Benzene, (2-propynyloxy)-											
5	4.33	4.51	4.77	433096	47566	4679	944	586	607	138	115
COMPOUND: 2-Cyclohexen-1-one, 3,5,5-trimethyl-											
6	5.04	5.24	5.38	1067984	129507	5703	949	852	863	146	126
COMPOUND: 2-Propyn-1-ol, 3-p-tolyl-											
7	6.10	6.24	6.34	666096	118253	7859	949	733	736	160	140
COMPOUND: 2-Cyclopenten-1-ol, 1-phenyl-											
8	15.60	15.77	15.98	367328	60132	29006	884	819	837	390	270
COMPOUND: 1,2-Benzenedicarboxylic acid, diisooctyl ester											

The Thermal Decomposition of Metal Acetates

JJ210 **47096, WA, FE-08-000D, 3' SP2250
 BASE 599,120 ZERO 14,256 SCALE 1.00
 TOTAL ION CHROMATOGRAM

** MAR 16, 1983 11:11AM



IRON — acetone soluble fraction — Fe-08-000D
 TABULATION OF RESULTS FROM THE NBS LIBRARY

PEAK	START	TOP	END	PEAK AREA	PEAK HEIGHT	ENTRY	FIT	PUR	RFIT	MOL. WEIGHT	TEMP (°C)
1	1.05	1.20	1.33	991936	207587	22074	555	428	506	282	66
COMPOUND: 1 <i>H</i> -Inden-1-one, 2,3-diphenyl-											
2	1.30	1.45	1.64	1834336	370661	2095	721	439	439	114	67
COMPOUND: 3-Pentenoic acid, 4-methyl-											
3	2.65	3.07	3.21	337024	51848	21995	921	780	833	282	83
COMPOUND: Benzaldehyde, 2,4-bis(trimethylsiloxy)-											
4	3.54	3.71	3.91	1220560	159164	1025	744	496	501	98	90
COMPOUND: Furan, 2,5-dihydro-2,5-dimethyl-											
5	3.88	3.95	4.15	182080	30697	5460	725	658	745	144	93
COMPOUND: 2-Pentanone, 5-(acetyloxy)-											
6	4.23	4.35	4.64	448960	45997	813	918	668	680	94	97
COMPOUND: Phenol											
7	5.03	5.11	5.28	181696	31987	1613	957	826	841	108	105
COMPOUND: Phenol, 2-methyl-											
8	5.28	5.43	5.65	2195648	269914	1613	968	885	899	108	108
COMPOUND: Phenol, 2-methyl-											
9	5.60	5.67	5.82	285568	82944	4679	927	781	839	138	111
COMPOUND: 2-Cyclohexen-1-one, 3,5,5-trimethyl-											
10	6.04	6.21	6.41	1170176	145988	2804	980	934	947	122	117
COMPOUND: Phenol, 2,3-dimethyl-											
11	6.41	6.55	6.90	2480896	225997	2802	986	932	936	122	121
COMPOUND: Phenol, 3,5-dimethyl-											
12	6.99	7.22	7.59	1052161	91904	4276	896	695	726	136	128
COMPOUND: 1,3-Cyclohexadiene, 1,2,6,6-tetramethyl-											
13	7.61	7.81	8.15	1732352	183552	4428	910	764	782	136	134
COMPOUND: Phenol, 2,4,6-trimethyl-											
14	17.35	18.23	18.78	674560	31974	6245	819	430	512	150	248
COMPOUND: Benzene, (1-methoxy-1-methylethyl)-											
15	18.78	19.16	19.42	292352	26842	17691	749	355	468	236	258
COMPOUND: 1,2-Benzenedicarboxylic acid, butyl methyl ester											
16	19.71	19.87	20.02	169344	26368	23666	343	260	543	300	266
COMPOUND: D-Homopregn-17a(20)-ene, (5.alpha.,17aE)-											
17	20.02	20.23	20.65	461440	50389	29004	833	414	470	390	270
COMPOUND: 1,2-Benzenedicarboxylic acid, dioctyl ester											

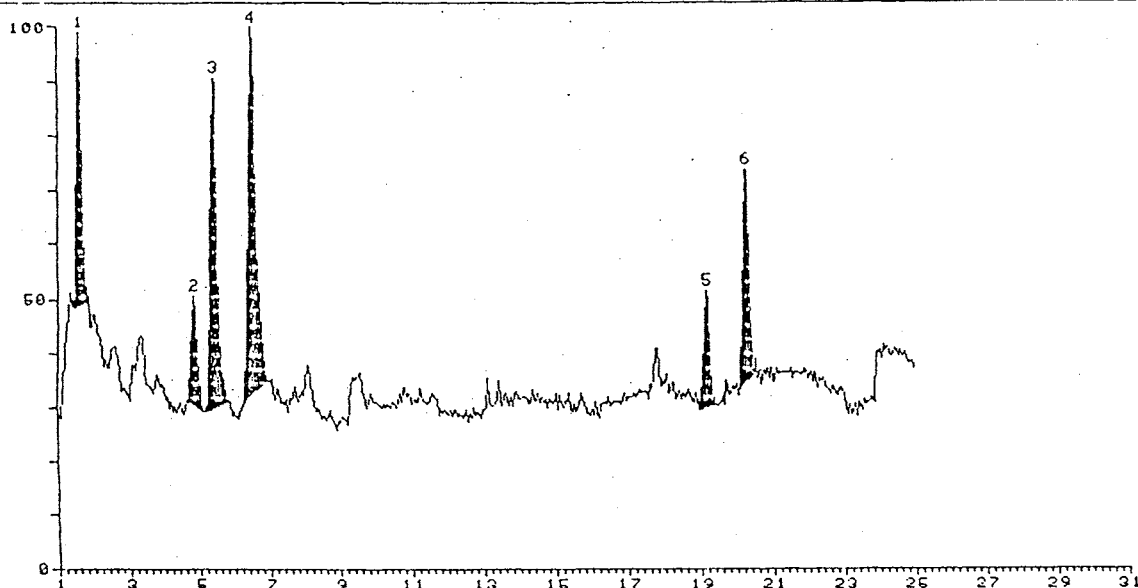
The Thermal Decomposition of Metal Acetates

LEAD — petroleum ether soluble fraction — Pb-08-001C

TABULATION OF RESULTS FROM THE NBS LIBRARY

IN 46 447095, WA, PB-08-001C, 3' SP2250
 BALC 267,360 ZERO 120,896 SCALE 1.00
 TOTAL ION CHROMATOGRAM

** MAR 23, 1983 11:17AM



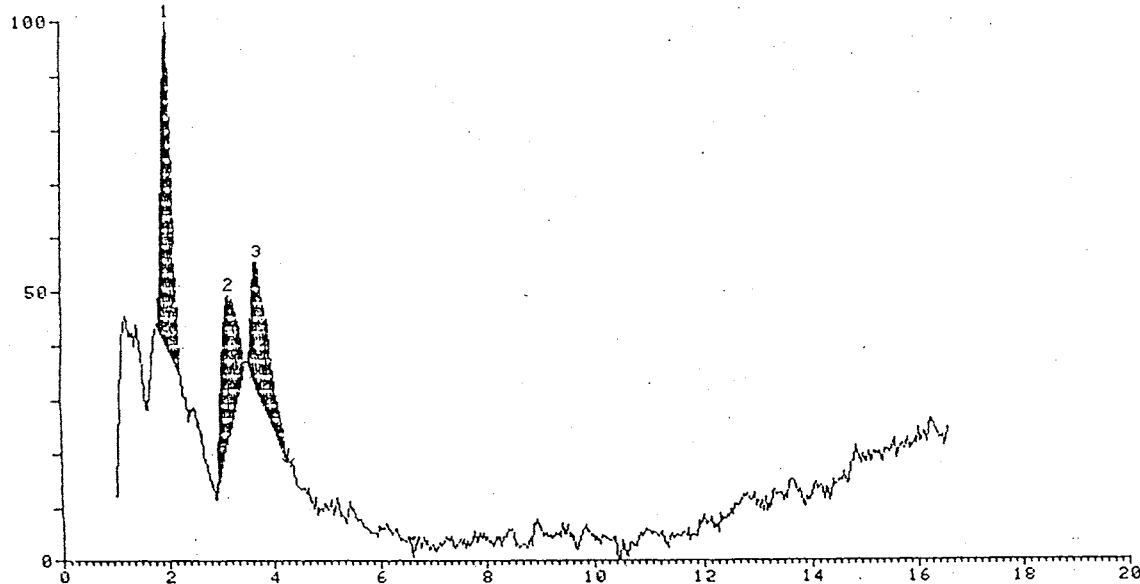
PEAK	START	TOP	END	PEAK AREA	PEAK HEIGHT	ENTRY	FIT	PUR	RFIT	MOL. WEIGHT	TEMP (°C)
1	1.44	1.64	1.87	845056	137192	2413	724	593	669	116	68
<i>COMPOUND: 2-Pentanol, 2,4-dimethyl-</i>											
2	4.65	4.89	5.16	510337	58317	4681	919	786	823	138	103
<i>COMPOUND: 2,5-Heptadien-4-one, 2,6-dimethyl-</i>											
3	5.14	5.43	5.81	1573888	167432	4679	929	749	781	138	108
<i>COMPOUND: 2-Cyclohexen-1-one, 3,5,5-trimethyl-</i>											
4	6.30	6.49	6.90	1867520	181914	2802	988	928	932	122	120
<i>COMPOUND: Phenol, 3,5-dimethyl-</i>											
5	18.96	19.17	19.40	427265	58033	25290	986	940	947	322	258
<i>COMPOUND: 1,2-Benzenedicarboxylic acid, 2-butoxyethyl butyl ester</i>											
6	20.09	20.27	20.50	768512	107546	29006	897	777	799	390	270
<i>COMPOUND: 1,2-Benzenedicarboxylic acid, diisooctyl ester</i>											

The Thermal Decomposition of Metal Acetates

LEAD — acetone soluble fraction — Pb-08-001D
 TABULATION OF RESULTS FROM THE NBS LIBRARY

J13066 **47096, WA, PB-08-001D, 3' SP2250
 BASE 94,022 ZERO 6,656 SCALE 1.00
 TOTAL ION CHROMATOGRAM

** MAR 16, 1983 8:15AM

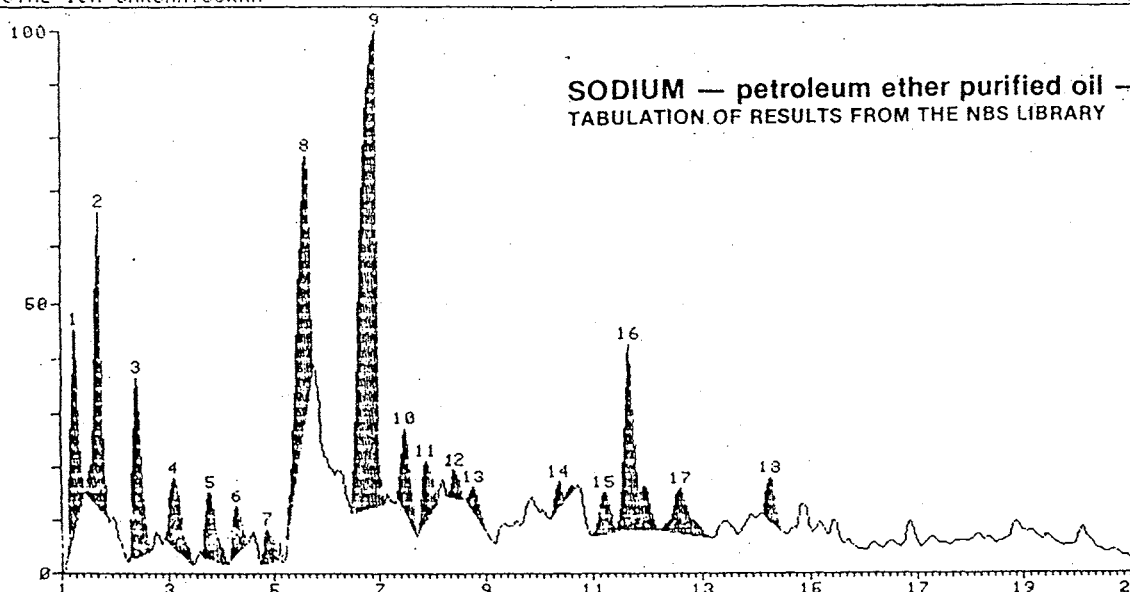


PEAK	START	TOP	END	PEAK AREA	PEAK HEIGHT	ENTRY	FIT	PUR	RFIT	MOL. WEIGHT	TEMP (°C)
1	1.80	1.99	2.29	377856	50706	4679	951	807	847	138	121
COMPOUND: 2-Cyclohexen-1-one, 3,5,5-trimethyl-											
2	2.94	3.18	3.50	269408	22569	2802	974	866	876	122	134
COMPOUND: Phenol, 3,5-dimethyl-											
3	3.50	3.72	4.31	280528	19989	4921	944	803	845	140	140
COMPOUND: 2H-Pyran-2-one, 5,6-dihydro-4,6,6-trimethyl-											

The Thermal Decomposition of Metal Acetates

JJ216 **47096, WA; NA-08-001C, 3' SP2250
 BASE 1,908,448 ZERO 76,736 SCALE 1.00
 TOTAL ION CHROMATOGRAM

** MAR 16, 1983 3:02PM



SODIUM — petroleum ether purified oil — Na-08-001C
 TABULATION OF RESULTS FROM THE NBS LIBRARY

PEAK	START	TOP	END	PEAK AREA	PEAK HEIGHT	ENTRY	FIT	PUR	RFIT	MOL WEIGHT	TEMP (°C)
1	1.09	1.26	1.44	4380673	676480	4276	967	921	936	136	67
COMPOUND: 1,3-Cyclohexadiene, 1,2,6,6-tetramethyl-											
2	1.44	1.71	1.90	6043649	993967	4309	963	874	886	136	68
COMPOUND: Bicyclo[3.1.0]hexane, 6-isopropylidene-1-methyl-											
3	2.25	2.42	2.66	4256897	606479	2685	993	957	960	120	75
COMPOUND: Benzene, 1,2,3-trimethyl-											
4	2.91	3.13	3.47	2414336	244686	4261	804	475	516	136	84
COMPOUND: 1,4-Cyclohexadiene, 1-methyl-4-(1-methylethyl)-											
5	3.64	3.81	4.13	1971264	226112	1963	875	545	573	112	91
COMPOUND: 3-Hexene, 2,2-dimethyl-, (Z)-											
6	4.16	4.31	4.48	1056896	162742	6026	928	875	891	148	97
COMPOUND: Benzene, 2,4-dimethyl-1-(1-methylethyl)-											
7	4.78	4.87	5.10	74880	106171	1729	994	758	760	110	103
COMPOUND: 2-Cyclohexen-1-one, 3-methyl-											
8	5.25	5.62	5.77	9060612	886711	4730	785	631	710	139	111
COMPOUND: 2-Pyrimidinamine, 4-methoxy-6-methyl-											
9	6.49	6.94	7.11	19207940	1576550	2802	978	869	872*	122	125
COMPOUND: Phenol, 3,5-dimethyl-											
10	7.34	7.49	7.71	2043393	296960	8225	629	579	746	162	131
COMPOUND: o-Xylene, 4,5-dimethyl-											
11	7.74	7.88	8.08	1766848	213394	4428	961	881	905	136	135
COMPOUND: Phenol, 2,4,6-trimethyl-											
12	8.28	8.43	8.58	578176	100736	6257	943	687	702	150	141
COMPOUND: Benzene, 1-methoxy-4-(1-methylethyl)-											
13	8.63	8.78	9.06	630528	88922	15415	777	417	477	215	145
COMPOUND: 2(3H)-Benzothiazolone, 3-methyl-, hydrazone, monochloride											
14	10.22	10.37	10.73	65150	89754	11979	809	621	701	188	163
COMPOUND: Naphthalene, 1,2,3,4-tetrahydro-2,3,5,7-tetramethyl-											
15	10.98	11.21	11.41	1039873	141222	14270	678	417	523	206	172
COMPOUND: Oxiranecarboxylic acid, 3-methyl-3-phenyl-, ethyl ester, cis-											
16	11.41	11.67	12.20	5723522	628505	13800	709	616	646	202	176
COMPOUND: 1(2H)-Naphthalenone, 3,4-dihydro-3,3,6,8-tetramethyl-											
17	12.23	12.61	13.19	2003265	153812	13975	501	386	455	204	187
COMPOUND: 3H-Naphtho[1,8-bc]thiophene-5-ol, 4,5-dihydro-2-methyl-											
18	14.12	14.27	14.47	904256	151241	16811	934	434	448	227	205
COMPOUND: Benzoic acid, 3-(phenylamino)-, methyl ester											

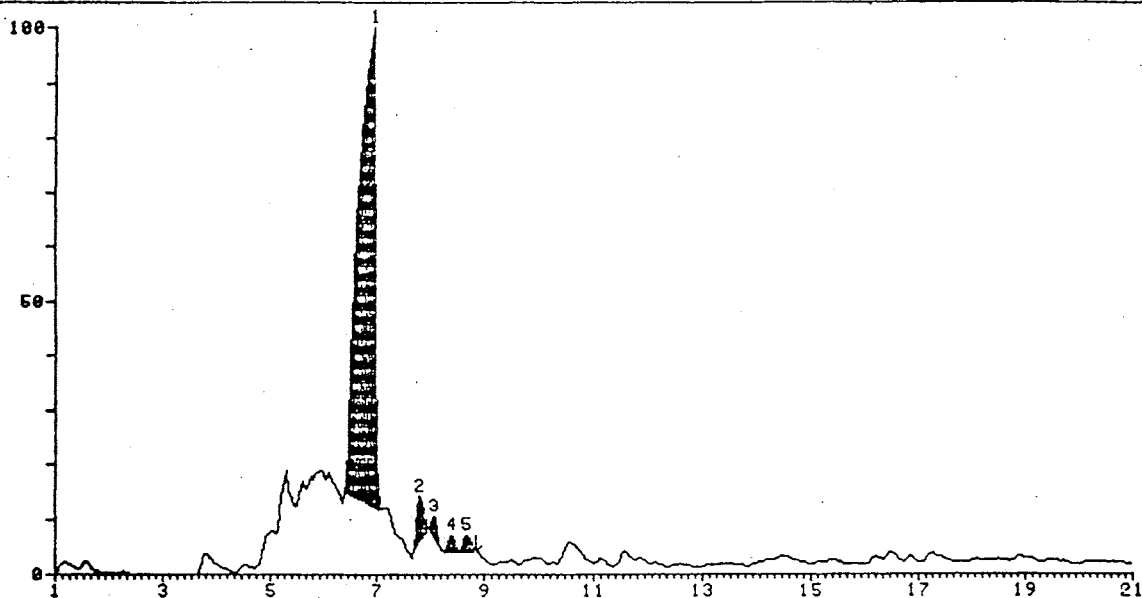
The Thermal Decomposition of Metal Acetates

SODIUM — acetone soluble fraction — Na-08-001E

TABULATION OF RESULTS FROM THE NBS LIBRARY

JJ212 ***47096, WA, NA-08-001E, 3' SP2250
 BASE 2,021,854 ZERO 2,594 SCALE 1.00
 TOTAL ION CHROMATOGRAM

** MAR 16, 1983 12:59PM



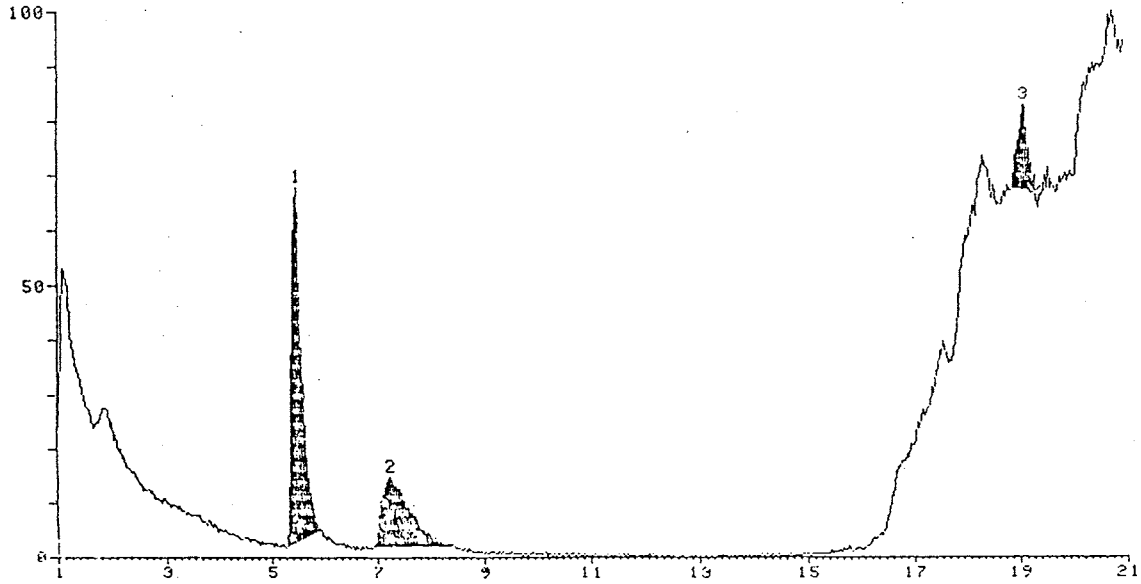
PEAK	START	TOP	END	PEAK AREA	PEAK HEIGHT	ENTRY	FIT	PUR	RFIT	MOL. WEIGHT	TEMP (°C)
1	6.40	6.94	7.11	25760768	1777152	2802	975	813	816	122	125
COMPOUND: Phenol, 3,5-dimethyl-											
2	7.70	7.83	7.98	1097920	178060	4428	965	916	947	136	135
COMPOUND: Phenol, 2,4,6-trimethyl-											
3	7.96	8.08	8.28	279744	72827	6502	928	896	936	152	138
COMPOUND: 2-Cyclohexen-1-one, 2-methyl-5-(1-methylethyl)-, (S)-											
4	8.26	8.40	8.55	357312	66624	6236	975	927	938	150	141
COMPOUND: Phenol, 2-methyl-5-(1-methylethyl)-											
5	8.53	8.67	8.85	346560	56379	4424	909	823	852	136	144
COMPOUND: Phenol, 4-(1-methylethyl)-											

The Thermal Decomposition of Metal Acetates

SODIUM — "phlegm" — Na-09-002A
 TABULATION OF RESULTS FROM THE NBS LIBRARY

JJ211. **47096, WA, NA-09-002A, 3' SP2250
 BASE 41,534 ZERO 65 SCALE 1.00
 TOTAL ION CHROMATOGRAM

** MAR 16, 1983 12:23PM



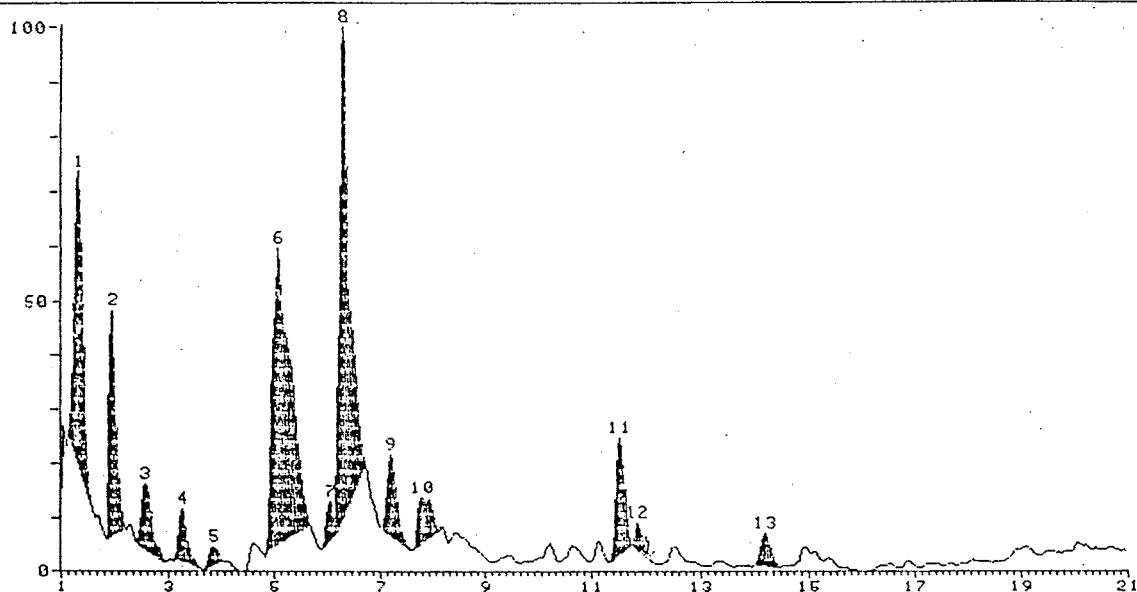
PEAK	START	TOP	END	PEAK AREA	PEAK HEIGHT	ENTRY	FIT	PUR	RFIT	MOL. WEIGHT	TEMP (°C)
1	5.30	5.47	5.94	221784	27125	4679	920	892	923	138	109
COMPOUND: 2-Cyclohexen-1-one, 3,5,5-trimethyl-											
2	6.97	7.26	8.40	124953	5331	2802	891	881	938	122	128
COMPOUND: Phenol, 3,5-dimethyl-											
3	18.91	19.11	19.31	49952	6496	20666	593	305	445	266	257
COMPOUND: 9-Octadecenal, (Z)-											

The Thermal Decomposition of Metal Acetates

SODIUM — "menstruum foetens" — Na-09-002B
 TABULATION OF RESULTS FROM THE NBS LIBRARY

JJ213 **47096, WA, NA-09-002B, 3' SP2250
 BASE 703,794 ZERO 9,432 SCALE 1.00
 TOTAL ION CHROMATOGRAM

** MAR 16, 1983 1:29PM



PEAK	START	TOP	END	PEAK AREA	PEAK HEIGHT	ENTRY	FIT	PUR	RFIT	MOL. WEIGHT	TEMP (°C)
1	1.17	1.39	1.63	2752320	396716	4309	967	895	907	136	75
COMPOUND: Bicyclo[3.1.0]hexane, 6-isopropylidene-1-methyl-											
2	1.85	2.00	2.19	1673520	293298	2685	994	967	971	120	80
COMPOUND: Benzene, 1,2,3-trimethyl-											
3	2.41	2.59	2.94	674848	85792	6256	870	697	708	150	84
COMPOUND: Benzene, 2-methoxy-1,3,5-trimethyl-											
4	3.14	3.28	3.63	524288	71830	4815	748	708	733	140	87
COMPOUND: Cyclohexane, 1,1,3,5-tetramethyl, trans-											
5	3.68	3.87	4.04	191568	25241	6026	977	897	905	148	92
COMPOUND: Benzene, 2,4-dimethyl-1-(1-methylethyl)-											
6	4.88	5.13	5.67	5663980	386449	4567	926	861	875	138	105
COMPOUND: m-Menth-1(7)-ene, (R)-(-)											
7	5.94	6.07	6.17	248704	51826	2806	972	608	617	122	116
COMPOUND: Phenol, 2,6-dimethyl-											
8	6.17	6.34	6.74	5189696	627467	2794	946	886	932	122	119
COMPOUND: Phenol, 2-ethyl-											
9	7.06	7.23	7.58	772512	106827	8242	940	860	874	162	128
COMPOUND: Benzene, 1,2-diethyl-3,4-dimethyl-											
10	7.63	7.80	8.20	703488	61331	4428	929	698	707	136	135
COMPOUND: Phenol, 2,4,6-trimethyl-											
11	11.34	11.54	11.76	1032416	149291	13800	909	806	862	202	175
COMPOUND: 1(2H)-Naphthalenone, 3,4-dihydro-3,3,6,8-tetramethyl-											
12	11.74	11.86	12.04	199096	35340	4773	631	322	368	139	179
COMPOUND: 2-Propynylamine, N,N-diisopropyl-											
13	14.00	14.20	14.45	335764	43861	16806	885	578	616	227	204
COMPOUND: 4(1H)-Pyridone, 3-acetyl-2-methyl-6-phenyl-											

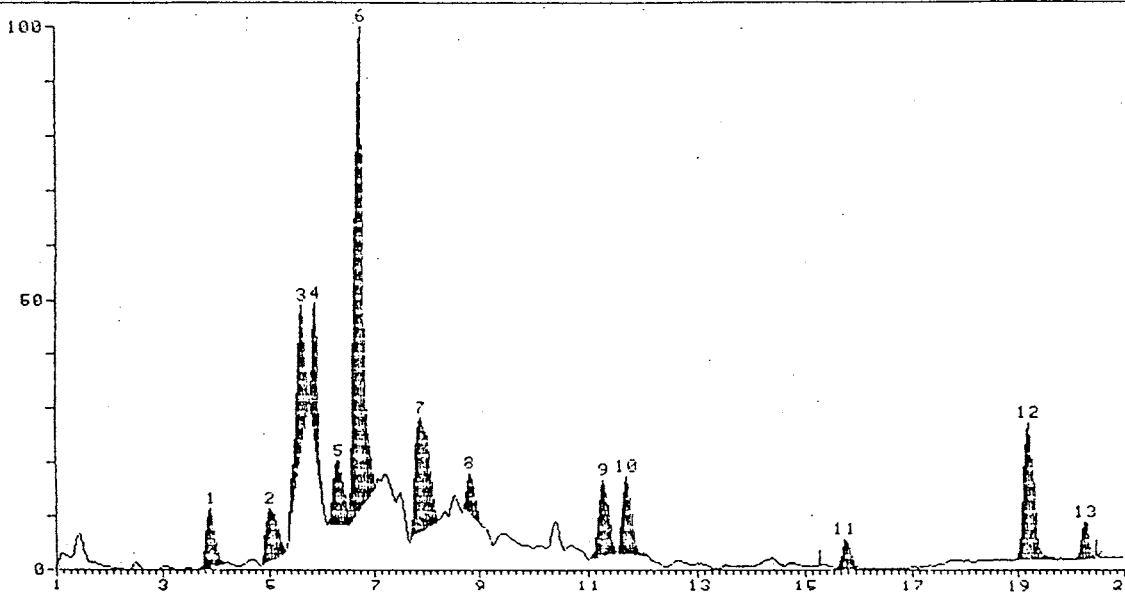
The Thermal Decomposition of Metal Acetates

POTASSIUM — petroleum ether soluble fraction — K-08-000A2

TABULATION OF RESULTS FROM THE NBS LIBRARY

JJ209 **47096, WA, K-08-000A2, 3' SP2250
 BASE 1,226,616 ZERO 8,328 SCALE 1.00
 TOTAL ION CHROMATOGRAM

** MAR. 16, 1983 10:15AM



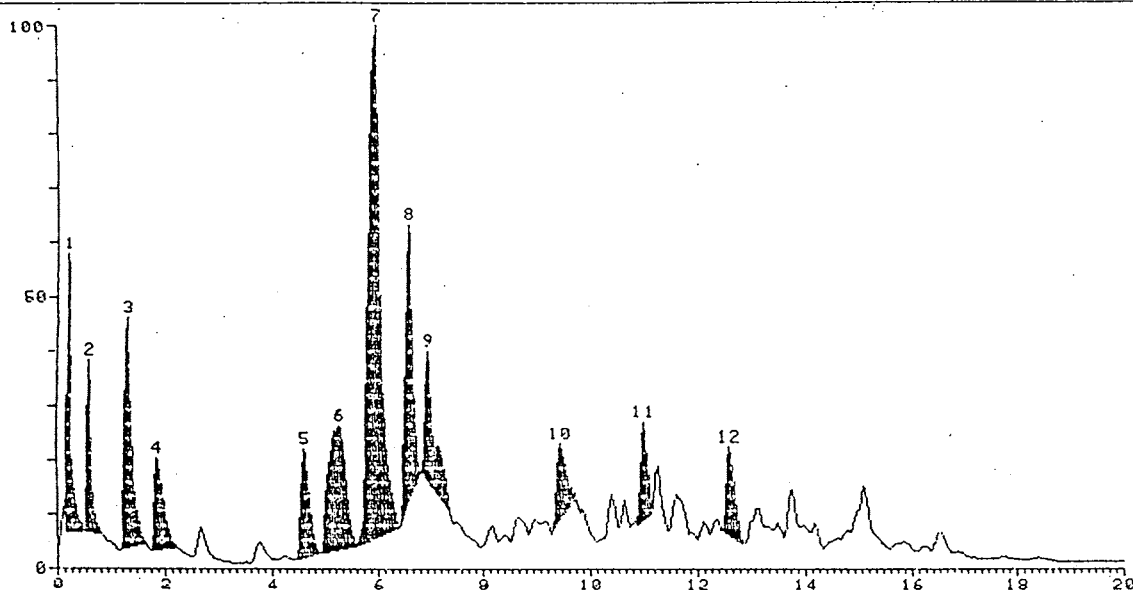
PEAK	START	TOP	END	PEAK AREA	PEAK HEIGHT	ENTRY	FIT	PUR	RFIT	MOL. WEIGHT	TEMP. (°C)
1	3.68	3.90	4.17	880320	130827	1963	852	568	594	112	92
COMPOUND: 3-Hexene, 2,2-dimethyl-, (Z)-											
2	4.88	5.03	5.32	1130544	119787	1729	994	912	913	110	104
COMPOUND: 2-Cyclohexen-1-one, 3-methyl-											
3	5.35	5.60	5.77	2313857	341784	4679	933	569	592	138	110
COMPOUND: 2-Cyclohexen-1-one, 3,5,5-trimethyl-											
4	5.75	5.87	6.04	1323520	325824	4567	879	793	810	138	113
COMPOUND: m-Menth-1(7)-ene, (R)-(-)-											
5	6.14	6.31	6.51	1312128	149649	2802	967	910	930	122	118
COMPOUND: Phenol, 3,5-dimethyl-											
6	6.51	6.74	7.01	7612801	1083917	2802	986	924	927	122	123
COMPOUND: Phenol, 3,5-dimethyl-											
7	7.71	7.88	8.26	3183328	269600	4428	961	896	928	136	135
COMPOUND: Phenol, 2,4,6-trimethyl-											
8	8.71	8.83	9.11	690496	101176	6273	837	712	750	150	145
COMPOUND: Phenol, diethyl-											
9	11.04	11.29	11.52	1269920	169113	7854	865	530	548	160	172
COMPOUND: 1(2H)-Naphthalenone, 3,4-dihydro-8-methyl-											
10	11.52	11.70	12.10	1248864	175617	13800	911	837	899	202	177
COMPOUND: 1(2H)-Naphthalenone, 3,4-dihydro-3,3,6,8-tetramethyl-											
11	15.61	15.76	15.97	371588	62670	21699	882	861	877	278	220
COMPOUND: 1,2-Benzenedicarboxylic acid, butyl 2-methylpropyl ester											
12	18.97	19.20	19.71	2455168	309500	25290	593	578	767	322	258
COMPOUND: 1,2-Benzenedicarboxylic acid, 2-butoxyethyl butyl ester											
13	20.08	20.28	20.49	461520	80048	29006	683	627	731	390	270
COMPOUND: 1,2-Benzenedicarboxylic acid, diisooctyl ester											

ZINC — petroleum ether soluble fraction — Zn-08-000A1

TABULATION OF RESULTS FROM THE NBS LIBRARY

JJ128 **47096, W.A., ZN-08-000A1, 3' SP2250
 BASE 1,704,959 ZERO 0 SCALE 1.00
 TOTAL ION CHROMATOGRAM

** MAR 8, 1983 8:43PM



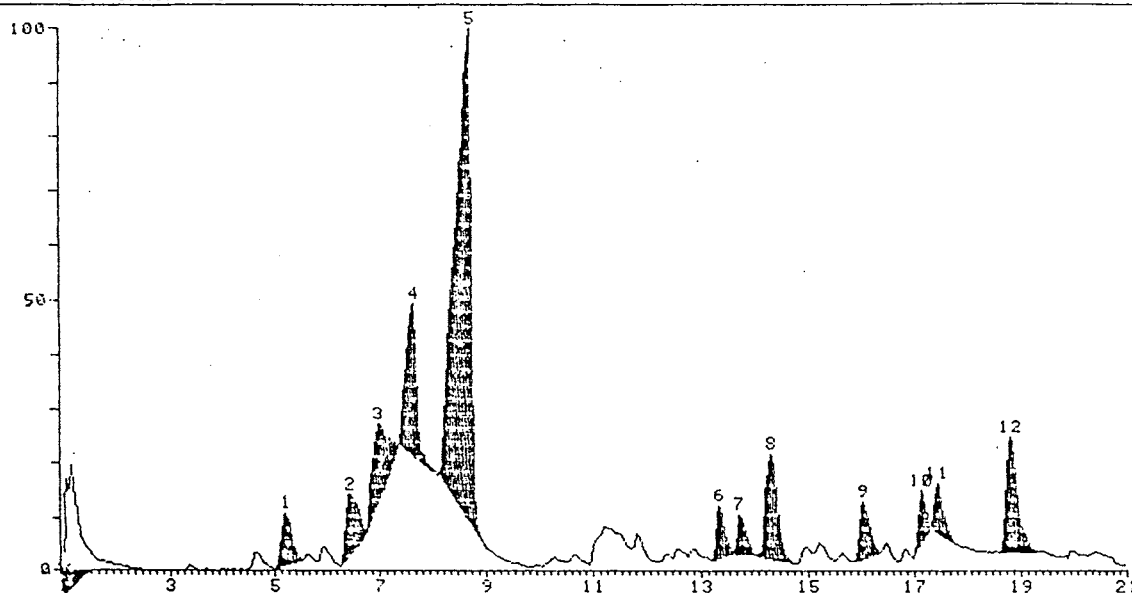
PEAK	START	TOP	END	PEAK AREA	PEAK HEIGHT	ENTRY	FIT	PUR	RFIT	MOL WEIGHT	TEMP (°C)
1	.16	.22	.51	3163872	801824	1245	862	162	167	100	59
COMPOUND: Pentane, 3,3-dimethyl-											
2	.53	.63	.86	2397696	538967	1426	623	610	908	104	62
COMPOUND: Butanoic acid, 3-hydroxy-											
3	1.21	1.34	1.66	5244416	722780	6811	811	760	800	153	68
COMPOUND: 2-Cyclohexen-1-one, 3,5-dimethyl-, o-methyloxime											
4	1.73	1.89	2.21	2368208	280704	2356	756	644	760	116	76
COMPOUND: 1,3-Dioxolane, 2,2,4-trimethyl-											
5	4.44	4.62	4.88	2703040	335753	4681	863	770	801	138	116
COMPOUND: 2,5-Heptadiene-4-one, 2,6-dimethyl-											
6	4.92	5.28	5.60	6558241	386366	4567	918	793	807	138	125
COMPOUND: m-Menth-1(7)-ene, (R)-(-)											
7	5.62	6.00	6.43	18186816	1603924	2802	985	921	925	122	136
COMPOUND: Phenol, 3,5-dimethyl-											
8	6.43	6.62	6.84	6029472	869584	8445	979	929	933	164	145
COMPOUND: Phenol, 3-ethyl, acetate											
9	6.82	6.99	7.37	4155522	410407	8183	989	930	935	162	150
COMPOUND: Ethanone, 1-(2,4,6-trimethylphenyl)-											
10	9.29	9.46	9.65	2062208	244228	6151	908	439	447	148	186
COMPOUND: 2(3H)-Benzofuranone, 3-methyl-											
11	10.88	11.03	11.16	1866368	308017	11961	901	758	811	188	209
COMPOUND: Benzene, 1-(1-cyclohexen-1-yl)-4-methoxy-											
12	12.46	12.63	12.86	1917856	275420	21910	757	471	527	280	233
COMPOUND: Cyclohexane, 1-(1,5-dimethylhexyl)-4-(4-methylpentyl)-											

The Thermal Decomposition of Metal Acetates

ZINC — acetone soluble fraction — Zn-08-000A2
 TABULATION OF RESULTS FROM THE NBS LIBRARY

JJ208 **47096, WA, ZN-08-000A2, 3' SP2250 .
 BASE 1,687,152 ZERO 19,856 SCALE 1.00
 TOTAL ION CHROMATOGRAM

** MAR 16, 1983 9:36AM

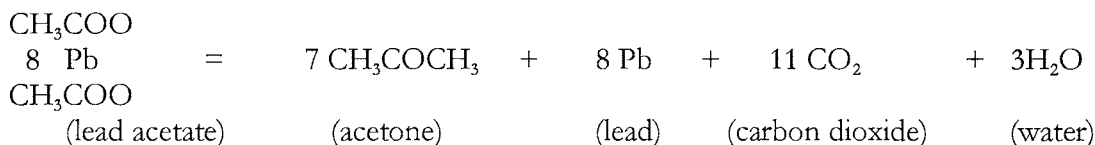
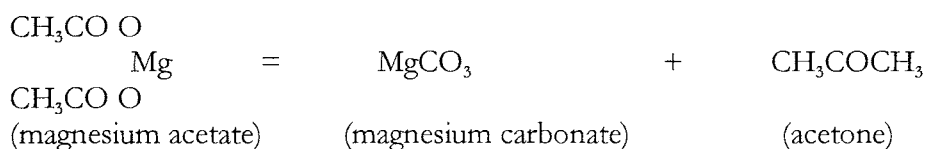


PEAK	START	TOP	END	PEAK AREA	PEAK HEIGHT	ENTRY	FIT	PUR	RFIT	MOL. WEIGHT	TEMP (°C)
1	5.03	5.22	5.55	1378160	169388	4679	869	750	802	138	106
COMPOUND: 2-Cyclohexen-1-one, 3,5,5-trimethyl-											
2	6.32	6.46	6.76	1817632	199196	2802	982	957	962	122	120
COMPOUND: Phenol, 3,5-dimethyl-											
3	6.76	6.98	7.37	2752384	247878	2802	983	947	951	122	126
COMPOUND: Phenol, 3,5-dimethyl-											
4	7.37	7.64	8.00	3546113	471374	2889	889	820	844	124	133
COMPOUND: Ethanone, 1-(4-methyl-1H-imidazol-2-yl)-											
5	8.14	8.72	8.96	18100164	1538496	4908	576	439	479	140	144
COMPOUND: 2-Pentynoic acid, 4-oxo-, ethyl ester											
6	13.21	13.35	13.60	1079392	168078	11961	902	800	850	188	195
COMPOUND: Benzene, 1-(1-cyclohexen-1-yl)-4-methoxy-											
7	13.60	13.73	14.03	884192	130868	14133	796	696	812	204	199
COMPOUND: 3H-1,2-Dithiole-3-thione, 5-tert-butyl-4-methyl-											
8	14.03	14.31	14.81	2732624	330256	11961	780	142	165	188	205
COMPOUND: Benzene, 1-(1-cyclohexen-1-yl)-4-methoxy-											
9	15.86	16.06	16.36	1343056	178772	11960	683	636	794	188	224
COMPOUND: 1(2H)-Naphthalenone, 3,4-dihydro-4,5,8-trimethyl-											
10	17.00	17.16	17.30	972448	172913	15453	856	428	445	216	234
COMPOUND: 3H-Pyrazol-3-one, 4-acetyl-2,4-dihydro-5-methyl-2-phenyl-											
11	17.30	17.44	17.77	1266688	168230	17556	780	450	539	234	238
COMPOUND: Phenanthrene, 9-butyl-											
12	18.60	18.82	19.32	3077088	358272	15320	798	682	711	214	253
COMPOUND: Naphthalene, 2-butyl-7-methoxy-											

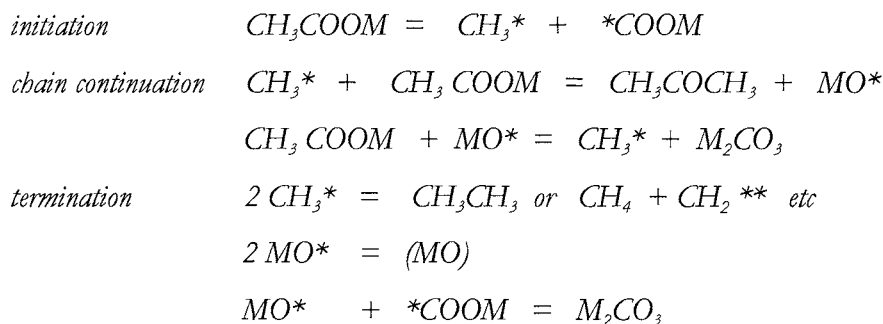
CONCLUSION

Chemical Views

The accumulated data indicates the occurrence of a highly complex sequence of reactions during the acetate distillation. Certainly all of the various fractions of pyrolysis, condensation, catalysis, etc., cannot be discussed herein. Many of these reactions can be drastically altered simply by changing the experimental set-up involving heating rates, cooling, flow design, and vapor contact time. Some of these reactions have been studied with the aid of radioisotopes^{29, 30} and models of reaction mechanisms proposed. A simple idea of the pyrolysis of a metal acetate is shown in the following equations:



A more generalized reaction sequence was suggested by Bell and Read³¹ as the following free radical mechanism:



NOTE: $M = \text{Li, Na, K, etc.}$

As shown by Ardagh et al,³² the yield of acetone thus produced can be increased by gently sweeping the reaction vessel with an inert gas.

Conversely, the oily residue increases if the acetone formed has a longer contact time with the hot mineral residue. In the latter case, the acetone itself is believed to undergo pyrolytic decomposition in the following manner:³³

The Thermal Decomposition of Metal Acetates

		E _{act.} (kcal)
(1)	$\text{CH}_3\text{COCH}_3 = \text{CH}_3^* + \text{CH}_3\text{CO}$	70
(2)	$\text{CH}_3\text{CO} = \text{CH}_3^* + \text{CO}$	10
(3)	$\text{CH}_3^* + \text{CH}_3\text{COCH}_3 = \text{CH}_4 + \text{CH}_3\text{COCH}_2^*$	15
(4)	$\text{CH}_3\text{COCH}_2^* = \text{CH}_3^* + \text{CH}_2^{**} = \text{CO}$	48
(5)	$\text{CH}_3^* + \text{CH}_3\text{COCH}_2^* = \text{CH}_3\text{COC}_2\text{H}_5$	8

These products can, in their turn, undergo reactions of pyrolysis or condensation. One of the major products is ketene (see equation 4) $^{**}\text{CH}_2 = \text{CO}$, an efficient acetylating agent ³⁴ capable of undergoing polymerization. ³⁵ The high occurrence of methylated compounds in the GC/Mass spectral data points favorably to the free radical mechanism.

As we can see then, a wide range of products are possible when we closely consider the reaction environment and even these are changeable by altering the apparatus design. Although the different metal salts should be capable of producing unique catalytic action and thus unique mixtures of reaction products, there are compounds which are common to nearly all. From the GC/Mass spectral data, a major compound in the metal oils appears to be 3,5-dimethylphenol. Qualitative analysis on the oil also indicates the presence of phenols helping to corroborate this identification.

The data from the GC/Mass spec. should not be considered the last word in the analysis of the reaction products. It represents a tabulation of the most probable compounds produced, but until each fraction is isolated in pure form and identified by derivatives, the data can only serve as a guide to future research. Also many of these compounds are obtained by the destructive distillation of wood and coal, and this gives rise to a generalized nomenclature when speaking of these oily residues as coal tars.

For a medical viewpoint, compound classes could be considered, or each compound listed could be referenced through the National Bureau of Standards registry number. In this manner some idea of the possible medical use could be discerned. For example, the compound 3,5-dimethylphenol, mentioned earlier as a major component in many of the oils, is listed ³⁶ as a member of a group of compounds collectively called cresols. The therapeutic category is disinfectant, though it can cause rapid circulatory collapse and death in larger doses (8 g.). Chronic poisoning from oral or percutaneous absorption may produce digestive disturbances, nervous disorders, faintness, vertigo, mental changes, skin eruptions, jaundice, oliguria, uremia. It is also listed as a general protoplasmic poison. Other methyl-, dimethyl-, and trimethyl phenols listed in the G C/Mass spec. data also fall in this category of cresols.

In such cases, dosage is a major consideration and perhaps use in homeopathic practice indicated. Again it may be the particular combination and ratio of compounds in an oil which have an intrinsic medical virtue. Few sources do more than indicate medical applications and this area of the topic is wide open for future research. Becker records the following statements concerning the more volatile fractions of the distillate:

“Since 1840 I have used the acetone very frequently. Since it contained not only acetone, but also oils, if prepared according to the old way, I called it spiritus aceti oleosus, so it could be distinguished. The preparation was good, but it did not correspond completely to the description of the old chemists since it was lacking the praised pleasant smell, which might be due to the fact that the preparation used then let the substance mature through long and repeated digestion and distillation...”

“The dehydration of the acetone through distillation over calcium chloride is chemically correct, but not the best for the medication. The pure acetone, like it can be obtained commercially, is not as strong, not in smell and taste, not in its medical effectiveness.

“...therefore, it appears that the etheric oil is essential for the medical constitution.”³⁷

Becker goes on to quote case histories of the successful treatment of flu, nervous complaints, rheumatism, headaches, fevers, and paralysis.

The Merck Index describes the toxicology of pure acetone stating that: “prolonged or repeated topical use may cause erythema, dryness. Inhalation may produce headache, fatigue, excitement, bronchial irritation, and, in large amounts, narcosis. Serious poisoning rare. LD in rats: 10.7 ml/mg orally”.³⁸

Alchemical Views

In the literature of alchemy, the process studied herein is more in the nature of a beginning or preliminary operation.

In the alchemical allegory, nature has infinite time at its disposal in the process of evolving towards perfection. By following the laws of nature, the alchemist hoped to accelerate this growth to perfection. As the Emerald Tablet states (on the frontispiece):

“As all things are derived from the one only thing, by the will and by the word of the one only one who created it in his mind, so all things owe their existence to this unity by the order of nature, and can be improved by adaptation to that mind.”

So in the acetate distillation was seen a partial demonstration of the course of nature. The crude and unrefined being (the minera) was subjected to forces and processes which purified and exalted its own nature ultimately making accessible its spiritual essence (distillate) on a material level. The process is one of consciously working with nature, purifying and refitting the vehicle of expression of the life force. It is the production of a better conductor for the spiritual light.

Using this process of acetate distillation as a foundation, Holland describes a more lengthy operation requiring 30-32 weeks eventuating, in the production of a substance he calls a stone...

“And if the party will take the like quantity of a wheatcorn every day for the space of nine days, I tell you, his body will be as spiritual as if he had been nine days in the terrestrial paradise eating every day of the fruit, making him, fair, lusty, and young. Therefore, use this stone weekly, the quantity of a wheatcorn with warm wine, so shall you live in health until your last hour of the time appointed for you by God.”

continuing further he says:

“My child, there are some people who have external distempers on their bodies such as fistulas, cancer, wolf, or evil biles or holes, be they what or how they will, etc. Give such a one the weight of one wheatcorn to drink in warm wine two days as is taught before. The whole body will, within and without, be freed from all which is adverse to nature.”³⁹

Certainly, such claims are worthy of extensive research even though today we are given the impression or mass opinion that alchemy has been thoroughly “debunked” giving rise to “modern chemistry”. As in any art or science, progress is the result of not one but many lives. Were there as many true alchemists today, as say, doctors or lawyers, we would be much farther along in either proving or disproving the validity of the hermetic science. However, at present, progress in this field is slow and often still shrouded in secrecy and mystery. Alchemy is not dead, but is quite possibly a sleeping giant.

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