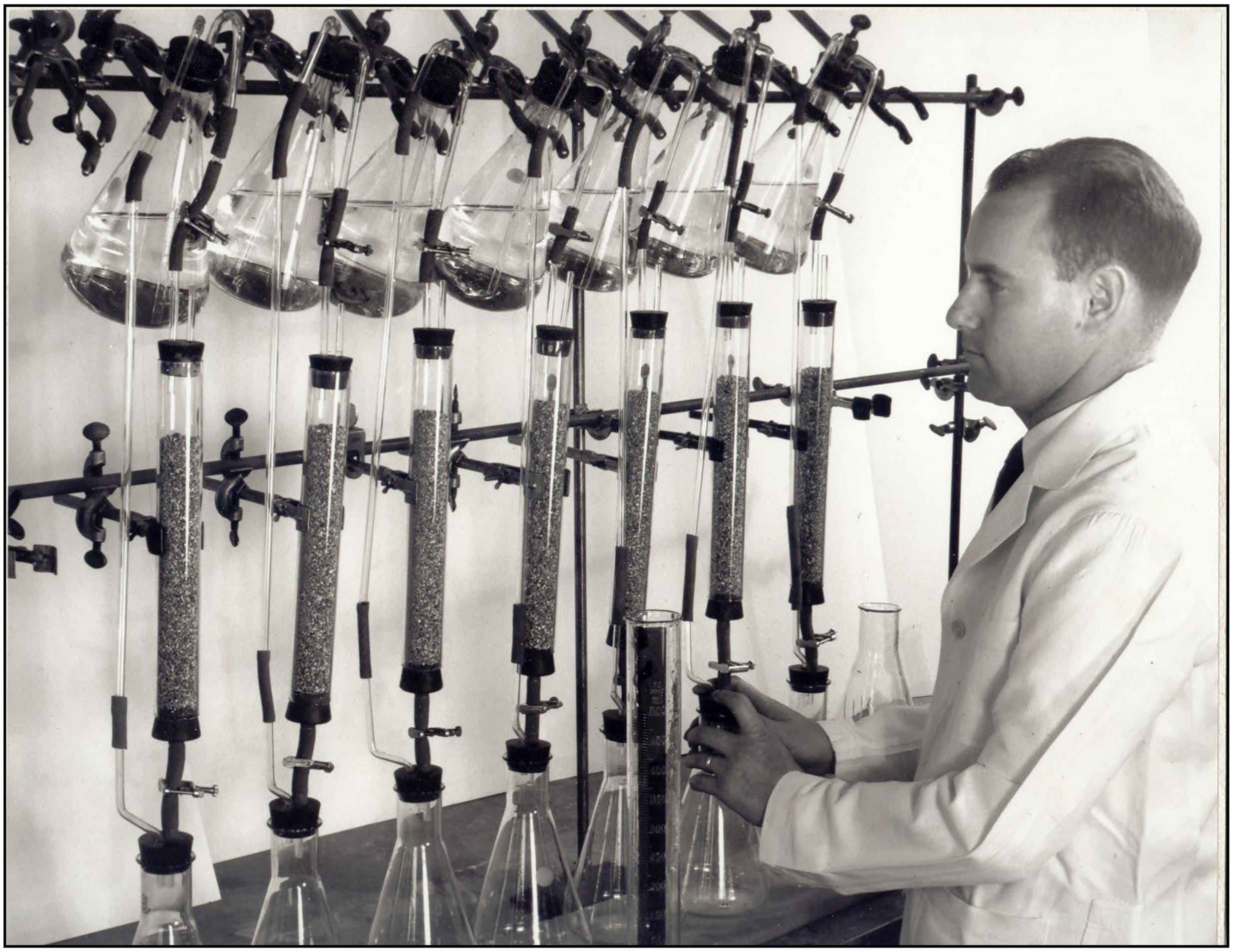
My Career



Midwest Research Institute
1951 to 1996

Gaylord Atkinson

IMPORTANT NOTICE TO ALL READERS!

The copy you have in your hands was shared with you
because you are a member of my family or are a close friend.

This is your copy and I hope you find it interesting,
but because of the sensitive nature of some of the material included
I request that you be discrete with it
and do not share this document with anyone else.

All of the work for the Central Intelligence Agency (the CIA) included in this document was once classified. I have not included anything that I believe could possibly hurt our country, much of the technology discussed is now outdated and has been replaced by digital and other technologies. I have deliberately excluded a few very interesting things that may still be sensitive.

The work was classified ranging from Confidential, to Secret, to Top Secret. My association with the CIA was "Confidential." But it gets a bit complicated and confusing. For example, in 1986 I received the CIA Agency Seal Medallion with a Citation that states in part "in recognition of his sustained outstanding assistance to the Central Intelligence Agency for over thirty years." I asked the individual, the Chief of Protocol, in charge of the presentation about the classification of the Citation. She told me that it was not classified and that I could show it to anyone. So my association with the Agency is classified, but my "assistance to" the Agency "for over thirty years" is not classified.

My original intentions were to write this only for my family. Others have learned that I was writing it and have requested a copy. This is your copy and I hope you find it interesting, but again, I request that you be discrete with the copy and do not make it available to others.

Thank you,

Gaylord
2007

Table of Contents

PREFACE

I often encounter individuals engaged in their employment and think how lucky I was to have had my work rather than theirs. I see doctors going from one examination room after another to spend a few minutes with patients. I see a dentist that sees one patient after another, drills out cavities and puts in fillings. I see pharmacists standing behind a counter filling prescriptions. Each of these professionals probably made more money than I did, but I can't understand what satisfaction they get out of their work. I understand that I see only part of their work and they may very much enjoy their work. They might not have enjoyed the work that I did. Certainly I saw colleagues at work that showed no enthusiasm for work similar to mine. But in general I enjoyed my work.

Not all of my work was enjoyable. I struggled with some reports and proposals, but when they were finished I thought they were good and I was proud of them. I thoroughly enjoyed most of my work in the laboratory, for the most part it was not routine, it was challenging with problems to solve. Three times I was offered a position in management. Three times I turned it down. I was outstanding in a laboratory; I would have been no better than average in management and possibly a failure and would have enjoyed it much less. Actually, in addition to my laboratory work, I was a program manager with responsibility and direction of several projects and the individuals working on them.

I love to play games and much of my lab work was like playing a game. Here is a problem, how can it be solved? Here is a game, how can I win?

I found my work interesting and hopefully I can present a description of it so you will find it interesting.

I started this description of my career at Midwest Research Institute as one part of a family history that I have been preparing. I have decided to let it be separate.

Most of my work was done under contracts with the Central Intelligence Agency, the CIA. The work was classified ranging from Confidential, to Secret, to Top Secret. My association with the CIA was Confidential. My colleagues without a security clearance did not know who we were working for. But the security clearance could get a bit complicated and confusing. For example, in 1986 I received the CIA Agency Seal Medallion with a Citation that stated in part "in recognition of his sustained outstanding assistance to the Central Intelligence Agency for over thirty years." I asked the Chief of Protocol in charge of the presentation about the classification of the Citation. She told me that it was not classified and that I could show it to anyone. So my association with the Agency was classified, but my "assistance to" the Agency was not classified.

All of the CIA work covered in this document was classified and technically may still be classified. Therefore I urge you to be discrete with it. There are a few topics that I would liked to have included but they may still be sensitive and they have not been included. I do not think that I have included anything that could possibly hurt our country now. I worked with many different individuals at the CIA. I mention several of them but for security reasons most are referred to by only their first name and last name initial.

I dedicate this memoir to my wife, Erma Atkinson, who put up with me the many times when I worked long enjoyable hours. She never complained and was proud of my work.

Like my MRI reports, I have struggled to organize and write this document. I wish to thank our daughter Kathy (Dhyan) Atkinson for help in editing.

Gaylord Atkinson
September 25, 2006

SUMMARY

Midwest Research Institute (MRI) is a not-for-profit contract research organization founded near the end of WWII for the purpose of providing technical support for the Kansas City area. Essentially all of the work is done for Clients (Sponsors). Typically a problem is presented to MRI by a Client and MRI responds with a written "Proposal" that states what we plan to do to solve the problem and how much time it will take and how much it will cost. If the client accepts the proposal, the work is conducted and reports submitted.

I started work at MRI on February 1, 1951. I worked on a variety of projects including:

- Determining the physical properties of fertilizers and how to keep them free flowing rather than hardening to form a solid cake in the bag
- Determining the properties of vermiculite as related to its use as a soil conditioner and growing media
- Developing a method for the detection and control of the moisture content of dry cleaning solvents
- The development of the process for producing Folgers Instant Coffee
- The development of a capillary ink reservoir for the Schaeffer Pen Company
- A project for the Department of Agriculture to determine the solubility of about twenty different sugars in six solvents at four different temperatures
- A series of projects with the Central Intelligence Agency (CIA) starting in April 1956 and continuing until my retirement
- Several projects for the EPA relating to the analysis of insulating materials for the presence of asbestos
- An evaluation of the Michigan State Crime Laboratory
- And numerous other small industrial projects

I started work on a project for the Central Intelligence Agency (CIA) in April 1956. My initial work for the CIA was related to the detection of secret writing. Secret writing was very important for the CIA from before the time I started working on it until the mid 1980s when electronic devises gradually took over. (I believe that secret writing is still in some use.)

A great deal of effort was spent on secret writing (SW). In the 1950s, 1960s and 1970s there was a Secret Writing Branch in the CIA with 15 to 20 individuals and probably an additional 100 people working on SW at about a dozen contractor facilities. MRI was the only contractor working on the detection of SW, and we had a considerable influence in the SW field. I was once introduced at a Secret Writing Conference as the Free World's leading expert in the detection of secret writing. It sounded impressive but on reflection I realized that my group was the only group in the United States concentrating on detection. In a field of "one" you should be outstanding! All of the other contractors were developing new SW systems and providing supplies and support for existing systems.

Arthur D. Little (ADL) of Cambridge Massachusetts had been the sole secret writing contractor during WWII and was the sole contractor from the time the CIA was formed in about1947 until 1956. They were primarily responsible for the considerable sophistication of secret writing and by the mid 1960s they were producing systems that were believed (and they claimed) to be completely secure. That is, they maintained that their secret writing systems could be made visible (recovered) only with their specific recovery treatment. In 1968, MRI was given several of the systems and we were asked to try to detect them. In about two weeks we detected messages from two or three systems and in three months we could detect messages of most of the systems and in six months we detected all but two of nearly one hundred systems. The CIA panicked. In six months they had gone from believing that their secret writing messages were completely secure to knowing that they were <u>not</u> secure.

During the next few years the major SW effort of the Client and several contractors was to defeat our detection procedure. Millions of dollars were spent with little success. We tried several approaches to defeat our own procedure that failed but we were the ones that finally came up with one that did work. I will go into more detail about this later.

Our success in the detection of secret writing led to several other secret writing-related projects including developing probe letters with microdots of chemical reagents to be sent through opposition mail channels. (Opposition meaning: The Soviet Block, Red China, and East Germany.) These letters were obtained and analyzed to determine what censorship procedures had been used by the opposition. We also detected and identified the chemicals used as secret inks by the opposition.

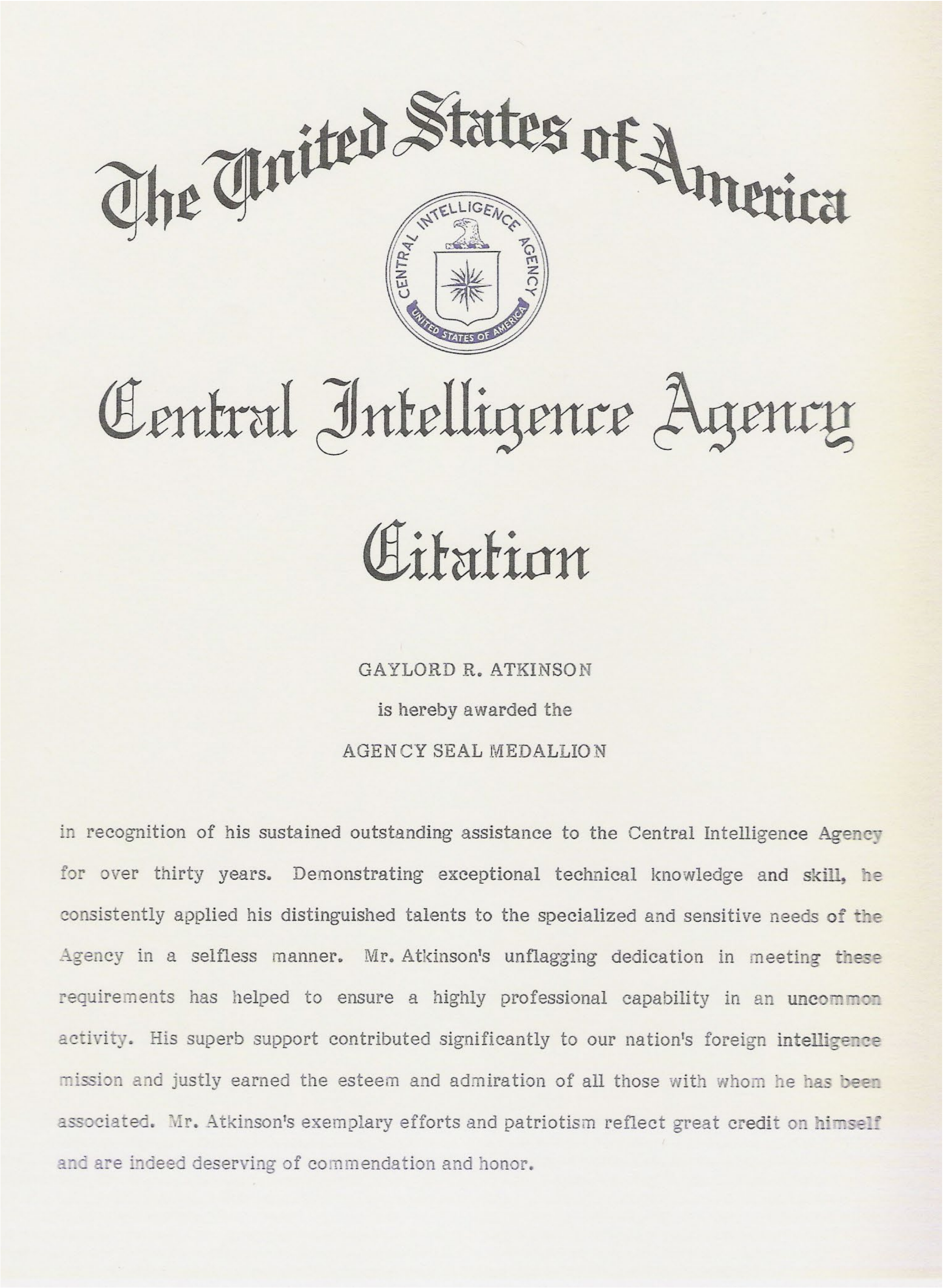
In 1972, I became the Project Leader of a series of general analytical projects for the CIA. This series continued for the remainder of my time at MRI. We received more than one thousand samples for analysis during the more than twenty years of these projects. The samples ranged from simple and easily analyzed to complex and/or challenging to analyze. They ranged from single samples that were unrelated to other samples to series of samples related to a particular subject. They ranged from not very important to <u>very</u> important.

Many of the samples came from on-going operations. Results were wanted as soon as possible. In many cases delayed results were essentially worthless. I accepted the analysis of samples as a game, to be solved, without error, as quickly as possible. The fast response was greatly appreciated.

In 1986, I was awarded the Central Intelligence Agency Medallion for “outstanding contributions to the intelligence effort of the United States.” I was only the fourth non-CIA employee to receive the Medallion. No specific contributions were included in the citation but I believe I received the award because of the work related to SW detection, timely submission of analytical results, and the work on and a report of one very important sample that provided information that indicated that the Soviet Union’s submarine fleet had a superior system for air purification. Erma was given special security clearance for the presentation of the award which was presented in the Directors Conference Room with about 50 CIA employees and several MRI employees present. Many from the CIA held high level positions. The Medallion and the accompanying Citation are shown on the following pages.

To
Gaylord R. Atkinson
In recognition of
your dedicated support
and outstanding contributions
to the intelligence effort
of the United States
1986

The Front and Back of the Medallion Presented August 28, 1986



The United States of America

Central Intelligence Agency

Citation

GAYLORD R. ATKINSON

is hereby awarded the

AGENCY SEAL MEDALLION

in recognition of his sustained outstanding assistance to the Central Intelligence Agency for over thirty years. Demonstrating exceptional technical knowledge and skill, he consistently applied his distinguished talents to the specialized and sensitive needs of the Agency in a selfless manner. Mr. Atkinson's unflagging dedication in meeting these requirements has helped to ensure a highly professional capability in an uncommon activity. His superb support contributed significantly to our nation's foreign intelligence mission and justly earned the esteem and admiration of all those with whom he has been associated. Mr. Atkinson's exemplary efforts and patriotism reflect great credit on himself and are indeed deserving of commendation and honor.

Citation presented with medallion

The Medallion presentation by Jim Hirsch,
Deputy Director of the Science and Technology Directorate
August 28, 1986

There are four Directorates in the CIA. The head of the CIA is the Director and there are four Deputy Directors. This presentation was in August 1986. Two years later when I had a heart attack I received a very nice letter from Jim. Unfortunately, the letter was classified secret so I could not keep it.

A group picture after the presentation including Erma
Jim Hirch, Gaylord Atkinson, Erma Atkinson, Jim's Deputy Dr. Munsenmyer

SECTION 1 INTRODUCTION

It is rather uncommon these days for an individual to spend an entire career with one organization, but, a few years after I started work at MRI, I became involved with a series of fascinating projects that I did not want to leave. These projects were for the Central Intelligence Agency, the CIA, during the time of the "Cold War," and much of the work was directly or indirectly related to the Cold War. These projects continued for the remainder of my 40 years of regular employment and an additional five years of part time employment because I found the work so interesting. I have always loved the challenge of games and the majority of my work was like a series of games. A problem was presented and the objective was to solve the problem. Some problems were solved within a few hours, some in a few days, and others after a few weeks or months, and one took more than twenty years.

Perhaps because I grew up on a farm during the depression when there was little or no money to buy new things, problem solving was a way of life. I developed a knack of solving problems that served me well in my career at MRI. Problem solving is a dynamic process. As information is obtained concerning the problem it is analyzed and the problem redefined. Often unexpected results from an experiment or analysis are the key to solving the problem. I was always alert for unexpected results and what they might mean. I will forever be amazed at many individuals, with science degrees, even PhD degrees, who hadn't the slightest concept of problem solving, or the ability to think though a problem. Often they dismiss any results that they were not expecting and missed important clues for solving the problem. Another important characteristic of being a good problem solver is how preliminary results are treated. If early results suggest a certain answer, most people will design additional experiments only to support the suggested answer. In addition to that type of experimentation, I also tried to design experiments to show that the anticipated answer was wrong. If I couldn't prove that it was wrong, it made the "right" answer much more certain. And, if I was wrong, I wanted to be the one to find out first.

People were part of my MRI experience and I will include some of them in the discussion. In general I worked with a very good group but there were exceptions. I especially enjoyed working with Paul Siewald I tend to give more negative coverage for the few than positive for the many. A few individuals did not live up to reasonable standards and they too contributed to my experience and MRI's reputation. I will include a couple of these negative examples. I will include one here.

I met many individuals from various parts of the CIA organization and for several years every new person that I met asked something like "Does Marty Wolf still work at MRI?" They might have forgotten her name but they all knew what she had done. Marty, Dr. Wolf, did not work in my group. She worked with a small group trying to develop a biological secret writing system. Her parents lived in New York City and she wanted a free trip home for Christmas so she arranged with her Project Leader to have a project meeting in Washington D.C. and deliver a report. The CIA individual that she normally contacted was off for Christmas and she met with the Deputy Branch Chief, John V. John was a very nice, low-key individual. Marty delivered the report and they went over it. John told her that it was unacceptable. (I later saw the report and, I agree, it was unacceptable.) She responded that she had worked on National Institute of Health (NIH) programs and her reports were accepted by them and then said, "by God if it is good enough for the NIH, it sure as hell is good enough for the CIA!" John called his boss, Bill S, the Branch Chief and asked if he could meet with him and Marty Wolf from MRI. I knew Bill for years and had an excellent relationship with him. When John and Marty entered his office, Bill stood up to greet this individual from MRI who he had never met. He extended his hand for a hand shake but got the following response. Marty threw the report on his desk and said "Here is your f----ing report and you can stick it straight up your a—". She then turned on her heel and walked out of his office. With that, MRI became well known throughout the CIA!

I had a few offers for other employment and nearly switched jobs in 1957 to go with a company in southern Illinois. I knew a few of the employees of that company and they knew of my work and ask me to join them. Their major work was with pyrotechnics and their major contractor was NASA. They were making pyrotechnic timing devises and such things as explosive bolts. For example, up to the moment of "lift off,"a satellite and rocket are held in place bolted to a tower and the launch pad. The bolts contain explosives and are blown apart in a timed sequence to release the satellite at the proper time. Fortunately I did not join the group because this company failed about two years later.

Off and on I worked with Hallmark Cards to solve several problems with inks and papers. When their Chief Chemist retired, he recommended me as his replacement. I seriously thought about it but decided not to accept their offer.

But let's go back to the beginning: How did I get to MRI? Dr, Militzer, my Major Professor at the University of Nebraska and Dr. Sherman Gillam who was at MRI had known each other while both were at Purdue University. Shortly before I finished my Masters Degree, Dr. Militzer had gone to MRI to have some samples examined with MRI's electron microscope. When he came back he told me about MRI, I applied for employment, and was hired.

I started work at MRI on February 1, 1951.

Midwest Research Institute is a not-for-profit contract research organization. It was formed by a group of six prominent Kansas City business leaders in 1944 near the end of World War II with the goal of providing a source of scientific research for the region, and thus promoting business growth in technical areas.

MRI started in a group of buildings in the Westport area of Kansas City. The headquarters building where I worked was located 4049 Pennsylvania. A

(About 1948) Gaylord working at the photo lab of the Soil Conservation Service while he was enrolled at the University of Nebraska.

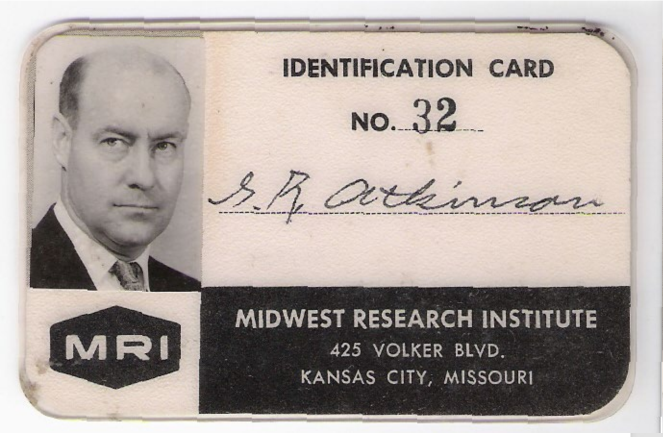
new building was built on the corner of Volker and Oak and we moved to that location in March 1955.

When I started work there were slightly more than 100 employees. I have a 1952 Directory of Personnel that lists 115 employees of whom 36 were in the Chemistry department. There were about 450 employees when I finally retired in 1996.

MRI Employment Photo - 1951

Earlier Picture of Gaylord

Front and back of ID Card, mid to late 1950s.



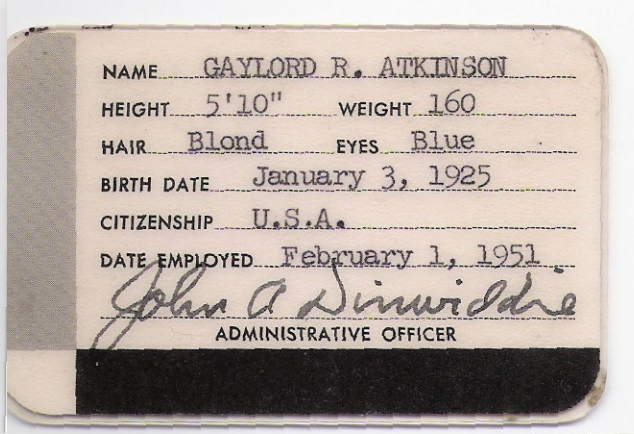
IDENTIFICATION CARD

NO. 32

G. R. Atkinson

MRI

MIDWEST RESEARCH INSTITUTE
425 VOLKER BLVD.
KANSAS CITY, MISSOURI



NAME GAYLORD R. ATKINSON
HEIGHT 5'10" WEIGHT 160
HAIR Blond EYES Blue
BIRTH DATE January 3, 1925
CITIZENSHIP U.S.A.
DATE EMPLOYED February 1, 1951

John A Dinwiddie
ADMINISTRATIVE OFFICER

SECTION 2 EARLY PROJECTS

My first work at MRI was related to the physical properties of fertilizer and how to keep it free flowing so as not to become a solid cake in the bag. This work was for the Spencer Chemical Company. The President of that company, Kenneth Spencer, was one of the six founders of MRI.

I then worked on the physical and chemical properties of vermiculite to determine factors that related to its use as a growing media. The photograph on the following page was related to this project.

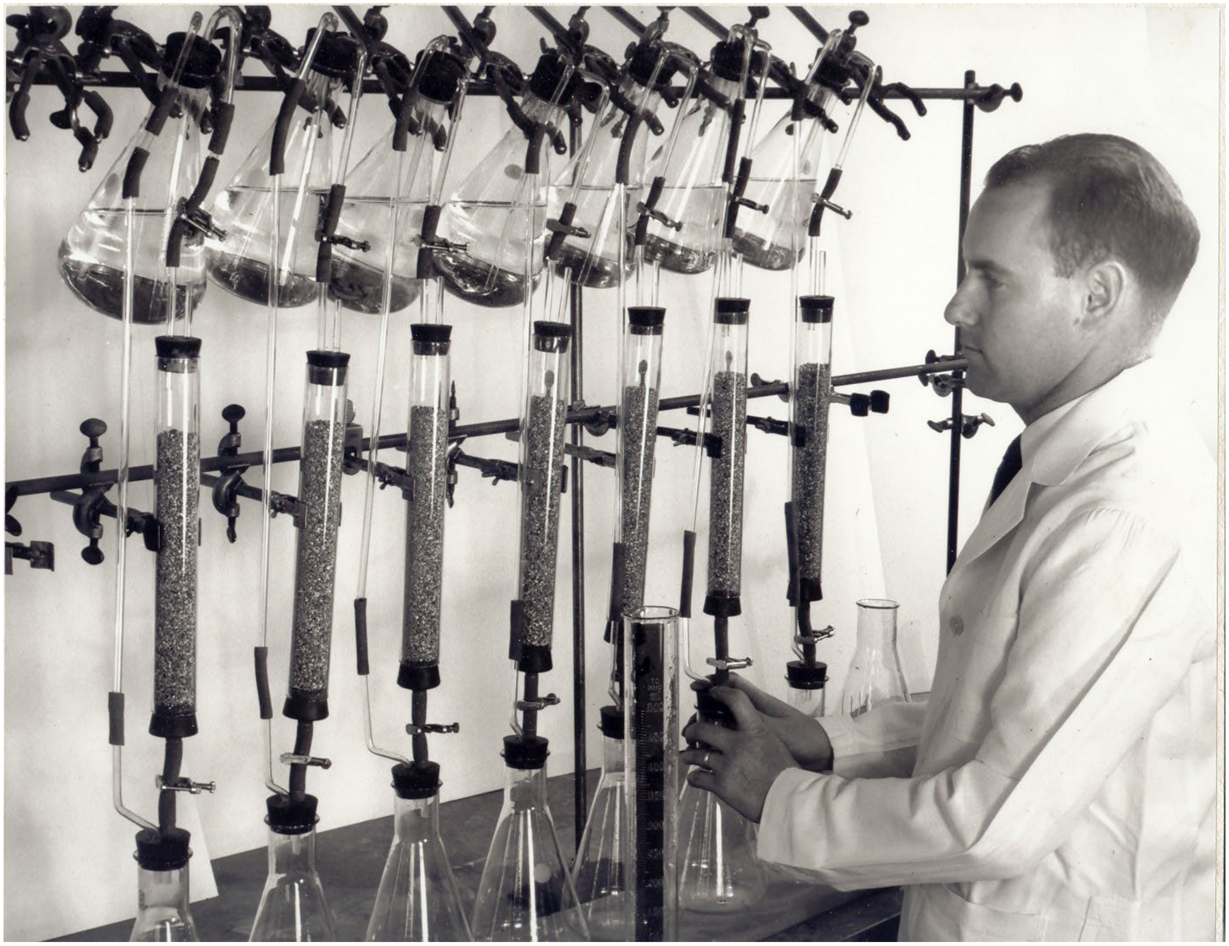
I next had a few small projects and a project with the Schaeffer Pen Company. This was before the days of ball point pens, and the standard fountain pen was causing problems as they tended to leak during air travel. The leakage was due to the change of atmospheric pressure as the plane changed altitude. As ink was depleted from the pen reservoir during usage, it was replaced by air. The volume of the air changed with change in atmospheric pressure. If the pen was positioned point down, the ink would be in the bottom of the reservoir and the air trapped at the top. With a decrease in pressure, the air would expand and force ink out which would result in leakage.

Schaeffer wanted to explore the development of a capillary pen, where the ink would be held in a reservoir by capillary forces. The principle would be to have a reservoir with capillary spacings from fairly large near the point and decreasing in size further from the point so the capillary attraction for the ink would be equal to, and opposite to, the force of gravity for any amount of ink that was in the reservoir. The reservoir would not be sealed and thus would have no trapped air and would be unaffected by pressure change. We developed a prototype capillary pen that worked fairly well, but about that time the ball point pen was replacing the liquid ink pens and Schaeffer lost interest in further development.

MRI worked for The Folgers Coffee Company during development of the process for making Instant Coffee. I was involved with some of that work. More about this later.

I next had a project for the Department of Agriculture. The project was to determine the solubility of about 20 different sugars, in six different solvents at four different temperatures. They wanted this information in order to develop procedures to extract and purify these various sugars for commercial use. With present day instrumentation, the determination would not be very difficult, but with the instrumentation available in 1955 it was very difficult. However we did obtain fairly good solubility values.

My assignment to the next project was fairly much by chance but indirectly related to the sugar solubility project. In the fall of 1955 two men from the Central Intelligence Agency (CIA) came to MRI and asked for a proposal for work on the properties of cellulose. They also asked for names of individuals who would be assigned to the project in order to investigate them for classified (secret) clearance. Cellulose is a polymer of glucose, a common sugar. My experience with the properties of sugars was the closest of anyone at MRI to that of cellulose and, unknown to me at the time, my Section Head (my boss) Howard Gadberry, and I were selected for the Project.



Apparatus that I set up for extracting soluble components from vermiculite in work relating the properties of vermiculite as a plant growing media. This picture was taken in 1952.

A few months later, in April 1956, I was told that I had been selected for a new project and that the Client would come for a kick-off meeting in a few days. In that meeting we learned that the cellulose was paper (paper is primarily cellulose) and the real subject of the project was to determine the mechanism of a procedure using an iodine solution for the detection of secret writing.

But before we go on to secret writing let me tell you about instant coffee. When I was in the army during WWII, “B” and “C” food rations contained packets of instant coffee. “B” rations came in a box slightly larger than a Kraft Macaroni and Cheese box. “C” rations were in cans. Some of the cans contained various dry material like crackers, instant coffee, cigarettes, etc. The coffee, as I remember, was made by Nescafe’. It was not very good and American service men and women associated instant coffee with “bad” coffee. There is less than a gram of solids in a brewed cup of coffee. This is about a fourth of a teaspoon. To provide more volume, Nescafe coffee was diluted with dextrins. Dextrin is a mixture of polysaccharides (polysugars) somewhat like starch, but with lower molecular weight and is much more soluble. One source of dextrin is to partially hydrolyze starch. It is essentially tasteless and its presence was not the reason for the poor quality of the army’s instant coffee but it came to be blamed for it.

After the war, Maxwell House put out an instant coffee that was fairly good but it took only about a fourth of a teaspoon to make a cup. Most people put in far more than that and it produced a very strong bitter coffee. It needed to be diluted. Dextrin was the obvious choice but unfortunately Maxwell House had advertised their instant coffee as 100% pure coffee with nothing added. They advertised that it was unlike the coffee you drank in the army with added dextrin. Their advertising put them in a corner. So what was their solution? They hydrolyzed the coffee grounds to produce a soluble dextrin-like material. So their instant coffee, diluted with hydrolyzed coffee grounds, was still “100% coffee with nothing extra added.” So Folgers had to follow and also hydrolyze their coffee grounds. MRI helped develop the Folgers’ procedure for the hydrolysis. It required a high temperature and high pressure with expensive equipment. I was involved in the analysis of the hydrolyzed material.

Readily available dextrin would have been just as good and cheaper but Maxwell House’s advertising dictated to the marketplace that the product be 100% coffee with nothing added.

SECTION 3 MY WORK WITH THE CIA - AN OVERVIEW

I was selected for the CIA project because of my work with sugars, but why did the CIA select MRI? That is an interesting, chance story. We need to go back to about 1927. Allen Gold and Perry Bistrup were high school students in Sedalia, Missouri. After high school the two friends both went to the University of Missouri (MU) and each received a degree in Chemistry. During WWII, Allen was in the Office of Strategic Services (OSS) and was involved with secret writing. After the war he returned to MU and earned a PhD in Chemistry. He joined the CIA when it was formed. His friend, Perry Bistrup, was one of the first MRI employees.

Allen Gold was one of the two men that came for the kick-off meeting mentioned above, and the one that would eventually be my Client contact for several years. Allen knew about MRI, because he knew Perry, but perhaps the real reason MRI got the project was that Allen's ageing mother still lived in Sedalia and with a project in Kansas City, Allen had the opportunity to visit his mother on trips to MRI. All of our project meetings at MRI were held on either Friday or Monday so Allan could spend the weekends in Sedalia. So that is how we <u>got</u> the project, but we <u>kept</u> it by doing good work.

Allen Gold and Dr. Max Thornton, MRI Vice President in charge of the technical divisions, got along very well. At one meeting near the end of our second year, Allen gave a rather long and detailed discussion to Dr. Thornton about budget problems and that all of the contractors, including MRI were going to have to adjust. Dr. Thornton sat there nodding his head. Then came the punch line. "So MRI will need to increase its effort." Dr. Thornton nearly fell out of his chair. Allen had set him up beautifully. Allen went on to explain that with limited funds, you concentrate the funds that you have where the best work is being done.

You may be surprised at the importance of secret writing during WWII and throughout most of the "Cold War." Nearly all of the information gathered (by spies) was sent back by secret writing and this continued until the mid 1980s when electronic means finally took over. Even then, secret writing continued to be important. In the 1960s and 1970s there was a Secret Writing Branch within the CIA of perhaps ten to fifteen individuals, and about a hundred others working at about eight contractor facilities, all working on secret writing. Secret writing was very important to the CIA at that time. MRI was the *only* contractor working on the development of procedures for the *detection* of secret writing. All of the others were working to develop new systems and for the support of existing systems.

During World War I and again in World War II, most secret writing inks consisted of very dilute aqueous solutions of inorganic compounds such as compounds of copper, nickel, and many others. The messages were recovered (made visible) by the application of solutions that were modifications of analytical chemistry reagents commonly used to detect these compounds. Thus the secret writing systems were modifications of commonly-used analytical chemistry materials and reagents.

During WWII a detecting solution was developed that detected not the secret ink, but the affect of the water in the ink on the paper. This solution detected any writing made with aqueous (water-based) inks. It was general and produced high contrast images. Thus even if you just wrote with water, that writing could be detected. I wrote test messages with distilled water in the 1950s and found a set of those samples shortly before I retired forty years later. I examined a few of the papers with distilled water writing and detected them with high contrast forty years after they were prepared. Our first project was to determine the mechanism of this detection.

The work leading to the understanding of secret writing detection using iodine took place during the first two-year project. Many projects generally related to secret writing continued until about 1990. Most of my work from 1956 until 1972 was related to secret writing. In 1972 I became project leader of a series of general analytical projects. That series of projects included a wide range of subjects and some very interesting and challenging work.

Before continuing with more detailed description of my work, I think it would be well to give an overview of all of the CIA projects to be discussed. *On the following page is a flow chart of 90 projects grouped into 12 subject areas. Nine of the subject areas are related to secret writing.*

In this overview those 12 subject areas will be briefly described. For some of the subjects, this brief description will be all that need be said about the subject. For others, the subjects will be covered in greater detail in later discussion.

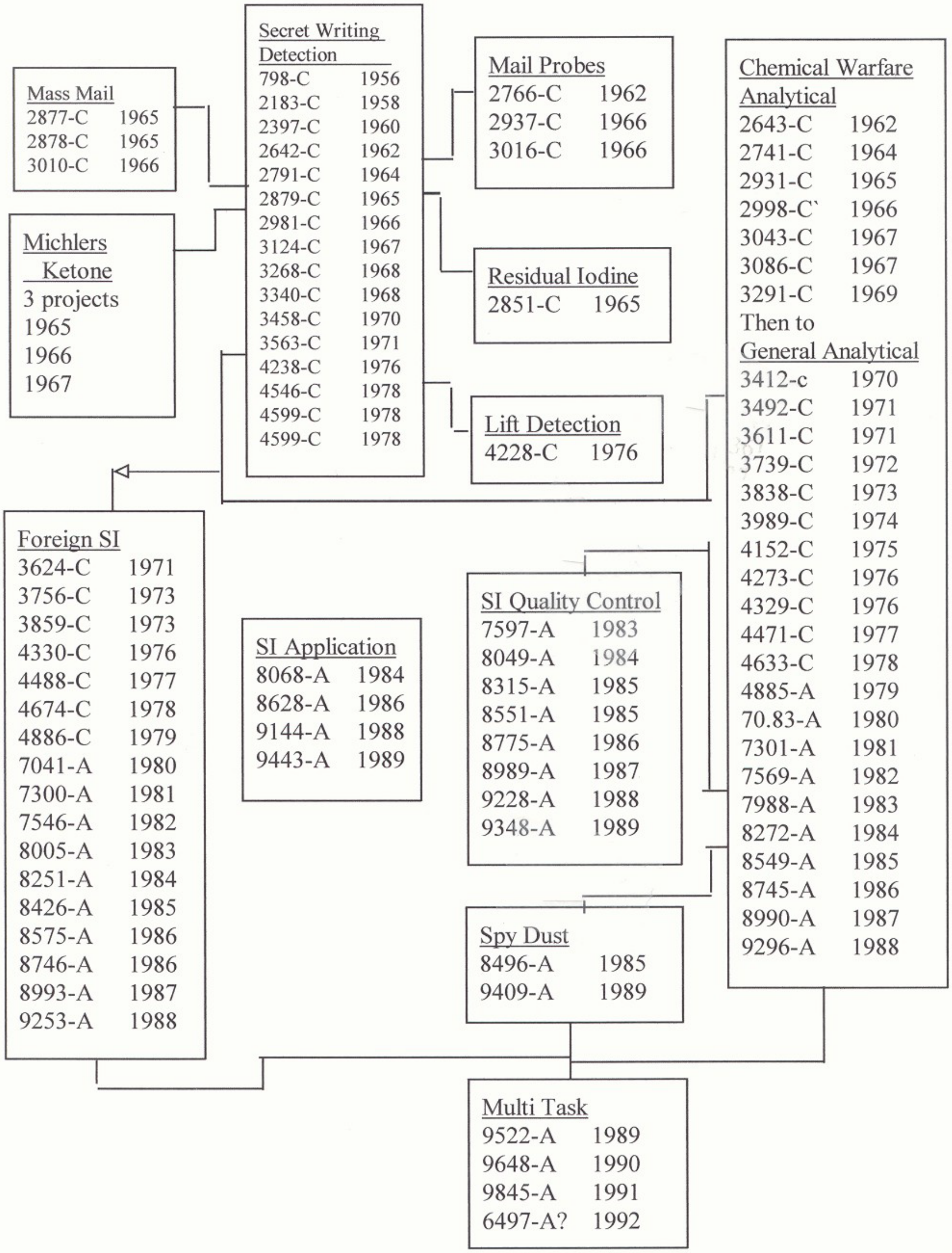
A certain amount of jargon and acronyms are in use and will be included in this and following sections. They are defined as follows:

Jargon and Acronyms

COTR	Contracting Officer's Technical Representative. This was the CIA employee that I reported to. The Contracting Officer had the ultimate authority, but in practice the COTR ran the projects. The Contracting Officer dealt with the MRI Contracts Department and I was seldom involved in this part of the work.

Secret Writing Related

Screed	As a verb: the act of writing a secret message. As a noun: the secret message itself, or the letter containing a secret message.
SW	Secret Writing
SI	Secret Ink
Recovery	The procedure used by the recipient of a secret message to make it visible. Recovery is when the recipient knows that he or she has a secret message and has the procedure and specific material needed to make it visible.
Detection	The procedure used by a censor looking for a secret message who has no knowledge as to whether a message is present or what secret ink was used if there is. Most of my work in secret writing was related to detection.



Mass Mail

2877-C	1965
2878-C	1965
3010-C	1966

Michlers Ketone

3 projects
1965
1966
1967

Secret Writing Detection

798-C	1956
2183-C	1958
2397-C	1960
2642-C	1962
2791-C	1964
2879-C	1965
2981-C	1966
3124-C	1967
3268-C	1968
3340-C	1968
3458-C	1970
3563-C	1971
4238-C	1976
4546-C	1978
4599-C	1978
4599-C	1978

Mail Probes

2766-C	1962
2937-C	1966
3016-C	1966

Residual Iodine

2851-C	1965

Lift Detection

4228-C	1976

Chemical Warfare Analytical

2643-C	1962
2741-C	1964
2931-C	1965
2998-C`	1966
3043-C	1967
3086-C	1967
3291-C	1969

Then to

General Analytical

3412-c	1970
3492-C	1971
3611-C	1971
3739-C	1972
3838-C	1973
3989-C	1974
4152-C	1975
4273-C	1976
4329-C	1976
4471-C	1977
4633-C	1978
4885-A	1979
70.83-A	1980
7301-A	1981
7569-A	1982
7988-A	1983
8272-A	1984
8549-A	1985
8745-A	1986
8990-A	1987
9296-A	1988

Foreign SI

3624-C	1971
3756-C	1973
3859-C	1973
4330-C	1976
4488-C	1977
4674-C	1978
4886-C	1979
7041-A	1980
7300-A	1981
7546-A	1982
8005-A	1983
8251-A	1984
8426-A	1985
8575-A	1986
8746-A	1986
8993-A	1987
9253-A	1988

SI Application

8068-A	1984
8628-A	1986
9144-A	1988
9443-A	1989

SI Quality Control

7597-A	1983
8049-A	1984
8315-A	1985
8551-A	1985
8775-A	1986
8989-A	1987
9228-A	1988
9348-A	1989

Spy Dust

8496-A	1985
9409-A	1989

Multi Task

9522-A	1989
9648-A	1990
9845-A	1991
6497-A?	1992

This is a list of the projects that are included in this write up. Individual projects are designated by Project Number and Division designation where "C" is for Chemistry and then after a re-organization "A" for Analytical Chemistry. The year is the date that the project started.

LIST OF SECRET WRITING RELATED PROJECTS

Secret Writing Detection: These projects involved the development of methods for the detection of secret writing (SW), both ours and the opposition's. The stated purpose of this work was to determine how vulnerable our secret messages would be to chemical censorship by the opposition. By law United States could not censor foreign mail (or domestic mail). During the period from the mid-1950s to late-1980s there were continuing improvements in both techniques and materials to produce secret writing and methods for the detection of secret writing. We would develop new methods of detection and then we, and other contractors, would work to find a way to defeat those methods. The detection of secret writing was a major part of my career and it will be covered in considerable detail

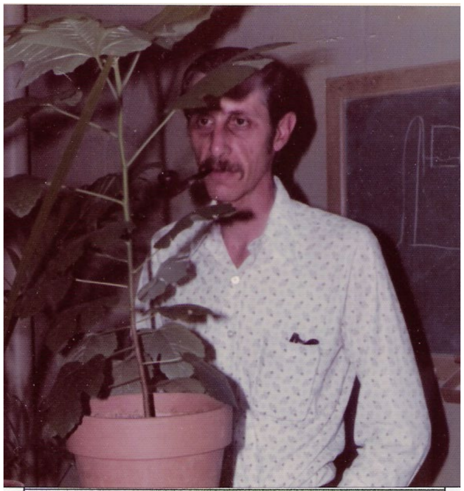
Mail Probes: It was known that most of the mail going to, or coming from, the Soviet Union, East Germany and China to-or-from the United States was being censored, but what was being done during the censorship process was unknown. There was a wide difference of opinion as to how secure our secret messages were. These projects were to design probes, or specially prepared letters, to send through opposition mail channels. The letters would them be recovered and examined to determine what had, or had not, been done to them.

Mass Mail: The results from the mail probes provided evidence of wide-spread censorship of mail from China. Thousand of letters that had come out of China which, for some reason could not be delivered to the addressee, were obtained from the "Dead Letter Office" of a friendly foreign country, since laws in the US prohibit the examination of such letters sent to-or-from the US. We learned a considerable amount about the extent and methods of Chinese censorship.

Residual Iodine: If a document is examined with iodine vapor (either at room temperature or hot) residual iodine remains on the document. This project was to determine if that residual iodine could be removed to below detectable levels. Most of the residual iodine is probably in the form of an iodide salt rather than free iodine. We found that essentially all of the iodine was removed if the document was placed in a hot chamber with high humidity and ozone. The ozone converted the iodide back to free iodine which was volatile in the hot humid conditions.

Michler's Ketone: Michler's Ketone is a material that was found by another contractor to detect some forms of secret writing. MRI was asked to investigate the material, determine the mechanism of the detection and in following projects look for other, possibly similar compounds that might detect secret writing. I was not involved with the first two of these three projects and they had been badly managed. What should have been an easy successful project produced no useful results because of preconceived conclusions, incompetence, and misdirection of the experimentations. This work will be covered in considerable detail later.

Lift Detection: In the late 1960s, MRI developed a powerful procedure for the detection of the secret writing then in use. The first step of this procedure was to transfer some of the ink of the secret writing to a very clean plastic film. This was done by pressing the film surface against the document being examined and was known as "making a lift." The focus of this Lift Detection project was to develop a probe to be sent through opposition mail channels to determine if the opposition was making lifts. Our earlier probes had obtained almost no evidence of censorship methods in Soviet and East German mail channels but they would not have detected whether or not a lift had been made. If we determined that they were making lifts, it could be concluded that they had developed a censorship technique similar to the one that we had developed**.**



Paul Seiwald
This is the only picture I have of Paul. Paul did excellent work on several projects.
I do not remember the significance of the plant.

Paul Seiwald conducted the work on this project and devised an excellent procedure to be used in a series of mail probes. The mail probes would have provided definite evidence as to whether lifts had been made and this would be very important information. We submitted a report on the procedure to the client which was then turned over to Operations. They modified a critical step in the procedure, got poor results in their trials, and abandoned the effort. We were very frustrated and disappointed.

Foreign Secret Ink: By 1971 we had developed a secret writing detection system that detected most of the secret writing used by either the United States or the Soviet Union. The objective of this series of projects was to isolate and chemically identify the foreign secret inks. We identified dozens of Soviet Block secret inks and, in many cases, concluded how they were being made visible. Again, Paul Seiwald conducted these projects for several years until he left MRI.

SI Quality Control: Each of the numerous secret writing systems in use required a different compound. Typically these were organic compounds with molecular weights of from 200 to 300. These compounds were made by different contractors. The purity of the compounds supplied by the contractors was, at times questionable and purity was very important.

We received several bulk SI samples for analysis under a general analytical project. We purified several of the impure materials. In this series of projects we received samples of all of the compounds produced by other contractors to determine their purity.

SI Application The objective of this series of projects was to be able to write secret messages with a computer printer. Special inks were made and computer ink cartridges were modified to print secret ink.

ANALYTICAL RELATED PROJECTS

The above mentioned identification of foreign secret inks and the SI quality control projects each involved analytical chemistry, but I have included them with secret writing. The following analytical projects are not related to secret writing.

Chemical Warfare Analytical: I had nothing to do with these projects at MRI that started in 1962 but mention them because I got involved 10 years later. These projects evolved into the General Analytical projects described in the next paragraph. The objective of the projects was to develop a facility for the quick analysis of samples related to chemical warfare. Most chemical warfare agents deteriorate rapidly when exposed to the environment and samples obtained following the use of the agents may contain little or none of the original agent. The samples may contain only degradation products and the chemical analysis and the interpretation of the results becomes complex. The analytical facility was developed and operational samples received. The group received a Presidential Citation for the identification of the chemical warfare agent used in a terrorist bomb that was exploded in the Middle East in about 1965. The facility, including the individuals and equipment, became a good general analytical facility for the analysis of samples including samples not related to chemical warfare. The emphasis of the facility gradually shifted from chemical warfare-related work to general analytical and numerous diverse samples were received for analysis.

General Analytical: I was involved in this series of projects which evolved from the Chemical Warfare Analytical projects mentioned above. Over the years more than a thousand samples of very diverse materials were received and analyzed. I helped in the analysis of some of the samples starting in about 1970 and became the Project Leader of the projects in 1972 when Dr. Don Rosebrook, who had been leading the projects, left MRI. Many of the samples were one-of-a-kind, and unrelated to other samples. Others consisted of a series of related samples. For example, we received numerous samples of explosives from letter bombs to analyze and from the results concluded which bomb explosives came from the same batch. As I recall, there were two or three different batch composition types, some yellow and some black, with minor variations between batches within a type.

The largest number of samples comprising a series of related samples concerned tracking. These were samples of materials that the Soviets were using to monitor the activities of Americans and our Soviet spies. This sample series started in about 1970 and continued until I left MRI in 1996. The highlight of the series came in the mid-1980s when the use of a particular tracking material that we had identified became the subject of a White House press conference where the U.S. accused the Soviet Union of exposing our embassy personnel to a potentially dangerous chemical. That chemical, which I named NPPD, an abbreviation of it’s chemical name, was called “Spy Dust” in the newspapers. *You can find copies of many of these articles in the appendix.*

Spy Dust: Spy dust was the name given to materials that the Soviet Union used to monitor the activities of certain US and Soviet Citizens. It was a very important operational and political issue and MRI and I were very involved.

Multi Tasks: In 1989, the ongoing work in the various previous projects were groups into a single multi-task contract.

SECTION 4 SECRET WRITING

SECTION 4.1 A BREIF HISTORY OF SECRET WRITING

The history of secret writing goes back hundreds, perhaps thousands, of years. It was widely used by Italian merchants hundreds of years ago. Later, the use of secret writing to send messages was very important during World War I (WW I – 1914 to 1918) and World War II (WWII – 1939 to 1945), and there was little improvement in the methods used from WW I to WWII. Secret writing systems were generally modifications of analytical chemistry reactions. The inks were generally dilute water solutions of any of various metal salts and the messages were recovered by the application of a reagent that would react with that metal to produce a visible image. Other secret inks were materials that scorched, or caused the paper to scorch when it was heated. Inks that could be recovered by scorch were milk, sugar, urine, alum and others.

Chemical censorship consisted of applying stripes of analytical reagents across the paper in the hope that the writing would appear. This procedure destroyed the sample which was probably a personal letter. Some countries had teams of forgers that could copy the letter to look like the original and this copy would be sent on while the original was destroyed by the censorship. The photograph on the following page is of a letter where the censor goofed and sent on the original letter that had been examined rather than the forged letter.

At some time, I do not know when but I believe it was during WW I, a censorship procedure was developed that used photographic negatives. These were the glass plates coated with the photographic emulsion that was in use before films were introduced. In this technique, the negative was first exposed to a low level of light. It was then dipped in water and allowed to drain but while remaining damp it was pressed against the document being examined. If the document contained secret writing of a metal salt, that salt would partially dissolve in the moisture and diffuse into the photographic emulsion. The photographic plate would then be placed in a bath of photographic developer. The developer was special in that it did not contain the usual buffers to control the pH of the solution. Photographic development is a chemical reaction where silver bromide is reduced to become silver metal. The rate of this reaction is extremely dependent on the pH of the solution. Ordinary developers are buffered to maintain a constant alkaline pH. Metal salt from the secret ink is likely to influence the pH of an unbuffered developer and therefore alter the rate of development. The secret writing would therefore be developed slightly faster or slower than the background area depending on the pH shift caused by the salt of the secret ink. The development would be observed under a safe light and if an image appeared the development would be stopped by placing the plate in an acetic acid "stop" bath

In addition to the possible pH shift, the salt could be an oxidation of reducing agent which could also influence the image development. Thus a detect would be obtained if the secret ink influenced the pH or the reducing potential of the developer.

This is a copy of a censored letter where the censor apparently made a mistake and sent this censored letter rather than a forged copy of the letter. The letter was photographed and then the chemicals in these stripes were analyzed and the findings noted on the photograph.

This photographic detection procedure was no longer used by the time that I was involved with detection but had evolved into a procedure that was very important for my work. At some time it was observed that some secret messages could be seen on the photographic plate before the plate was treated with the developer. By 1956 a procedure known as "photo plate" was in use.
A photographic film, whether it is on glass or a plastic base consists of silver bromide and other additives in a solution of gelatin. This solution is applied to the glass plate or plastic film to produce the light sensitive film. For the "photo plate" procedure, glass plates were coated with just the gelatin without the silver bromide. In use, the gelatin plates were dipped in water for about a minute, and then allowed to drain and dry until the surface became tacky. It was about as tacky as Removable Scotch Tape. The document being examined was then pressed against the gelatin surface and removed. If the document contained particles of secret ink, some of the particles would be transferred to the gelatin. The gelatin surface was then examined with a bright light against a dark background. Any secret ink particles on the surface would scatter the light and be visible.

Sometime during WWII, an iodine solution was developed that would detect any secret writing prepared with an aqueous ink. The water of the ink altered the surface structure of the cellulose of the paper and this was detectable. My first project was to explain the mechanism of this detection. Because of this iodine detection, two things happened. The first was that any secret message prepared with an aqueous ink needed special treatment to minimize the probability of the iodine detection. After the message was written, the sheet needed to be steamed and then pressed between blotters for a few hours. Even with the pressing, the sheet tended to appear slightly wrinkled indicating that it had been moistened. Second, there was an attempt to develop writing procedures that did not include aqueous inks.

The Client had periodic contractor meetings with representatives of each of the contractors working on secret writing. At the first meeting that I attended in 1957, one of the contractors was working on solid writing devises to avoid the action of water on the paper. For example they had made books of matches where the head of certain matches in the book contained secret ink. Combs were made with certain teeth contained secret ink. The selected teeth were to be broken off and used as a pencil to write the message. Several other items were also being made.

Our first report on the iodine solution detection, which was submitted just before the meeting in 1957, contained a short paragraph that changed the direction of the secret writing program. We reported that friction on a paper surface was readily detected, but pressure was not. A 1 mm glass rod with a smooth rounded tip moved over a sheet of paper with a downward force of one gram was readily detected but a grid pressed on the paper with a force of 1,000 pounds per square inch was not detected. With that report, the Client stopped work on the secret ink pencils and started making secret ink carbon paper. These papers looked like ordinary paper but were impregnated with secret ink. They were used like an ordinary carbon paper.

There were lots of problems in making the carbons. For example a very small amount of the secret ink (SI) needed to be placed on the sheet. At first it was decided to dilute the SI with an inert material to better obtain a uniform coating. Talc is sometimes used as a filler in paper and it was considered inert. Since it is often in paper, if it were found in an analysis it would not be cause for suspicion. So sets of SI carbons were produced that included talc. These carbons produced secret messages that gave good recoveries and passed most of the censorship tests. Then a test message made with one of these carbons was examined with the gelatin plate, or "Photo plate." When the photo plate was examined with a bright light against a dark background, the talc in the message was detected like a neon light. Photo plate became a critical test for the carbons to pass. Forget the talc, any particle larger that the wavelength of light was detected. Eventually the gelatin photo plate was replaced with a sheet of polystyrene which produced even more sensitive detection. A polystyrene sheet was pressed against the document being examined and then the sheet was observed with an intense collimated light against a dark background.

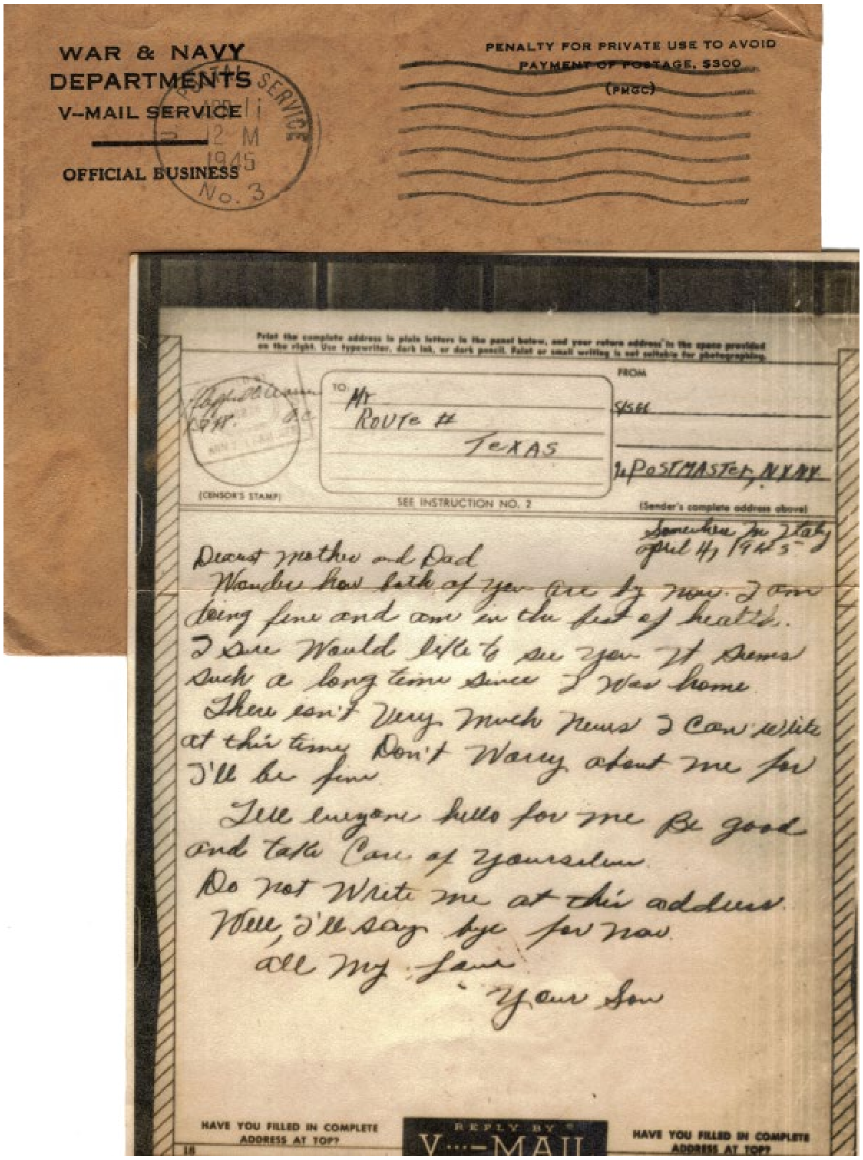
Production of SI carbons became a complex operation. It required that the SI particles be ground to sizes smaller than the wavelength of light, and distributed uniformly over the surface of the paper. Typically, in the 1950s and 1960s a carbon sheet contained about 3mg of SI on each side. By the 1980s the concentration had been reduced to less than 1 mg per side. Nearly all of this work was done by Arthur D. Little (ADL) of Cambridge Massachusetts.

ADL was the only non-government group working on secret writing during WWII. They came up with "V-Mail" as a way to defeat secret messages. V-Mail was widely used during the war. It consisted of paper forms with an area for writing a letter. The letter was then folded to form an envelop, addressed, and mailed. The envelops went to a photographic processing center where they were opened, and photographed on 16 mm film. The roll of film was then taken overseas to other processing centers where the message was photographically printed and sent on to the addressee. V-Mail not only defeated secret writing, it greatly reduced the bulk of the mail going over seas and was faster than regular mail as the roll of film was generally flown overseas while other mail went by boat. The amount that could be written was limited. As I remember the space for the letter was about 6 X 8 inches and this was printed out about half that size. The next page is a copy of one V-Mail that I sent home during the war while I was in Europe.

During WWII ADL developed a battery of censorship reagents and procedures as well as many secret writing systems. After the war all of this information was written up in a large report and archived by the government. A few years later when the CIA was formed, a search was made for this report. It was never found, but the story goes that a copy of it was purchased on the black market in Eastern Europe.

ADL was the only contractor working on secret writing from shortly after the formation of the CIA until 1956. In fact several of the early CIA employees came from ADL. ADL remained the main contractor for developing new secret writing systems the whole time I was involved with secret writing.

This is an example of what V-Mail looked like. This is the approximate size of the original.



WAR & NAVY
DEPARTMENTS
V–MAIL SERVICE

OFFICIAL BUSINESS

POSTAL SERVICE
APR
12 M
1945
No. 3

PENALTY FOR PRIVATE USE TO AVOID
PAYMENT OF POSTAGE, $300
(PMGC)

Print the complete address in plain letters in the panel below, and your return address in the space provided on the right. Use typewriter, dark ink, or dark pencil. Faint or small writing is not suitable for photographing.

TO: Mr
Route #
Texas

FROM
S/Sgt
c/o Postmaster, N.Y. N.Y.

(CENSOR'S STAMP)

SEE INSTRUCTION NO. 2

(Sender's complete address above)

Somewhere In Italy
April 4, 1945

Dearest mother and Dad
Wonder how both of you are by now. I am
doing fine and am in the best of health.
I sure Would like to see you It seems
such a long time since I Was home
There isn't Very much News I Can write
at this time Don't Worry about me for
I'll be fine

Tell everyone hello for me Be good
and take Care of yourselves
Do not Write me at this address.
Well, I'll say bye for now.
All My Love
Your Son

HAVE YOU FILLED IN COMPLETE ADDRESS AT TOP?

REPLY BY V-MAIL

HAVE YOU FILLED IN COMPLETE ADDRESS AT TOP?

SECTION 4.2 "LIQUID IODINE" DETECTION SOLUTION

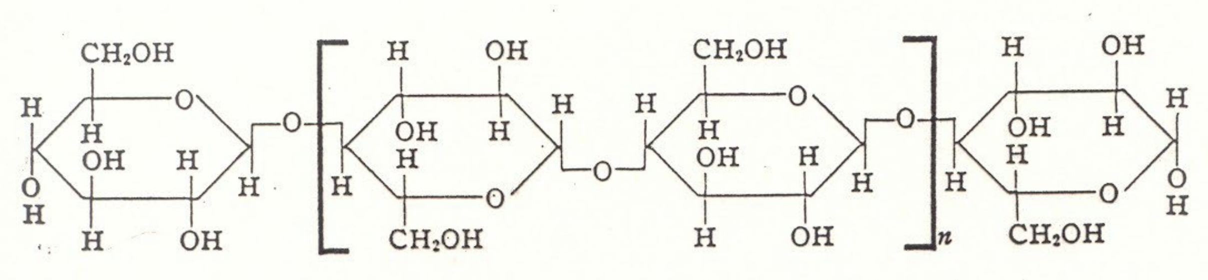
As I previously stated, our first assignment with the CIA was to determine the mechanism of the SW detection using the solution containing iodine There is a long history of the use of iodine vapor for examine documents for the presence of fingerprints and document modifications, as well as for the detection of secret writing. For example, for the detection of fingerprints, some iodine is placed in a chamber along with the document in question and the iodine vapors are absorbed by the skin oils of the fingerprint. Iodine is very dark and the resulting treatment generally produces good contrast images.

There are three techniques for detecting secret writing using iodine. They are:

1. The use of iodine in a closed chamber. This is known as "iodine vapor chamber".
2. The use of a solution of iodine, potassium iodide, calcium chloride in water. This is known as "The General Censorship Reagent" (GCR) or commonly as "liquid iodine".
3. The application of puffs of hot iodine vapors to the surface of the document. This is known as "hot iodine".

At first we were involved in work to understand the action of "liquid iodine" but we were soon working to understand the mechanism of "hot iodine"and after these were understood, how could the detection be defeated.

Liquid iodine detected any writing made with an aqueous ink, including distilled water.
It was soon readily apparent that the detection was the result of some change in the paper due to the action of the water of the ink. To understand the detection we needed to understand paper. Paper can be made from any plant material which contains cellulose. Wood, generally pine, is now the primary starting material, but centuries ago cotton from old rags was used. In China, rice straw was used. In the case of wood as the starting material, the wood is chipped into small pieces and then cooked with chemicals to dissolve and remove the resins and rosins and other non cellulose materials. The cellulose pulp is then mechanically "beat" in a water slurry to essentially separate the pulp into individual fibers.



Cellulose

As I stated earlier, cellulose is poly glucose and each glucose unit has six carbon atoms. Each glucose unit in cellulose has three hydroxyl groups (-OH). These hydroxyl groups are closely related to water (HOH) and during the beating, the hydroxyl units on the fiber surface attract water and become “hydrated”. Each surface hydroxyl becomes surrounded by, and loosely bonded to, a number of water molecules.

To make paper, this slurry of cellulose pulp is poured onto a moving wire screen. Much of the water passes through the screen and the fibers form a mat on the screen. Some the remaining water is sucked through the screen by vacuum and then the fiber or paper mat is passed from the wire screen to between two felt belts that take much of the remaining water that is not bonded to the cellulose hydroxyl groups. The paper mat next passes over a series of hot rotating drums to complete the drying of the mat and the mat becomes paper. This is a critical operation and most of the water that had been bonded with the cellulose hydroxyl groups is driven off by the heat. The cellulose hydroxyl groups now bond to each other by what is know as hydrogen bonding. These bonds give paper its strength. When paper is wet, the action of the water breaks many of the hydrogen bonds between the cellulose fibers and the hydroxyl groups again become hydrated. That is why wet paper has very little strength. When this wet paper is then dried at room conditions, most of the water evaporates but many of the surface hydroxyl groups remain hydrated and do not form hydrogen bonding with adjacent fibers.

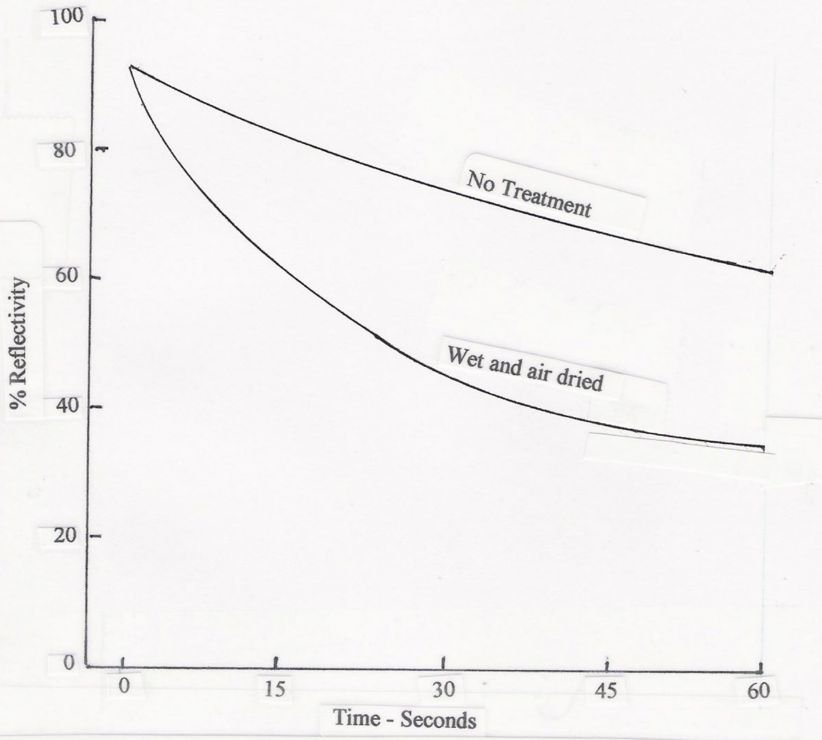
So what does this have to do with the detection of secret writing? When a secret message is written with an aqueous ink, the cellulose fibers that are wetted by the ink are partially hydrated, hydrogen bonds are broken and only partially reform as the ink dries at room temperature. The difference between the number of un-bonded hydroxyl groups in the written line and the background paper remains indefinitely.

As it turned out, the iodine detection solution is a reagent that reacts to form a colored complex with the free hydroxyl (OH) groups of the cellulose (those not hydrogen-bonded to neighboring hydroxyl groups) on the surface of the paper.

Several staining solutions are used, in the pulp and paper industry, to either identify the source of the fiber, (pine, oak, etc) or to determine the degree of “cooking” of the pulp during preparation to making paper. One of these stains is know as the “Graph C” stain and is a solution of zinc chloride, potassium iodide and iodine in water. This staining solution provides a variety of different colors for different fibers and is used to identify the source and past treatment of the fibers The solution that was so effective in the detection of the secret writing was a modification of that stain with the zinc chloride replaced with calcium chloride and the concentrations somewhat modified. It took us about a year to fully understand the mechanism of the detection but during that time we were also working on the “hot iodine” detection technique.

For secret writing detection, a highly colored complex is formed when the free cellulose hydroxyl groups combine with the iodine, iodide, and water. The color is a dark brown. As I said earlier, messages written in distilled water were detected with high contrast forty years later.

The rate and intensity of the color formation when the detection solution is applied to the paper is essentially determined by the temperature of the last drying of the paper. When the paper was made, it was dried at a high temperature. When the secret ink dried, it dried at room temperature. The difference in the rate of darkening of an untreated paper and an area of the same paper that had been wet and air dried is shown in a plot of the reflectivity of the two areas following the application of the detection solution.



Darkening rates as indicated by the surface reflectivity vs. time for a paper with no prior treatment and an area of the same paper that had been wet and air dried. These rates are as the background and the secret writing of an aqueous ink.

We had critics that questioned our proposed mechanism and so we continued our investigations. We did lots of work, had lots of fun and interesting experiences. I will briefly describe one experiment. It was based on how commercial dyes react with different fabrics which, like paper, are made of cellulose. We obtained a blue dye that will dye cotton to form a "fast" color that will not wash out, but this dye will not dye cellulose acetate (commonly known as "acetate"). We also obtained a yellow dye that will dye cellulose acetate but not cellulose. Cellulose acetate is formed when the free hydroxyl groups of paper that has been wet and dried at room temperature are then exposed to acetyl chloride vapor. There is almost no reaction on the original paper that had not been wet as most of the hydroxyl groups remained bonded to each other. So we prepared a message written with just distilled water, allowed it to dry, and then exposed it to acetyl chloride vapors to react with the free hydroxyl groups to form cellulose acetate. The sheet was then placed in a solution of a mixture of the blue cellulose dye and the yellow acetate dye. The results were spectacular. The writing was bright yellow against a dark blue background. I wish I had saved one of those samples.

Our understanding of how paper reacted to these and other reagents lead to a very interesting situation. Some early secret writing (using dilute blood, milk, saliva, urine and many other materials) could be recovered or detected by heating the sheet until the ink or the paper with the ink scorched. This produced dark writing against a lighter background. A devise was in use for the examining secret writing with new experimental secret inks to determine if they would be detected if they were subjected to scorching. This device was a metal bar ½ inch wide and about 4 inches long it was heated to a temperature that would produce a scorch. The heated bar had legs to support the bar about 2mm above the surface of the paper. If it was left on the paper long enough it would slightly scorch the paper and produce a half inch wide brown band.

We received many samples of various kinds for analysis from our client. One day we received a piece of paper with a faint ½ inch wide brown band on it. It looked very much like it had been scorched. We were asked to determine if this was a scorched area. When a paper is heated to a temperature that produces discoloration (scorch) some the hydroxyl groups of the cellulose are driven off, and the cellulose is permanently altered chemically Many of the hydroxyl groups are no longer present. We scorched papers to look like the sample, and found that the difference between a scorch and un-scorched area of a paper was drastically different when examined with either the iodine solution or the blue dye. If a paper with a scorched area is wet and then dried, the scorched area has few hydroxyl groups to hydrate and does not react with the iodine reagent to form the colored complex, but the area that was not scorched will react normally and darkens very rapidly. Thus, a scorched area is very readily detected by the iodine reagent with very high contrast as a light colored "resist" against a very dark background. Similarly the blue cellulose dye will not attach to the scorched surface where the cellulose has been damaged. And the scorched area remains light colored against a dark blue background. The "scorched" area from the client's sample was not detected by either of these treatments. We tested the Client's sample with two other reagents that also detected controlled scorch areas but did not detect the band of the Clients sample.

Our results were definitive. The brown band on the clients sample definitely was not scorch.

So what might it be?

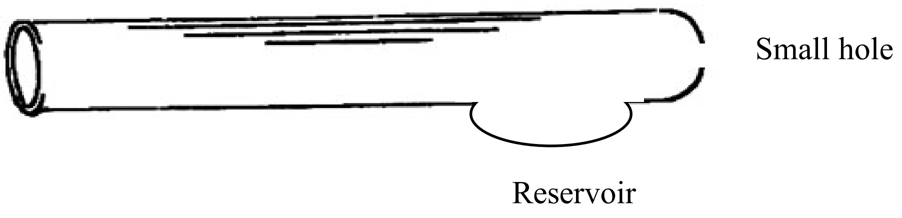
Early Scotch tape was ½ inch wide and books that were repaired with this tape aged to produce a brown stain on sheets even several pages away from the mended page. The same is true of the contents of an envelop that had been sealed with Scotch tape. I had some books that had been mended with Scotch tape years earlier that had brown coloration several pages away from the mended page. I sacrificed part of a page of one of the books and treated strips of it with both the iodine solution and the blue dye. The Scotch Tape stain was not detected with either treatment. We reported that the brown stain definitely was not scorch and was probably "Scotch, not scorch".

Several years later our COTR told us the following story. Portions of the "scorch" sample had been sent to four contractors. The other three contractors each reported that the discoloration was the result of scorch, and one of them, probably ADL, provided a detailed report giving the time and temperature that produced the scorch. Their report also stated that none of the secret writing using the secret inks that they had developed would be detected by this scorch treatment. Obviously, although each of the contractors had the iodine detection reagent and other reagents that we used, none had bothered to use them to examine their “scorched” sample. Our client would not answer my direct question “Did you know the dark band was from Scotch tape when you sent out the samples?” but I think our reputation was greatly enhanced from our reported results of the analysis of this sample.

SECTION 4.3   HOT IODINE VAPOR

Hot Iodine. The technique known as hot iodine was developed by the Chinese. It was an extremely powerful technique and, with a skilled operator, detected many of the secret writing systems in use by the United States. It was demonstrated to MRI in late 1956 and we were asked to determine how it worked.

The apparatus that we were shown for hot iodine was simple. An ordinary 6 inch test tube was modified by blowing a bulge in one side of the test tube to form a reservoir and then a small hole was blown in the end of the test tube. A crude depiction of the modified test tube is as follows:



In use, a small amount of iodine crystals was placed in the reservoir. A stopper with a glass tube through its middle, a rubber tube attached to the glass tube, and a squeeze bulb attached to the other end of the rubber tube, was placed in the mouth of the test tube. It took a bit of practice to effectively use the apparatus. The test tube was held with the index finger and thumb of the right hand with the squeeze bulb also held in the palm of that hand. The document to be examined was held in the left hand. The reservoir with the iodine was then heated in the flame of a Bunsen burner until the iodine melted at 113°C. At this temperature the test tube is fairly well filled with the dark purple iodine vapors. The apparatus is then positioned at an angle near the surface of the document and the bulb is squeezed to send a puff of iodine vapor against document. We later designed a modified apparatus that was not hand held and easier to use. The glass blower at the Chemistry Department at Kansas University made numerous of these “iodine puffers” for the Client.

A “hot iodine” detection of a secret writing message may appear in a variety of ways depending on the characteristics of secret message paper and the iodine puff. Writing with the same secret ink on the same paper might appear:

a. As dark lines against a relatively light background, known as a “positive detect”
b. A light lines against a dark background, known as a “resist”.
c. Momentary light lines against a dark background, known as a “flash resist” followed by a positive detect or a resist detect, or nothing at all. The flash resist lasted only a few hundredths of a second. For some secret writing the flash resist was the only detect to be observed, so it was important to watch for it.

The detect was generally high contrast but a very short duration. We were asked to explain these different detects. Explaining the "positive detects" was relatively straight forward and simple. Secret ink particles provided nucleation sites for the formation and growth of iodine crystals. Thus the dark lines against a light background.

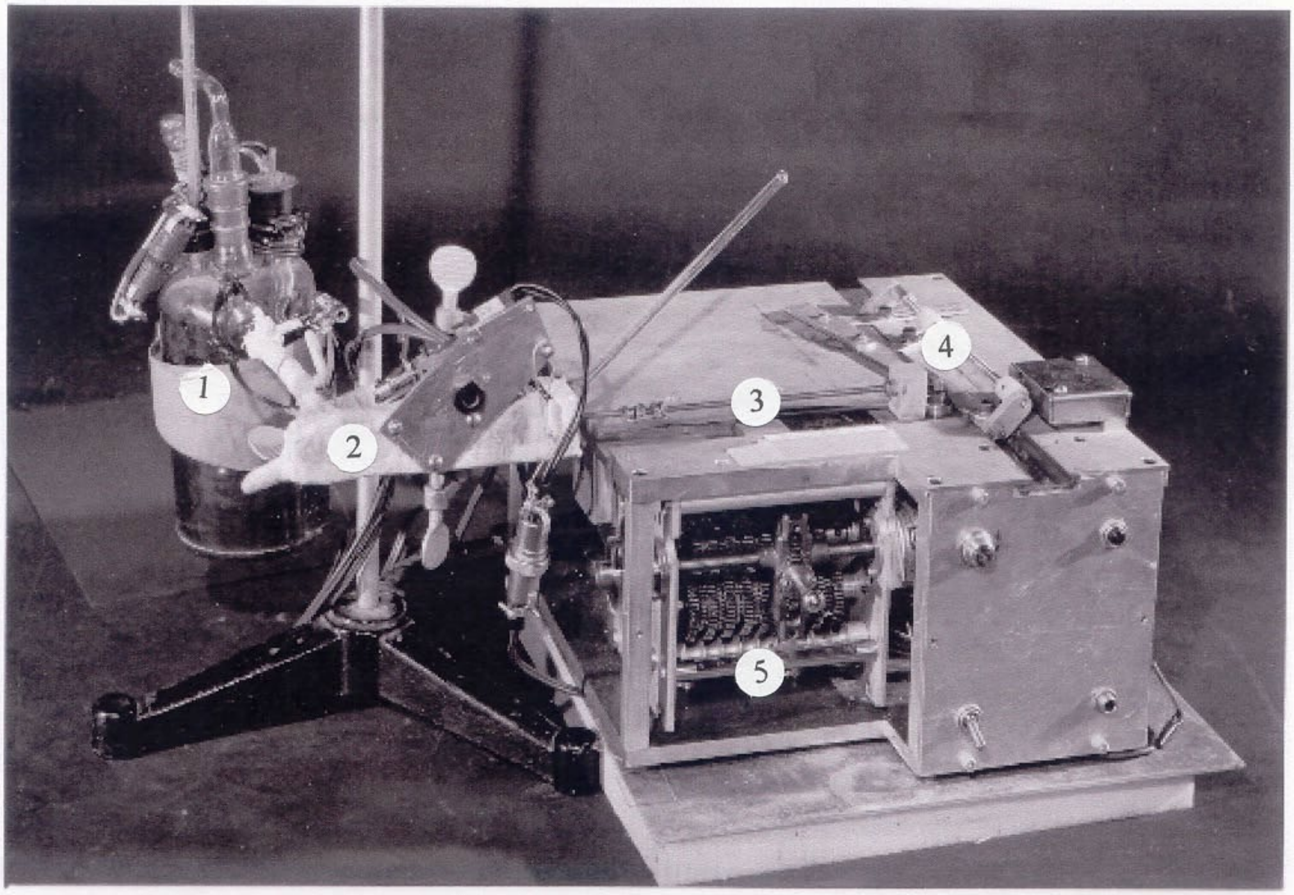
Explaining the other detects was not so simple and after much work with inconclusive results, we finally built a rather complex apparatus to deliver a controlled and programmed puff of iodine to the sample surface. The puff intensity and duration was controlled and the intensity could be changed during the puff from high to low or from low to high. The iodine puff was produced from a heated glass hypodermic syringe with the rate and duration of the puff controlled by cams that drove the syringe plunger. The puff was directed towards a sample that was positioned on a microscope stage with the microscope fitted with a motion picture camera. Pictures of detects were taken at 64 frames per second. Thus we could see the sequence of the detection at intervals of 1/64 of a second.

The apparatus used, and photographs of the detection are shown on the following pages. These photographs are the only ones that I have now. They are from the only roll of color film that I took. I had black and white pictures that showed the detect better but I did not keep them.

The resulting photographs showed that initially, with a moderately intense puff, the entire surface was coated with an opaque layer of super-cooled liquid iodine. This layer was very dark and also very unstable. If the secret ink provided nucleation sites, iodine crystals formed at those sites. At first the crystals were too small to have optical significance but iodine traveled from the surrounding unstable liquid layer to the stable crystals to create an iodine free halo around each crystal. These halos grew rapidly and very soon became the dominate feature of the lines of secret writing resulting in the "flash resist". This all took place on 3 or 4 frames of the movie film which represented less than one tenth of a second.

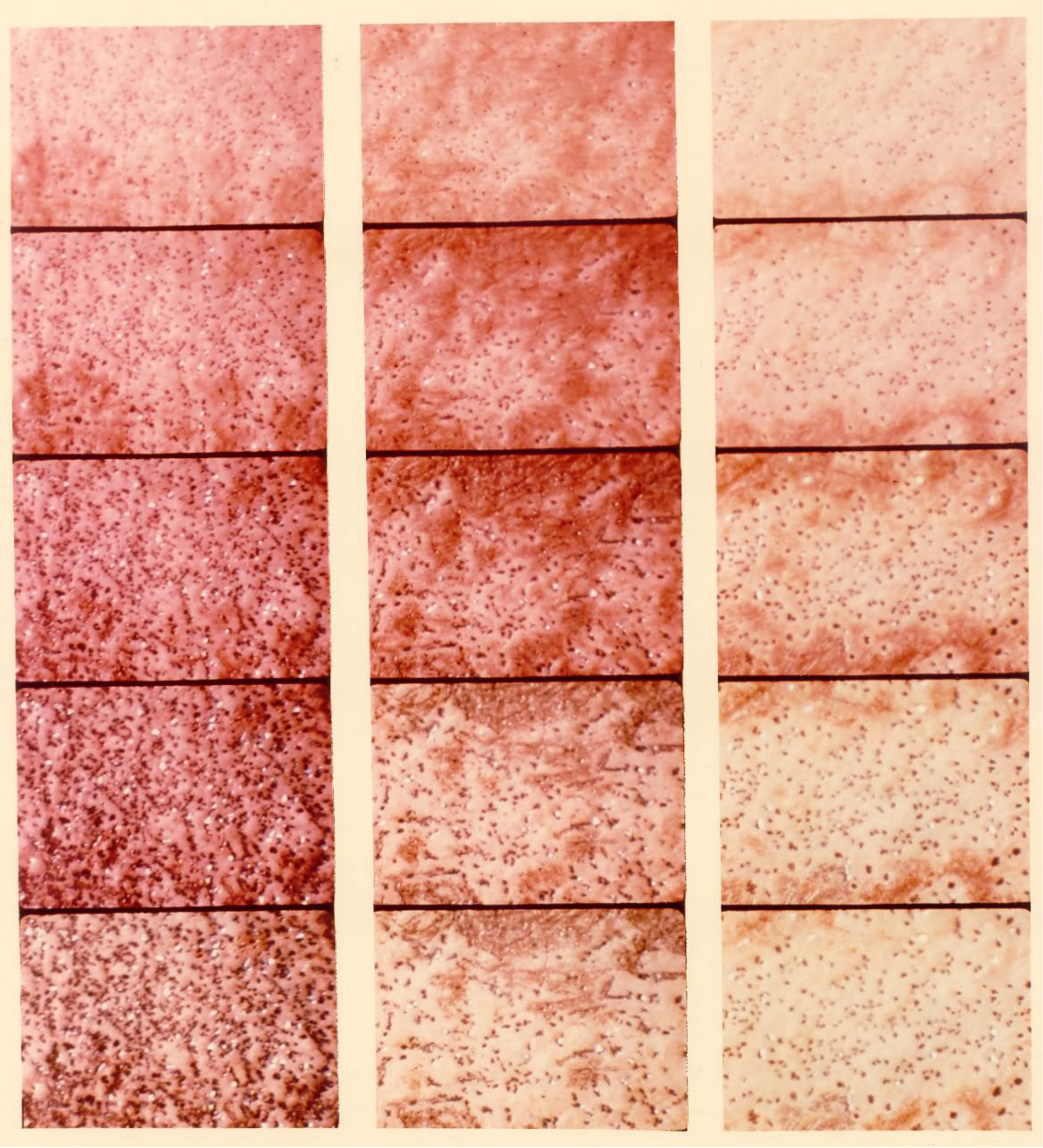
Now, if the iodine puff stopped, the layer of liquid iodine would rapidly evaporate from the surface. This would result in either, essentially, no further detect or a slight positive. If the puff duration was continued at a lower intensity, the liquid in the background would still be lost by evaporation, but the crystals in the line of secret writing would continue to grow, resulting in a positive detect. If the puff was maintained at a fairly high intensity, but for a short duration, the liquid layer would eventually crystallize to produce a dark background with the line relatively clear because of the dominants of the halos around the small iodine crystals. This is the resist detect.

Thus we explained the mechanism of the iodine detection methods. It was a lot of good work and getting the results was very satisfying.



APPARATUS FOR PRODUCING PROGRAMED IODINE VAPOR PUFFS

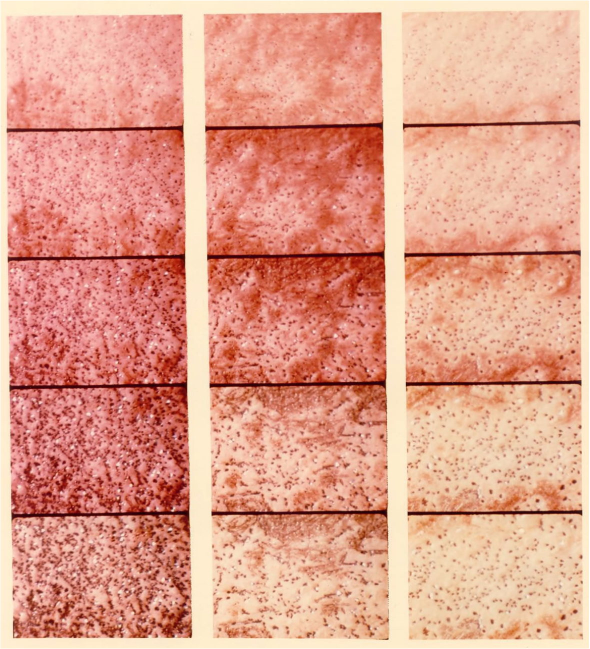
1 Iodine chamber maintained at a temperature of 125° C with heated transfer tube.
2 Heated block that contained a 5 cc glass syringe with a solenoid operated valve. The iodine puff was ejected from the tip on the left
3. Cam driven rod which was attached to the syringe plunger
4. Cam carriage that moved from back to front. Different cam shapes were used to provide controlled puff intensity profiles.
5 Gear train to provide different rates of cam carriage movement which determined the puff duration.

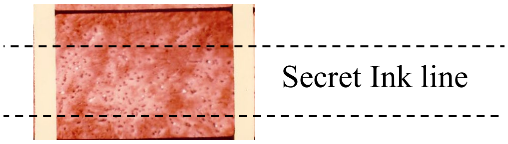


High speed motion picture views of Hot Iodine screed line detects. See next page for details.

Explanation of pictures on the previous page

Five consecutive frames taken at 64 frames per second of each of three Hot Iodine detects of screed lines. Each image represents an area of approximately 1 X 0.7 mm. The screed line runs horizontally and is about 0.5 mm wide so there is little of the background outside of the line shown. A lower magnification would have been better but unfortunately this is the only set of pictures that I saved.





The images in the middle set best show the hot iodine detection. This set was produced by an iodine puff of moderate intensity and short duration. By the second frame, the background is coated with a thin layer of amorphous iodine and iodine crystals have formed on the Secret Ink particles in the screed line. Halos have formed around each crystal. The halos form because the crystalline iodine has a much lower vapor pressure than the very unstable amorphous iodine and the iodine moves from the amorphous layer to the crystals. By the fourth frame the halos have grown to dominate the screed line. Some of the amorphous layer has crystallized. A small area of amorphous iodine remains near the center bottom of the frame. It is gone by the fifth frame and now all of the iodine is crystalline. Much of the surrounding background was coated with amorphous iodine and it crystallized/ It eventually crystallized rather than evaporated because of the duration of the puff. This resulted in a negative screed line with a dark background.

The set to the right was the result of a puff of low intensity and short duration and was a flash resist detect. The halo effect rapidly cleared the screed line while the background was momentarily dark. The short duration of the puff allowed the amorphous layer to evaporate rather than crystallize. You can see that the amorphous layer along the bottom of the frames is decreasing in intensity from the third to the fourth to the fifth frame would be gone by the next frame.

The set on the left is of a puff low intensity and extended duration.
Note that at 64 frames per second the five frames shown represent only about 7/100 of a second.

SECTION 4.4 ALTERNATE REAGENTS

Search for Alternative Reagents Is iodine the only material that can be used for secret writing detection or are there other materials that would do the same or possibly better?

We spent considerable effort looking for other materials and techniques. Since some secret writing used metal salts as secret inks we tried vapors of various metals and metal salts as a detecting agent. For this work, documents with lines of various materials were placed in a vacuum chamber and the test reagents evaporated from a hot tungsten filament. Gold, silver and copper uniformly coated the documents and gave no detects. Zinc was the only metal that gave detects and it was fairly good for the detection of various metals and metal salts. We got some interesting results but the procedure was not very practical and was abandoned.

Numerous organic compounds were tried. We used the modified test tube that had been used for hot iodine to test these compounds. Several of these compounds produced detects but most of them were white and could not be readily seen on white paper. A few of them were fluorescent and the detects were seen using ultraviolet lights. Good detects were obtained on papers without fluorescent brighteners but by this time brighteners were being added to most writing papers and there was little contrast between the fluorescence of the detected writing and that of the paper. None of these materials were as good as iodine and, in 1957, didn't seem to be of much value and the search was discontinued. Eleven years later, in 1968, with a different examination procedure, several of them, antranilic acid, benzoic acid, pyrene and a few others became powerful detection reagents. The 1968 work is covered in Section 4.10 "Zephyr."

SECTION 4.5 ATTEMPTS TO DEFEAT IODINE DETECTION

We spent the next few years trying to develop treatments of paper to defeat iodine detection, either by liquid iodine or hot iodine. We developed treatments and procedures that nearly defeated detection but not completely, and nearly was not good enough.

We found that secret writing made with a mixture of water and alcohol would not be detected by liquid iodine. However humidity was critical. A water-alcohol mixture that worked in a dry environment was readily detectable in a humid environment and one that worked in a humid environment was readily detectable in a dry environment.

We treated papers to react with or coat the reactive hydroxyl groups. Detection contrast was greatly reduced but not eliminated.

We tried to mask hot iodine detection by adding nucleation sites to the paper surface. From this work it became standard procedure to pre-rub the message sheet with a cloth before writing the secret message. The rubbing produced nucleation sites for iodine that eliminated or greatly reduced the possibility of detection by hot iodine but the recovery of the message was not affected.

By this time secret writing was done by using secret ink carbons rather than liquid inks and these secret messages were not detected with the iodine detecting solutions and pre-rubbing the message sheet before the application of the message essentially defeated hot iodine detection.

SECTION 4.6 INDIGO

During the first several years, starting in 1956, the client held secret writing conferences with representatives from each of the contractors and several CIA employees in attendance. Papers were presented by the contractors on various SW subjects. A conference in early 1957 is of particular interest. At that time secret writing was shifting from liquid inks and pencils to the use of secret ink carbon sheets. The carbons were ordinary sheets of writing paper with applied secret ink. There were questions concerning how to produce a carbon and how long a carbon could be used before the secret ink (SI) would be exhausted. ADL was the contractor working on carbon production and they gave a paper on the subject. The SI was generally applied by a very fine spray of a suspension of finely ground SI particles in a volatile liquid.

They described the SI on the carbon paper as residing in three locations; the "Pad" which would be the top of the surface fibers, a name of a location that I don't remember at an intermediate location, I will call it "Intermediate" and "Reservoir" which was deep into the paper between fibers. They proposed equilibrium of SI concentration between these three locations as follows: Pad ↔ Intermediate ↔ Reservoir. They assumed that 50% of the SI would transfer from the carbon pad to the message sheet from the actual area of the written message and they calculated the fraction of the sheet area that consisted of actual text. With these assumptions they calculated the expected life of the carbon.

I thought that their presentation was unrealistic almost to point of being ridiculous. Several things were wrong. The SI consisted of individual solid particles and there is no basis for an equilibrium movement from one location to another. Microscopically the paper surface is rough and looks about like a plate of cooked spaghetti. During writing with a carbon and message sheet, primarily only the top surface fibers of carbon sheet and the top fibers of the message sheet would make contact. Much of the top plane of a paper surface consists of voids between the fibers so the actual area of contact is less than the width of the line times its length.

A lively discussion followed their presentation. I, or someone else, pointed out the microscopic roughness of the surface and that only a fraction of the area of the written letters would have made contact between the carbon and the message sheet. I will never forget the response of Don Lindsey from ADL, "Since the paper surface is rough you can have more than 100% contact. It is like two sheets of corrugated roofing in contact with one another." Ridiculous! The ridges and valleys of corrugated roofing are parallel and equally spaced. The fibers of paper are not straight and parallel just as the strands of cooked spaghetti are not straight, parallel and equally spaced.

At the same conference Don Grabar, from another contractor, presented a paper proposing the use of the compound indigo as an SI. He described a procedure for producing an indigo carbon by a fairly high temperature, short duration sublimation of indigo from a hot surface to a sheet of paper. *(Sublimation means to convert a substance from a solid state directly to its vapor by heat and allow it to solidify again.)*

He showed photomicrographs of the indigo carbon and he had determined that there were 300,000 indigo particles per square millimeter. This produced a very lightly colored carbon. Test messages had been made with the carbon, producing nucleation sites for the message, and the message was recovered by using an ordinary iron and ironing a blue handkerchief with blue indigo dye onto the message sheet. Excellent recovery resulted. I was fascinated with his presentation.

When I got home I made an indigo carbon by the procedure described by Don. I counted the particles on a portion of the carbon and I also determined that there were approximately 300,000 particles per square mm. I wrote a test message with it and obtained a good recovery. I used a hot plate rather than an iron, and indigo sprayed from a solution on an aluminum sheet rather than a handkerchief.

A microscopic examination of the recovered message showed that the indigo had sublimed only to the top surface of the top fibers in the text area. That is, where the message sheet had contacted the carbon surface and indigo particles had transferred to the message sheet and served as nucleation sites for the indigo vapors during recovery development. Thus I had a procedure to obtain information relative to the life of a carbon and the presentation by ADL. (I had remained troubled by their presentation) While I had no authorization to so, I proceeded with an interesting and informative experiment. (I did much of this work on my own time rather than charged time.)

If you calculate the pressure created during writing with a normal writing force of about 125 grams, (about 4.5oz) the writing pressure is about 1000 lb/square inch. In my experiment, I pressed my indigo carbon sequentially against 100 sheets of paper with a force of 1,000lb/square inch. Thus I had obtained 100 “message sheets” from the same area of the carbon. While the carbon and message sheet were still in contact and not moved relative to one another, I inserted a pin through the carbon and message sheet to make two pin holes 3 mm apart. I used the same two holes in the carbon for each of the 100 message sheets. With the pin holes in the recovered message sheets I could locate the exact area of carbon contact for each of the 100 message sheets.

I photographed the areas between the pin holes and made a same size transparency of the corresponding area of the carbon. By placing the transparency on the photographs of the recovered message sheets I was able to identify the exact contacting positions, fiber to fiber, for most of the message sheets. Reference points were placed on each message sheet photograph.

Then one night, when I would not be disturbed, I made sequential exposures from the negatives of selected messages sheets onto photographic papers. To do this I first placed the photograph of the area of interest in the paper holder for the enlarger, and with the aid of a transparent overlay positioned the two reference marks in fixed positions relative to the holder. I then placed the negative in the enlarger and superimposed the projected image on the photograph. The paper holder was moved to obtain a perfect match. Then without moving the paper holder, the photograph was removed and the photo paper placed in the holder. And the paper exposed to the image. This sequence was repeated until I obtained multiple exposures from four, six and eight message sheets. I started at about 8 o’clock in the evening and finished after two in the morning.

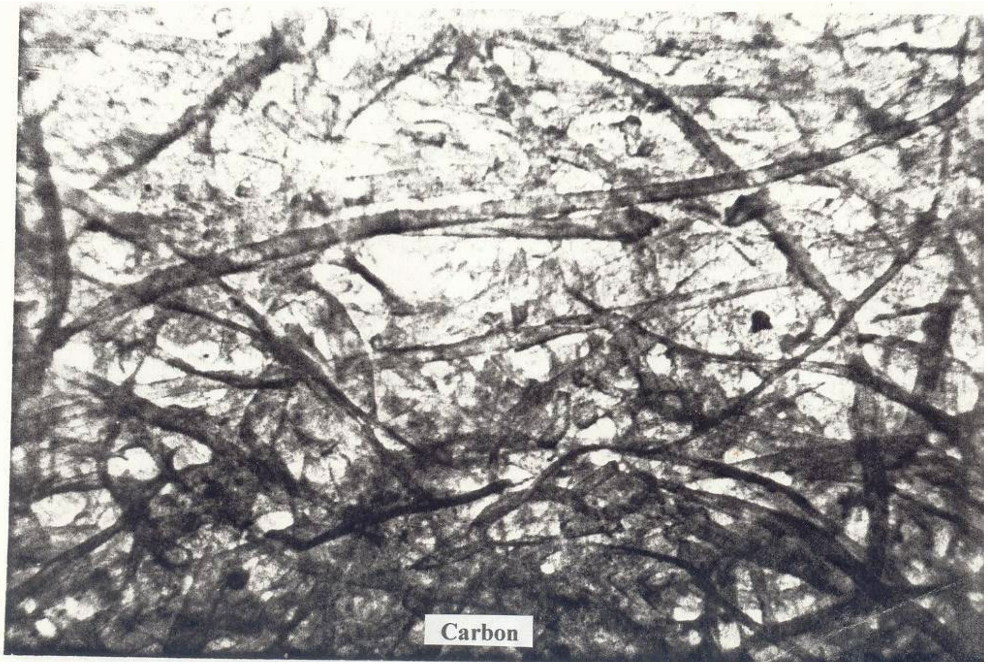
I was a nervous wreck. At the end, I had hours invested in what was to become one photograph (for each of the four, six and eight exposures). Any little mistake would have ruined it.

But I made no mistake. The corresponding areas of the carbon (mirror image) and the photograph made with eight exposures are shown on the following page. Two indigo developed sheets of an area adjacent to the composite image are shown on the page after that. (Unfortunately I apparently saved the wrong set of photographs, not the areas used for the composite exposure.)

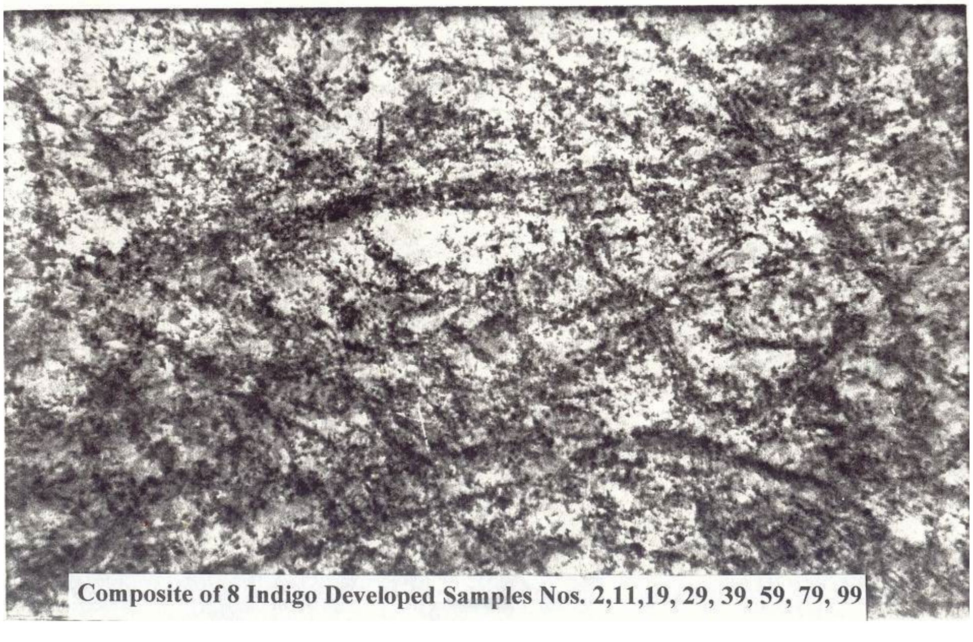
This work showed that only the indigo on the top fibers of the carbon paper, contacted and transferred only to the top fibers of the message sheet. The intermediate and reservoir layers were not involved in the transfer.

I presented this work and the composite images at the next SW conference. Allen Gold, my COTR was very excited with the presentation, and very complementary ADL came up with lots of objections.

Considerable information was obtained from this and related indigo work. For example, for the paper that was used for this work it was determined that about 15% of the surface areas of the carbon and message sheet made contact at a pressure of 1000 lb per sq in.

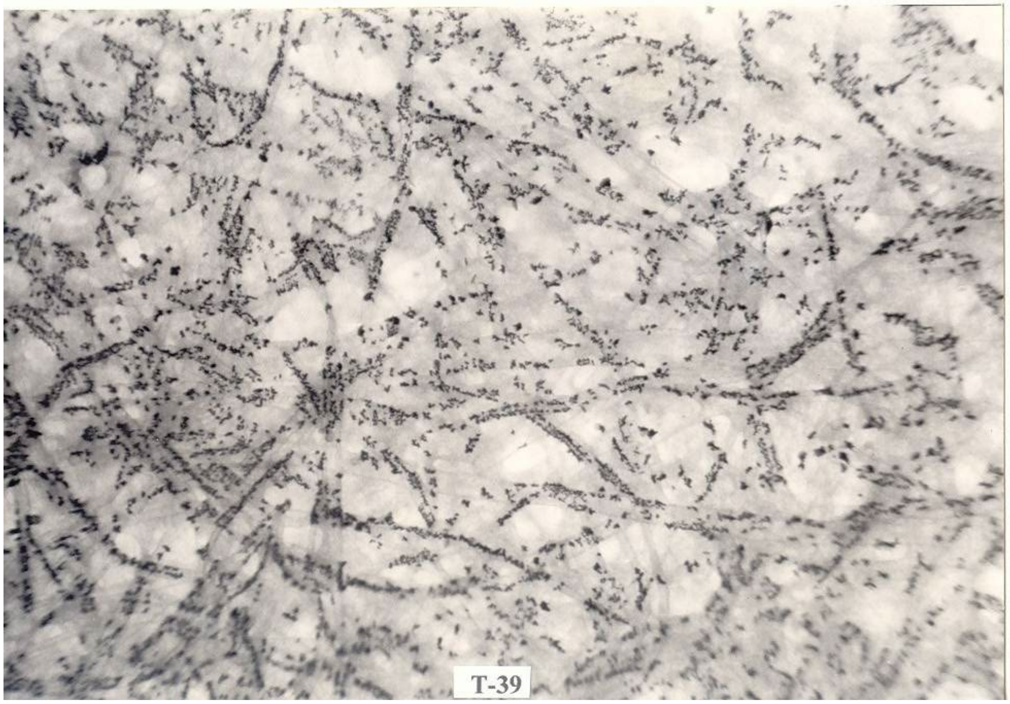


Carbon

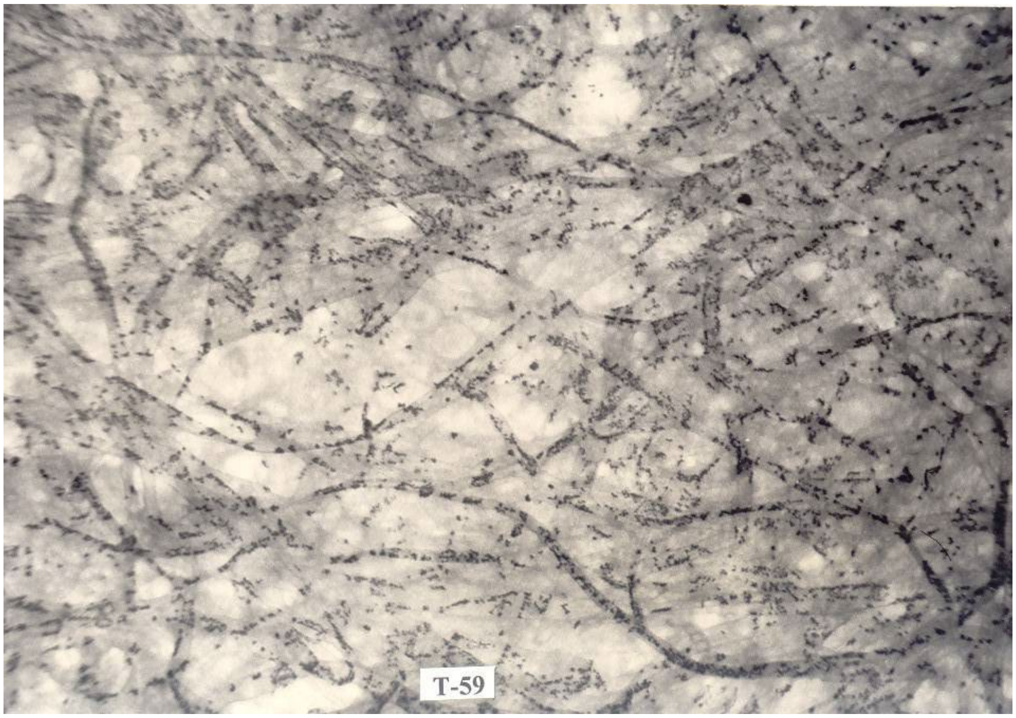


Composite of 8 Indigo Developed Samples Nos. 2,11,19, 29, 39, 59, 79, 99

Top: Mirror image of the indigo carbon that was pressed against 100 message sheets.
Bottom: The image produced by the sequential exposure of photographic negatives of eight message sheets of the exact area that contacted the above carbon. Note that the pattern of indigo deposits of the composite closely resembles the pattern of fibers of the carbon. See text for further explanation. Indigo deposits on individual message sheets are shown on the following page. Each photograph represents an area approximately 1 X 1.5 millimeters.



T-39



T-59

These indigo deposits are from areas of two message sheets that were included in the composite photograph in the preceding page but not of the same area. Unfortunately I saved the wrong set of photographs. The appearance is similar to the areas that were used for the composite.

SECTION 4.7 MAIL PROBES

During some of the early secret writing conferences there were discussions concerning the security of our secret messages. A wide range of opinions were expressed. Some thought that the opposition was doing very little censorship and security was not a problem. Others thought that most of our letters were censored and that the opposition was very good at it. Strong opinions were expressed but they were only opinions. There was little evidence one way or another about what was being done to our letters and what might be detected.

Frank Laubinger was my COTR at the time and he suggested designing a set of mail probes to be sent though opposition mail channels and then returned for analysis. He had very little support but managed to obtain funds for a small feasibility study that was conducted in 1962. Our approach included putting microdots of reagents on the letter that would respond to various chemical or physical treatments. We submitted a report and it generated no interest. Then in 1966, someone saw our report and wanted to put mail probes in operation immediately.

We went from a feasibility study to operations without much time for a development stage to refine our procedures. In the feasibility study we had tried many things with varying degrees of success. We now needed to decide which to use and which to discard. We needed to decide what to put on the letters, where and how to put it and how to locate it and analyze it when it was returned. We needed to prepare the sheets of paper with the microdots and measurements of various paper properties such as thickness, reflectivity and exact size. The client would use these sheets to write a letter, mail it, recover it and return it to us for analysis.

We selected eight reagents and placed 0.5 mm diameter micro dots of the reagents on the letters. Some were to detect chemical treatments and some physical treatments. A copy of the template that we used for recording the microdot locations on the letters and photographs of the apparatus for placing the microdots appears on following pages.

We learned quite a bit. The Chinese were applying numerous reagents to the letters, including one that was previously unknown to us. There was much less evidence of treatment in the Soviet block.

Two of the dot materials were emulsions of two different paraffins. These were paraffins with fairly sharp melting points. One melted at 90ºC, the other at 140 ºC. I made the emulsions by pouring a thin stream of hot molten paraffin into a high speed blender containing cold water. The dots were "read" on the return probes by applying "liquid iodine." In a letter that had received no treatment the dots would appear dark against a light background. This was the result of the water of the emulsion wetting the paper. If the letter had been heated to above the melting point of the paraffin, the paraffin would have coated the surface of the paper and prevented the iodine solution from wetting the paper and the spot would appear as a resist, a light spot against a darker background. For example, if the letter had been steamed, and then dried between blotters, the 90 ºC melting paraffin would have melted and appear as a light resist while the 140 ºC paraffin spot would not be detected because the paraffin had not melted but the steaming would result in the background being dark. But if the paper had been steamed the background would be relatively dark as compared to a control. If the letter had been wet for any reason and then ironed dry, both paraffin spots would appear as resists.

We obtained all of the above possible results in the operational samples from China.

Two of the stripes shown in the photograph of the chemical censored letter (Section 4.1) were labeled "oxine". Oxine is the common name for 8- hydroxyquinoline. It is a common analytical reagent for the detection of several metal ions and reacts with the metals to form a fluorescent complex. Any of various metal compounds used in secret writing can be detected with oxine as fluorescent writing when viewed in ultraviolet light.

Our probe letters contained two metal compounds. They were nickel sulfate and magnesium hydroxide, $Mg(OH)_2$, commonly known as milk of magnesia. The $Mg(OH)_2$ dots on most of the letters returned from China were very fluorescent. We first assumed that the letters had been treated with oxine, but when we extracted the material from a letter and compared it to oxine by thin layer chromatography it was evident that the material was not oxine. It turned out that the material was 2-methyl-8-hydroxyquinoline. It was oxine with an added methyl group. It was methyl oxine and soon became known as "MOX".

Many papers have trace amounts of aluminum and oxine reacts with this aluminum to produce a general fluorescence of the paper which tends to mask the detection of secret writing. MOX produces much less fluorescence with aluminum but fluoresces with several other metals. Therefore it is a better censorship reagent than oxine. Some years later it was determined that MOX also was a fairly good censorship reagent by nucleation and crystal growth on secret ink particles.

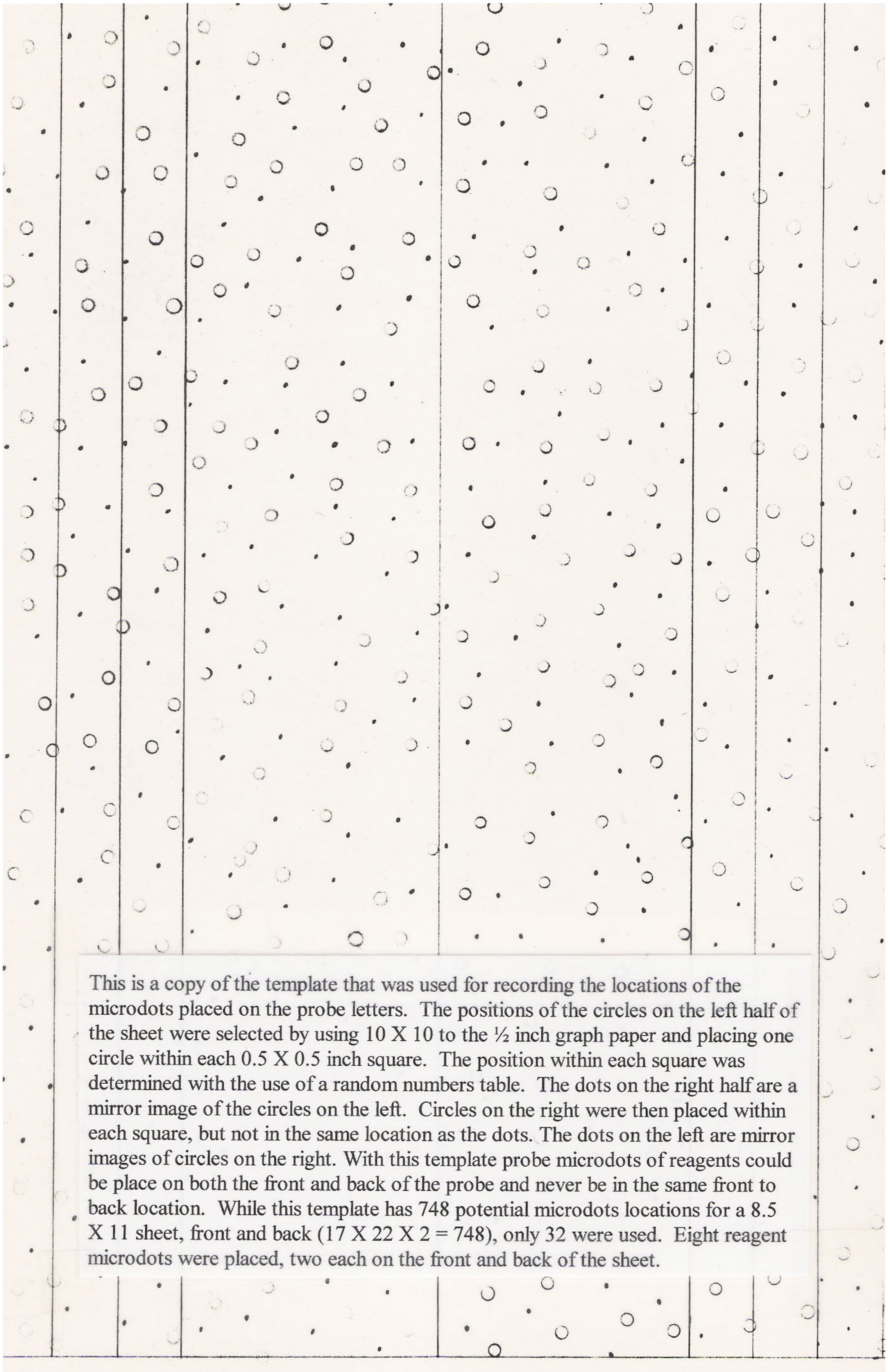
One of the microdots consisted of a compound that was destroyed if it was exposed to ultraviolet light. Nearly all censorship procedures include an examination with ultraviolet light, either alone or in conjunction with the application of some chemical. As expected, most of the letters from China had been exposed to UV.

We examined the returned letters for residual censorship treatments not related to our microdots. Many of the letters from China contained trace amounts of iodine, ammonium ion (residual from fuming with ammonium hydroxide), and chloride ions (residual from fuming with concentrated hydrochloric acid).

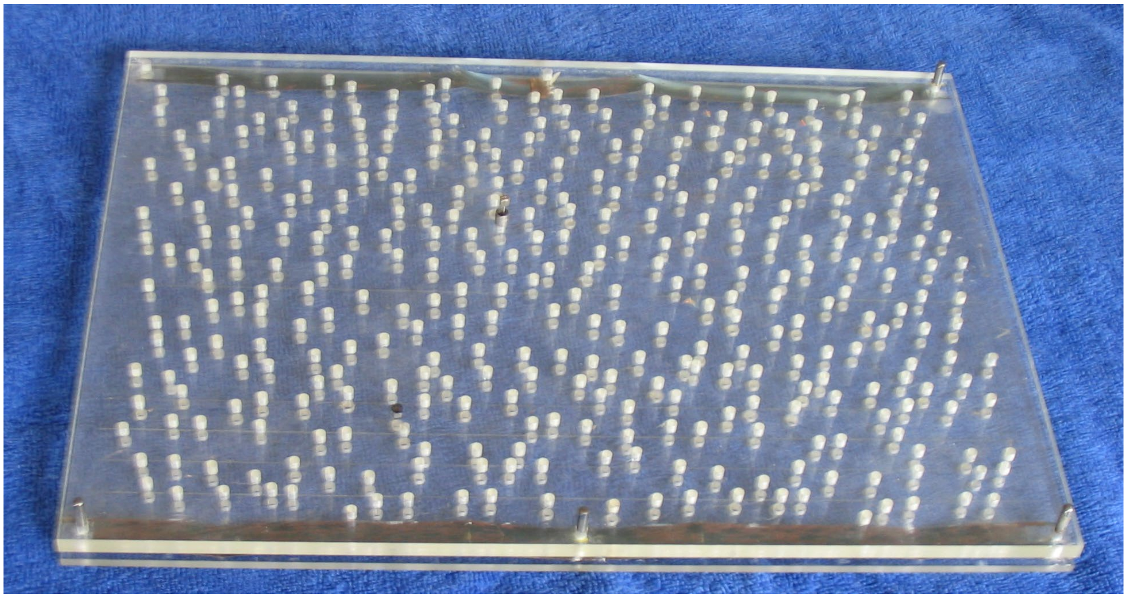
It was concluded that the Chinese were censoring most of the mail, but their techniques were not very sophisticated. They were probably primarily interested in internal secret messages rather than secret writing by the United States.

The fact that we found very little evidence of censorship from the Soviet Block was, in my opinion, much more significant. It didn't mean that they were not censoring, it meant that they were probably much more sophisticated and left little evidence of their procedures.

The probes were considered to have been a success and Frank asked if I would take a year off from MRI to teach the techniques in their labs around the world. He said a formal request would be made only if MRI management approved. Van Morriss, MRI's Technical Vice President vetoed the idea. He said that if I left for a year I probably would not return to MRI. I guess that was a compliment but I was disappointed. In the end, people were brought to MRI from around the world for training.



This is a copy of the template that was used for recording the locations of the microdots placed on the probe letters. The positions of the circles on the left half of the sheet were selected by using 10 X 10 to the ½ inch graph paper and placing one circle within each 0.5 X 0.5 inch square. The position within each square was determined with the use of a random numbers table. The dots on the right half are a mirror image of the circles on the left. Circles on the right were then placed within each square, but not in the same location as the dots. The dots on the left are mirror images of circles on the right. With this template probe microdots of reagents could be place on both the front and back of the probe and never be in the same front to back location. While this template has 748 potential microdots locations for a 8.5 X 11 sheet, front and back (17 X 22 X 2 = 748), only 32 were used. Eight reagent microdots were placed, two each on the front and back of the sheet.



The top picture: This application apparatus consisted of two 9x10 inch plates of plexiglass with holes at the locations of the circles in the template shown on the preceeding page. Leroy drafting pens were used to apply the reagent dots. The pens, with a reagent solution, were placed in the designated positions. The two plexiglass plates were held apart with springs and the tips of the pens did not go through the lower plate. This apparatus was then positioned on the probe sheet and the upper plate was momentarily pressed down so that the tips of the pens touched and deposited reagent dots on the probe sheet. By moving this apparatus from sheet to sheet, multiple identical probe sheets were produced.

Bottom right picture: This is an enlargement of the one pen shown in the top photograph.

Bottom left picture: Before computers, every draftsmen had a set of these Leroy drafting pens. They ranged in size to produce very narrow lines to fairly wide lines. A plunger wire, just smaller than the point opening, prevented the ink from leaking. The pens were used in various holders. This picture is about two-times actual size.

(2007: I went online and discovered that Leroy pens are still available.)

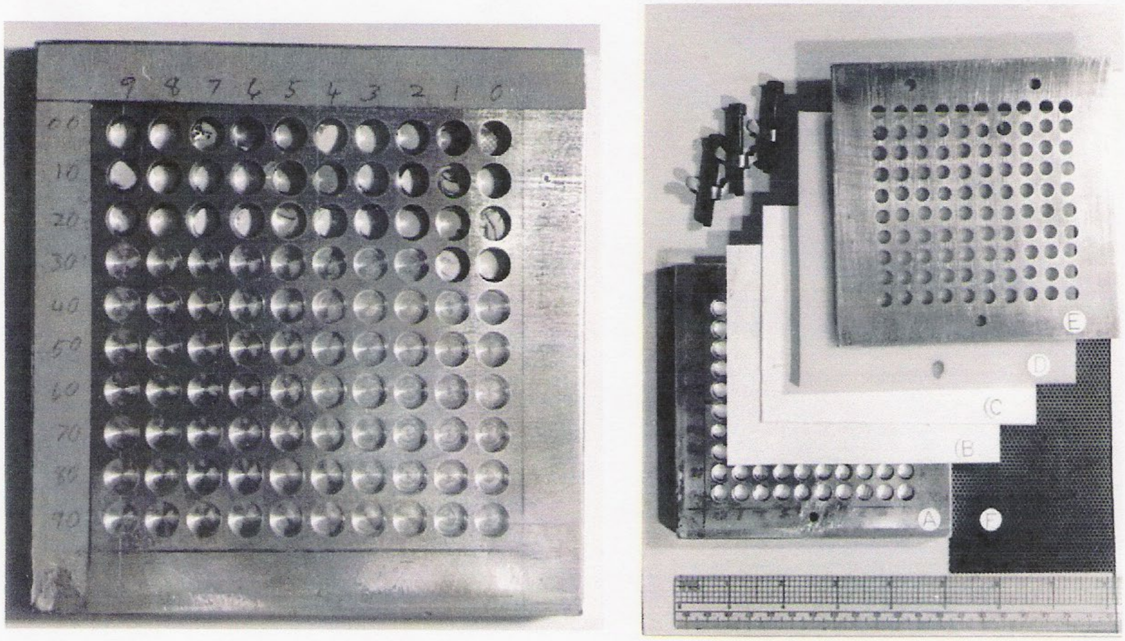
SECTION 4.8 EXAMINATION OF MASS MAIL

The CIA obtained thousands of letters from the British dead letter office in Hong Kong. Most of these letters had been mailed into China and for some reason were returned but could not be delivered to the sender. The primary objective of this project was to search for new censorship reagents used by the Chinese and determine the frequency of their censorship. It was a large effort involving several analytical techniques. Huge amounts of data were collected but no new reagents were found.

I will describe two things related to this project that might be of interest. The first is how the data was handled and the second, shown on the following page, is the apparatus we made to analyze for MOX on the letters.

This project was done before computers were in general use. Instead of a computer we used punch cards. Briefly, we used about a thousand 10 X 10 inch punch cards. Each card represented a characteristic such as one possible result of an analytical procedure, for example the presence of residual iodine or MOX. A hole was drilled through the card for a sample with that characteristic. Each card had 100 X 100 sample positions for up to 10,000 samples. Thus each sample was assigned a specific position on the cards. Correlations were determined by placing a characteristic card or cards on a light table and then placing other cards on top of it to determine which samples had that combination of characteristics.

Aren’t you glad you live in the age of computers?



This is the apparatus we used to detect the presence of 2-methyl-8-hydroxyquinoline (MOX) on letters from China.

- On the left is a 5X5 inch aluminum plate one-half inch thick. The plate has 100 holes, 5/16 inches in diameter and ¼ inch deep. This is "A" in the picture on the right.
- With this apparatus we didn't even need to open the envelopes for the analysis. We just punched a ¼ inch sample from the letters and placed the sample in one of the holes. The analytical procedure was as follows:
    - A ¼ inch punch of a blotter was placed in each of the 100 holes of the plate "A" and each was slightly wet by adding 10 micro liters of water (a very small drop.)
    - A punch from each of 100 letter samples was added to the holes in the plate.
    - A 5 inch square of filter paper "B" that had been sprayed with a solution of zinc chloride was placed on the plate, followed by a sheet of blotter paper "C," a sheet of polyurethane foam "D" and an aluminum cover plate "E."
    - The assembly was held together with three bolts shown at the upper left.
    - The assembly was then placed on a hot plate. The steam from the heated, wet blotters "steam distilled" the MOX from the letter samples and the MOX vapor reacted with the zinc on sheet "B." The MOX was detected by examining the sheet with ultra violet light. Any MOX present resulted in fluorescence.
- With this apparatus, hundreds of samples could be analyzed in a few hours.

SECTION 4.9 MICHLER'S KETONE

In 1964 MRI received another project to study a mechanism of detection, in this case, for Michler's Ketone, MK for short also known as 4, 4'-bis (dimethylamino) benzophenone. Arthur D. Little (ADL) had come up with this reagent and they were not happy that the project was given to MRI but their major assignment was to develop new secret writing systems, not detection. MRI had done good work in determining the mechanism of iodine detection. The CIA had several projects at MRI in various areas other than the ones I had. I was not involved with the early portion of this work.

The procedure for this detection, as presented to MRI, was to spray a solution of MK in acetone on the paper being examined and view the results under long wave ultra violet light (UV). With the MK, the paper fluoresced a bright yellow. Usually no detection was seen at this stage. The paper was then repeatedly sprayed with a very fine mist of chloroform. A detect generally appeared in one to two minutes as a dark blue message against the bright yellow background.

Dr. Florence Metz wrote the proposal. Dr .Ron Butler along with Lane Young did the lab work. The literature on Michler's Ketone was primarily related to energy transfer and Florence concluded that the detection mechanism was energy transfer. (Energy transfer is when light is absorbed by a molecule to increase its energy, alter the position of its electrons, and then this energy is transferred to another molecule, which in this case results in fluorescence.)

At the time, I was in Dr. Earl Barney's Section. Florence and Ron were in another section. The turf battles between Florence and Earl were classic. They hated each other, each with considerable justification. So, as far as Florence was concerned, I was in the enemy camp. Therefore I knew little about what they were doing until the beginning of the third year. This project was titled something like a search for MK-like reagents. I learned that ADL had been given a copy of their reports and strongly disagreed with MRI's results and conclusions.

Florence had proposed that the mechanism of detection was energy transfer and their experiments were designed to prove it. Their work had followed procedures from the literature related to energy transfer. This work was with solutions. They identified several materials that gave classic energy transfer characteristics**, in solution**. They then made screeds using these materials and tested them with MK. Some detects were obtained. As far as they were concerned that proved that the MK detection mechanism was energy transfer.

ADL suggested that the mechanism was nucleation.

Dr. Florence Metz was not one who wanted the truth of her theories to be tarnished by experimental results. ADL's suggestion was ignored and Florence dug in with work that could not indicate a mechanism other than energy transfer.

The real beauty of research is to see the unexpected, and to look for the unexpected. You conduct an experiment with expected results and get something else. I was often successful because I noticed the unexpected which often was much more important than the expected. Ron and Florence, like many in the field wore blinders and saw nothing other than what they wanted to see. If something else appeared, they ignored it. In this instance, they got some good results but they failed to interpret the results properly.

Colin R was the project monitor for both my projects and the MK project. Colin was with ADL during WWII and joined the CIA shortly after it was formed. He had a PhD from Kentucky. He was very good. On Colin's next trip for a project meeting, he brought Marty Koocher of ADL. Marty was a very aggressive, out-spoken individual. He demonstrated what he thought was convincing evidence of nucleation. That meeting and demonstration was my first real involvement with MK, and I was convinced that nucleation was at least part of the detection mechanism. Florence and Ron ignored Marty's demonstration and redoubled their efforts to prove energy transfer.

After the meeting, I ran a ten minute experiment. I made a screed line with an ink that I knew would be detected with MK. Beside the line I placed a wire about the size of the line and sprayed the sheet with MK. The wire was a mask to produce a line without MK. Then the wire was removed and the paper sprayed with chloroform to obtain the detect of the screed line. The detected SI line and the line free of MK, masked by the wire, appeared essentially identical. Both appeared to be blue. The lines appeared to be blue to the eye as a complimentary color of the bright yellow background. If the background was covered so only the screed line was seen, it was white.

I told Ron about it and suggested that they should consider more than one mechanism, perhaps both energy transfer and nucleation. A short time later I wrote a five page memo to Ron making suggestions for what I thought they should include in their work I suggested they do some microscopic examinations of the detects, which had indicated nucleation. I thought I convinced him, but the work that I had suggested was vetoed by Florence.

After the next project meeting with Colin, when we were cleaning up, we found a sheet from a note pad on the floor where Colin had been sitting. The message was:

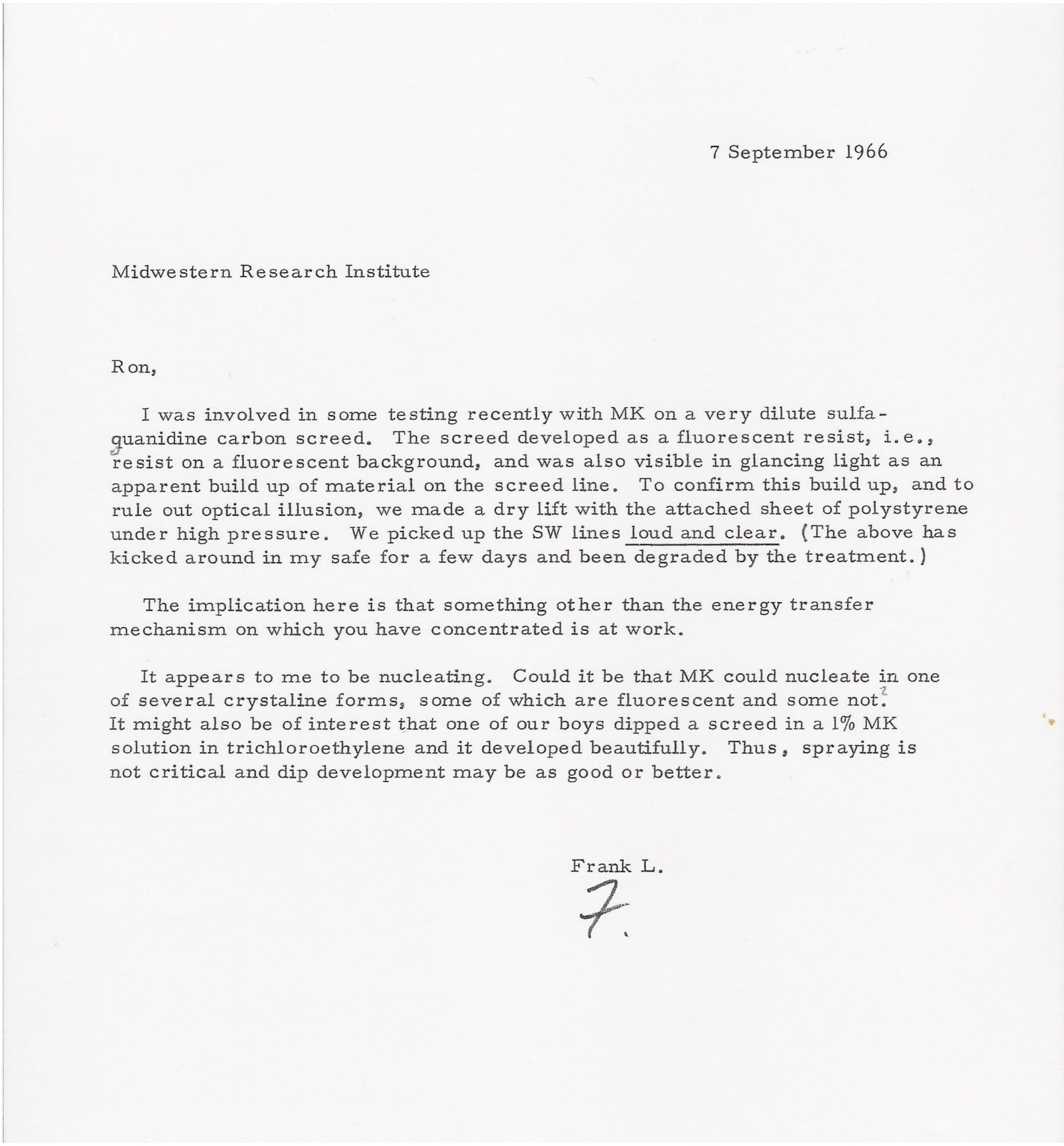
From the desk of
MARTY KOOCHER

SOMETHING IS ROTTEN IN DENMARK

And in MRI

Did Colin intentionally drop it?

Ron and Florence got a chuckle from it and nothing more. Years later I went through the MK files and found the letter on the following Page. It was from Frank L who was my project monitor a few years earlier. That letter had been written in 1966, during the second of the three projects. It seems that any sensible person would have followed up on Frank's information but Florence and Ron apparently completely ignored it.



7 September 1966

Midwestern Research Institute

Ron,

I was involved in some testing recently with MK on a very dilute sulfaguanidine carbon screed. The screed developed as a fluorescent resist, i.e., resist on a fluorescent background, and was also visible in glancing light as an apparent build up of material on the screed line. To confirm this build up, and to rule out optical illusion, we made a dry lift with the attached sheet of polystyrene under high pressure. We picked up the SW lines <u>loud and clear</u>. (The above has kicked around in my safe for a few days and been degraded by the treatment.)

The implication here is that something other than the energy transfer mechanism on which you have concentrated is at work.

It appears to me to be nucleating. Could it be that MK could nucleate in one of several crystaline forms, some of which are fluorescent and some not? It might also be of interest that one of our boys dipped a screed in a 1% MK solution in trichloroethylene and it developed beautifully. Thus, spraying is not critical and dip development may be as good or better.

Frank L.

F.

Near the end of the third year of their project Florence went to Washington to present a proposal outline for a continuing project. Colin told me some time later that her real objective was to have my work canceled and the funds transferred to her. Colin said that he had often had one contractor bad mouth another, but had never experienced the intensity of Florence's attack on me. Among other things, she pointed out that if Colin funded her proposal he would be getting two PhDs while Gaylord only had a Masters Degree. Her attack backfired. My proposal was funded, hers was not. Their final report never mentioned the possibility of nucleation.

But there was nothing personal in Florence's attack on me. I was just fair game in her turf battle. A short time later Earl Barny left MRI and the department was reorganized, Florence became a Section Head and I was placed in her Section. Now, I was a very talented, nice guy on her turf and I had her full support.

Colin asked if I could document nucleation for MK. Microscopic observation of the detects of many SW systems clearly showed that the MK on the background was amorphous and the MK in the detected text was crystalline. I found no MK detects that were other than by nucleation. It looked about the same as the resist detect or flash resist for hot iodine, only the time frame was minutes rather than fractions of a second. Photomicrographs were taken and a report submitted.

And why did the amorphous MK in the background fluoresce? I don't know. Maybe it was energy transfer.

A few years later at one of the SW conferences Marty Koocher was asked some hard questions about some problems with ADL's work. His response was to chide the Client for wasting three valuable years and considerable money by giving the MK work to an incompetent contractor. I assume his remark was also for my benefit, and it hurt, but silently, I had to agree with him.

SECTION 4.10 ZEPHYR

Zephyr is the code name that was given to the secret writing detection procedure described in this section. It was the best and most important secret writing work that we did. Paul Seiwald and I worked as a team and within a few months developed a procedure and found reagents that detected nearly all of the secret writing systems in use at that time. Secret writing was very important to the Client at that time and our results were devastating.

During the time of the MK work I wanted to get back into detection work. I certainly wanted to work in detection after I realized that Florence Metz and Ron Butler were doing a very bad job in detection. Colin R returned detection research to my group in June 1968 after he canceled the MK follow-on project. He told us that essentially all of the SW systems in use were recovered by nucleation and crystal growth. Most of these systems had been developed by ADL and they assured the client that they were completely secure and could only be visualized by the specific developer. Colin was not so sure.

By now SW carbons were in general use. The carbons were used just as an ordinary carbon paper but looked like an ordinary sheet of paper. Colin brought us twenty or thirty carbons of different systems and asked us to see if we could find a method to detect screeds produced by them. He suggested trying supersaturated solutions of various materials.

Paul Seiwald and I started work. We started with solutions of colored materials so the detects could be easily seen. Supersaturated solutions are obtained by putting an excess of the material being tested in a solvent and heating it while it is stirred. This produces a saturated solution at the heated temperature which is generally the boiling point of the solvent. The excess solids were then allowed to settle and the clear saturated solution poured into tray or pan and allowed to cool. Almost all materials increase in solubility as the temperature increases and decrease as the temperature decreases. In this example the solution has a concentration of saturation at the higher temperature but as the solution cools it becomes supersaturated. Many materials can exist as a supersaturated solution for a considerable length of time; in fact in organic chemistry it is often necessary to “seed” a solution with crystals of the material to start crystallization. In secret writing detection, the SI on the message sheet acts as the seed for nucleation sites. At some point during the cooling, the paper with the secret writing is slipped into the supersaturated solution and hopefully the secret ink particles act of nucleation sites for the crystal growth of the test material. If is does, a detection of the writing results.

This procedure is the same as for the recovery of a secret message. The difference is that for secret message recovery, the supersaturated solution is the same material as the secret ink and nucleation and crystal growth is essentially certain. For detection, the secret ink is unknown and It may or may not be an effective nucleation material for the supersaturated solution of another material.

Our first success was with 2-nitro anisidine (methoxy nitro aniline or just nitro anisidine). It is a dark orange brown that could be readily seen. A couple of the systems were detected when they were dipped in a 2-nitro anisidine supersaturated solution. Nitro anisidine is fairly volatile so I tried vapor phase nucleation. I placed some nitro anisidine in a Petri dish, placed the dish on a hot plate and a sheet of paper with some secret writing on the dish. Detects were obtained. The hot nitro anisidine vapor had nucleated and grew crystals on the secret ink particles.

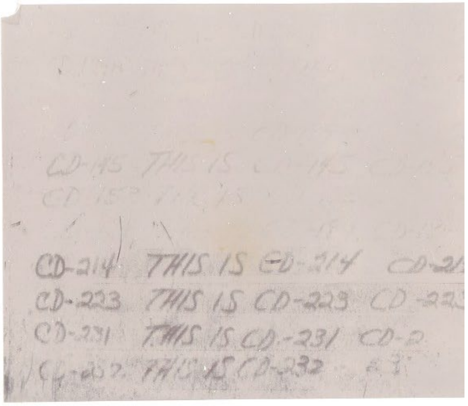
The systems that Colin brought us were not detected using a plastic lift because the SI particles were smaller than the wave length of light. A lift was obtained by pressing a sheet of plastic, for example polystyrene, against the message sheet. Some of the SI transfers to the plastic surface and if the particles were larger than the wave length of light they were visible when viewed in an intense light against a dark background. All of carbons that Colin had supplied us were made with SI particles smaller than the wave length of visible light. However when lifts of the various systems were exposed to the nitro anisidine vapors from the Petri dish, several were detected. With the detection taking place on the clear plastic sheet rather than on paper, the detecting agent no longer needed to be colored. Any crystal growth on a clear plastic sheet is readily seen when viewed with a bright collimated light.

More of the systems were detected from the vapor than from supersaturated solution. This may be because the solvent of the solution may have dissolved the SI particles before they had time to serve as nucleation sites for the detecting material.

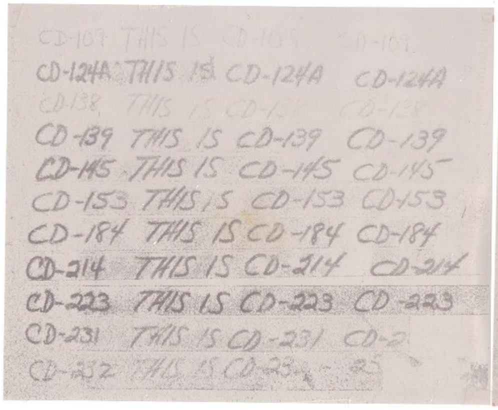
Back in 1957 we searched for materials that acted like hot iodine detection. We had found several materials that appeared to be fairly good but they were colorless and while detects were obtained they couldn’t readily be seen on white paper. We next tried those materials. We hit the jack pot. Within about two weeks we had materials and a technique that detected nearly half of the carbons that we had. By September, three months after we started, we had a technique, and a battery of six reagents that detected over 90% of the entire stock of systems in use. The technique was simple and the detection was high contrast.

See next page for examples of our early detection:

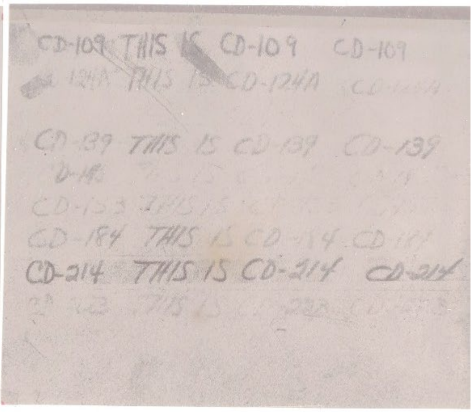
No Treatment



Pyrene



Anthranilic Acid



Benzoic Acid

Each line indicates the use of a different Secret Ink identified by its own CD number.

These are some of our very early Zephyr detects of Secret Inks in actual use at the time. Note that different Zephyr reagents detect different inks.

In these examples, Pyrene, Anthanilic Acid and Benzoic Acid were used to detect the Secret Inks. These photographs were printed as negatives so they appear as dark letters with a light background.

The Client was in panic. In three months they went from believing that their systems were secure, to knowing that they were not secure. The technique was code named Zephyr and was classified at the highest level, above **TOP SECRET.** Only a limited number of individuals within the CIA were briefed on the procedure. It was that important to them.

We continued to look for other reagents and to refine the procedure. At first it took several hours to screen a new compound. Temperatures were critical, and we had to change temperatures until we reached an optimum range. By this time we were using plastic coated glass plates for the lift surface and the temperatures of both the test reagent surface and the glass plate were important. We then built and used two gradient temperature hot plates to quickly find the optimum temperature combinations for the reagent source plate and for the message lift. Each of these plates consisted of an aluminum plate 18 inches long and seven inches wide and 3/8 inch thick. One end was thermostatically heated to a selected temperature, for example 100 °C, and the other end cooled with circulating water from a controlled temperature water bath for example 30°C. There was a uniform temperature gradient along the length of the plates.

The two plates were positioned perpendicular to one another so a wide range of temperatures and temperature differences existed between the two plates in a single two minute examination. A new reagent could now be screened in minutes, and the results were much better than the previous trial and error procedure. The configuration and temperature relations of the two temperature gradient plates and the detection of a test screed are shown on the following pages.

The optimum temperature for the reagent source plate and the lift surface varied somewhat from one reagent to another depending on the vapor pressure of the reagent. In general the best detects were obtained when temperature differences between the reagent source plate and the lift surface were thirty to forty degrees Celsius. This represented a super-saturation of about 20 at the surface of the lift. That is, the vapor pressure (vapor concentration) of the reagent at the source plate temperature is twenty times that of the lift surface temperature.

The Client had acquired nearly 100 Soviet Block carbons, and they were detected as well as were ours. Our Client therefore reasoned that if we could detect their systems by this technique, the Soviets did not have a similar technique. They would not be using systems that they knew could be detected, therefore it was safe for us to continue using our systems. Do you sense a flaw in this reasoning? I am almost certain that the Soviets had a similar detection system from the fact that our earlier mail probes showed almost no evidence of chemical censorship. Our probes would not have provided evidence of a Zephyr type procedure. In later work, (Section 4.11) we identified numerous Soviet secret inks and from the nature of these materials it was fairly obvious that their recovery method was also nucleation and crystal growth. So, in my opinion it was very likely that they had their own “Zephyr” procedure. I would like to know if they used the same reagents that we did.

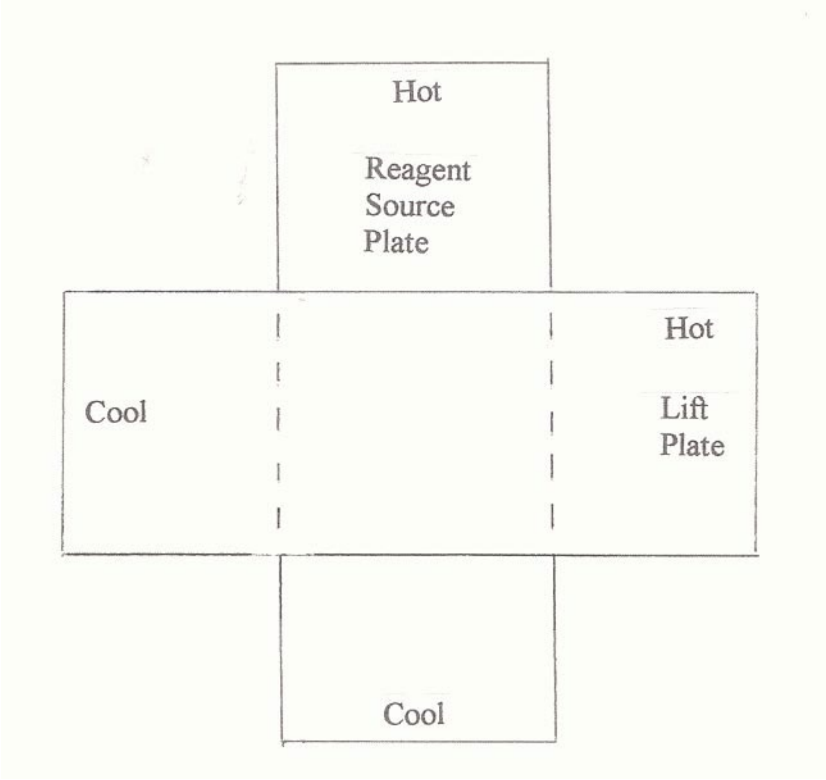
Zephyr became a major concern for selected groups working in SW.

We worked for the next several years both on how to improve the technique, but most of the effort was how to defeat it. Numerous approaches were taken by us and other groups. Many millions of dollars were spent. It took more than ten years to finally defeat it. And we were the ones that came up with the technique that did.

But before discussing our attempts to defeat Zephyr I will describe the apparatus and procedure.

The lift surface. We wanted as clean a plastic surface as possible. Any commercial plastic sheet that we tried had flaws that served as nucleation sites for the reagent and interfered with the detection. We finally standardized on glass plates coated with a very thin layer of Lexan (polycarbonate). The glass was ordinary window glass. A dilute solution of Lexan in methylene chloride was streaked along one edge of the glass plate and then spread across the plate by drawing a "doctor" blade with a spacing of about 0.1 mm between the glass and the blade. The methylene chloride quickly evaporated to produce a clean Lexan surface without flaws.

ADL was given the Zephyr procedure and they made some modifications including the use of commercial polystyrene sheets for the lift surface. They had used these polystyrene sheets for plastic lift detection for several years. They modified other steps in the procedure as well. There were some differences in the detection obtained by our and ADL's procedures. These differences will be briefly mentioned later.



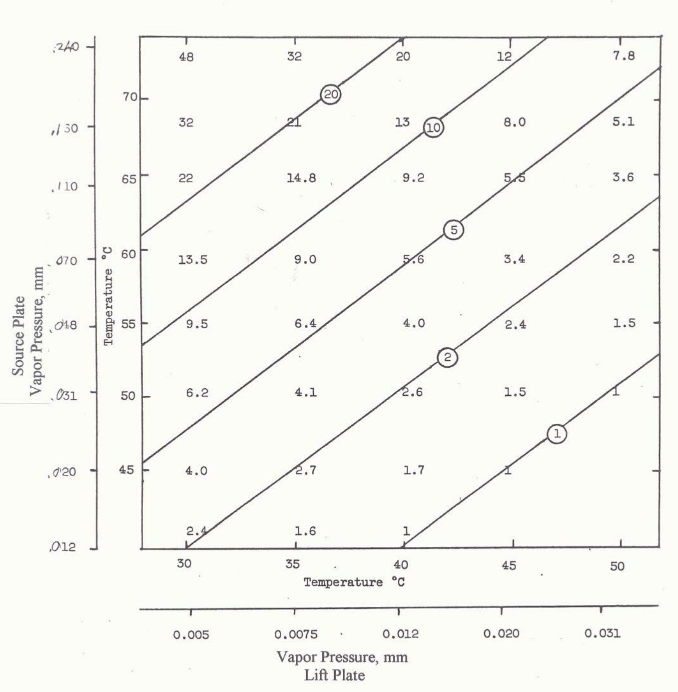
On Possible Configuration and Temperature Relationship of the Two Temperature Gradient Hot Plates.

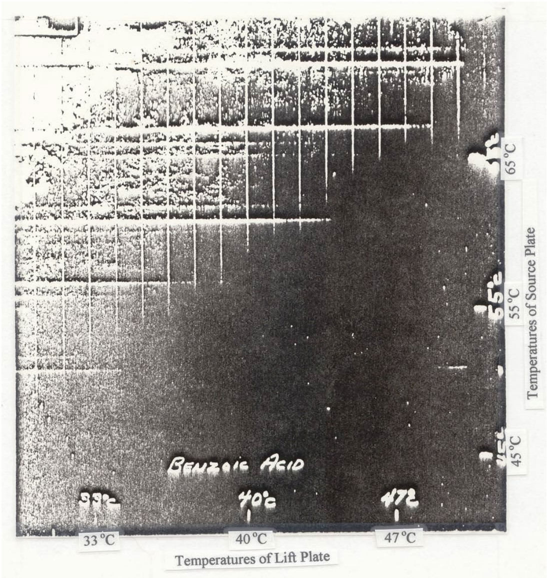
Each plate was 7 X 18 inches with one end heated and the other end cooled to provide a linear temperature gradient along the length of the plate. A solution of the reagent that was being tested was sprayed onto a thin 7 X 7 inch aluminum plate which was positioned on the Reagent Source plate covering the desired temperature range. The test screed was “lifted” onto the center 7 X 7 inches of a 7 X 9 glass plate with a thin coating of Lexan. The lift was obtained by pressing the test screed against the Lexan coated glass at 1,000 psi, with a total force of 50,000 pounds.

The lift was positioned on the gradient temperature Lift Plate covering the desired temperature range and attached to the plate with clips at each end. The Lift Plate was then inverted and placed, with the center of the lift over the Source Plate. The reagent coated plate had 0.5 mm spacers on each corner to provide a space between the two surfaces. After two minutes the Lift Plate was removed from the Source Plate, the glass plate with the lift was removed and examined for a detect.

Note that the upper left corner of the 7 X 7 inch square of the test has the maximum temperature of the source plate and the minimum temperature of the lift. Thus this corner has the maximum temperature difference between the two surfaces. The lower right corner has the coolest source temperature and the warmest lift temperature. This gives a gradient temperature difference from the lower right to the upper left.

An example of a resulting detection is given on the following page.





Left: Temperatures, Vapor Pressures, and Degrees of Supersaturation of Benzoic Acid at Various Positions for the Crossed Gradient Temperature Plates.

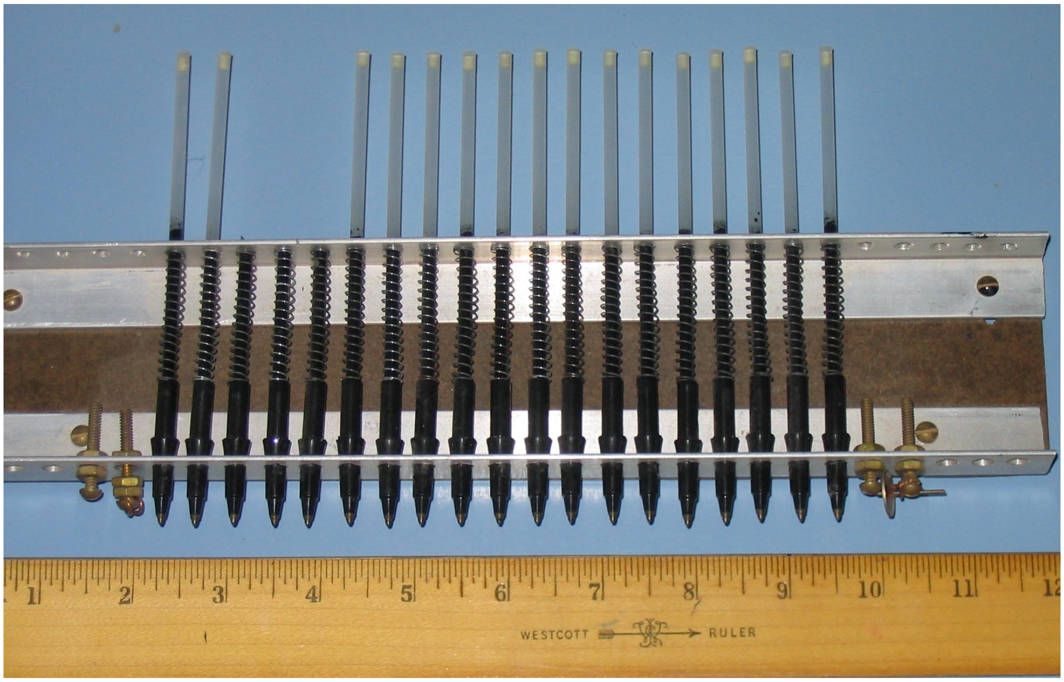
Right: The Detection of a Test Screed by Benzoic Acid with the Conditions Indicated on the Left.

For this example, as shown above, the gradient temperature plate for the reagent was vertical, the hottest end to the top. The plate with the screed lift was placed in a horizontal position on top of the reagent plate as described on the previous page.

The temperatures of the reagent source plate are given along the vertical axis and the temperatures of the lift plate are given on the horizontal axis. The vapor pressure of benzoic acid at the various temperatures are given to the left of the temperatures for the reagent source plate and below the temperatures for the lift plate.

The degree of supersaturation at any point is the vapor pressure produced at the source plate divided by the vapor pressure that would be produced at that temperature on the corresponding position on the lift plate. The degrees of saturation at various positions are indicated by the numbers within the 7 X 7 inch square. The diagonal lines represent approximately the same degree of supersaturation.

The vertical lines in the image to the right are from screed lines of graphite. The horizontal lines are of various operational secret inks.



This is the device that was used to produce the test screed lines that are shown on the previous page.

Each of these ball point pens were spring loaded to produce a writing force of 125 grams. As you can see this devise has been damaged. Three of the pens have been broken off and the left wheel is missing. The wheels were slightly higher that the pen tips so, in writing, the pens were pushed up to the level of the bottom of the wheels and pressed down by the force of the springs.

The lift. We had a press with 7 X 7 inch plates so our work was limited to that size. That is why our temperature gradient hot plates and glass plates were 7 inches wide. The message sheet was placed on the Lexan coated glass and the combination placed in the press and pressed at 50,000 pounds force, which is slightly more than 1000 lb/sq inch.

The glass plates were 7 X 9 inches and that size was selected for the initial gradient temperature work. The glass plate was attached to one of the gradient temperature hot plates with clips at each end and then the assembly was inverted and placed on the other gradient temperature plate which had the reagent source plate. The two gradient temperature plates were positioned at right angles to one another with the middle 7 X 7 inch area common to the two plates. Spacers of about 0.5 mm separated the two surfaces

The source plate. The source plate consisted of an aluminum plate that was lightly sprayed with a solution of the detecting reagent. This plate had corner spacers of about 0.5 mm which determined the spacing between the source plate and the lift surface.

The detect. For the initial gradient temperature work the development was timed for two minutes which was about the optimum time. For later work, with uniformly heated hot plates we could watch the development of the detection and stop it at the desired time which was generally about two minutes. We built a special lighting devise that directed light into the edge of the lift glass plate. The light was totally reflected from one surface to the other as it traveled through glass. Looking down on the plate before detection had started, it looked dark. When a crystal of reagent formed on the surface it allowed light to escape at that point and appeared as a pin point of light. When multiple crystals formed along the written text, the writing appeared as bright lines against a dark background. This lighting system was one advantage of using glass rather than a thin plastic sheet. However, light will travel inside of a thin plastic sheet for a considerable distance. Or the plastic sheet can be optically coupled to a glass plate with a film of liquid, usually water, so that the light passes into the plastic sheet and reflects from the lift surface.

ADL used thin polystyrene sheets as the lift material and since the sheet was not ridged as was glass they used a spacing of about one centimeter. The polystyrene was not temperature controlled and was backed by room temperature air but the sheet was heated from the hot source plate temperature. The sheet was somewhere between the source plate temperature and room temperature. We were sent carbons for Zephyr examination of each new SW system that was developed, generally by ADL. There were some differences in detection results that they obtained and those that we obtained. Some years later we compared the two procedures. We found that some of the battery of Zephyr reagents worked better with a Lexan lift and others were better with Styrene.

Zephyr Defeat. Most of our early attempts to defeat Zephyr was to try to mask the detect. That is, to apply something to the entire paper surface that would provide nucleation sites over the entire sheet and thereby reduce the detect contrast to zero. Graphite was detected by all of the battery of reagents so it was one masking material tried.

We obtained graphite masking by first darkening about 2 square inches of a sheet of paper with a No. 2 pencil (sheet No. 1) This area was then rubbed with a Kleenex -like tissue (tissue No. 1) A new sheet of paper (sheet No.2) was then rubbed with the tissue (tissue No.1). That tissue was discarded and the second sheet (sheet No.2) was rubbed with a new tissue (tissue No. 2). A third sheet (sheet No. 3) was then rubbed with the second tissue (tissue No.2) to produce a masked message sheet. There was no visual indication of the graphite on this sheet. Incidentally this third sheet could be used as a carbon to produce writing that could be detected with high contrast by any of the battery of Zephyr reagents.

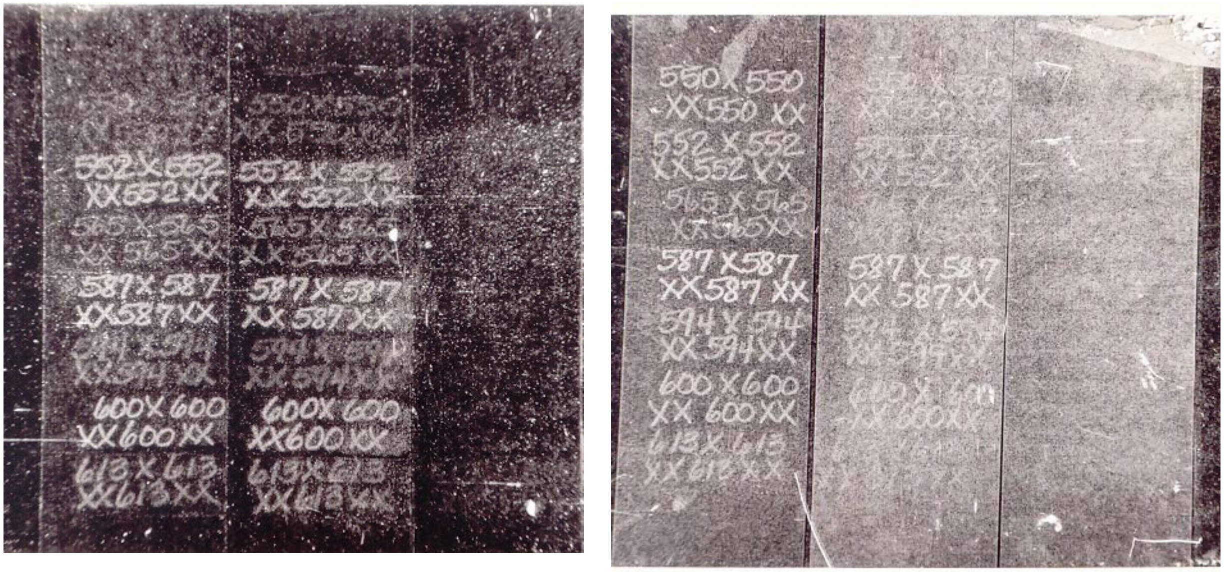
Masking greatly reduced the “signal-to-noise ratio” and reduced the detect contrast but it did not defeat Zephyr.

ADL and others were making fluorinated secret inks with the idea that they would be less polar and less likely to be detected. These compounds were detected with fewer of the battery of reagents but did not defeat Zephyr.

We finally considered the entire Zephyr procedure in a search for a vulnerable step and found the lift step as a likely target. If we could prevent the transfer of SI from the message sheet to the lift surface we would defeat Zephyr. We first tried spraying the message sheet with a solution of film-forming material. We could essentially defeat Zephyr, but it caused problems with recovery of the message. The solvent used for the spray could not dissolve the SI particles and the film needed to be soluble in the solvent used in the recovery procedure. Close, but no cigar.

Pre-rubbing the message sheet before the message was written had become standard procedure primarily to defeat Hot Iodine. In this case the paper was simply rubbed in a prescribed manner with a piece of flannel. We tried "post-rubbing," meaning rubbing after the message was written. The intent was to remove the SI particles from the top surface of the top fibers. The results: This defeated Zephyr. For about half of the systems, the SI was smeared over the entire sheet to also defeat recovery. For the other half, Zephyr was defeated and good recovery was obtained. Electron microscope images of the good post-rubbed messages indicated that the SI particles were simply moved from the top fiber surfaces to the edge of the fibers, the voids in the paper surface. Since recovery is accomplished with a supersaturated solution of the same material as the SI, the SI did not need to be on the very top of the paper surface.

Below are examples of pre-rubbed and post-rubbed screeds.



The Detection of 7 Secret Inks on Three Paper Treatments with Two Zephyr Reagents.

552 X 552 XX552XX	Each set of numbers and Xs above indicate one of the 7 Secret Inks that were being tested. For example, on the left you see sample 552.

The paper treatments for the three columns were:

left -	no treatment;
middle	the paper was pre-rubbed with flannel before it was screeded;
right -	the paper was pre-rubbed, screeded, and then post- rubbed.

These seven systems were recovered well on post-rubbed screeds.

As can be seen, rubbing the paper had little effect with the reagent shown on the left, but significantly increased the background and thereby decreased the detection contrast with the reagent on the right.

SECTION 4.11 FOREIGN SECRET INKS

The Client had obtained many opposition SW carbons and they wanted to know what materials they were using as secret inks. One contractor had been analyzing the carbons for the presence of metals but they were generally finding nothing other than the metals present in ordinary paper. Another contractor was using mass spectrometry by putting a sliver of the carbon in a direct inlet cup, heating the cup and obtaining the spectra of volatile components. The spectra of the SI were generally lost in the maze of junk from the paper. Spectra of dozens of components were usually observed. Many of them were common to many papers and could be disregarded but by this procedure the only separation of components was their difference in volatility as the sample was heated and the data obtained at any moment represented a mixture of material. The success by this procedure was very limited.

We obtained this series of projects based on the fact that we could detect the SI by Zephyr and therefore we would be able to follow it through an isolation and purification procedure leading to instrumental analysis. I came as close to making a hard sale for this contract as any that I had ever made. I was confident that we would be successful.

We got the project and started work. Only then did I realize that being able to detect the secret ink when it was present as secret writing, by Zephyr, did not give us a means to follow the SI through a purification procedure. Once the SI had been extracted from the paper, Zephyr was of no use. The probability of failure loomed large in my mind. I had placed myself deep in a corner and needed to find a way out.

I intended to use thin layer chromatography (TLC) to isolate the SI from other components in an extract of the carbon. TLC that we were using consisted of the use of a glass plate coated with a thin layer of silica gel. A small portion of the sample extract was spotted near the edge of the silica gel coated plate and the plate was placed in a chamber with a small quantity of a solvent or a mixture of solvents. The solvents slowly wicked up through the silica gel and through the sample spot. Each component of the extract mixture had a degree of affinity to the silica gel and affinity, or solubility, to the moving solvent. If a component had a great affinity to the silica gel and little affinity for the solvent it would move very little. If the component had equal affinity for the silica gel and the solvent it would be stationary half of the time, moving with the solvent half of the time and would move half as far as the solvent front. If the component had little affinity for the silica gel and a great affinity for the solvent it would move to near the solvent front. Thus, each component moved depending on its relative affinity for the silica gel and the moving solvent, and the extract components were separated one from another. There were numerous ways to visualize the components on the plate. The most common was to use a TLC plate that contained a fluorescent dye in the silica gel that fluoresced when exposed to short wave ultraviolet light. Nearly all aromatic organic compounds adsorb this wave length of UV and in so doing reduce the intensity reaching the dye. The spot appeared dark relative to a bright surrounding background. This is an oversimplification of TLC but it should give you a general idea of the separation procedure.

Our first work was an attempt to find some properties of the SI to aid in determining what component of the extract was the SI. Solubility was the first property that we explored. We did this by placing narrow strips of the carbon in vials with a small amount of various solvents. The solvent was allowed to wick up the paper strips for about 20 mm (3/4 inch). The strips were then removed, dried, pressed against a Zephyr plate and examined by Zephyr. If the SI was not soluble in a particular solvent it would be detected for the full length of the strip. If it was slightly soluble in the solvent the detection would start a short distance from the wetted end of strip and continue to the top of the strip, and if the SI was very soluble there would be no detection there the solvent had wet the paper. This gave us considerable information concerning the solubility of the SI and, at times, allowed us to select a solvent to remove non-SI components in a preliminary extraction, or to use the mildest solvent to extract the SI without much of the paper components.

But our real break came after considering the properties of all SW carbons. The one property common to all carbons was that the SI must readily transfer from the carbon to the message sheet during the preparation of the secret message. Essentially no paper components are transferred when it is pressed against another surface. The procedure that we finally developed was to press the carbon sample against a polyethylene sheet to transfer some SI to the polyethylene. The SI was then washed from the polyethylene and the wash concentrated by evaporating most of the solvent.

The concentrated wash was then spotted on a TLC plate beside a spot of an extract of the carbon. The extract contained much more of the SI than did the lift, but the extract had many other components and resulted in numerous spots on the developed plate but generally there was only one spot from the polyethylene lift. That spot identified the position of the SI from the extract. The SI from the extract was then recovered from the TLC plate for instrumental analysis.

In a little more detail, we pre-washed the polyethylene sheet in solvents to remove soluble components before it was used to make the lift. The extract from the carbon contained lots of paper junk and the extract was actually streaked along several inches of the 8 X 8 inch TLC plate with the lift wash spotted at the end of the streak. After the TLC plate was developed and the position of the SI located it was recovered. To recover the SI from the TCL plate, the silica gel with the SI was carefully scraped from the glass and placed in a tube with a plug of glass wool as a filter. The SI was then eluded (washed) from the silica gel with an appropriate solvent. The SI was then identified from mass spectrometry and infrared spectrometry data.

Paul Seiwald ran these projects for several years until he retired. He was very successful in isolating and identifying the SI from most of the samples. They were typically organic compounds with molecular weights of between 200 and 400. He confirmed the identification by synthesizing the compound if it was not commercially available and using it to recover secret writing from the original Soviet Block carbon.

SECTION 4.12   INK JET PRINTER SECRET WRITING

The objective of this series of projects was to be able to prepare secret messages with a computer and an ink jet printer. There were almost an endless number of problems to be solved. We had to find a solvent that (1) would dissolve the SI, (2) not modify the message sheet in a detectable manner, and (3) would work in an ink jet cartridge. The SI, when printed on the paper needed to crystallize to serve as nucleation sites for the recovery of the message. Many of the inks that we tried remained amorphous rather than crystallizing. We were successful but it took five years of fairly intense work to do so.

This project started soon after ink jet printing was developed and introduced by Hewlett Packard. The original project plan was for me to work with individuals at Hewlett Packard, but I was soon able to modify the cartridge for our experimental development work and later modify cartridges that were supplied to the client for operational use.

An ink jet cartridge has a series of closely spaced tiny orifices in a plate with a tiny electrical heater behind each orifice. Ink fills the orifices and the small amount of space between the orifices and the heaters. During printing, a jet of ink is ejected from the appropriate orifice at the appropriate time by a heater which vaporizes some of the ink and its sudden increase in volume forces a small jet of ink from the orifice onto the paper. (I believe some printers now use piezoelectric crystals rather than heaters to produce the ink jets.)

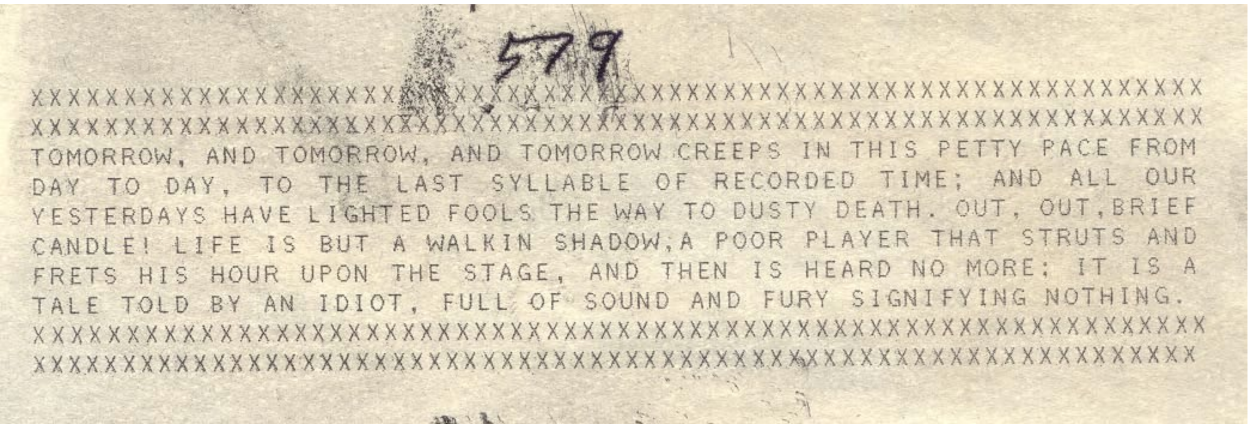
Many ink solvents that we tried did not produce a jet of a single droplet that was needed for good resolution and none of the solvents that both dissolved the SIs and did not give detectable messages were compatible with the plastic components of the ink cartridge. I had to modify the ink cartridge by removing the existing reservoir and replacing it with a glass vial with a glass capillary going from the vial to the print head.

On the following pages are images of the ink cartridge and copies of recovered ink jet secret writing. As stated earlier, this work was done with the first HP print cartridge and the work was completed just before I left MRI in 1996. I do not know if later cartridges have been adopted for secret writing.

The inks that we developed did not produce iodine detectable SW. Therefore it could be loaded into a felt tip pen to write on paper and it would not be detected with iodine. So what I had tried to do and failed in the 1950s and 1960s was a success as a side effect of this work.

The HP ink Jet Cartridge

Top left:	The unmodified cartridge
Top right:	Disassembled cartridge with the rubber bladder removed, part of the top fin removed and a vertical hole drilled though the cartridge
Middle left:	Back view of the front plate. The hole to the orifice plate is at the top of the tapered channel going from the center upward and slightly to the right. The top of this channel was milled away to gain access to the hole.
Middle right:	A small vial used as the ink reservoir, and the reassembled cartridge with glass tube cemented into the body of the cartridge and a capillary placed and cemented in the hole of the orifice plate.
Bottom:	The modified cartridge with the reservoir in place. The reservoir held enough ink to print about twenty sheets.



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TOMORROW, AND TOMORROW, AND TOMORROW CREEPS IN THIS PETTY PACE FROM
DAY TO DAY, TO THE LAST SYLLABLE OF RECORDED TIME; AND ALL OUR
YESTERDAYS HAVE LIGHTED FOOLS THE WAY TO DUSTY DEATH. OUT, OUT,BRIEF
CANDLE! LIFE IS BUT A WALKIN SHADOW,A POOR PLAYER THAT STRUTS AND
FRETS HIS HOUR UPON THE STAGE, AND THEN IS HEARD NO MORE; IT IS A
TALE TOLD BY AN IDIOT, FULL OF SOUND AND FURY SIGNIFYING NOTHING.
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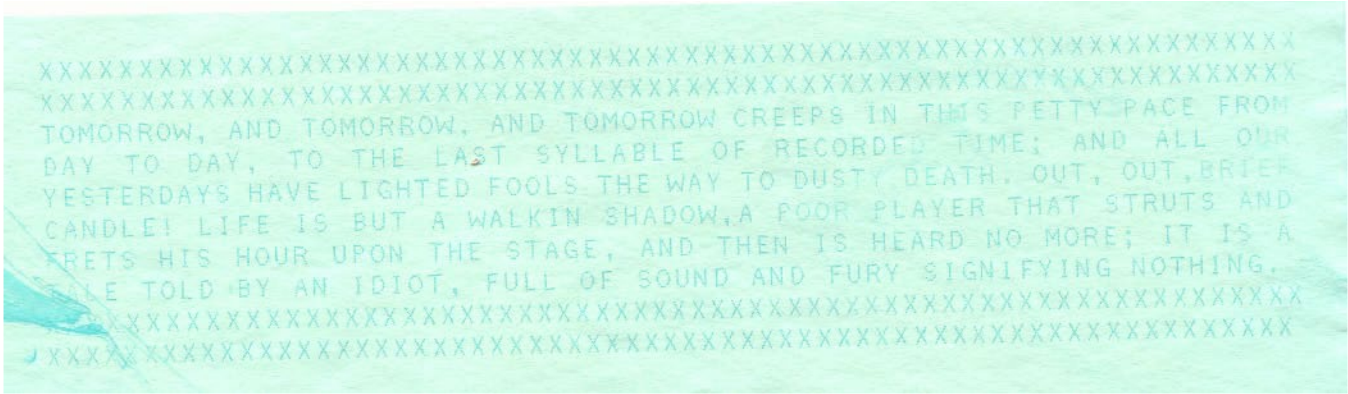

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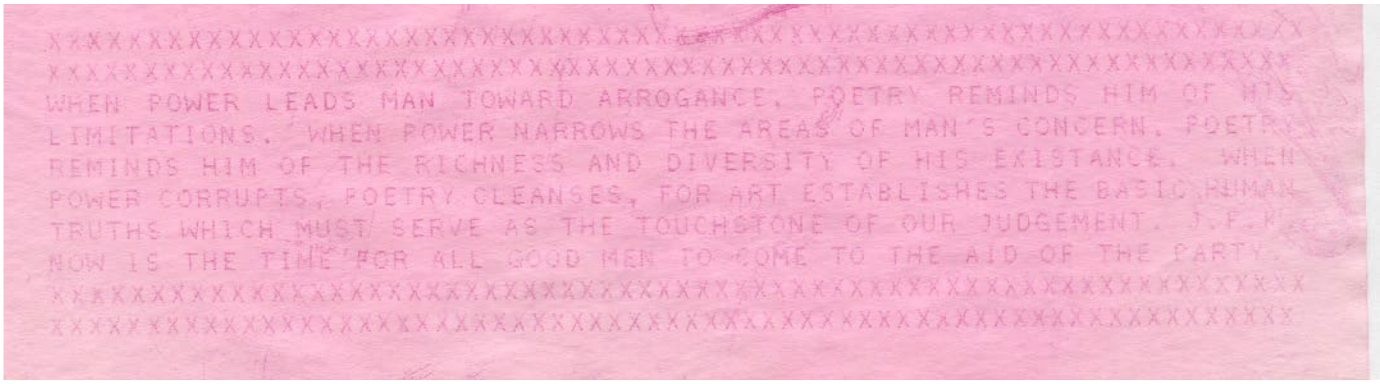

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REMINDS HIM OF THE RICHNESS AND DIVERSITY OF HIS EXISTANCE. WHEN
POWER CORRUPTS, POETRY CLEANSES, FOR ART ESTABLISHES THE BASIC HUMAN
TRUTHS WHICH MUST SERVE AS THE TOUCHSTONE OF OUR JUDGEMENT. J.F.K.
NOW IS THE TIME FOR ALL GOOD MEN TO COME TO THE AID OF THE PARTY.
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Screeds of three systems prepared with Secret Inks from a modified HP Ink Jet printer. The top example was visualized with carbon black in the developing bath; the lower two had dyes in the bath.

Text selections by Leigh Laber, who had a great sense of humor.

On the following two pages are full pages of developed screeds. Both had dyes in the bath. The pale blue dyed the text darker than the paper. The second one the dyed the paper but not the recovered text, thus light on dark.

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THERE SHE WAS JUSTA WALKIN DOWN THE STREET, SINGIN DO WA DIDDY DIDDY DUM DIDDY DE. SNAPPIN HER FINGERS AND SHUFFLIN HER FEET, SINGIN DO WA DIDDY DIDDY DUM DIDDY DE. SHE LOOKED GOOD, SHE LOOKED FINE, SHE LOOKED FINE, AND I NEARLY LOST MY MIND. BEFORE I KNEW IT SHE WAS WALKIN NEXT TO ME, SINGIN DO WA DIDDY DIDDY DUM DIDDY DE. HOLDIN MY HAND JUST AS NATURAL AS COULD BE, SINGIN DO WA DIDDY DIDDY DUM DIDDY
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Leigh Laber, who chose the quotes used above, conducted most of the laboratory work for the last two years of the project. Leigh was a technician without a college degree; as such she was expected to do assigned tasks but was not expected to contribute to the direction of the tasks. However, Leigh did much more. I was very fortunate to have Leigh working with me on this project. Below is a copy of a memo that I submitted at the time of Leigh's performance evaluation.

June 28, 1990

To: Stan Spurlin

From: Gaylord Atkinson

Subject: Leigh Laber's performance

Leigh Laber's work has been consistently outstanding. She is dependable and takes an active interest in the project to which she is assigned. She has performed beyond her assignments. She has made numerous substantive suggestions for the current project.

She has outlined material for the quarterly reports, organized the data for the reports and drafted sections of the reports. She has prepared material for presentation at meetings with the sponsor and actively participates in the meetings.

During the past month Leigh has observed, noted and called attention to two sets of unexpected results. The first seemed to be simply an interesting observation, of little or no practical importance. The second was devastating to the progress of the project. Leigh managed to relate the two and with the first, helped explain the second. She further dug out reports from the previous project (of work done before she was in the group) and related the current results with data in the old reports. These correlations have helped us to partially understand the current unexpected results and have opened the way for a new experimental approach which hopefully will allow us to overcome the impasse in reaching a major project objective in a timely manner.

Leigh's performance is an excellent example of the quality work that is a goal of MRI. In Leigh's case the quality is self imposed and spontaneous. She should be recognized and encouraged.

SECTION 5 GENERAL ANALYTICAL

This series of projects evolved from the Chemical Warfare Analytical projects at MRI with the CIA as project sponsor. Project Leaders, starting in 1962, had been Dr. Earl Barny, Dr. Ted Herman and Dr. Don Rosebrook. I became the Project Leader in 1972 when Don left MRI and I was the Project Leader for the remainder of the time that I was at MRI. More than a thousand samples of very diverse materials were received and analyzed. Many of the samples were one-of-a-kind, unrelated to other samples. Others consisted of series of related samples.

While most of the activities in this series of projects were related to chemical analysis, we also conducted other services for the client. We developed procedures, formulated materials and built equipment for special uses and tested various items for the Client.

For this section I have selected fifteen samples or sample groups that I believe you will find interesting. There were hundreds of other samples. Following discussion of the fifteen selected samples I mention some of the others in a last miscellaneous section.

There are a few other interesting items that I have omitted for security reasons.

SECTION 5.1   TRACKING

NPPD

Don Rosebrook was the Project Leader of the General Analytical project in 1970 when a doormat from our American embassy in Moscow was received for analysis of an oily substance on the mat.
A previous doormat had been found with a similar oily substance, was discarded and replaced with this mat. Within a few days this mat was observed to also be wet with an oily substance. It was now assumed that the doormat contained some sort of a tracking agent and it was submitted to MRI for analysis. The results of the analysis indicated that the major component of the oily material was oleic acid and other lesser components were consistent with the composition of a substance known as “tall oil.” Tall oil is a by-product of the wood pulp industry and “tall” is Swedish for pine. Tall oil has the consistency similar to olive oil or cooking oil. Don submitted a report with the results and concluded that the oily substance was tall oil. He concluded that the tall oil was a solvent or carrier for the tracking agent but no tracking agent was found. Some months later another doormat was received and the analytical results were the same as for the earlier sample. Again, only tall oil and no tracking agent was found.

A third doormat, again containing an oily substance was received in August 1972 about a week before Don left MRI and I became the Project Leader. Again the oil seemed to be only tall oil. I submitted a report with the analysis indicating that only tall oil was found but unlike Don who found no tracking agent, I suggested that the major component of tall oil is oleic acid and oleic acid is a component of animal fat. I suggested that the oily material might be a tracking agent for a trained dog. Jinx Anderson (his name was Glenn, but he was known as Jinx) was my COTR for this project. He requested that I furnish him with a sample of tall oil for testing. I sent it and they got a dog trainer and dog for a trial. Jinx placed some of the sample that I supplied on a towel, stepped on the oily towel, and then walked for eight miles around the streets of Washington. The dog followed his trail for the entire eight miles. That was my first sample on the general analytical project. I was involved with samples related to tracking for the remainder of my time at MRI.

Shortly after that, Jinx woke up in the middle of the night to an apartment full of smoke. He found a sofa burning and tried to carry it outside. He was badly burned and never returned to work.

Additional tall oil containing doormats or paper towel wipes of doormats were received in May and October 1973, and March 1975.

Next we received paper wipes in July 1975. The wipes were from various places inside of a car. They were visibly stained a yellow-brown and produced a yellow fluorescence. The material was not identified but may have been the same material that was identified from the next sample described.

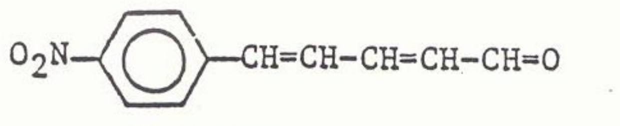
In March 1976 we received part of a facial tissue with a slight orange smudge. The tissue had been used to wipe the steering wheel of a car in Moscow. Something unusual appeared to be on the steering wheel so it was wiped and we received half of it for analysis. (We were told some time later that another contractor received the other half and they found nothing.)

This sample arrived on a Friday afternoon. Microscopic examination showed that the material was a powder and a couple of particles were removed and a melting point obtained. A small portion of the tissue was extracted with acetone and the extract examined by thin layer chromatography. A single spot was seen on the developed plate indicating a single component. It was readily extracted in acetone so we extracted the entire sample in acetone. By that time it was five o'clock and time to go home. We had enough information to know what to do Monday morning. We put the acetone extract in the refrigerator and went home for the weekend.

Monday morning I ran another TLC examination of part of the acetone extract, and to my horror we now had dozens of components. The sample had reacted in, and with, the acetone. The component that matched the location and characteristics of the spot that we had on Friday now appeared to be a minor component of the mixture. We isolated several components from the TLC plate, including the component that appeared to be the original sample and obtained reasonably good mass spectra (MS) and infrared spectra (IR) of the components.

There are MS and IR reference spectra for thousands of compounds in the literature but nothing was found that matched the spectra of this compound. We had to identify the compound from information of the MS and IR spectra. Years later I became fairly good at interpreting spectra and determining the molecular structure from spectra but in 1976 it was new for me. I had identified many unknown compounds by matching their spectra to reference spectra but not by interpreting a spectrum by itself. I worked on the data for weeks without coming up with a reasonable structure. I knew that the molecular weight was 203 which is an odd number indicating that the molecule had an odd number of nitrogen atoms, 1, 3 or 5 etc. It was fairly certain that it had a nitro group, $-NO_2$, an aldehyde group, -HC=O and an aromatic group which would be a six carbon benzene ring or phenyl group. Dan McCausland, an MRI employee in the Organic Synthesis Group, gave me the final clue when he said that the IR spectrum indicated a trans -CH=CH-CH=CH- group. "Trans" indicates that the hydrogen atoms alternate from the top to the bottom of the carbon chain.

So how could the parts fit together? The correct structure turned out to be as follows:



NPPD

***trans,trans*-5-(4-Nitrophenyl)-2,4-pentadienal,**

Its chemical name is 5(4-nitro phenyl) pentadieneal. The '4' indicates the location of the nitro group on the benzene ring; 'pentadien' indicates a five carbon group with two double bonds; and the 'al' indicates an aldehyde group. I created the acronym of NPPD.

While I thought that I knew the structure, I wanted to confirm it. I sent the above structure to Aldrich Chemical Company and asked for a bid for making some of it. They specialized in synthesizing custom chemicals, but for some unknown reason they turned the bid down. (Ten years later when the compound, then known as "Spy Dust" when it became national news, it became available in their next catalog and was listed as Spy Dust.) The synthesis was not difficult so I made some, which produced MS and IR spectra and a melting point identical to the sample. These results confirmed the structure of the compound.

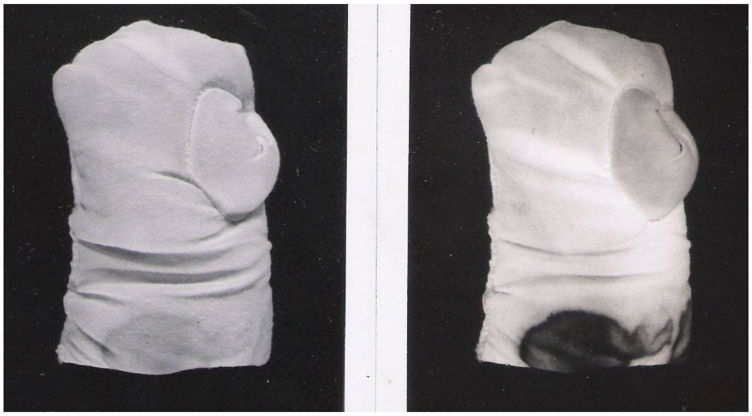
Our next tracking agent sample arrived more than a year later in June 1977. A cotton ball had been used to wipe the interior of a car in Leningrad. The wipe was yellowish and similar in appearance to that of the previous NPPD sample. This sample gave analytical results that were fairly simple to interpret. Chemically it was entirely different than NPPD. It turned out to be the di-napthylhydrozone of terephthalaldehyde. This material was never seen again.

You might wonder, as I did, how did the material get on the inside of the car? These cars were from the US Embassy and were clearly identified by their license plate. The windows were left open a couple inches so anyone that wanted to leave a message could slip it into the car through the slightly open window. The steering wheel could also be sprayed through the window opening.

It was nearly three years before we received the next car interior wipe. We received gauze wipes from cars in March and September of 1980. Both contained NPPD. These samples were different in that they also contained considerable quantities of a common plasticizer, Di-2-ethylhexyl phthalate (DEHP). Apparently the plasticizer was used as a carrier for the NPPD.

Two years later, in November 1982 we received a canvas handbag that we found to be heavily loaded with NPPD. A couple of weeks later we received a ski glove that had come from the same individual. The glove was very contaminated with NPPD. This female employee was nearing the end of her tour of duty, and before returning home she had gone on a ski trip. When she returned to her Moscow apartment she found that many of her things were stained yellow.

(Photographs of the glove are shown on the next page.)



Visible light Ultraviolet

The Inside of the Ski Glove with NPPD

Visually the stain on the bottom of the glove was a pale brownish yellow. The visual intensity of the stain is seen in the black-and-white photograph on the left. NPPD has a very high absorption of long-wave ultra-violet light, and if we could see in the ultraviolet range, NPPD would very much more intense than in visual light. This intensity is seen in the photograph on the right which was taken with the glove illuminated with ultra-violet light and the camera fitted with a filter to allow only ultraviolet light to be photographed.

In December 1982 we received a yellow stained handkerchief that had been used to collect a wipe sample. It contained NPPD along with the large amounts of the DEHP plasticizer previously found.

Problems and questions arose about some of the results because wipes had been obtained using a variety of wiping materials. Some time in 1982 we were requested to obtain and supply a standard material for obtaining wipe samples. We found a company, The Texwipe Company, that sold a variety of cleaning materials, primarily for the electronics industry. We purchased their Texwipe cotton twill 3 X 3 inch cloths. These were to be used to collect wipe samples and returned for analysis. Preliminary evaluation of these cloths showed that there were considerable materials in the wipes that would be extracted in the solvents that would be used in the final analyses. We therefore washed the Texwipe cloths in acetone to remove the potential interfering materials. The resulting cloths were placed in envelops printed for recording sampling information and sent to the client.

The first Texwipe sample was returned in January 1983. It was of a car interior. It was stained yellow and was found to contain both NPPD and DEHP.

Not all Texwipe wipes contained NPPD. In June 1983 we received a wipe of an oily spot on a carpet which was identified as castor oil.

Another sample received in September 1983 was a wipe of a car interior with a paper towel. It was very heavily loaded with the pesticide Malathion. This was probably simply a harassment of the Americans by the Soviets.

In April 1985, a car interior, including a child's carseat was found to contain very high levels of NPPD. The car belonged to a high ranking official who had his wife and one-year-old baby with him in Moscow.

It became obvious that Americans, including children, were being exposed to NPPD. An evaluation of the potential health risks had been made by the Client. This included evaluating NPPD with the Ames Test for mutagenic properties. I know very little about the Ames Test, but I was told that it consists of exposing seven different strains of bacteria to the material being tested and then determining which, if any, underwent genetic mutations. NPPD was reported to be the only material ever tested to cause mutations in all seven strains of the bacteria. If a material is mutagenic it is likely to also be carcinogenic.

Some time, probably in April 1985, systematic sampling of various surfaces was started and twenty of the Texwipe samples were received in May. Eight of them, from Moscow, tested positive for NPPD.

In that same month we received two Ziploc bags that contained material from a spray can. Apparently a KBG agent was paid-off to get these samples. One of the bags contained luminol which is the most common chemical in light sticks used for emergency lighting and the light tubes that are popular at parties. Luminol was detected on wipes from Moscow, East Berlin, Leningrad, and Helsinki. In contrast, NPPD was only found in samples from Moscow.

The other Ziploc bag contents were determined to be closely related to NPPD with a five member, sulfur-containing ring replacing the six carbon benzene ring of NPPD. This material was never detected in any of the wipe samples.

Two things happened in August of 1985:

- MRI received a sole source, $1,000,000 contract to analysis wipe samples taken from various places in the Soviet Block.
- The State Department (and CIA) had become increasingly concerned about the possible health hazards of NPPD exposure and made a formal protest to the Soviet Union. The State Department issued a statement including "The United States deplores the Soviet Union's use of chemical substances against its diplomatic representatives in the U.S.S.R. We have protested the practice in the strongest terms and demanded that it be terminated immediately."

On Wednesday, August 21, 1985, a meeting of the United States Embassy staff in Moscow was held and they were told that the Soviets had been using NPPD as a tracking agent and that there was a potential health risk. Later that day the State Department and the White House issued statements and the White House held a News Briefing.

Ed H. was the project COTR at the time and warned me several days before that the White House News Briefing that it was going to take place. He had had several meetings at the White House and at the State Department providing information concerning NPPD and its use. He told me that while both the White House and the State Department knew of MRI's and my involvement with NPPD, that this information would not be revealed. However, as with all stories in Washington, information leaks were possible. We were told what to say or not say. I was to transfer all requests for information to the MRI security office. They had a statement ready in case MRI was contacted.

Fortunately there were no information leaks. If there had been, that might have ended MRI work for the CIA since our association was classified.

The news media came up with the name "Spy Dust" for NPPD and it was the headline or at least front page news on August 22, 1985 for essentially every newspaper in the country. The cartoonists had a field day. For samples of these articles and cartoons see the appendix.

Some time before the news briefing it had been decided that a team headed by the EPA would go to Moscow and determine the extent of the NPPD use. I prepared a detailed protocol for the sampling and analysis of the samples. My protocol was given to Karen Hammerstrom of the EPA. I knew Karen from the time I worked on asbestos for the EPA but she did not know of my involvement with the CIA or with NPPD. She did not know who had prepared the protocol. Karen made one critical change to the protocol. My instructions were to take dry wipes. In Karen's procedure, the Texwipes that were taken to Moscow were pre-wet with a solvent (cyclohexane) and placed in cap-sealed vials. Everyone, including Karen, knew that a damp cloth is better than a dry one for picking up dust but what she didn't know was that with the solvent the NPPD would become attached to the fabric and become undetectable.

So 418 surface wipe samples were taken and the still wet wipes were resealed in the vials and brought back and delivered to Versar, a laboratory in Springfield, Virginia for analysis. Not surprisingly, no NPPD was detected. By using wipes wet with solvent, the NPPD had either degraded or more likely became attached to the fibers of the cloth. The first step of the analysis was to extract the NPPD from the wipe with methylene chloride. NPPD is very soluble in methylene chloride but it is a mild solvent and would not displace NPPD that had become attached to the wipe fabric. Although Karen had changed the protocol, she apparently never ran a control of the modified procedure to determine if NPPD would be detected. If she had wiped a surface known to have NPPD with the wet wipes and then stored them for a few days, she would have found that NPPD would not be extracted with methylene chloride and therefore would not be detected by her modified sampling procedure. Without a recovery control, she was conducting very bad science.

The CIA managed to get about 50 of the wipe samples from Versar after the wipes had been analyzed and no NPPD detected. Some of these wipes were still slightly visibly yellow and

NPPD was readily seen on many of the wipes with ultraviolet photography. By modifying the extraction procedure, we were able to confirm the presence of NPPD on several of the 50 samples. The official (non CIA) results were that no NPPD was present and the case was closed.

Continuing evaluation of the health risks of NPPD exposure indicated that the risks were minimal if any.

MRI received and analyzed 793 wipe samples from May 1985 through February 1986. We were specifically looking for NPPD and Luminol. The results are given in the following table.

ANALYTICAL RESULTS OF WIPE SAMPLES TESTED FOR NPPD AND LUMINOL

The city of origin of samples that tested positive is indicated as follows:
M = Moscow, EB = East Berlin, L = Leningrad and L = Helsinki

Month(s)	Samples	Samples with	
Month(s)	Received	NPPD	Luminol
May 1985	20	8 M	
June – July 1985	88	3 M	1 EB 4 M
Aug. – Sept. 1985	300	16 M	2 M 1 H 1 L
October 1985	85	3 M	5 M
November 1985	67	3 M	1 M
December 1985	39	1 M	4 M 1 L
January 1986	93	1 M	-
February 1986	101	2 M	8 M 5 H 3 EB

Luminol was found from all four cities, NPPD was found only from Moscow samples.

The turnaround time from when wipe samples were taken until the results were received back in Moscow or other Soviet block cities was longer than was desired. We were therefore requested to develop a field kit for analyzing the wipes in the various Embassies with no laboratory facilities. To comprehend the enormity of this request I must tell you the procedure that we were using to analyze the samples. Our procedure was adapted from standard procedures in use for the preparation of environmental samples for instrumental analysis. We purchased and used the accepted equipment for doing the accepted procedures. The procedures were labor intensive.

First the Texwipe sample was placed in a special piece of glassware for extraction. I wish I had a picture of the glassware, but I do not. The glassware had a glass chamber about ¾ inches in diameter and three inches long centered in a glass tube about 2 inches in diameter. Most environmental samples were placed in a paper or fiberglass thimble and inserted into this chamber. The Texwipe sample wipe was placed directly in the chamber. The bottom of the outside tube consisted of a ground glass joint to join with a small round flask. The top of the outer tube was connected to a water-cooled condenser. In operation, 25 milliliters of extracting solvent was placed in the lower flask which was heated to boil the solvent. The resulting hot vapors went up past the inner chamber to the condenser where it was cooled and condensed to a liquid. The bottom of the condenser was constructed so that the descending solvent dripped into the top of the chamber with the wipe. A small siphon tube was attached to the bottom of the chamber and looped up to a level near the top of the chamber. In operation the hot solvent would drip into the top of the chamber and onto the wipe and fill the chamber until it reached the level of the top of the siphon tube at which time the solvent would siphon out of the chamber, return to the flask and the cycle would be repeated. This repeated extraction continued for about 30 minutes which was about ten cycles.

Following the extraction, the solvent volume needed to be reduced from 25 ml to about 0.1 ml for it to be introduced into the mass spectrometer and other analysis. Another set of standard glassware was used for volume reduction to about 1 ml and again the extract was transferred to another smaller container for the final volume reduction to 0.1 ml, about two drops.

This entire procedure was run first with methylene chloride to extract the NPPD and then repeated with methyl alcohol to extract the luminol.

This was not something to be worked into a field kit.

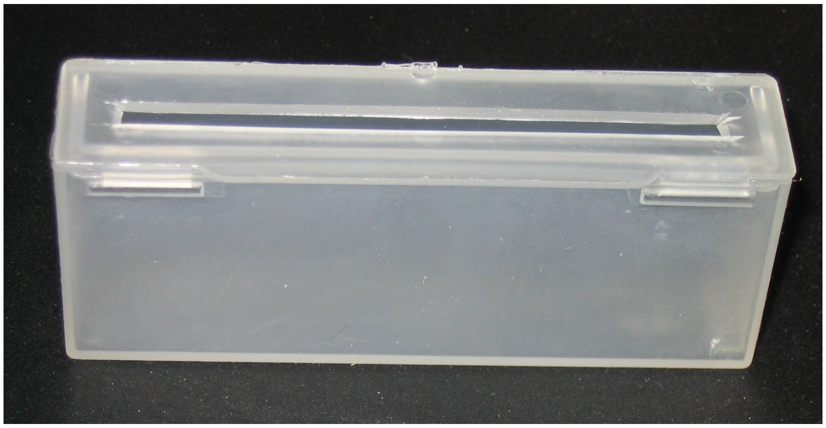
It took me about two weeks of thinking and trying many things before I came up with a procedure that could be run with minimal facilities. The procedure was to abandon the Texwipe cloths for collecting samples and use a triangle of filter paper. (specifically, Whatman Chromatogaphy paper No.2) The new wipe material consisted of equilateral triangles of a filter paper 2 inches on a side.

We were specifically testing only for NPPD and luminol. NPPD is very soluble in methylene chloride and luminol is not soluble in methylene chloride but is very soluble in methyl alcohol. So the NPPD was first extracted from the wipe with methylene chloride and then the liminol was extracted with methyl alcohol. Modified microscope slide mailing containers were used for the extraction. The modification consisted of cutting a slit in the top of the container to produce an extraction chamber, so that one edge of the triangle could be slipped into the chamber. For NPPD extraction, a small amount of methylene chloride was placed in the chamber and the triangle inserted. The methylene chloride wicked up the filter paper, dissolved the NPPD and took it to the very tip of the triangle. The triangle was removed from the container, allowed to dry for a few minutes and the tip cut off and placed in a small vial. A small drop of solvent was then added to the vial and the sample it was ready for I insturmental analysis. The extraction procedure is illustrated on the following page.

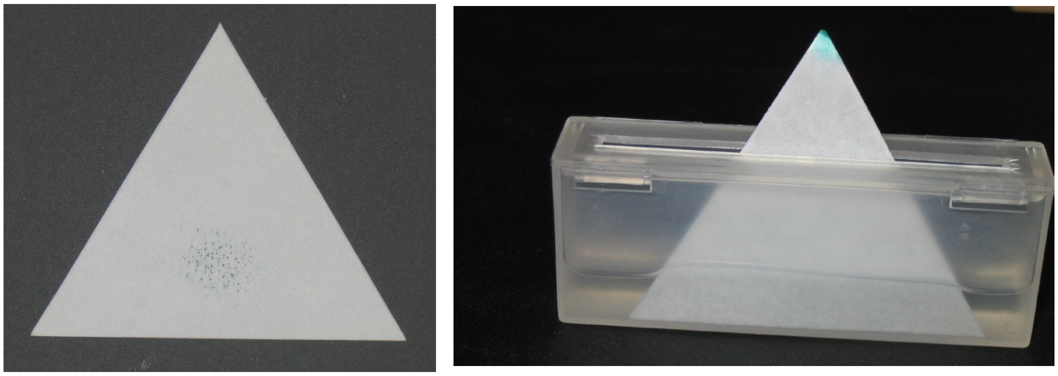
After the tip of the triangle containing the NPPD was removed, the triangle was rotated 120 degrees so that another tip was at the top and it was placed in a holder with a small amount of methyl alcohol. Now the methyl alcohol wicked up the triangle dissolving the luminol and transferring it to the new top tip of the triangle. Again the tip was cut from the triangle, placed in a vial with a small amount of solvent for further analysis.

We went from a procedure where one person could work up about eight samples in a day (an average of one hour per sample) to a procedure that took only a few minutes per sample. The amazing thing was that the sample recovery was better from the triangles and the simple sample recovery than the Texwipes and the elaborate standard recovery procedures.

At MRI the analysis for NPPD was by mass spectrometry. Since mass spectrometry was not available in foreign embassies we had to come up with a simple detection procedure. NPPD has a very high absorption of long wave ultraviolet light. If we could see in ultraviolet, NPPD would look very black. We had photographed wipes illuminated by UV light with a camera fitted with a filter to block visible light but pass UV light. These photographs indicated that very low amounts of NPPD could be seen. For the field kit we purchased small surveillance cameras, with monitors, and fitted the camera lenses with ultraviolet filters. With this camera system, triangle wipes could be examined before anything else had been done to them. If no dark material appeared on a wipe, no NPPD was present and no further treatment for NPPD detection was required. If dark material was seen in this examination, it may or may not have been NPPD, and further work was required. In this case the triangle would be placed in the container with methylene chloride and allowed to wick to the tip. The triangle would again be examined with the camera. If the dark material did not move, it was not NPPD and it could be concluded the sample was negative for NPPD. If it did move to the tip, it was probably NPPD but for further confirmation the tip could be cut off, extracted and a simple TLC examination would provide information as to whether NPPD was or was not on the wipe.



Microscope Slide Mailing Container Modified to be an Extraction Chamber.



An Illustration of the Triangle Extractions

For this illustration, green ink from a felt tip pen is a substitute for NPPD or luminol.

Left:	The green ink was placed on a sheet of plastic and allowed to dry. The sheet was then wiped with the triangle. The ink can be seen near the lower center of the triangle.
Right:	The green ink has been moved to the top tip of the triangle. For this illustration, acetone was used and as it wicked up through the paper it dissolved the green ink and moved it to the tip of the triangle. For the real procedure, the tip of the triangle would then be cut off, placed in a small vial and made ready for instrumental analysis by adding a small drop of the appropriate solvent.

Luminol

When a solution of hydrogen peroxide (or other oxidizing reagents) with a catalyst is added to a solution of luminol it produces chemiluminescence, meaning it "gives off light." Eventually we were authorized to purchase fairly expensive illuminometers for analysis of sample extracts for the presence of luminol. For the first few samples analyzed for luminol, mass spectrometry was used, but that was expensive and not very satisfactory. So some other method that detected the chemiluminescence was needed.

I developed a procedure that used a Polaroid film pack. You may or may not be familiar with Polaroid cameras which are not very prevalent anymore. A picture could be taken and a print produced in about one minute.

Understanding the Polaroid possess is not important for this discussion on the detection of luminol, but I have always been fascinated by it and so I am included a brief explanation here.

When a picture was taken the optical image exposed a surface that became a negative. After the exposure, a tab was pulled which brought the surface that became the negative (the exposed surface) and the surface that would become the positive photograph together with a controlled, very thin, coating of developer between them. The amount of developer was critical. The "negative" surface and the "positive" surface competed for the developer. I believe that there was a coating on the positive surface that gave the negative surface a head start. For a light area of the image the negative used up all of the developer so there is none left for the positive surface and it remained white. Where the image was black, no developer was used by the negative and it remained for the positive image which became black. For areas of the image between black and white, part of the developer was used for the negative and part for the positive surfaces.

The final photograph size was the same as the exposed image in the camera. I only used the film pack without a camera. The film pack that I used produced a picture that was approximately 2.5 X 3.75 inches. For luminol detection, I made a wooden block the same size as the Polaroid picture and drilled 12 holes, in a 4 X 3 pattern through the block to hold glass vials. A glass plate was attached to the bottom of the block to hold the vials but let light pass through.

For the examination of the extracts of wipes for the presence of luminol, the extracts for the various samples were placed in the vials and the vials placed in the holes in the block. A beaker of hydrogen peroxide and catalyst was prepared and an eye dropper placed in the beaker. The next steps were conducted by feel in the dark. The cover was removed from the Polaroid film pack exposing the film surface and the block holding the extract samples positioned on that film surface. A dropper of the mix of hydrogen peroxide and catalyst was added to each vial. If luminol was present, light would be emitted and the Polaroid surface exposed. We placed numbers, on the glass on the bottom of the block so a positive detect would record its number on the developed photograph. There were 12 positions for vials. The two upper left positions were for controls and the other ten were for samples. The position numbering was as follows:

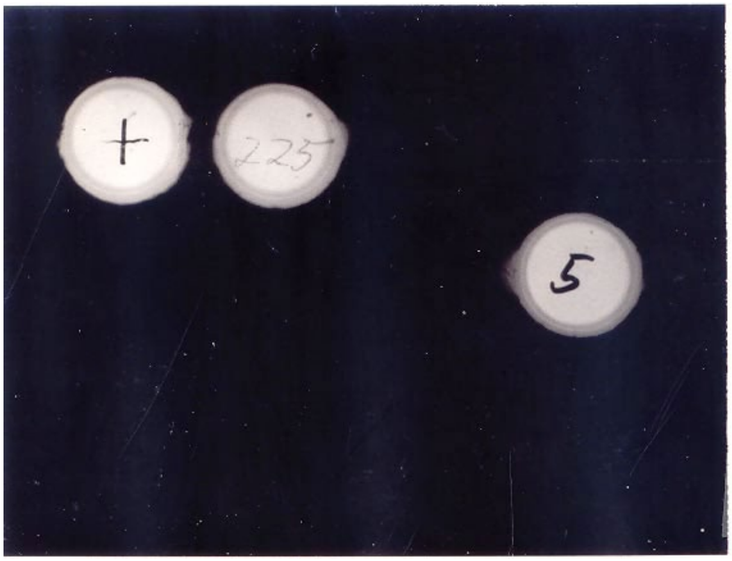
C1	C2	0	1
2	3	4	5
6	7	8	9

- C1 generally had the date of the run and was a control with 20 ng (nanograms, billionths of a gram) of luminal.
- C2 was a 5 ng control and had the first three numbers of the 4 digit sample number
- The remaining positions had the fourth digit of the sample number

Examples of the luminal detects are shown on the following page:

Polaroid detects of the chemiluminescences of various control amounts of luminol.

Position	ng of luminol	Position	ng of luminol
C1 (with date)	20	4	2
C2	5	5	1
0	50	6	0.7
1	20	7	0.5
2	10	8	0.3
3	5	9	0.0



Polaroid examination results of triangle wipe samples 2250 to 2259.
Sample 2255 was positive for luminol. The other nine samples were negative.

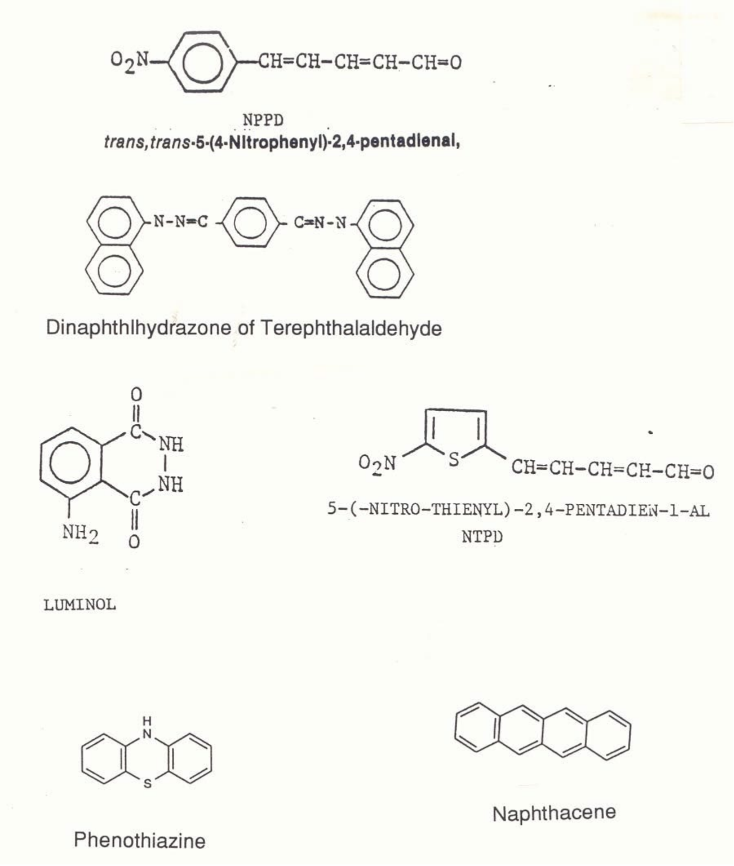
Raduga

The Client had learned that there was a battery of several tracking agents that, when sprayed with a certain material, each fluoresced with a characteristic color. It was reported that the system was called “raduga” (spelling?), which translated to “rainbow” in English. Thus different tracking agents could be applied to different individuals and the material, inadvertently transferred to something or someone else, could be identified by the color of the resulting fluorescence. We had known about raduga for several years and we were asked to try to figure out what it was.

If there was a battery of several tracking agents, it was probable that some of these would have been on the wipes that we had received and analyzed for NPPD and luminol. All of the sample extracts had been saved in a freezer. We went back and re-examined these extracts for the presence of other materials. We found two materials, each from several samples, that were unusual for the environment. These were naphthacene and phenothiazine.

Finding naphthacene provided the clue for solving the identity of raduga. I knew that naphthacene fluoresced a bright yellow when it was treated with anthracene. We now knew of six tracking agents and spots of each were sprayed with a solution of anthracene. All but luminol fluoresced giving a variety of colors. Luminol would have been detected by its chemiluminescence’s.

I retired as a full time employee in 1991 and worked part time for an additional five years. We solved raduga in 1993. If I had not continued to work part time I would never have known the complete raduga story. The key to solving raduga was my knowledge that naphthacene fluoresced when treated with anthracene. Would anyone else have come up with that information? Who knows?



These are the six Soviet tracking agents that we identified.

My final work related to tracking was to test and document the color and intensity of several other materials that fluoresced with anthracene. Some are shown below. I also formulated a cleaning solution and developed a procedure to wipe surfaces to destroy the tracking agents.

Fluorescence of several chemicals when treated with antheracene.

The fluorescence was somewhat more intense than shown in this photograph. The columns right to left are:

- Not sprayed with anthracene
- Aged at room conditions for seven days then sprayed
- Sprayed without aging

Epilogue

The work that was done relating to tracking agents was extremely important to the Client. They had some excellent CIA agents who had recruited key Soviet citizens and they were very careful when making contacts to avoid detection. In spite of their care, several of their recruits (also known as “assets”) were arrested and executed. It was concluded that Soviet tracking techniques must be how they were finding our assets. That was why it was so important to learn as much as possible about their tracking techniques.

Shortly before I retired it was learned that that the Soviets had a much simpler way of finding our assets. The Soviets had recruited two U.S. citizens who had access to the names of the assets that we had recruited. Aldrich Ames was a high ranking CIA employee and betrayed the United States for money. Robert Hanssen decided when he was a teenager that he wanted to be a spy for the KBG. He tailored his education so that he could join the FBI and worked his way up to a position that gave him access to information that would be valuable to the Soviet Union. So in his case, it was not that he was recruited by the KBG but rather he contacted the KBG. Hanssen received a considerable amount of money from the KBG but his real incentive was the thrill and excitement of what he was doing. Apparently neither of them was concerned that their action was resulting in the death of others.

Both Ames and Hanssen are serving life sentences. They avoided execution by agreeing to tell the U.S. details of what they had done.

Apparently the KBG obtained names from Ames and/or Hanssen of our CIA agents working in the Soviet Block and the names of the assets that they had recruited. Apparently, the KBG then used the tracking agents to confirm the relationship.

An interview of Aldrich Ames by Tim Weiner appeared in the July 31, 1994 New York Times Magazine. A copy of this interview is included in the appendix.

*****

In 2007, a movie of the life of Robert Hanssen was released. It was called “Breach.”

SECTION 5.2 POTASSIUM PEROXIDE K_2O_2

In about 1980 a container washed up on the shore of a Greek island. It was a sealed metal container and was about 21 x 21 x 25 cm (approximately 8 x 8 x 10 inches.) It had Russian printing on the outside. The Greek authorities turned it over to the US Navy. The Navy cut the container open and saw nothing that made any sense to them and left it open on the deck exposed to the weather. It got soaked.

Eventually, it got turned over to the CIA and was identified from the printing as material used in a submarine to purify the air and supply oxygen for the crew.

We received the container and identified the active material as Potassium Peroxide (K_2O_2) and it not only supplied oxygen but removed carbon dioxide and water from the air. It's function was the opposite of respiration.

Potassium peroxide reacts with water to produce oxygen and potassium hydroxide. The potassium hydroxide then reacts with the carbon dioxide to form potassium bicarbonate.

$$2K_2O_2 + 2H_2O \rightarrow 4KOH + O_2$$

$$4KOH + 4CO_2 \rightarrow 4KHCO_3$$

The combined reactions:

$$2K_2O_2 + 2H_2O + 4CO_2 \rightarrow 4KHCO_3 + O_2$$

The US had observed the Soviets loading submarines and knew how many of these containers were on a sub, but they didn't know what was in the containers and how much air they could purify. We were ask to determine the contents of the container and determine how much oxygen they could supply.

The analysis was not easy since the Navy had left the open container on the deck in the rain. About two thirds of the potassium peroxide had reacted with the rain and by the time we received the container the exposed surface was a relatively thin layer of potassium bicarbonate over rock hard potassium hydroxide. Twenty steel dividers that turned out to be holders for twenty 20 x 20 x 1 cm slabs of the potassium peroxide, extended above the solid cake of potassium hydroxide.

We cut the container open and could readily see the layers of three components, at that time unidentified. The bottom third was a bright canary yellow and was the potassium peroxide. Most of the remainder was potassium hydroxide with a thin top layer of a mixture of potassium carbonate and potassium bicarbonate. The sample was placed in a "glove box" to protect it from exposure to the moisture of the atmosphere and portions of each layer were removed and the composition identified. The layers were then separated and weighed. The separation was not easy since the work was done in a glove box and the potassium hydroxide was very hard.

The sheets of potassium peroxide contained about 5% of asbestos fibers to hold it together. The rain water that the navy allowed to get in the container reacted with about two thirds of the potassium peroxide but the asbestos was unchanged. We determined the exact fraction of asbestos in the undamaged portion of the sample, and the fraction of asbestos in each of the other layers. With this information we calculated the total amount of asbestos in the container and from that we calculated the original total amount of potassium peroxide.

We also determined the weight of a known volume (area of a portion of the 1cm thick sheet) of the potassium peroxide from portions of the undamaged sample. From the size and shape of the steel holders it appeared likely that the original sheets were 20 x 20 cm. The results of each of the independent analysis and calculations were in excellent agreement for the total amount of the original potassium peroxide. It was concluded that the container consisted of twenty 20 x 20 x 1cm sheets.

Weighed amounts of the potassium peroxide sheet were exposed to water and the volume of oxygen that was produced was then measured. The measured amount was in excellent agreement with the expected results from the equations given above.

A detailed report with numerous photographs was prepared and submitted.

The story doesn’t end here. It turned out that this Soviet system was superior to the system the US was using and the determined oxygen producing capacity of the container was about 15% higher than had been assumed. Thus the Soviet submarines could stay submerged 15% longer than had been believed. This was important intelligence information.

I was told that at that time, there was a biweekly publication that included the most important intelligence for that period from each of the various US intelligence organizations. These included the CIA, the FBI, and the intelligence branches of each of the armed services. I was told that my report was the feature article in the next issue. It was classified at a level well above my Secret Clearance so I never saw it. My COTR told me that the CIA Analyst who received my report simply removed our report cover and the page that identified MRI and me as the author of the report and added a page with her name as the analyst and another page or two stating the significance of the results My COTR was unhappy that he (and I) were given no credit.

Anyway this was a short time before I received the Agency Seal Medallion and this work and the report were probably strong contributing factors. So I think, in that way, I did receive credit.

Photographs of this sample are on the following page.

Photographs of material from the Soviet Potassium Peroxide container.

The top photograph is of the very hard potassium hydroxide at the top of the container, the yellow in the bottom photograph is the potassium peroxide.

SECTION 5.3 DROP ROCK

This was a very important sample and required some detective work. Unfortunately I was working against a CIA employee, not against the Soviet Union. The work extended over several months.

A drop rock is a container made to appear as a rock. It is used to pass information or supplies to a person who is a spy for the U.S. when it is not desirable or safe to meet with the individual. In this case the rock contained a small camera, some film, some medicine for the individual and, most importantly, micro film with the entire intelligence operation plan for a country in the Soviet Block for the following year.

The "rock" had been placed next to a tree in a park and should have been picked up the same day, or night. A few days later, the individual who had placed the rock recognized it, now part of a nearby rock wall. Apparently someone had thought that it was a real rock and added it to the wall… or had something else happened?

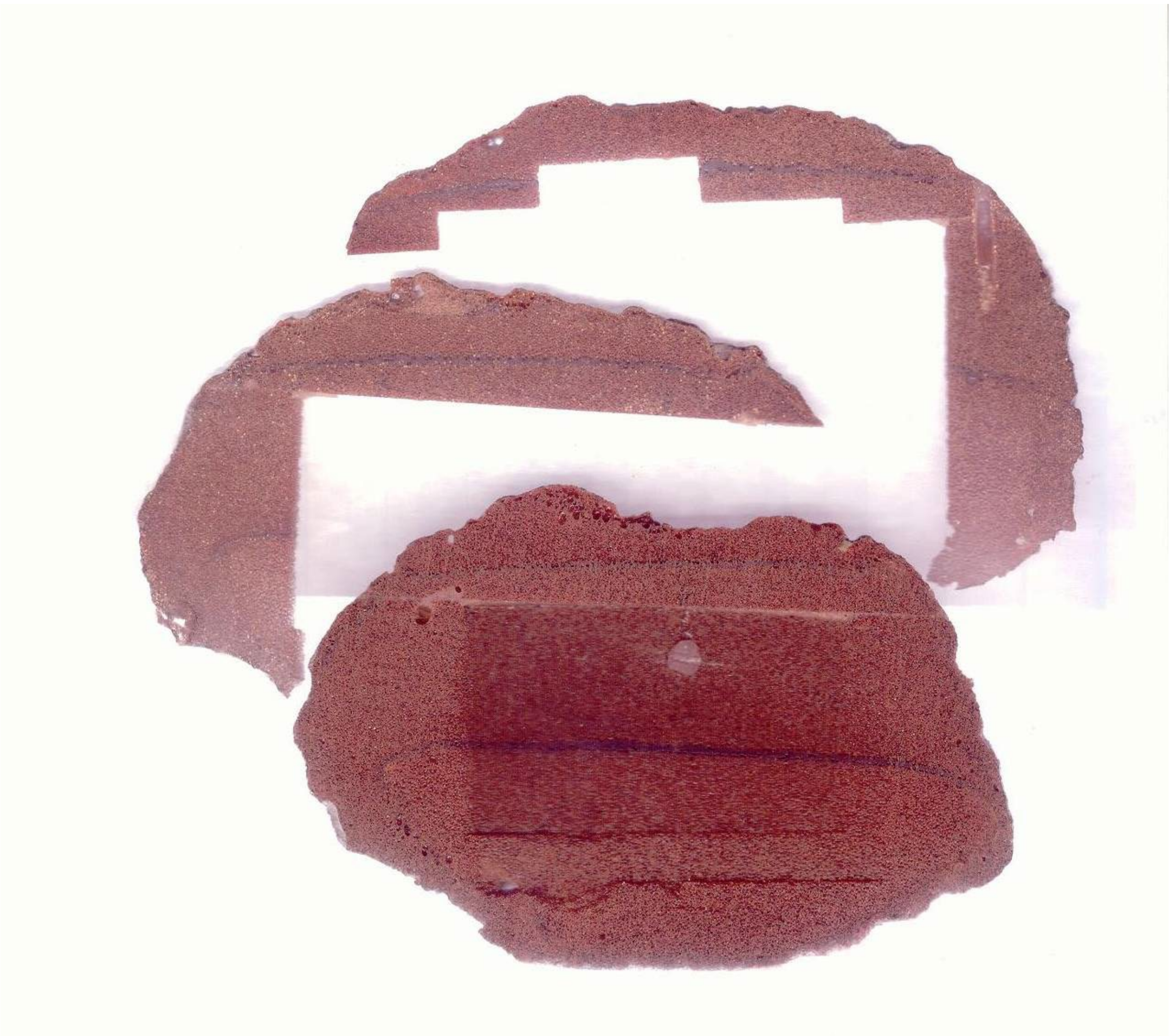
The rock was retrieved and returned to the US and examined by the individual who made the rock. He concluded that the rock had been cut open and cemented back together. The implication was that the information on the microfilm had been compromised, and the rock repositioned in a place that would be found by the US so we would not think that it had been opened. If, in fact, this is what had happened, the opposition would know our plans and think that we did not know that they knew.

MRI was asked to identify the adhesive used to reseal the rock.

The drop rock was made by starting with a real rock the size needed to hold the intended contents of the rock. A silicone rubber mold was made of the real rock and the mold was then used to cast a rigid polyurethane foam rock. The foam rock was then cut into two parts and a cavity milled out for its intended contents. The contents were then placed in the rock and it was cemented back together. This particular rock was then sprayed with an adhesive and tumbled in fine sand to make it appear as a sandstone rock.

The location of the adhesive used for the original sealing of the rock was obvious and formed a straight line. The line that was alleged to be adhesive when the rock was resealed was not straight.

After a few minutes of visual examination of the sample, Paul Seiwald's comment was "This is going to be interesting". It was obvious to Paul and me that the second line was not of a cut that had been resealed but a skin that had formed on the top of a first patch of polyurethane that had been added to the mold. Additional urethane had then been added. In fact there was another skin line near the top. Photographs showing the lines in the polyurethane appear on the next page.



Cross Sections of the Drop Rock

The polyurethane foam rock was cut into slices to reveal its properties.

After the “rock” was cast, holes were drilled around the outside top of the rock to aid in the alignment of the top when it was reassembled. The top was then cut off and a cavity was milled out of the rock to hold its contents. The top was then reattached with epoxy adhesive. The epoxy is the straight, light-colored line at the top of the cavity. Alignment holes can be seen on the right of the top slice and on the left of the bottom view.

The dark line slightly below the middle of the rock in the bottom view was the original item of concern. The individual who made the rock thought this was an adhesive to reseal the rock after it had been cut open by the opposition. It seemed to us that this was a skin formed at the top of a batch of urethane that had been poured into the mold. The skin formed because surface bubbles break leaving a layer of urethane with few bubbles. Note that this line curves down at each edge of the rock.

When urethane is mixed with a catalyst, a gas is produced in the reaction, and that results in the polyurethane foam. (With other starting materials, gas is not formed and the resulting polyurethane is a hard rubber like material. We used this type of polyurethane in the work described in Section 5.13) If the gas bubbles of the foam break at the surface, a skin is formed. In this sample, while there was a definite skin line of material with few bubbles, there were some bubbles within the about 1mm-wide line. The fact that there were bubbles in this line was reasonable proof that the line was not an adhesive used to reseal the rock.

We removed material from (1) the foam, (2) from what we considered to be the skin, that they thought was an adhesive and (3) the adhesive originally used to seal the rock and obtained infrared spectra of them. The foam (1) and the skin (2) were identical and were polyurethane. The adhesive (3) was an epoxy adhesive.

We submitted a report with the spectra and with numerous photographs with conclusions approximately as I have just described. That should have been the end of it. It wasn't.

The individual that made the polyurethane rock stated emphatically that the urethane had all been added to the mold at one time.

Further, the photographs in our report showed a series of 1/8 inch holes that had been drilled into the rock around the outer edges. We assumed, and concluded, that these holes were drilled before the polyurethane rock was cut prior to milling the cavity and were used to place pins for alignment when the top was reattached. The maker of the rock responded by saying, while he sometimes used holes and pins for alignment, they were not used for <u>this</u> rock.

His conclusion was, if the second line was not adhesive, and if there were alignment holes, it was not the rock that he had made. It was a copy!

If it was a copy, the implications were tremendous.

For about the next three months we would analyze some property of the sample, send in a report suggesting that it was not a copy, and he would counter with a statement that the characteristics that we described did not describe his rock. It had to be a copy.

He had sealed the top to the body of the rock with Devon Two Ton Epoxy cement. We purchased that epoxy and about ten other epoxy adhesives. There were significant variations in the infrared spectra (IR) of each of the samples, and the epoxy from the rock only matched that of the Devon Two Ton Epoxy.

His counter statement: "They" must have analyzed the epoxy and used the same.

The rock had been sprayed with "3M 77" adhesive and tumbled in a fine limestone sand to give the appearance of a sandstone rock.

We were sent a sample of the sand that was used to coat the rock. We dissolved the adhesive on the rock in a solvent and recovered both the adhesive and the sand. The IR spectrum of the adhesive matched that of 3M 77. The sand from the sample matched that supplied by the Client in composition and size distribution.

The maker of the rock still maintained that it was not the rock that he had made. He insisted that the urethane had been added to the mold in one continuous operation and no alignment holes and pins had been used. He said, “They had to have made a mold of the original polyurethane rock and made a copy.” My view was that “they” would have had to have been extremely talented and have outstanding facilities. Within a very few days they had analyzed the rock and acquired American made Devon epoxy and the 3M adhesive and acquired matching limestone sand.

The rock maker did not back down.

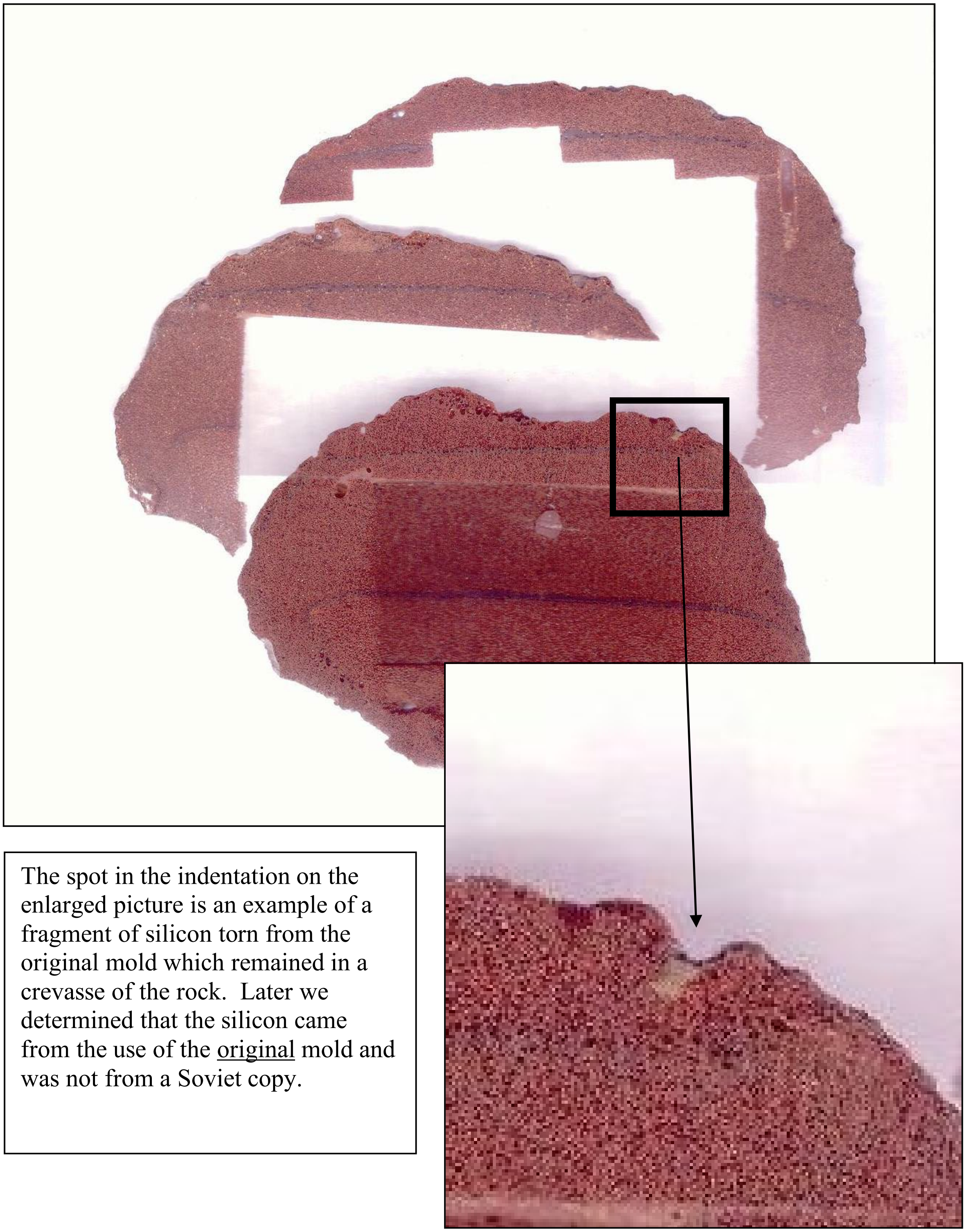
Finally, we received the silicone mold that was made to make the original polyurethane rock. We now had the following alternatives:

- The original silicone mold. I’ll call it M1
- The original polyurethane rock, or R1
- The silicone mold that “they” supposedly made from R1 which will be called M2
- And the polyurethane rock that “they” made in M2 which will be called R2

If the maker was correct we now had in our hands M1 and R2.
If he was not correct we had M1 and R1.

The question was, did I have R1 or R2? Since at that point which rock it was was not established, I called it RX. From RX, it was apparent that the real rock that was used to make M1 was sandstone with a considerable amount of gravel in it. The surface was very rough and there were fairly deep indentations or crevices in the surface. These crevices would become projections on the surface of the silicone mold. There were a few locations on RX where the silicone had not released in these crevices and the silicone had torn leaving some of the silicone in the crevice. *See photographs on the next page.*

Most of the torn silicone fragments on the surface of RX were small and no larger than about one or two millimeters across. However a few were larger and one was much larger and was roughly a triangle with sides of 3 x 4 x 5 mm. The torn surface was about 2 mm below the surrounding rock surface and a thin needle inserted down along the boundary of the silicone and the polyurethane indicated that the crevice extended about 4 mm below the torn silicone surface. So the crevice in the original, real rock, from which M1 was made, was about 6 mm deep. Thus M1 originally had a corresponding projection of 6 mm.



The spot in the indentation on the enlarged picture is an example of a fragment of silicon torn from the original mold which remained in a crevasse of the rock. Later we determined that the silicon came from the use of the <u>original</u> mold and was not from a Soviet copy.

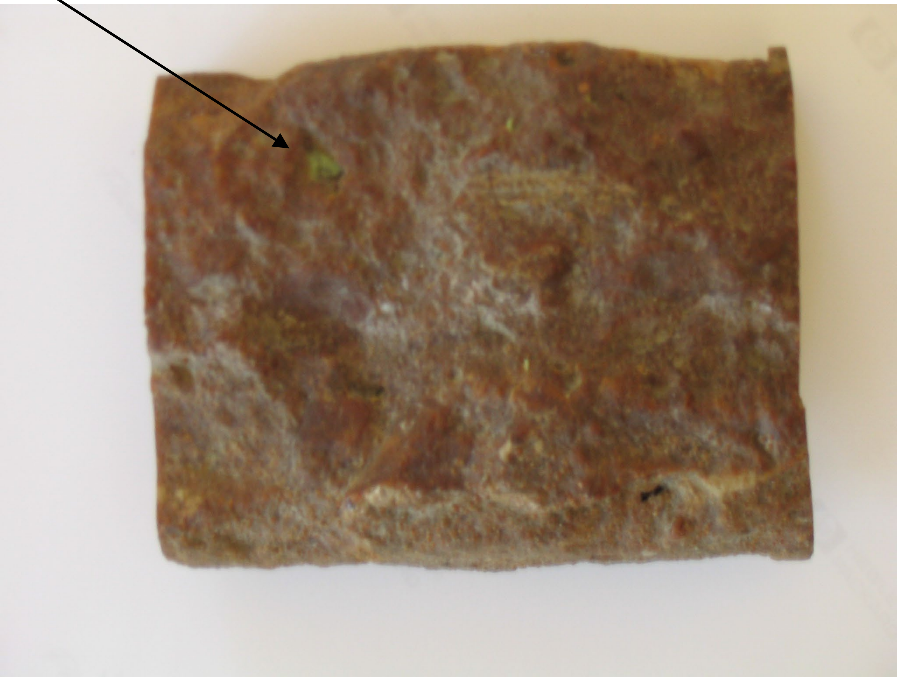
When silicone is mixed with the curing catalyst, air bubbles are often introduced into the mix. In fact it is difficult to keep from getting air bubbles in the mixture. An air bubble was in the silicone that was in the crevice described in the preceding paragraph and the silicone tear was through that bubble. Half of the 0.8 mm bubble was with RX the other half was with either M1 or M2.

If RX was R2 (the rock supposedly created by the opposition) and came from M2, M2 would have had to have had the 6 mm projection which tore when R2 was removed. For that to be the case, that corresponding projection could not have torn from M1 when R1 was made.

The examination of the corresponding location of M1 clearly showed that there was not a 6mm projection. There was a 2 mm projection and the torn surface had the other half of the bubble.

That was conclusive proof that RX was R1.

I went to Washington to present my results. I was taken to a conference room by my COTR where there were about a dozen individuals who were unknown to me. I was introduced to the group, but they were not introduced to me. (They were under cover agents.) I gave my presentation which included numerous slides of photographs. I answered a few questions and was escorted from the room where the meeting continued. I was told later that the maker of the polyurethane rock was present and after I was gone life was not pleasant for him.



This is THE piece of silicon that helped solve the puzzle.

SECTION 5.4 VITALY YURCHENKO

This is a very strange and bizarre story. It should have been made into a movie.
There are variations of the story as are indicated in a couple of newspaper articles that are included. (See the appendix) Vitaly Yurchenko was front page news for some time in 1985 but I didn't know at the time that MRI would be involved and those papers were not saved.

I will give the story as I understand it. It may or may not be totally correct.

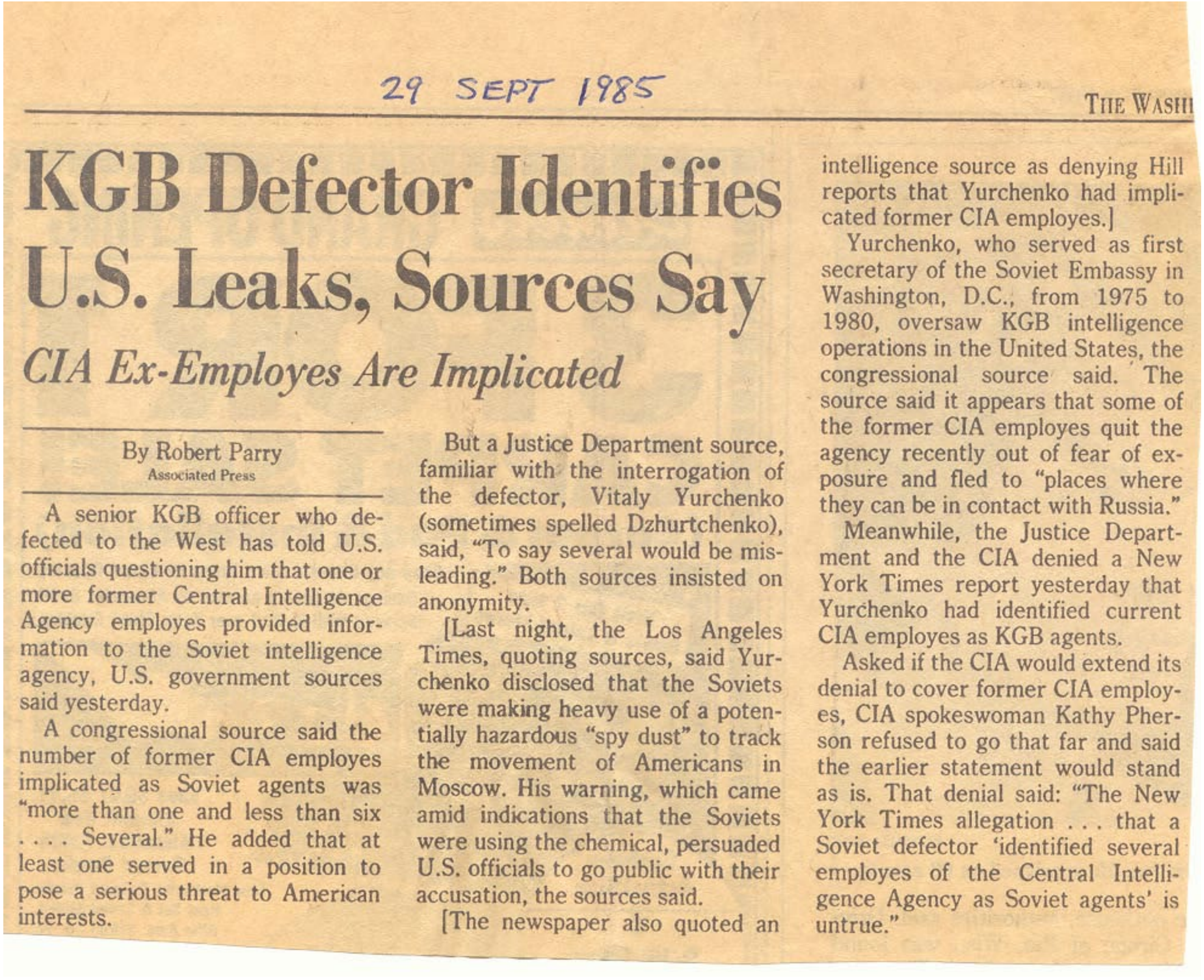
Vitaly Yurchenko was a senior KGB official who defected to the United States. The story goes that he had fallen in love with a Canadian when he was working at the Soviet Embassy in Canada. So he wanted to abandon his wife in the Soviet Union, come to the United States and marry the Canadian woman. He was brought to Washington and was interrogated day after day. One day, he and his interrogator went to a Georgetown restaurant for dinner. Vitaly excused himself, went to the restroom and never came back. He went out a window of the restaurant and apparently went to the Soviet Embassy in Washington and, from there, back to Moscow.

There were newspaper reports that he was executed soon after his return to Moscow. However a short time later a reporter recognized him on a street in Moscow and interviewed him.

Apparently this entire event was well planned by the Soviets to determine how a defector was treated by the United States.

His apartment in Washington was searched and 18 medicinals were found. These were sent to MRI for analysis. Donna Nichols did an excellent job analyzing them. Several of them were legitimate pharmaceutics which could probably also have been used to recover a secret writing message. Many of the chemicals used in secret writing were not unlike many pharmaceuticals.

Copies of two newspaper articles that I have are on the following pages.



29 SEPT 1985

THE WASHI

KGB Defector Identifies U.S. Leaks, Sources Say

CIA Ex-Employes Are Implicated

By Robert Parry
Associated Press

A senior KGB officer who defected to the West has told U.S. officials questioning him that one or more former Central Intelligence Agency employes provided information to the Soviet intelligence agency, U.S. government sources said yesterday.

A congressional source said the number of former CIA employes implicated as Soviet agents was "more than one and less than six . . . . Several." He added that at least one served in a position to pose a serious threat to American interests.

But a Justice Department source, familiar with the interrogation of the defector, Vitaly Yurchenko (sometimes spelled Dzhurtchenko), said, "To say several would be misleading." Both sources insisted on anonymity.

[Last night, the Los Angeles Times, quoting sources, said Yurchenko disclosed that the Soviets were making heavy use of a potentially hazardous "spy dust" to track the movement of Americans in Moscow. His warning, which came amid indications that the Soviets were using the chemical, persuaded U.S. officials to go public with their accusation, the sources said.

[The newspaper also quoted an intelligence source as denying Hill reports that Yurchenko had implicated former CIA employes.]

Yurchenko, who served as first secretary of the Soviet Embassy in Washington, D.C., from 1975 to 1980, oversaw KGB intelligence operations in the United States, the congressional source said. The source said it appears that some of the former CIA employes quit the agency recently out of fear of exposure and fled to "places where they can be in contact with Russia."

Meanwhile, the Justice Department and the CIA denied a New York Times report yesterday that Yurchenko had identified current CIA employes as KGB agents.

Asked if the CIA would extend its denial to cover former CIA employes, CIA spokeswoman Kathy Pherson refused to go that far and said the earlier statement would stand as is. That denial said: "The New York Times allegation . . . that a Soviet defector 'identified several employes of the Central Intelligence Agency as Soviet agents' is untrue."

There are a couple of very interesting items in this article which appeared a little more than a month after the US protested about the use of "spy dust." This article implies that the US went public about "spy dust" because of a Yurchenko statement. Actually, they went public because of the fear that it was a health hazard.

Yurchenko claimed that the KGB had spies within the CIA. In the article above, the last sentence states: "The New York Times allegation . . . that a soviet defector identified several employees of the Central Intelligence Agency as Soviet agents' is untrue." Apparently the only part of the statement that was untrue was the word "several." A short time later Aldrich Ames of the CIA and Robert Hanssen of the FBI were arrested and convicted as being Soviet spies.

Kansas City Times

15 March 1986

Section E

Ex-Soviet defector shown on television

The Associated Press

FRANKFURT, West Germany — A West German television network Friday showed an interview from Moscow with a man it described as Vitaly Yurchenko, the Soviet official who returned home last summer after reportedly defecting to the West.

The film clip lasted less than a minute. The network did not say when the interview was conducted.

The U.S government said Mr. Yurchenko defected in Rome last year and was brought to the United States for questioning. After several months, he ditched his CIA chaperone in a restaurant and went to the Soviet Embassy in Washington.

At a news conference then, Mr. Yurchenko said he had been abducted and drugged by the CIA, and he denied that he had defected.

The U.S. government said he was a high-ranking KGB officer, but Mr. Yurchenko said he was merely in charge of security at Soviet embassies.

The brief film shown by the ARD national network and repeated by the regional Frankfurt television station showed the man identified as Mr. Yurchenko walking on a Moscow street.

ARD reporter Lutz Lehmann said he had run into Mr. Yurchenko by chance.

According to dialogue shown on television, Mr. Lehmann asked the man, "Are you really Vitaly Yurchenko?"

"Do I have to show you my passport?" Mr. Yurchenko replied.

"I believe you," the reporter said.

Mr. Yurchenko said he was "doing the same thing as before — securing Soviet missions abroad."

Some West German newspapers had reported last week that Mr. Yurchenko was shot in a Soviet prison after his return to the Soviet Union.

So ends the strange story of Vitaly Yurchenko. It was a very embarrassing story for the United States.

SECTION 5.5 DELAYED APPEARING MARKING SYSTEMS (DAMS

When one of our CIA agents has recruited a foreign individual, for example a Soviet citizen, to spy for us that individual is referred to as an “asset” rather than a spy. A CIA employee will be referred to as an agent.

From time to time an agent wishes to meet with his or her asset and needs a way to signal that a meeting is desired. One way to signal is to put a mark on a pre-selected location such as a post or building wall. The asset would monitor that location and when a mark appeared he or she would know that a meeting at a pre-selected time (hour) and location was desired. However our agents are often followed when they are out in public and it would be noticed if they placed a visible mark on any public surface.

We were asked to develop a material that could be applied to a surface that was initially invisible but would become visible later. A device was desired that could be used to apply the marking signal on a surface by just walking by with some type of applicator. The application must not be noticed by someone trailing the agent.

I worked on the problem for a considerable time with some success but the results were not very good. Finally, Donna Nichols came up with a series of commercial leuco dyes. These dyes are colorless in the leuco stage but become colored when they are oxidized or otherwise activated. They are also light sensitive and turn from colorless to colored when exposed to light and air.

The various colors of the dyes are shown on the following page.

The client was delighted with these dyes. They were used for many years in various ways. We first bought broad felt-tip markers and modified them by removing and replacing the ink reservoir and the felt tip. The markers were originally five inches long and we cut them to three inches so they could be concealed in the hand. We bought same thick felt and cut new tips from it and stuffed cotton in the barrel of the marker to serve as a reservoir. We made dozens of these markers and they were used successfully.

Then the Client wanted to be able to apply the DAMS marks without actually touching the surface being marked. They wanted to be able to squirt a stream of the dye as they walked by. Again it had to be small enough to conceal in the hand. For this devise we bought cases of Visine (eye drops), emptied the bottles and modified the bottles so they would squirt the liquid as a stream. We then spray painted the bottles an opaque brown to protect the dye from the light and make the bottle less visible in the hand. A spot of the dye could be placed on a surface from a distance of from one to two feet with these modified Visine bottles.

These are the colors of the various dyes used it the Delayed Appearing Marking Systems. All of these were colorless when they were applied to this paper and became colored after a few minutes exposure to light. Color would appear within seconds when exposed to direct sunlight and within several minutes in room light.

Some time later they wanted to apply the marker from a moving automobile. They came up with a spring action BB pistol and they wanted a dye-filled projectile that could be fired from this pistol. The projectile had to be strong enough to withstand the force of firing and fragile enough to break when it hit the target. It also had to be heavy enough to travel a considerable distance. We finally came up with a 15 mm length of very thin glass tubing the diameter of a BB. A BB was cemented to one end of the tubing with epoxy cement, the tube filled with the dye and another BB cemented to the other end. The two BBs gave the tube enough weight to be fired a considerable distance. A problem was that the thinnest glass tubing that we could find was too strong to break reliably went it hit the target. Hydrofluoric acid dissolves glass so we started with the thinnest walled tubing that we could buy and placed the 15 mm lengths of the tubing in hydrofluoric acid until it was etched down to the desired thickness.

Chester B. was our COTR at the time and one night we drove around Kansas City and shot about two dozen of these projectiles at various surfaces. The next day, after daylight converted the colorless mark to a colored mark, we drove around and saw marks on all but two of the targets.

How about that one for fun and games?

SECTION 5.6 A Blouse

The story behind the sample in this section is that I received articles of clothing belonging to a woman that was reported to have been our best agent in Moscow. She was wearing these garments on her way to a rendezvous with an asset (spy) that she had recruited. Her route was designed to foil trackers. She left the U.S. Embassy and went to a department store, changed clothes there, went out a different door of the store, took a bus for some distance into another shopping area, went into a building and out another door, took a streetcar, walked for a considerable distance and finally came to the rendezvous location when all at once she realized that she was surrounded by KGB. She yelled, “KGB!” and her asset apparently escaped but she was arrested and deported.

The question was how did they follow her through this entire, very elaborate path? I received her outer clothing and her purse to see if I could find a tracking agent that had been placed on either. I spent hours, and used many analytical procedures but found nothing.

The blouse was a very distinctive print pattern which I felt was rather novel. A year or so later I was on a plane flying to Washington reading Time Magazine. I turned a page and here was a picture of this woman, Martha Peterson, in that blouse. This was during the Carter Administration and we had just captured a Soviet spy in Washington. Carter was going to make an example of him and, with that threat, the Soviets released this picture of “our spy”.

Spying is fair game, and both sides understood that and they usually simply expel someone that gets caught. Carter was trying to go against this tradition. Neither the CIA nor the KBG wanted to change the game rules and the Soviet Union responded to his threats by releasing the picture of Martha Peterson along with a protest to the Carter Administration. Time picked up the story and printed the picture. I apparently did not save that issue of Time but my daughter, Dhyan, obtained a copy of the article at the Boulder, CO library. The quality of this copy is poor and the pattern of the blouse can not be seen.

So how did they track her? We were unable to find any tracking agents on her clothing. It was concluded that the Soviets knew that she was a very effective agent and they wanted to catch her in a spying operation. They may have had as many as a 1000 agents monitoring every place that she might be going. They all knew who she was and probably surrounded the buildings that she went through. They simply had Moscow saturated with agents to follow her wherever she went.

A photograph of the blouse, and the copy from Time are on the following pages. There are some variations between what I understood happened and Time’s version.

A few years later I met Martha Peterson in a meeting discussing a new problem. I do not know if she knew that I was the one that examined her clothing for tracking agents.

This is the blouse that our CIA agent was wearing when she
was arrested by the KBG and charged as being a spy.
She was wearing this blouse in the photograph
that appeared in Time Magazine

Episodes in a Looking-Glass War

Soviet and U.S. spies expose each other's capers

The short, slick spy thriller had been written to order by Russia's famed de-ective novelist, Julian Semyonov—the Soviet Ian Fleming. Spread over five columns of *Izvestiya* last week, it had some of the suspense but none of the humor of a James Bond story. The tale began as Martha Peterson, 32, a tall, blonde vice consul in the U.S. embassy in Moscow, drove her car to a deserted street in the Soviet capital. Quickly changing from a white dress to a black outfit that would meld into the shadows, she boarded in rapid succession a bus, a streetcar, a subway and a taxi. Satisfied that she was not being tailed, she walked to a bridge over the Moscow River and deftly thrust a stone into a chink in the wall.

Suddenly, the area was alive with agents of SMERSH—the celebrated Soviet counterintelligence service. As the lady yelled "I am a foreigner!" to alert her Russian accomplice, who was lurking near by, the agents examined the stone she had left at the dead drop. Cleverly concealed inside were espionage instructions, miniature cameras, Soviet currency and gold. Most damning were two ampuls of a deadly poison. Peterson was charged with passing them to a Russian contact who allegedly had used the same poison in an earlier CIA plot to kill an innocent man.

Russia's Enger after indictment

There was some truth to *Izvestiya's* fiction. As some Washington officials tacitly conceded last week, the lady vice consul had indeed been involved in some Moscow capers of a type that are more or less routine in the murky world of espionage. She was a CIA agent operating under diplomatic cover in Moscow. Nabbed by Soviet counterintelligence last July, she was photographed with an array of spy gear and quietly allowed to leave the U.S.S.R. under diplomatic immunity. She was reassigned to Washington. Hours after the appearance of the *Izvestiya* story, the State Department instructed the CIA to put Peterson on leave. She immediately dropped out of sight. In answer to queries about the *Izvestiya* charges, a CIA spokesman denied only that Peterson had been involved in murder—a crime that U.S. intelligence agents are prohibited from committing by Gerald Ford's 1976 presidential order.

The *Izvestiya* story was the most dramatic salvo in a Le Carré-like "looking-glass war" that has developed between Russian and American spooks; in a sense, it is the mirror image of the East-West battle of words being conducted on the

Soviet photograph of Peterson being confronted with spy equipment by KGB

Time June 26, 1978

diplomatic front. The Soviet decision to make a sensational public-issue of the Peterson case was apparently prompted by U.S. disclosures four weeks ago that the FBI had captured three Soviet spies in Woodbridge, N.J. One of the Russians, a staff member of the Soviet mission to the U.N., had diplomatic immunity and was swiftly sent home. The other two, United Nations Employees Rudolf Chernyayev and Valdik Enger, were indicted by a grand jury on charges of passing U.S. Navy secrets and jailed with the unusually high bail of $2 million each. FBI leaks to the press ridiculed the agents as ham-fisted operatives who had been caught with an orange-juice carton full of phony antisubmarine warfare documents that had been prepared for them by the feds.

Though the Justice Department had a strong case against the Russians, the decision to prosecute them (rather than hustle them out of the country) was made by the White House. "The Soviets were agitated, really ripped off," one State Department official said. "They accused us of changing the rules of the game." Indeed, the U.S. had deliberately violated an informal understanding between Soviet and American intelligence services that each other's spies will be discreetly ferreted out of the country when they are caught. Soviet Foreign Minister Andrei Gromyko complained angrily to Secretary of State Cyrus Vance about the indictment of the spies, threatening that "two can play this game."

In addition to brandishing Peterson's transgressions, the Soviets have coolly demanded indemnification for damage done to their equipment by American security officers who had discovered KGB devices bugging the U.S. embassy in Moscow. Furious about the U.S. discovery of the eavesdropping equipment and subsequent news stories about it, the Soviets countered by declaring that the Americans had actually been using the apparatus to spy on the Russians.

The spy war intensified last week when the Soviets arrested F. Jay Crawford, 37, a Moscow representative of the International Harvester Co., and accused him of selling foreign currency to Soviet citizens at speculative prices—a charge that could cost him eight years in a forced-labor camp plus a five-year term of exile in the U.S.S.R. Crawford, a genial Alabaman, was driving to a cocktail party with his fiancée, U.S. Embassy Secretary Virginia Olbrish, when policemen accosted him at a traffic light and dragged him from his car. When his fiancée resisted the cops, she was bruised in the scuffle. Late last week, U.S. Consul Clifford Gross was allowed to visit Crawford at Moscow's Lefortovo Prison. Crawford appeared to be in good health but was distraught. U.S. officials insist that the Soviet allegations are trumped up. "There is no indication that he was into anything that wasn't completely aboveboard," said a senior State Department official.

Crawford's arrest worried American businessmen in Moscow. Many fear that another representative of a U.S. firm will be arrested by the KGB so that they can have two Americans on hand to trade for the two Soviet spies held in the U.S. Washington has been adamant in advance about rejecting such a trade. Meanwhile, American firms doing business with the U.S.S.R. were reassessing the pros and cons of U.S.-Soviet trade.

Many were alarmed by the fact that the Russians picked on International Harvester, which has sold the Soviets more than $300 million worth of much needed heavy construction equipment and gas turbines. Moreover, Harvester's board chairman, Brooks McCormick, has been one of the U.S.'s most active boosters of trade between the two countries. Declared a White House aide: "Crawford's arrest is not the kind of move designed to inspire confidence in the American business community." ■

SECTION 5.7 BORAX ($Na_2B_4O_7$-$10H_2O$)

Occasionally the client needed the results of the analysis of a sample very quickly. They would receive a sample, take it to the airport right away, and send it to Kansas City on "the next flight out." I, or someone for MRI, would go to the Kansas City airport and we would have the sample only a few hours after it was sent. I made a game of it to see how quickly I could get an answer as to what the sample was. Often I could call in a tentative result the next day and confirm it on the third day.

The story of one particular sample is interesting. I went to the airport, got the sample and returned to MRI about 12:30 noon. The sample was a white granular material. As usual I made some simple tests to determine the general classification of the material. I placed a small amount of the material on the tip of a spatula and he heated it in a flame. By observing, if it melted or burned and, if it burned, whether it produced a colored flame or smoke or an odor, and what if any residue was left, conclusions could be made as to whether it was organic or inorganic as well as an indication of some of the atoms present.

For this sample, the material melted almost as soon as I put the spatula in the flame. It then boiled and left a white residue. I concluded that it was a highly hydrated inorganic compound. I next placed a bit of the sample on the tip of a platinum wire and put it in a flame. It produced a green flame. A green flame is most likely produced by either copper or boron, but hydrated copper compounds are blue. This compound was white. The most likely highly hydrated boron compound would be borax. So in less than 10 minutes I had a tentative conclusion that the sample was borax. We had some borax in the lab. I made a melting point comparison of the sample and borax, and both melted at 75° C, which was the literature value for borax. I then placed small amounts of the sample and borax in weighed small test tubes and weighed them. I then heated both of them to drive off the water of hydration. I cooled the test tubes and again weighed them. Both the sample and borax lost 47% of the original weight, which is the amount expected for borax.

By now, less that an hour from starting the analysis, I was 99+% certain that the sample was borax.

I called the client to report my results. I got a rather strange and unusual response but he thanked me for the result and the quick turn-around.

Many months later, when my COTR was moving on to a new position, he told me the following story: When they send me a sample by next flight out, the group in the office of about ten individuals each chipped in a dollar to form an office pool to see who came closest in guessing when Atkinson would call in the results. That day they were in the middle of making up the pool when I called.

SECTION 5.8 LETTER BOMBS

During the mid-1970s there were numerous letter bombs sent to various people. They were common enough that any mail that was received that appeared like it might contain explosives was carefully examined. A sheet of plastic explosive that was perhaps 3 x 6 inches and 1/8th inch or less thick could easily fit in an envelop and would be enough to blow a hand off when the envelop was opened. I never saw any of the firing mechanisms but I was told they generally consisted of small tube with a spring loaded pin. The pin was retained by a small wire going through the tube. When the letter was opened, this wire would be pulled out of the tube and the spring would send the pin forward into a small amount very sensitive explosive primer that, when set off, would cause the plastic explosive to explode.

I received many samples of the explosives to analyze. These samples were from letter bombs that were detected and had not exploded. They were generally mixtures of two common explosives, PETN and RDX, along with mineral oil to make them plastic. They had the consistency of putty. Some samples contained carbon black and were black; others contained a yellow dye and were pumpkin yellow.

The objective of our analyses was to determine the composition of the explosives so that it could be determined which letter bombs were made from a single batch of explosive. In addition to the obvious color variations there were minor variations in the proportions of the PETN, RDX and mineral oil. As I remember, we probably received and analyzed about twenty samples and they came from four different batches.

SECTION 5.9 SUICIDE CAPSULES

Many terrorists, when captured, were found to be carrying some kind of material which enabled them to commit suicide. We received several samples of these materials to analyze. They ranged from 'sophisticated and definitely lethal' to 'crude' and in some samples 'non-toxic.' The most sophisticated one consisted of a glass ampoule containing about two milliliters of liquid Hydrogen Cyanide (HCN) which had been placed in a short length of latex rubber tubing with a small disc of stainless steel screen at each end. Apparently the intended use was to place the container in the mouth and bite it. This would break the thin walled ampoule and release the Hydrogen Cyanide. Apparently the screen was to keep the broken glass in the tube so the individual wouldn't get broken glass in his or her mouth. Without the screen the mouth might be cut a few minutes before death. I thought that was very considerate.

Less sophisticated, but just as lethal, were capsules of either sodium- or potassium cyanide. These would be lethal but probably not pleasant to take since they are very caustic and probably would produce considerable mouth and stomach discomfort before death.

We received two or three capsules that were filled with potassium ferrocyanide. It sounds pretty toxic if you don't know about the compound but actually the cyanide is so tightly bound to the iron atom (ferro-) that is less toxic than common table salt. So anyone that took one of these capsules, in an attempt to kill themselves would certainly be surprised and would continue to live.

SECTION 5.10 DETERIORATED RUBBER PRODUCTS

For this task we received several rubber-containing items with the rubber severely damaged. We were told that the items came from an important government office and sabotage was suspected. We were asked to determine what had caused the damage. The items included an electrical power cord, a coiled insulated wire, some rubber bands and a few other rubber items. The power cord apparently had been looped back and forth and tied in that position to shorten the cord. In this position there were straight sections of the cord and sections where the cord was in a tight bend. The straight sections appeared to be undamaged, the bends were badly damaged.

Back in the early 1950s, MRI had a project with the city of Los Angeles related to the cause of smog. Ozone was determined to be a major contributor to smog production. That was back before analytical instrumentation was very sophisticated and a very simple, but effective "ozonometer" was in use. It consisted of an ordinary rubber band stretched over two pulleys. One pulley was fixed and the other allowed to turn. One leg of the rubber band was exposed to the atmosphere and the other shielded from the atmosphere. A pointer was attached to the moveable pulley and a scale fixed behind the pointer.

When this ozonometer was exposed to the atmosphere containing ozone, the ozone would react with the exposed leg of the rubber band and cause deterioration of the rubber and weaken it. It became weaker than the protected leg of the rubber band and the resulting difference in the tension of the two legs of the rubber band would turn the pulley with the pointer. The readings of this devise were surprisingly accurate in indicating the atmospheric ozone concentration.

The point of this story about the ozonometer is that I was familiar with the damage that ozone would do to rubber. The damage of the items received was typical of ozone damage.

But why would an important government office have a high level of oxone? The answer to that question was fairly obvious. It was an office with many copy machines and poor ventilation. A check in the literature provided information concerning both the production of ozone by copy machines and the damage that ozone will do to rubber. The most damage is done when the rubber is stretched, as was the case for the power cord looped back and forth.

We bent undamaged sections of the power cord, and placed them along with stretched rubber bands, in a chamber with ozone and produced the same type of damage seen on the submitted samples.

We took numerous photographs and submitted a report.

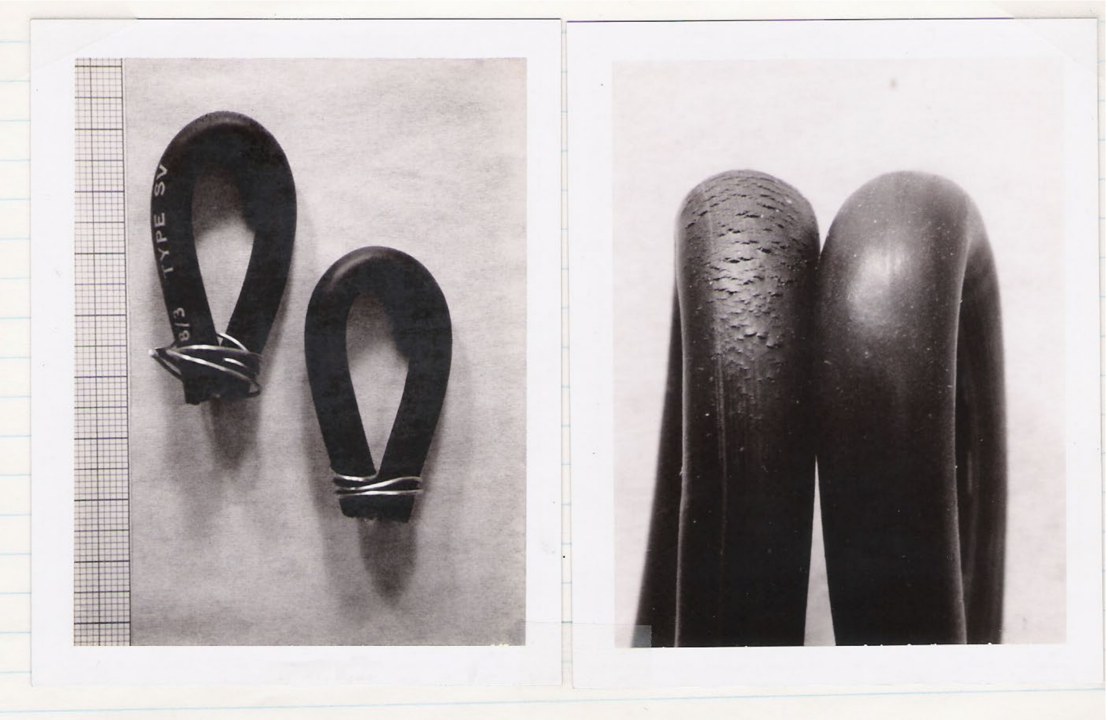
We latter learned the items had come from the secure government hideaway for use by the President and other high level government officials during times of threatened nuclear attack.

That office complex had lots of copy machines and very little ventilation.

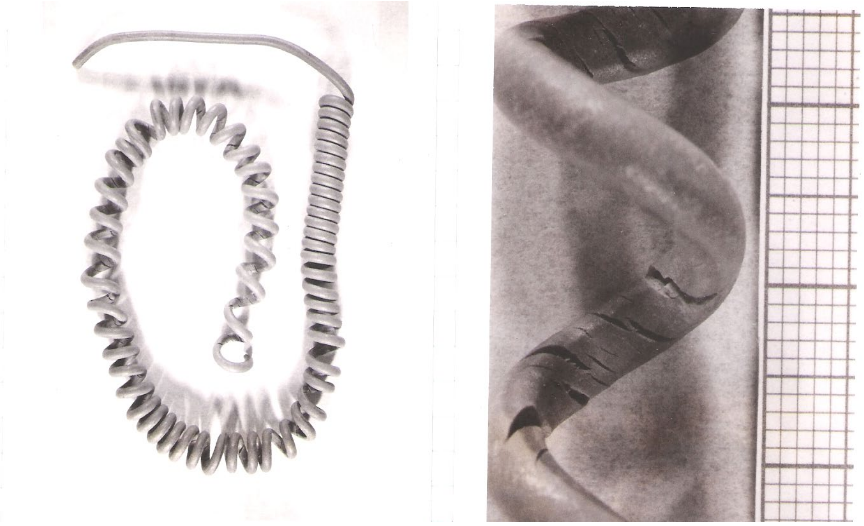
Pictures of a few of the rubber samples follow:

Eight rubber items received for analysis.

The power cord had been looped back and forth to shorten it. The damage had occurred at the turn of each loop, area of maximum tension. On the right, is a picture of damage that occurred at the turn of one loop.



These pictures show portions of the power chord that were originally undamaged. We damaged the one on the left in the second picture by bending it the same way the looped chord had been and then exposing it to ozone. The other one, not exposed to ozone, served as a “control.”



On the left is a piece of telephone chord and its damage is shown in an enlarged view on the right.

SECTION 5.11 NEW CHEMICAL WARFARE AGENT

By about 1980, none of the individuals that had worked on the chemical warfare agents were still at MRI but we were still expected to be able to receive and analyze chemical warfare (CW) related samples. We received an urgent message that a new Soviet Union chemical warfare agent had been obtained and we were to receive it to determine its composition.

We knew the characteristics, dangers and treatments for known CW agents but obviously nothing was known about a new agent. Dr. Jim Downs was given the lead for this task. He wrote a detailed protocol for handling and analyzing this sample. It was sent to the client for review and approval. We spent about a month of work preparing a laboratory with the needed equipment. We assembled a team of Jim, me and one other technical staff from MRI plus an MRI photographer to document every step of the activity. A doctor at a nearby hospital Emergency Room was briefed of the situation so he would know what had happened in case there was an accident and one of us was exposed to the new CW agent. We went through several trial runs to be sure everything would go right. The project COTR, Ed H. came for a final trial run.

The client took this sample very seriously. The sample was transferred from Washington DC to Kansas City in a convoy of five vehicles. The sample was in a van with two lead cars and two following cars.

The sample was in a heavy stainless steel container about 12 inches in diameter and 18 inches high with a lid bolted to a flange at the top of the container. The container was packed in a wooden crate filled with vermiculite. Apparently, after the sample had been obtained the special container and crate were fabricated and sent overseas for the shipment to the U.S.

The crate was brought to our lab and stored until the following day when Ed and a translator arrived. It had been indicated that printing in Russian was on the sample.

We wore gas masks as we opened the crate and transferred the container to a glove box and the glove box was sealed. The glove box was about 36 x 24 x 24 inches with a sloped window in the front. Two arm-length gloves were attached to openings in the front of the box. Tools had been placed in the box to open the container. There was an opening in the box to allow a controlled amount of air to enter the box and air was withdrawn from the box to maintain a slight negative pressure in the box. The air that was withdrawn passed through a chamber with two mice (like the canary in a mine) and then to a bubbler of potassium chromate in concentrated sulfuric acid to destroy any toxic gas that might be present.

Jim worked in the glove box. I had a tape recorder to record the activities. Ed and the translator observed the action and the photographer took numerous photographs of the activities.

The lid was removed from the container and a cardboard box removed. It was opened to reveal four different colored plastic boxes about 8 x 4 x 1 inches. There were labels on the boxes with considerable information. Jim held a box up to the window of the glove box for the interpreter to read. His Russian wasn't very good and he really struggled reading it, he spent about 15 minutes trying to make it out. However, it soon became fairly evident that the sample was not a new chemical warfare agent. It apparently was a new field kit for detecting chemical warfare agents. Then, Jim turned the plastic container over, and there on the bottom of the container was the same information in Czech, the translator's native language.

The different colored boxes were for material to detect different types of agents. I don't remember what happened to the mice, but they did not die after being exposed to what was <u>not</u> a Soviet chemical warfare agent.

We went on to determine the various chemicals in the kits. That was a considerable effort.

One wonders how this new field test kit could have been obtained and thought to be a new chemical warfare agent.

SECTION 5.12 EMBASSY WINDOW

This sample was a 300 pound window from a U.S. Embassy. It was said to be sledge hammer proof and with its construction I am sure that it would be. It consisted of three layers of glass and Lexan. The outer layer was a ¼ inch pane of safety glass like a car windshield glass which is a sandwich of a tough plastic between two eighth inch sheets of glass. Next there was a ¼ inch air space followed by a ¾ inch thick plate of Lexan, then another ¼ inch of air followed by a ¼ inch of tempered glass for a total thickness of 1.75 inches. The window components were held together with a ¾ inch band of a very tough band of black silicone around the edges.

```
 __________________________________________
|///////////////////////////////////////////     Safety glass
|XXXXXX____________________________________     X = Silicone seal, then air
|\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\
|//////////////////////////////////////////      Lexan
|\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\
|XXXXXX____________________________________     X = Silicone seal, then air
|///////////////////////////////////////////     Tempered glass, inner surface fogged
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The problem was that this, and other windows in the Embassy, had become fogged with some coating on the inside surface of the tempered glass. We were asked to determine what the material was that caused the fogging, and how did it get there. These were very expensive windows and someone was going be held responsible and pay for replacement.

The glass part of the window was 58.75 x 17.4 inches with about a three inch aluminum frame around the glass. The glass extended into a ¾ inch deep channel in the aluminum frame. The frame was bolted together and I first removed the frame so I could work with the window itself.

The material in question had some interesting patterns which in places appeared almost like frost on a window in a cold winter day. In other places it had streaks and in one place there were what almost appeared to be a flock of birds or insects in flight. Before proceeding with separating the panels, I spent considerable time photographing the deposits. It was rather difficult to get the illumination right to get photographs, but the time I spent was well worth while and the photographs were important in concluding the cause of the fogging. I also obtained a photograph that I had in my office for my remaining time at MRI that was quite a conversation piece. “What is it?” See first picture at the end of this section.

I spent hours carefully cutting through and removing the silicone band around the edge of the window. When the tempered glass pane was finally cut free and lifted there was an odor of acetic acid. A sample of the deposit on the surface of the glass was removed and determined to be sodium acetate.

So how did the sodium acetate get on the glass, and why was it only on the surface of the tempered glass and not on the Lexan or safety glass surfaces? My conclusions:

1. The silicone that was used around the edges of the window as a seal and adhesive was of the type that released acetic acid as it cures. Most silicone formulations do release acetic acid but there are other silicone formulations that do not release acetic acid. Years earlier we had received a microwave antenna that was badly corroded. It turned out that the assembly had been sealed with acetic acid-releasing silicone adhesive and the acetic acid had caused corrosion. I learned then that there were other silicone adhesives specifically designed for electronics that did not release acetic acid.

2. Photographs of the deposit clearly showed circular patterns in places that indicated that the glass surface had been buffed in a circular motion leaving streaks of a residual sodium containing material. In other places the pattern of sodium acetate indicated something like brush strokes or a wipe with a cloth.

My conclusion was that the tempered glass, but not the safety glass, had been cleaned or buffed with some sodium containing material, possibly sodium bicarbonate, (baking soda) and some residual material reacted with the acetic acid vapor to form the sodium acetate.

I have no information about who was held responsible for the fogging. It appeared to be a combination of leaving some residual sodium containing cleaning agent on the surface of the glass and the use of an acetic acid releasing silicone.

Pictures are on the following pages.

Approximately one square inch of the sodium acetate fogging on an inner surface of the Embassy window. Note that all of the lines originate at the “eye” of these insect-like images and all are curved. I concluded that the “eyes” were deposits of some sodium compound of a cleaning material which was streaked by a circular buffing motion.

This is the picture that I had on my wall at MRI.

A slightly larger area of the images shown on the previous page. Note the brush or cloth marks on the upper left of the top view.

SECTION 5.13 LAMP COATING

Our agents in places like Moscow used various electronic devices containing batteries that needed to be recharged. As part of their job in recruiting assets or just getting information agents did a considerable amount of entertaining in their apartments. They didn't want the devices to be seen by their guests so ginger jar lamps were modified to contain recharging equipment inside of the lamps.

In order to aid in obtaining information it helps to have parties with plenty of alcohol. But at parties with plenty of alcohol a lamp might be knocked over and the ceramic ginger jar broken. If that happened the electronic devices would be exposed. That would be very undesirable.

I was asked to find a material to coat the inside of the ginger jar so it would not shatter if it were knocked off of the table and broken.

I tried several materials and settled on polyurethane. Polyurethane is a very tough, strong material something like hard rubber. It is formed by adding a catalyst to urethane to get it to polymerize. The material that I used was a commercial urethane with a viscosity about that of Karo Syrup. The viscosity would gradually increase after it was mixed with the polymerizing catalyst, would no longer flow in about ten minutes, and was completely cured in about 30 minutes.

Rather than buying lamps to coat and break, I choose much cheaper, ordinary, eight inch diameter, red clay flower pots. By trial and error the right amount of urethane could be mixed with the catalyst, allowed to stand for a short time to slightly increase the viscosity and then be poured into the pot. The pot was then rotated to evenly coat the inside with a one, eighth-inch thick coating.

A very strong bond was formed between the polyurethane and the pot. When the pot was dropped it would crack but would maintain its shape. Repeated blows with a hammer shattered the pot and some of the clay came off but the pot shape was retained.

Several of the pots were coated and sent to the client. Ed H.,our COTR at the time, said that he took one of the pots to the office of the individual who had requested this item. This man was head of a group and was at least two levels of management above Ed. Ed walked in the office and said "look at this" and threw the pot on the floor. In shock, this individual said something like "What in the hell are you doing?" Then he realized what he had just seen and he was delighted.

That was the end of our involvement in lamp coating but obviously lamps were successfully coated and put in use and MRI was given credit. I received compliments about the lamps from various individuals for several years.

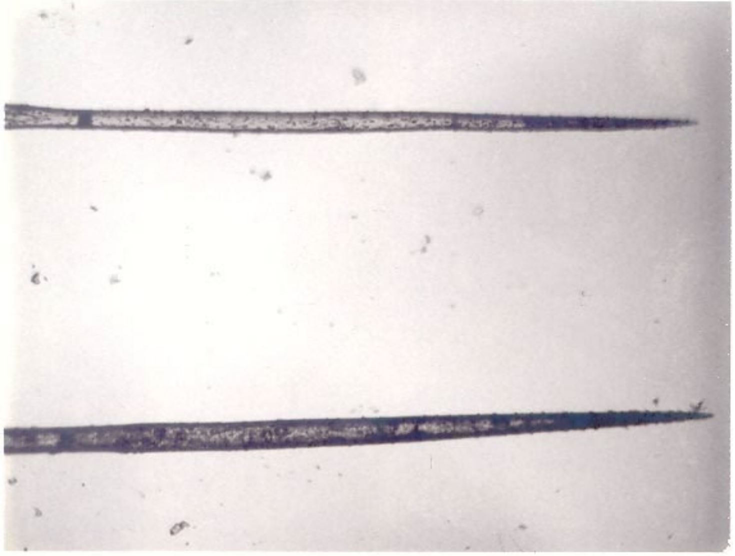
SECTION 5.14 IRRITANT POWDER

This was as bizarre a sample as I ever received. It was reported to be an “Irritant Powder” that could be dispersed in a room to disrupt a meeting. We received the sample in an ordinary rubber squeeze bulb. It was stated that, in use, the squeeze bulb would be shaken and then squeezed to expel a puff of the powder.

The bulb was cut open and about 3 grams of dark brown material was obtained. Microscopic examination of the material indicated that it consisted of needles about 2 mm long. Part of the sample was extracted with solvents and the extracts examined by thin layer chromatography. There were bright red fluorescent components detected that were determined to be chlorophyll.

Chlorophyll absorbs red light and reflects green, that is why plants appear green, when exposed with ultra violet light it fluoresces red. This indicated that the needles were from a plant, undoubtedly a cactus.

It so happened that I had some prickly pears growing in my back yard and I collected some needles from the prickly pear for comparison. They were very similar as seen in this photograph.



The needle at the top is from the squeeze bulb,
The needle at the bottom is from a prickly pear.
The needles shown in this photograph were
slightly longer than 1 mm.

It seems very likely that a few puffs of these needles into the air would disrupt a meeting.

SECTION 5.15 JOE K.

Over the years I worked directly or indirectly with 25 to 30 different employees of the CIA. They ranged from competent to incompetent, from pleasant to unpleasant, and all combinations in-between.

Joe K. was not in the Branch that I had been involved with and I had not done any work for him. However I had been in several meeting with him and other individuals from MRI had done some work for him. I had heard several stories about him, none were good. One of my COTRs referred to him as Crazy Joe.

One day I received a telephone call from Joe requesting a proposal for the microscopic examination of an environmental sample. He was very vague about the sample, but the work was urgent and he wanted the proposal soon. A few days later I had finished the proposal and called him to tell him that it was ready to be mailed and asked him for mailing instructions. This was on a Thursday. He said mailing would take too long. He needed the proposal tomorrow. He told me to hand carry it to Washington and deliver it to him on Friday.

Friday morning I arrived at his office and asked the receptionist for Joe. She informed me that Joe's mother was in Washington for the weekend and Joe had taken the day off. So there I was in Washington and I couldn't even talk to him about the sample.

I returned home and a couple of weeks later the sample arrived. It was half of a filter from a window air conditioner. Joe's request was to examine the material on the filter to determine if there was anything unusual present. He would not give me any information concerning what I should be looking for. The filter was covered with about 1/8 inch of filth. It contained soil dust, insects, insect parts, seeds, animal hair, and all kinds of plant debris. The only thing that I found that might be considered unusual and that I couldn't specifically identify were a few fibers or rods a few millimeters long. I isolated some of these and photographed them. I didn't know if these fibers were what Joe was looking for but I found nothing else that seemed to be unusual.

I next received a message from Joe's secretary that I was to attend a meeting at Lawrence-Livermore National Laboratory east of San Francisco to present my findings. So I flew to San Francisco, rented a car, and drove to Livermore. I went through the security checks to enter the complex and found the site of the meeting. The receptionist escorted me to outside of the meeting room and she went in. She came back out and told me to have a seat. I would be called when they were ready for me. After about 15 minutes another individual showed up. We talked and it turned out that he had received the other half of the air conditioner filter. He also had been told nothing more than to look for something unusual. We compared notes and he too had found the same fibers that I had found. I felt a lot better.

Eventually we were asked to come into the meeting room. There were about ten people there. I knew three or four of them. We were asked what we found. We gave our answer, showed the pictures and then were escorted from the room. We were in the meeting no more than five or ten minutes and were given no indication concerning what we had found or why it was important.

I later learned, not from Joe, that the sample had come from Kabul, Afghanistan at the time that the Soviets were occupying Afghanistan. What we had found was chaff expelled from Soviet aircraft during take-off used to disrupt ground-to-aircraft missile radar.

This is the photograph that I presented at the meeting at Lawrence-Livermore

SECTION 5.16 MISCELLANEOUS

This is just a very brief mention of some of the other hundreds of samples that were received.

- Part of a bouillon cube, it was soon fairly obvious that it was bouillon, but difficult to prove.
- The determination of the vapor pressure of heroin at various temperatures.
- The analyses of solutions in a bomb timing device that could be timed from a few hours to several days by changing the ratio of two components of the solution. The solution reacted with a wire connected to a spring-loaded firing pin. When the wire was corroded-to-failure the firing pin was released to detonate the bomb.
- Numerous samples of various drugs both legal and illegal.
- Drug samples from two heads of state to determine their medical condition.
- A sample of a potentially illegal substance taken by a US Senator from his daughter's room to find out what she was on.
- Several wine samples from our agents who got sick after drinking wine given as a gift, which caused them to think they had been poisoned. We never found a poisoned wine. They probably just drank too much.
- A sheet of paper to determine if it had been wet with sea water. It had.
- Some white powder found in a high level official's mail box that was determined to be potassium bicarbonate. This is not a poisonous substance but very unusual to find in a mailbox.
- The formulation of olive drab paint to exactly match paint of a Soviet tank

Appendix

The Client sent me dozens of newspaper clippings about NPPD that appeared following the White House News Briefing of August 21, 1985. I have included a few of them here along with some later articles up to February 1986 when Karen Hammerstrom of the EPA issued her report on the results of the Moscow sampling trip. The items included along with comments are:

1. **Thursday, August 22, 1985, The Kansas City Times**
   U.S. protests Soviet 'spy dust'
    a. This was the first article that I saw
2. **Thursday, August 22, 1985, The Washington Post (three pages)**
   At Moscow Embassy, Continuous Shadow War
   Substance Unfamiliar to Chemists
   State Department Statement
3. **Cartoons Related to "Spy Dust"**
4. **September 2, 1985, Newsweek**
   The Soviet Spy-Dust Scare
5. **September 16, 1985**
   Jack Anderson Article
    a. The article includes "Nosenko didn't know the exact chemical used, but he reported that Mr. Langelle's maid had dusted the diplomat's shoes with it.
    b. The KBG, using a sniffer dog, tracked Mr. Langelle to the mailbox - -"
    c. This was probably the tall oil that had been placed on embassy doormats that we identified in 1972.
6. **Date and source not recorded, probably August of September 1985**
   "Spy Dust" Irritates Diplomats
    a. I found this article interesting in that The Aldrich Chemical Company "had no information on NPPD". Ten years earlier they had rejected my request that they synthesize some of it.
7. **Monday, September 16, 1985, The Washington Post, Jack Anderson and Dale Atta**
   Soviets Sprinkle 'Spy Dust" for years
    a. This article includes another mention of the use of a sniffer dog and probably tall oil as the tracking agent.
8. **February 24, 1986, Chemical and Engineering News**
   Tests show spy dust not a health hazard
9. **Probably February 1986, Science**
   After the Spydust Settled
    a. This article was after, and about the EPA's report on Moscow samples.

The Kansas City Times

Thursday, August 22, 1985

U.S. protests Soviet 'spy dust'

Potential cancer-causing powder used to track Americans' movements in Moscow

The New York Times

WASHINGTON — The United States accused the Soviet Union on Wednesday of using a mysterious powdery substance as an aid in tracking the movements of Americans and possibly other foreigners in Moscow.

"We have protested the practice in the strongest terms and demanded that it be terminated immediately," a State Department spokesman said.

Officials said the United States was more concerned about a possible health risk from the substance than about espionage questions.

The State Department spokesman, Charles Redman, raised the possibility that the chemical might have the potential to cause cancer. He said one chemical agent, apparently developed by the Soviets for tracking people, was a mutagen known as nitrophenylpentadienal. A mutagen is a substance known to cause genetic change.

The chemical is so little known that it does not appear in standard chemical reference books, and interviews with chemists disclosed that nearly everything known about the substance in the United States is secret.

In Moscow, 500 American residents were informed about the situation in briefings at the residence of the United States ambassador. They were told that the yellowish powder was being used by the KGB, the Soviet internal security agency, to keep track of the movements of foreigners.

The reaction was divided between skepticism, tinged with humor, and apprehension that the substance might cause harm to children or pregnant women.

See U.S., A-9, Col. 1

Continued from Page A-1

The substance instantly was dubbed "spy dust" by some members of the American press corps in Moscow who took a lighthearted view of the disclosure.

But others were worried. Jane Thatcher, wife of correspondent Gary Thatcher of the *Christian Science Monitor*, said she was concerned about the possible effect on the health of her 2-year-old son, Jason.

In Washington, Democratic Sen. Dave Durenberger of Minnesota, chairman of the Intelligence Committee, said the Soviets tracked people who came in contact with the powder either by lifting samples off their clothes or by illuminating them with ultraviolet light, making them glow.

He said the United States had known of the powder since 1976 but did not know of its potential health dangers until recently.

Statements by officials left many questions unanswered, including where or how the use of the substance had been discovered, why an alarm was being raised now and why the use of tracking agents had not been examined before.

Officials said it was "entirely possible" that President Reagan would raise the issue when he met with Mikhail S. Gorbachev in November.

In Santa Barbara, Calif., White House spokesman Larry Speakes said, "We will certainly discuss, in various forums, the serious dangers to the relationship caused by the actions of the Soviet military and security services, which seem to act

the political authorities."

He said Mr. Reagan was told of the situation Monday and had directed the American response.

The accusation is the latest in a series of pointed gestures toward the Soviet Union this week. On Monday, the White House said the Soviet Union was hampering arms control talks, and the next day, the United States announced that it would proceed with a test of an anti-satellite weapon against an object in space.

In the State Department, Mr. Redman, discussing the possibility that the chemical tracking agent could cause cancer, said "mutagens can be, but are not always, carcinogens in human beings."

The United States has no proof that the substance was absorbed into the blood. Extensive testing will be necessary to determine whether it poses a cancer threat or any other kind of health hazard, he said, adding that no one had fallen ill as a result of exposure, which he described as very low.

He said a task force under the National Institutes of Health and the Environmental Protection Agency would go to Moscow to conduct an investigation.

An administration official who declined to be identified said the substance was thought to have been deposited in places that embassy personnel frequently touch — "on your car seat, steering wheel, door knobs, literally anywhere."

"The embassy employee comes in contact with it," he said. "It is a

not disappear from him wherever he happens to have touched it. He then, in theory, transfers this substance to anything he may come in contact with."

The official would not say how the Soviets traced the deposits left by the substances.

"We have known of the general use or existence of such sorts of chemical tracking agencies since the 1970s," he said. "Their use, however, was very sporadic, infrequent, to the best that we could determine. In fact, we believed that the Soviets had terminated using such agents, even in these limited amounts that we had detected, in 1982. We simply did not detect any use between 1982 and the resurgence of more widespread appearances in the spring and summer of this year."

Last year, a laboratory test known as the Ames Test found that the substance might be harmful, and this year the United States found evidence that its use was more widespread than previously thought. He said the United States regularly "runs all kinds of tests for various kinds of activities which may be mounted against us."

The official said the United States did not use tracking substances to monitor the movements of foreigners.

He also minimized the possibility of health risks from the chemical, saying it was not "a life and death situation."

The Washington Post

60 • © 1985, The Washington Post Company THURSDAY, AUGUST 22, 1985 P Higher From D

At Moscow Embassy, Continuous Shadow War

Charges Are Latest in 30-Year Spy Saga

By John M. Goshko
Washington Post Staff Writer

Charges that the Soviet Union used a chemical dust to keep track of U.S. diplomats mark the latest chapter in a saga of espionage activities directed against the U.S. Embassy in Moscow for more than 30 years.

It generally is assumed in diplomatic circles that U.S. intelligence agencies routinely use various tricks of their trade to keep a similar eye on what goes on inside the Soviet Embassy here.

While electronic bugging and surveillance are an accepted part of the superpower rivalry, it is the Soviets, with their willingness to use more brazen techniques, who periodically are caught doing things that make headlines.

Perhaps the best-remembered incident occurred in 1960, when the United States was trying to deflect attention from the fact that its U2 spy plane had been downed in the Soviet Union.

Henry Cabot Lodge, then U.S. ambassador to the United Nations, dramatically displayed to a session of the United Nations a large, carved wooden replica of the Great Seal of the United States that the Soviets had presented as a gift in 1952 and had hung in the U.S. Embassy in Moscow for six years.

Lodge said the plaque was a listening device, with a hollow inside chamber that produced resonance and permitted the Soviets to overhear conversations in the room by bouncing radio waves off the plaque.

Lodge charged that more than 100 similar devices had been uncovered in U.S. missions and residences in the Soviet Union and East European communist nations.

In the ensuing years, as advances were made in eavesdropping techniques, reports of Soviet espionage efforts became increasingly exotic.

In 1969, a security officer at the U.S. Embassy in Bucharest detected the voice of a senior diplomat talking business. It turned out that the diplomat had sent his shoes to be repaired a few days earlier and they had come back with a tiny but powerful transmitter in a new heel.

During the early 1960s, U.S. officials discovered that the Soviets were bombarding the Moscow embassy with microwaves to tap telephones and interfere with telephone and cable traffic. The bombardment continued through much of the 1970s and finally forced a strong public U.S. protest in 1978 about a possible health hazard to embassy personnel.

Disclosure of the microwave assault caused such a stir that the State Department asked the Johns Hopkins University School of Public Health to make a two-year study of more than 2,000 Americans who had worked at the embassy. It concluded that they suffered no apparent ill effects.

Despite the protests, there have been U.S. allegations that the Soviets periodically resort to using microwaves and more advanced laser techniques to hear what goes on behind the embassy's walls.

At one point, Washington formally accused the Soviets of installing a "secret listening post" in the chimney of the chancery building and linking it to a neighboring Soviet apartment building by a tunnel through embassy property.

As recently as last March, U.S. officials said that Soviet agents had installed tiny sensing devices in several of the embassy's typewriters. The devices reportedly could pick up the contents of documents typed on machines and transmit them to antennas in the embassy walls.

In 1978, Soviet officials claimed to have caught an employe of the embassy consular section, Martha Peterson, in an apparent attempt to poison an unnamed Soviet official. Because she had diplomatic immunity, the United States was able to rush her out of the country and she dropped out of sight, with U.S. officials refusing to discuss the circumstances of the incident.

The espionage allegations have extended to this country, where U.S. officials frequently have charged the Soviet Embassy here and its consulate general in San Francisco with spy activities. Sometimes the FBI surveillance produces such dramatic results as the recent disclosure of the Walker family spy ring, charged with selling Navy secrets to the Soviets.

Sometimes the results are bizarre. When an aide to the Soviet naval attache delivered a package to the commander of the Washington Navy Yard last Christmas, suspicious explosives experts detonated it—destroying two bottles of vodka intended as a present for the admiral.

Staff writer John Mintz contributed to this report.

Washington Post – August 22, 1985 – Front Page Article

Soviets Said To Imperil Diplomats

'Potentially Harmful' Tracking Chemical Used, U.S. Charges

By Don Oberdorfer
Washington Post Staff Writer

The United States charged yesterday that Soviet secret police have employed "potentially harmful" chemical dust to track the movements and contacts of U.S. diplomats in Moscow and Leningrad, and demanded that the practice be stopped.

The surprise disclosure, in White House and State Department news briefings and U.S. Embassy sessions in Moscow for diplomats, their families and other Americans who might have been exposed, brought to the fore a new, bizarre and emotion-laden problem in U.S.-Soviet relations just three months before the scheduled summit meeting of President Reagan and Soviet leader Mikhail Gorbachev.

Official sources said the chemical dust—said to be odorless, colorless and with no visible residue when properly sprayed—was placed on steering wheels of diplomatic cars and other places where U.S. attaches would come into contact with it. The diplomats unknowingly would then leave tiny amounts of the long-lasting chemical on the hands, clothing or possessions of Soviet citizens with whom they met—telltale traces that could be identified by the KGB, or secret police.

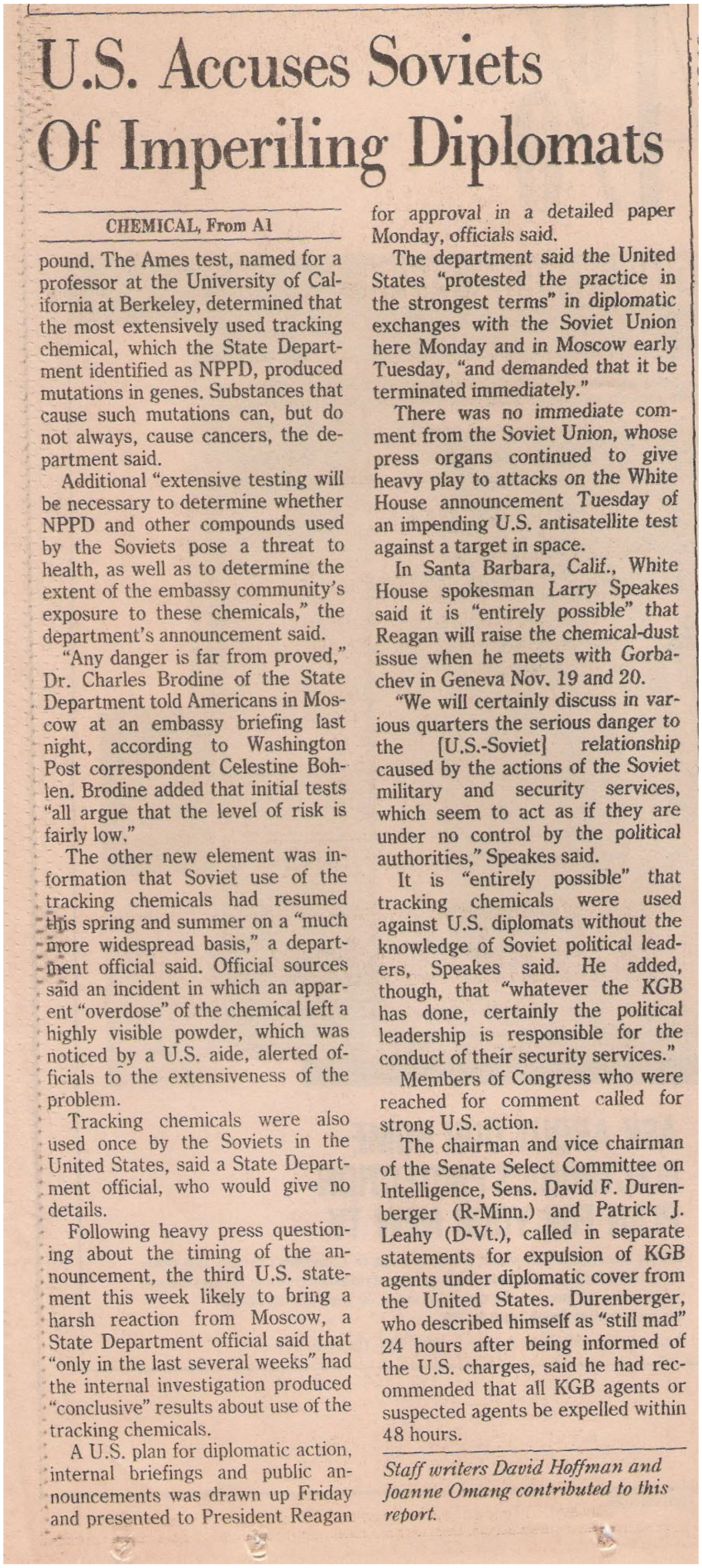
A few reports of use of such "tracking chemicals"—fewer than 10 in a decade—are said to have been in the files of the U.S. Embassy in Moscow before 1982, when their use was believed to have been stopped. No announcement of these "very sporadic" incidents was made, officials said.

Two new elements brought the problem to high level of concern, according to State Department accounts.

One was the result of a biological screening test applied in a U.S. laboratory for the first time last year to samples of the obscure com-

See CHEMICAL, A28, Col. 1

■ *Soviet "tracking agent" appears unknown to chemists. Page A28*



U.S. Accuses Soviets Of Imperiling Diplomats

CHEMICAL, From A1

pound. The Ames test, named for a professor at the University of California at Berkeley, determined that the most extensively used tracking chemical, which the State Department identified as NPPD, produced mutations in genes. Substances that cause such mutations can, but do not always, cause cancers, the department said.

Additional "extensive testing will be necessary to determine whether NPPD and other compounds used by the Soviets pose a threat to health, as well as to determine the extent of the embassy community's exposure to these chemicals," the department's announcement said.

"Any danger is far from proved," Dr. Charles Brodine of the State Department told Americans in Moscow at an embassy briefing last night, according to Washington Post correspondent Celestine Bohlen. Brodine added that initial tests "all argue that the level of risk is fairly low."

The other new element was information that Soviet use of the tracking chemicals had resumed this spring and summer on a "much more widespread basis," a department official said. Official sources said an incident in which an apparent "overdose" of the chemical left a highly visible powder, which was noticed by a U.S. aide, alerted officials to the extensiveness of the problem.

Tracking chemicals were also used once by the Soviets in the United States, said a State Department official, who would give no details.

Following heavy press questioning about the timing of the announcement, the third U.S. statement this week likely to bring a harsh reaction from Moscow, a State Department official said that "only in the last several weeks" had the internal investigation produced "conclusive" results about use of the tracking chemicals.

A U.S. plan for diplomatic action, internal briefings and public announcements was drawn up Friday and presented to President Reagan for approval in a detailed paper Monday, officials said.

The department said the United States "protested the practice in the strongest terms" in diplomatic exchanges with the Soviet Union here Monday and in Moscow early Tuesday, "and demanded that it be terminated immediately."

There was no immediate comment from the Soviet Union, whose press organs continued to give heavy play to attacks on the White House announcement Tuesday of an impending U.S. antisatellite test against a target in space.

In Santa Barbara, Calif., White House spokesman Larry Speakes said it is "entirely possible" that Reagan will raise the chemical-dust issue when he meets with Gorbachev in Geneva Nov. 19 and 20.

"We will certainly discuss in various quarters the serious danger to the [U.S.-Soviet] relationship caused by the actions of the Soviet military and security services, which seem to act as if they are under no control by the political authorities," Speakes said.

It is "entirely possible" that tracking chemicals were used against U.S. diplomats without the knowledge of Soviet political leaders, Speakes said. He added, though, that "whatever the KGB has done, certainly the political leadership is responsible for the conduct of their security services."

Members of Congress who were reached for comment called for strong U.S. action.

The chairman and vice chairman of the Senate Select Committee on Intelligence, Sens. David F. Durenberger (R-Minn.) and Patrick J. Leahy (D-Vt.), called in separate statements for expulsion of KGB agents under diplomatic cover from the United States. Durenberger, who described himself as "still mad" 24 hours after being informed of the U.S. charges, said he had recommended that all KGB agents or suspected agents be expelled within 48 hours.

Staff writers David Hoffman and Joanne Omang contributed to this report.

Washington Post – August 22, 1985
page 28

Substance Unfamiliar to Chemists

'Tracking Agent' Seen as Unlikely to Harm Embassy Employes

By Boyce Rensberger
Washington Post Staff Writer

The "tracking agent" that the State Department said yesterday was being used to monitor the activities of U.S. diplomats in Moscow appears to be unknown among forensic chemists and other specialists in the use of science for surveillance.

The Soviet powder, chemically called nitro phenyl pentadiene aldehyde, is not listed in any standard chemical reference book.

Although special powders are used by police in this country to mark valuables, preliminary indications were that the Soviet powder was of a different sort—one that may persist longer on the skin or in clothing and one that may elude detection by the usual means.

U.S. "marking powders," which glow under ultraviolet light, the so-called "black light," are used by police to mark money and other valuables so that thieves may pick up smudges that are visible only under ultraviolet light.

Several chemists said the molecular structure of the Soviet tracking agent suggested it might be similarly fluorescent, but a State Department official maintained that it was not and that the powder could be detected only by subjecting it to laboratory analysis.

Some forensic scientists suggested that although fluorescent powders would be easier to use in tracking people, they would also be easy for the marked person to detect and wash off.

The State Department also said the substance caused mutations in bacteria in the Ames test, a standard method of screening chemicals for cancer-causing potential.

But all indications are that Moscow embassy personnel picked up quantities too small to be hazardous. Dr. Charles Brodine, a State Department specialist in environmental health, told embassy employes that it could be measured in quantities of only a few billionths of a gram.

"Substances that are positive on the test," said Bruce Ames, the inventor of the test, "are often carcinogenic but there's no way you can tell without further tests." Ames, a professor at the University of California in Berkeley, said the amounts reportedly used probably pose less of a health threat than a variety of other substances naturally present in foods and which do cause cancer in large doses.

A cup of coffee, Ames said, contains natural carcinogens in amounts far larger than those of the substance picked up by embassy personnel.

The State Department said specialists from the National Institutes of Health and the Environmental Protection Agency would go to Moscow to evaluate the degree of exposure and that their findings would not be available for some time.

When Brodine briefed several hundred embassy employes and family members last night he said "any danger is far from proved" and added that tests so far "all argue that the level of risk is fairly low." Many said it was the first they had heard of the powder and several expressed concern about possible health effects, especially about the danger to pregnant women.

"I have been here two years now and I want to know what it is I should do," one young mother told Washington Post correspondent Celestine Bohlen.

Another embassy employe after the briefing said people were "concerned but quite understanding that the information at this point had to be incomplete."

Several forensic experts in this country said they knew of no comparable use of marking powders to track people. "Marking powders are used by security firms to mark valuables but to my knowledge there's no chemical substance that's put on a person to tell who he's in contact with," said John Hicks of the FBI laboratory in Washington. "But it does sound perfectly conceivable that this could be done."

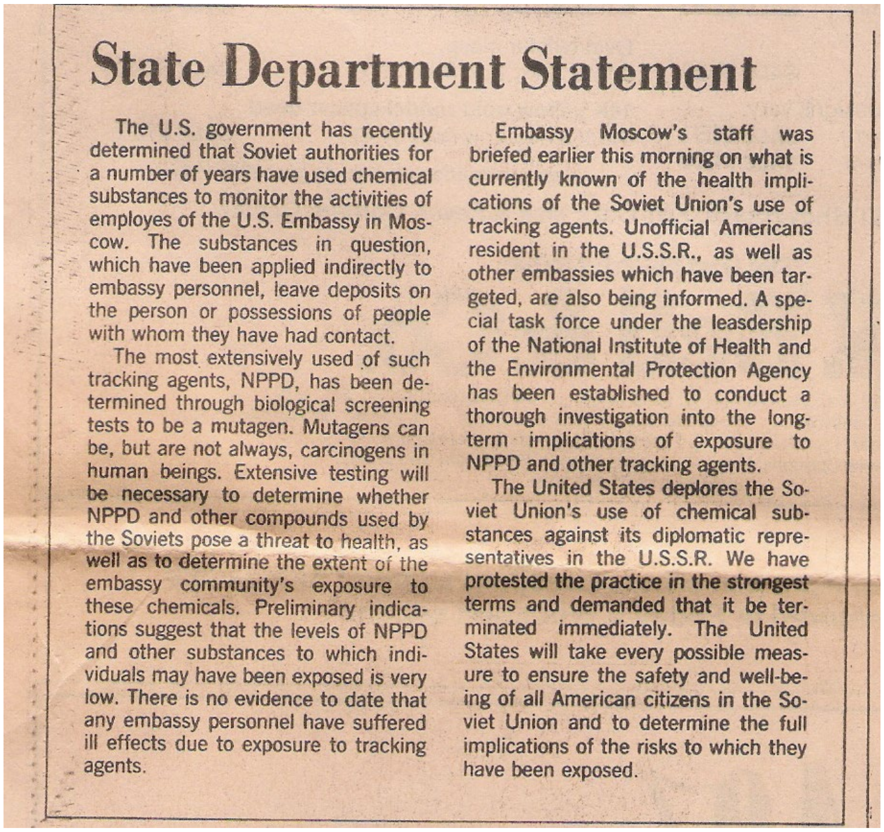
Another forensic scientist, Walter F. Rowe of George Washington University, expressed skepticism about the State Department's announcement.

He recalled a previous incident in which the State Department claimed the Soviet Union was spraying a toxic "yellow rain" in Southeast Asia. The substance turned out to be bee feces, he said.

"After the yellow rain business," Rowe said, "I don't have a bit of faith in the State Department. I don't say it's impossible the Soviets are doing it. I just say I would like to see a lot more evidence."

Attempts by The Washington Post to learn more about the Soviet powder from various federal agencies with expertise in such areas were unavailing.

Washington Post - August 22, 1985
page 28



State Department Statement

The U.S. government has recently determined that Soviet authorities for a number of years have used chemical substances to monitor the activities of employes of the U.S. Embassy in Moscow. The substances in question, which have been applied indirectly to embassy personnel, leave deposits on the person or possessions of people with whom they have had contact.

The most extensively used of such tracking agents, NPPD, has been determined through biological screening tests to be a mutagen. Mutagens can be, but are not always, carcinogens in human beings. Extensive testing will be necessary to determine whether NPPD and other compounds used by the Soviets pose a threat to health, as well as to determine the extent of the embassy community's exposure to these chemicals. Preliminary indications suggest that the levels of NPPD and other substances to which individuals may have been exposed is very low. There is no evidence to date that any embassy personnel have suffered ill effects due to exposure to tracking agents.

Embassy Moscow's staff was briefed earlier this morning on what is currently known of the health implications of the Soviet Union's use of tracking agents. Unofficial Americans resident in the U.S.S.R., as well as other embassies which have been targeted, are also being informed. A special task force under the leasdership of the National Institute of Health and the Environmental Protection Agency has been established to conduct a thorough investigation into the long-term implications of exposure to NPPD and other tracking agents.

The United States deplores the Soviet Union's use of chemical substances against its diplomatic representatives in the U.S.S.R. We have protested the practice in the strongest terms and demanded that it be terminated immediately. The United States will take every possible measure to ensure the safety and well-being of all American citizens in the Soviet Union and to determine the full implications of the risks to which they have been exposed.

BY DANA SUMMERS FOR THE ORLANDO SENTINEL

"THE CLEANING LADY IS HERE TO DUST, SIR..."

GRIN & BEAR IT WAGNER

"Is news to us about chemicals in spy dust. We simply follow the people who are sneezing."

BY AUTH FOR THE PHILADELPHIA INQUIRER

Tass Cites a Motive

■ MOSCOW—The official Soviet news agency Tass said charges of potentially cancer-causing chemicals at the U.S. Embassy here were prompted by motives of "personal gain."

Tass said the State Department would press for an increase in hardship pay for embassy personnel here in the wake of revelations about a chemical allegedly used to track U.S. diplomats.

Chemical espionage in Moscow: Is the KGB carrying surveillance of Americans to a dangerous new extreme?

The Soviet Spy-Dust Scare

Even in Moscow's forbidding political climate, last week's news came as a distinct chill. Hastily summoned to the U.S. Embassy by phone, the 500 or so Americans over the age of 18 who live in the Soviet capital learned that they and their families had what chargé d'affaires Richard Combs delicately called "a potential health problem." Off and on for a period of years, and with increasing frequency in recent months, they had been exposed by agents of the KGB to a chemical powder that reportedly enabled the Soviets to keep track of their movements. That was spooky enough, but there was more, Combs said: the chemical, known by its initials NPPD (for nitrophenylpentadienal), was a mutagen, capable of causing chromosomal change and perhaps even cancer. An undercurrent of alarm swept the assembly of diplomats, journalists, business people and spouses: "When we go to Beirut we know we are facing terrorism," said one later. "No one ever told us this was a problem here."

Fear soon gave way to anger, however. The official briefing raised more questions than it answered. Who in particular had been exposed to the powder? How often and in what circumstances—restaurants, cars, theaters? There was no substantive response. One correspondent's wife wanted to know if the stuff could be hidden in the Soviet roach powder in her kitchen, or whether she should be suspicious of her maid. No good answer. Another woman asked if it would endanger unborn children. It came down to the same thing: "All I can tell you is there is no doubt that the KGB is using the substance," said Combs. "I can't go beyond that." The stonewalling made some Americans wonder if they were pawns in a larger geopolitical game. "They could have issued a two-sentence memo for all they said," snorted Gay Kimelman, wife of another journalist. "These are the games [both] the Soviets and Americans play, and this time they used us."

World Opinion: The timing of the revelations did seem suspect. U.S. officials had by their own admission known about the powder's use long before, but had said nothing until the very week the Reagan administration announced it would test an antisatellite weapon (ASAT) this autumn. Coming just before the Ronald Reagan-Mikhail Gorbachev summit meeting in November, the test was sure to provoke criticism, and speculation was that the administration aimed to dull any Soviet edge in the war for world opinion. The Soviets themselves, predictably, made much the same argument. The news agency Tass said that charges of chemical espionage were "absurd" and "totally unacceptable." It accused the United States of "preparing the ground for another slander campaign against the Soviet Union [and] poisoning the atmosphere in relations between our countries."

American officials denied any political motivation. In Washington the State Department said that while NPPD traces had occasionally been found on Americans in the Soviet Union as long ago as 1976, concern had risen lately because of increased KGB use of the chemical. An even more important consideration in the timing of the briefings, one administration official said, was new health fears. Among the potential effects of inhalation of NPPD in large doses, according to a still-unreleased CIA report based on animal tests, are nerve damage, respiratory failure, loss of bladder control and impotence. "The [timing] is based solely and totally on the discovery that this stuff might be dangerous," said the official. "We want the Soviets to stop using it."

Not enough is known about NPPD to evaluate the danger with any confidence. According to Purdue University chemist Mark Cushman, there are in the entire scientific literature only a few references to the substance—most from the Soviet Union and Eastern Europe, none from the United States. It is one of several related compounds investigated in the 1960s and '70s as potential antibacterial agents. It crystallizes as a white-yellowish powder and would be easy to dispense from an aerosol spray can—allegedly the method favored by the KGB. Two properties give NPPD value as a tracking agent: it comes from a class of chemicals that absorb infrared and ultraviolet light and would be detectable by a spectrometer in a lab; and it is electrophilic, meaning that it binds quickly and easily to protein molecules like those on skin and natural-fiber clothing, particularly woolens. The binding leaves a stain that cannot be washed off with ordinary soap and water. Even so, says Melvin W. Sargent, an Australian chemist and one of five Westerners to have synthesized NPPD, "it doesn't seem very practical" for surveillance teams to use a chemical that requires the bulky spectrometer. "Electronics are so much more efficient."

If in fact the Soviets do use NPPD for tracking Americans in both Moscow and Leningrad, it may be that they are unaware of any potential hazards. Some U.S. officials in Washington speculate that the Soviet police agency is acting on its own, and as one said last week, "We may not know much, but I bet we know 10 times more than they do about what this stuff can do, and it's their people running around with cans of it in their pockets." In any event, the KGB will soon have some new targets: this week a team of U.S. government medical investigators arrives in Moscow to begin addressing the unanswered questions.

PETER McGRATH with JOYCE BARNATHAN in Moscow and JOHN WALCOTT and Kim Willenson in Washington

NEWSWEEK/SEPTEMBER 2, 1985

**Newspaper? -
Sept 16, 1985**

Sept. 16, 1985

KGB may have been using spy dust as far back as '59

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Jack Anderson

Washington—Apparently the KGB has been sprinkling "spy dust" on diplomats, dissidents and other suspicious characters for years. As far back as 1959, an infernal chemical was reportedly used to track one of the CIA's most valuable "moles" inside Soviet intelligence.

The story of how he was caught was brought to the CIA by a KGB defector in 1964. Yet. Adm. Stansfield Turner, director of the CIA from 1977 to 1981, said he'd never heard of "spy dust." Neither had Malcolm Toon, who was U.S. ambassador in Moscow from 1976 until 1979.

While they were kept in the dark, the chemical dust was used to trace the movements of U.S. diplomats around Moscow. It has been detected several times since 1976, but its use against U.S. diplomats has increased greatly since last May.

The worry is that the stuff can be harmful to their health. It has been identified as nitrophenylpentadienal—or NPPD—which can cause cancer.

The diplomats pick up the surveillance chemical on their skin from, say, a car steering wheel. Then they leave an invisible trail on every doorknob they touch or hand they shake. The KGB simply administers another chemical to a suspicious hand or doorknob, and the telltale NPPD becomes fluorescent.

This is how a U.S. mole, known as Colonel Popov, reportedly was detected. The story, which was both a triumph and a tragedy for the CIA, is locked in the agency's "top secret" files. But here are the essential details:

Colonel Popov was an officer of the GRU, Soviet military intelligence. He made contact with the CIA while he was stationed in Vienna in 1953. Later he was transferred to East Berlin, where he continued to pass secrets to the CIA.

After he was called back to Moscow he continued to provide important intelligence to the CIA through Russell Langelle, an intelligence officer working under diplomatic cover. On Oct. 16, 1959, while exchanging a note on a Moscow bus, Colonel Popov and Mr. Langelle were arrested by the KGB. A Soviet defector later reported that Mr. Langelle's Russian maid had dusted the diplomat's shoes with it. The KGB, using a sniffer dog, tracked Mr. Langelle to a mailbox where the police found a note he had mailed to Popov.

Footnote: We interviewed the CIA case officer, George Kisvalter, who handled Popov in Berlin. Now 75 years old, he said he couldn't comment because he is still employed by the CIA.

"Spy Dust" Irritates Diplomats

On the evening of 21 August, U.S. diplomats in Moscow and their families were called to a special meeting at the embassy to learn that some of them might have been exposed to a "spy dust" used by the Soviet police. Charles Brodine, a State Department medical officer, had flown in from Washington to let them know that this powder, which contains 5-(4-nitrophenyl)-2,4-pentadienal (NPPD), proved to be a mutagen in the Ames test and was possibly a carcinogen. He had little else to report, except that some preliminary data showed the health risk to be "fairly low."

As U.S. diplomatic families received this warning in private, the State Department went public at the same time with a press briefing in Washington. Immediately, the news went out that Americans were being threatened with cancer because of the KGB's practice of dusting steering wheels, doorknobs, and appliances with NPPD-laced powder. A few senators clamored for retaliation. Strom Thurmond (R–S.C.) led the bidding, saying the embassy in Moscow should be closed.

> The government conceded it had known about the Soviets' use of spy powder for nearly a decade.

Meanwhile, Soviet officials flatly denied the accusation, calling it "absurd," a "provocation," and reminiscent of a "cheap detective story." Adjectives flew in all directions, but facts were few and hard to find.

Tracking powder was designed as an aid to police surveillance. American banks and robbery detectives employ a similar method for marking and tracking stolen money. But in the United States a different (also toxic) chemical is used—zinc sulfide. In the Soviet Union, according to the State Department, the powder was used to verify meetings between foreigners and Soviet citizens, among other purposes. The Soviet chemical NPPD is said to be very persistent and detectable in minute quantities with analytical methods which the department would not discuss. It's not known whether these include scanning for flourescence under an ultraviolet light, a common approach in the United States.

Scientists at federal agencies reported that they could find no listing of NPPD in the registers that name tens of thousands of toxic compounds. The Aldrich Chemical Company of Milwaukee, a major producer of organic chemicals, had no information on NPPD. Peter Andrews of the American Chemical Society made a search of chemical journal abstracts and found only seven articles on NPPD and related compounds. Six were by Soviet scientists; one was by Australians.

The timing of the announcement aroused skepticism, in part because the government conceded it had known about the Soviets' use of spy powder for nearly a decade before alerting employees. Thus the press briefing on NPPD came at a bad moment, in the same week the White House had announced that it would permit an antisatellite weapon test to go forward, despite the strain this would put on U.S.-Soviet relations. A summit meeting is scheduled for November. Reporters asked whether the Administration was trying to undermine the meeting. State Department spokesman Charles Redman replied: "There is absolutely no U.S. attempt in any way to sabotage prospects for the Geneva meeting." He added, "The timing of this whole issue was driven by the humanitarian concerns for our personnel in Moscow."

A State Department official said the government had known "since the 1970's" that the KGB was using the powder. But the Soviets' use of NPPD was "sporadic" and "infrequent," according to State, until recently. In the spring and summer of this year there was a "resurgence of more widespread appearance." Although the government had run some tests on NPPD in 1984, it decided not to sound the alarm then because it believed exposure levels were low. "Exposure is the key word here," the State Department briefer said. In "the last few weeks only" the use has been significant enough to warrant a public alarm and protest. He would not describe the levels of exposure other than to say they were at least "an order of magnitude greater than" they had been in the 1970's when the spy dust was first noticed.

Officials at the Environmental Protection Agency and the National Institutes of Health say they have been instructed to refer all calls to the State Department's press office. As a key EPA staffer said: "They're trying to make sure everything funnels through one source to make sure you don't get inconsistent answers." State's press office declined to give out detailed information on NPPD's toxicity tests, saying the data are either "too tentative" or deal with classified "operational" matters.

Most mutagens are carcinogens, and that in itself raises a concern about exposure to NPPD. However, Bruce Ames, the Berkeley biochemist who invented the Ames test, has written that many natural foods—including coffee—are mutagenic and that the range of potency for carcinogens is enormous. One must know about the potency and the level of exposure before it is possible to make a good guess about the threat to public health. Ames said the cancer risk is likely to be small, and he pointed out that "Every meal is full of natural carcinogens."

Until better data come along, not many will take issue with the State Department's decision to sound the alarm. Philip Landrigan, director of the Environmental Sciences Laboratory at Mount Sinai Hospital in New York, found it "perfectly reasonable" to warn people that they have been exposed to a mutagen. He saw this as fitting in with recent legislation that requires manufacturers to advise employees of chemical hazards in the workplace. But Landrigan said that unless exposure levels have been high, there probably is not much to worry about.

If the chemical under investigation is obscure, the same can be said about its investigators. However, it is known that Robert Goyer, deputy director of the National Institute of Environmental Health Sciences, will coordinate work by Public Health Service scientists, and that the EPA's office of pesticides and toxic substances will be sending people to Moscow as well. The White House science adviser has not been involved.

—ELIOT MARSHALL

TON POST MONDAY, SEPTEMBER 16, 1985 B13

JACK ANDERSON and DALE VAN ATTA

Soviets Sprinkled 'Spy Dust' for Years

Apparently the KGB has been sprinkling "spy dust" on diplomats, dissidents and other suspicious characters for years. As far back as 1959, an infernal chemical was reportedly used to track one of the CIA's most valuable "moles" inside Soviet intelligence.

The story of how he was caught was brought to the Central Intelligence Agency by a KGB defector in 1964. Yet Stansfield Turner, director of the CIA from 1977 to 1981, said he'd never heard of "spy dust."

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While they were kept in the dark, the chemical dust was used to trace the movements of U.S. diplomats around Moscow.

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The worry is that the stuff can be harmful to the diplomats' health. It has been identified as nitro phenyl pentadiene aldehyde—or NPPD—which can cause cancer.

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Later he was transferred to East Berlin, where he continued to pass secrets to the CIA.

After he was called back to Moscow, Popov continued to provide important intelligence to the CIA through Russell Langelle, an intelligence officer working under diplomatic cover.

On Oct. 16, 1959, while exchanging a note on a Moscow bus, Popov and Langelle were arrested by the KGB.

Langelle was interrogated and kicked out of the country. Popov was executed.

Not until 1964, when KGB Maj. Yuri Nosenko defected in Geneva, did the CIA learn how the KGB monitored Popov's movements.

Among the wealth of information Nosenko gave his debriefers was the disclosure that Popov had been caught through the use of a chemical.

Nosenko didn't know the exact chemical agent used, but he reported that Langelle's Russian maid had dusted the diplomat's shoes with it.

The KGB, using a sniffer dog, tracked Langelle to a mailbox where the police found a note he had mailed to Popov.

Footnote: We interviewed the CIA case officer, George Kisvalter, who handled Popov in Berlin. Now 75 years old, Kisvalter said he couldn't comment because he is still employed by the CIA.

Chemical and Engineering News – Feb 24, 1986

Tests show spy dust not a health hazard

Six months after the State Department charged the Soviets with exposing U.S. citizens living in the Soviet Union to a possible carcinogenic tracking agent comes the declaration that its use is targeted to a select group of diplomats and poses no health problem.

Last August, the U.S. said NPPD or 5-(4-nitrophenyl)-2,4-penta-dienal, was being used as a tracking agent and was mutagenic in bacteria and, therefore, possibly carcinogenic. To allay the fears of the U.S. communities in Moscow and Leningrad, the U.S. fielded a team of four scientists from the Environmental Protection Agency, the National Institute of Environmental Health Sciences, and the Centers for Disease Control. The team sampled 20% of the homes, offices, and cars of U.S. citizens living in the two cities.

EPA took 418 wipe samples from surfaces of objects that people are likely to touch, such as door knobs and car steering wheels. An additional 18 bulk samples were taken from dryer lint and vacuum cleaner dust. Using mass spectrometry, an EPA contract lab was unable to detect NPPD, better known as spy dust, in any of those samples.

Last month, an unspecified number of additional samples were taken by a medical technologist in the U.S. Moscow embassy's health unit. The survey focused on vehicles of embassy employees likely "to be targets for use of a tracking agent," State Department spokesman Charles E. Redman said. This time, samples were taken from cracks and crevices. An unknown number of samples from five vehicles tested positive for NPPD and another chemical, luminol. The U.S. believes that luminol is also a tracking agent.

Redman tells C&EN that fewer than 100 samples were taken in the followup survey. He said "I believe that the five positives were subjected to MS as well as a recently developed chemical test," but he is not certain. This chemical field test involves the reaction of aromatic unsaturated aldehydes, like NPPD, to the color reagent naphthoresorcinol, explains another State Department spokesman.

The simple chemical test was developed by a federal agency, possibly NIEHS, says the second State Department spokesman. Redman says a similar test might be used by the Soviets to detect NPPD. This test is now being used on a regular basis to survey U.S. embassies in the U.S.S.R., Redman says.

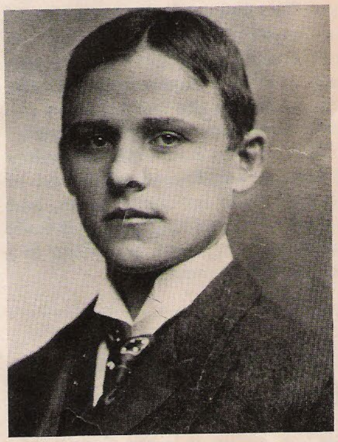
NIEHS ran a battery of preliminary toxicological tests on NPPD. Using four strains of *Salmonella typhimurium*, NPPD was mutagenic in two, weakly so in one, and not mutagenic in the fourth. Sister chromatid exchange tests in Chinese hamster ovary cells showed that NPPD does not damage chromosomes in mammalian cells in test tubes. NPPD was poorly absorbed through the skin. But when applied to the skin, NPPD did not cause birth defects in the rat. It was not an allergen in the mouse or guinea pig. Once in the bloodstream, it is metabolized and excreted within 48 hours. NIEHS concludes: "Based on the above results, contact with NPPD when used as a tracking agent does not appear to be a health risk." Even last summer, before NIEHS testing began, a government scientist told C&EN that long-term toxicity tests would not be needed. ☐

Du Pont to screen applicants for drug use

Beginning next week, people applying for jobs at Du Pont will have to submit to urine tests aimed at detecting use of illegal drugs. Current employees also may be asked to provide urine samples for testing if the site manager at their facility thinks they should, explains Bruce W. Karrh, Du Pont's vice president for safety, health, and environmental affairs.

The tests are intended to improve

Centennial of electrolytic process for aluminum marked



When Charles Martin Hall, working in a woodshed behind his family's home on East College Street in Oberlin, Ohio, discovered the electrolytic process for extracting aluminum from its oxide, he was only 22 years old. That discovery, on Feb. 23, 1886, of an inexpensive, practical way to produce aluminum was something chemists had long been seeking. Last week, Oberlin College began a series of celebrations marking the centennial of the occasion.

Hall graduated from Oberlin College only eight months before making his discovery. And, the school notes, a major factor in the achievement was Hall's mentor relationship with Oberlin College chemistry professor Frank Fanning Jewett. It was a relationship that began in 1880, when Jewett, a teacher with worldwide experience, and Hall both arrived in Oberlin. Jewett came directly from Tokyo's Imperial University, bringing with him a sample of aluminum that had been extracted by the costly sodium-reduction process and puzzling over the problem of an inexpensive approach. Hall, a freshman, went to Jewett to buy small items of equipment and chemicals for his home laboratory, and Jewett, inspired by Hall's industriousness, took the young man under his wing, giving him work space in his college lab.

Following his discovery, Hall went on to cofound Pittsburgh Reduction Co., which later became Aluminum Corp. of America. He died a multimillionaire in 1914, bequeathing extensive gifts to his community, Oberlin College, and higher education in general. ☐

After the Spydust Settled . . .

THE "spydust" crisis in U.S.–Soviet affairs has ended. The fanfare was less than deafening on 14 February when the State Department released its final report on the case, perhaps because there was so little to release.

The State Department says that Soviet agents have been sprinkling a chemical called NPPD* in places where Americans would come in contact with it, creating a chemical trail they could follow later. Last year, the U.S. government warned that NPPD might pose a cancer threat and spent 6 months researching the proposition. In February, the department came up empty handed. The bottom line, said department spokesman Charles Redman on 14 February, is that NPPD "does not pose a health hazard" to anyone.

Arthur Hartman, U.S. Ambassador to Moscow *Ordered a "pointed" search for spydust after EPA found none.*

Six months earlier, on 21 August, Redman told the press that the United States was protesting "in the strongest terms" the "use of chemical substances against its diplomatic representatives in the USSR." Redman said that NPPD tested positive in the Ames test, which uses bacteria to check a chemical's ability to cause genetic mutations. U.S. diplomats, it seemed, were working in a biohazard zone. The department's assistant medical director, Charles Brodine, flew to Moscow to break the news to the American community and give counsel to those who might be alarmed. U.S. senators inveighed against the assault. One said the Moscow embassy should be closed, not a good omen for the Reagan-Gorbachev summit scheduled to take place 3 months from then.

In late August, following the initial blast of invective, the United States sent a team of scientists to Moscow to find the evidence. The experts collected samples, analyzed the data, and wrote up several reports in December. The central paper, by Karen Hammerstrom and Richard Levy of the Environmental Protection Agency, was not released until February.

Hammerstrom directed the sample collecting effort, which she described in her paper as a random survey aimed at discovering the extent of exposure to NPPD in the entire U.S. community. The scientific team collected 418 "surface wipe" samples and 18 samples of lint or vacuumed material. Each was analyzed at Versar, Inc., a laboratory in Springfield, Virginia. The results were negative. "NPPD was not found in any of the samples," the report said. It concluded that "no purpose would be served by further random sampling of the general population."

However, the authors guessed that the State Department might be dissatisfied with the results and might want to continue looking for evidence. In this case, it said, the department should "identify those individuals and locations most likely to be exposed to NPPD and conduct sampling only among the members of that group." That is just what happened, on orders from U.S. Ambassador Arthur Hartman, who demanded "a more pointed sample."

Accordingly, the embassy in Moscow resumed the search for NPPD in January. A technician who runs medical tests for the embassy collected 189 additional samples from 30 cars used by officials who might be of interest to the Soviets. The Versar lab analyzed the samples in January and February and found five positive for NPPD. However, the lab noted that the NPPD in the samples had a slightly different spectrographic signature from the laboratory standard NPPD issued by the State Department.

Their Sampling destroyed The NPPD

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*The chemical is an aromatic aldehyde, 5-(4-nitrophenyl)-2,4-pentadien-1-al.