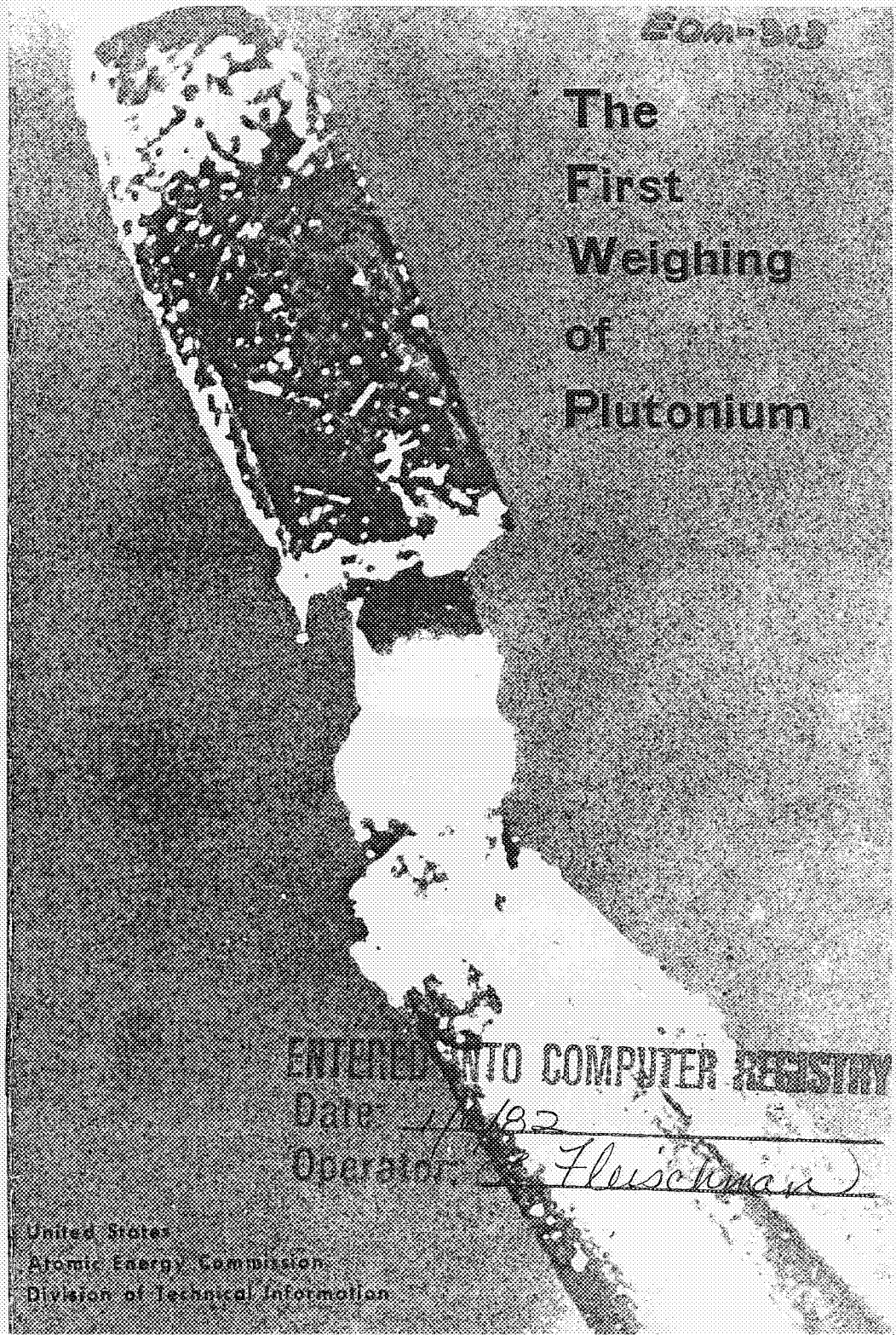
The First Weighing of Plutonium



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Recollections and Reminiscences

at the

25th Anniversary of

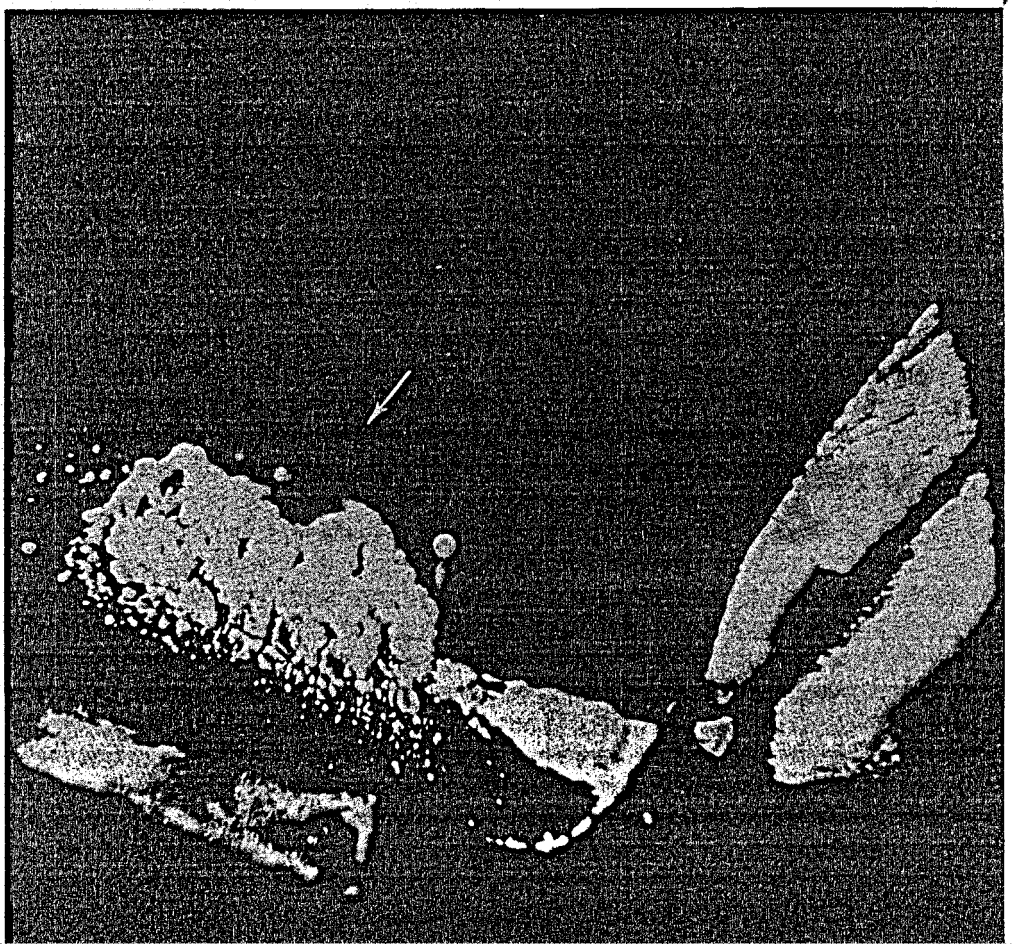
THE FIRST WEIGHING OF PLUTONIUM

University of Chicago
September 10, 1967

"These memorable days will go down in scientific history to mark the first sight of a synthetic element, and the first isolation of a weighable amount of an artificially produced isotope of any element."

Glenn T. Seaborg

On the cover is the plutonium compound (2.77 micrograms of oxide), which was the first to be weighed by man on September 10, 1942. It is on a platinum weighing boat and is magnified approximately 20 fold. The plutonium oxide appears as a crusty deposit (upper left hand part of the photograph) near the end of the platinum weighing boat, which is held with forceps that grip a small handle (lower right hand part of photograph). Below is another picture of the same compound magnified 85 times. The plutonium is indicated by the arrow.



INTRODUCTION

On September 10, 1967, a group of scientists held a reunion to celebrate the 25th anniversary of an important scientific event, and to be present when the room where the event took place was designated as a National Historic Landmark.

The event, which took place on September 10, 1942, was the first weighing of plutonium. The room where it took place was Room 405 Jones Laboratory at the University of Chicago, one tiny room of the wartime Metallurgical Laboratory of the Manhattan Project.

Two previous experiments had proved that plutonium-239 was even more fissionable than uranium-235, and thus it appeared possible that either of these isotopes might serve as the basic ingredient for a nuclear weapon. These crucial experiments were conducted on March 28 and May 18, 1941, in Berkeley. The mission of the Met Lab was to develop (1) a method for the production of plutonium in quantity and (2) a method for its chemical separation on a large scale.

The key to the solving of the first problem was the demonstration by Enrico Fermi and his colleagues of the first sustained nuclear chain reaction on December 2, 1942.

Important to the solution of the second problem was the determination of the chemical properties of plutonium, an element so new that little was known concerning these characteristics. This work was done with the extremely limited quantities available, and this first weighing of a 2.77-microgram example was a key step in this crucial aspect of the project.

The solution of these two critical problems led to the construction of the large plutonium production reactors and chemical separation plants in Hanford, Washington, and to the success of a program that culminated with the detonation of a plutonium weapon over Nagasaki that brought World War II to an end.

Plutonium still serves as a major ingredient of nuclear weapons, but perhaps more significant today is its peaceful potential as the fuel for the "breeder" type nuclear power reactor. Nuclear power stations using the breeder reactors now under development may someday be our main source of electricity, capable of supplying most of the world's growing power needs for centuries to come.

The following text, transcribed from the remarks of those scientists who gathered at the University of Chicago on September 10, 1967, to celebrate the 25th anniversary of the first weighing of plutonium, tells an important part of the story of this fascinating new element that is destined to play an increasingly significant role in the future of man.

(Left to right) Louis B. Werner, Glenn T. Seaborg, Burris B. Cunningham, and Michael Cefola in Room 405 Jones Laboratory at the University of Chicago on September 10, 1967, the 25th anniversary of the first weighing of plutonium.

The First Weighing of Plutonium

GLENN T. SEABORG The first weighing of plutonium was a significant event in the history of science and technology. A number of scientists assembled here in the Metallurgical Laboratory at the University of Chicago in the spring of 1942. Among those were a group of chemists working with me on a process to extract plutonium from uranium and its fission products. The uranium would be irradiated in the huge production reactors at some site not yet chosen.

It occurred to me that central to achieving of such a separation process would be chemical work on concentrations that would exist in the chemical extraction plant. This seemed a very far out idea, and I can remember a number of people telling me that they thought it was essentially impossible, because we had no large source for plutonium. But I thought we could irradiate large amounts of uranium with the neutrons from cyclotrons since the indications were that we probably could produce sufficient plutonium, *if* we could learn to work on the microgram or smaller-than-microgram scale. That way we could get concentrations as large as those that would exist in the chemical extraction plant.

I knew rather vaguely about two schools of ultramicrochemistry—the School of Benedetti Pichler at Queens College in New York, and the School of Paul Kirk in the Department of Biochemistry at the University of California at Berkeley.

I went to New York in May 1942, looked up Benedetti Pichler, and told him that I needed a good ultramicrochemist. He introduced me to Mike Cefola and I offered him a job, which he accepted immediately. He was on the job about three weeks later, which is illustrative of the pace at which things moved in those days.

Then I took a trip to Berkeley, early in June, where I looked up my friend Paul Kirk and put the same problem to him. By the way, I couldn't tell any of these people why we wanted to work with

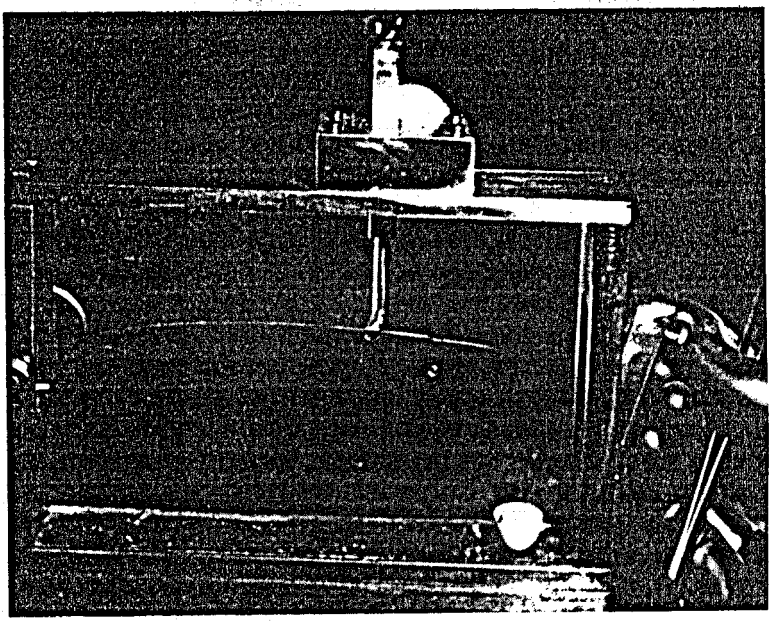
microgram amounts, or what the material was, but this didn't seem to deter their willingness to accept. Paul Kirk introduced me to Burris Cunningham. I asked him if he would come to Chicago. He accepted and was in town by the end of the month. He told me as soon as he arrived that he had a fine student, Louie Werner, whom he would like to invite and I was, of course, delighted. Louie Werner came along in a few weeks.

These, then, are the people who began the task of isolating plutonium from large amounts of uranium. We had a little cyclotron-produced sample, prepared by Art Wahl, that we brought from Berkeley. It had a microgram or so in it, mixed with several milligrams of rare earths. Using that sample the ultramicrochemists, Cunningham, Cefola, and Werner isolated the first visible amount—about a microgram—of pure plutonium. I guess it was a fluoride or perhaps a hydroxide. It was not weighed, but it could be seen! We were all very excited when we saw this first man-made element on August 18, 1942.

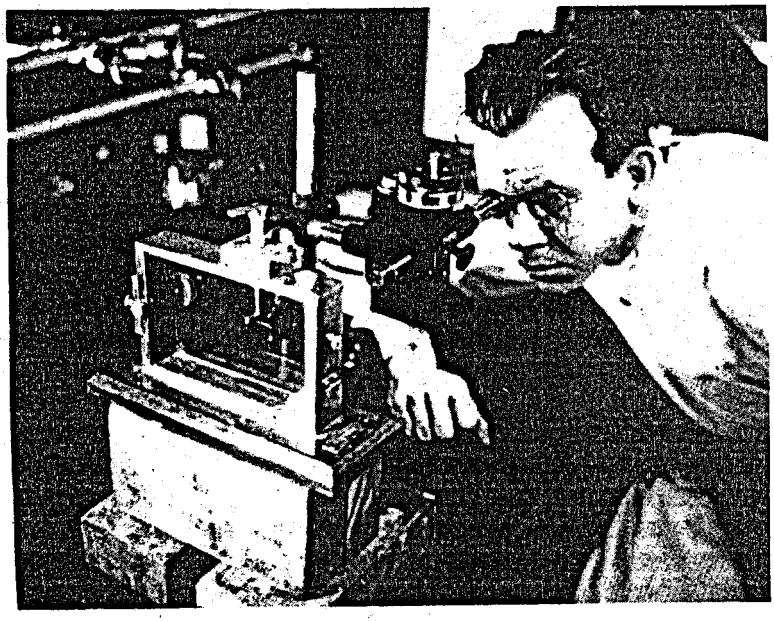
In the meantime, hundreds of pounds of uranium were being bombarded with neutrons produced by the cyclotron at Washington University, under the leadership of Alex Langsdorf, and at the 60-inch cyclotron at Berkeley, under the leadership of Joe Hamilton. This highly radioactive material was then shipped to Chicago. Art Jaffey, Truman Kohman, and Isadore Perlman led a team of chemists who put this material through the ether extraction process and the oxidation and reduction cycles to bring it down to a few milligrams of rare earths, containing perhaps a hundred micrograms of plutonium, and this was turned over to Cunningham, Werner, and Cefola. These men prepared the first sample in pure form by going through the plutonium iodate and the hydroxide, etc., on to the oxide.

This 2.77-microgram sample was weighed on September 10, 1942. The first aim was to weigh it with a so-called Emich balance, which was somewhat complicated and had electromagnetic compensation features. As it turned out, due to the heavy load in the shops, this weighing balance would have taken perhaps 6 months to build.

Cunningham then had the idea of just a simple device consisting of a quartz fiber, about 12 centimeters long and 1/10 of a millimeter in diameter, suspended at one end, with a weighing pan hung on the other end. Then the depression of that end of the fiber with the pan containing the sample would relate to the weight of the sample that was weighed. Cunningham then measured the depression of the quartz fiber with a microscope. He invented this balance himself, although he found out later that an Italian named Salvioni had invented it earlier, and so it became known as the Salvioni balance.



The Salvioni balance. Below Burris B. Cunningham operates the instrument.



I thought it might be appropriate if I called on the ultramicrochemists first, and then I shall call on those who played important roles, perhaps equally important roles, although secondary to the weighing, in the Plutonium Project here at the Metallurgical Laboratory.

BURRIS B. CUNNINGHAM As I look at this assemblage, I can't resist remarking that rarely have so many made so much about so little.

The weighing experiments were only a part of a broad program of research on the properties of plutonium carried out under Glenn Seaborg's direction in the summer of 1942.

The experiments, which immediately preceded the weighings, and the weighings themselves represented two important scientific "firsts". They afforded the first human glimpse of a man-made element and they were, to the best of my knowledge, the first ultramicro, gravimetric, chemical experiments carried out in the United States.

Now after all these years, it is difficult to recall the psychological impact of these events. Today alchemy is a thriving, commonplace business. But at that time we, who had been brought up in an older tradition, saw it as a miracle and just a little bit difficult to believe in.

More than a year after the first isolation of plutonium, I recall one of the members of our group arguing vehemently that plutonium* wasn't really plutonium at all; it was just an oddly behaving isotope of uranium. And the ultramicro work met with similar skepticism. When I first showed Dr. Seaborg the data on the reproducibility of the balance that we intended to use for weighing the plutonium, he thought that I had slipped a couple of decimal places, and that these deviations must surely be in micrograms and not in hundredths of a microgram. And I recall a long conversation with Truman Kohman, in which I vainly tried to convince him that it was possible to measure a microliter of solution to 1% accuracy. I'm not sure that he believes it even to this day.

Mike and Louie and I believed in plutonium, but wondered constantly if the stuff that we were precipitating from our little cones was genuinely pure material. There was always the possibility that it might be grossly contaminated with other material.

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*"Plutonium is so unusual as to approach the unbelievable. Under some conditions it can be nearly as hard and brittle as glass; under others, as soft and plastic as lead. It will burn and crumble quickly to powder when heated in air, or slowly disintegrate when kept at room temperature. It undergoes no less than five phase transitions between room temperature and its melting point. Strangely enough, in two of its phases, plutonium actually contracts as it is being heated. It also has no less than four oxidation states. It is unique among all of the chemical elements. And it is fiendishly toxic, even in small amounts." Glenn T. Seaborg

And, of course, everybody worried about the calibration of the balance. How could you calibrate a balance to a hundredth of a microgram when you didn't have microgram weights to do it with? Well, these doubts dissipated gradually, and we came to accept the obvious. Plutonium *was* a little complicated in its chemical behavior perhaps, but much easier to purify than many elements which had been discovered a half century earlier. And after we had calibrated the balance in three independent ways, and had come out with the same answer, we realized that Bureau of Standards certified microgram weights were not essential.

Looking back on these early experiments, one sees that they were not really glamorous or high-flown at all. They were straightforward and pretty simpleminded, really. And in a way, this seems a pity, because in the case of an event of such historical importance, one feels that it should have involved at least one esoteric principle of chemistry or physics. The balance ought really to have been something much more complicated than a quartz glass fiber enclosed in a homemade case of wood and glass. But that's the way it was. I suppose that most of us looking back on those early days and recalling the challenge and excitement would not change them if we could. I became hooked. I've since had the great pleasure of being the first to do experiments with other new elements. And, in a way, I'm still doing business at the same old store. Only the atomic numbers have changed.

GLENN T. SEABORG Now it seems rather commonplace that it should have been easy to handle materials on this scale. But at that time, it was very doubtful in our minds whether you could even separate or purify and handle, as a chemical entity, materials as small as micrograms and submicrograms.

MICHAEL CEFOLA My initial knowledge of the existence of the project was disclosed when Dr. Seaborg contacted me in May 1942 and wanted to discuss my joining his group at the Metallurgical Laboratory.

The outcome of the meeting was that I learned very little about the nature of his research except that ultramicrochemical techniques would be involved. He strongly stressed the importance of the work without giving specific details.

As I could not join the group until June, I had ample time to speculate about my role in the laboratories. My first guess was that I would be carrying out analyses of impurities on trace levels. That I was wrong became most obvious when, on a visit to the Met Lab before the official starting date, I saw one of the rooms lined with counting

equipment set up by Dr. Spofford English and on the roof huge evaporating dishes filled with ether extractions of uranyl nitrate solutions.

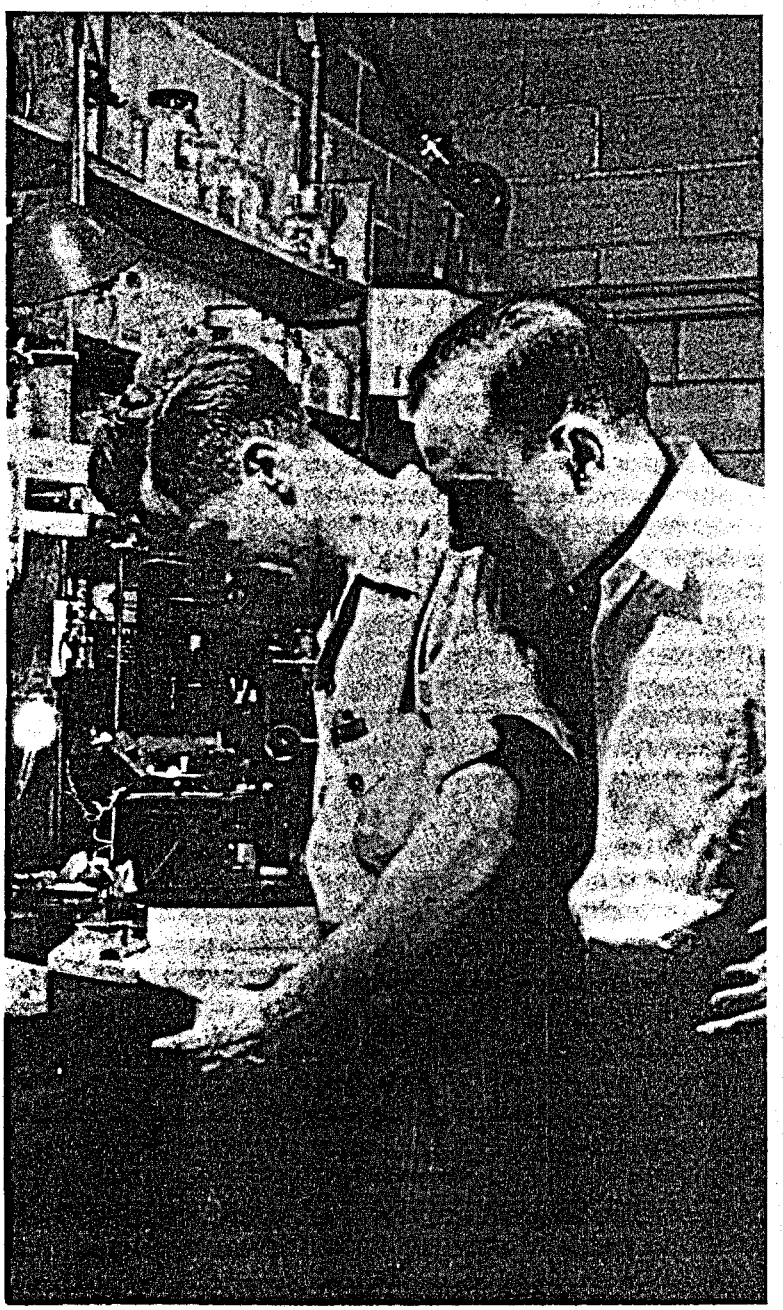
Since much of my work would center around ultramicrochemical techniques, I was instructed to line up available related equipment. This included microscopes which were already becoming scarce because of the large demand and the halt of imports from Germany. When equipment trademarked Zeiss later arrived at the laboratory, I was reminded that we were at war with Germany and that U. S. currency would reach Germany through South America.

Initial attempts at restricting unauthorized persons from entering the laboratories on the top floor of the Jones Laboratory were feeble. Later a partition was installed in the corridor leading to Dr. Seaborg's laboratories and the responsibility for admittance was assigned to some recent high school graduates and a technical assistant. This, at times, created amusing situations such as when Dr. Schlesinger, Chairman of the Chemistry Department at the University of Chicago, knocked on the door and walked in without properly identifying himself. He was immediately stopped by one of the more aggressive young men who gruffly said, "Where do you think you're going?" The reply was a meek "But I am Dr. Schlesinger." "I don't care if you're Roosevelt. You don't get in here without proper identification," the young man retorted, and, not realizing the stranger's identity, pushed him outside the entrance. The difficulty was resolved when Dr. Seaborg identified Dr. Schlesinger.

The equipment from Zeiss and Bausch and Lomb, which included microscopes and micromanipulators allowed us to carry out reactions on sub-micro amounts of materials. Even before the isolation of the relatively pure plutonium, many experiments, to test the efficiency of different precipitates as carriers and using tracer amounts of ^{238}Pu, were executed on this scale to conserve the material for experimentation by other members of the group.

Essentially the reactions were performed in capillary cones (similar to centrifuge cones) of about 1 millimeter diameter, 3 to 4 mm in length and a capacity of approximately 1 microliter. Because of the cone size, reagents were transferred into them by means of handmade micropipets with tips as small as 0.01 microliter.

As a consequence of the rapid evaporation of water from such small volumes of solutions, the reaction vessels were placed in a specially designed moist chamber. The entire assembly was then put on a microscope stage where the progress of the reaction could be witnessed.



Isadore Perlman and Michael Cefola work in Room 405 in 1942.

Immediately following the isolation of microgram amounts of plutonium, this scale of experimentation became extremely useful especially in the examination of chemical reactions. Conservation of the material was of utmost importance because we needed much information and had so little with which to work.

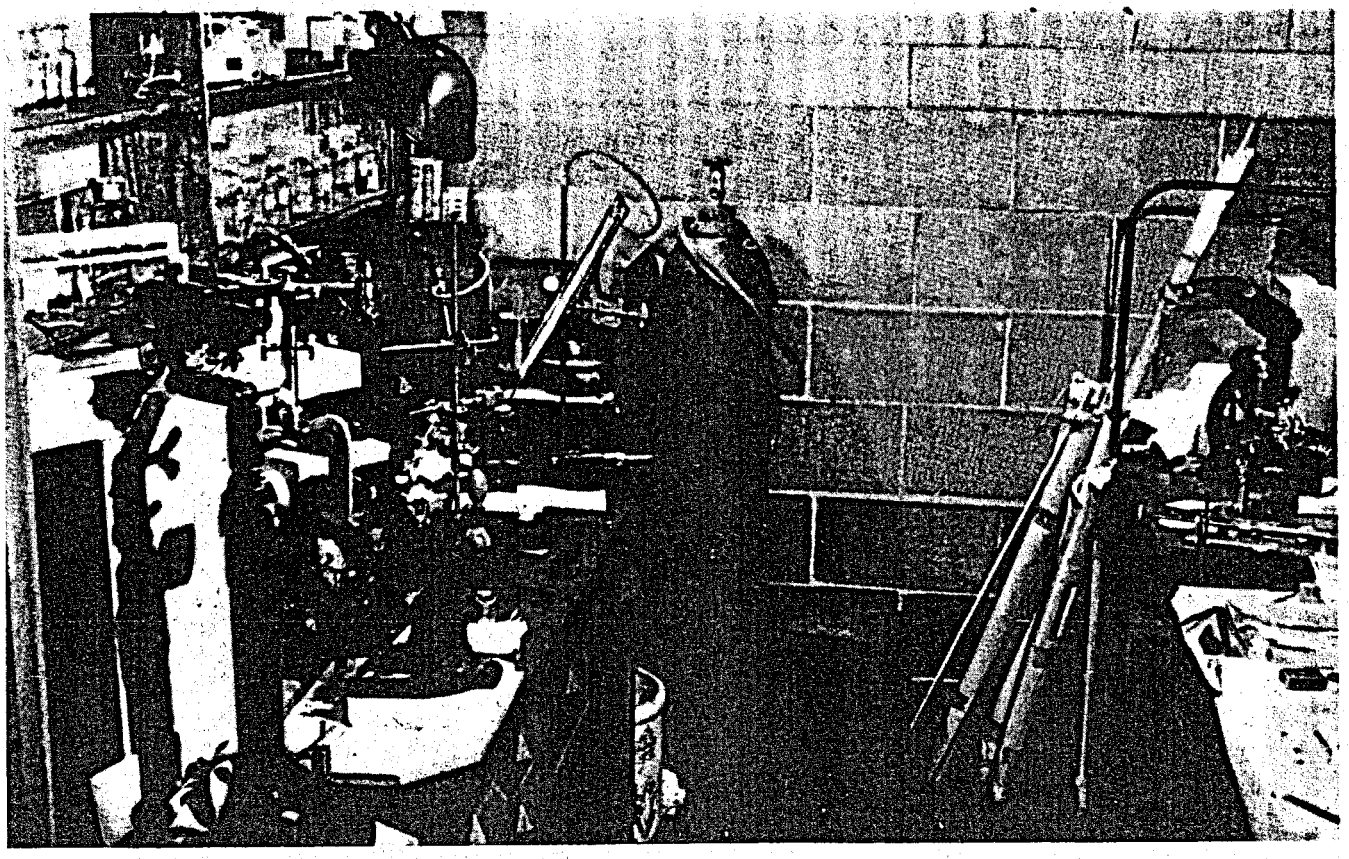
This milligram-to-submicrogram experimentation substantially reduced the time required to achieve our goal.

GLENN T. SEABORG Mike's remarks also reminded me of the curiosity that people had as they joined the project. I used to have the new man, the neophyte, come into my office, and then I had the pleasure of explaining to him that we were working on a new element and watching his consternation and almost unbelievable surprise. In fact, I would often ask them what they thought we were working on, and sometimes the answer would be, "Well, I don't know, but at least I'm sure that it's one of the 92 elements."

I'm going to call on Louie Werner next, and that reminds me of the size of the room in which this work was carried on. This was in Room 405 of the Jones Laboratory, and this is a room 6 feet wide and 10 feet long. I have taken pleasure on a number of occasions when I describe this work to point out that one of the ultramicrochemists who worked in this room was 6 feet 7 inches tall–that's Louie Werner.

LOUIS B. WERNER Everything didn't go smoothly by any means. The most serious problem occurred when I was in the microchemical laboratory and heard a violent clatter in the main part of Jones Laboratory. I went to investigate and found that the centrifuge in which I had placed the world's supply of plutonium had come apart, and the solution of plutonium was dripping down through greasy motor bearings onto the floor of the laboratory. That was a black day. Fortunately, by sopping it up with towels and sponges and digesting them, we were able to recover almost all the plutonium. As it turned out, this experience came in quite handy for purifying the first batches of reactor-produced product, which the chemical engineers turned out at Clinton Laboratories in Oak Ridge, and which seemed to contain a little of almost everything the Jones Lab material had picked up.

Once the pure product was isolated, everyone naturally wanted to see what it looked like. However, there wasn't much to see, and there was some skepticism as to whether there was anything there at all. Rather than tie up the tiny plutonium supply for exhibition, we thought we might engage in a harmless deception, and make up a somewhat more impressive solution of simulated plutonium from green



Room 405 Jones Laboratory at the University of Chicago in 1942.

ink. Green seemed to be the predominant color of aqueous solutions of plutonium. And so, the day before an important visitor was scheduled to arrive, we made up a colored solution and set it aside. The next morning, just before our guest arrived, we found to our horror that the green color had turned purple! Unfortunately, we did not have the courage of our convictions, because it turned out later that the +3 oxidation state of plutonium *was* purple in color.

There were also educational aspects of our activities during that period. A technical seminar was held weekly to which, however, we junior scientists were not invited. Undaunted by this, an independent seminar was organized. A suitable round conference table and meeting room were found in the back of Hanley's Bar on 55th Street, and, with the aid of Hanley's technicians and chemical supplies imported from Milwaukee, these seminars became very popular. The subjects of these conferences tended heavily toward the theory of games and applied statistics. An exercise that was practiced occasionally during these sessions was one which was attributed by his disciples to Professor Charles Coryell. It was called "five card draw, clubs wild".

On a serious note, it was an inspiring experience to be associated with the Plutonium Project, and an influence never to be forgotten. It was an opportunity to meet with, and be inspired by, the current and future famous scientists of the world, many of whom are assembled here today.

GLENN T. SEABORG It's true that we did fib a bit and use green dye and even aluminum hydroxide dyed green to represent the plutonium hydroxide. But I remember we mitigated this a little by carefully saying to visitors, at least on occasion, that "this represents a sample of plutonium hydroxide". I don't believe the visitor completely understood the significance of that, but it wasn't our fault if they thought it actually was a plutonium hydroxide sample.

Now I want to introduce "Iz" Perlman, who played a very important role in the leadership of this plutonium chemistry group. He was an administrator but at the same time a laboratory man, and in fact, in later stages of the ultramicrochemistry, he actually trained himself as an ultramicrochemist and carried on these intricate experiments, which is illustrative of his experimental chemical capability.

ISADORE PERLMAN The splendid group we have here today all share the peculiar sense of wonder that was aroused by seeing a new element for the first time. It was the first man-made element seen with the naked eye, and an element, indeed, which was to be fateful in the

Louis B. Werner and Burris B. Cunningham in Room 405 in 1942.

history of humanity. We're commemorating a weighing, and for those of you who are not chemists, I think the full impact of what it means to weigh something perhaps doesn't register. Although there have been many elaborate and very important techniques evolved to probe into the nature of chemical matter, the touchstone for the evolution of an element's chemistry comes from the preparation of a pure compound, and weighing plays a fundamental part in knowing that one has a pure compound.

I would like, if I may, to share a little reflective glory in this by telling a small story. I don't know what the libel laws are in the State of Illinois, so I will make this short. As already mentioned there are others who carried out some of the initial steps in the separation of plutonium from rather bulky amounts of material down to where the micro-chemists could take over. A number of us worked on this. And at one stage some of this fell into my care. As I remember, the material was in a beaker of perhaps a half a liter or so when it was put away for the night. The next morning the beaker was broken. A lead brick had fallen over, and here was this precious material spilled. Fortunately, it happened to be sitting on a Sunday edition of the *Chicago Tribune*. A half a liter of anything is nothing for this paper to absorb and we proceeded to get the very largest evaporating dish we could find. It was larger than a bathtub and smaller than a swimming pool, and we dumped this in with the idea of subjecting it to what the chemists call wet ashing. We digested it with nitric acid and kept this witches brew going for days. Which reminds me that this is September 10, and if it weren't for my participation in the isolation of plutonium, this anniversary would probably be celebrated in August.

Well, we finally got all this in the solution and I remember vividly that the print still floated. I was very grateful for having that newspaper there, and I cannot avoid reflecting that among the Democrats of left-wing persuasion, I'm probably the only one that digested an edition of the *Chicago Tribune* so thoroughly.

GLENN T. SEABORG We have been reviewing the work of one of the sections in the Chemistry Division of the Metallurgical Laboratory, namely the Plutonium Section. But there were three other important sections. These sections were under the overall leadership of the successive Chemistry Division Leaders. First there was Frank Spedding, then James Franck, and then Thorfin Hogness, who played a vital role in the direction of the overall chemistry program.

George Boyd was the section chief of the Analytical Chemistry Section, which performed the analyses that were so vital at all stages to

the success of the project. George is a scientist's scientist. There are many things about him that impress me. One is that I know of no one who is more familiar with chemical literature. He seems to absorb and remember just about everything that he's ever read or heard. I don't know how he does it but if you want to get information on some early phase of this work or any related field, you can always get it from George.

GEORGE E. BOYD One of the great things about these early days to those who were associated in this intellectual voyage were the people that we came into contact with. I was an undergraduate, a graduate student, and an instructor in the Chemistry Department under Herman Schlesinger's chairmanship, and I remember clearly how we were impressed by the way in which the whole chemistry effort moved after these brilliant fellows came to Chicago.

I got into the Plutonium Project because in the State of Michigan anyone who goes deep into a copper mine in the upper peninsula must be accompanied. Volney Wilson, one of Arthur Compton's graduate students, was working on the penetrating meson component in cosmic rays and needed someone to go with him into one of those mines, which go down several miles through thick layers of dense basalt rock. Later, Compton, knowing me as a chemist, asked if I would help out in chemical matters in connection with the study group on uranium fission that he had organized in the Chicago Physics Department.

I remember those days in 1942 because Professor Compton, after great study of the literature on uranium fission, decided that the rare earths were going to be very important. He was really right in a way that no one could have foreseen, because as we know now plutonium and the other heavier elements form a second "rare-earth-like" series.

He invited a great chemist, Herbert N. McCoy, who was an expert on the chemistry of europium and was known as the co-discoverer with Frederick Soddy of the concept of isotopes. McCoy had worked for a long time in radioactivity and also knew a great deal about rare earths. My apprenticeship to him consisted of going out to the Lindsay Light and Chemical Company plant and working with him on uranium. It's hard to believe how ignorant we were chemically of the then known heavy elements. I, for one, had never seen a uranium compound before!

Fermi* wanted some uranium dioxide. The black oxide was available in West Chicago as a by-product so McCoy and I were to make

*Enrico Fermi, the Italian physicist, led the team of scientists who built the first nuclear reactor at the University of Chicago under the West Stands of the Stagg Field stadium. This reactor sustained a chain reaction on December 2, 1942.

the red oxide. In this plant there were huge vats filled with rare earths in varying states of separation by fractional recrystallization. They were extremely beautiful with their typical delicate colors. It was at Lindsay Light and Chemical that the first uranium dioxide was made and brought back to the University for Fermi to use in one of his early experiments.

In the late winter of 1941-42, McCoy advised Compton that he needed someone who knew something about rare earths to guide the chemistry effort. He named Frank Spedding who came to Chicago in March 1942. Spedding was the first director of the Chemistry Division, which was organized into the four sections that Glenn Seaborg has just mentioned. Glenn, Charles Coryell, Milton Burton, and I were the four individually responsible for these sections.

As the person responsible for analytical chemistry, I came into direct contact with the physicists on the Plutonium Project. They were interested in obtaining extremely pure uranium and extremely pure graphite. Fermi was building one "criticality experiment"* after another in the handball court under the West Stands of Stagg Field. The reproduction factor, k, kept going up–0.96, 0.97, 0.98, etc.–as increasingly pure uranium oxide or metal and graphite were used in his experiments. The levels of purity required seemed fantastic and there was the need to analyze for impurities at part-per-million concentrations. The field of trace analysis in analytical chemistry had scarcely been developed, and we had the job of not only analyzing for many unaccustomed elements as impurities, but also doing it at levels that had never been attempted before.

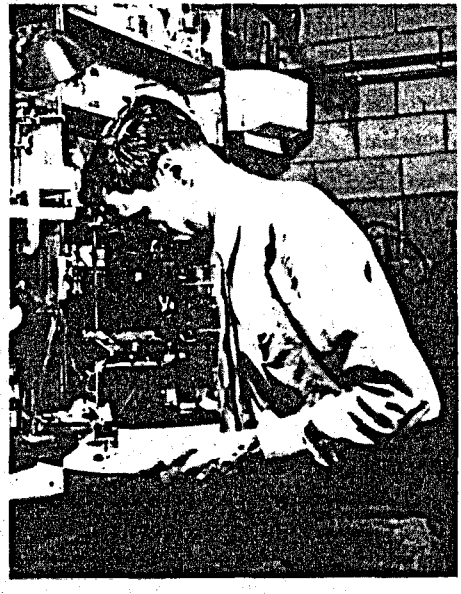
Professor Compton asked our section to work with the Mallinckrodt Chemical Company in St. Louis whose chemists had devised an ether extraction process for the purification of uranium. A method was needed to show that this solvent-extracted uranium had a high degree of purity.

One of the greatest assists we got was with a nonchemical procedure devised by Fermi. This was the so-called "shotgun" method wherein a kilogram of uranium was extracted and the impurities were collected and measured in aggregate. It was described as shotgun because it measured all the impurities at once. Even with the purest of uranium, if you extract a kilogram you will recover a milligram to a fraction of a gram of impurity. These impurities were pressed into a pellet and placed in a paraffin geometry with a radium-beryllium source. The

*Criticality describes the state of a nuclear reactor when it is sustaining a chain reaction.

neutron absorption by the impurities was measured by the activation produced in indium metal foils. This was an extremely elegant method which Fermi improvised in one day, it seemed, just out of discussion of this problem of control analyses for the purity of uranium.

The problem of the purity of uranium was solved. The problem of pure graphite was solved. The chain reaction did run, and plutonium is now made in very large quantities. It was a very great experience for all of us to have been in contact with people like Arthur Compton, Leo Szilard, Enrico Fermi, Sam Allison, Herbert McCoy, James Franck, Thorfin Hogness, Warren Johnson, and many, many others that regrettably I cannot name. It was a great human experience!



Glenn T. Seaborg in Room 405 in 1942.

GLENN T. SEABORG Next, I would like to call on Milton Burton who was the chief of the section on Radiation Chemistry. Milton and his group of some 40 or 50 chemists tested, under the radiation conditions that would exist in the actual production, all the materials from the production reactor to the chemical extraction process. In order to do this they used Van de Graaff accelerators at Notre Dame University and MIT and cyclotrons at Berkeley and Washington University. This was an important part of the project because these materials had to stand up under the radiation in order for the project to be successful.

MILTON BURTON I remember May 15, 1942, the day that I arrived to go to work on the job. It was in the early evening. I checked in at the Miramar Hotel–that first (slightly strange) resort of nearly all of us on Woodlawn Avenue–and a little while later was greeted in the lobby by Harry Spedding, Glenn Seaborg, and one other person (it may have been Charles Coryell). I was in my natural state of ebullience and began talking about some work I had been doing. I used the word "photochemistry". Immediately there was a hush. Spedding whispered to me that I mustn't use that word because people would associate it with me and begin wondering what I was doing in Chicago. And, I presume, thus arrive at the conclusion that we were working on the development of an atomic bomb.

Security was an enigma to me then and I won't say what it is to me now. I remember some time later that Coryell and I were reproved for referring to K in a sidewalk conversation. We were talking about potassium* but the woman who overheard us knew k only as a mysterious, secured and thus sacred symbol and promptly reported us. I am certain that k as the reproduction factor for a nuclear reactor meant nothing at all to her.

I remember Lewis and Randall's book *Thermodynamics*† in which they told how release of atomic energy could be involved in the production of the sun's energy. In the 1920s we had all taken it for granted that a similar process was ultimately to be produced on earth, but not in our lifetimes. Then Taylor's *Treatise on Physical Chemistry*‡ described the von Halban suggestion for obtaining atomic energy. Again we put this down as something very, very far in the future. Well, those things weren't nearly as far in the future as we had thought.

In our group were a number of young men–all of them impudently young. And it wasn't merely that they had the audacity, they had the effrontery to assume that a thing like large-scale plutonium production could be accomplished. I think that effrontery is the peculiar quality of genius–this willingness to say "The past has nothing to do with what I now propose to do. Prejudice that we may have absorbed from our teachers has nothing to do with what we are going to do. We are just going ahead and do it because it can be done". Seaborg's people did these things.

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*K is the symbol for potassium.

†*Thermodynamics and the Free Energy of Chemical Substances*, Gilbert N. Lewis and Merle Randall, McGraw-Hill Book Company, 1923.

‡*Treatise on Physical Chemistry*, Hugh S. Taylor (Ed.), D. Van Nostrand Company, Inc., 1924.

I was on the faculty at New York University at the time Mike Cefola submitted his thesis for his doctorate and was one of the examiners of that thesis. Several of us, equally and similarly troubled, got together and said substantially, "Oh, my God! What do you do with a thesis like this?" The thesis was addressed to the question of the limits of extension of microchemistry. That seemed to us to be an essentially speculative document which didn't qualify properly as a Doctor's thesis. However, since Benedetti Pichler had given his blessing, and he was the faculty supervisor involved, we felt that we couldn't properly reject the subject. So we approved and accepted it. Thus Cefola got his degree, and Seaborg got Cefola because the thesis was far more important than we realized.

Another incident involves Louis Werner and Burris Cunningham. Now, in the course of my work here at the Met Lab, I became Chairman of the Building Committee. It followed, as the night the day, that I was asked to assume some responsibility for the first chemistry building at Oak Ridge, where Lou Werner was supposed to go. We planned a big microchem room—big enough to contain people like Lou (already 7 or 8 feet tall at the time) and people like Mike Cefola at his present girth.

There was the inevitable complaint from Oak Ridge, "Why do you need such a big room for microchemistry?" And I said, "This is for microchemistry, not for *micro chemists*. You should see the size of the chemist who is to be in charge". The point won its way.

We did have security here at the Met Lab and very fine security, I later discovered. Also we had a sort of security which protected itself from "unnecessary" information at times.

I think it was Leonard Trieman who visited a friend at Northwestern University and was asked, "Say, what is this about the critical mass of plutonium?"

Well, poor Leonard wasn't ready for that one at all. Remember this was during World War II. Not only was the word "plutonium" very, very secret, but the idea that we were talking about the critical mass of any element was certainly a highly "secured" bit of information even though the words "critical mass" were hardly likely to be interpretable to an outsider. Leonard couldn't do anything but ride with the punch. "I haven't the vaguest notion of what you're talking about. Tell me why you're asking it and maybe I can figure it out."

And his friend explained, "Well, I was riding on an elevated train on 63rd Street. Two fellows in the seat ahead of me were talking about it. I didn't see who they were".

Leonard's next ploy was, "Why do you ask me?"

"Well, I figured that they were from the University of Chicago and you might know".

Leonard said, "I don't know a thing about it, never heard of it". However, he didn't forget. When he came back here he told Warren Garrison who passed it to Gus Allen, who came to me and inquired, "What should we do?"

I reached for the telephone right then and got the security office–someplace downtown I thought–and told them that a matter had come up that I felt ought to be discussed. I thought that maybe in a half-hour someone ought to be walking in. Well, within a minute a fellow came into my office, put an identification card in front of me, and introduced himself as Captain so-and-so of Counter Intelligence–I think they called it CIC at the time. I don't know whether any of you people knew it but this fellow was one of the workers in our chemistry stock room. A very efficient man whom we took for granted as a useful fixture. He wasn't the chief of the stockroom. I thought earlier that he seemed rather superior for his modest job. That this apparent underling was a captain in the Army was a shocker! When he asked about the problem I, of course, told him about the incident in detail. His response was totally unpredictable: "What is plutonium?" That was another shocker! I had to tell him that that's what he was there to protect.

Now, the reason I like this last story particularly is because it later appeared in a modified version in *The New Yorker.* It was in an article by Daniel Lang* telling about atomic energy incidents involving the Army. And, of course, you would know that the Army doesn't demean itself in such matters. They told the story factually, how they really couldn't do anything about the incident except to pray that such things wouldn't happen again. However, they didn't know how to prevent them and could depend only on the good sense of the people involved. All that is true. But the thing that was not mentioned in this story was the question, "What is plutonium?" I think now they know.

GLENN T. SEABORG You had me worried for a moment. I didn't realize we had such a fellow in our stockroom. Maybe it wasn't so serious if he didn't know what plutonium was.

Next, I'd like to introduce Charles Coryell who was the chief of the section on fission products. Nathan Sugarman was one of his associate section chiefs and Tony Turkevitch was one of the other associate leaders. This is the section that played a very vital role also in the

*Daniel Lang's stories from *The New Yorker* were collected and published in *Early Tales of the Atomic Age,* Doubleday & Company, Inc., 1948.

success of working out a chemical process and other aspects of plutonium production. It was their responsibility to determine the radioactive characteristics and the relative yields of the fission products. And they did a first-class job on this. Their yield curve was published soon after the war was over. This was a very definitive work that stood for many years and it took a long time to improve upon it.

CHARLES D. CORYELL When I arrived at the Metallurgical Laboratory from UCLA on May 5, 1942, I had absolutely no previous experience in nuclear chemistry. I was charged by the Project Director, Arthur H. Compton, and the Director of Chemistry, Frank H. Spedding, with building up a group to study the fission products—that batch of 36 elements from zinc through terbium formed in the fission of uranium.

It was impossible to find nuclear chemists, so beginning with Nathan Sugarman we collected a group of physical and analytical chemists. Self education was a very important part of our job. We also learned a lot in the very excellent course Glenn Seaborg gave evenings in July and August 1942. I worked long and late to prepare a respectable set of lecture notes to help later newcomers.

Seaborg appointed Isadore Perlman and Spofford English as chemical and instrumental liaison between his plutonium group and ours. Spof taught me to count, using ^{238}Pu and alpha counters that were very sensitive to shock. If you sneezed once, you got 10 extra counts.

I had been recommended for the project by Compton's first chemistry advisor, Herbert N. McCoy, apparently as a consequence of heated discussions we had had about oxidation states of rare earths. My Ph.D. thesis involved divalent silver, a very powerful oxidant. It is not quite as powerful as ozone, but it is much more dynamic. Ozone was then being used in an effort to oxidize plutonium beyond the hexavalent oxidation state. Our great hope was to get an octavalent state that might have a volatile tetroxide. That hope hasn't yet been realized, and Seaborg doesn't think that it will be. My experiments with divalent silver helped get plutonium oxidized faster, and led to a secret patent, now declassified.

My group had another occasion to be involved with plutonium after the first reactor CP-1 started on December 2, 1942. One could see the pile under the seats in the stadium if one had business. Shortly after start-up, Sugarman and I made a visit to see what constructive pile research we fission-product chemists could do. Walter Zinn and George Weil showed us around cordially, and we saw the huge irregular pile of

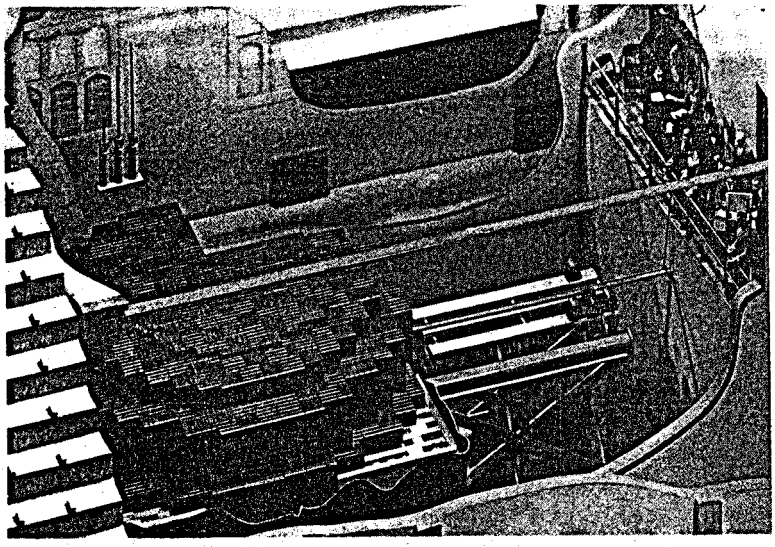
graphite blocks seeded with uranium dioxide "eggs" and, in the center, uranium metal "eggs". We decided to try an experiment to determine the ratio of ^{239}Pu formation to ^{235}U fission* in uranium in the reactor and, if intensity of radioactivity permitted, also the yield of 7-day ^{237}U formed by the (n, 2n) reaction.

In February 1943 half of the young fission-product chemists got to see the pile when they carried over about five Spedding "eggs"–5-kilogram cylinders of uranium. These were exposed for 5 hours to the neutrons on a graphite ledge a few feet from fuel in the pile, with power level of 50 watts. A few days later the other half of the group made a visit to the pile and brought back the irradiated uranium to our laboratory. With the aid of plutonium procedures Arthur Jaffey furnished to Buck Rubinson, the ^{239}Pu was laboriously separated from the uranium and fission products. Its initial counting rate was 5×10^{-8} that of the uranium. Don Engelkemeir separated and purified the ^{239}Pu and Larry Glendenin determined the yield of 12.8-day barium-140 from which the fission rate could be established. A figure was obtained for the ratio of neutron capture rate in ^{238}U to fission rate in ^{235}U in natural uranium exposed to pile neutrons. When corrected for the modern fission yield of ^{140}Ba it was 1.4 ± 0.3, higher than the modern value by 0.4. The yield of beta-emitting ^{237}U was too low to be seen above the radiation background of freshly purified uranium. Later studies gave its yield, compared to fission, as about 0.002.

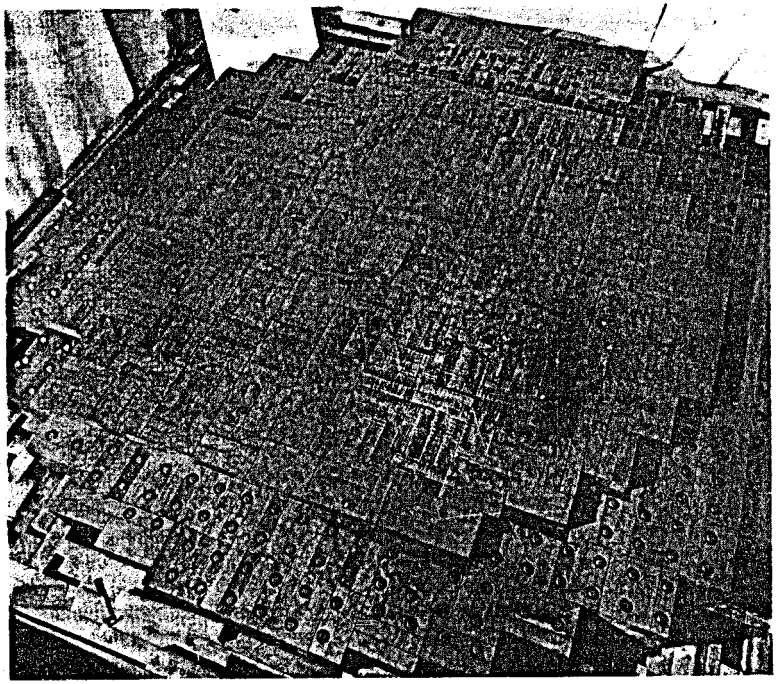
Those days on the Plutonium Project were exciting days for all of us, and two of the greatest features were the generosity of the experienced with the unexperienced and the democratic atmosphere of the whole project.

ARTHUR JAFFEY As Charles Coryell mentioned, many of us who came in the early days didn't know beans about radiochemistry, and certainly not about plutonium. Most of us hadn't had experience in radiochemical separation techniques of any sort. I spent the first month learning how to shift plutonium ions from the lower to the upper state, and trying to get rid of some of the fission products by precipitating them with lanthanum ions plus hydrofluoric acid.

*About one atom of ^{239}Pu is formed per atom of ^{235}U undergoing fission and fission products are formed along with the plutonium. The fission products include highly radioactive isotopes of several elements. Their presence complicates the separation and recovery of plutonium from uranium since the operation must be performed remotely from behind thick shielding to protect the technicians from the radiation.



Above is a cutaway model of the West Stands of Stagg Field showing the first reactor in the squash court beneath it. Below is a photograph of the reactor taken during construction.



In the midst of these training exercises, we got a shipment of uranium nitrate hexahydrate which had been bombarded in a cyclotron. This was the first shipment we got, and I think it came from Berkeley. (The later ones all came from the Washington University cyclotron in St. Louis.) This shipment had been packaged in little plywood boxes, out of whose joints the material was creeping. The standards of packaging then were quite relaxed, on a level the AEC would never accept today. The boxes were of all shapes and sizes to fit around the cyclotron snout with its neutron source. We had an estimate from the bombardment history that there were probably 100 micrograms of plutonium in this batch.

There we were confronted with something like 200 pounds of uranium nitrate hexahydrate and no place to work it up. The laboratory that we had been using was the university's large inorganic chemistry room on the fourth floor. But it wasn't large enough for the mining operation we had before us. Someone in the chemistry department proposed the old attic junk room, which was on the same floor. It was euphemistically called the storage area. All the equipment that had ever been used in research, and that no one wanted to throw out, was up there. We found stuff that dated way, way back to a time long before the building was built. And there were a lot of old crates lying around. We were told that if we could work around equipment and crates, we were welcome to the room.

Then we had to find equipment to use for a large-scale extraction. The first problem was to concentrate the plutonium from the uranium; an ether extraction seemed the only practical means for this. We snooped around the building and found a stock room with lots of goodies. Again we were very thankful that nothing was ever thrown away in this chemistry department. Somebody in days of yore had bought a quantity of large separatory funnels, 1 and 2 liter ones. At that time nobody did research requiring such funnels, but here they were, and we used them.

We placed uranium nitrate crystals in these separatory funnels, added ether, and shook them violently in front of our bodies with one finger on the glass stopper and another on the stopcock to prevent spillage. Seaborg gave us as many men as he could spare from the other work, and it was quite a sight to see the gang of us in that old attic shaking those funnels. The operation required keeping the funnel close to the body, and it was recognized that this could lead to sizable gamma-ray exposure from the fission products. No dosimeters were available (we would probably have been frightened by their off-scale readings if we'd had them), so Dr. Nickson, our medical man, had daily

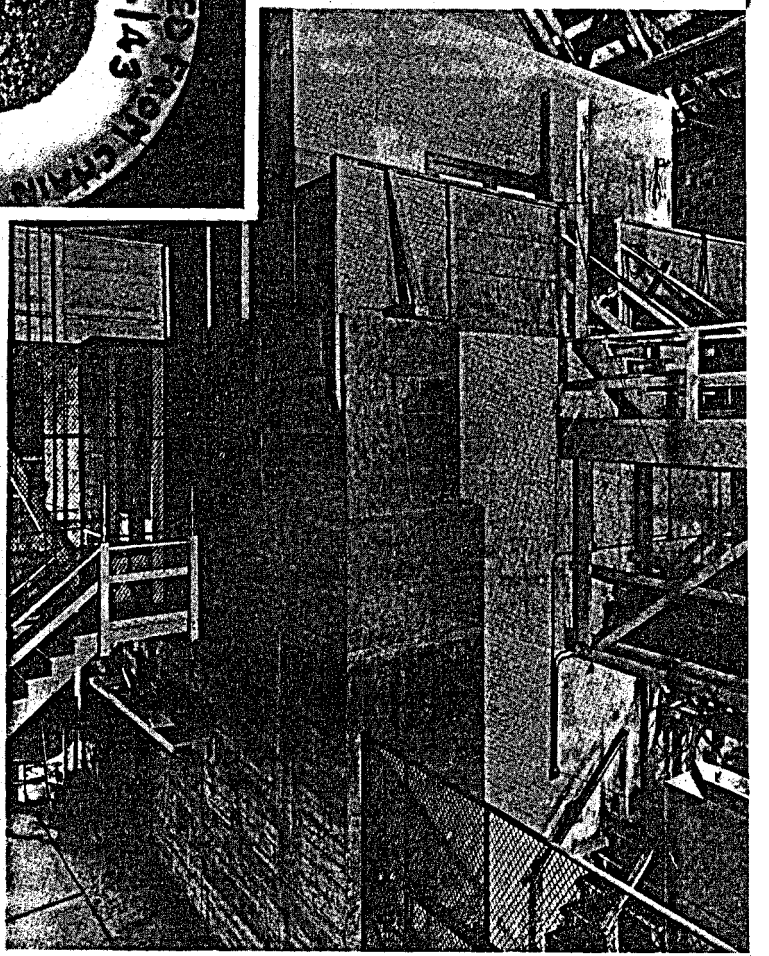
white blood corpuscle counts taken of those doing the separation. In addition, the separation job was rotated among members of Seaborg's section.

Part of the hydration water from the uranium nitrate hexahydrate settled out to the bottom of the funnel, and with it came the plutonium, together with many of the fission products. We separated the water phase and saved it, and dumped the ether phase (with most of the uranium) into 5-gallon carboys, and let somebody else take care of that.

But we still had to handle the water phase; since the separation factor of the ether extraction was only 90% (that is, 90% of the uranium went into the ether phase), we still had 10% left. While each separatory funnel didn't yield much water phase, all the many separations yielded quite a bit of solution and a lot of uranium, I suppose something on the order of 20 pounds, still too much for desk top chemistry. So we had to start over again to separate the plutonium from a quantity of uranium.

We didn't know any other way of doing it as efficiently, so we had to do another ether extraction. Since this meant starting with uranium nitrate hexahydrate crystals, we had to evaporate the water solution. Here again, we had to do it on a big scale. We didn't have hoods, but we did have an unknown benefactor who had left us big evaporating dishes about 24 to 30 inches in diameter. Then we found that there was a little open roof area outside the attic. It was August and balmy, so we just set up some hot plates on a table on the roof and started evaporating. This was as good as a hood since the wind blew away the fumes.

But we did have one serious problem. We had to know when to stop evaporating. The solution temperature was around 80 to 90 degrees centigrade, but we couldn't get the uranium nitrate hexahydrate crystals until we allowed the whole solution to cool. This was tedious because the entire mass had to be vigorously stirred as the solution cooled and the crystals settled out. If we allowed it to cool after having evaporated too much water we would get a lower hydrate than the hexahydrate, and this wouldn't dissolve in the ether. If we left too much water in the solution, then the crystals would come out, but they would be wet and we would get too large a water phase in the ether extraction. This would carry too much uranium with the plutonium. We didn't have the time or the facilities to do an accurate calibration of the necessary density, but we discovered empirically a method that worked as well. Little porcelain pieces from a small smashed evaporating dish were being used as boiling stones. We found that if we lifted



On the left is the first pure plutonium (about 30 millionths of a gram) produced in the CP-2 reactor (below) near the present site of the Argonne National Laboratory in Illinois on October 12, 1943. The photograph was taken through a microscope. The sliver is less than a half inch long.

one of the pieces to the top of the solution and it took 10 seconds to fall from top to bottom, then that was the right concentration. (I've forgotten the exact time, but it was around 10 seconds.) The crystals formed were uranium nitrate hexahydrate; they weren't wet and were soluble in ether.

We allowed the hexahydrate to crystallize out, and did the ether extraction over again; then we had our solution of plutonium with only a few pounds of uranium nitrate.

I think that Perlman lays too much blame upon himself for having lost the entire stock. As I recall, we split the final water phase into two or more portions (there were at least two portions). It was Seaborg's intuition that a beaker or a centrifuge might break, and he was right. Although we desperately wanted the material that had been lost from that broken beaker, we still had the ace-in-the-hole of that other portion, and it was this plutonium that went into the supply which Burris Cunningham and co-workers worked on later.

GLENN T. SEABORG I have the impression in retrospect that not all these problems were called to my attention at the time.

I should say that although we didn't succeed in getting Professor Benedetti Pichler to come to Chicago, as I indicated earlier, we did succeed in luring Paul Kirk. I would like to recognize the contributions that he made to the ultramicrochemical investigations with plutonium in the subsequent stages of the project.

Now I would like to call on John Willard who came with us from the University of Wisconsin and who played one of the leading roles in the leadership of the plutonium section of the Metallurgical Laboratory.

JOHN E. WILLARD We all know that we are commemorating an occasion which had its origins in Glenn Seaborg, that he was responsible for bringing many of us here 25 years ago, and that his genius and foresight were responsible for catalyzing the best efforts of all associated with him.

As I think back to those years, some of his methods of doing this appear in a sharper, though perhaps apocryphal, light.

There was the initiation ceremony. For me, having been accustomed to working with nothing smaller than a vacuum line, this took the form of being presented with 1 microgram of plutonium with the suggestion that I work out a method for separating a kilogram a day from 10^5 curies of fission products—and do this within a week or two.

Then there was the immunization process of daily lunches at a certain 55th Street restaurant to which members of the group were subjected, apparently on the theory that if they survived there would be no danger of losing them to illness at some future time of crisis.

Glenn anticipated by two decades the value of the telephone-booth-packing craze of a few years ago by use of his cubbyhole office to hold group meetings. This developed esprit des corps. It was here that heated and exciting discussions of decontamination, scavenging, and waste storage occurred.

But Glenn had his human side. I remember that when he came back from his first trip to Oak Ridge to see the industrial separations plant designed to scale up to 1 gram per day from the microgram laboratory scale, his first remark was, "I felt positively irresponsible".

He also had his human side in other ways—as when he left suddenly on a business trip to California and came back with a bride.

And there was the time he took an evening off to go down to a radio studio as a guest of the Quiz Kids—and casually announced two new elements to the world.

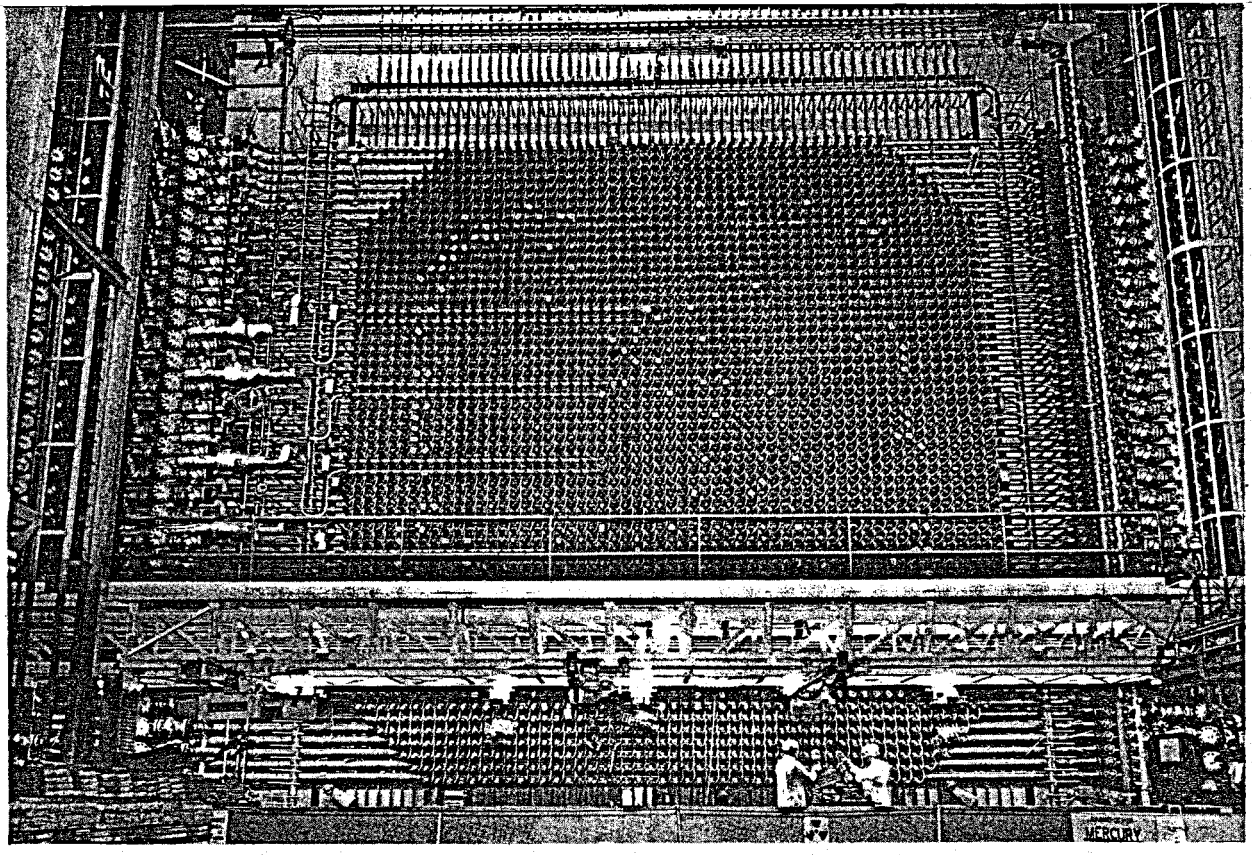
Also there was his scientific hobby with which he liked to refresh colleagues who showed signs of going stale. This consisted of late afternoon investigations of the properties of small white balls with respect to potential wells to be found in the grass of the Jackson Park golf course.

Another aspect of the Plutonium Project was the outstanding success that Glenn and the other section chiefs had in impressing the purchasing office with the importance of meeting the scientists' every request instantly. This was demonstrated when a harried purchasing agent called a scientist, who had just put in a rush order for a platinum boat, to ask, "Do you really want a PT boat?"

GLENN T. SEABORG I thought for sure, John, you were also going to remark on the requirement that a fellow ought to be able to go out on the golf course on short notice at almost any time.

Before concluding, I want to make special mention of the contributions of the leaders of our project, Arthur Holly Compton, and those who worked with him such as Norm Hilberry and Dick Doan.

During this 25th anniversary of the first weighing of plutonium we have looked back at some of the events that helped to begin the Nuclear Age. I hope that during the next 25 years we will see that age becoming one of peace and plenty for all men, and that plutonium will prove to be a major asset in helping us to achieve that universally desirable end.



Face of a plutonium-production reactor at the Hanford Plant in Richland, Washington.

PHOTO CREDITS

Page	
1	Lawrence Radiation Laboratory (LRL)
iii	Argonne National Laboratory (ANL)
7	LRL
11	LRL
15	LRL
21	ANL
24	ANL