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REMEMBERING THE EARLY DAYS OF THE MET LAB

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Historians of the nuclear age have for the most part focused their attention on the development of the atom bomb. The discovery of nuclear fission, the successful achievement of a controlled nuclear chain reaction, and the design and detonation of nuclear weapons. This is not at all surprising, considering the role that nuclear weapons have played during the second half of the twentieth century. However, from the long term historical point of view, the military aspects of nuclear energy may very well turn out in the long run to be among the less important consequences of the successful liberation of nuclear energy. The recent adjournment of the Cold War, if, as we all hope, it becomes permanent, may at long last remove the constraints that have prevented nuclear energy from being put to work.

It must be realized that between the laboratory demonstration of nuclear fission, and nuclear energy as we know it today, enormous scientific gaps had to be filled. One of the earliest of such efforts, and perhaps the most important in terms of its long term potentialities, was to develop practical methods to produce on the large industrial scale the then newly discovered man-made element plutonium.

The venue for this effort was the Metallurgical Laboratory at the University of Chicago. The circumstances attending the creation of the Metallurgical Laboratory are described by Smyth in the famous Smyth report and by Rhodes in his marvelous *The Making of the Atomic Bomb*. The Met Lab was set up by the war-time Manhattan District, U.S. Corp of Engineers to i) find a system using normal uranium in which a chain reaction would occur; ii) to show that if such a chain reaction did occur, it would be possible to separate plutonium chemically from the uranium matrix and the fission products formed in the chain reaction; and iii) to prepare plans for the large-scale production of plutonium.

Chemistry Section C-1 of the Met Lab was assigned the responsibility for developing separation methods for plutonium production on the industrial scale. This was a problem of extraordinary complexity. Not only was plutonium radioactive and toxic, but it was accompanied by

a host of highly radioactive fission products comprising a sizeable fraction of the Periodic Table. For its anticipated uses, it was essential to reduce the concentrations of these and of low atomic weight impurities to very low levels. To succeed, it was essential to have a clear and thorough understanding of the chemical properties of the new element plutonium, which had never before been seen by the eye of man. In short the chemical research would have to be carried out on an element of largely unknown chemical properties, in a lethal environment, and under the tremendous pressures imposed by the war.

The research could not be only an exercise in pure chemistry, important as this was. The industrial process for plutonium production would have few precedents. The gram amounts of plutonium produced in a nuclear reactor would have to be separated from tons of uranium accompanied by such huge amounts of radioactive contaminants that only chemical reactions and operations that could be carried out by remote control could be considered. These practical problems were to be solved by engineers, but the unusual demands on the process imposed serious constraints on the chemical laboratory research. Whichever process was developed in the laboratory, it would have to work perfectly the first time it was used. For the chemists, it would be a little bit like improvising at the piano wearing gloves (in our case, actually rubber gloves).

It is not my intention to discuss scientific minutiae here. Rather, I will try to describe some aspects of daily life in Section C-1 as experienced by one of the workers. Since I never had any administrative responsibilities, I knew little of what went on at the higher levels. If Wagner had written an opera about the Met Lab, I would have been cast as a Nibelung. Consequently, the view here is distinctly from below.

What makes it possible to resurrect the past of Section C-1 is that it is one of the best documented scientific enterprises in the history of science. G.T. Seaborg was an assiduous note taker, and he clearly had a strong sense of history. He kept a daily journal, and thus there exists a record of the activities of Section C-1 for every day between April 19, 1942 and May 15, 1946. These records are assembled in 4 volumes, each approximately the size of a Chicago telephone directory.

I do not know of any other scientific inquiry which has been recorded in such detail. It is as much of interest for the inferences that can be drawn from its more than 2500 pages as for its overt contents. It must surely one day attract the attention of historians of science. In any event, it makes it possible to resurrect the past with reasonable accuracy.

Although GTS was only thirty years old when he assumed his new responsibilities in 1942, he was already an experienced researcher. In addition to numerous other radionuclides, he and his small group of collaborators had discovered plutonium in the Berkeley Radiation Laboratory in 1940, and had begun to study its chemical properties. A description of Glenn Seaborg's research in the Berkeley Rad Lab can be found in Martin Kamen's fascinating memoir *Radiant Science. Dark Politics.* GTS was thus the logical choice to head up the chemical research program in the Met Lab. His first priority was to assemble a staff. He gave this a high priority. I believe it is fair to say that GTS was personally involved in the selection of every scientist, technician, and secretary who joined the Section.

In the early days of the war, recruiting scientists was by no means a simple matter. There were many competing research programs, and the extreme secrecy in which the Manhattan Project operated made it impossible to tell prospective candidates for employment just what sort of research they would be engaged in. Further, inorganic chemists were in very short supply. Inorganic chemistry before World War II was not a particularly flourishing branch of chemistry, and practitioners were scarce.

Consequently, GTS was more interested in the personal characteristics of candidates than in their academic training. Adaptability was perhaps the most important qualification. As a result the staff was extremely heterogeneous. There was a sizeable population of retreaded organic chemists, of whom I was one. There were two mammalian physiologists, an entomologist, a limnologist, am oceanographer, an expert in forensic chemistry, biochemists, physical chemists, at least one electrical engineer, and an expert in photography. There was a large population recruited from colleges and universities. There was even a properly qualified inorganic chemist. There was a core of

radiochemists who came with GTS from California, but even these had learned their craft on the job.

Because much of the research with plutonium was carried out on the ultramicro scale, people who had practiced microchemistry were in great demand. The entomologist was a specialist in the kidney pigments of mosquitoes, and was very good at removing a kidney from a mosquito, extracting the pigments and separating them by chromatography. The forensic chemist was also experienced in working with very small amounts and was a recognized expert in ultramicro chemistry. These skills were soon disseminated throughout the Section.

There were of course a few recruits who turned out not to be receptive or adaptable to new demands. There was also an occasional nay sayer who knew that we were destined to failure. Because the Section management followed the work of everyone very closely, these would soon be identified. There were, as far as I know, no reproaches or ultimata. There might be a reassignment. If this failed, such a person would soon discover that a new staff member across the hall was working on the same problem, and the square pegs would fade from the picture.

I happened to be one of the few members of the Section who had actually worked with uranium. Prior to joining GTS's Section, I was a member of a small group assembled by H.I. Schlesinger and H.C. Brown to prepare volatile compounds of uranium that would be suitable for the separation of the uranium isotopes. In the course of this work, I learned something about uranium chemistry. The Volatility Project did some very important chemical research, but none of the compounds we synthesized turned out to be useful for isotope separation, and the group disbanded.

Acting on the advice of a friend who was already a member of Section C-1, I introduced myself to GTS, and was given an appointment. I thought for a long time that this was due to the good impression I had made during the interview. On reading the Seaborg *History of the Met Lab Section C-1*, I found that GTS was well aware of the Volatility Project, and that I had been slated to become a member of the Section well before the group disbanded.

I have indicated above that the progress of the research in Section C-1 was followed very closely. An internal review process was always under way. This took the form of regularly scheduled

meetings. The entire scientific staff of Section C-1 met at least once a week. There were also weekly group meetings, and there was a regularly scheduled meeting of the Section council, which consisted of senior scientists of the staff. All of the meetings were held in the evening after hours, or early in the morning before work started. As we worked a six day week, additional meetings early Saturday morning were not uncommon.

There were also general Met Lab meetings, generally held in the evening. These were particularly interesting from a sociological point of view. In the Met Lab, the physicists were the lords of creation. This was not surprising, as people the likes of Enrico Fermi, Eugene Wigner, Leo Szillard, and Arthur Holly Compton, to name but a few, were involved. The meetings, it seemed to me, were pretty well monopolized by physicists and physics problems. Some of physicists had very curious ideas about what chemists did. It became apparent at one meeting that at least one physicist had the notion that extracting plutonium from irradiated uranium metal was something like scraping the skin off a carrot. On another occasion, after Eugene Wigner had given a disquisition on process chemistry, I came up to him after the meeting was adjourned, and asked him a question that indicated that I had some problems with his chemistry. His reply to the question, delivered ever so politely, was "Young man, I have a Ph.D. too". I lost the argument, but I learned not to ask questions at meetings.

At the Section meetings, the lower orders were by no means confined to a passive role. Chemists working at the bench were frequently called on to describe their progress and their future plans. All of this made it possible for the management of the Section to keep in close contact with the progress of the research. It served to prevent scientists from being bogged down without anyone knowing about it, but it also made for a highly competitive environment.

The frequent meetings and the close monitoring of the research made it possible to plan ahead in an informed way, and planning ahead was one of the distinctive features of Section C-1. The planning of research was very effective. Although the principal charge to the Section was a separation process for plutonium that was practical on the large scale, the basic chemistry of

plutonium was by no means ignored. Assigned an importance at least equal to and probably greater than the process development were investigations on the nuclear and chemical properties of plutonium: nuclear properties, oxidation states, redox potentials, ions in solution, thermochemistry, metal preparation, solid state chemistry, all received attention.

The basic research on the chemistry of the new element plutonium proved very successful. How successful can be judged by comparison to the history of the closely related element uranium. Uranium was discovered in 1789, plutonium in 1940. In a five year period almost as much about the basic nuclear and chemical properties of plutonium was discovered as had been learned about uranium in 150 years. The power of organized research, sometimes disparaged as Big Science, should not be underestimated.

In the process work, there was an unstated determination to explore all possible approaches to the separation of plutonium. Although it became evident quite early that a conservative precipitation process would become the method of choice for the large scale production of plutonium, other possible approaches were not ignored. Work on volatility process was started almost from the beginning. Solvent extraction (partition between two immiscible liquids), adsorption, ion exchange, and a wide range of precipitation processes received attention. In November, 1943, GTS listed 17 possible approaches to plutonium extraction and decontamination, most of which were under study. Practically every technical advance in plutonium technology since the war had its prototype in Section C-1.

My own experience of the Met Lab began in March, 1943. At that time, Section C-1 was housed in a new building on the campus of the University of Chicago. The building had a pleasant rustic appearance in contrast to the massive academic Gothic structures on the campus. The entrance to New Chem was manned by a platoon of guards. Because the building was under negative air pressure to prevent inadvertent leakage of radioactivity to the outside, entrance to the laboratories was through an air lock. All of the windows in the building were covered with white paint, so that once inside one was effectively cut off from the outside world.

The laboratories were conventional in the style of the time, and lacked any of the features that would distinguish a present day facility that handles radioactive materials. My first assignment in the Met Lab was to investigate a volatility method for the separation of uranium, plutonium and fission products, the mixture produced by the chain reaction in a nuclear reactor. It was conceptually possible to effect a separation by taking advantage of the differences in volatility of the fluorides of uranium, plutonium and the fission products.

In those days, the techniques for handling fluorine, which is one of the most reactive substances known, could at best be described as primitive. Plutonium itself was not as yet available for chemical research, and it was therefore necessary to do the experiments with uranium-232 and plutonium-238 as tracers. The experiments were encouraging, but progress was painfully slow, and it became evident that it was unlikely that anything that would be useful to the war effort was likely to emerge from the work. Years later, as a matter of honor, the late H.H. Hyman and I found a practical way (by the use of the liquid halogen fluoride bromine trifluoride rather than elemental fluorine) to convert a mixture of uranium, plutonium and fission product to their fluorides, and to separate them by fractional distillation. This process, unfortunately, became an early victim of nuclear technology immobilization.

As soon as plutonium-239 became available in milligram amounts, I was put to work preparing and characterizing the halides of plutonium. To minimize the hazards of plutonium toxicity, the experiments were carried out using microchmical techniques, and the products were characterized by X-ray crystallography. By now it was realized that enclosed systems were necessary to avoid dissemination and ingestion of plutonium. Closed walk-in cubicles had been constructed containing vacuum-line equipment in advance so that work could begin when plutonium became available. The cubicles prevented scattering of plutonium throughout the laboratories, but it required unceasing vigilance on the part of the experimenter to avoid accidents. For a while I shared a cubicle with Isabella Karle, which remains among the most pleasant of my recollections about New Chem.

In the winter of 1944, I was instructed by GTS to take a small group consisting of Larry Magnuson, Ted LaChappele, Walter Beard and myself to Oak Ridge to isolate the first macroscopic amounts of the long-lived isotope neptunium-237. Why I was chosen for this task was a mystery to me as I had little first hand experience with the wet chemistry to be used for the separation. The Oak Ridge pilot plant was now operational, and several special processing runs were scheduled for the neptunium recovery.

Since only a few milligrams of ^{237}Np would be present in a large amount of plutonium, it was decided to prepare a large amount of the short-lived ^{239}Np that would be added to the starting material to serve as a tracer for neptunium during the isolation procedures. We thereupon proceeded to prepare one curie of neptunium-239. We worked in room on the second floor under the West Stands of Stagg Field, where Fermi and his co-workers had constructed the first nuclear reactor. We had constructed a primitive cave of lead bricks that stood on a table in the middle of the room. The structure was in such a state of repair that inspectors came every morning to check for new cracks in the floor and walls. We were under strict instructions not to bring in even one additional lead brick into the room for fear of collapsing the floor.

We carried out the chemical reactions required to separate the meptunium-239 form cyclotron-irradiated uranyl nitrate wearing rubber gloves with our arms extended over the wall of lead bricks. I had the advantage of having short arms, so my exposure was minimized. The hood containing the centrifuge was along the wall, so the centrifugations, which were required from time to time, made it necessary to transfer the sample from the cave in the center of the room to the hood along the wall. Carrying the centrifuge tube once from the cave to the centrifuge resulted in a radiation dose considerably in excess of the then permitted daily dose, a radiation dose which would now be considered criminal. We took turns, and one transfer finished you for the day. The curie of neptunium-239 was then shipped by military courier to Oak Ridge. As ^{239}Np undergoes radioactive decay with a half-life of 2.3 days, we could not afford to dawdle.

We proceeded to Oak Ridge by train. For me it was a great adventure. I had spent the whole of my life up to then in Michigan and Illinois. When the train crossed the Ohio River, it was night. When it made its first stop, I looked out the window, saw a small sign, and announced to my companions that we were now in White, Kentucky. The trip was to be truly educational.

One morning, well before dawn, I was taken by car to the Oak Ridge pilot plant in the X-10 area where the plutonium pilot plant was situated. It was a frosty morning. I was a committed reader of science fiction at that time and the X-10 area looked other worldly to me in the dark. I climbed on to the roof of the separations plant, one of the engineers opened a valve, and I poured the neptunium-237 tracer into a pipe connected to the dissolver unit. Some hours later, we were handed a dark green, viscid solution containing the neptunium-237 and 17 grams of plutonium.

The laboratory to which we were assigned was not equipped with enclosed cubicles or any of the special facilities that were already in use at Chicago for handling plutonium. It was hard to be casual about the 17 grams of plutonium. However, our ^{239}Np was decaying at a rapid rate, and so we went ahead, using the fume hoods when possible, otherwise using open beakers out on the desk tops. We carried out successive oxidation-reduction cycles to make the separations. One step required the conversion of Pu (IV) and ^{237}Np (IV) fluorides to their hydroxides by addition of a sodium hydroxide solution. For this we used a 2 liter beaker out on the desk top. One of my colleagues stirred and I poured. The stirrer got enthusiastic and I suddenly felt a burning sensation on my lip. It was not possible to decide whether the drop came from the sodium hydroxide solution (unpleasant but otherwise harmless) or from the solution containing the 17 grams of plutonium. We did not consider it either prudent or essential to notify the authorities. I swabbed my lip with Kleenex and we continued with our operations.

After several days of intensive work, we succeeded in obtaining a very pure ^{237}Np preparation. After some conversations with Chicago we discovered that our efforts to remove all of the plutonium from the neptunium had been misguided. Plutonium was then in great demand for research, and not easy to obtain. We therefore added the largest amount of plutonium to the

neptunium sample that we thought would not raise questions, and headed back to Chicago. When we left, every surface in the lab in which we had worked was covered with a thin film of plutonium.

People worked hard in Section C-1, as I imagine they did everywhere in the Manhattan Project. The work came first. I can give a personal example. In Volume III of the Seaborg compendium I have found the following entry, dated Sunday, October 1, 1944: "I was told that Joe Katz and Sonia Weiner, Thorfin Hogness'[the director of the Chemistry Division] secretary are being married today in Grand Rapids, Michigan. He will be back to work tomorrow."

I knew in the Met Lab that we were doing important work, but I had no conception of its far-reaching implications. It was an experience that made a deep impression on me that affected all of my future scientific life. This may well have been the case for most of the people who participated in the work of Section C-1. We were certainly not unaware of the possible consequences of our research. But we were also in mortal fear of Hitler in possession of an atom bomb. A vivid and perceptive description of the psychological atmosphere at this somber time can be found in Richard Rhodes' *The Making of the Atomic Bomb*. Today there are some who think that only people with a "genocidal mentality" could have carried out this work. We did not make the sacrifices that people in the military did, but we worked toward the same goals. Today we can look forward with some optimism to the time when the world finally dispenses with weapons of mass destruction, and to a day when the inexhaustible source of energy latent in plutonium can be put to use for the benefit of all mankind.

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