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D. W. Pearce
METALLURGY AND CONTROL DIVISION

A REPORT ON A TRIP TO STUDY THE MASS SPECTROMETER AND ITS USEFULNESS AS AN ANALYTICAL TOOL

Introduction

The writer visited, in a two weeks tour, the laboratories of A. O. Nier in the physics department of the University of Minnesota, of A. J. Dempster and Harold C. Urey at the University of Chicago, and the laboratories at K-25, Y-12, and X-10 at Oak Ridge, Tennessee. Nier, Dempster and Urey are men whose names have been associated closely with the development of the mass spectrometer and Nier and Dempster have each designed and constructed many instruments. At Oak Ridge a large number of mass spectrometers are used as analytical instruments for the routine control of the respective plant processes in K-25 and Y-12. The instruments in use at Oak Ridge are of particular interest to the Hanford Works because they were designed for the separation of heavy masses, namely U^{235} and U^{238}.

On this tour, the writer paid particular attention to the types of mass spectrometers in use at the various places, the specific merits of each type, their applicability to analytical problems, their space and staff requirements, and the commercial availability of the instruments or their components. The time spent at the various laboratories was distributed as follows:

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1. November 8, 9 and 10; the laboratories of A. O. Nier in the Physics Building at the University of Minnesota, Minneapolis, Minnesota.

2. November 12; the laboratories of A. J. Dempster and H. C. Urey at the University of Chicago, Chicago, Illinois.

3. November 15, 16 and 19; the chemistry and physics laboratories at K-25, Oak Ridge, Tennessee.

4. November 17; at X-10, Oak Ridge, Tennessee.

5. November 18; at Y-12, Oak Ridge, Tennessee

In addition to the mass spectrometers at Oak Ridge, the writer was shown other laboratories at K-25, the Redox control laboratories, Redox pilot plant, and pile building at X-10 and laboratories and production facilities at Y-12.

Some samples of Hanford metal waste solutions have been submitted to K-25 for isotopic analysis. These samples arrived during the time that the writer was at Oak Ridge and the proposed analyses were discussed with the men concerned at X-10 and K-25.

<u>Basic Principles of the Mass Spectrometer</u> *

"A mass spectrometer is essentially a very special vacuum tube through which a
gas to be investigated is continuously flushed at a low pressure. By bombarding
this gas with a beam of electrons, positive ions are formed, which, if sent
through an appropriate combination of electric and magnetic fields will be
separated according to the respective masses of the ions formed. Thus, if a
complex mixture of gases having different molecular weights is introduced, ions
of different masses are produced, separated in the magnetic field and the
individual ion currents measured. Thus, an analysis of the gas is achieved."

"Figure 1 shows a schematic view of a typical mass spectrometer. The ion source
consists of an electron gun in conjunction with a suitable combination of plates
which serve to draw the ions from the electron beam where they are formed into an
ionic lens for focusing and acceleration before they are sent into a magnetic
analyzer to be separated according to mass." A charged particle entering a
magnetic field is acted on by a force at right angles to its direction of motion
and proportional to its velocity. The charged particle is accordingly forced to
follow a circular path while in the magnetic field. In the type of apparatus
shown, all ions fall through the same difference of potential in the source and

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* See "The Mass Spectrometer as an Industrial Tool", Alfred O. Nier, Proceedings of the National Electronics Conference, Vol. 2, 1946.

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hence acquire the same kinetic energy, $\frac{1}{2} mv^2$, where m is the mass and v is the velocity. A detailed analysis of the system by means of electrostatics and electromagnetics leads to the equation:

$$R^2 = \frac{20,720\, mV}{eB^2}$$

where R = radius of curvature of the particle path in the magnetic field (in centimeters),

m = mass of the particle in atomic weight units,

V = the difference in potential in volts through which the ions fall in the accelerator,

e = the charge of the ion in terms of the number of electrons removed,

B = strength of the magnetic field in gauss.

In practice, the radius of curvature, R, is fixed by the design of the spectrometer tube. Nier usually uses a 6 inch radius of curvature through 60°. A particular mass is focused upon the slit in the first collector plate by selecting the proper ratio of accelerating voltage, V, and magnetic field strength, B. A doubly charged particle (e = 2) will appear to have a mass of one-half that of the same particle having a single charge (e = 1). A typical ion source is shown in Figure 2, and a typical ion collector in Figure 3.

In practice, a scan of a range of masses is usually made by using a fixed accelerating voltage and varying the magnetic field, B, by changing the current in the coils. On some instruments, a permanent type magnet is used and the voltage is varied for scanning. It is not necessary to know the magnetic field strength since the instrument is easily calibrated in terms of voltage, magnet current, and mass number. A typical scan over the range of the mercury lines is given in Figure 4. Such a scan can be made by feeding the output of the amplifier from collector plate #2 to a Brown recorder and varying the magnet current at a constant and known rate with a constant speed motor on a helipot. This curve was taken from the manual of the G.E. Assay Machine (Cat. No. 6933969 G2) and is an indication of its resolving power. This is the control instrument used at Oak Ridge, Tennessee at both K-25 and Y-12. The resolution of a mass spectrometer is a measure of its ability to separate neighboring mass numbers. It is expressed as the ratio of the height of one of two adjacent peaks to the intervening valley height. For example, at a mass of 200, this G.E. equipment should give a resolution of at least 200.

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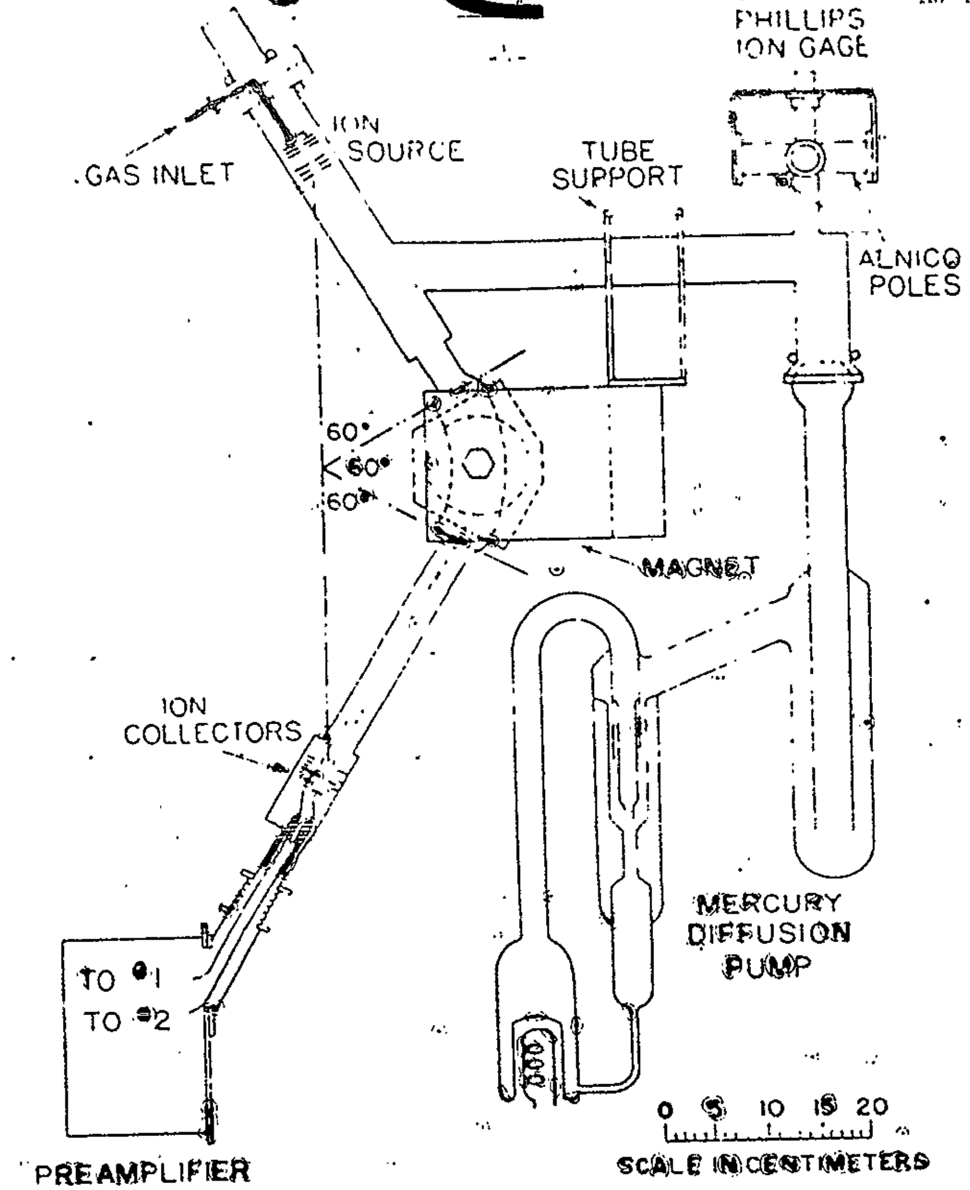

FIG. 1. Schematic drawing showing mass spectrometer tube, magnet, pressure gauge, and pumping system.

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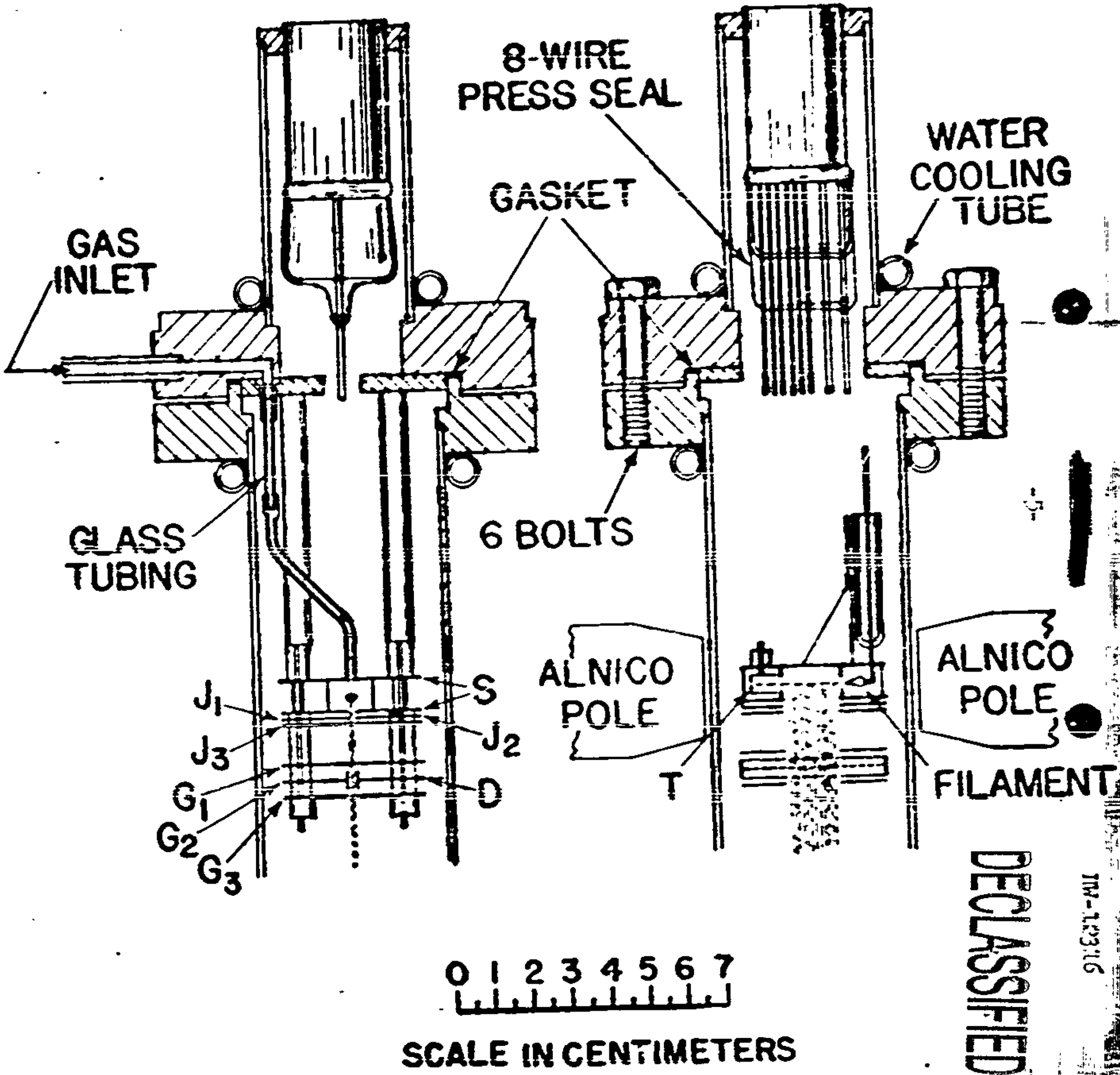

FIG. 2. Ion source. Filament made of tungsten ribbon, 0.75 mm wide and 0.025 mm thick. Electron beam defining slit $= 0.5 \times 2.0$ mm. Distance between filament and defining slit $= 1.0$ mm. Slit in lower plate of shield $S = 1.0 \times 14$ mm. Spacing between plates J_1 and $J_2 = 1.0$ mm. Slit in $J_3 = 1.0 \times 14$ mm. Slits in G_1 and $G_3 = 0.25 \times 14$, and 0.5×14 mm, respectively. Spacing between G_2 and D is 3 mm. The electron beam is 1.5 mm above the lower shield plate S. All plates made of Nichrome V 0.025″ thick.

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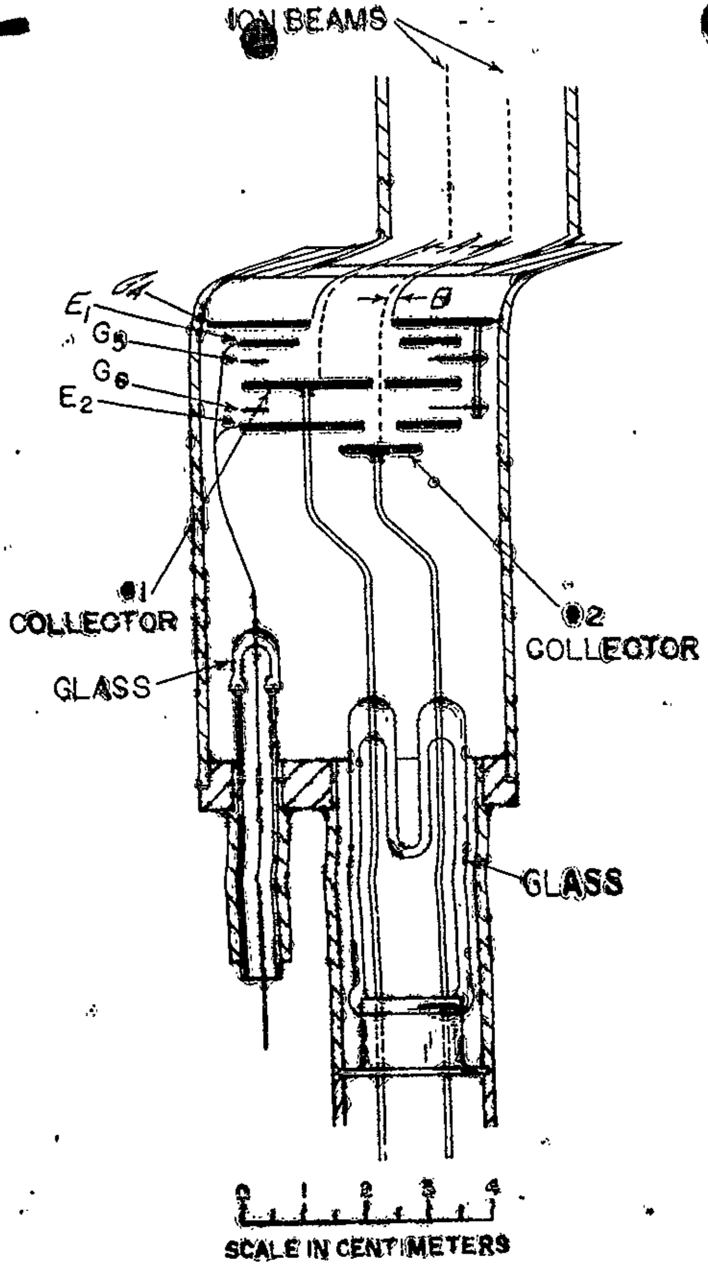

FIG. 3. Ion collector assembly. All joints are silver soldered and Kovar to glass seals are employed for bringing electrical leads into the housing. Distance A is 12.5 mm and B 1.6 mm. Slit in collector No. 1 is 1.25×14 mm. The opening defining the ion beam admitted to the collectors is square, so that when measured perpendicular to the paper it is 12.5 mm long. The various plates are supported by two posts using a construction similar to that employed in the source. Plates E_1 and E_2 operate at a potential of −45 volts in respect to ground. They serve to suppress secondary electrons emitted by ion bombardment. The openings in these plates are sufficiently large to prevent ions from striking them. G_5 and G_6 are grounded rings inserted between collector No. 1, E_1, and E_2, respectively. Their presence prevents ground leakage currents from E_1 and E_2 from reaching collector No. 1.

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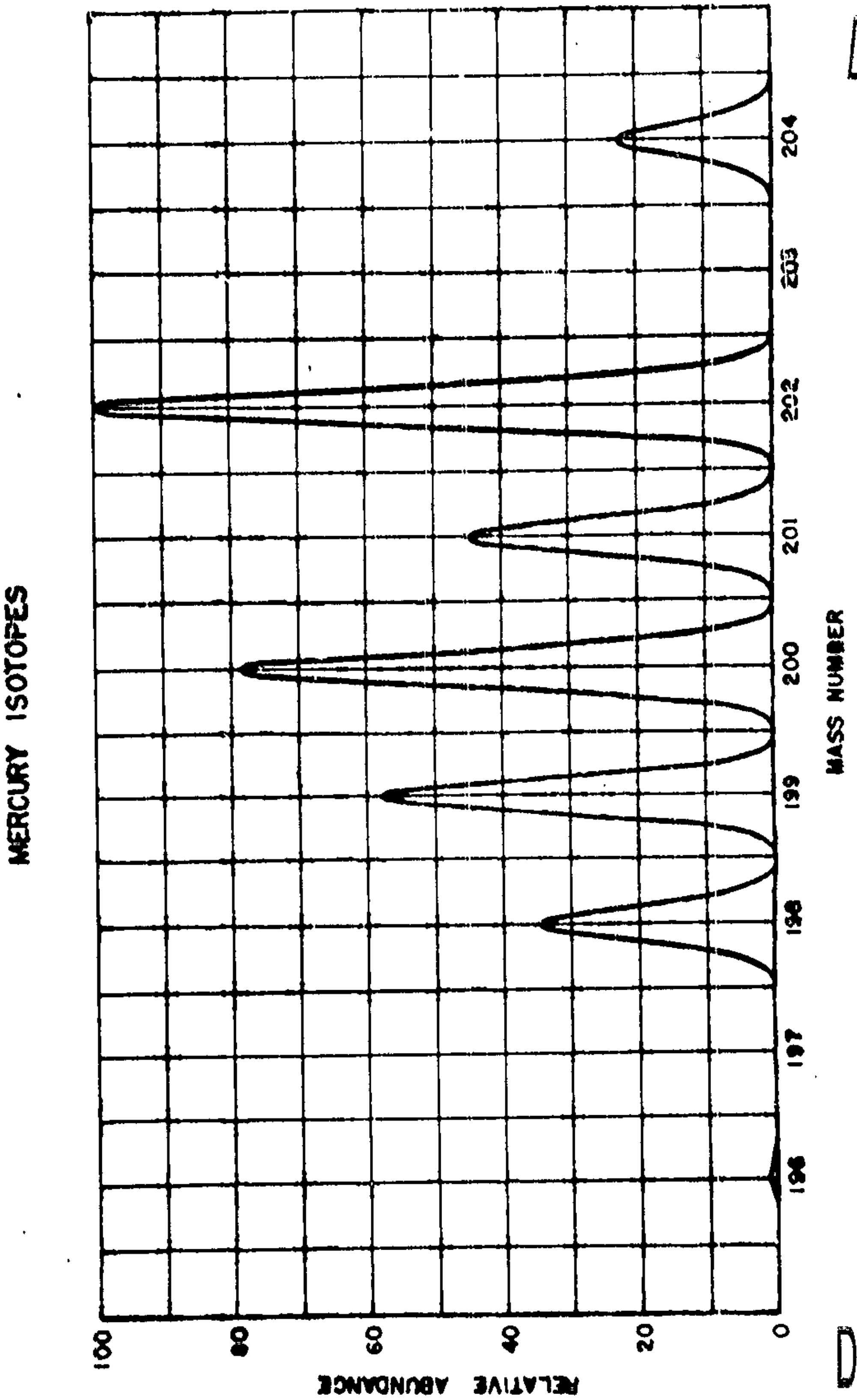

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The principles and diagrams shown above are basic although many mass spectrometers are built with larger or smaller angles of magnetic deflection and may have double focusing by using electrostatic deflection in addition to magnetic deflection. This is done by passing the ion beam between charged plates to deflect it through a given angle, e.g. 30°, before it enters the magnetic field. One of Aston's mass spectrographs used an electrostatic deflection of 1/6 radian and a magnetic deflection which depended on the mass. The collector was a photographic plate and the various masses produced a "spectrum" on the plate.

Double focusing is resorted to for work on atomic masses and packing fractions when very high resolution is essential. Photographic methods are undesirable in quantitative analysis and isotopic ratio work because of the need for accelerating voltages of about 20,000, correspondingly higher magnetic field strengths, greater ion currents, and the accurate measurement of line densities in order to obtain quantitative data. This is true because a rather large number of high energy ions are required to sensitize the photographic plate. Ions of such energies as 20,000 volts obviously require a correspondingly stronger magnetic field for resolution.

The Laboratories and Equipment of A. O. Nier

Dr. Nier, at the University of Minnesota, has been working in the field of mass spectrometry since about 1935 and has designed some of the best spectrometers for quantitative analysis in use at the present time. His chief interest has been in isotopic ratios and therefore his instruments are well suited for analytical work.

Dr. Nier has about eight mass spectrometers in operation in his laboratories. Many of them have been constructed for the analysis of gaseous mixtures of low molecular weight, e.g., abundance of C^{14} in CO_2 - air mixtures. Two of his instruments have larger magnets and are capable of good resolution of atomic masses above 200. Most of his instruments are the single focusing type using either a 60° or a 180° magnetic deflection and up to 4000 V. ion acceleration. He is now constructing a double focusing spectrometer which will have a 90° electrostatic deflection followed by a 60° magnetic deflection. He hopes to obtain magnetic and electrostatic fields stable enough to measure atomic masses and packing fractions using electrometer measurement of the ion beam. All previous work on atomic masses and packing fractions has been done with instruments using photographic detection of the ion beam.

The Nier ion collector is shown in Figure 3. The ion currents collected by the two collector plates are usually fed through identical amplifiers and the amplified current from collector #2 measured with a lamp and scale galvanometer. Frequantly, the signal from the #2 collector plate is led to a Brown Recorder and either the magnet current or accelerating voltage varied continuously to give a "scan" or "spectrum". Figure 4 represents such a scan of the mercury isotopes.

When the ratio of two constituents or the ratio of two isotopes is desired, the best precision is obtained by feeding a portion of the signal from the #1 collector to the input of the 2 amplifier through a standard resistance decade in such a manner as to oppose or balance out the #2 signal. The decade setting needed to restore the galvanometer reading to zero is proportional to the ratio of the two ion currents. This method is called the "total pull method". Calibration curves are obtained with known mixtures. Absolute measurement of the ratio of isotopes is possible but calibration with standards is necessary whenever different molecules are involved. This is necessary because the ionization efficiency varies for different substances; that is, the ratio of CH_4^+ and O_2^+ in the ion beam is not necessarily the same as the ratio of CH_4 and O_2 in the gas being analyzed.

Nier has converted his equipment to inconel mass spectrometer tubes with flanged fittings and aluminum metal gaskets. The only glass on the tubes themselves is in the press seals through which the electrical leads enter the tube. Kovar seals are used to join the glass portions to the inconel. The metal tubes are wrapped with heating wire for baking out when evacuating them. Nier works with a vacuum of about 2 x 10^{-7} mm. of mercury, obtained with a mercury diffusion pump. Whenever it is necessary to shut down and break vacuum on a tube, it is best to fill the tube with dry helium or nitrogen. The pump-out time is thereby kept to about 8 to 10 hours.

A machinist in the Physics Shop at the U. of Minnesota, Mr. [illegible] D. Thorness, builds all of the spectrometer tubes and ion sources for Nier and since the source is the most critical part of the instrument, he makes a variety of them for every tube, experimenting with electrode spacing, slits and other features. The writer inspected many of the sources and spoke with Thorness at length about tube and source design. Thorness does considerable machine work in his own shop and he would contract to build spectrometer parts for the Hanford Works. It would be highly desirable, however, to train a machinist for such work in our own shops because experimentation with special sources would be difficult without constant personal contact with the machinist.

Nier gave his opinion regarding several points which directly concern us at Hanford. He believes that a single focusing instrument like those built for Oak Ridge by General Electric (Assay Machine, Cat. No 5933949 G2) would be best for quantitative work and he saw no need for double focusing. Electrostatic deflection increases the complexity of the instrument greatly. He believes that the instrument will resolve adjacent masses in the region of 240 units. The analysis of solid samples of inorganic materials would be more tedious than gas analysis, but seems to Nier to be within the realm of possibility and to be worth an extended study. He suggested the use of micro furnaces in the source so that solid samples could be heated to the sublimation or boiling point and distilled directly into the electron gun. This method requires the breaking of the vacuum after each sample and therefore would permit only about one analysis per day. Nier felt that an adequate staff, including an electronics man and a machinist, could put a mass spectrometer into operation within a year.

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The Laboratories of A. J. Dempster and Harold C. Urey

At the University of Chicago, the author visited Mark Inghram and A. E. Shaw. Unfortunately, Dr. Dempster and Dr. Urey were not available. There are about 6 mass spectrometers and mass spectrographs (photographic type) in operation in Dempster's department. A couple of Dempster's earlier instruments are also still set up although they haven't been used for several years. Several of the instruments are single focusing with 60° magnetic deflection and current measurement with electrometers very similar to those in the Nier instruments. The original Nier instrument which General Electric used as a model for building the Assay Machine was in Inghram's laboratory. Inghram is doing considerable research with solid inorganic samples, both halides and oxides. He has used the Dempster spark source (on a Dempster spectrograph) but now uses a microfurnace or a heated filament. The heated filament method has proved to be suitable for many substances, especially rare earths, which undergo thermal ionization. Plutonium salts do this to some extent, but the efficiency is low. (At the Hanford Works, Garrison (Notebook HEW-225-T) experimented with this method in 1945). Inghram has used tantalum and beryl crucibles in his sources to vaporize solid samples and he stated that the rate of ionization can be controlled well enough by the crucible temperature. Inghram prefers vibrating reed electrometers instead of D.C. amplifiers for ion current measurement.

For the determination of isotopic masses and packing fractions, photographic methods are generally used because the constancy of the ion current and the density of the lines are not important but the distances between lines must be measured accurately. High velocity ions are needed to make a photographic image and therefore spectrographs always use accelerating voltages of about 20,000 to 30,000 volts. Dempster's spark source was devised for solid samples. Ions from a spark source enter the source region with a wide range of initial velocities (up to 4000 volts, according to Inghram). Therefore, a correspondingly higher accelerating voltage must be used to keep the relative range of energy of the ion beam small. Such high voltages are disadvantageous for obvious reasons. Another disadvantage of the spark source is that it is wasteful of sample; at Hanford there was a loss factor of about 10^{-7} with the spark source spectrograph (See the File of G. W. Struthers, September 21, 1945).

For quantitative analysis and isotopic ratio determinations, Inghram recommended a single focusing type of spectrometer with electrometer measurement of the ion currents. A Nier instrument would probably be better. Solid samples should be vaporized in heated crucibles within the source.

H. C. Urey has two Nier mass spectrometers for low molecular weight gas analysis. He is using them for the determination of the isotopic abundance of O^{18} in nature. He has been able to correlate the O^{18} - O^{16} ratio in calcium carbonate deposits with the temperature of formation of rocks and fossils.

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Visit to the Laboratories at Oak Ridge, Tennessee

At Oak Ridge, the writer spent three days at K-25 and one day at each of Y-12 and X-10. At K-25, F. W. Hurd discussed the problems involved in the handling, purification and isotopic analysis of the uranium in the Hanford metal waste samples which were shiped to K-25 in November. Plans were made to submit the samples to M. T. Kelley at X-10 for purification. The uranium nitrate extracted from them will be returned to K-25 for fission counting and isotopic analysis.

Angus Cameron guided me through the laboratories at K-25 and explained the mass spectrometry procedures in considerable detail. He described some of the differences between the instruments at K-25 and those at Y-12. He pointed out that although the instruments in use at Oak Ridge were obtained from General Electric Co. several changes in circuits and in tube construction were made at Oak Ridge. Most of these changes were minor adjustments necessary to put the instruments into satisfactory operation. An automatic switch was installed in each instrument which would turn the instrument off in case a leak occurred and the vacuum in the tube was lost. A large standard-taper glass joint was installed on the tube above the source to permit rapid access to the source for exchange of parts.

Lester Smith, a supervisor of spectrometer laboratories, showed me the instruments in the control laboratories and the sample preparation laboratories. I read the laboratory manual and watched a laboratorian run several routine samples from the diffusion plant.

The mass spectrometers used at Oak Ridge are G. E. Assay Machines. They have a typical Nier source (see Figure 2) with an adjustable leak for gaseous samples. The spectrometer tubes are copper and glass. All electrical leads enter the tube through glass press-seals on tungsten wire. The collector is essentially the same as that shown in Figure 3. An FP54 amplifier on collector plate #2 feeds into a sensitive galvanometer, while a 954 feedback amplifier on collector plate #1 is used to balance out the signal on #2 plate through a decade; it is thus a "null" method of comparing ion currents. The instrument was designed for application to a mass range of 40 to 400 atomic weight units. All measurements of U^{235} - U^{238} ratios are done by the "null" method, or a modified or "partial null" method, and all analyses are made by comparison with standards. Standards have been prepared at Oak Ridge from natural uranium, pure U^{238}, and very highly concentrated U^{235} (high Q product) of accurately known composition. The standards have been carefully checked and duplicated, and the entire range from 0-100% U^{235} is covered. Each mass spectrometer is used for only one narrow range of U^{235} concentration in order to avoid the errors due to the "memory effect" of the instruments, which causes any given uranium analysis to be affected by the composition of the previous sample. For the same reason, the samples must be compared to standards having nearly the same concentration. In routine operation, the "memory" error amounts to about 10% of the difference in the isotopic ratio between standard and sample. The "memory" can be eliminated by heating the source under vacuum but that is too troublesome for routine analysis.

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The samples come from the Diffusion Plant as UF_6 in nickel sample tubes fitted with Hoke-Phoenix packless valves (refrigerator valves). These sample tubes are connected to a manifold on the mass spectrometer along with a suitable standard. The manifold is kept under high vacuum and when the Hoke valve of a sample tube is opened, the UF_6 enters the maniford and then passes through an adjustable gas leak into the mass spectrometer source at a controllable rate. When isotopic measurements are completed, the residual UF_6 is drawn completely back into the sample tube by freezing it out with liquid nitrogen. The routine procedure is to run the sample first, scanning the peak of the $(U^{235}F_5)^+$ signal five times, then run the standard the same way, and finally, re-run the sample.

Samples of uranium oxide are converted to the fluoride for analysis by direct fluorination or by the cobalt trifluoride procedure. The latter is used at Y-12 and is suitable for samples of 100 - 200 mg. U_3O_8, whereas the former requires larger samples. Direct fluorination consists of treating U_3O_8 in a combustion tube under vacuum with pure dry fluorine gas at 1200°F. The apparatus is usually nickel or copper. The UF_6 passes out of the reaction tube in the gas stream and is condensed in a U-tube at dry ice slush temperature. The fluorine and oxygen exhaust gases are passed through a soda ash-salt tower before being expelled into a hood. Transfer of UF_6 from U-tube to sample tube is done by connecting the two together and warming the first while chilling the second in liquid nitrogen. The amount of UF_6 collected in a sample tube is determined by weighing.

The cobalt trifluoride method for conversion of U_3O_8 to UF_6, as used at Y-12, is carried out in an all-glass apparatus by reacting a mixture of finely divided U_3O_8 and CoF_3 at 350 - 400°C. The UF_6 evolved is frozen out in a U-tube immersed in liquid nitrogen. A glass manifold and glass sample tubes having a break-seal are used on the spectrometers at Y-12, but the techniques in the analysis of UF_6 are essentially the same as those at K-25 as described above.

I spent a day with Sabin and Thom in a spectrometer laboratory under the direction of Boardman. These men analyze special samples and do research on spectrometer equipment. I studied their two spectrometers in detail, obtained some circuits and drawings, and operated one of their instruments, measuring the "drop through", checking the focusing adjustments, and measuring the U^{235} - U^{238} ratio of a sample of UF_6 by the null method.

At X-10, M. T. Kelley conducted me through the analytical research laboratories and discussed many of the methods of analysis being developed for the Redox control laboratories. Ed Frederick, supervisor of Redox control, conducted me through his laboratories and then tours through the Redox pilot plant and the pile building were arranged. "Hot" runs were in progress in the pilot plant using slugs obtained from the Hanford piles.

The samples of waste solutions from 200-E and 200-W at Hanford arrived at K-25 on November 16. They were transported by truck to the X-10 pile building on Wednesday, November 17, where Frederick and I unpacked them that day. Frederick will probably be responsible for purifying the uranium and will probably submit it to K-25 as uranium nitrate. The samples, called "Metal Waste Solution" in the H.E.W. Manual, Section C, P. 533, have the following approximate analysis:

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UNH = 21.5%
HNO_3 = 0.9
H_2SO_4 = 3.6
H_3PO_4 = 5.7
$NaNO_3$ = 0.7
H_2O = 67.6

There was about 3.5 ml. of each sample, a total of six samples, labeled B-4102 B-4103, B-4104, T-4105, T-4106, and T-4107, collected during the months of September and October in 200-E and 200-W. They were shipped in stainless steel sample cans enclosed in lead-lined casks. Upon arrival at X-10, the radiation level outside the sample can was about 30 mr/hr. at one foot, while the level a few inches above the solution itself was about 20 r/hr. The samples were stored temporarily behind a lead shield. Frederick expected to separate the UNH from the fission products by solvent extraction, probably with ether. The effect of the phosphoric acid on the extraction efficiency will need to be checked so it will be necessary to prepare a synthetic standard first and use it to test the procedure. Frederick hoped to have the samples purified within two weeks.

At Y-12, the writer reported to Dr. C. E. Larson. A tour through the plant control laboratories was conducted by Roger Hibbs. He explained the mass spectrometers, the fission counters and the attendent chemistry and as mentioned above, the CoF_3 method as used at Y-12 in the routine preparation of UF_6 samples.

The fission counters at Y-12 are essentially the same as those at K-25. A radium-polonium-beryllium source of neutrons is used in an instrument which counts five samples simultaneously. The samples are prepared on nickel plates by electroplating. The preparation of the plates consists of the following steps:

1. Dissolve the sample of uranium in nitric acid in a platinum dish.
2. Evaporate the solution to dryness, ignite to U_3O_8, and weigh.
3. Prepare a standard solution of the uranium by dissolving the oxide in nitric acid and diluting to volume.
4. Select a suitable aliquot of the standard solution, add oxalate, and electroplate hot at 3 amperes using a nickel plate (approx. 3 inch diameter) as the cathode. Uranous oxalate is collected on the plate.
5. Ignite the deposit to the oxide (1 to 8 mg. deposit).
6. Mount the plate in the counter, back side to the neutron source.

The ionization caused by the fission products produces the impulses which are counted.

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I was conducted through one of the beta track buildings by Mr. Mitchell at Y-12. Most of the "tracks" in the area are shut down and are kept in stand-by condition. The original electromagnetic process involved the separation of U^{235} from U^{238} in the "track", which is a large 180° mass spectrometer. The starting material was UCl_4, which was vaporized into an electron beam from a furnace within the source. The electromagnetic separation of U^{235} was carried through several cycles. The alpha tracks were much larger than the beta tracks and started with natural uranium. Early in the history of the process, the alpha tracks were shut down and enriched uranium from the diffusion plant was used as a starting material for the beta tracks. At the present time the few tracks in operation are being used for experimental work.

Considerable experimental work has been done at Y-12 on mass spectrometry with solid samples. Mr. Hibbs is conducting research on solid samples at the present time. With salts or oxides which vaporize below the softening point of pyrex, he leads the vapors into the spectrometer source through a heated pyrex tube from an external furnace. For salts and oxides of higher boiling points, he has constructed micro furnaces which mount inside the cap of the spectrometer tube in such a position that the mouth of the furnace fits into the ion source of the spectrometer. The vapors of samples contained in the furnace are discharged into the source at a rate controlled by the furnace temperature.

Summary

A trip was made to study the mass spectrometer as an analytical tool and to inspect the facilities of various laboratories which use mass spectrometers. The following places were visited:

1. Physics Department, University of Minnesota, Minneapolis, Minnesota, Laboratories of A. O. Nier.
2. University of Chicago, Laboratories of A. J. Dempster, Mark Inghram and H. C. Urey.
3. Oak Ridge, Tennessee, Carbide and Carbon Chemicals Corp., K-25, X-10 and Y-12.

Special attention was given to the types of mass spectrometers in use, the specific merits of each type, their applicability to analytical problems, space and staff requirements and the commercial availability of the instruments.

Mass spectrometers can be broadly classified as single focusing and double focusing. The single focusing types use magnetic deflection of the ion beam and are most commonly used for isotopic abundance work and quantitative analysis. Double focusing instruments use combined electrostatic and magnetic deflection. They are more complex, but capable of greater resolving power, and are preferred for work on isotopic masses and packing fractions. They are made in a variety of types, the best known being the Aston, Dempster and Mattauch types of instruments.

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Nier is also constructing a double focusing instrument for packing fraction determinations.

The ion beam collector of a mass spectrometer may be a photographic plate or a pair of collecting plates connected to electrometers. The latter is better for quantitative analysis. Photographic methods require higher accelerating voltages, stronger ion currents, and periodic breaking of the vacuum to remove and replace the plates.

Mass spectrometers are being manufactured by General Electric Co., Schenectady, N.Y., Consolidated Engineering Corp., Pasadena, Calif., and Process and Instruments, Brooklyn, N.Y. These instruments are constructed for the analysis of petroleum products of relatively low molecular weights. General Electric also constructed, under contract with the Manhattan District, a large number of Nier mass spectrometers for uranium analysis at Oak Ridge. These instruments were designed for a 40 - 400 mass unit range, and are being used in the routine analysis of UF_6 for the U^{235} - U^{238} ratio. It was the opinion of most of the men consulted that this instrument would be the most suitable one available for the higher mass ranges. The instrument is reasonably adaptable to modification for solid samples. Nier, and also the people at Oak Ridge, have modified and improved this mass spectrometer in the past few years. Most of these modifications have been published in project literature.

A great deal of work has been done on the use of mass spectrometers for the analysis of gaseous mixtures, especially of organic materials. However, its application to inorganic analysis, especially of relatively non-volatile compounds, is less common. Such applications are much more difficult here as provision must be made within the ion source for the vaporization of the sample. This has been done by sparking, by vaporization from a hot wire (for rare earths and other substances which undergo thermal ionization) and by vaporization from a micro crucible in a furnace within the spectrometer tube. The latter method was recommended by both Nier and Inghram, and considerable research work has been done with micro furnaces for solid samples in the spectrometer by Nier, Inghram, and by various workers at Y-12 at Oak Ridge. In the analysis of solid samples, the vacuum must be broken for recharging after each sample. This limits the capacity of an instrument to about one sample a day.

Isotopic abundance analyses present much fewer problems than analyses of mixtures of compounds and if a gaseous or relatively low-boiling compound of the element can be prepared the analysis is greatly simplified. Techniques with such samples have been well established. The isotopic ratio of U^{235} - U^{238} is determined at Oak Ridge on a routine scale using UF_5 which sublimes at 190° at 10^{-6} mm. pressure. Therefore, the vapor is easily drawn into the source from an external sample tube through a gas leak and the vacuum need never be broken on the spectrometer tube.

The fission counters and the attendant chemistry were investigated at Oak Ridge. Fission counting does not give as accurate a determination of U^{235} content as mass spectrometry, but it is a valuable companion instrument. Fission counting is indispensible for plutonium isotope analyses.

Possible Applications of Mass Spectrometry to Hanford Works Problems

The mass spectrometer can be readily applied to the analysis of gaseous materials and organic substances. Some specific applications of particular interest at Hanford are listed below. The application to solid samples, on the other hand, will be more difficult, but a thorough study of this problem is in order. Mass spectrometry is advantageous for the analysis of chemically inert materials (the rare gases), elements difficult to separate chemically, and particular isotopes which are of importance in fission product studies.

Problems which should be attacked by mass spectrometer methods are:

1. The Metal Waste Recovery Program

   U^{234}, U^{235}, U^{236}, U^{238} isotope abundance measurements are desired. The men at Oak Ridge have resolved U^{234} in the presence of U^{235} and U^{238} but have, as yet, been unable to resolve U^{236} using UF_6 in the General Electric Assay Machine. This problem may be solved by the use of a compound (possibly UCl_4) which will ionize to U^+ in the source, thereby reducing the mass number of the ion by nearly 100.

2. The 234-5 Project

   Plutonium isotope abundances will be needed.

3. The determination of hexone and hexone oxidation products.

4. The determination of iodine and xenon in the air stream from the metal dissolving tank.

   The mass spectrometer can readily give a continuous recording of the composition and provide a means of automatic control.

5. The determination of isotopic abundances among the fission products.

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Recommendations.

It is recommended that at least two mass spectrometers of the type used at Oak Ridge (that is, the G. E. Assay Machine) be obtained for research at the Hanford Works. One instrument is needed for studies with gaseous samples and a second is needed for the study of the mass spectrometry of solid samples.

It will be necessary to plan a staff consisting of:

1. Two competent chemists or a chemist and a physicist.
2. An electronics engineer.
3. A machinist, part time.
4. A glass blower, part time.
5. At least one spectrometer operator.

The men who are experienced with the mass spectrometer warn that it will probably require six months to one year to put the mass spectrometer into satisfactory operation after it has been assembled.

R. J. Brouns
R. J. Brouns
Analytical Section
Metallurgy and Control Division

RJBrouns/AB

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