Present Status of the Reconstructed Large Mass Spectroscope

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After completely destroying the old large mass spectrograph by the high-tide typhoon in 1961, the reconstruction of a new large instrument, which has the quite similar dimensions in the main parts to the old one's, was planned. The machinery works of the main parts was finished in 1964, and their setting up works was begun in 1964 in the new building of our Science Faculty.

The newly constructed large machine has the following points improved against the old one: in order to have better focus stabilities, the effective areas of the electric field and the main magnetic field are both somewhat extended; for eliminating charging up effect on the slit blades as much as possible, the blades can be baked.

With the new machine, electric method can be used together with photographic method simultaneously, for measuring double mass differences. And, we call the new machine "the large double focusing mass-spectroscope".

The preliminary focus adjustment was carried out with photographic method. In this case, even in the best condition, the resolution obtained was only about 100,000 or somewhat less, and in most cases it was only the half or one third of the above best value. In order to find out this cause, the electric method was adopted. And as the results, the rather large a.c. stray magnetic field, filling in the room which affected the ion beam oscillation, was found. At present, as the results of eliminating such these stray magnetic field as much as possible, the half-width resolution of about 300,000 has been obtained with the widths of main and collector slits of about 10μ respectively.

In order to increase the absolute accuracy of doublet mass difference measurements, some preliminary experiments with electric method have been carried out. A few results obtained so far will be described in this report.

I. Introduction

In 1951, the successful result in resolution was obtained with a modified Bainbridge-Jordan type mass spectrograph at Osaka University.^{1), 2)}

Immediately after the success, we were intending to construct a new large mass spectrograph, in order to make extensive developments in resolution and mass measurement accuracy.³⁾ In 1956, the constructing and final

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focus adjustment works of the large machine were completed, and the maximum experimental resolution of about 900,000 was obtained.⁴⁾ However, the accuracy of doublet mass differences obtained with the machine was found to be considerably lower than that expected. The cause of such difficulties might be mostly ascribed to the fact that the charging up effect on the edges of blades of slits, especially located at the position of energy spectrum.⁵⁾

During that the improvement works in that points were carrying out, in the fall of 1961 this large machine was completely damaged by the high-tide typhoon which attacked our laboratory at the center of Osaka city.

After the typhoon, we were intending to reconstruct a new large mass spectrograph again. Because of that such high resolution as mentioned above could be obtained with the previous large machine in its best condition, on designing the newly planned large machine, the ion-optical dimensions of the machine were adopted to be quite similar with the old one's. However, in order to increase the stability of the focusing characteristics of the machine for longer period, some improvements were made for vacuum envelope materials and effective areas of analysing fields, and the blades of slits and baffles were made to be bakable for preventing the charging up effect on their edges.

With the previous mass spectrograph, only the photographic method was used for measuring doublet mass differences, but with the new one, in order to make easy to find out systematic errors and to reduce them, we planned to be able to do the measurements by electric

method together with photographic method using the same instrument.

At the middle of 1964, the newly constructed large mass spectroscope was set up in the new laboratory situated in the suburb of Osaka city. Since then, the focus adjustment works have been carried out and the preliminary investigations for finding out the most accurate method to determine doublet mass differences with electric method have been done. Here, some results so far obtained will be described, together with the brief descriptions on some characteristics of the newly constructed large machine.

II. Instrumentation

Dimensions of the newly constructed instrument are quite similar to the old one, which characteristics were described in detail previously,³⁾ but here some basic characteristics of the instrument are tabulated, and the schematic diagram and the general view of the instrument are shown in Fig. 1 and 2 respectively.

<u>electric field</u>	<u>magnetic field</u>
cylindrical $\varphi_e = 84^\circ 50'$ $a_e = 1092 \text{ mm}$	uniform $\varphi_m = 60^\circ$ $a_m = 1200 \text{ mm}$
$\ell'_e = 1340 \text{ mm}$ $\ell''_e = 0 \text{ mm}$	$\ell'_m = 1200 \text{ mm}$ $\ell''_m = 4480 \text{ mm}$

The dispersion of 1% mass difference and the magnification are calculated to be 22.4 mm and 1.37 perpendicular to the central ion beam, and to be 76.7 mm and 4.6 along the mean focal

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Fig. 1 Schematic diagram of the instrument

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Fig.2 General view of the instrument

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plane, respectively. The resolving power for the main slit of 0.001 mm width is calculated to be 1.64×10^6 . The focal planes of direction and energy have the angle of 17° and 21° respectively with the direction of central ion path, so a photo-plate is set at about 19° through a double focusing point. And, for using electric method, a collector slit and a ten-stage electron multiplier are installed.

In the following sections, the important parts of the instrument and the performance are described in detail.

2-1 Ion source assembly

In Fig. 3 the ion source assembly present used is shown. The ion source is a kind of low voltage arc type with a hot filament. Ions are extracted through a circular hole of 2 mm diameter from the ionizing chamber and are roughly focused on a main slit by means of the lens system. The lens system consists of three cylindrical electrodes which have inner diameter of 36 mm. The first and the third ones are grounded, and an adequate potential is applied to the second electrode. The converging rate of ion beam is adjusted by changing the potential of the second one. The ion source assembly can be slided to horizontal and vertical directions, and turned about each axis so that the ion current passes through the main slit as much as possible.

The operation of the ion source is as follows. A filament is of 0.4 mm diameter tungsten wire and its current supplied by a 220Ah 6V lead storage battery is about 10 A. Electrons emitted from the filament are accelerated to an energy of about 100V, which is applied to a hollow cylindri-

Fig. 3 Ion source assembly

cal anode by a 5Ah 100V lead storage battery. The current through this anode is about 10 mA. A source magnetic field of about 200 gauss parallel to the anode axis serves to increase the running pathes of electrons and the ionizing efficiency. The source magnetic field is produced by a coil which is wound outside the evacuated source region. The gas sample is ionized which is admitted into the ionizing chamber at about 10^{-4} mmHg. The ions are extracted and accelerated out of the chamber by the electric field which penetrates through the circular hole into the chamber from the first grounded lens electrode. Total ion current extracted from the ionizing chamber is 150~350 μ A for the gas sample of N_2 and CO . The profile of the ion beam extracted

from the ion source is critically affected by the following factors; the sample pressure, the strength of source magnetic field and especially the total ion current. The batteries and the associated circuitry are all located in the high voltage cabinet and controls are brought out of the cabinet *via* insulator rods.

The accelerating high voltage regulator present used has the following characters,

voltage (continually variable)	
	$1 \sim 5 \times 10^4$ volt
maximum current	2.5 mA
d.c. stability (drift)	1×10^{-5} /min.
ripple	0.2 volt

When, first, a.c. powers were used for the filament current supply and the electron accelerating potential, the ripple, 60 Hz, of the high voltage which is usually used at about 35 kV was about 10 V. Thereafter a.c. power supplies were replaced by the storage batteries above mentioned and the ripple became only 0.2 V. The machine has the energy selecting electric field of about one meter radius, so ions accelerated by about 35 kV with about 10 V ripple will oscillate by the amount of about 0.3 mm at the energy difining slit (β -slit) located at the exit of the electric field. Accordingly, the ion intensity after passing through the β -slit with 0.1 mm width varies with twice a frequency of high voltage ripple, that is 120 Hz.. If 0.2 V, the oscillation width corresponds to about 6×10^{-3} mm. In that case, the ion intensity modulation does not almost observed. On the other hand, as ions have about 5 eV energy spread due to the ionizing process,

so the intensity modulation of the amount above mentioned is not so serious. But, of course, it is desirable that the accelerating voltage ripple is as small as possible.

Fig. 4 High voltage switching circuit

The accelerating and lens potentials are supplied from a high voltage switching circuit shown in Fig. 4. The lens potential applied to the second electrode by varying R_2 , which are 4 W carbon resistors ($100\text{k}\Omega \times 100 + 200\text{k}\Omega \times 200$) soaked in oil to prevent the corona discharge. R_1 is $200\text{k}\Omega$ 20-turn helipot, which is adjusted so that the higher member of mass doublet passes through the β -slit as much as possible in the peak matching method. R_1 and R_2 can be controlled by means of insulated rods from ground.

2-2 Main slit assembly

Fig. 5 schematically shows the main slit assembly which consists of a shutter, a z-difining baffle, a main slit, and a monitor. When it is necessary to measure the total ion current extracted from the ionizing chamber, the shutter can be insulated from the ground potential. The diameter of ion beam cross section on the main slit is about 1 mm. So when ions have energy of 35 kV and total ion current is $300 \mu\text{A}$, the dissipated power on the slit becomes to be about 14 W/mm^2 . The z-baffle of 0.1 mm width is movable to verti-

Fig. 5 Main slit assembly

cal direction.

The main slit consists of two rods of 1mm diameter stainless steel wire. The maximum width of the main slit is about 10μ . By rotating it around the vertical axis at the center of the slit from outside of the vacuum envelope, its slit width can be adjusted from its maximum to zero continuously. The vertical location of the slit is variable within ± 2 mm. The vacuum box including the slit is movable to the direction along the ion path and to the horizontal direction within ± 40 mm and ± 5 mm respectively. The width and height of the main slit generally used in the present experiment are about 5μ and about 0.1 mm respectively. The inclination of the slit can also be adjusted. The ion current through the main slit can be monitored by a Faraday cup.

The main slit and ion source assemblies are mounted on a iron bench and can be moved in any direction as a whole. We can easily direct the ion beam to the proper position on the main slit, by looking the beam by naked eyes through the window. A well-collimated ion beam looks like to be a fine wire which is gleaming blue.

2-3 Energy analyzing field

The energy analyzer is a conventional cylindrical condenser, the radial cross section of which is shown in Fig. 6. The circular arc electrodes

of the condenser are made of nonmagnetic stainless steel, each having a cross section of 50 mm thick and 180 mm high. The gap between the two electrodes is 30 mm. So the outer radius of the inner electrode is 1078 mm and the inner radius of the outer electrode 1108 mm. In the old instrument, the height of electrode was 80 mm and it was supposed that even at the central part of gap the field would not be ideal field. In the present one, the improvement extending the effective radial field area has been done.

The field defining slits (Herzog end plates) are located at the entrance and exit faces of the condenser so that the effective field boundary roughly equals to that of the condenser. In an asymmetrical instrument like the present one, it is important that the actual effective field boundary is as equal as possible to the ideal one. Because if not so, the focusing point may shift considerably.

The direction and the energy difining slits are located at the entrance and the exit of the condenser respectively. These slits can be moved to lateral direction by the micro-meter screws. Also their widths can be adjustable, but ordinarily are 0.1 mm. When these slits are spoiled by oil vapour, the oil will be carbonized by ion bombardment and insulating film will be produced on the slits. So the surfaces of the slits are charged up irregularly and this effect not only reduces the resolution but also introduce the systematic error in the mass measurement. Then the improvement to prevent the contamination of oil vapour has been doing, that is heating the blades of

Fig. 6 Radial cross section of energy analyzer

slits and baffles.

In front of the direction defining slit, there is a monitor system which determines the lateral and vertical position of the beam at the entrance of the selector. The material of all parts including the vacuum chamber are non-magnetic stainless steel, except for vacuum seals and some insulators.

The carefully stabilized voltage is supplied to the electrodes of the energy

selector through the Nier circuit shown in Fig. 21 from a set of twenty-two 90 V layer dry batteries (BL 260) with the stabilizing circuit. In Fig. 7, the stabilizing circuit is shown. As current more than 1 mA must be drawn to the Nier circuit, supplying only from the batteries without the stabilizing circuit, the drift in supplied voltage was more than 10^{-5} per minute. With the stabilizing circuit, the drift of less

Fig. 7 Stabilizing circuit for deflecting potential

than one part in 10^7 per minute can be easily achieved and this stability in supplied voltage is completely enough for the operation of the instrument. In Fig. 7, the error signal between the voltages of twenty 90 V dry batteries (BL 160) and the output is fed to a 12AX7 tube which is used as a cathode follower. Its output signal is amplified by a Philbrick T52 and this amplified error signal controls the plate current of a 2C53 tube. In the operating state, switches S_1 , S_2 are turned on and S_3 off. At the beginning of the operation of the circuit, all switches are turned on, and a 200 k Ω helipot and a 1 M Ω variable resistor are adjusted so that the current through a micro-meter becomes to be zero, that is, the error signal equals to zero. Then S_3 is turned off and the power supply is connected to T52 amplifier. After these processes, the circuit becomes to be in proper operating condition. In the normal oper-

ation, the current through the 2C53 tube is about 0.3 mA. The 12AX7 is used to draw out no current from the standard cells (BL 160). For filament power supplies of 2C53 and 12AX7 tubes and other power supplies, storage batteries or dry cells are used in order to prevent the 60 Hz hum noise and they are all located in the negative portion of the high voltage.

2-4 Momentum analyzing field

The uniform magnetic field is obtained from the large electro-magnet, the radial cross section of which is shown in Fig.8. Except for pole pieces and exciting coil, the magnet is one used in the old instrument. The pole gap is 18 mm which is maintained by brass spacers, while in the old one it was 9 mm. The surfaces of the two pole pieces are smoothed and flattened to the degree of 0.01 mm and are placed parallel to each other with the same degree of accuracy. An analyzer tube is used and inserted in the

pole piece gap, although in the old one the space surrounded by the two pole surfaces and brass pieces had made a vacuum chamber. In addition, the effective field area is increased, that is, the radial width of pole piece is increased to 15cm while 12cm in the old

one. The analyzer tube is made of SUS 27 stainless steel, the inner wall of which has a cross section of 10 mm high by 72mm wide. The exciting coil consists of 4500 turns with a total resistance of 120 ohm.

Fig. 8 Radial cross section of magnetic analyzer

Fig. 9 Cross section of magnetic shields along ion path

At the entrance and exit of magnet, there are three types of shields to reduce the stray field. The cross section of the shields along the ion path is shown in Fig. 9. The first one is a Herzog shield made of pure iron block and is located by the pole gap distance namely 18mm apart from the pole face. The second one is a permalloy wall which is 12mm high, 70 mm wide and 170mm long and is installed in the the Herzog shield, moreover in the vacuum envelope. This permalloy shield reduces the stray field in the region

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near the magnet, that is in the Herzog shield. The third one consists of four iron screens standing at the Herzog shield, like surrounding the pole face. These iron screens which are 1 mm thick are separated each other magnetically. These screens reduce the stray field in the long place apart from the pole face. Fig. 10 indicates the distribution of the stray magnetic field strength along the ion path. Curve A is the case with only the Herzog shield, B is with the Herzog shield and the iron*

becomes to be an undesirable form.

The high stability of magnetic field is realized by powering the magnet coil by a set of thirty 2 V 220Ah lead storage batteries with the stabilizing circuit which is a current stabilizing type shown in Fig. 11. A magnetic exciting coil current passes through a resistor R_2 shown fully in Fig. 12, and the voltage drop in R_2 is compared with two 1.3 V Hg cells. The resultant error signal is amplified by a chopper amplifier (Yokogawa VN-11) followed

Fig. 10 Distribution of magnetic stray field

* screen shields, and C is the case with those three shields. It is desirable that the stray field is as small as possible and the field at the pole face is sharply cut off. Even though the alignment of the apparatus were right, the focus lines would be shifted by the stray field, among them especially by magnetic stray field. By the way, however, when the permalloy shield is approached to the pole piece over the Herzog shield, the field distribution

by a I. C. amplifier (Philbrick T52).

Its amplified error signal controls the current through a transistor 2SC376 which is by-pass of the coil. In normal operation, the current through the transistor is $150\sim 200\mu\text{A}$. R_1 shown in Fig. 11 consists of two parts, one of which is variable from 0.01Ω to 500Ω by 0.01Ω step and a main current passes through, and the other is $30\text{ k}\Omega$ 10-turn helipot which is used to control a slight change of the exciting current.

Exciting the coil for one day past when the instrument is operated and taking proper care, the drift in field strength of less than five parts in 10^7 per minute can be achieved. While without the stabilizing circuit, the drift is about one part in 10^5 . Using the current sta-

ture variations of e.m.f. of Hg-cell and of resistance of R_2 , they are temperature controlled within $\pm 0.1^\circ\text{C}/\text{min}$.

2-5 Beam scanner

In order to observe the mass peak shape on the synchroscope screen, the ion beam must be scanned before a *

Fig. 11 Stabilizing circuit for magnetic exciting current

Fig. 12 Details of R_2 resistor
in Fig. 11

bilizing circuit, the ion beam drift ascribed to that of the magnetic field is negligible small in operation. For reducing the drift due to the tempera-

* collector slit by a saw tooth field. In the present case, a magnetic field is used, which is produced by feeding the saw tooth current to a Helmholtz type coil located at 2 m apart from the exit of the main magnet. The cross section of this coil is shown in Fig.13. The ion beam is scanned at the front of the collector slit from left to right or *vice versa* by operation of a reverse switch. the scanning width, usually 200μ , can be changed by varying the gain of the voltage to current converter and the scanning frequency also can be varied from 0.001 Hz to 100 Hz.

The d.c. magnetic field which rides on the saw tooth field is compensated by feeding a d.c. current to an another Helmholtz coil placed at the same place.

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Helmholtz Coil

Vacuum Envelope

Fig.13 Cross section of scanning coil in the direction perpendicular to ion path

stainless steel plates of 0.5 mm thickness, forming knife edges. Ordinarily, 10μ aluminu foil lies between the plates. So the slit width is about 10μ and its height is 2 mm. The vertical location of the slit can be adjusted from outside of vacuum chamber and also the vertical inclination is adjustable. If

Fig.14 Voltage to current convertor

The d.c. current can be adjusted in order that ion beams are collected when the combined field strength is zero. If ion beams are collected when the combined field is not zero, it may be that the systematic error is brought in the mass measurement.

The voltage to current convertor circuit is shown in Fig.14.

2-6 Collector slit and ion detector systems

The collector slit consists of two

necessary, the slit can be removed from the ion path and analysed ions are directly collected.

To detect the ion beam current less than 10^{-12} A, a 10-stage Ag-Mg secondary electron multiplier and a fast response d.c. amplifier (Keithley Model 300) are used. This amplifier is 100 % feed back type as well known. The high voltage applied to the electron multiplier is supplied by eighteen 90 V dry batteries (BL 160). The gain of the

multiplier is about 3×10^3 . Under this applied voltage the noise level is very low, but for higher applied voltage the noise increases much more than the signal. The d.c. amplifier is placed as near as possible to the multiplier output to reduce the stray capacity. Using $10^{10} \Omega$ high resistance for the d.c. amp. input, the scanning rate of 10 Hz can get good result without any peak shape deformation.

The vacuum envelope including the slit system and the detector system can be moved along the direction of the ion path and also perpendicular to it.

2-7 Evacuating system

The vacuum envelopes are made of stainless steel and for vacuum seals vitons are used. Two gate valves are installed; one is placed between the main slit and the energy selector, and

the other between the main magnet and the collector slit. Therefore three parts of the instrument can be evacuated independently. For evacuation of the whole system, seven sets of 7 inch oil diffusion pump backed by 2 inch oil diffusion are used. The rough vacuum lines for each pump-set are centralized to a low vacuum tank evacuated by a 2 inch oil diffusion pump and an oil rotary pump. The chambers of the ion source and the main slit system are evacuated by two sets of 4 inch oil diffusion pump independently. To prevent from oil back diffusion, liquid nitrogen trap is installed between each pump-set and the vacuum envelope of the machine. The vacuum is roughly estimated to be 3×10^{-7} mmHg by the B-A gauge. In Fig.15 is shown a schematic drawing of evacuating system.

Fig.15 Schematic diagram of evacuating system

III. Experiment

3-1 Photographic method

At the time when we planned to reconstruct the instrument, although the peak matching method was adopted in many laboratories of the world for the precise mass measurement, we were intending to adopt the two method jointly in the new instrument. However, as we had only photographic technique but no knowledge about peak matching, we first adopted the photographic detection in the instrument. On the other hand, preliminary experiment for peak matching was carried out using remodeled Bainbridge-Jordan type instrument which was originally constructed thirty years ago as a mass spectrograph.⁶⁾ Thus, since the end of 1964, the focus adjustment of the present instrument had been carried out using the photographic detection.

Before the collector slit, there is a camera chamber in which a photographic plate is mounted. The size of the

plate is 45mm high and 300mm wide.

Shifting the plate holder vertically by external means, six series of the mass spectra can be taken. The camera chamber can be moved along and perpendicular to the ion path.

Unfortunately, the minimum width of the image obtained was about 0.1 mm, and the width ordinarily obtained was about 0.3mm which corresponded only to the resolving power of 30,000. In addition, the reproducibility of the image was not good. In order to find the cause of such a de-focusing, the photographic method was replaced by the electric method for ion detection, in last spring.

3-2 Ion beam oscillation

Adopting the electric detection, immediately the synchroscope display showed that the mass peak had been subjected to a serious modulation as shown in Fig.16. Such a modulation might be not that of ion intensity, but be ascribed to oscillation of ion beam at the collector slit.

Fig.16 Ion beam oscillation behaviour with 0.1 Hz scanning rate

It was inferred that the ion beam had been oscillating with a small amplitude, roughly that of the same order with the image width, in the front of the collector slit. Moreover, it was found that the majority of the component of this oscillation consisted of sine wave of 60 Hz synchronized with a.c. power line and the rate of oscillating amplitude was roughly estimated at 3×10^{-5} in terms of the reciprocal of resolution. Such effect might not be able to find out by photographic detection. Accordingly, the failure to obtain a sharp mass line on photographic plate might be mainly ascribed to such ion beam oscillation. With the old large mass spectrograph, such an oscillation effect was not experienced. The reason may be that all the vacuum envelopes of the new apparatus were made of nonmagnetic stainless steel, instead of ordinary iron for the old one.

The main cause of such a rather large beam oscillation was considered to be in the a.c. magnetic field filled in the laboratory where the apparatus is installed. So the strength of such stray field in the room was measured by a searching coil. And, it was found that the a.c. stray magnetic field, mainly including sine wave of 60 Hz which was synchronized with a.c. power line, existed and this field was not localized but widely distributed in the room. This magnetic field may affect the ions throughout their whole path in the instrument.

Until recent date, the source of this large a.c. stray field had not been found. However, we could cancel out this beam oscillation rather well by the compensation method. This method is divided into the following two parts.

One is that this oscillating field is picked up by the searching coil placed at the exit of the main magnet, and then the phase and amplitude of its output are changed adequately, and the compensation current thus obtained is feeded to a Helmholtz coil placed at the position of a beam scanning coil. The other is that the phase and amplitude of a.c. 60 Hz line current are changed adequately and feeded to another coil placed to the same position. On the other hand, it was not so effective for cancelling the beam oscillation to feed back the above output to the auxiliary coils which are wound around the pole piece of the main magnet. With such compensation, the total width of mass peak on synchroscope screen was decreased by about one third of that without compensation.⁷⁾

A main source of this a.c. stray field was found to be 200 V three-phases a.c. power cables running in the line-duct through the passage outside the room. One of the cables has ordinarily a slightly different potential from the ground. So if its cable touches to the ground, a large loop is made up around the room and considerable current flows in the loop. In consequence the a.c. magnetic field is produced over the room. At an adjacent room, one of the power cables for a cooling device had touched the window frame made of steel. Also as a main switch for cooling device was jointly used for another device, the operation of the switch was irregular, except that the switch had been stationarily turned on in summer. Even if the ion beam proper has the width corresponding to the resolution of 10^5 at image position, the image to be detected will have that corresponding

to 3×10^4 due to the ion beam vibration, when this switch is turned on. However, when this switch is turned off, the pure image not disturbed by the vibration is detected. It may be mainly due to the matter just mentioned that we had not been able to establish the reproducibility in regard to the resolution in the photographic detection. Besides the cause above mentioned, such an effect, almost the same as the strength above mentioned, had been observed. Fortunately, it was found at once that a part of the heater used in one of diffusion pump which was supplied by 200 V power line touched its metal wall.

After eliminating the a.c. stray magnetic field above mentioned, a small ion beam oscillation still remained. The amount was estimated to be about 2×10^{-6} in terms of the reciprocal of resolution. For such a small oscillation, the compensation device above mentioned was completely powerless. However, it was found that fluorescent lamps in the room and power supplies for 7 inch oil diffusion pumps had great influence on this small field. By covering the ion beam duct with iron pipe, and reconnecting the cables of power supplies to pump heaters properly, the small ion beam oscillation could be reduced considerably well. However, the beam oscillation of about 1×10^{-6} in terms of the reciprocal of resolution still remains at present, and such a very small oscillation has no longer the pure sine wave form. The cause of this oscillation has not yet been found. Now, in order to decrease the amplitude of this small oscillation by one order of magnitude, more complete shielding of a.c. stray mag-

netic field along the ion beam path with permalloy and soft iron is planned.

3-3 Focus adjustment

By means of the photographic detection, we had not been able to perform the focus adjustment successively. It may be mainly due to the ion beam vibration caused by a.c. stray magnetic field. With beam scanning and electric detection technique, the performance of focus adjustment may be considerably easy compared with the photographic detection. Many informations on the ion beam profile can be obtained on a synchroscope screen. So the focusing conditions can immediately recognized by adjusting several parameters and the image width can also be roughly estimated

For our large mass spectrometer, the position of double focusing point is calculated to be 4480mm apart from the exit edge of the main magnet, and the direction and the energy focusing lines lie at the angles of 17° and 21° to the central ion path respectively, as previously mentioned. At first, the main parts of the instrument; the magnet, the energy selector, the main and the collector slit, were aligned exactly at their calculated positions as much as possible. Even when the geometrical setting could be exactly realized at calculated positions, many other factors; the stray fields, the field inhomogenities, the space charge effect, and the residual gas scattering etc. would have rather large affections on ideal focusing. In the present instrument, once the energy selector and the magnetic analyzer were aligned at their proper positions, this relative position was never changed. Therefore, the focus adjustment was carried out by adjustment

Fig.17 Schematic views of direction and energy focusing in the instrument

Fig.18 Flat top and sharp mass peak

of the positions of the main slit and the collector slit, mainly by that of the latter.

As well known, ion optics shows how the ion beam leaving a main slit focuses on the direction or the energy focusing line, accordingly the double focusing point. The schematic views of the direction and the energy focusing of the present instrument are shown in Fig.17. Now, let us slide the direction difining slit (α -slit) only a few to the positive radial direction. If the collector slit is placed before the direction focusing line, the mass peak observed on synchroscope screen shifts toward the low mass side. Conversely if the collector slit is behind its line, the mass peak shifts toward the high mass side. In the case that the collector slit is just on the direction focusing line, the mass peak does not shift but only its height changes. Thus we can estimate whether the collector slit is placed before, behind, or on the direction focusing line, qualitatively or to a certain extent quantitatively.

Next, let us change the energy of ions slightly by varying the ion accelerating voltage. If the collector slit is not placed on the energy focusing line, the mass peak shifts on synchroscope screen toward any side. For instance, if, when the accelerating voltage increases only a few, the mass peak shifts toward the high mass side, the collector slit is placed before the energy focusing line. In the case that there is the collector slit on the energy focusing line but not on the direction focusing line, the mass peak shape forms a flat top as shown in Fig.18, since the ion beam distribution at the α -slit is expected to be almost homogeneous. In this case, the α - and β -slit together have 0.1 mm width. Here, if the α -slit is narrowed to less than 0.1 mm, the height of the mass peak is not almost changed but its width is narrowed and the sharp peak is obtained. In the contrary case that the collector slit is on the direction focusing line but not on the energy focusing line, the similar behaviour is observed.

Fig.19 Two mass peaks which are deviated each other by one part in 150,000

According to these principles, the focus adjustment has been performed. In order that the collector slit rides on the double focusing point, the collector slit has been moved along or perpendicular to the ion path. At present, the resolution of about 150,000 which is estimated at the full width of mass peak is obtained under the following condition; the width of the main and the collector slits are about 0.007 and 0.010 mm respectively, and the α - and β -slits together 0.1 mm. Fig.19 shows two mass peaks which are deviated each other by one part in 150,000. In the peak matching method, the collector slit should be placed on the double focusing point. If not so, error will be introduced in the precise mass measurement due to the energy or the direction de-focusing.

3-4 Mass difference determination

For the precise mass measurement with electric detection of ion beam, the peak matching method is exclusively used.⁸⁾ This method is based on the following principle. For a fixed magnetic momentum analyzing field, if every electric potential applied in the instrument are varied inversely proportional to the mass to charge ratio of charged particles, trajectories of particles are unchanged. Now let us consider two ions which have masses M_1 and M_2 and the same charge, and which are detected respectively when the deflecting potential applied to the energy analyzer are V_1 and V_2 . Then, the condition above described is replaced by

$$M_1 V_1 = M_2 V_2.$$

Here, if $M_1 = M$, $V_1 = V + \Delta V$ and $M_2 = M + \Delta M$, $V_2 = V$, above equation follows that;

$$\Delta M / M = \Delta V / V.$$

In practical application, this deflecting potential is supplied through the Nier circuit shown in Fig.20 to the electric analyzer. Therefore the potential ratio may be replaced by the resistance ratio, i.e.

$$\Delta M / M = \Delta R / R.$$

Thus, the measurement of the mass doublet difference is reduced to that of the resistance. Also, for proper operation of the instrument, it is necessary that the ion accelerating potential is changed at the same rate.

Fig.20 Nier circuit

In order to determine that two ions having masses M_1 and M_2 strictly follow the identical trajectory, the two mass peaks are observed on synchroscope screen by means of the device described below.

Fig.21 shows a block diagram of the peak matching circuitry. The saw tooth signal, produced in a function generator, is feeded to the scanning coil through a voltage to current convertor shown in Fig.14. The pulses, synchronized with the start of the saw tooth signal, trigger a horizontal sweep of synchroscope and a relay driver shown in Fig.22 simultaneously. The

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output of electron multiplier is amplified and feeded to the vertical input of synchroscope. The relay driver

actuates three sets of relays. The first set causes the deflecting potential

Fig.21 Block diagram of peak matchig

Fig.22 Relay driver circuit

Fig.23 Construction of reed switch in Fig.4

Fig. 24 Construction of mercury relay in Fig.20

supplied to energy analyzer to switch between V and $V + \Delta V$, and consists of a mercury wetted relay which is separated to a relay coil and a contacting portion to eliminate a leakage current to the ground as shown in Fig. 24. The second one causes the ion accelerating potential to produce a similar change, and consists of a reed switch which is tolerable for the high voltage as shown in Fig. 23 and a mercury wetted relay installed in the high voltage. The third one is a mercury wetted relay which change the d.c. amplifier gain by a gain switching circuit with two 20 k Ω helipot.

Switching operation of these three relay sets makes it possible to superimpose the two members of the mass doublet on synchroscope screen.

Relationship of these phases is shown in Fig. 25. Thus, on sweep 1, 3, 5, ---, one can observe one member of doublet and on sweep 2, 4, 6, --, the other member.

Fig.25 Phase relation of peak matching

A new method of the peak matching has been tried which we call the peak differentiation⁷⁾. This method may somewhat reduce the matching error caused by personal judgement compared with the orthodox peak matching as described above. In the peak differentiation method, the output signals of the multiplier are amplified and then differentiated. On alternative sweep, any differentiated peak is inverted. The superposition of a differentiated peak with its inverted one is shown in

Fig.26 Superposition of a differentiated peak with its inverted one

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Fig. 26. In orthodox method, the matching point is found by the superposition of peak shapes as a whole. While, in the differentiation method, this point is found by intersecting three lines i.e. zero volt line and two differentiated peaks one of which is inverted. Using N_2-CO mass doublet, matching errors caused by the orthodox and the differentiated peak matching have compared, which are shown in Fig. 27. In Fig. 27 as well as Fig. 28 each observation consists of ten or so of matchings, and the arithmetic mean of these matchings is adopted, and the errors attached to the means are standard deviations of one

matching. But, in the present stage, it may be not better to mention about numerical values of the means. Three groups are shown in Fig. 27, which were observed in different days. In any group, the observations by the two method were done respectively by two different observer. In this stage, the values observed in different days were more deviated over their errors.

But the values observed in any day by the differentiated method are consistent compared with those by the orthodox one, and the standard deviation of one matching is also somewhat small.

In Nier circuit in Fig. 20 and accel-

Fig. 27 Comparison with orthodox and differential peak matchings

erating voltage switching circuit in Fig. 4, operating reverse switches, one can observe a high or a low mass peak when relay coils are exciting. Let us call the former and the latter by A- and B-states respectively. In the measurements of N_2-CO mass doublet, a large systematic deviation among values of the mass difference observed*

found that the reverse switching operation in accelerating voltage switching circuit affected less to this deviation. So the effect of such a deviation was inferred to be mainly due to the reverse switching operation in Nier circuit which was located in the vicinity of the energy selector. First, as the cause of this effect, a contact^x

Fig. 28 Large systematic deviation; circle is in B-state and triangle is A-state.

* in A- and B- states was found, shown in left side of Fig.28. Observations in A- state indicated by triangles are much larger than that in B-state indicated by circles. The difference between these observations are sometimes larger than their errors, and is of about 2Ω in ΔR . This quantity corresponds to about 0.1 mV in ΔV and to about $20 \mu u$ in ΔM . Until recent date, the cause of this effect had not been found. But it was

^xpotential and resistance of contact points of relay are considered. The former is less than 0.01 mV measured by chopper amplifier and the latter is about $5 m\Omega$ measured by double bridge. These quantities are too small compared with the deviation observed. Next, a magnetic field produced by relay coil exciting is considered. By such a field, a high mass ion beam will be caused to deflection in A-state and a

low mass ion beam in B-state. In this case, values of mass difference obtained in A- and B- states may deviate to the opposite direction each other by the same quantity. In order to confirm this consideration, the exciting current through the relay coil was reversed, *i.e.* the deflecting directions caused to ion beams was reversed. Then the behaviour as shown in the figure was inverted. At last, the box of the Nier circuit was kept away from the beam trajectory. The results thus obtained is shown in right side of Fig.28. still a very small deviation remains, but within the standard deviations of one matching.

III. Conclusion

Obstacles have been eliminated or reduced, which are made it impossible to perform the precise mass measurement in the present instrument. The focus adjustment has been performed successfully using electric detection. The drift of ion beam is reduced to less than five parts in 10^7 per minute, which is negligible small in operation. Also, the ion beam oscillation is reduced to one part in 10^6 estimated in terms of the reciprocal of resolution. This reduction is a little unsatisfactory in present operation. The disturbance of the magnetic field produced by relay coil exciting is almost eliminated.

At present, it is possible to perform the mass measurement in precision of one part in 10^7 . But, at the present status, it would not be better to mention carelessly about the numerical values of mass doublet. Yet, some causes, which may bring systematic errors in the observed values of mass doublet, remain unresolvedly.

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