UPPSALA UNIVERSITY

INSTITUTE OF PHYSICS

The design of a new experimental equipment for photo electron measurements.

by

Kai Siegbahn

Contract number DA-91-591-EUC-2899

Final Technical Report. 1/6 1963 - 31/5 1964.

The research reported in this document has been made possible through the support and sponsorship of the US Department of Army, through its European Research Office.

Abstract

The photo electron method for determining the binding energies in atoms has been used at the Institute of Physics of Uppsala for some years. The method has been refined and now yields data more accurate than the X-ray absorption method. The photo electron method is furthermore very sensitive, an atomic layer of 100 Å thickness is enough to get good signal-to noise ratio. Signals have been obtained from practically all elements in the periodic system from 5 Boron up till 92 Uranium. These investigations have been made with an iron free double focussing spectrometer.

In order to investigate the possibilities of the photo electron method as an analytic tool for determining the relative abundance of elements a semicircular electron spectrometer with permanent magnet pieces has been made. The pole pieces of the spectrometer have a diameter of 60 cm. Two additional airgaps have been introduced in the spectrometer in order to achieve a better homogenity in the magnetic field and also to extend the homogenity over a greater part of the pole pieces.

Attached to the spectrometer is an evaporation chamber which makes it possible to introduce an evaporated source into the spectrometer without exposing the source to air.

The X-rays which are used for expelling electrons from the source are monochromatized by means of a bent crystal. It is possible to use both Cu$K\alpha$ and Mo$K\alpha$ radiation.

The detection is made with photographic plates and the intensity ratios will be determined by counting electron tracks.

Photo electron lines have been obtained from the K-shell of Manganese the electrons being expelled by Mo$K\alpha_1$ and Mo$K\alpha_2$ X-radiation.

Introduction

The binding energy of electrons from various shells in atoms can be accurately determined in two ways, the X-ray absorption and the photo electron method.

In the middle of the 1950's a research group at the Institute of Physics of Uppsala started a systematic study of electron binding energies which now covers pratically the whole periodic system /1/. The resolving instrument is a 30 cm iron free double focussing spectrometer, which was constructed for this purpose /2/. The high resolving power of this instrument together with some refinements in the techniques have made it possible to get determinations of binding energy values much more accurately than those of the X-ray absorption method. Up till now 215 energy levels in 76 elements have been determined with an accuracy of a few tenths of an eV. The lightest element which has been examined is 5 Boron and the heaviest 92 Uranium. A binding energy table covering the whole periodic system based on photo electron measurements has been published /3/.

Since the photo electron method has proved to give good electron signal-to-noise ratio from all elements it would be of great importance if the method could be used in quantitative analysis of the relative abundances of elements in compounds especially for the light elements where the X-ray fluorescence method is impossible to use.

The double focussing spectrometer is equipped with an X-ray tube, especially built for the spectrometer. From this tube the X-radiation consisting of characteristic and continuous radiation irradiates the sample. The contribution from electrons expelled by the continuous X-radiation gives a rather high background.

In order to get an accurate determination of the relative intensities of different electron lines it would therefore be desirable to use a curved crystal for monochromatizing the X-radiation. This would give an increased signal-to-noise ratio. However, this is not possible with the present geometry of the spectrometer. We have therefore built a semicircular spectrometer of the permanent magnet type with a fairly large diameter, i.e. 60 cm. This type of spectrometer has the advantage of being able to analyse a large energy region at the same time, using photographic registration. This would of course be of great practical importance when studying compounds of different elements. With this type of instrument one can also use commercial X-ray tubes and curved crystals for the monochromatisation. This combination of monochromatized X-rays and a spectrograph with high resolution have never been tried before.

Design of the permanent magnet electron spectrometer

The magnet

Since this spectrometer would be used preferably in analysing electrons with low energy it was given a rather unconventional design. The common way of building a semicircular spectrometer of the permanent type is demonstrated for instance by Slätis /4/.

In the present type the vacuum chamber of the spectrometer also serves as iron yoke for the magnetic return flux. The chamber consists of a 15.0 mm thick cylindrical shell of diameter 680.0 mm and height 106.0 mm and two plates of thickness 10.0 mm. The cylindrical shell has 6 openings which will be explained later on, see fig. 1. Since it has been our purpose to make a spectrometer as light and neat as possible without disturbing the magnetic field, the plates had to be strengthened in order to restore the air pressure when the spectrometer is under vacuum. Two aluminum plates of a special design have therefore been glued on the ironplates of the vacuum chamber. The epoxy consisted of 100 parts of Araldit AY103 and 8 parts of Araldit HY951.

The permanent magnets were delivered from the Swedish firm Fagersta and of their quality Ticonal Gg. These magnets are made in cylindrical pieces with diameter 22.5 mm and height 14.0 mm. The magnets were placed upon the iron plates of the vacuum chamber. Each layer contains 73 pieces. As seen from fig. 1 the permanent magnets are kept fixed in their positions through an aluminum matrix. Upon each layer a circular iron disk of 600 mm diameter and 5 mm thickness is placed. The pole pieces have a diameter of 600 mm and a thickness of 15 mm and are placed with an airgap of 5 mm to the thin iron disk in question. We believe that these extra airgaps have had a substantial influence on the homogenity of the magnetic field.

The distance between the pole pieces is 29 mm. The two pairs of iron disks have been polished flat to within 0.01 mm and the variation in thickness is less than 0.005 mm. Spacers made of brass have been inserted between the disks.

The magnetization coils contain each 64 windings of cotton-covered copper wire. The wire has a rectangular intersection with dimensions 3x2 mm^2. The average value of the radius of the coils is 323 mm. The position of the coils can be seen in figure 1. A power supply, which gives a maximum DC current in the coils of 85 Amperes, is available.

The manufacturing of the coils has required a complicated procedure. Since in this design the coils have to be placed inside the vacuum chamber it is necessary that the coils have a low degassing effect. The problem was solved in the following way:

The copper wire was wound on a bobbin made of brass. An aluminum foil was placed about 3 mm above the last winding layer on each coil and tightened to the bobbin. Then the coils were placed in a vacuum chamber which was evacuated down to about 10 torr. After a reasonable time of pumping the coils were filled up to the aluminum foil with an epoxy (100 parts of Araldit F (CY 205), 100 parts of HY 905, and 10 parts of DY 040) maintaining the same pressure 10 torr. The coils were then placed in an owen at a temperature of 150°C for about 30 hours. After that the aluminum foils were taken away. In making the coils in this way it was possible to avoid enclosings of air which would have damaged the desired vacuum properties of the coils.

As mentioned earlier, the vacuum chamber also serves as iron yoke. It has 6 openings which can be seen in fig. 2. Connected to these are

1. Ionisation gauge head and an air inlet valve.
2. Connections to the magnetization coils and a turnable coil for measuring the magnitude of the magnetic field.
3. The plate holder.
4. The vacuum pump system.
5. The evaporation chamber through which the source holder can be slid into the source position.
6. An air tight window for the X-radiation and a directing equipment into which the plate holder can be slid. The position of the plate holder is very well defined.

The plate holder

The position of the plate holder can be seen in fig. 2. In front of the photographic plate which has the dimensions 17x298 mm^2 a shutter system is placed. It consists of two doors which can be operated from outside the spectrometer. Each of them can be put closed, half-open or open which makes it possible to expose the photographic plate by 3 sections along its length. Behind the photographic plate an insulated brass plate is placed. It can be put at high tension in order to accelerate electrons with low energy. Through a hole in the back of the plate holder the source holder can be slid into its position 20 mm behind the plane of the photographic plate. The source position relative the photographic plate is reproducible to around 0.01 mm. In front of the source position and in the same plane as the photographic plate two movable baffles of brass are situated. Lead shields are placed between the source position and the plate position and also behind the position of the plate holder in the spectrometer /fig. 2/.

The evaporation chamber

The evaporation chamber can be seen in fig. 3. A valve makes it possible to shut off the evaporation chamber from the spectrometer. The source holder can be slid through the evaporation chamber into the spectrometer. The source holder can also be taken out from or put into the evaporation chamber through a valve without breaking the vacuum in the system. The evaporation unit, i.e. tungsten boat plus a slit system which makes it possible to chose any desired source width, can be slid into the evaporation position through a valve in the bottom of the evaporation chamber without breaking the vacuum. The source holder can be slid through a hole in the evaporation unit towards the slit and placed in a fixed position which is reproducible to around 0.01 mm. The heating of the boat is made with DC current from the power supply which is also used for the magnetization. The vacuum in the evaporation chamber is better than 10^{-6} torr.

The vacuum system

The vacuum system consists of an Edwards one stage rotary pump with a displacement of 8.7 m^3/h and a permanent gas ultimate vacuum better than 0.005 torr and an Edwards vapour diffusion pump of the fractionating type with a pumping speed of 300 l/s and a total ultimate vacuum better than $5 \cdot 10^{-7}$ using Edwards Silicone oil No 705. The vacuum in the spectrometer chamber is better than $2 \cdot 10^{-5}$ torr after 1 hour of pumping.

Temperature control

In order to get a magnet field which is constant in time it is very important that the permanent magnets maintain constant temperature. Two covers made of plastic have therefore been placed outside each aluminum plate /see fig. 3/. The covers are connected to each other. Temperature controlled air is then allowed to circulate in the system.

X-ray equipment

A Philips PW1009 D.C. X-ray diffraction generator is available for the high tension to the X-ray tube. We have now two different tubes, one with copper anod and the other with molybdenum anod. They are both of Philips fine-focus type. The monochromatizing crystals are bought from Seifert. The crystals are made of quartz with reflecting plane $(10\bar{1}1)$.

Measurements of the magnetic field

The homogenity of the magnetic field has been measured for various magnitudes of fields.

We have found that the absolute deviation from the average value of the magnetic field is almost the same for 20 and 35 gauss and only slightly more for 100 gauss. Most of these deviations were found to be due to imperfections in the pole pieces. The greatest field gradient is less than

0.035 %/cm	B = 20 gauss
0.030 %/cm	B = 35 gauss
0.014 %/cm	B = 100 gauss

The field is homogenous up till the last 2 cm of the pole pieces. This excellent result is due to the extra airgaps.

Preliminary results

Since it is our purpose to detect low energy electrons (5-10 keV) we have been studying the sensitivity of many different emulsions. At this time we have got photolines with 2 different plates, namely Kodak SWR and CEA X-ray film. We are now going to test Ilford G5 and K5 emulsions.

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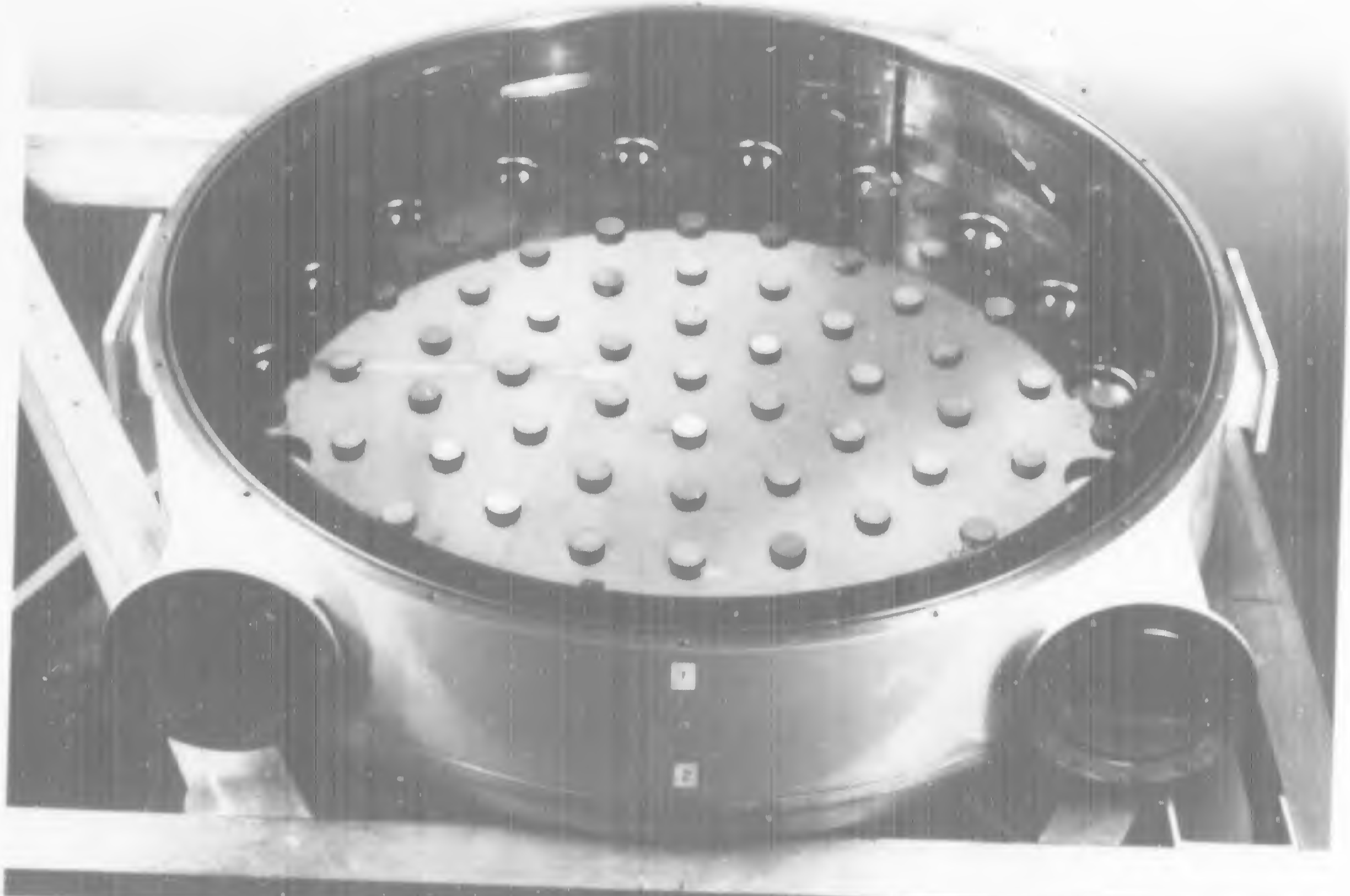

Fig. 1.

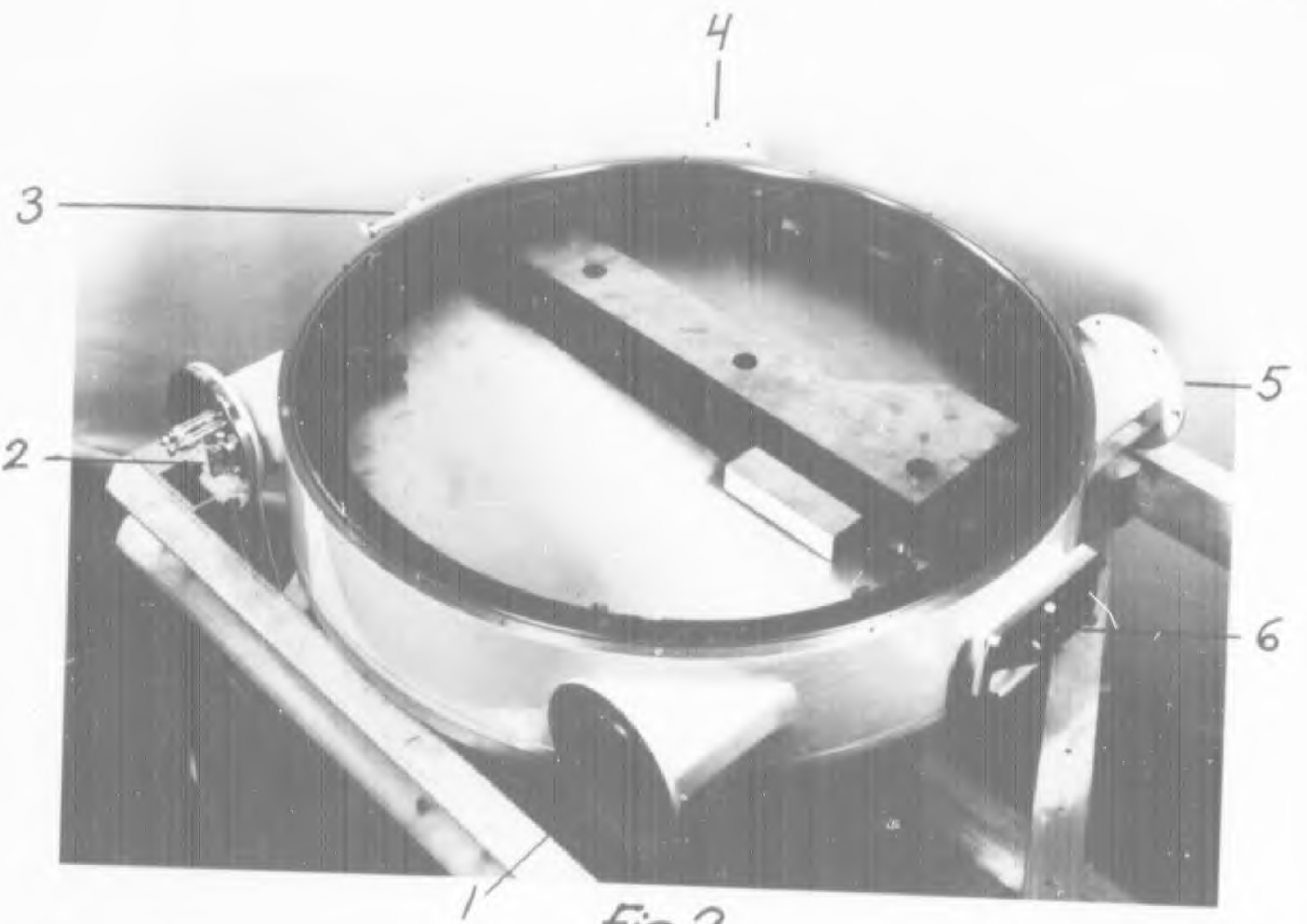

Fig. 2.

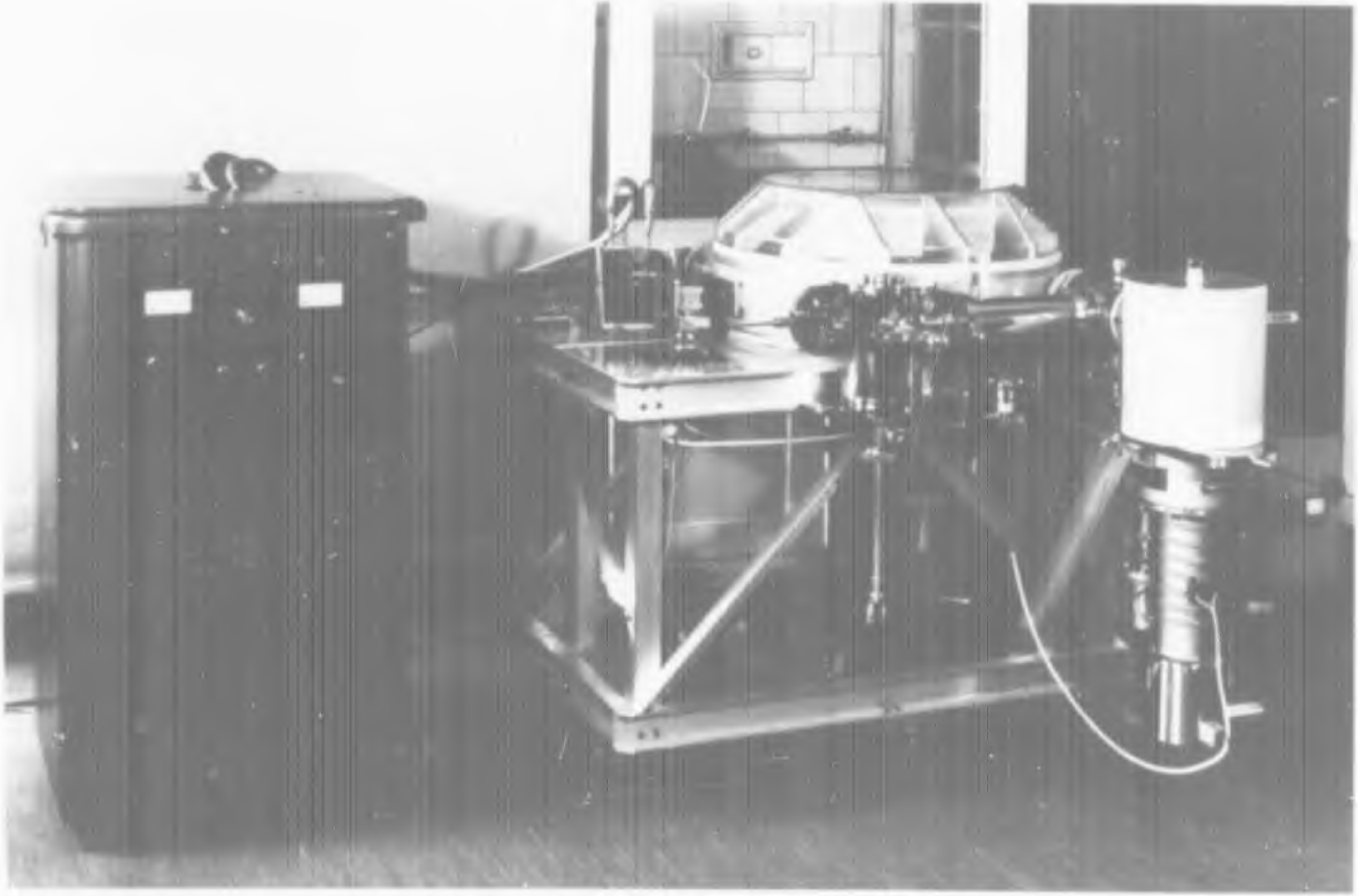

Fig. 3.

ANNEX

1. Personnel utilized during June 1, 1963 - May 31, 1964:

   Kai Siegbahn

   Carl Nordling

   Stig Hagström

   Anders Fahlman

   Sven Kumlin

   Gösta Larsson

   Jan-Olof Forsell

   Sune Isberg

2. Number of manhours expended of the contract:

   11,900.

   Expendable materials:

   10,300 Sw. crowns.