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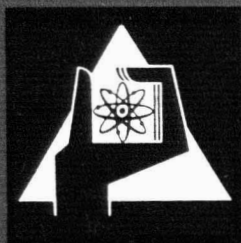
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A Very Flexible Pulsed Beam Spectrometer for Studying  
Neutron Scattering in Solids and Liquids

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Neutron Scattering in Solids and Liquids

by

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## 1. Introduction

Work carried out at research reactors during the last decade demonstrates that slow neutrons are a powerful tool for studying the properties of solids and liquids. A comparison of the neutron with other possible probes, for example photons, electrons or protons, shows easily its advantages.

Coherent neutron scattering offers excellent means for studying crystal and magnetic structures. But it is in crystal and liquid dynamics where the availability of neutrons has opened a new field of research. For structure investigations now various types of double-axis crystal spectrometers are in use as standard equipment at many reactors. For dynamic studies the beryllium filter technique, the triple-axis crystal spectrometer and different types of pulsed beam spectrometers have been developed.

The apparatus which will be discussed in this paper is of the pulsed beam spectrometer type. It was originally designed for scattering law measurements in isotropic materials [1], but it has proven to be also very useful for other types of experiments.

## 2. General features of the rotating-crystal time-of-flight spectrometer.

The arrangement is similar to that of a normal crystal monochromator, a set of Soller type collimators being mounted in front of and behind the single crystal. The crystal, however, rotates at high speed, so that the Bragg law will be fulfilled in each revolution only during very short time intervals. Energy resolution and time-width of the neutron pulse are determined by several parameters: collimation, geometrical dimensions and mosaic spread of the crystal and its speed of rotation. The quantitative relations cannot be discussed in detail here.

Fig. 1 shows the set-up of a crystal chopper that we have been operating for about two years now. The crystal is a 3.5 cm dia. aluminum cylinder, 8 cm in height, which is fixed directly to the shaft of a 75 watt, 4 pole hysteresis synchronous motor. The three-phase-split output of an audio-power-amplifier controlled by a variable frequency RC-oscillator feeds the motor. In most of the experiments done so far, the rotor ran at 14,500 rpm.

Fig. 1 illustrates the technical simplicity of the rotating crystal compared, for example, with a several-rotor chopper system. Only one compact rotor, the crystal, with dimensions of the utilized beam is necessary, and no phasing problem between different driving units exists.

If we compare the transmission of both systems under equal conditions, the result is that in the energy range below 0.5 eV the crystal is competitive in spite of its incomplete reflectivity. The reason for this is found in the two different mechanisms of monochromatization. Lattice parameters, Debye-Waller factors, mosaic spreads and absorption set the limits for crystals; for mechanical choppers a severe limit is given by the mechanical strength properties of the rotor material.

Two advantages of the rotating crystal are apparent:

- 1) The monochromatic beam is inclined to the reactor beam, which is an important point in background considerations.
- 2) Several monochromatic beams at different reflecting angles, i.e. with different neutron energies, can be used simultaneously. This particular feature largely enhances the utilization of a reactor beam hole.

Fig. 2 is a scheme of our present experimental arrangement with four different types of experiments. Before describing these in detail, a few words on the production of the monochromatic beams should be said.

### 3. Methods for filtering monochromatic beams

One disadvantage of the crystal rotor is that it does not only reflect neutrons of the desired energy. Higher orders reflected at the same planes and low intensity contamination cannot be suppressed completely. Although this is a question of the reactor spectrum available and of the crystal planes used, we found the quality of the monochromatic beam quite satisfactory for scattering experiments whenever a (111) plane was used and the neutron energy higher than 0.05 eV. For lower energies, additional means for cleaning the beam are necessary. Three different methods have been used for doing this:

- 1) large single crystals of quartz and bismuth as filters,
- 2) total reflection of neutrons at polished metal surfaces,
- 3) a rotating collimator of the mechanical velocity selector type.

Method (1) is simple, but unfortunately connected with a considerable loss in intensity even when the filter is operating at low temperature. The second method is most efficient for energies below 0.01 eV (3 A). A pair of Soller collimators (collimation 10'), inclined with an angle greater than twice the geometrical collimation, is used. The efficiency depends largely on the quality of the polished surfaces. Although we have used so far only mechanically polished surfaces, a transmission of about 50 % and an improvement of peak-to-background ratio in the monochromatic beam of a factor 10 has been reached. For higher energies a rotating collimator was built (method C). Fig. 3 shows the layout. The rotor has a length of 50 cm, an outer diameter of 42 cm and 222 straight slits, 10 cm in height and 0.3 cm in mean width with slit walls 0.03 cm thick, made of stainless steel. Speed of rotation is between 3600 and 7000 rpm. Rotation speed and inclination of the rotor axis relative to the beam determine the transmitted energy. We have used this system down to 0.016 eV where we get a transmission of 50 % for the monochromatic pulse as well as an improvement of the order of 20 in both peak-to-background and first-to-second order intensity ratios.

#### 4. Scattering law measurements in isotropic materials

The purpose of this arrangement is the measurement of the double differential scattering cross section in polycrystalline solids, liquids or gases, for a wide range of momentum and energy transfers. From this the so called "scattering law", which is only a function of the dynamic properties of the scatterer, can be extracted. Besides its basic interest, the knowledge of the scattering law is of importance for thermalization calculations in reactor materials.

Fig. 4 shows an artist's sketch of the device. The neutron detectors are placed on the upper half of a vertical circle at a distance of two metres from the sample.

The whole set-up is able to turn around the axis of the crystal, so that the incident neutron energy can be changed without much trouble. Detectors can be separately moved on the circle. A total of 15 different detector positions can be used simultaneously. At present two different types of detectors are in use:

1. Scintillation detectors with  $\text{Li}^6\text{F-ZnS}$  scintillators [2], 0.5 mm thick and 5 inch diameter;
2.  $\text{He}^3$  counters, 1 inch diameter, forming banks with an effective area of  $155 \text{ cm}^2$ .

To calibrate the detectors vanadium scattering experiments are done. Inelastic scattering, absorption and, to some extent, multiple scattering are taken into account in the processing of the vanadium data.

In this type of experiment, a relatively large beam cross section is used, the samples being 4.5 cm wide and up to 7 cm high. With the second collimator a typical pulse width at the sample is 20  $\mu\text{sec}$ .

In order to get the highest intensity available, up to now most of the experiments have been done without this second collimator. Under these conditions, the intensity at the sample, at 0.04 eV, is  $400 \text{ n/cm}^2\text{sec}$ . In order to reduce multiple scattering contributions, samples scattering only about 10 % of the incident neutrons are used.

To eliminate background, measurements without sample are done. Due to the rotational symmetry of the crystal monochromator, a large part of the background is constant in time or uncorrelated and can, therefore, be easily corrected.

Typical results of three day runs are shown in Fig. 5. The lower curve is for vanadium, the upper one for graphite. Incident energy was 0.042 eV, scattering angle  $125^\circ$ , channel width 8  $\mu\text{sec}$ .

##### 5. Arrangement for measuring small momentum transfers

Slow neutrons offer a valuable tool for studying diffusion processes in liquids on a microscopic scale. Experiments of this type done so far [3] indicate that measurements at smaller momentum transfers with a higher energy resolution are necessary to get conclusive information concerning the different proposed diffusion models.

For studying small energy and momentum transfers we have set up an experiment using a monochromatic beam from the rotating crystal in the energy range from 5 to 10 meV. The length of the flight path is 3.5 metres. The bank has a total of 60 detector positions between

$1^\circ$  and  $30^\circ$ . At each position up to three 1 inch  $\text{BF}_3$  or  $\text{He}^3$  counters, 8 inch in length, can be mounted. With this arrangement, an energy resolution of about 1 % at  $5.10^{-3}$  eV is attained.

A remarkable technical feature of this arrangement is that in the case of three counter tubes in series, i.e. behind each other, the corrections for time-of-flight differences in the tubes are made by a short programme in the on-line computer which is used with the spectrometer and which will be described in sect. 8. Using a computer programme, it should also be possible to optimize the peak to background ratio for different energy regions.

#### 6. Arrangement for the determination of dispersion laws in single crystals.

Today mainly the triple-axis spectrometer [4] is used for measuring phonon frequencies in coherent scattering single crystals. This spectrometer could definitely not compete with time-of-flight methods if there were not the possibility of mechanical programming and its use in the "constant Q" and other special methods. Modern time-of-flight techniques, however, offer great potentialities also in this direction.

In our present experimental arrangement six  $\text{He}^3$  counters, with a diameter of 1 inch and an active length of 4 inch., were placed side by side at a distance of 2 metres from the crystal under investigation.

The experimental procedure, the "scattering surface" method [5], reminds of the method of "successive approximations" [6]. Should phonons be measured in a certain direction, a phonon wavevector is selected and the associated phonon frequency estimated on the basis of some previous knowledge. The parameters of the arrangement are set so that an inner counter meets this choice. If the measured phonon does not fall in the selected direction, phonons measured in slightly different ones with the outer counters, help to construct a part of the scattering surface whose intersection with the desired direction gives the phonon looked for. Although this is the procedure now accepted, ways could be thought of which can combine the measurements at neighbouring angles in a different manner, for instance, to keep the end-point of the wavevector transfer in a certain direction. With time-of-flight this is merely a reduction of data which can be done effec-

tively with the on-line computer.

### 7. Diffractometer using time-of-flight

In a provisional set-up, we have tried to use the time-of-flight method for structure research. This means using a pulsed monochromatic beam from the crystal chopper and determining scattering angles by time-of-flight. A scheme of the arrangement is given in Fig. 6. A wide range of angles of interest can be covered in one run. The geometrical arrangement of the detectors (we use at present two  $\text{BF}_3$  counters, 1 inch diameter and 117 cm active length) determines the angular resolution. A typical pattern for lead is shown in Fig. 7.

Although this method at present is not competitive with the conventional diffractometer, it should be of interest for pulsed reactors and it has the advantage of separating the second and higher orders by time-of-flight so that lower incident energies can be used. Finally, this method is suitable for studying relaxation effects in structures.

### 8. Acquisition and reduction of data with the on-line computer

The effectiveness of the various experimental facilities described depends largely on the availability of a capable data acquisition system. More than 20,000 time channels are needed simultaneously. At the FR 2 reactor we have met these and also the requirements of other multiparameter experiments by the installation of a multiple input data acquisition system [7] using a Control Data 160-A computer, with 8 K. core memory, as a central unit.

Besides the time-of-flight spectrometers described in this paper, three two-parameter experiments in nuclear physics use this system.

The events recorded by all the time-of-flight detectors mentioned above are fed to a multiparameter coding unit which converts them to digital words of 20 bits length and which consists of:

1. a binary decoder of the detector number and experiment,
2. a time-of-flight digitizer with the possibility of digitizing 256, 512 or 1024 time channels with widths from 1 to 64  $\mu\text{sec}$ ,
3. a device for recording the status of the reactor and of the scattering samples.



The connection between this unit and a multiplexer unit involving a 4 x 24 bit derandomizer at the central station is made by an one-24-bit-word buffer station which identifies all information coming from the rotating crystal spectrometer.

Transfer of the data from the derandomizer to the computer memory, checking and eventually reduction by means of a subroutine, is governed by one control program. Depending on the number of channels required, the information is stored in the memory or written on magnetic tape.

At present, totalizing is performed in the CDC 160-A and output is in the form of punched paper tape. In a not too distant future, the IBM compatible tapes will be fed directly into the central Karlsruhe IBM 7070 for further data processing.

A flexible display, generated by the computer, is available [8] at the place of the experiment. Using digital switches, a wide variety of different display patterns can be chosen.

#### Acknowledgements

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#### References

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Legends to figures:

Fig. 1: Crystal chopper with cylindrical aluminum crystal.

Fig. 2: Schematic diagram of the rotating crystal spectrometer arrangement. The detector set-ups 2 - 4, here drawn in the horizontal plane, are in vertical planes.

Fig. 3: Scheme of the rotating collimator.

Fig. 4: Sketch of the device for scattering law measurements.

Fig. 5: Typical results with vanadium and graphite.

Fig. 6: Scheme of the time-of-flight diffractometer.

Fig. 7: Typical diffraction pattern.

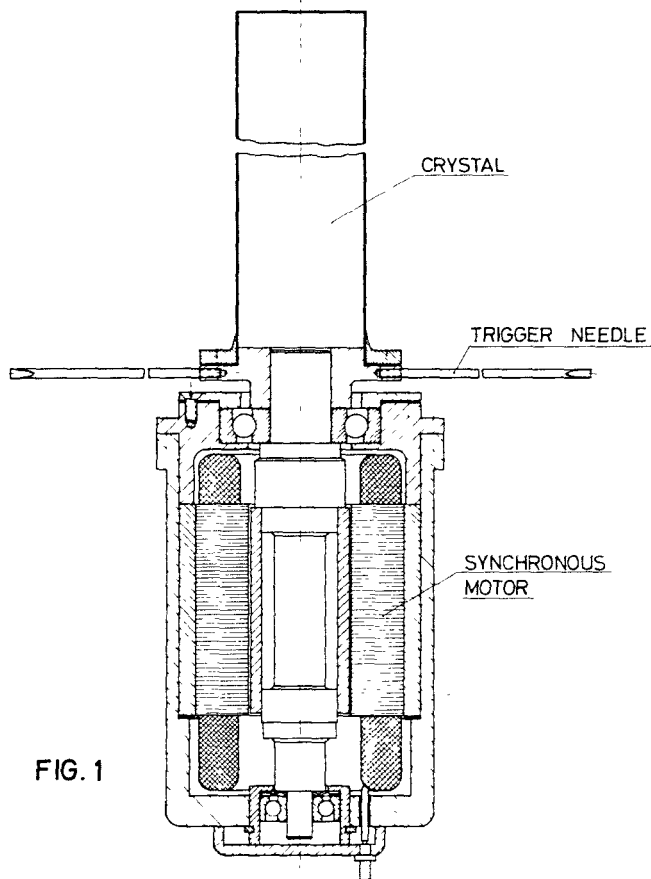


FIG. 1

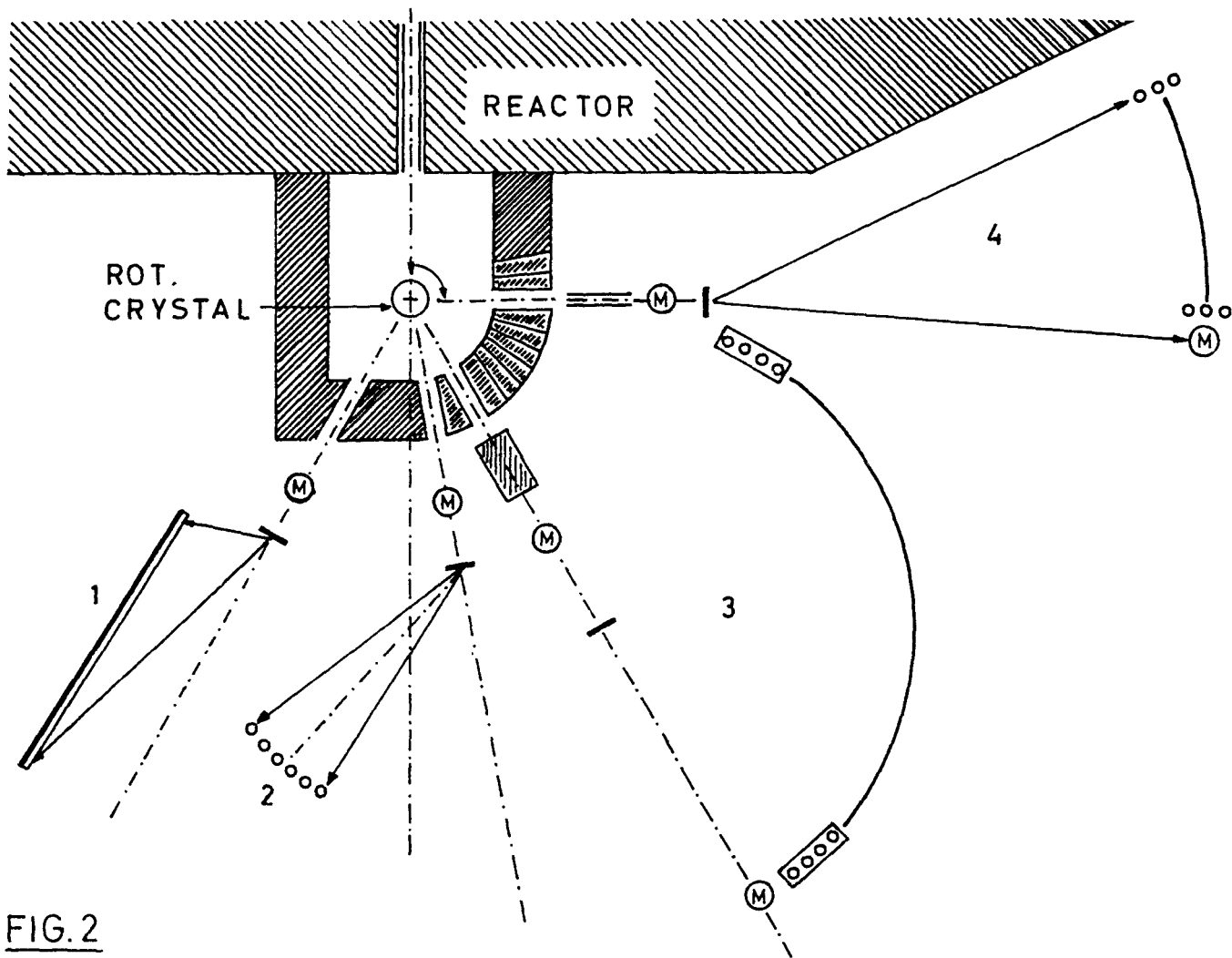


FIG. 2

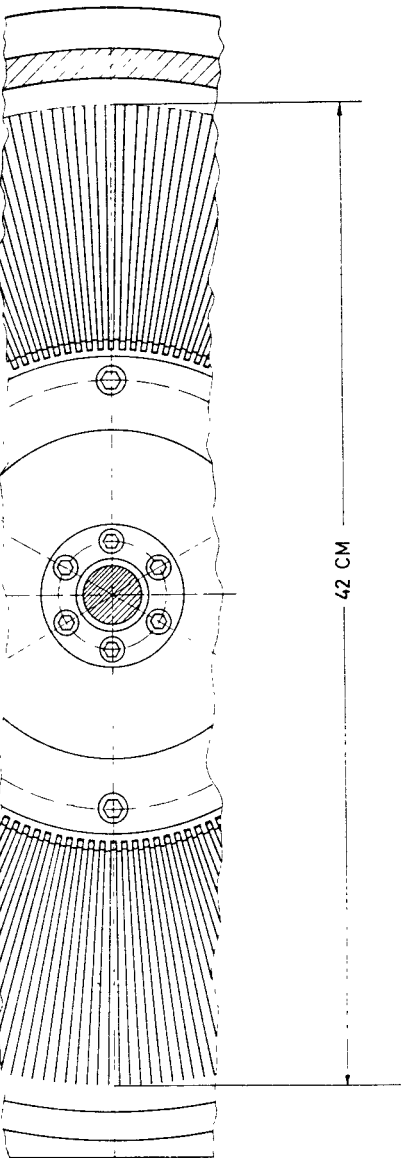
1 = DIFRACTOMETER                      2 = DISPERSION LAW SPECTROMETER

3 = SCATTERING LAW SPECTROMETER

4 = LOW MOMENTUM TRANSFER SPECTROMETER

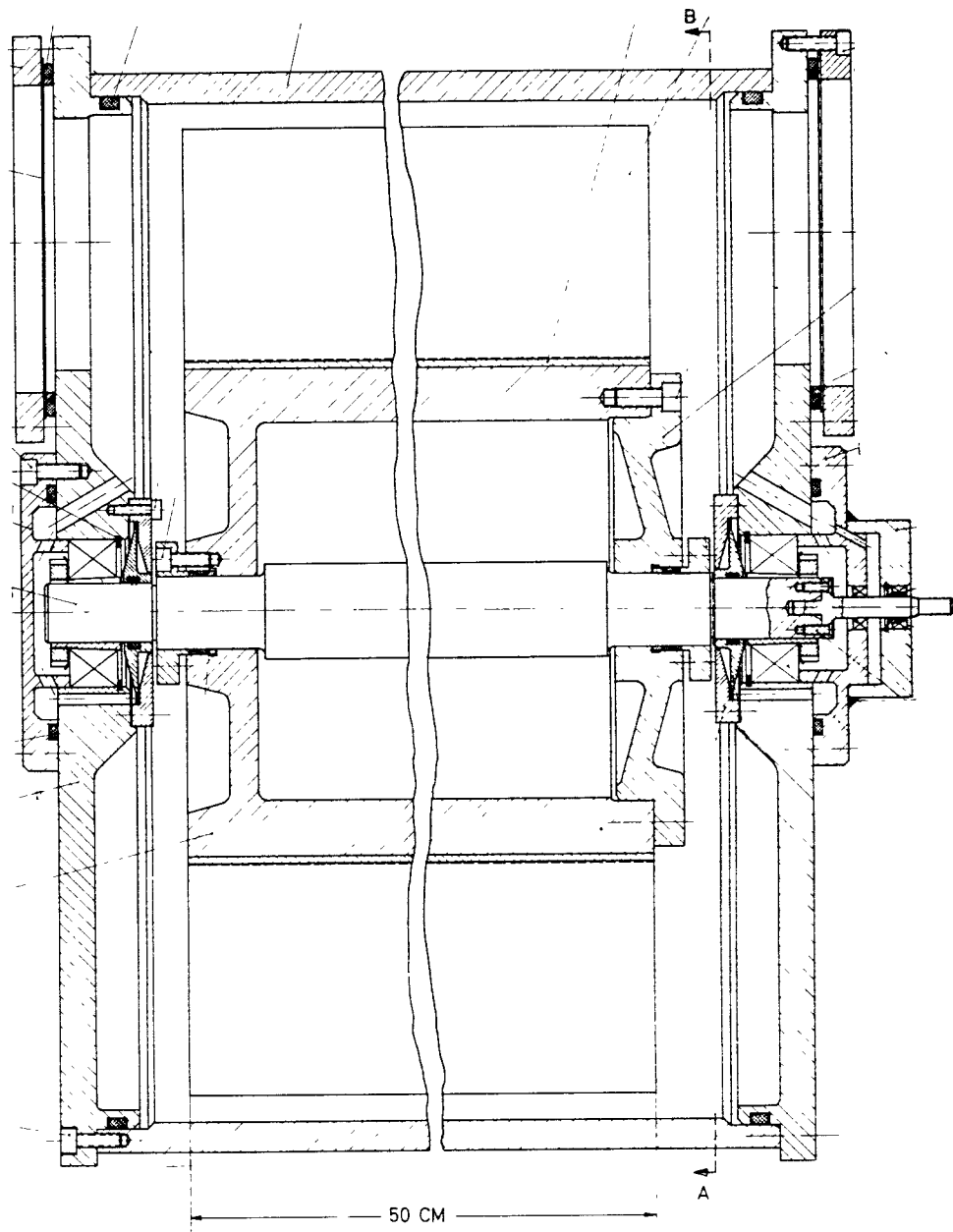
Ⓜ = MONITOR, ○ = DETECTOR, | = SAMPLE

SCHNITT A-B



42 CM

FIG. 3



50 CM

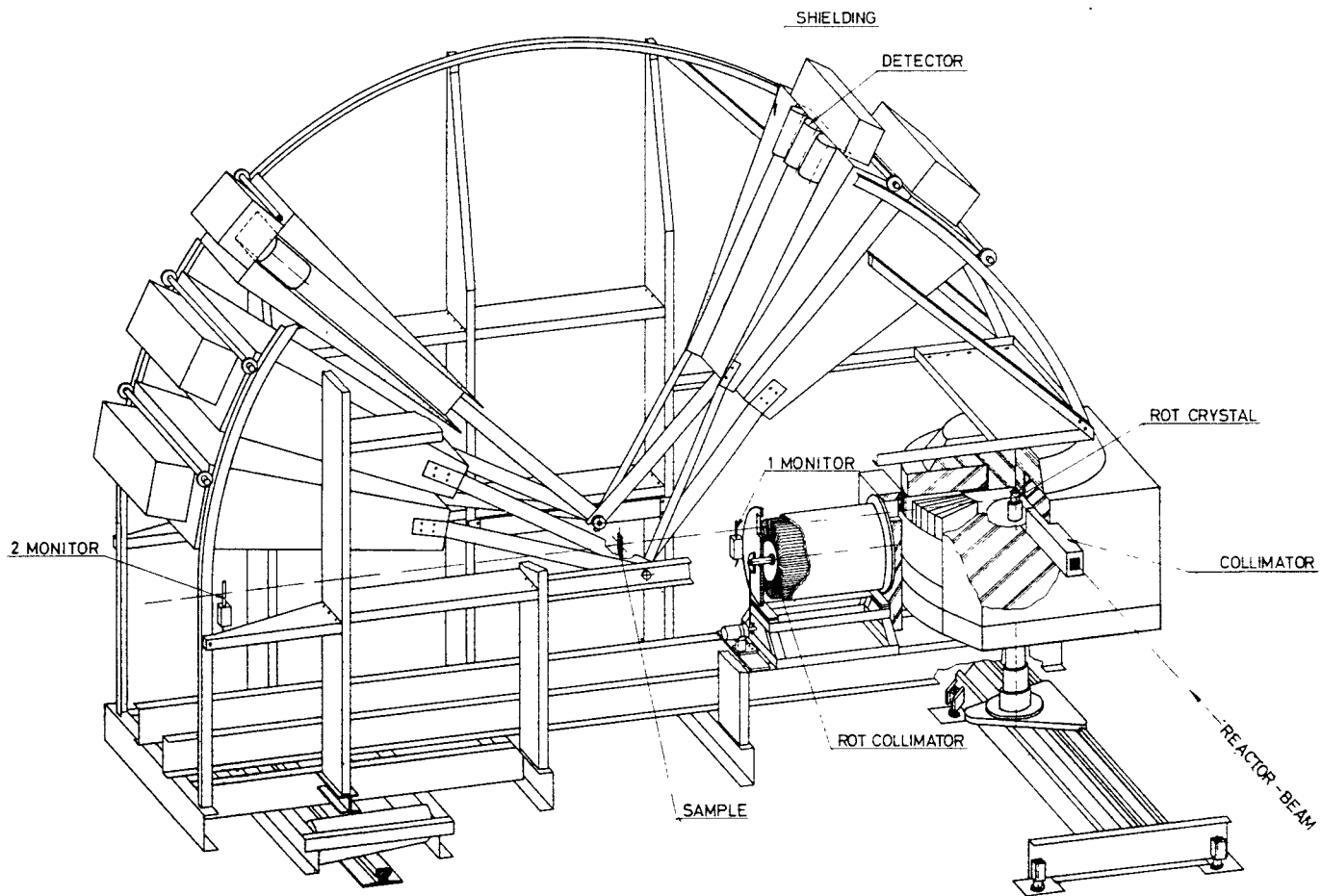


FIG. 4

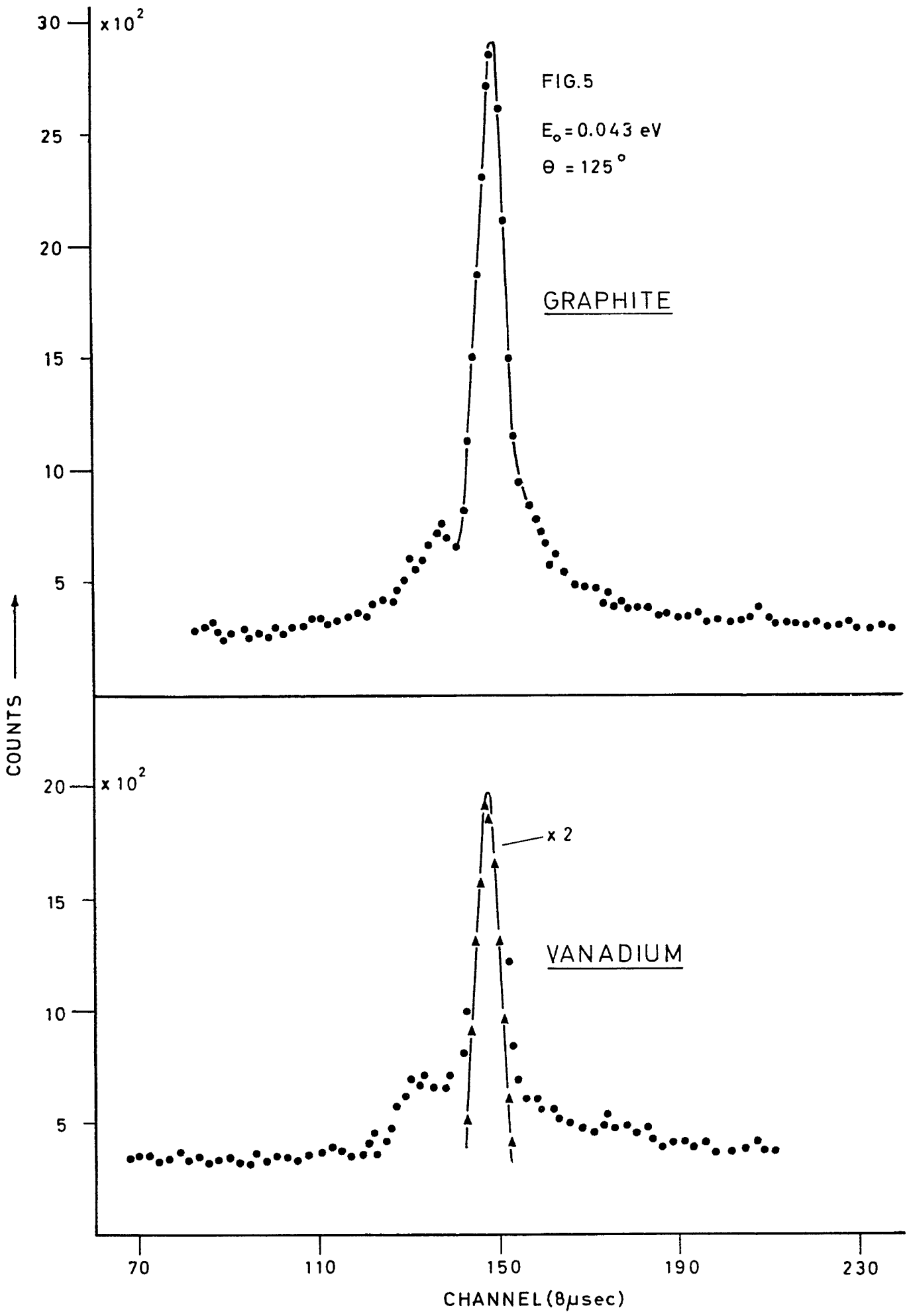


FIG. 6

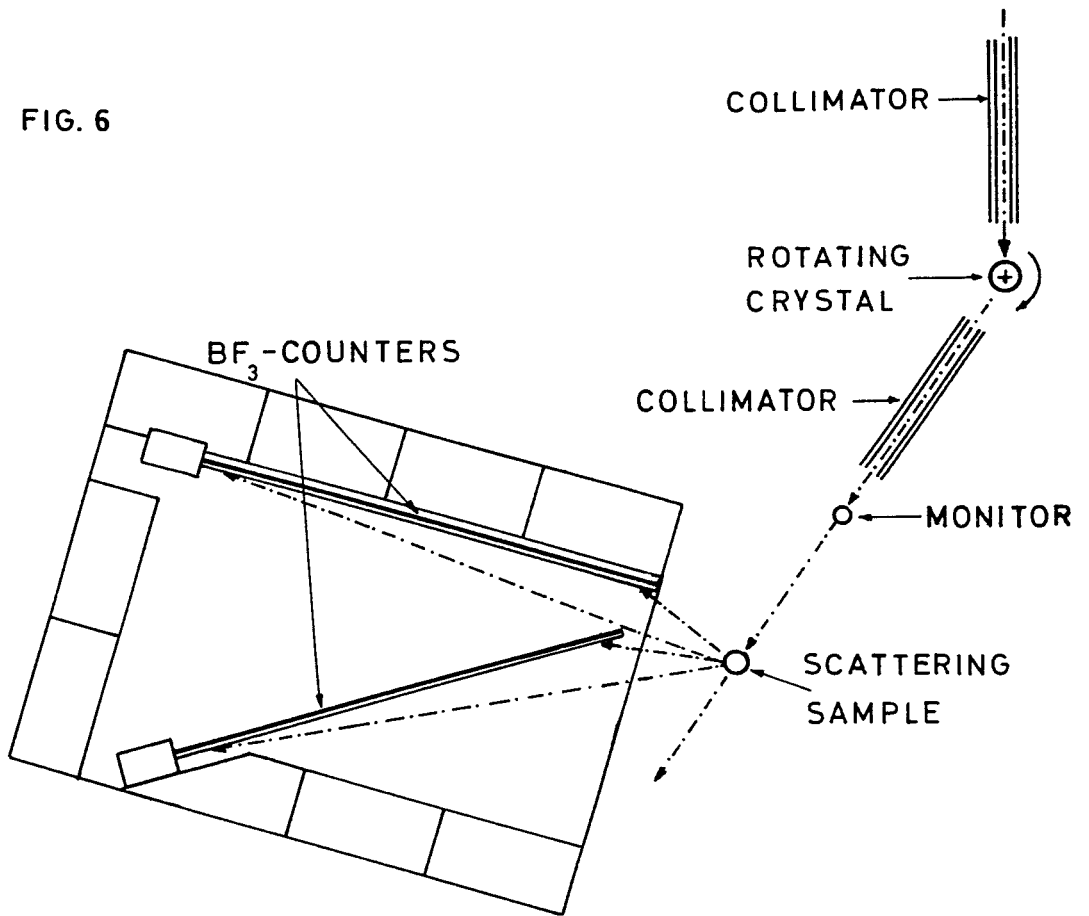


FIG. 7

$$\lambda_0 = 1,42 \text{ \AA}$$

