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RESEARCH PERFORMED AT THE ET-RR-1 REACTOR USING THE NEUTRON SCATTERING EQUIPMENT*

M. Adib, R.M.A. Maayouf and A. Abdel-Kawy
Reactor and Neutron Physics Department
Nuclear Research Centre, A.E.A., Cairo, Egypt

(* The Equipment was Supplied by the IAEA According to the Technical Assistance Project EGY/1/11/10)

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Abstract

This report represents the results of studies and measurements, performed at the ET-RR-1 reactor, using the neutron scattering equipment supplied by the IAEA according to the technical assistance project EGY/1/11/10.

The results of these studies, starting in 1980 and continuing to date, are discussed; the use of the equipment, both as a neutron monochromator and fixed scattering angle spectrometer, is also assessed.

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INTRODUCTION

According to the IAEA neutron scattering project EGY/1/11/10, still agreed about in 1976, the equibment were installed infront of one of the ET-RR-1 reactor horizontal channels in 1980. The project's equibment consists of a double rotor system and the electronics required for it.

The system consists of two rotors, suspended in magnetic field, spinning at speeds up to 16000 rpm with a constant phase angle relative to each other and producing bursts of monochromatic neutrons at the sample. Each of the rotors, 32 cm in diameter and 27 Kg in weight, has two slits to produce two neutron bursts per revolution. The slits are with radius of curvature 65.65 cm and 7x10 sq.mm cross-sectional area.

The installation of the equibment, at the ET-RR-1 reactor, was followed by studies and measurements, using the system, with the following intentions :

A) To use the system as a double rotor neutron monochromator.

B) To use the system's equibment as a fixed-scattering angle spectrometer.

The present work represents the results of these studies, starting from 1980 to date.

2. STUDIES CONCERNING THE DOUBLE ROTOR SYSTEM

The studies concerning the double rotor system started with theoretical calculations /1/, in order to assess its use, as neutron monochromator, for studying the dynamics of solids and liquids.

This was followed by measurements of the main parameters of the double rotor neutron monochromator system. A schematic diagram of the geometrical arrangement of the double rotor, as installed infront of one of the ET-RR-1 reactor horizontal channels, is given in Fig.1 for scattering angles 0° and 90°. Neutrons are emitted, from the reactor's channel, through an in-pile collimator(60 cm long and with beam hole 2.5 cm in diameter) made from lead, paraffin and boric acid. The double rotor system is surrounded by shielding from boron carbide and paraffin (see Fig.1). The distance between the rotors could be varied from 1 - 3 meters. The sample is located at distance 40 cm from the centre of the second rotor. Two ^{3}He - neutron detector batteries are mounted at the ends of two mobile flight tubes (each 1.6 m in length) which could be situated at different angles with the neutron beam direction.

The double rotor system consists of two similar rotors; each of them is mounted on its mobile platform.

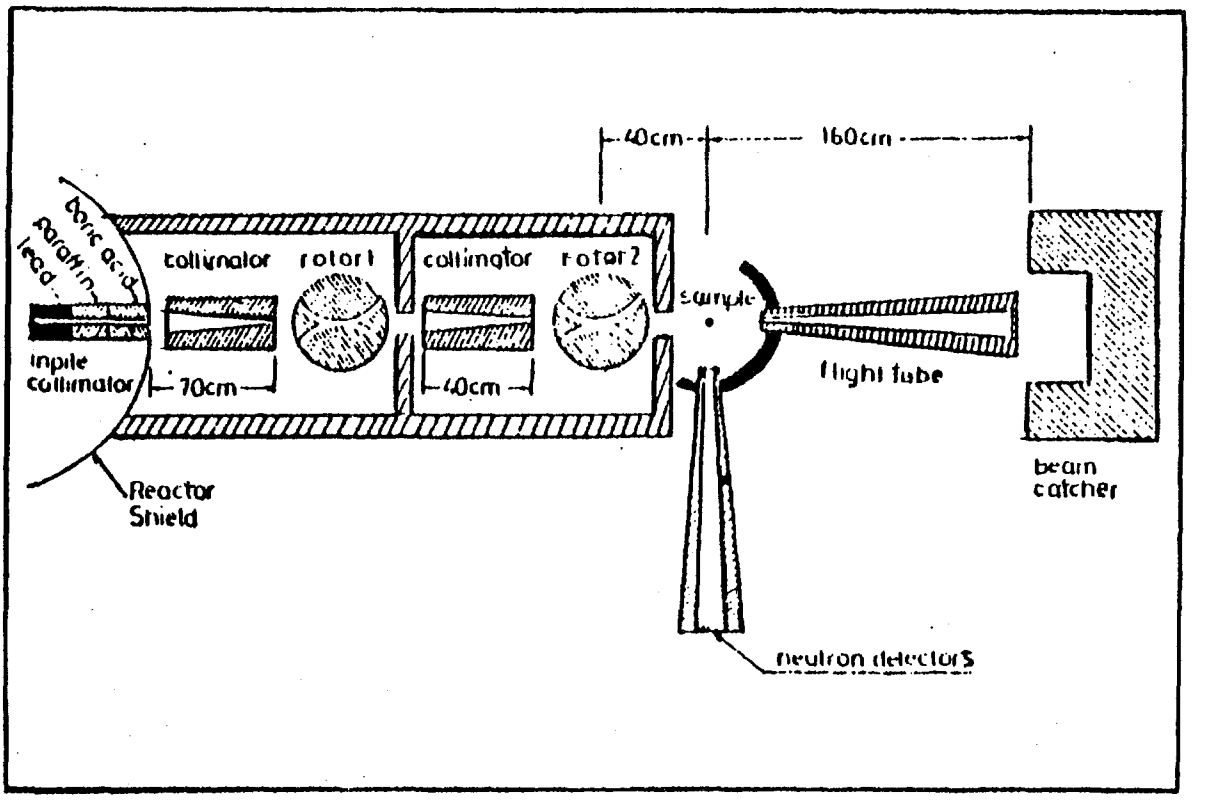

Fig. 1 : The Geometrical Arrangement of the Double Rotor System.

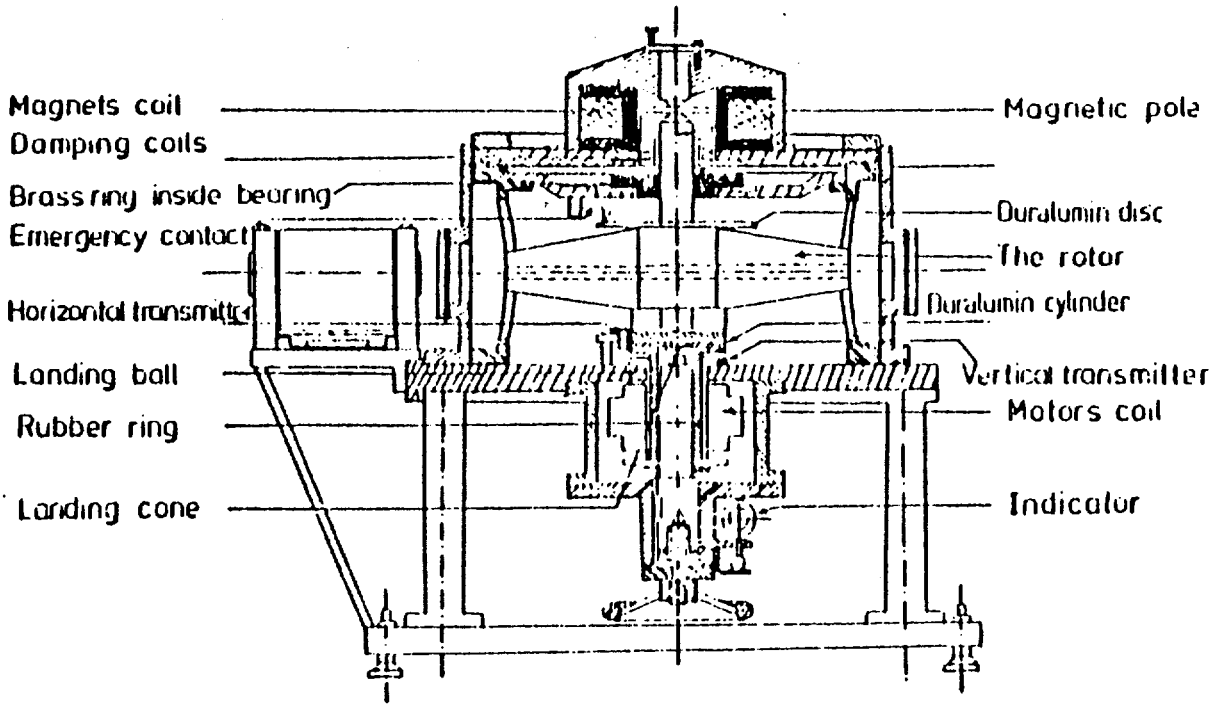

Fig. 2 : Schematic Diagram of One of the Rotors.

The general view of the rotor system is given in Fig.2. The rotor (1) is installed in evacuated chamber, in order to minimize its friction during rotation. The rotor is made from nickel alloy and has two slits. The magnetic pole (2) is manufactured from magnetic iron material; this helps its suspension in the magnetic field. The rotor tail (3), duralaluminium, is for rotating the rotor when a rotating magnetic field is induced in the motor's coil (4). The steel ball (5) is used for the rotor's landing while the position of the landing point is controlled by the indicator (6). The transmitter (7) feeds the current into the magnet's coil (8), in a negative feedback loop. Consequently it controlls the height level of the suspended rotor in space within an accuracy better than 5 μm. The horizontal position of the rotor is controlled by both the transmitter (9) and damping coils (10). The mechanical contact (11), along with a light duralaluminium disc (12) is used for avoiding collision of the rotor with the magnet's pole in case of emergency. In such case the cone (14), made from brunze and surrounded by a rubber ring (13), is used for damping the vibrations of the rotor, while the horizontal vibra-

tions are limited by a brass ring (15). The safety of rotation is ensured while the rotor is rotating up to a maximum velocity 16000 rpm and during long time of operation. More details about the double rotor system, as well as the spectra transmitted through one rotor system at different rotations and the jitter of the phase between the two rotors, are given in Ref./2/.

The jitter phase between the rotors is considered one of the most important parameters of the double rotor system. Because of the damping unit used, the jitter of the phase between the two rotors, reported in Ref./2/, had a double peak behaviour around the zero phase and did not exceed 1.5 μs. Accordingly a new damping unit has been designed specially to decrease such fluctuations. The jitter of the phase between the two rotors, measured with the new damping unit, was reported along with parameters of the system in Ref./3/ ; it is represented in Fig.3,

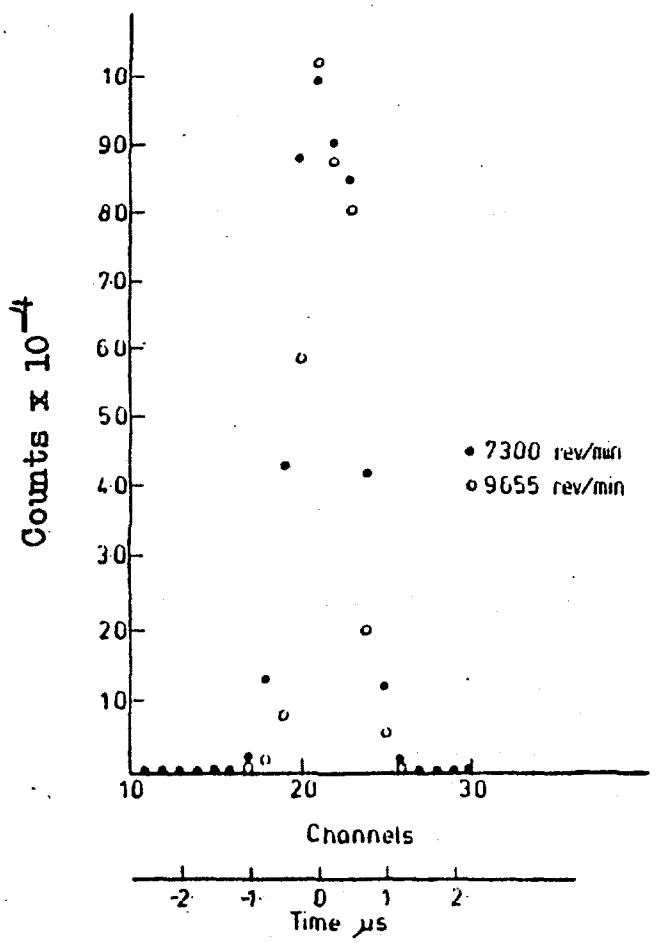

Fig. 3 : Jitter Phase Between the Rotors.

where it can be noticed that the double peak has disappeared, The new value of the jitter phase does not exceed ± 1.0 μs.

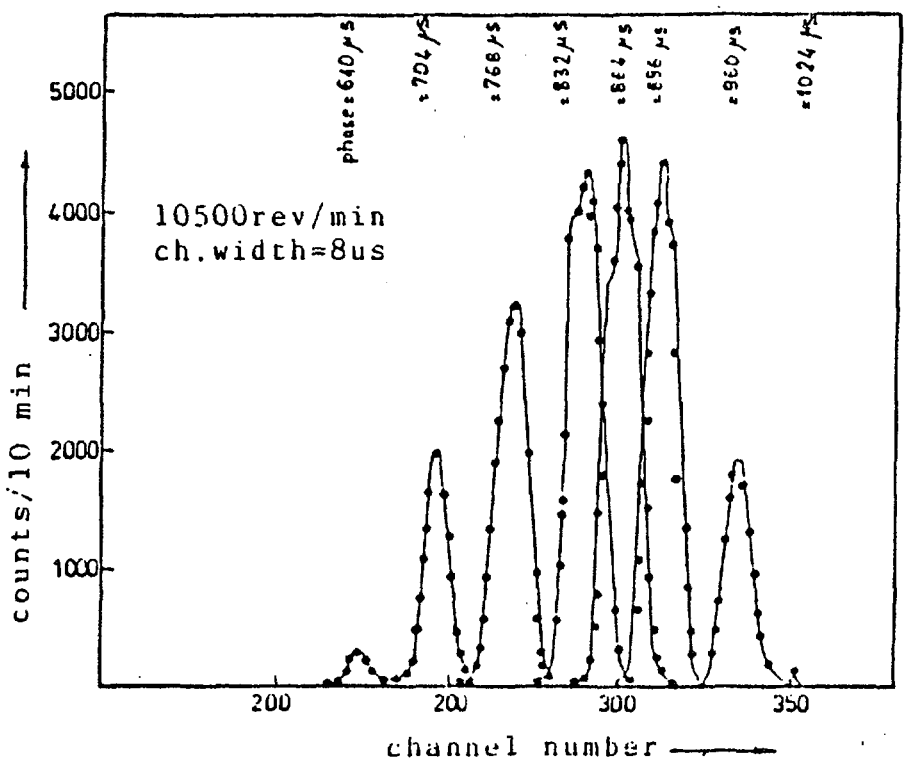

Fig. 4 : Observed Neutron Monochromatic Beam.

The neutron monochromatic beam, typically observed at a rotation rate of 10500 rpm is represented in Fig. 4 for different values of the phase between the two rotors. The time-of-flight is measured from the centre of the first rotor to the detector where the flight path is 350 cm. The maximum transmission for neutrons of wavelength 2.74 Å was found to correspond to 864 μs difference in phase between the rotors.

Figure 5 shows the time distribution of neutrons in the pulse at the detector position, along with the calculated one for maximum transmission /4/. One can notice that both distributions are in good agreement. This means that the standard deviation of the pulses at a sample position 40 cm from the centre of the second rotor will be 11 μs. The neutron time-of-flight distribution in pulse obtained for maximum transmission at rotation rates 10500 and 11650 rpm are represented in

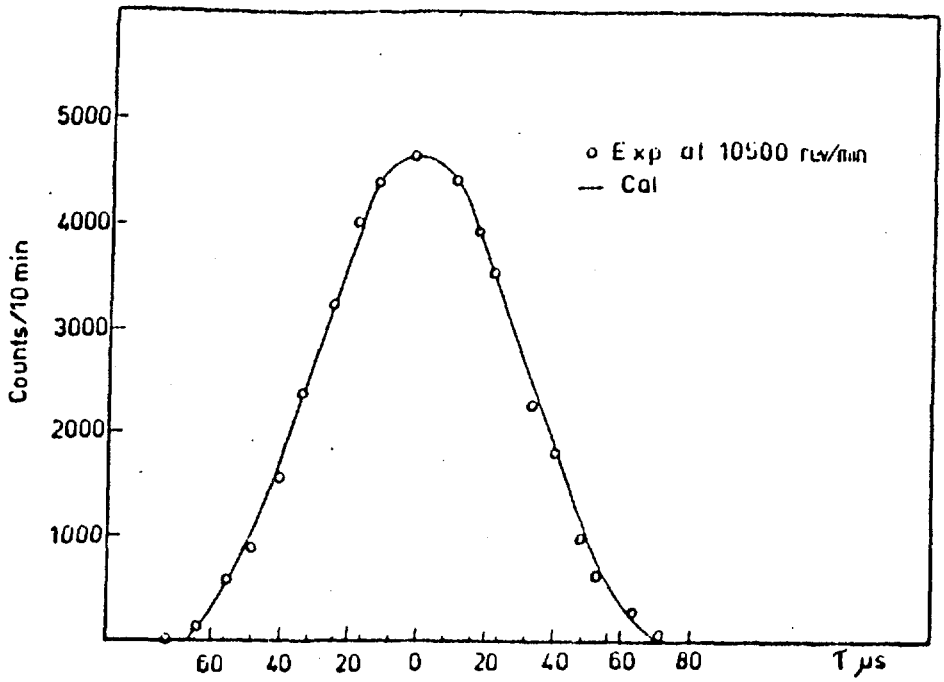

Fig. 5 : Time Distribution of Neutrons at Detector Position.

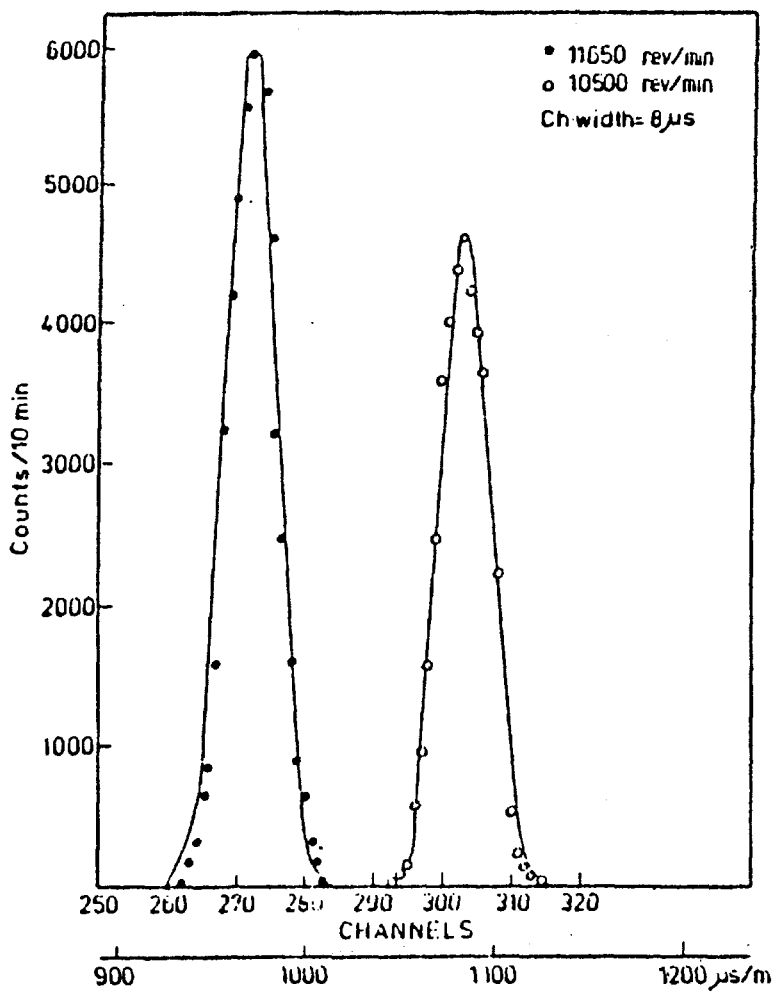

Fig. 6 : Neutron TOF Distributions.

Fig . 6. The solid lines are the distributions calculated following Ref./4/. One can see that good agreement exists between both the experimentally measured and calculated distributions. The wavelength resolution $\Delta\lambda/\lambda$, obtained from Fig. 6 at $\lambda = 2.74$ Å , was 3.2%. The observed intensity of the monochromatic neutrons, obtained using the double rotor system, of wavelengths (2.74 ± 0.09) Å and (2.47 ± 0.08) Å were found to be 66.8 and 92.2 n/s respectively; the intensities calculated, considering both the detector efficiency and attenuation in air, were 370 and 507 n/s respectively.

Because of the low intensity of the monochromatic neutrons, obtained with the present set-up, it is required either to use neutron guide collimators or multislit rotors instead of the two slit rotors, presently used, in order to increase the neutron intensity. The expected increase in neutron intensity, when using the neutron guide collimators, will not be sufficient for carrying out neutron inelastic scattering experiments within reasonable accuracy ; because the cross-sectional area of the present dimensions of the rotor slits will provide neutron beams with divergence higher than the cretical angle of the total internal reflection from the material of the neutron guide collimator at the available wavelengths. This makes the application of multislit rotors the only suitable way for raising the intensity of monochromatic neutrons.

3. STUDIES CONCERNING THE FIXED SCATTERING ANGLE SPECTROMETER

The studies concerning the use of the double rotor equibment as a fixed scattering angle spectrometer almost started with those ones mentioned in the previous section. This was further supported by the low intensity of the obtained monochromatic neutrons. The basic idea of the fixed scattering angle spectrometer is based on applying the method reported in Ref. /5/, for studying the crystal

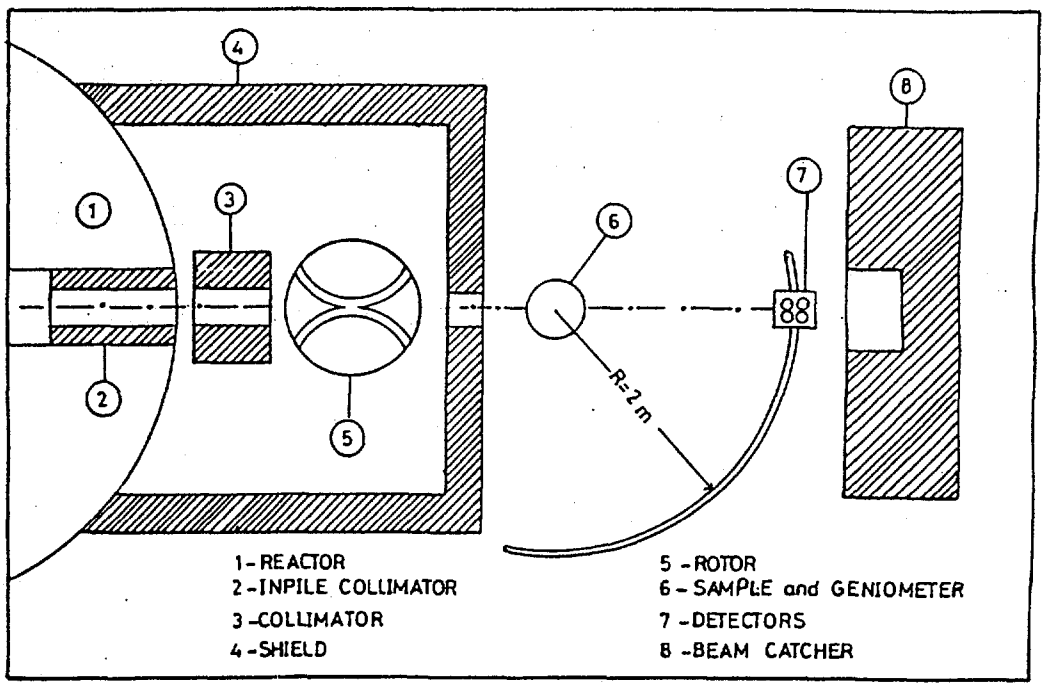

Fig. 7 : Schematic of the Fixed Scattering Angle Spectrometer.

structure with neutrons. This could be done by diffraction of timed bursts of polychromatic beam of neutrons by the crystal sample and measure, by time-of-flight, the intensity and position of the diffraction peaks at a constant scattering angle. The present arrangement for using the double rotor equibment as a fixed scattering angle spectrometer was reported, along with the parameters measured and calculated for the spectrometer, in Ref. /6/.

The horizontal view of the general arrangement of the fixed scattering angle spectrometer,applies a single rotor, is represented in Fig. 7.

It has been proved /6/ that the wavelength resolution for the fixed scattering angle spectrometer, is a function of h/wr, while the intensity of neutrons with maximum transmission is a function of h^2/wr; h, w and r are the width of the rotor's slit, its angular velocity and radius respectively. This makes the neutron intensity, for a given wavelength resolution and radius of the rotor, higher at high angular velocities. The magnetic suspension of the rotor makes it able to spin around the free axis of inertion, and no dynamic forces can

arise. Moreover the friction in such case is negligible, since the rotation can easily be realized in a vacuum chamber. The calculations /6/ has proved that the construction of the present rotor system, as well as its dimensions, allow maximum transmission for neutrons of wavelengths 0.1809 nm (E = 0.025 eV) at resolution $\Delta\lambda/\lambda$ = 2.0% and w = 16000 rpm. More details about the experimental checkup of the spectrometer's parameters are given in Ref. /6/ .

The reactor spectra, transmitted through Be sample and measured at rotor velocities ranging between 500-9100 rpm are represented in Fig. 8. It is noticeable that the sharp Bragg cut-offs, due to Be planes with Miller indices (002) and (100), with the respective double interplaner distances 0.358 and 0.396 nm, appear in all the measured spectra at the same channels; not depending on the rotor's rotation rate.

The value of the half time width Δt of the resolution function was determined from the spectrum measured

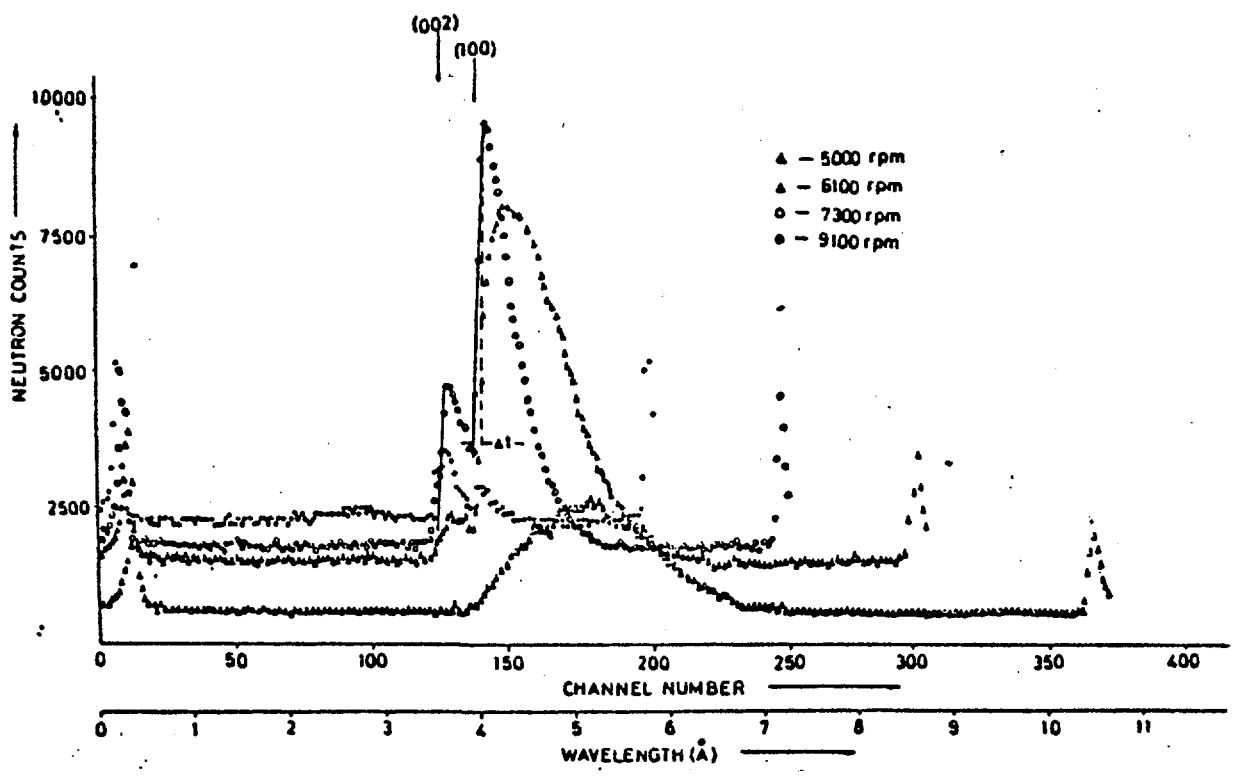

Fig. 8 : The Observed Be Filtered Neutron Spectra.

at 7300 rpm and found to be (42 ± 4) μ sec, this value is in good agreement with the calculated value 40.6μ sec. This yields /6/ a value 1.82% for the wavelength resolution at $\lambda = 0.4$ nm.

The background spectra, measured with a Cd sheet placed infront of the rotor's slit, at different rotation rates were almost the same /6/, this means that the background is almost constant.

4. MEASUREMENTS USING THE FIXED SCATTERING ANGLE SPECTROMETER

It was found that the fixed scattering angle spectrometer could provide polychromatic neutrons of reasonable intensity, within the wavelength band from 0.15 nm - 0.75 nm, with high wavelength resolution and almost constant background. However the effect to background ratio is low at wavelengths higher than 0.5 nm. This is due to the fact that the thickness of the rotor's material, transmitted by the neutron beam, is small. The effect to background ratio could be increased, by applying a synchronized rotating collimator- rotor system. Regardless, the present fixed scattering angle spectrometer, which applies only one rotor, was used successfully, as will be shown, for studying basic characteristics of single crystals.

4.1 Measurements Performed for Pyrolytic Graphite Crystals.

The filtering characteristics of pyrolytic graphite (PG) crystal have been reported by several authors /7-11/. The study of the filtering properties of PG /12/ lead to the conclusion that it can also serve as an effective higher order filter for neutron wavelengths between 0.37 - 0.43 nm.
Besides, it was shown /7/, that the highly aligned PG may be tuned for optimum scattering of second-order neutrons of wavelengths between 0.112-0.425 nm by adjusting the filter in an appropriate orientation. However, all the reported measurements, for PG crystal, have been carried

out using triple-axis spectrometers; consequently the measured transmission data are essentially corrected /7/ for higher-order reflections. However transmission measurements, using time-of-flight (TOF) spectrometers are free from such corrections. Transmission measurements were performed at the ET-RR-1 reactor /13/, applying the two TOF spectrometers reported in Ref. /14,15/, for a PG crystal. The measurements covered the wavelength band from 0.03-0.5 nm, and at 7 different angles between 0° and 63.7° inclusively. These measurements supported that the PG crystal is indispensable filter /13/.

The experimental measurements, performed at the ET-RR-1 reactor with the fixed scattering angle spectrometer, were for studying the characteristics of the PG crystal when used either as a neutron monochromator or analyzer for long wavelengths. Therefore, measurements were performed, using the transmission method, for both integrated intensity and the wavelength spread of neutrons reflected from the PG crystal at wavelengths between 0.4 nm - 0.65 nm. The PG crystal is a plate 8 mm thick, 30x67 mm^2 cross-sectional area and cut along the (002) plane which is parallel to the 30x67 mm^2 side. The PG crystal was produced by Union Carbide Corporation. The transmission measurements has the advantage, over the usual reflection method, that they are carried out under the same geometrical conditions, using the same detector. Besides, the very small neutron absorption cross-section ($<$ 6 mb) makes it possible to evaluate the value of the reflectivity, from the measured crystal's transmission T, simply as (1-T).

Fig. 9 represents the obtained neutron reflectivity (1-T) of the PG crystal's plane, as a function of neutron wavelength. The neutron reflectivity is represented, for the (002) plane, at 7 different values of the angle ψ between the neutron beam direction, and the normal to the sample's surface, i.e. the normal to the (002) plane. It is noticeable that the position of the neutron distribution's peak, due to the Bragg reflection from the (002) plane, shifts towards small wavelengths and with broadening of the dis-

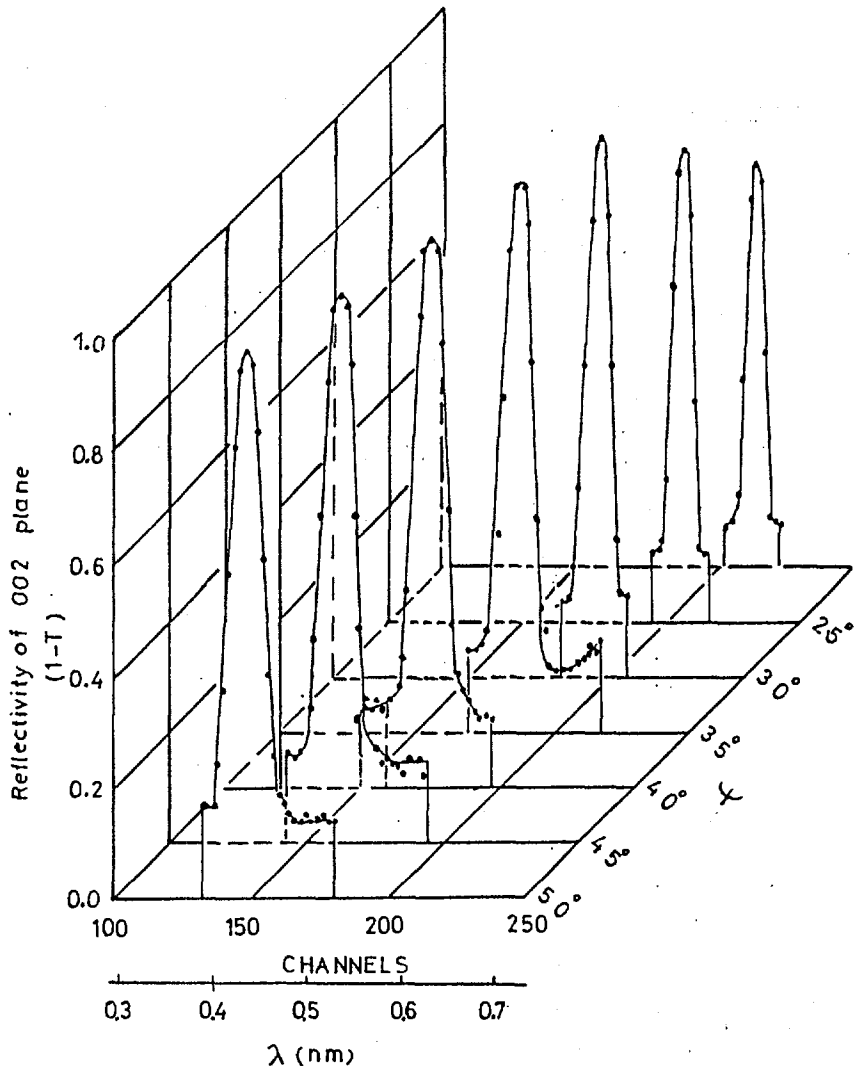

Fig. 9 : The Neutron Reflectivities, Obtained for PG at Different Angles.

tributions width. This shows the effect of the spectrometer's resolution, as well as ψ , on the observed distribution of reflected neutrons.

The Gaussian epproximation /16/ was used in order to compute the halfwidth of the observed distribution, which is a convenient quantity for comparison with the experiment; the value of the dispersion of the observed gaussian distribution σ was then calculated applying the formula given in /17/. The resulting values of σ^2 are represented in Fig. 10 , as a function of $\sin^2\psi$. The solid line is the best fitting of σ^2, using the least square method. The mosaic spread of the used PG sample was deduced from the slope of this line, and found to be $(4.00 \pm 0.07)^\circ$.

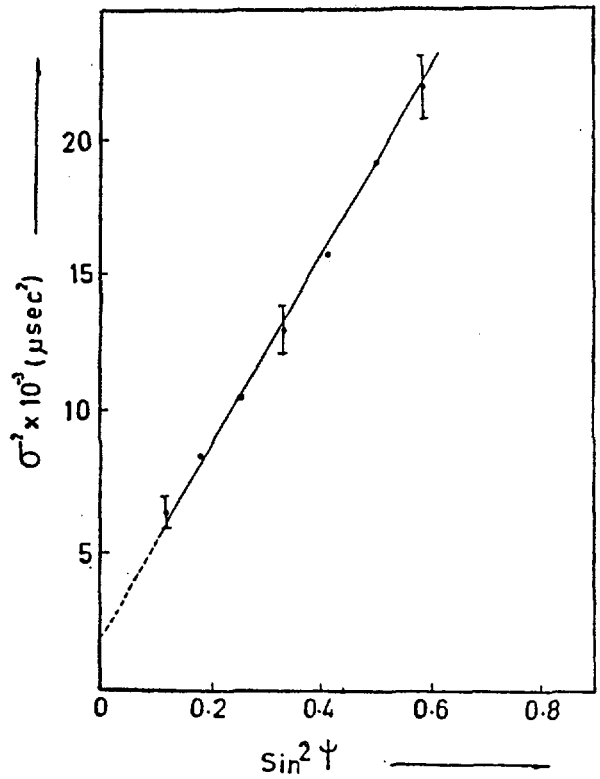

Fig.10: The Time Dispersion of the Distribution of Neutrons Reflected From PG.

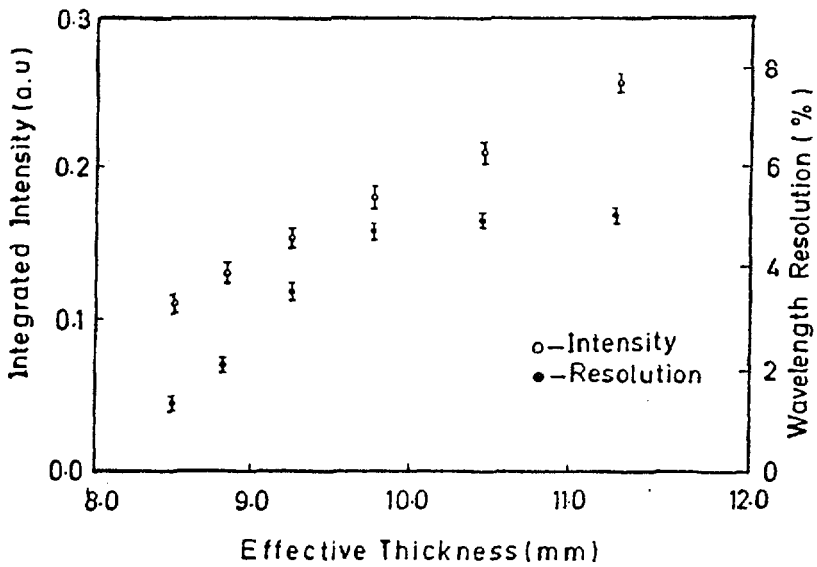

Fig.11 : The Intensity and Resolution of the Neutron Beam Reflected From PG, at Different Sample Thicknesses.

The integrated intensity of neutrons reflected from the (002) plane, at each angle ψ , was computed as the area under the (1-T) peak being corrected for the incident neutron flux (assumed to be Maxwellian one with neutron gas temperature T = 330 K /18/), the transmission fuction of the spectrometer and the efficiency of the neutron detector. Fig. 11 represents the computed integrated in-

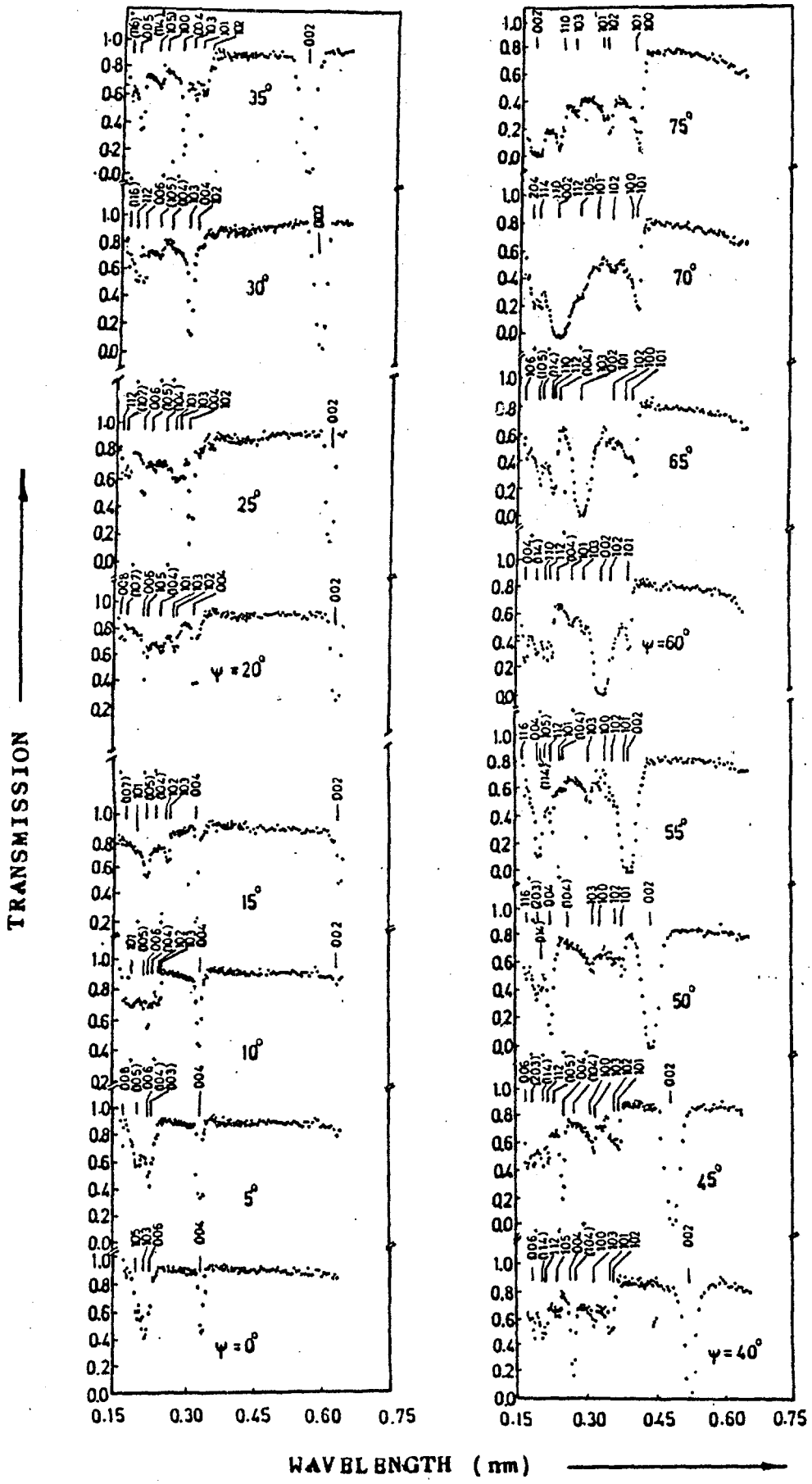

Fig.12: The PG Neutron Transmission Measured at Different Reflection Angles

tensities (open circles) versus the effective sample thickness $t_{eff} = 8/\cos\psi$ mm. In the same figure are also represented (closed circles) the values of the wavelength resolution $\Delta\lambda/\lambda$ of neutrons reflected from the (002) plane at different angles ψ . It is noticeable that the behaviour of the integrated intensity values is in good agreement with that one predicted in Ref. /8/ for idealy imperfect crystal; the integrated intensity reaches almost a maximum value, at $\psi = 35°$, for neutrons reflected from the (002) plane with wavelength ~ 0.55 nm and resolution $\Delta\lambda/\lambda = 5.0\%$.

In order to investigate the filtering characteristics of the PG crystal and to verify experimentally the tuned intervals for optimum scattering of second-order neutrons, predicted in Ref. /4/, the neutron transmission of the PG crystal was measured, by the spectrometer, in the wavelength range from 0.15 nm - 0.65 nm, and at different values of the angle ψ . The results of measurements /19/ are displayed in Fig. 12. It was concluded /19/, from Fig. 12 , that the PG crystal can be used as a second-order filter at the wavelength intervals : (0.29 ± 0.018) nm, (0.232 ± 0.017) nm, (0.214 ± 0.003)nm and (0.185 ± 0.003) nm.

The fixed scattering angle spectrometer can also be used for determination of the mosaic spread; this could be done by recording all neutrons reflected from a certain crystal plane, under Bragg condition. This method /17/ is simple and alike the x-rays, could be efficient for studying the crystal's mosaic spread of light elements /17/.

The schematic diagram of the experimental arrangement used for determination of the mosaic spread is given in Fig. 13. A large number of neutron detectors is used, see Fig. 13, in order to record all the neutrons reflected from the crystal's plane. The required number of detectors /17/, depends mainly on both the value of the mosaic spread and the divergence of the incident neutron beam. In order to decrease the number of the used detectors and con-

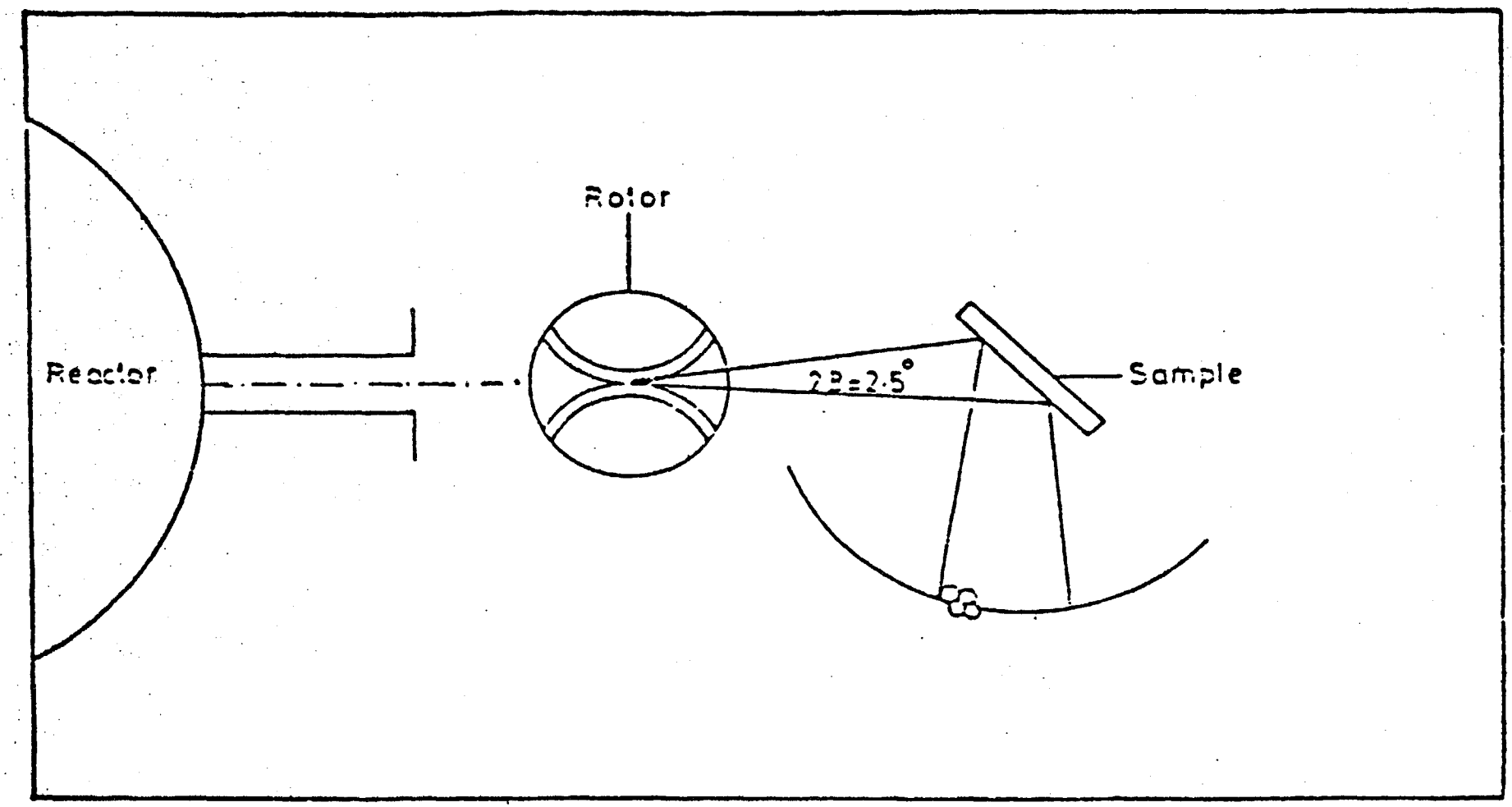

Fig. 13 : Schematic of the Arrangement Used for Mosaic Spread Determination.

sequently the background level, a battery of four similar detectors, mounted on a holder which could be moved on an arc, is used during the measurements. Thus the measurements are carried out in steps, moving the detector battery in order to cover all the angular range subtended by reflected neutrons. All the steps are carried out under the same operating conditions. The summation of these steps, taken channel by channel should provide, after subtraction of the background, the required wavelength distribution of reflected neutrons.

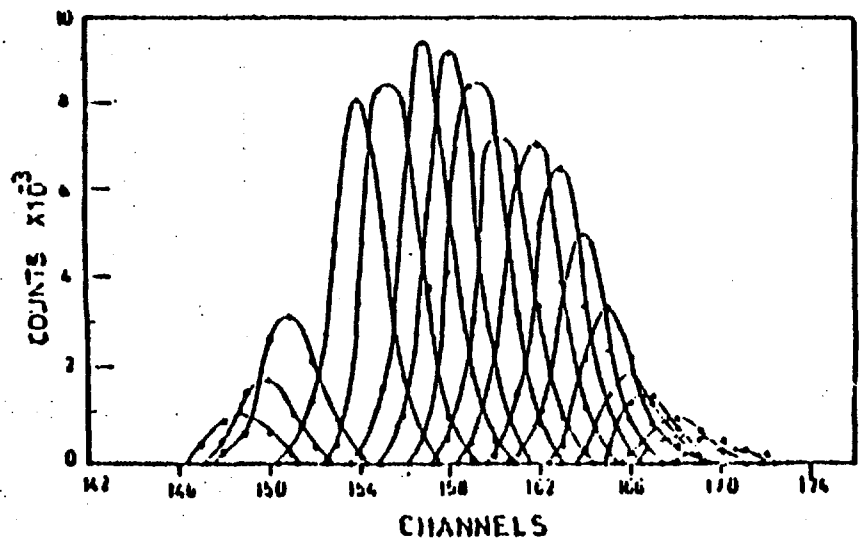

Fig.14: Neutrons Reflected From the (002) Plane of PG at Different Steps.

The detectors used, are ^{3}He of diameter 1.7 cm and 17 cm effective length. The four detector battery could cover an angle 0.97°. The observed distributions of neutrons reflected from the (002) plane, of the PG crystal, versus channel number and at different positions of the detector battry on the arc of the flight path, are represented in Fig. 14 . The distribution resulting from the summation, after subtraction of the background, is displayed in Fig. 15. The solid curve, in Fig. 15, is the best Gaussian fit calculated /17/ for a time dispersion $\sigma = (112 \pm 5)$ μsec. The mosaic spread of the PG crystal was calculated, using the equation given in /17/, for the operating conditions used during the measurements /17/.

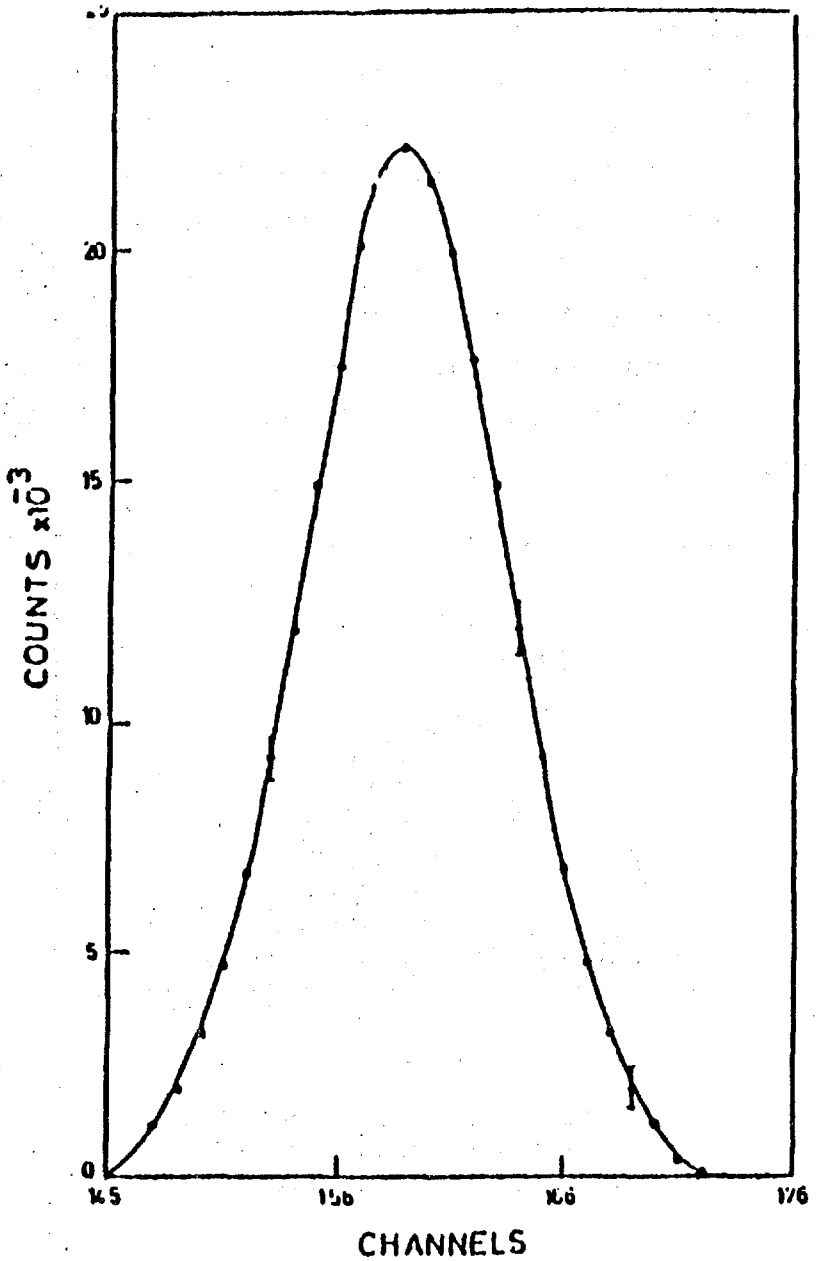

Fig.15: The Distribution of Neutrons Reflected From (002) Plane of PG.

The resulting value of the mosaic spread, due to neutrons reflected from the (002) plane is (3.60±0.16)°, while the value (3.67 ± 0.18)° is deduced for the (004) plane. Both values are consistent with each other, and with the value (4.00 ± 0.07)°, mentioned before, deduced from transmission measurements. Such good agreement proves the adequacy of the method applied for determination of the mosaic spread.

The mosaic spread of the Zn single crystal was also determined using the same method, used for the PG crystal. Accordingly, the distribution of neutrons reflected from the (002) plane of the Zn crystal was measured with the fixed scattering angle spectrometer, at rotor's speed 8230 rpm, the width of the multichannel time analyzer was 24.4 μ sec, and the Zn crystal was fixed at an angle $\psi = (46.4 \pm 0.1)°$ with respect to the neutron beam direction, i.e. with respect to the normal to the (002) plane. The reflected neutrons were measured in 0.97° steps.

The observed distributions of reflected neutrons, as measured at different steps, are represented in Fig. 16. The summation of the number of reflected neutrons, detected for the same channel number at different steps, after subtraction of the background, resulted with the distribution represented in Fig. 17. The solid curve , given in Fig.17 is the best Gaussian fit, calculated

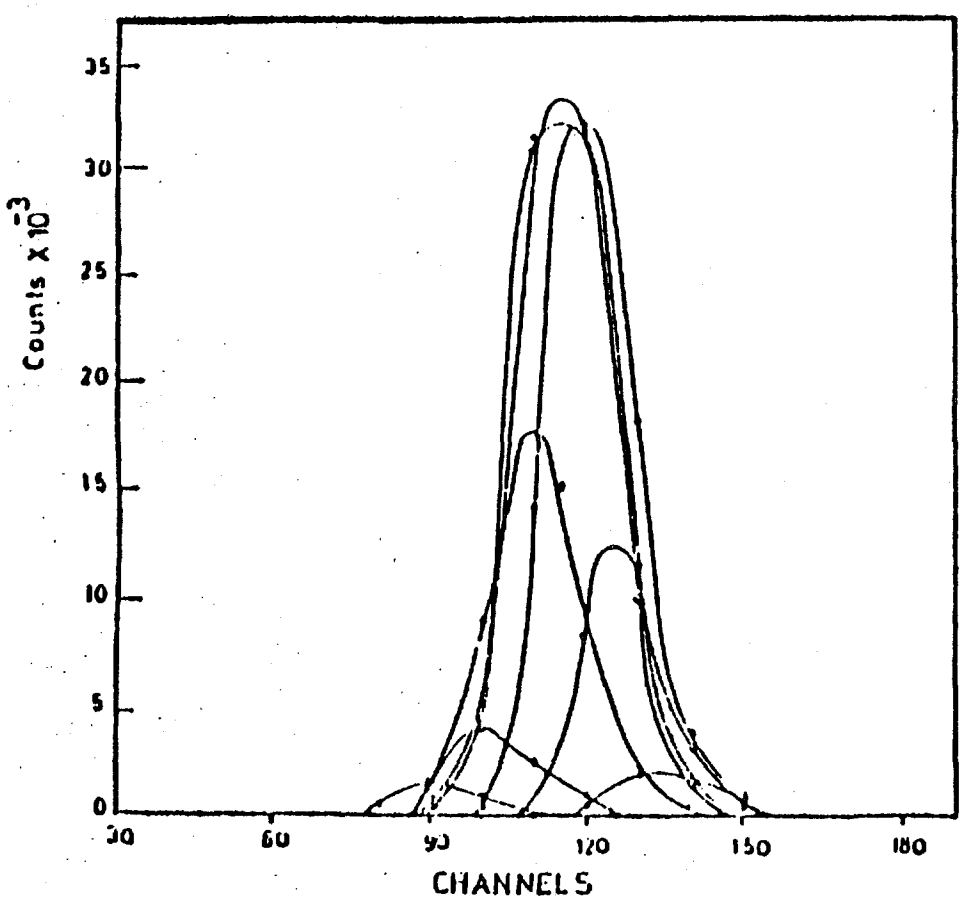

Fig.16: Neutrons Reflected From the (002) Plane of Zn at Different Steps.

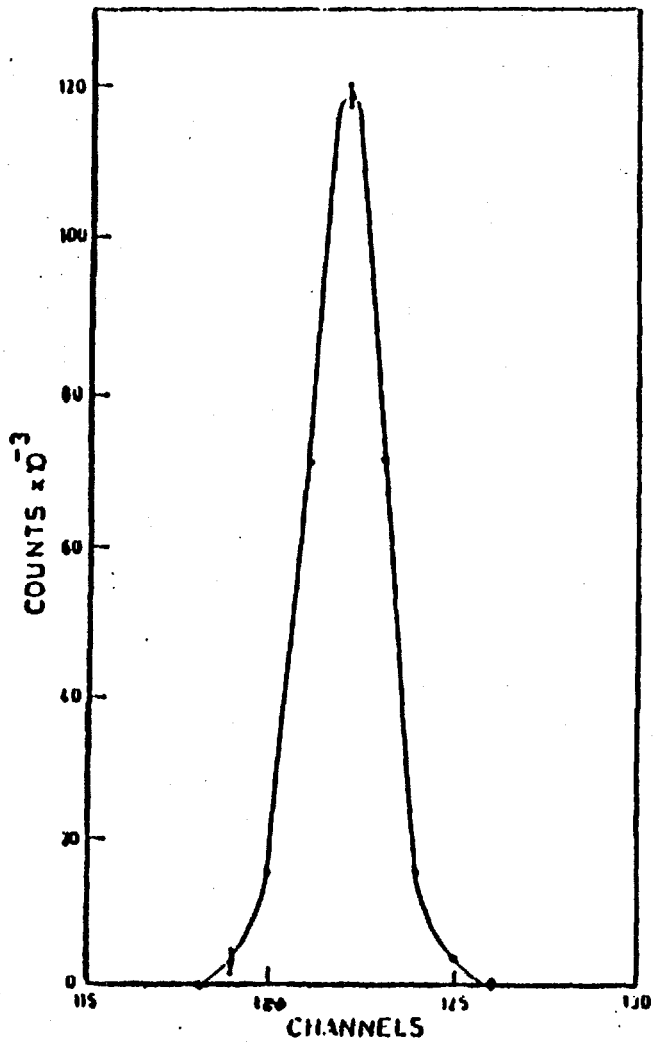

Fig.17: The Distribution of Neutrons Reflected From (002) Plane of Zn

as described in /17/. The value of the mosaic spread, for the Zn crystal, was found to be (10'± 6'). The order of this value is in good agreement with that one given by the manufacturer.

4.2 Current Measurements with the Spectrometer

At the time being measurements are carried out at the ET-RR-1 reactor, concerning the reflectivity characteristics of Cu monocrystals cut along the (111) and (200) planes. The measurements are carried out, with the fixed scattering angle spectrometer, for studying the use of such single crystals both as neutron monochromators and analyzers.

Measurements, concerning both the design and reflectivity characteristics of neutron mirrors are also carried out using the fixed scattering angle spectrometer. The measurements are carried out using thin films, of different thicknesses, coated on glass plates. The thin films are of both natural and enriched ^{58}Ni. The measurements should help in choosing the proper thickness, as well as the reflectivity characteristics, required for the design of neutron guide collimators, to be used, with the neutron scattering equibment, at the ET-RR-1 reactor.

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