**SOME MEASUREMENTS OF THE NEUTRON
SCATTERING LAW FOR LIGHT WATER
AT 95 °C**

ROBERT B. SMITH

AUGUST, 1967

**AEC RESEARCH &
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FOR LIGHT WATER AT 95 °C

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ABSTRACT

Measurements have been made of the double-differential scattering of slow neutrons from H₂O at 95 °C using the Hanford triple-axis spectrometer. Scattering cross-section values were obtained by normalization to the measured scattering from vanadium and were further reduced to the Egelstaff Scattering Law presentation. With initial energy values from 100 to 377 milli-eV(meV), results were obtained for neutron energy changes of 40 to 175 meV and scattering angles of 4 to 70 degrees.

During the course of these measurements several sources of systematic errors in triple-axis spectrometer measurements were investigated. These include the effect of spatial nonuniformities in the neutron beam intensity and in analyzer sensitivity as well as multiple diffraction in beryllium monochromator crystals [simulated (0003) reflections were noted]. The magnitudes of these effects and methods of minimizing their contributions are discussed. Also described is a copper monochromator crystal in which mosaic was increased by mechanical deformation.

INTRODUCTION

Slow neutron scattering studies of water are motivated by the need for knowledge in two areas. The slow-neutron differential scattering cross section is of direct interest in making accurate calculations of neutron thermalization processes in reactor design. The same scattering cross section also leads to understanding of the dynamics of the liquid and the motions of the principal scatterer, hydrogen. Dynamical models for water may be developed, tested against the measured scattering properties, and in turn used to extrapolate the cross-section values into regions beyond those of the measurements.

Several laboratories have reported relative and absolute differential scattering cross-section results for water.⁽¹⁻⁷⁾ Several types of time-of-flight and crystal spectrometers have been used, and results have been ob-

tained for both light and heavy water at different temperatures. From this laboratory* Kottwitz and Leonard⁽⁶⁾ reported results for light water at room temperature. These results were obtained using a triple-axis spectrometer. Agreement between workers has, in some cases, been relatively good within known uncertainties. However, a desired 10% accuracy in the value of the Scattering Law has been rarely, if ever, assured; and systematic disagreement and errors of much greater amount are often evident.

Representative of this situation is a recent article by McMurray, Russell, and Brugger.⁽⁷⁾ They report new measurements for water at room temperature and also compare their and previous results with a new extension of

* Previously Hanford Laboratories, operated by General Electric Company for the AEC.

a model for calculating the differential cross section.

The purpose of the present work was to extend, to water at 95 °C, the previous measurements made here at room temperature and, at the same time, explore possible sources of systematic errors.

The lack of agreement between various slow neutron scattering measurements is largely due to limitations of available neutron flux and counting rates. Available neutron flux has been relatively low in that it has been necessary to adopt lower resolution, thicker samples, and other poorer geometries than would be desirable. The testing and repetition of data and other tests for systematic errors is also inhibited.

During the early course of this work some inconsistencies were noted which led to identification of some possible sources of systematic errors when triple-axis spectrometers are used. One source of error was peculiar to the beryllium analyzer crystal being used. Multiple Bragg diffraction could cause spurious results. Another source of error is the lack of uniformity of neutron flux over the area of a sample or a monochromator crystal. This can cause errors in the measurement of scattered intensity. The nature of these possible systematic errors is discussed in two sections of the report.

After identification of these systematic errors, the apparatus and procedures were modified to eliminate or minimize them, and then further measurements were made. For this reason, results obtained under the dif-

ferent configurations are reported. Data for earlier measurements which are believed relatively free of any systematic effects are included with all the results obtained with the later configuration. Limitations on available time and counting rates have not permitted the results to be as complete as desired; but new results for water at 95 °C are presented and, hopefully, will be found useful.

THEORY

The quantity of central interest in this work is the differential neutron scattering cross section for water. It has become common, however, to reduce and normalize the cross section by various factors to a function $S(\alpha, \beta)$, the "Scattering Law", which depends only on the properties of the scattering material. Thus, it has been shown^(8,9) that the differential scattering cross section may be written as

$$\frac{d^2\sigma}{d\Omega dE} (E_1 \rightarrow E_2, \phi) = \frac{\sigma_b}{4\pi kT} \left(\frac{E_2}{E_1} \right)^{1/2} e^{-\beta/2} S(\alpha, \beta) \quad (1)$$

which may be considered a defining equation for $S(\alpha, \beta)$.

$\frac{d^2\sigma}{d\Omega dE}$ is the differential cross section, (b/sr-eV).

E_1 and E_2 are the initial and final neutron energies, (eV).

ϕ is the scattering angle.

k is the Boltzmann constant, (eV/°K).

T is the absolute temperature, (°K).

σ_b is the bound atom total scattering cross section, (b).

$$\alpha = \frac{m(E_1 + E_2 - 2[E_1 E_2]^{1/2} \cos \phi)}{MkT}$$

$$\beta = \frac{E_2 - E_1}{kT} \text{ (negative for energy loss by neutron).}$$

m and M are the masses of the neutron and hydrogen atom, respectively.

$S(\alpha, \beta)$ is a function of only two variables--the energy change and the square of the momentum change. The α and β are these quantities but are normalized to the mean thermal energy and momentum of the scatterer and are, therefore, dimensionless. The value of $S(\alpha, \beta)$ does not depend on the particular values of E_1 , E_2 , and ϕ , but depends only on the resultant values of α and β . This may serve as a powerful check on the consistency of experimental results. An experimental measurement may be made with several choices of E_1 , E_2 and ϕ which lead to the same α and β . The values of S should show agreement. Such consistency would not be made evident by comparison of cross-section results.

Equation 1 may be inverted to give S explicitly from the measured cross section.

$$S(\alpha, \beta) = \frac{4\pi kT}{\sigma_b} \left(\frac{E_1}{E_2} \right)^{1/2} e^{+\beta/2} \frac{d^2 \sigma}{d\Omega dE} \quad (2)$$

In principle, the scattering cross section is obtained from a measured intensity distribution in final energy and scattering angle. It is also necessary to provide a normalization to reduce a relative measurement to true cross section. If the resolution in solid angle and energy is known, and

the data is complete enough, then the measured intensity may be integrated over all angles and final energy, and the normalization chosen to give the proper total cross section. An alternative is to normalize the measured intensity against the intensity scattered by a material of known cross section. Vanadium may serve as such a material because it is almost a perfectly isotropic and elastic scatterer. It should be noted that this is not necessarily a direct substitution experiment because a different initial energy may be used in each measurement. However, under certain reasonable assumptions, it is possible to derive an expression for the cross section and for the Scattering Law in terms of the observed count rates from water and vanadium measurements.

The only assumptions necessary are the following:

- 1) The monitor chamber for the incident beam has $1/v$ sensitivity.
- 2) The water cross sections being measured are slowly varying functions within the instrumental energy and angular resolutions.
- 3) The vanadium scattering cross section is perfectly elastic, isotropic, and not dependent on energy.

Under these assumptions it is a straightforward matter to derive the following expressions for the cross-section and scattering law.

$$\frac{d^2 \sigma}{d\Omega dE} (E_1 \rightarrow E_2, \phi) = \frac{\sigma_V}{4\pi} \frac{n_V}{n_H} \cdot \left(\frac{E_2}{E_1} \right)^{1/2} \frac{C_H(E_1 \rightarrow E_2, \phi)}{A_V(E_2)} \quad (3)$$

$$S(\alpha, \beta) = kT \frac{\sigma_V}{\sigma_H} \frac{n_V}{n_H} e^{\beta/2} \frac{C_H(E_1 \rightarrow E_2, \phi)}{A_V(E_2)} \quad (4)$$

σ_V is the total vanadium scattering cross section, (b).

σ_H is the total bound scattering cross section for hydrogen, (b).

n_V and n_H are the corresponding area atomic density for vanadium and hydrogen samples, (atoms/cm²).

$C_H(E_1 \rightarrow E_2, \phi)$ is the observed scattered counts from water, (counts).

$A_V(E_2)$ is the area of what will be called the vanadium scattering peak. This is the peak observed in an energy analysis of the neutrons scattered at some fixed scattering angle by the vanadium sample. The area is a measure of the sensitivity of the analyzer. The monoenergetic beam is set to the energy value for which the sensitivity is desired. (A measurement more closely paralleling the situation during a water measurement would be to set the analyzer to the fixed final energy, scan the initial energy across this neighborhood, and integrate the resulting peak. This is very inconvenient with the spectrometer; and, under assumptions similar to 2 above, the measurements are equivalent.) A_V will be in units of counts-eV.

Both C_H and A_V are in terms of counts observed for the same preset number of beam monitor counts rather than for a fixed time interval. Because of the $1/v$ sensitivity of the monitor, this results in shifting the occurrence of the factor $(E_2/E_1)^{1/2}$ in Equations (3) and (4) from where it might be expected from Equations (1) and (2). Note that it is not necessary

to know the resolution in scattering angle or energy for the two spectrometers or even the detector sensitivity or crystal reflectivity.

Neutron scattering by the vanadium is, of course, not perfectly elastic. There can be considerable inelastic scattering by the phonons in the vanadium. For small scattering angles, the inelastic component will be quite small. At larger scattering angles, the inelastic scattering will increase as the purely elastic component is reduced by the Debye-Waller factor. However, the inelastic component will be nearly all due to one-phonon scattering; and the magnitude of the possible energy changes will be limited. In addition the total scattering, elastic plus inelastic, will still be closely isotropic. Thus, if the vanadium measurement can be made in such a way as to include both elastic and inelastic components, then Assumption 3 is reasonable.

A sample calculation was made of the scattering by the vanadium using the program LEAP.⁽⁹⁾ For an initial energy of 107 meV and a scattering angle of 15°, the inelastic component is only 3% of the total. Approximately 95% of this component is within 30 meV of the initial energy. At a 57° scattering angle, the inelastic component increases to about 30% of the total, which is within 2% of being equal to the total at 15°. Under these circumstances it is easy to make the vanadium peak measurement over a region wide enough to include all neutrons in the integration of the peak. With the resolution used in the experiments reported here, a large portion of the

inelastic component is included within the width of the vanadium peak; and the region of integration need not be very wide. One high resolution measurement, made with a scattering angle of 60° , verified the features indicated above for 57° .

Equations (3) and (4) are somewhat different than those used by Kottwitz (e.g. Ref. 6, Eq. 5). Previously, it was assumed that all resolution functions were Gaussian in shape. Instead, the above $A_V(E_2)$ appeared in terms of its peak height, peak width, and a numerical factor. The observed vanadium elastic peaks were assumed to be properly characterized by the measured peak height and width. A Gaussian assumption is not necessary. It is only necessary to accept Assumption 2 and that resolution peaks are, in that sense, narrow. The peaks are then sufficiently characterized by their zeroeth moment (area), first moment (location of its center), and perhaps the second moment (width squared). Equations (3) and (4) then follow easily from the fact that the area of the convolution of two resolution functions is just the product of the individual areas. (10,11)

APPARATUS AND PROCEDURES

The Hanford Triple-Axis spectrometer has been described briefly in reference 6. The monochromating spectrometer is heavily shielded, and the scattering angle and crystal orientation are set manually to provide the monoenergetic beam emergent from the shield. The monochromator crystal was, for all measurements, a beryllium crystal of exceptional uniformity. The (0002) and

(11 $\bar{2}$ 0) reflections were the only two used in this work. Though the beams obtained from the (0002) reflection are subject to contamination by half-order (0001) reflected neutrons (of energy $E_1/4$), intensity considerations favored their use. For example, when $E_1 = 0.377$ eV, even with gadolinium filters present to remove half-order neutrons (also attenuating the desired neutrons), the intensity available was still greater than that from the (11 $\bar{2}$ 0) planes. The (11 $\bar{2}$ 0) planes had lower reflectivity and also provided a higher resolution than necessary. For comparison purposes a few water scattering measurements were nevertheless made with these higher resolution conditions. Similarly, the monochromator was operated in a variety of configurations (choice of planes and angular resolution) for the purpose of consistency checks in the vanadium elastic peak measurements. Removable cadmium collimator strips could be easily changed in the exit collimator, making available overall resolutions, $\Delta\theta$ in Bragg angle, of 0.26, 0.43, and 0.53° . These are calculated values. (12)

The intensity of the monoenergetic beam was monitored by a boron-loaded pulse ionization chamber at the exit end of the collimator. The monitor chamber was very transparent to the neutron beam and was of low efficiency--about $6 \cdot 10^{-5}$ at 0.1 eV. The efficiency is quite uniform over the area of the beam and is assumed to vary accurately as $1/v$ in the energy region used.

The scattered neutrons from the sample at the second axis are selected in energy by the analyzing spectrometer. The collimator on the second arm serves

the dual purpose of defining the scattering angle and being the first collimator of the analyzing spectrometer. The resolution in scattering angle (as compounded from the angular widths of the first and second arm collimators) was between 0.7 and 1.8° . Water measurements commonly were characterized by the larger values, 1.3 to 1.8° . Poorer resolution in scattering angle could have been afforded for the sake of counting intensity; however, one of the limitations of triple-axis spectrometers is that because of common collimators, good resolution in energy is necessarily accompanied by good resolution in scattering angle.

The analyzing spectrometer was the subject of greatest change from the configuration previously used.⁽⁶⁾ Initially, it differed only in the use of a 1 in. diam, 8 atm, He^3 proportional counter for the third arm detector. The analyzing crystal was the same 1/4 in. thick beryllium slab parallel to the (0002) planes. These planes, in reflection geometry, were the only ones of sufficient reflectivity for making these measurements. Subsequent observations indicated that the use of these planes in this manner could result in the systematic errors in some of the data (This is discussed in the next two sections.) and motivated a change to a copper analyzer crystal. The copper (200) planes were used in transmission geometry. Partly for the same reasons, a change was made in the detector. An end window (2 in. diam), 1 atm BF_3 proportional counter was used in a larger and more effective cylindrical shield.

The analyzer collimators also had removable cadmium collimating strips which permitted a choice of energy reso-

lution. The available values of $\Delta\theta$ in Bragg angle were 0.48 , 0.77 and 1.05° . Typical values for the resultant energy resultant energy resolution are included in the tables of the results in Appendix B.

The energy scales and resolution of both spectrometers were taken to be as calculated from arm angle setting and collimator dimensions. The measurement of a vanadium peak serves as a check of each spectrometer against the other. Close agreement between the two was indicated by consistency and reproducibility in the vanadium data, the center of the peaks being always within 0.10° , and usually within 0.05° (arm angle), of the value expected. Arm zero was checked in each case by scanning the undiffracted beam. An upper limit to the uncertainty in initial and final energy might reasonably be one-tenth the resolution width in energy for each. This is consistent with the 0.10° value above. The width of the vanadium peak was usually somewhat greater than predicted from calculated resolution values.

Automatic equipment could program either the second arm (scattering angle) or the third arm and crystal (final energy) through equal increments. At each setting, the detector count was accumulated and recorded as well as a background count (analyzer crystal "off-Bragg"), and then the spectrometer advanced to the next step. Nearly all water scattering data were accumulated as distributions in scattering angle for fixed final energy. The measurement of the vanadium peak was in the form of energy distribution for one or more fixed scattering angles.

The water samples were confined in a heated holder assembly between two layers of stretched aluminum foil. Two foil windows were first secured to a main frame by clamping rings; the initial foil separations was about 1/2 in. Separate tensioning rings then forced the central portion of the windows toward each other, stretching the foils and reducing the space between the two windows to the desired water thickness. Pure aluminum, 0.006 in. thick, worked well at room temperature. Stretching of the foil resulted in work-hardening the aluminum to sufficient strength and tension to prevent sag and variations in water sample thickness. At 95 °C creep occurred which reduced the tension, and some variation in thickness was noted after several days at temperature. With subsequent use of 0.005 in., type 5052 aluminum alloy, no variations in thickness were observed during a month's time.

Some precautions were necessary to prevent gas evolution in the water at temperature. Aluminum in contact with the water was lightly anodized beforehand; and the water supply, triply distilled, was deaerated before use. With these precautions, no evidence could be found for possible bubble formation in the portion of the sample in the beam.

The sample region was 2 in. diam and nominally 0.033 in. thick. The geometry was such that scattering angles up to 70° could be used without the frame of the sample holder interfering with the scattered beam. Sample thickness was measured periodically with an electrical contacting micro-

meter with allowance made for window thickness. The uncertainty in sample thickness was 0.001 in. or less. For neutrons of the range of energies used, the measured transmission of the sample was close to 82% while the transmission of empty holder was 99% or greater. In all cases the sample was mounted normal to the monoenergetic beam.

The heavy aluminum frame of the holder contained electrical heaters, and a simple regulator-circuit held the temperature at 95 °C. The holder was moderately insulated, and additional 1/2 mil (0.001 in.) aluminum foil radiation and convection shields were placed over the windows to reduce the effect of changes in room conditions. Temperature was monitored by a thermometer and also by a thermocouple with chart recorder. Temperature variations with time and over the sample are estimated to have been a maximum of 1/2 °C. The same holder was used for an occasional room temperature run.

The vanadium sample was a very fine grained polycrystalline piece, 0.253 in. thick and 3 in. square. The measured density was equal to the accepted value. The measured neutron transmission was close to 72% for several energy values used. All vanadium measurements were made at room temperature; and, as for water, the sample was always normal to the monoenergetic beam.

MULTIPLE DIFFRACTION EFFECTS⁽¹³⁾

Multiple (or simultaneous) diffraction situations are coming to be recognized as a frequent possibility in all

work involving crystal diffraction of X-rays and neutrons. Ordinarily, a crystal is oriented for diffraction by a particular choice of planes and for a particular energy (or wave-length). Accidentally, but frequently, it happens that one or more other planes of the crystal are oriented to diffract, in some other direction, the same radiation intended for the primary beam. For a given set of crystal planes, oriented to some value of Bragg angle, the occurrence of multiple diffraction depends only on the remaining degree of rotational freedom of the crystal. The particular orientations for which multiple diffraction occurs are readily calculable;^(14,15) and, with the help of computers, it is possible to systematically catalog and diagram all occurrences of importance. It is, thus, possible to predict the locations, (crystal orientations) where the effect may be expected in the normal conduct of an experiment. Many workers have reported these "competition" dips in observations of a reactor spectrum with a single crystal spectrometer.⁽¹⁶⁾

Alternatively, it is possible for a double diffraction process to diffract into the primary beam a greater number of neutrons than normal. The energy may be of the same or different (a different "order") energy than intended for the primary beam. By this means, ordinarily "forbidden" reflections are possible, and a beam of desired energy may be contaminated with different order neutrons. Such is the case for beryllium (0002) planes and forbidden (0001), (0003), . . . reflections.

The magnitude of these effects depends on many factors--the number, type, and reflectivity of the planes involved; the relative intensities of different energy neutrons; resolution in both horizontal and vertical planes; etc. These effects were encountered in several aspects of the present work.

For the beryllium (0002) planes, calculations of multiple diffraction conditions were made; both for competition situations (where available intensity would be reduced) and for simulation of the (0001) planes (which would introduce half-order neutrons). These calculations were applicable to both the monochromating and analyzing spectrometers when using the beryllium (0002) planes. With these results in hand, choices were made of initial and final energy values to be used in the inelastic scattering measurements--avoiding, as much as possible, Bragg angles at which effects of greatest magnitude could be expected. This is the reason for the apparently odd values of energy which were used. Even so, at the higher initial energies, (because of reactor spectrum shape and degree of angular resolution) a significant amount of half-order neutrons could be present in the monoenergetic beam and needed to be removed by filters or corrected for.

A more serious, and slightly more involved, situation was found to sometimes exist when using beryllium (0002) planes for the analyzing spectrometer. It was found that simulated (0003) reflections ("three-halves order") could contribute, under some circumstances, a component of measured intensity in the inelastic scattering measurements. This component

could be mistakenly accepted as inelastically scattered neutrons which it was desired to count. In fact, it was due to elastically scattered neutrons. This may be understood as follows.

The spectrum of neutrons scattered by water includes a relatively large component at energies very close to the initial incident energy. This is the so-called quasi-elastic peak, and is most intense at small scattering angles. The neutrons of interest in the present work are those which have suffered a greater energy loss (30 to 200 meV). They are much fewer in number and must be measured in the presence of the quasi-elastic neutrons. If at some scattering angle, the analyzer energy (with a beryllium (0002) set of planes being used) is set equal to the initial energy value, E_1 , then the large quasi-elastic component will be detected. When the analyzer is set to lower energies, the inelastically down-scattered neutrons of interest will be counted. At an even lower energy setting of $E_1/4$, the elastic peak will again be observed--now however, as the result of a second order diffraction by the beryllium (0002) planes (i.e., diffraction by the (0004) planes).

The (0003) reflections are ordinarily forbidden. However, at some analyzer energy setting E_2 , if multiple diffraction conditions should exist which permit simulation of the (0003) reflection, then the analyzer will also accept (if only inefficiently) neutrons of energy $(9/4)E_2$. This value may turn out to be equal to the initial energy and the quasi-elastic peak would again be weakly observed.

Thus, if the analyzer is set to a final energy of $E_2 = (4/9)E_1$ and if multiple diffraction conditions also exist, then elastically scattered neutrons may be detected along with the desired inelastic component. This situation was found to exist in some inelastic scattering measurements as a result of the particular choice of E_1 and E_2 . For a fixed, small scattering angle, an energy analysis of the scattered neutrons would result in a small peak in the region of interest. This peak could be mistakenly interpreted as a result of scattering by a discrete level in water. However, the energy at which the peak occurred bore a fixed fractional $(4/9)$ relationship to different values of initial energy rather than a fixed energy-loss relationship. This peak could be at times comparable in intensity to the inelastic component of interest. Examples of the observed peaks are shown in Figure 1 for two values of the initial energy. Identification of the effect was further affirmed by filter measurements and direct comparison against calculations predicting the conditions for which multiple diffraction occurred.

The problem was subsequently avoided (in experiments using Be (0002) planes for the analyzer) by choice of values of E_1 and E_2 . No attempt was made to predict the non-occurrence of the multiple-diffraction conditions. Data for the questionable ratio of $E_2/E_1 = 4/9$ was not used. This problem further motivated the abandonment of beryllium as an analyzing crystal. For the monochromator crystal the situation is different and this $3/2$ -order reflection is not believed to present a problem.

FIGURE 1. Energy Analysis of Neutrons Scattered by Water at 95 °C [Region covered is between quasi-elastic peaks seen in first and second order by analyzer. (These would occur at points marked by arrows.) Curves are shown for two different values of initial energy. The peaks are due to quasi-elastic neutrons detected as a result of simulated (0003) reflection in the beryllium analyzer crystal while the "background" for the peaks is the true inelastic (down-scattered) component of interest. The scattering angle was 12 degrees.]

GEOMETRICAL EFFECTS

Many measurements were made during the course of the work to check on the accuracy and reproducibility of the results. The scattering law for some values of α and β was obtained under different conditions of initial and final neutron energy as well as under different resolutions for the same energy values. These measurements showed inconsistencies in the magnitude of $S(\alpha, \beta)$ to often be 10 to 15% and occasionally 20 to 25%. An even simpler measurement, the energy analysis of the vanadium elastic peak, sometimes resulted in 15 to 20% differences in the area of the peak. These differences occurred (for a fixed analyzer configuration and same nominal neutron energy) between determinations made with different types of monoenergetic beams, i.e., beams obtained from different monochromator crystal planes and with different degrees of collimation. Such a situation obviously calls into question the assumptions which form the basis of the measurement and the extent to which they are met.

The principal explanation for these discrepancies was found to lie in the geometry of the incident beam, its intensity distribution over the sample, and similarly, the sensitivity of the analyzer over the sample area. It was possible to minimize much of the discrepancy originally observed, but it is apparent that there are inherent limitations to the accuracy of intensity measurements made with crystal spectrometers. Some of the limitation is due to the use of multiple-slit Soller collimators.

The analysis of crystal spectrometer resolution, intensity, and sensitivity, etc., is commonly limited to consideration of angular variables describing the acceptance of collimators, mosaic spread of the crystals, angular divergence of beams, etc.⁽¹²⁾ Factors which are not usually included are vertical divergence, distances between collimators and crystals or samples, and, in particular, the possible variation in crystal reflectivity and beam intensity over the cross section of collimators. Such a treatment is adequate for most situations--for crystal structure studies with relatively simple spectrometer geometry, or for other diffraction studies where only peak occurrence and locations, not relative intensities, are important. However, when (for the sake of intensity) it is necessary to use large collimators and beam areas, it is also necessary to use large monochromator crystals and multiple slits in the collimator. In this case it is important to consider questions of beam uniformity; because, in addition to the assumptions mentioned earlier, it is ordinarily assumed also that the analyzer sensitivity is the same for all scattered neutrons regardless of their point of origin in the sample. This assumption may be difficult to realize in practice.

Measurements were made of the incident beam distribution at the sample by using a narrow vertical slit and counter. In addition the analyzer sensitivity was measure by moving a narrow plastic rod scatterer across the field of view of the second collimator. Some of the features to be expected were observed.

The cadmium collimator strips caused dips in the intensity of the monoenergetic beam at the sample, and there were similar depressions of the analyzer sensitivity. These depressions are, of course, different for each choice of collimator resolution. The sample was sufficiently far from the ends of the collimators that the dips were not severe--up to 20% of peak intensity. Furthermore, the incident beam width at the sample could at times be narrower than the collimator width, and more often the intensity distribution could be asymmetrical. The latter case occurs when the monochromator crystal is used in reflection or when its angle is not carefully set. An added complication is that maximum intensity and beam symmetry do not necessarily occur at the same crystal angle setting.

The width of the region of uniform analyzer sensitivity was found to be much narrower than expected. This was attributed mostly to the use of a 1 in. diam detector in the third arm. Narrowness is also to be expected when a thin slab monochromator crystal is used in reflection geometry. Oriented for reflection, a thin crystal presents only a narrow profile to the second collimator and sample and does not intercept all scattered neutrons accepted by the collimator. The analyzer sensitivity could also be quite asymmetrical.

These nonuniformities clearly can result in systematic differences in measured intensity. For the same total incident beam intensity, a higher counting rate will occur if, in the sample, points of highest incident intensity are viewed by points of greatest analyzer sensitivity; the converse is also true.

In principle the measured intensity may be expressed as the integration, over the sample region, of the product of beam intensity and analyzer sensitivity. There is, however, a changing relationship between the two factors at different scattering angles; and the overall sensitivity can become dependent on scattering angle in addition to its dependence upon incident beam shape. In practice, a substitutional experiment of comparing the scattered intensities from a water sample and a vanadium sample may largely circumvent the problem. To do so completely, however, would require equal sample thickness, measurement of the vanadium scattering peak at several scattering angles, and small differences in the incident beam distribution for the two measurements. Since the very use of Soller collimators results in nonuniformities of intensity and analyzer sensitivity, the accuracy of any intensity measurements may be limited in situations where such collimators are necessary.

Several precautions are possible for minimizing the effects discussed. The monoenergetic beam should be explored and gross asymmetry avoided. It is usually easy to assure equal division of the intensity about the center-line of the sample even though the intensity may otherwise be skewed and a slight loss of total intensity may occur. Any beam monitor should have uniform sensitivity over the area of the beam. The sample should be carefully centered. The analyzer spectrometer should have collimators wide enough to view the whole illuminated portion of the sample at any scattering angle, and the analyzer crystal and detector should be wide enough to provide

equal sensitivity for all of the multiple channels of the collimators. For small Bragg angles, analyzer monochromators should, thus, be limited to a transmission (preferably symmetrical) geometry.

When precautions of this type were taken the inconsistencies first described were reduced to about the 10 to 15% level, but it was not possible to do much better while using the Be(0002) planes in reflection. The use of other planes of the beryllium crystal was explored; but, because of low reflectivity and relative thinness of the crystal, sufficient sensitivity could not be obtained.

When the copper monochromator crystal was used, in transmission, the width of the analyzer sensitive region was increased but not to the full width of the analyzer collimators. With the copper crystal as analyzer, the vanadium scattering peak was measured with a wide variety of incident beam conditions. The areas of the vanadium peaks so obtained agreed within 5%. A similar agreement occurred for two water scattering experiments made with different resolution conditions for both the incident beam and the analyzer (but same values of initial and final energy). It is unlikely that any greater accuracy could be realized for such measurements with crystal spectrometers. The effect of intensity dips due to the collimator strips could, alone, result in errors of 5%.

TREATMENT OF THE DATA

As mentioned earlier, all data were accumulated for a standard number of preset monitor counts or normalized to

this number. Background counts were taken at each point. The data for each measurement run was processed by computer for background subtraction, normalization, calculation of the statistical error, and calculation of energy scales and α and β values from the spectrometer angles.

Some sample-out background runs were made to correct for scattering by air and sample holder. However, with the thin aluminum windows used, this correction was not significant except at the smallest scattering angles, where neutrons from the unscattered beam were starting to be counted directly. Such data points were often rejected and an air scattering correction was not in general applied. As discussed by Kottwitz,⁽⁶⁾ there is no need to make explicit corrections for resolution effects--only a need to avoid values of the final energy where any part of the tail of the quasi-elastic peak may be counted. Cadmium and gadolinium filter measurements were made to determine the fractional and higher order content of the monoenergetic beam, and corrections were made in every case where significant. This was viewed as a correction to the monitor count rate. The correction for higher orders was, at most, 5%, and for higher order neutrons was 30%. For most experiments it was less than 5%.

Two types of results are reported: some obtained with the beryllium analyzer crystal and some with the copper crystal. The vanadium peak data for the former was obtained in terms of the peak height and the full width; and the normalization and data reduction were performed as described previously. With the copper analyzer crystal, the

vanadium peaks were measured over a wide enough range that the area could be obtained directly by summation. The summation was made on the angle scale (of the analyzer arm) and converted to energy scale. For several of these peaks, the first moment was calculated and found to agree with the center of the peak defined by the midpoint of the full width. It should be noted that the integrated areas were from 0 to 5% greater than would have been obtained from the peak height and width values. No attempt was made to make a correction of this nature to the peaks obtained using the beryllium crystal. In all cases the vanadium peak data were corrected for multiple scattering and absorption effects as described for the previous work.

In the present measurements there was evidence of need to use a normalization which was a function of scattering angle. Previously a constant normalization was found to be reasonable after application of multiple scattering corrections. The reason for the difference is not known; but the present results showed a sensitivity drop-off of, at most, 15% at the largest scattering angles. This occurred for both the beryllium and copper analyzer crystals. For the former an angular distribution of the peak value was combined with the width values to give the sensitivity as a function of scattering angle. For the copper crystal, the full peak and area was obtained at two scattering angles, 10° and 60°, say, and a linear variation between them was assumed.

Using the vanadium results described above, the water scattering results were reduced to cross section values and

the scattering law using Equations 3 and 4. For the other parameters in the equation, the following values were used: $\sigma_H = 81.6$ b; $\sigma_V = 5.05$ b; and $kT = 31.72$ meV.

No corrections were made to the water scattered intensity for multiple scattering in the water sample.

RESULTS AND DISCUSSION

The results of the measurements are presented in Figures 2 to 5 as graphs of S versus α for several values of β . (The same data is listed in tabular form in Appendix B.) These results include all measurements made with the copper analyzer crystal. The latter are believed to be largely free of systematic errors of the type discussed. Unless otherwise indicated the graphs are for copper analyzer results. Also shown are some representative results obtained with the beryllium analyzer crystal. These are for energy values and resolution which should rule out any possibility of 3/2-order effects. However, they may suffer from systematic errors of unknown amount arising from geometrical effects. The data shown are those which indicated least disagreement with each other and with the copper analyzer data. (Other measurements with the beryllium analyzer were made and results exist for energy changes up to 250 meV. However, these show serious lack of internal agreement and large unknown amounts of systematic errors and are not reported.)

The error bars shown represent only the statistical uncertainty in the count for the water measurements. Statistical errors are not necessarily shown for all data points. Sometimes these

FIGURE 2. $S(\alpha, \beta)$ Versus α for $\beta = -3.15$ for Room Temperature Water (6)
 [The line drawn is identical to that shown by Kottwitz and Leonard. (6)]

FIGURE 3. $S(\alpha, \beta)$ Versus α for Several Values of β . Water at 95 °C

FIGURE 4. $S(\alpha, \beta)$ Versus α for Two Values of β . Water at 95 °C
(The line shown in the lower two graphs for $\beta = -3.15$ is identical to ease comparison of the two sets of data.)

FIGURE 5. $S(\alpha, \beta)$ Versus α for Several Values of β . Water at 95 °C

are omitted for clarity or the errors are covered by the size of the symbol. Other errors are perhaps best judged by the degree of agreement between the different measurements. The lines drawn on each figure are only visual estimates and heavily favor the results for the copper analyzer.

Figure 2 shows the results for two measurements made on room temperature water ($\sim 23^\circ\text{C}$) for a single value of β . They are compared to the result obtained by Kottwitz and Leonard for the identical value of β . This comparison serves as a measure of the consistency and reliability of both measurements. Agreement is seen to be quite good (about 10%) both between the two new measurements and with the previous result.

Figures 3 to 5 show the remaining results for the various values of β . The many measurements for $\beta = -3.15$ are some of those made in the course of consistency checks. To avoid crowding, the results for the two analyzer crystals are shown separately but the line drawn in the lower two graphs is identical and may be used for comparisons. Clearly, the consistency obtained is not always as good as that in Figure 2.

It should be noted that, although it is reasonable to assume the cross section varies slowly with energy and scattering angle, the same cannot be said for $S(\alpha, \beta)$. Because of the exponential factor, $e^{\beta/2}$, the value of $S(\alpha, \beta)$ is very sensitive to changes and uncertainties in the energy change, β , especially at large values. Thus, on a fractional basis, variations $\delta\beta$, through the exponential factor alone, give rise to variations δS of

$$\frac{\delta S}{S} = \beta^2/2 \left(\frac{\delta\beta}{\beta} \right).$$

For this reason the statistical errors, uncertainties in normalization, and other errors evidenced by comparison of different measurements may not represent the largest uncertainty in the values stated for $S(\alpha, \beta)$. For example, a value of $\beta = 5$, known with an accuracy of 5%, results in an uncertainty of 60% in the value of $S(\alpha, \beta)$. This is the approximate situation for one of the measurements reported here. This sensitivity to β is of consequence when comparisons are made between theoretical models and experimental results. Measured intensity is not very sensitive to errors in energy, and the exponential factor does not influence comparisons between different experimental results--even those obtained with different apparatus. For this reason it might be suggested that for many purposes experimental results could be presented, compared, and discussed as a function of the form $\left[e^{-\beta/2} \cdot S(\alpha, \beta) \right]$, i.e., the scattering law with the exponential factor removed. This form retains many of the advantages of $S(\alpha, \beta)$, being still a function of the two dimensionless variables α , β , and no other external variables. The uncertainty in the value of β could be stated separately, and its effect would remain distinct from other sources of error until a conversion to S is made for comparison with theoretical results.

ACKNOWLEDGMENTS

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work for guidance and for long discussions on many aspects of inelastic scattering and diffraction effects. He also wrote the program and performed calculations of the multiple diffraction effects in beryllium. This same program was modified to perform the same calculations for the cubic copper crystals. Thanks are also due to him and B. R. Leonard for occasional help in running the spectrometer.

The author is also grateful to B. R. Leonard for unfailing support and continued encouragement throughout the work.

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APPENDIX A
COPPER MONOCHROMATOR

The purpose of this appendix is to briefly describe a copper monochromator crystal in which the reflectivity was increased significantly by deliberately induced mosaic. The choice of copper was governed largely by its availability and its reasonable monochromator properties. It has an adequate coherent scattering cross section and is readily available in large ingots of high perfection. Its disadvantages are that it possesses significant incoherent and absorption cross sections. Experience has also indicated that severe secondary extinction was present in copper crystals. This was expected from calculations and was dramatically apparent in comparing measured reflectivities of crystals in transmission and reflection geometry. The need of the present work was for a crystal able to fill most of the field of view of the analyzer collimators, 1.5 x 2.5 in., and to do so at small Bragg angles. This dictated a transmission geometry. In view of the secondary extinction, it appeared that efforts to improve reflectivity by increasing the mosaic would be justified.

Many suggestions have repeatedly been made for increasing single crystal mosaic spread, and most laboratories have probably had a try at several methods. The most direct method is probably the use of many pieces of crystal, each sufficiently thin that secondary extinction is small. These pieces must then be mounted together slightly misoriented with respect to each other--but not so greatly misoriented that each piece does not contribute to the diffracted beam. There are obvious me-

chanical difficulties in such a scheme. Other methods such as mechanical deformation have not been very successful in assuring uniformity in the induced mosaic. Barrett, Mueller, and Heaton⁽¹⁾ have reported increases by a factor of 20 to 40 in the intensity available from germanium monochromators. The most effective method appeared to be simple compression (at 650 °C) to a reduction of up to 2.5%. That the increase was so great is perhaps a tribute to the original perfection of the germanium and the severity of the secondary extinction. It was not expected that such an increase would be available from copper. The above work suggested the methods which were used here.

The monochromator was assembled from five thin pieces of copper crystal. The pieces were 2.5 in. in diameter and cut parallel to one set of (200) planes. Due to difficulties in cutting, pieces were not all of the same thickness but ranged from 0.063 to 0.088 in. In the symmetrical transmission position, either of the remaining (020) or (002) set of planes were used; and all subsequent observations refer to reflections from these planes. The five pieces were mounted together in an assembly and were adjustable in orientation relative to each other. One central copper piece was mounted in a main supporting frame of the assembly and two additional pieces were mounted in thin brass frames closely on each side of the central one. These four outer pieces had screw adjustments which permitted them to be made parallel (or misaligned slightly) with respect to

the central piece. Figure A-1 shows two views of the completed assembly. All adjustments were independent, and it was possible to have the (020) reflecting planes of all pieces very nearly parallel while the (002) planes were misaligned, or conversely. [This permitted the novelty of having a monochromator crystal with two different "mosaic" spreads available. The crystal could be easily mounted with either the (020) or (002) set of planes in the vertical reflecting position.] It is not known how effective this technique, alone, is in reducing secondary extinction because concurrently a method was evolved for introducing further mosaic in each piece by bending.

True integrated reflectivity measurements were made of the individual pieces and of the final assembly at different stages of the work to judge the improvement achieved. The crystals were "rocked" through their Bragg reflection in a monoenergetic beam and with the detector collimator wide open to accept all diffracted neutrons. The area of such a Bragg peak, when normalized to the open beam counting rate, gives the integrated reflectivity, R^0 , in radians.⁽²⁾

With the two crystals (monochromator and analyzer) in a parallel configuration, the resulting peak width is a minimum and, in principle, is dependent only on the mosaic spread of the two crystals. The mosaic changes of the copper pieces could be judged from any increase in the width over the minimum width, 0.18° , seen with the most perfect copper piece. A neutron imaging phosphor together with a Polaroid film holder could be used to photograph the

twice diffracted beam. With the resulting photographs, taken at different points on the crystal rocking curve, it was possible to identify the portions of the crystal contributing to the diffracted beam and, consequently, judge slight misorientations within the crystal and the homogeneity of induced damage. This method is essentially that reported by Bearden and Henins.⁽³⁾

The thin copper pieces were all cut from the same 2.5 in. diam ingot (originally about 3 in. long) by spark erosion machining. Contrary to reputation, this method does not necessarily yield strain free specimens and the first attempts resulted in thin (0.070 in.) pieces with a large curvature toward the freshly cut surface. This was attributed to the highly stressed (though very thin) sputtered and fused surface layer and the very low plastic yield strength of single crystal copper. Undeformed pieces were obtained by attaching a thick backing plate to support each piece during the cutting operation. Each piece remained attached to this support while the sputtered surface was removed by etching. Sometimes the etch was preceded by wet grinding to level the surface. The sputtered surface remaining on the ingot was similarly treated before cutting the next piece.

The integrated reflectivity of the individual pieces, as cut, varied somewhat and was probably due to different amounts of imperfections resulting from the cutting operation. For two pieces, the reflectivity of one set of planes, the (020), say, was 30% higher than that of its other set (002). This difference could be correlated with the direction

FIGURE A-1. Two Views of the Copper Analyzer Crystal Assembly [The circular area is one of five 2.5 in. diam single crystal pieces. The other pieces are mounted in frames whose edges are just visible through openings in the edge of the assembly. The small round head screws adjust the orientation of the individual frames. (The large set screws are part of the large goniometer in which the assembly is mounted, and in the one view the exit end of the second arm collimator appears.)]

of cutting and the expected nature of the residual damage. These differences in reflectivity were not accompanied by a significant increase in width of the rocking curve.

Damage was induced in the thin copper pieces by bending them to a cylindrical surface of 12 in. radius. This results in approximately 1% strain at the surface. The pieces were rolled to the 12 in. radius and then unrolled to flatten them. The damage was thus induced by a straight bend line progressing across the disk of the crystal from one side to the other, leaving, it was hoped, a relatively homogeneous distribution of the deformation.

Interestingly, copper is a popular sample material in the study of single crystal deformation; and more is known about it, perhaps, than any other material.^(4,5) All initial deformation of the single crystal occurs by glide along simple crystallographic planes and directions in the planes. Combinations of planes and directions are termed slip systems. For copper the planes are of the form (111) and the direction [110], resulting in 24 possible slip systems. Intuitively, it was believed that bending in several directions in such a way as to activate all possible slip systems would result in the best homogeneity of the residual damage. From the view point of the direction of the surface strain, this is best achieved by rolling along the [011] and [0 $\bar{1}1$] directions in the plane of the slab. By rolling and unrolling along these two directions, and doing so also with opposite curvature, all slip systems are active several times.

After this much treatment, the copper was sufficiently work-hardened that some curvature remained after the last unrolling action. This curvature was nearly all removed by pressing briefly at 500 °C between flat steel plates. For use as a reference surface it was of course necessary that at least one side of each piece must have been flat and plane before any deformation existed. Not all pieces used in the final monochromator were given the identical treatment--some were bent more times than others and some along other directions. A few measurements indicate that most of the increase in reflectivity of an individual piece had occurred after the first two bends. Any further bending did increase the mosaic and broaden the rocking curve.

In every case the reflectivity of the single pieces increased by about a factor of two. The thicker pieces increased somewhat more. Those which increased somewhat less showed some signs of damage before the deliberate bending. The reflectivity of all pieces, after damage, was closely proportional to the thickness, indicating little secondary extinction. Conversely, the factor of two increase in reflectivity is a measure of the amount of extinction possible in pieces of copper as thin as 0.050 in.

The width of the rocking curves increased to values between 0.25 and 0.35°. If rms compounding of mosaic and resolution widths is assumed, this means that the mosaic of the damaged pieces was increased by 0.17 to 0.30°. Photographs of the beam from the copper crystals showed that the damage was

reasonably uniform over the crystal on a scale comparable to the thickness. The brightness of the diffracted beam had nonuniformities of millimeter dimensions. Some pieces, those of widest rocking curve, showed evidence of residual curvature not fully removed and some larger misoriented regions. These contribute to the rocking curve width but not necessarily to local reduction of secondary extinction. Even so, a mosaic value determined as above would probably serve well in resolution calculations.

The separately damaged pieces were mounted together in the composite 5-piece assembly and studied in several settings. The total thickness of all five pieces was 0.36 in. The orientation of the separate pieces could be easily set to yield a rocking curve with five separated peaks, or with a little care it was possible to orient the pieces to yield a single-peak rocking curve as narrow as 0.34° (mosaic 0.29°). In this latter case some secondary extinction was present, and the reflectivity was reduced by one-third from that existing for separated peaks. Because of attenuation by the total copper present, the reflectivity of the composite was less than the sum of the individual reflectivities. At best, for the displaced orientations, the total reflectivity was only three times that of the average piece. Very probably, for the superimposed peak, the

point of diminishing returns in thickness has been reached and equal reflectivity could be obtained with three or four pieces--unless greater mosaic were induced in each piece.

The composite crystal was used for water scattering experiments only as adjusted for minimum rocking curve width. Because of questionable reproducibility, changing the mosaic for different collimator widths was not considered. As used, the composite is estimated to have three times the reflectivity otherwise possible with copper crystals in transmission.

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APPENDIX B

TABULATION OF RESULTS

In this appendix the results are presented in tabular form more convenient for any future use. Each table is distinguished primarily by the value of β , and for some values of β there are separate tables for different degrees of resolution and for the two analyzer crystals used. For each case the initial and final energy values are given, their calculated resolution, and the crystal planes used. The tabulated quantities are the scattering angle (in degrees), the value of α (dimensionless), the value of S (also dimensionless), and the differential cross section (in barn/hydrogen atom-steradian-eV). The indicated errors

are only those due to counting statistics.

Each table represents the results of one to three measurement runs made under identical conditions and the data processed together. (The case numbers shown served only for identification during data processing.)

Below is a summary list of the measurements reported. Note that the first two tables are for the two room temperature measurements. All the rest are for the 95 °C water sample. The reference to resolution is only general and does not refer to any standardized configuration. It may refer to a relatively high resolution in the analyzer only.

SUMMARY LIST OF TABLES

Table No.	E_1 , meV	E_2 , meV	β	Analyzer	Resolution	Remarks
1	214	114	-3.93	Cu	High	~23 °C, E_1 from Be(11 $\bar{2}$ 0)
2	214	114	-3.93	Cu	Low	~23 °C
3	99.5	62	-1.18	Be	Low	95 °C
4	99.5	62	-1.18	Cu	Low	95 °C
5	124	62	-1.97	Cu	Low	95 °C
6	137	62	-2.36	Cu	Low	95 °C
7	149	62	-2.76	Cu	Low	95 °C
8	162	62	-3.15	Cu	Low	95 °C
9	214	114	-3.15	Cu	High	95 °C
10	214	114	-3.15	Cu	Low	95 °C
11	214	114	-3.15	Be	Low	95 °C
12	214	114	-3.15	Be	High	95 °C, E_1 from Be(11 $\bar{2}$ 0)
13	162	62	-3.15	Be	Low	95 °C
14	239	114	-3.94	Be	High	95 °C
15	239	114	-3.94	Cu	Low	95 °C
16	264	114	-4.73	Cu	Low	95 °C
17	289	114	-5.52	Cu	Low	95 °C
18	377	202	-5.52	Cu	Low	95 °C
19	377	202	-5.52	Cu	High	95 °C, E_1 from Be(11 $\bar{2}$ 0)

TABLE 1.

<u>Room Temp., H₂O</u>		<u>$\beta = -3.93$</u>	<u>Case 531</u>
$E_1 = 214 \text{ meV}$		$E_2 = 114 \text{ meV}$	$\Delta E = 100 \text{ meV}$
$\delta E_1 = 14$		$\delta E_2 = 8$	
$\text{Be}(11\bar{2}0)$		$\text{Cu}(002)$	
<u>ϕ</u>	<u>∞</u>	<u>$S \cdot 10^3$</u>	<u>$d^2\sigma/d\Omega dE$</u>
4	0.638	4.6 ± 1.5	6.2 ± 2.0
8	0.727	5.6 ± 1.0	7.5 ± 1.3
12	0.875	6.5 ± 1.0	8.7 ± 1.3
16	1.08	5.6 ± 0.9	7.4 ± 1.2
20	1.34	7.2 ± 0.9	9.6 ± 1.2
24	1.66	7.9 ± 1.0	10.5 ± 1.4
28	2.03	9.1 ± 1.0	12.1 ± 1.3
32	2.46	9.7 ± 1.0	12.9 ± 1.3
36	2.93	10.1 ± 1.0	14.6 ± 1.4
40	3.46	9.9 ± 1.0	13.2 ± 1.4
44	4.03	11.9 ± 1.1	15.9 ± 1.4
48	4.64	11.5 ± 1.0	15.4 ± 1.4
52	5.29	11.3 ± 1.1	15.0 ± 1.5
56	5.98	11.1 ± 1.1	14.8 ± 1.4
60	6.70	11.0 ± 1.1	14.7 ± 1.5
64	7.45	11.9 ± 1.1	15.9 ± 1.5
68	8.22	10.2 ± 1.0	13.6 ± 1.5

TABLE 2.

<u>Room Temp., H₂O</u>		<u>$\beta = -3.93$</u>	<u>Case 530</u>
$E_1 = 214 \text{ meV}$		$E_2 = 114 \text{ meV}$	$\Delta E = 0.110 \text{ meV}$
$\delta E_1 = 23$		$\delta E_2 = 17$	
Be(0002)		Cu(002)	
<u>ϕ</u>	<u>α</u>	<u>$S \cdot 10^3$</u>	<u>$d^2\sigma/d\Omega dE$</u>
8	0.727	4.1 ± 0.3	5.5 ± 0.3
12	0.875	5.0 ± 0.3	6.7 ± 0.3
16	1.08	5.9 ± 0.3	7.9 ± 0.3
20	1.34	6.6 ± 0.3	8.8 ± 0.4
24	1.66	7.3 ± 0.3	9.8 ± 0.4
28	2.03	8.1 ± 0.3	10.8 ± 0.4
32	2.46	10.1 ± 0.3	13.4 ± 0.4
36	2.93	10.7 ± 0.3	14.2 ± 0.4
40	3.46	11.8 ± 0.3	15.6 ± 0.4
44	4.03	11.7 ± 0.4	15.6 ± 0.6
48	4.64	12.7 ± 0.4	16.7 ± 0.6
52	5.29	12.5 ± 0.3	16.5 ± 0.4
56	5.98	12.4 ± 0.3	16.4 ± 0.4
60	6.70	12.2 ± 0.3	16.2 ± 0.4
64	7.45	11.1 ± 0.3	14.8 ± 0.4
68	8.22	10.9 ± 0.4	14.6 ± 0.5

TABLE 3.

<div> <div>H₂O 95 °C</div> <div>E₁ = 99.5 meV</div> <div>δE₁ = 7.3</div> <div>Be(0002)</div> </div>		<div> <div>β = -1.18</div> <div>E₂ = 62 meV</div> <div>δE₂ = 6.7</div> <div>Be(0002)</div> </div>		<div>Average</div> <div>Case</div> <div>354-366</div> <div>ΔE = 37.5 meV</div>
ϕ	∞	$S \cdot 10^2$	$d^2\sigma/d\Omega dE$	
3	0.146	3.10 ± 0.17	9.04 ± 0.5	
4	0.151	3.41 ± 0.15	9.94 ± 0.4	
8	0.188	3.82 ± 0.11	11.11 ± 0.3	
12	0.248	4.48 ± 0.12	13.1 ± 0.3	
16	0.331	4.65 ± 0.12	13.6 ± 0.3	
20	0.44	5.10 ± 0.12	14.9 ± 0.3	
24	0.57	5.81 ± 0.12	16.9 ± 0.3	
28	0.72	6.21 ± 0.13	18.1 ± 0.4	
32	0.89	6.93 ± 0.12	20.2 ± 0.3	
36	1.09	7.48 ± 0.12	21.8 ± 0.3	
40	1.30	8.07 ± 0.13	23.5 ± 0.3	
44	1.53	8.20 ± 0.13	23.9 ± 0.3	
48	1.78	8.74 ± 0.13	25.5 ± 0.3	
52	2.05	8.65 ± 0.13	25.2 ± 0.3	
56	2.32	8.75 ± 0.13	25.5 ± 0.3	
60	2.62	8.55 ± 0.14	24.9 ± 0.4	

TABLE 4.

<div> <div><u>H₂O 95 °C</u></div> <div><u>β = -1.18</u></div> <div><u>Case 557</u></div> </div>			
<div> <div>E₁ = 99.5 meV</div> <div>E₂ = 62 meV</div> <div>ΔE = 37.5 meV</div> </div>			
<div> <div>δE₁ = 3.8</div> <div>δE₂ = 6.8</div> </div>			
<div> <div>Be(0002)</div> <div>Cu(002)</div> </div>			
<u>φ</u>	<u>∞</u>	<u>S · 10²</u>	<u>d²σ/dΩdE</u>
4	0.15	4.9 ± 0.6	14.3 ± 1.7
6	0.17	4.2 ± 0.5	12.3 ± 1.4
10	0.21	5.1 ± 0.5	14.9 ± 1.5
20	0.44	5.5 ± 0.6	16.2 ± 1.6
30	0.80	7.3 ± 0.7	21.1 ± 2.1
40	1.30	10.1 ± 0.7	29.6 ± 2.1
50	1.91	9.5 ± 0.7	27.9 ± 2.0
60	2.62	10.9 ± 0.7	31.8 ± 2.2
70	3.40	9.8 ± 0.7	28.6 ± 2.1

TABLE 5.

<u>H₂O 95 °C</u>		<u>$\beta = -1.97$</u>	<u>Case 548</u>
$E_1 = 124 \text{ meV}$		$E_2 = 62 \text{ meV}$	$\Delta E = 62.5 \text{ meV}$
$\delta E_1 = 5.4$		$\delta E_2 = 6.8$	
<u>Be(0002)</u>		<u>Cu(002)</u>	
<u>ϕ</u>	<u>α</u>	<u>$S \cdot 10^2$</u>	<u>$d^2\sigma/d\Omega dE$</u>
4	0.35	3.1 ± 0.4	11.9 ± 1.5
6	0.37	2.9 ± 0.3	11.2 ± 1.3
10	0.42	2.9 ± 0.3	11.1 ± 1.3
20	0.67	4.0 ± 0.4	15.7 ± 1.4
30	1.08	5.3 ± 0.4	20.4 ± 1.6
40	1.64	5.8 ± 0.4	22.3 ± 1.7
50	2.32	6.2 ± 0.5	23.9 ± 1.8
60	3.11	5.8 ± 0.5	22.4 ± 1.8
70	3.99	4.9 ± 0.5	19.0 ± 1.7

TABLE 6.

<div> <div><u>H₂O 95 °C</u></div> <div><u>E₁ = 137 meV</u></div> <div><u>δE₁ = 6.3</u></div> <div><u>Be(0002)</u></div> </div> <div> <div><u>β = -2.36</u></div> <div><u>E₂ = 62 meV</u></div> <div><u>δE₂ = 6.8</u></div> <div><u>Cu(002)</u></div> </div> <div> <div><u>Case 550</u></div> <div><u>ΔE = 75 meV</u></div> </div>			
<u>φ</u>	<u>α</u>	<u>S · 10²</u>	<u>d²σ/dΩdE</u>
4	0.477	2.8 ± 0.3	12.5 ± 1.6
6	0.495	2.8 ± 0.3	12.6 ± 1.4
10	0.552	2.9 ± 0.3	13.3 ± 1.4
20	0.814	2.9 ± 0.3	13.1 ± 1.4
30	1.24	3.6 ± 0.3	16.3 ± 1.5
40	1.82	4.5 ± 0.4	20.2 ± 1.7
50	2.54	4.2 ± 0.4	18.9 ± 1.6
60	3.37	4.7 ± 0.4	21.2 ± 1.7
70	4.29	4.3 ± 0.4	19.4 ± 1.7

TABLE 7.

<div> <div>H₂O 95 °C</div> <div>$\beta = -2.76$</div> <div>Case 551</div> </div>			
<div> <div>$E_1 = 149 \text{ meV}$</div> <div>$E_2 = 62 \text{ meV}$</div> <div>$\Delta E = 87.5 \text{ meV}$</div> </div>			
<div> <div>$\delta E_1 = 7.1$</div> <div>$\delta E_2 = 6.8$</div> </div>			
<div> <div>Be(0002)</div> <div>Cu(002)</div> </div>			
ϕ	α	$S \cdot 10^2$	$d^2\sigma/d\Omega dE$
6	0.63	1.8 ± 0.2	9.6 ± 1.1
10	0.69	1.8 ± 0.2	9.3 ± 1.1
20	0.96	2.3 ± 0.2	12.2 ± 1.2
30	1.41	2.8 ± 0.3	14.9 ± 1.4
40	2.02	3.1 ± 0.3	16.2 ± 1.4
50	2.77	4.1 ± 0.3	21.6 ± 1.6
60	3.64	4.2 ± 0.3	21.9 ± 1.7
70	4.65	3.5 ± 0.3	18.4 ± 1.6

TABLE 8.

<div> <div>H₂O 95 °C</div> <div>$\beta = -3.15$</div> <div>Case 549</div> </div>			
<div> <div>$E_1 = 162 \text{ meV}$</div> <div>$E_2 = 62 \text{ meV}$</div> <div>$\Delta E = 100 \text{ meV}$</div> </div>			
<div> <div>$\delta E_1 = 8.1$</div> <div>$\delta E_2 = 6.8$</div> </div>			
<div> <div>Be(0002)</div> <div>Cu(002)</div> </div>			
ϕ	α	$S \cdot 10^2$	$d^2\sigma/d\Omega dE$
4	0.76	0.95 ± 0.22	7.4 ± 1.7
6	0.78	1.52 ± 0.21	9.3 ± 1.3
10	0.84	1.68 ± 0.22	10.2 ± 1.4
20	1.13	1.72 ± 0.24	10.5 ± 1.4
30	1.59	2.53 ± 0.28	15.5 ± 1.7
40	2.22	2.69 ± 0.29	16.5 ± 1.8
50	3.00	2.68 ± 0.28	16.4 ± 1.7
60	3.91	2.92 ± 0.30	17.9 ± 1.8
70	4.91	2.87 ± 0.30	17.5 ± 1.9

TABLE 9.

<u>H₂O 95 °C</u>		<u>$\beta = -3.15$</u>	<u>Case 543</u>
$E_1 = 214 \text{ meV}$		$E_2 = 114 \text{ meV}$	$\Delta E = 100 \text{ meV}$
$\delta E_1 = 12$		$\delta E_2 = 8.0$	
Be(0002)		Cu(002)	
<u>ϕ</u>	<u>α</u>	<u>$S \cdot 10^2$</u>	<u>$d^2\sigma/d\Omega dE$</u>
4	0.52	0.84 ± 0.29	6.0 ± 2.1
6	0.55	0.89 ± 0.21	6.5 ± 1.5
10	0.64	1.11 ± 0.19	8.0 ± 1.4
20	1.09	1.62 ± 0.20	11.7 ± 1.5
30	1.81	2.06 ± 0.23	14.9 ± 1.7
40	2.80	2.38 ± 0.25	17.1 ± 1.8
50	4.02	2.26 ± 0.24	16.3 ± 1.7
60	5.42	2.30 ± 0.27	16.6 ± 1.9
70	6.98	1.84 ± 0.26	13.2 ± 1.8

TABLE 10.

<u>H₂O 95 °C</u>		<u>$\beta = -3.15$</u>	<u>Case 558</u>
$E_1 = 214 \text{ meV}$		$E_2 = 114 \text{ meV}$	$\Delta E = 100 \text{ meV}$
$\delta E_1 = 12$		$\delta E_2 = 17$	
Be(0002)		Cu(002)	
<u>ϕ</u>	<u>α</u>	<u>$S \cdot 10^2$</u>	<u>$d^2\sigma/d\Omega dE$</u>
6	0.55	0.74 ± 0.11	5.3 ± 0.8
10	0.64	0.85 ± 0.11	6.2 ± 0.8
20	1.09	1.15 ± 0.13	8.3 ± 0.9
30	1.81	1.62 ± 0.13	11.6 ± 0.9
40	2.80	2.42 ± 0.16	17.5 ± 1.1
50	4.02	2.16 ± 0.17	15.5 ± 1.2
60	5.42	2.22 ± 0.17	16.0 ± 1.2
70	6.98	1.71 ± 0.16	12.3 ± 1.2

TABLE 11.

<u>H₂O 95 °C</u>		<u>$\beta = -3.15$</u>	<u>Average: Case 371 & 391</u>
$E_1 = 214 \text{ meV}$		$E_2 = 114 \text{ meV}$	$\Delta E = 100 \text{ meV}$
$\delta E_1 = 23$		$\delta E_2 = 17$	
Be(0002)		Be(0002)	
<u>ϕ</u>	<u>α</u>	<u>$S \cdot 10^2$</u>	<u>$d^2\sigma/d\Omega dE$</u>
4	0.517	0.72 ± 0.12	4.4 ± 0.7
8	0.589	1.19 ± 0.06	7.3 ± 0.4
12	0.708	1.14 ± 0.05	7.0 ± 0.3
16	0.875	1.16 ± 0.05	7.1 ± 0.3
20	1.09	1.36 ± 0.06	8.3 ± 0.4
24	1.35	1.75 ± 0.06	10.7 ± 0.4
28	1.65	1.92 ± 0.06	11.8 ± 0.4
32	1.99	2.16 ± 0.07	16.3 ± 0.4
36	2.38	2.28 ± 0.07	14.0 ± 0.4
40	2.80	2.36 ± 0.07	14.5 ± 0.4
44	3.26	2.58 ± 0.07	15.8 ± 0.4
48	3.76	2.68 ± 0.06	16.4 ± 0.4
52	4.28	2.74 ± 0.06	16.8 ± 0.4
56	4.84	2.63 ± 0.06	16.1 ± 0.4
60	5.42	2.51 ± 0.06	15.4 ± 0.4

TABLE 12.

<u>H₂O 95 °C</u>		<u>$\beta = -3.15$</u>	<u>Case 406</u>
$E_1 = 214 \text{ meV}$		$E_2 = 114 \text{ meV}$	$\Delta E = 100 \text{ meV}$
$\delta E_1 = 11$		$\delta E_2 = 7.9$	
Be(11 $\overline{2}$ 0)		Be(0002)	
<u>ϕ</u>	<u>α</u>	<u>$S \cdot 10^2$</u>	<u>$d^2\sigma/d\Omega dE$</u>
4	0.517	1.07 ± 0.22	7.7 ± 1.5
8	0.589	0.78 ± 0.20	5.6 ± 1.5
12	0.708	1.28 ± 0.17	9.2 ± 1.2
16	0.875	1.16 ± 0.19	8.3 ± 1.4
20	1.09	0.81 ± 0.21	5.8 ± 1.5
24	1.35	1.55 ± 0.21	11.1 ± 1.5
28	1.65	1.50 ± 0.22	10.8 ± 1.6
32	1.99	1.99 ± 0.24	14.3 ± 1.7
36	2.38	2.41 ± 0.23	17.3 ± 1.6
40	2.80	2.71 ± 0.25	19.5 ± 1.8
44	3.26	2.40 ± 0.23	17.2 ± 1.5
48	3.76	2.41 ± 0.23	17.3 ± 1.6
52	4.28	2.45 ± 0.22	17.5 ± 1.6
56	4.84	2.74 ± 0.24	19.7 ± 1.7
60	5.42	2.18 ± 0.26	15.6 ± 1.9
64	6.03	2.32 ± 0.31	16.7 ± 2.0
68	6.66	2.32 ± 0.31	16.6 ± 2.2
70	6.98	1.90 ± 0.31	13.7 ± 2.2

TABLE 13.

<u>H₂O 95 °C</u>		<u>$\beta = -3.15$</u>	<u>Case 369</u>
$E_1 = 162 \text{ meV}$		$E_2 = 62 \text{ meV}$	$\Delta E = 100 \text{ meV}$
$\delta E_1 = 8.1$		$\delta E_2 = 6.7$	
Be (0002)		Be(0002)	
<u>Φ</u>	<u>α</u>	<u>$S \cdot 10^2$</u>	<u>$d^2\sigma/d\Omega dE$</u>
3	0.752	1.20 ± 0.09	7.3 ± 0.6
4	0.759	1.42 ± 0.08	8.7 ± 0.5
8	0.805	1.45 ± 0.06	8.9 ± 0.3
12	0.882	1.56 ± 0.06	9.5 ± 0.3
16	0.989	1.73 ± 0.06	10.6 ± 0.4
20	1.13	1.88 ± 0.06	11.5 ± 0.4
24	1.29	2.16 ± 0.08	13.2 ± 0.5
28	1.48	2.17 ± 0.07	13.3 ± 0.4
32	1.71	2.36 ± 0.07	14.4 ± 0.4
36	1.95	2.46 ± 0.08	15.1 ± 0.5
40	2.22	2.55 ± 0.07	15.6 ± 0.4
44	2.52	2.64 ± 0.07	16.2 ± 0.5
48	2.84	2.78 ± 0.07	17.0 ± 0.4
52	3.18	2.77 ± 0.07	17.0 ± 0.4
56	3.53	2.79 ± 0.07	17.1 ± 0.4
60	3.91	2.65 ± 0.07	16.2 ± 0.4

TABLE 14.

<u>H₂O 95 °C</u>		<u>$\beta = -3.94$</u>	<u>Case 404</u>
$E_1 = 239 \text{ meV}$		$E_2 = 114 \text{ meV}$	$\Delta E = 125 \text{ meV}$
$\delta E_1 = 15$		$\delta E_2 = 7.9$	
Be(0002)		Be(0002)	
<u>ϕ</u>	<u>α</u>	<u>$S \cdot 10^3$</u>	<u>$d^2\sigma/d\Omega dE$</u>
4	0.747	4.7 ± 1.8	4.7 ± 1.8
8	0.823	4.5 ± 1.2	4.5 ± 1.2
12	0.950	4.4 ± 1.1	4.4 ± 1.1
16	1.13	9.1 ± 1.1	9.1 ± 1.1
20	1.35	6.0 ± 1.1	6.1 ± 1.1
24	1.62	10.0 ± 1.1	10.1 ± 1.1
28	1.94	11.4 ± 1.5	11.5 ± 1.1
32	2.31	10.6 ± 1.7	10.7 ± 1.1
36	2.71	8.1 ± 1.8	8.2 ± 1.1
40	3.16	12.6 ± 1.9	12.7 ± 1.9
44	3.65	11.1 ± 1.8	11.2 ± 1.8
48	4.17	14.0 ± 1.9	14.1 ± 1.9
52	4.73	15.0 ± 2.1	15.1 ± 2.1
56	5.32	16.1 ± 2.2	16.2 ± 2.2
60	5.93	15.1 ± 2.2	15.2 ± 2.2
64	6.57	15.3 ± 2.3	15.4 ± 2.3
68	7.24	17.3 ± 2.2	17.4 ± 2.2

TABLE 15.

<u>H₂O 95 °C</u>		<u>$\beta = -3.94$</u>	<u>Average: Cases 559-560</u>
$E_1 = 239$ meV		$E_2 = 114$ meV	$\Delta E = 125$ meV
$\delta E_1 = 15$		$\delta E_2 = 17$	
Be(0002)		Cu(002)	
<u>ϕ</u>	<u>α</u>	<u>$S \cdot 10^3$</u>	<u>$d^2 \sigma / d\Omega dE$</u>
6	0.78	5.4 ± 0.6	5.5 ± 0.6
10	0.88	4.7 ± 0.5	4.8 ± 0.5
20	1.35	7.9 ± 0.9	8.1 ± 0.9
30	2.12	9.1 ± 0.8	9.3 ± 0.8
40	3.16	11.0 ± 0.8	11.2 ± 0.8
50	4.44	13.1 ± 0.7	13.3 ± 0.7
60	5.93	13.6 ± 0.7	13.9 ± 0.7
70	7.58	12.1 ± 0.8	12.3 ± 0.8

TABLE 16.

<u>H₂O 95 °C</u>		<u>$\beta = -4.73$</u>	<u>Case 547</u>
$E_1 = 264$ meV		$E_2 = 114$ meV	$\Delta E = 0.175$ meV
$\delta E_1 = 16$		$\delta E_2 = 17$	
Be(0002)		Cu(002)	
<u>ϕ</u>	<u>α</u>	<u>$S \cdot 10^3$</u>	<u>$d^2 \sigma / d\Omega dE$</u>
4	1.01	2.9 ± 0.8	4.3 ± 1.1
6	1.04	2.7 ± 0.8	4.0 ± 0.9
10	1.15	2.3 ± 0.6	3.4 ± 0.8
20	1.64	3.4 ± 0.6	5.0 ± 0.8
30	2.45	4.1 ± 0.6	5.9 ± 0.8
40	3.54	6.9 ± 0.7	10.0 ± 1.0
50	4.89	6.3 ± 0.8	9.1 ± 1.1
60	6.46	8.1 ± 0.8	11.8 ± 1.2
70	8.19	7.5 ± 0.8	10.9 ± 1.2

TABLE 17.

<u>H₂O 95 °C</u>		<u>$\beta = -5.52$</u>	<u>Case 544</u>
$E_1 = 289 \text{ meV}$		$E_2 = 114 \text{ meV}$	$\Delta E = 175 \text{ meV}$
$\delta E_1 = 18$		$\delta E_2 = 17$	
Be(0002)		Cu(002)	
<u>ϕ</u>	<u>α</u>	<u>$S \cdot 10^3$</u>	<u>$d^2_{\sigma}/d\Omega dE$</u>
4	1.29	2.3 ± 0.5	4.6 ± 1.1
6	1.33	2.2 ± 0.4	4.4 ± 0.8
10	1.61	2.6 ± 0.4	5.4 ± 0.8
20	1.95	2.0 ± 0.4	4.1 ± 0.8
30	2.80	3.3 ± 0.5	6.7 ± 1.0
40	3.94	4.6 ± 0.5	9.3 ± 1.1
50	5.36	5.7 ± 0.6	11.5 ± 1.1
60	6.99	5.5 ± 0.6	11.2 ± 1.2
70	8.80	5.1 ± 0.6	10.3 ± 1.1

TABLE 18.

<u>H₂O 95 °C</u>		<u>$\beta = -5.52$</u>	<u>Case 544</u>
$E_1 = 377 \text{ meV}$		$E_2 = 202 \text{ meV}$	$\Delta E = 0.175 \text{ meV}$
$\delta E_1 = 54$		$\delta E_2 = 40$	
Be(0002)		Cu(002)	
<u>ϕ</u>	<u>α</u>	<u>$S \cdot 10^3$</u>	<u>$d^2_{\sigma}/d\Omega dE$</u>
6	0.95	1.2 ± 0.3	2.4 ± 0.8
10	1.12	1.1 ± 0.3	2.2 ± 0.6
20	1.91	2.0 ± 0.3	4.4 ± 0.7
30	3.19	3.2 ± 0.3	6.6 ± 0.6
40	4.93	4.0 ± 0.3	8.1 ± 0.7
50	7.08	4.6 ± 0.4	9.2 ± 0.8
60	9.57	3.4 ± 0.4	6.8 ± 0.8

TABLE 19.

<u>H₂O 95 °C</u>		<u>$\beta = -5.52$</u>	<u>Average Case 393 & 395</u>
$E_1 = 377 \text{ meV}$		$E_2 = 202 \text{ meV}$	$\Delta E = 175 \text{ meV}$
$\delta E_1 = 27$		$\delta E_2 = 19$	
Be(11 $\bar{2}$ 0)		Be(0002)	
<u>ϕ</u>	<u>α</u>	<u>$S \cdot 10^3$</u>	<u>$d^2\sigma/d\Omega dE$</u>
8	1.02	0.86 ± 0.72	1.7 ± 1.5
12	1.24	0.65 ± 0.76	1.3 ± 1.5
16	1.53	1.22 ± 0.63	2.5 ± 1.3
20	1.91	1.20 ± 0.67	2.4 ± 1.4
24	2.36	2.32 ± 0.63	4.7 ± 1.3
28	2.89	3.97 ± 0.63	8.0 ± 1.3
32	3.50	4.05 ± 0.67	8.2 ± 1.4
36	4.18	5.52 ± 0.67	11.2 ± 1.4
40	4.93	6.02 ± 0.67	12.2 ± 1.4
44	5.74	6.24 ± 0.67	12.6 ± 1.4
48	6.62	6.47 ± 0.72	13.1 ± 1.5
52	7.55	6.02 ± 0.72	12.2 ± 1.5
56	8.53	6.60 ± 0.72	13.4 ± 1.5
60	9.57	5.48 ± 0.67	11.1 ± 1.4
64	10.6	5.28 ± 0.67	10.7 ± 1.4
68	11.7	4.84 ± 0.67	9.8 ± 1.4

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