



THE UNIVERSITY
of LIVERPOOL

Department of Physics

PHYS378

Advanced Practical Physics (BSc)

PHYS478

Advanced Practical Physics (MPhys)

Laboratory Handbook

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Advanced Practical Physics

This handbook applies to both modules PHYS378 Advanced Practical Physics (BSc) and PHYS478 Advance Practical Physics (MPhys). In this handbook the term ‘BSc students’ refers to all students taking PHYS378 and ‘MPhys students’ refers to all students taking PHYS478, regardless of the actual degree programme for which a student is registered. BSc and MPhys students work side-by-side on the same experimental apparatus and follow essentially the same scripts. However, the level of understanding attained by MPhys students is expected to be higher, as outlined in the laboratory report guidelines below.

The laboratory is divided into three 4–week blocks labelled *A*, *B* and *C*. In each block students carry out:

- (i) Group work, led by a demonstrator, to become familiar with the concepts, instrumentation and software associated with a group of experiments.
- (ii) Experiments, working individually or in pairs, that apply the knowledge and understanding gained in the group work.

Block A **Radiation Detection**

Group work on the use of radiation detectors followed by two experiments selected from four on samples that have been activated by a source of thermal neutrons

Block B **X-Ray Diffraction**

Group work on computer modelling to simulate x-ray diffraction from crystals followed by experiments to determine the crystal structures and lattice constants of two unknown materials

Block C **Quantum and Waves**

Group work on paramagnetic resonance followed by two experiments selected from three demonstrating quantum or wave phenomena

In Block *C* it may be necessary, depending on the number of students taking these modules, for students to timeshare experimental apparatus. This means that two students may be assigned to one experiment for a two-week period, and will be expected to organise their time accordingly. Each student will take their *own* data and do their *own* analysis, interpretation and write-up. Discussion between students is of course allowed, but sharing of data is *not*. This also applies to experiments in Blocks *A* and *B* unless it is explicitly stated that students work as a pair — check with a demonstrator if you are unsure.

Students will carry out the experiments during the 12-week semester in the order *ABC*, *BCA* or *CAB*. Each block is self-contained, and all work in a block must be written up and handed in to the Student Office at the end of weeks 4, 8 and 12, as indicated below.

Week 1 2 3 4 5 6 7 8 9 10 11 12

<i>A</i>					<i>B</i>					<i>C</i>			
<i>B</i>					<i>C</i>					<i>A</i>			
<i>C</i>					<i>A</i>					<i>B</i>			

Assessment: 30% from each of the three blocks of experiments

(each block includes 5% for group work)

10% for the laboratory workbook, which must contain all raw data, analysis, plots, calculations and results.

The remainder of this handbook contains guidelines for conducting and writing up experiments, the material required for the group work, and scripts for each of the experiments.

Laboratory Report Guidelines

A student who makes a minimal attempt to follow the experimental scripts, acquires the relevant data, analyses and interprets the results and submits a report that is considered to be barely satisfactory, will obtain a mark of $\sim 4/10$.

A report that is well written (according to the guidelines given below) and indicates that the student has been diligent and conscientious in acquiring, analysing and interpreting the data will obtain a first class mark of $\sim 7/10$.

To obtain higher marks a student must demonstrate that he/she has contributed more than was required by the scripts and has mastered some or all aspects of the experiment. In this respect, there is greater expectation for the level of understanding attained by MPhys students, who will be expected to do more background reading, to expand on the physics concepts mentioned in the scripts, and to present the experiment in a wider context. To give some guidance on which areas are suitable for such treatment, the scripts have been annotated with asterisks (*).

Some general guidelines for the successful completion of laboratory experiments are given in this handbook. The instructions for individual experiments may offer more detailed advice.

A useful reference for practical physics is *Practical Physics* by Squires.

Finding Out About the Experiment

- Read the instructions thoroughly once. Do not worry if you do not understand every aspect of an experiment initially.
- Identify the pieces of apparatus and familiarise yourself with them.
- Try some test measurements to get a feel for the experiment.
- If there are still some points you feel you need to understand more clearly before taking the data ask a demonstrator. This should not be a substitute for thought – the demonstrator will expect you to have thought about the problem.

Taking the Data

- Establish the range and number of readings to be taken.
- Before starting to take a series of serious measurements, it is worth spending a few minutes to lay out the apparatus in an orderly way and tidy any wiring and cables so that you can read and record the instruments efficiently and comfortably.
- Record the raw data with their units directly into your workbook, not on scraps of paper. The raw measurements should be included in your report.
- Check your observations as you proceed, look out for any measurements that are out of line with the rest. Plotting a graph as you take the data is often the simplest way to identify any rogue measurements.
- You may need to repeat some measurements to determine the random error associated with the measurement.

Doing the Analysis

- Write the analysis directly into your workbook, not on scraps of paper.
- Show the key intermediate mathematical steps of the calculation in your report. This will help both you and the demonstrator who assesses the work if you make a mistake in the calculation.
- Consider the experimental errors in the measured quantities and estimate the error in any derived quantity. This may require repeating measurements to estimate the spread in the reading.
- Measurements should be presented in a sensible form and include an error and a percentage error. The reading may be $1.23567468 \times 10^{-18} \text{ m} \pm 0.534873678 \times 10^{-20} \text{ m}$ but presented correctly would appear as $(1.236 \pm 0.05) \times 10^{-18} \text{ m} (0.4\%)$

Plotting Graphs

- When investigating the relation between two measured quantities, always plot the data on a graph. It is the easiest way to judge the consistency of the data.
- Label the axes of the graph clearly, including the units of the quantities plotted.
- Make the graph large enough to illustrate the quality of the data. As a rule of thumb, graphs with real data points should be at least half a page of A4. It is increasingly common to see small graphs in word-processed reports. Whilst these are adequate as illustrations, they are a poor visual indication to the assessor of how well you have taken the data.
- As general rule use chose the range of the scale on the graph to match the range of the data. Including zero on one or both axis often helps the visual interpretation of the graph, but not usually at the expense of large areas of unused graph paper. Common sense and experience are the best guides.
- Use error bars on the data points.

Presenting Your Work

The primary deliverable of an experiment is a measurement of a physical quantity together with its error. The written report should contain all the evidence for this result in a form that can be checked by a third party.

A good report should include the following

- A summary page at the start of the report showing the results and conclusions
- An introduction
- A brief outline of the method
- The raw data and any derived data including errors
- A proper error analysis of the derived results, including the error presented as a percentage and the largest/most significant error identified
- Easy to follow examples of all calculations
- Any graphical interpretations of the data including error bars
- Any line fit results and interpretations of graphs
- A conclusion that includes derived numbers quoted to a sensible number of decimal points
- Answers to any questions asked in the script should be given. It is up to you how these appear in the report

Credit will be given for further discussion demonstrating an insight and total understanding of the experiment.

Errors and Their Treatment

Why Estimate Errors?

A measurement of a physical quantity is unlikely to yield exactly the true value. An estimate of the size of the likely difference between the true and measured value is important if significant conclusions are to be drawn from the result. Since the true value is not known, the most likely size of the error on the measured value must be estimated and quoted along with the result.

Random and Systematic Errors

A random error occurs when repeated measurements vary around the mean value without a pattern. For example, reading the position of a pointer on the scale of a meter has a random error associated with it. Random errors can usually be estimated from the spread in repeated measurements.

Systematic errors are usually more difficult to estimate and must be identified by consideration of the experimental method. A simple example of a systematic error is one that is the same throughout a set of readings. For example if a meter is not zeroed correctly, all the readings taken with it will be offset by the same amount. More subtly, a systematic error can vary during an experiment perhaps due to drift in the calibration of an instrument.

At the research level, rigorous error analysis can be difficult and sometimes impossible. It is however important to attempt an error analysis and to state clearly in any report of an experiment how the error has been estimated so that the reader can understand the significance of the result and compare it with the results of the experiment. This is why it is important to develop an understanding of error analysis and why you will always be expected to perform an error analysis of experiments.

Estimating Errors in Practice

- In experiments in the undergraduate laboratories, you should identify possible sources of systematic errors and eliminate or reduce them by correct use and calibration of the instruments. An estimate of remaining systematic errors should be made considering the operation of the instruments. For example the zero scale can be set to within, say ± 0.2 of the smallest division.
- Random errors can be estimated from the spread in repeated readings
- All the errors can then be combined to determine the error on the final result using the rules outlined in this document.
- Estimates of errors are liable to have errors themselves! As a rule of thumb it is usually adequate to determine the errors themselves to within about 10%. In consequence, error calculations can often be oversimplified by ignoring errors that are small compared with other errors. Some care is needed here as the analysis may be more sensitive to errors in particular quantities than others.

Errors in Reading Instruments

The error in reading a scale, for example on a ruler or on an analogue meter, can arise from a number of sources:

- a) ***Parallax error***: If the line of sight is not at right angles to the scale, a gap between the object being measured (the pointer in the case of the meter) and the scale will cause an error. This can be reduced by careful alignment of the eye, a process aided in better quality meters by a mirror built into the scale so that the pointer and its image can be lined up to ensure the scale is viewed at right angles.
- b) ***Zeroing error***: Most instruments have the provision to set the reading to zero when zero input is present. If the instrument is not correctly zeroed, actual reading will be offset by the offset of the zero. This offset can be measured and a correction applied but it is good practice to always zero the instrument so that the reading can be used without correction.

- c) **Scale reading errors:** The scale can only be read to some accuracy which depends on how finely the scale is engraved. A conservative rule of thumb is assume the scale can be read to a half of the smallest division. However a fifth of the smallest interval can often be achieved. In practice, you make a judgement based on your use of the particular instrument tempered with experience and common sense. A digital scale can be read ± 1 in the least significant digit displayed provided the reading is stable. As for analogue instruments there will be a zeroing error.

Calibration Errors

The accuracy of the reading of every instrument, analogue or digital, will depend on the calibration. Manufactures will usually supply details of the accuracy of the calibration of the instruments at the point of manufacture. A data sheet from the manufacturer with this information should be available close to the equipment in the laboratory. Unless told otherwise in the instructions, you can assume the calibration is correct. However, possible calibration errors should not be ignored if you have to do any trouble-shooting on the data.

Calibration errors may take the form of an overall multiplicative constant. Instruments will often have some internal adjustment to set this. More commonly, calibration errors will manifest themselves as small deviations around the marked scale due, for example, to the quality of construction of the instrument. These can be accounted for by calibrating the scale against an (expensive) standard instrument. If this is advisable, the instructions will tell you.

General Comment

It is very useful indeed to quote the errors both as a number with units and as a percentage e.g.

Measured density of nitrogen = $(811 \pm 2) \text{ kg m}^{-3}$ (0.25% error)

This lets you identify easily the largest error in the experiment, hence find which measurement should be improved to obtain a more accurate final result. Working through your error calculations using errors expressed as percentages (rounded to the nearest integer %) is one way the demonstrators spot mistakes in your error calculations when marking your report.

The Distribution of Measurements

A set of measurements of the same quantity free of systematic errors will typically show a distribution about the true value. The best estimate of the true value of the quantity will be given by the mean of these readings:

$$\text{Mean:} \quad \bar{x} = \frac{\sum x_i}{N}$$

where $N =$ number of readings

$x_1, x_2, \dots, x_i, \dots, x_N =$ the N values of x

The error on a single reading is given by the spread of a distribution.

$$\text{Standard error on a single reading: } \sigma_{n-1} = \sqrt{\frac{\sum (x_i - \bar{x})^2}{N-1}}$$

Multiple readings reduce the error on the mean by the factor $\frac{1}{\sqrt{N}}$

$$\text{Standard error on the mean:} \quad \Delta x = \sqrt{\frac{\sum (x_i - \bar{x})^2}{N(N-1)}} = \frac{\sigma_{n-1}}{\sqrt{N}}$$

Quoting Results

The result of an experiment should be quoted as:

$$(\bar{x} \pm \Delta x) \text{ UNITS} \quad \left(\left(\frac{\Delta x}{\bar{x}} \times 100 \right) \% \text{ error} \right) \quad \text{for example, } (811 \pm 2) \text{ kg m}^{-3} \quad (0.25\% \text{ error})$$

Round the value of the measured figure to the same significance. For example:

$$980.5 \pm 1.2$$

When large or small powers of ten are involved, it is neater to use brackets:

$$(8.033 \pm 0.015) \times 10^{10}$$

$$(6.6726 \pm 0.0009) \times 10^{-11}$$

DO NOT FORGET TO INCLUDE THE UNITS

Consistency

Two independent measurements of the same quantity $x_1 \pm \Delta x_1$ and $x_2 \pm \Delta x_2$ are said to be consistent if:

$$|x_1 - x_2| < 3\sqrt{\Delta x_1^2 + \Delta x_2^2}$$

Rules for Combining Errors

The rules below show how an error in a measured quantity propagates into an error in a derived quantity. ΔA refers to an error in A and is assumed small compared to A .

1. Addition or subtraction

i.e. $Z = A + B$ or $Z = A - B$ $\Delta Z = \sqrt{(\Delta A)^2 + (\Delta B)^2}$

Errors added in quadrature

2. Multiplication or division

i.e. $Z = A \times B$ or $Z = A \div B$ $\frac{\Delta Z}{Z} = \sqrt{\left(\frac{\Delta A}{A}\right)^2 + \left(\frac{\Delta B}{B}\right)^2}$

Fractional errors added in quadrature

3. Multiplication by a constant

i.e. $Z = kA$ $\Delta Z = k\Delta A$ or $\frac{\Delta Z}{Z} = \frac{\Delta A}{A}$

Either multiply the actual error by k or equate fractional errors.

4. Indices

i.e. $Z = A^n$ where n is a constant $\frac{\Delta Z}{Z} = n \frac{\Delta A}{A}$

Fractional error multiplied by n

5. Functions

i.e. $Z = f(A)$ where Z is a function of the measured value A $\Delta Z = \frac{dZ}{dA} \Delta A$

The error in the measured value multiplied by the analytical differentiation of the function with respect to the measured value.

For more complicated functions the above rules must be applied in sequence.

Note: Care must be taken if a quantity appears more than once in the calculation to avoid the effects of correlated errors.

The Law of Combination of Errors

If $z = f(x, y)$, then a small error δz in z is related to small errors δx in x and δy in y by (the following expression is only valid if x and y are independent):

$$\delta z^2 = \left(\frac{\partial f(x, y)}{\partial x} \right)^2 \delta x^2 + \left(\frac{\partial f(x, y)}{\partial y} \right)^2 \delta y^2$$

This says that the error on x and y , multiplied by suitable scaling factors, are added in quadrature. Adding two positive numbers in quadrature gives a smaller result than the usual arithmetic addition. Intuitively you can see that this is reasonable: errors of overestimation in x have a fair chance of being compensated by errors of underestimation in y .

Further information on combination of errors can be found in *Statistics A Guide to the Use of Statistical Methods in the Physical Sciences* by R J Barlow, published by Wiley.

Examples

The following examples are intended as a reference and a reminder of how to apply the rules laid out in the above text.

i) $f(x, y) = z = x + y$

Using the combination of errors rule $\frac{\partial f}{\partial x} = 1$ and $\frac{\partial f}{\partial y} = 1$

\therefore plugging the above into the combination of errors rule gives $\delta z^2 = \delta x^2 + \delta y^2$

This should be easily recognised as adding errors in quadrature as is written in rule 1

ii) $z = kx^n \quad \Rightarrow \quad \frac{\partial z}{\partial x} = knx^{n-1} \quad \Rightarrow \quad \delta z = knx^{n-1} \delta x$

Dividing through by z gives $\frac{\delta z}{z} = n \frac{\delta x}{x}$

This is a combination of rules 3 and 4

iii) $z = k \sin \theta \quad \Rightarrow \quad \frac{\partial z}{\partial \theta} = k \cos \theta \quad \Rightarrow \quad \delta z = k \cos \theta \delta \theta$

An example of the use of rule 5

iv) $z = kxy \quad \Rightarrow \quad \frac{\partial z}{\partial x} = ky \quad \text{and} \quad \frac{\partial z}{\partial y} = kx$

$\delta z^2 = k^2 y^2 \delta x^2 + k^2 x^2 \delta y^2 \quad \Rightarrow \quad \left(\frac{\delta z}{z}\right)^2 = \left(\frac{\delta x}{x}\right)^2 + \left(\frac{\delta y}{y}\right)^2$

A combination of rules 2 and 3

Computing Information

Computers are available in T2 for the use of physics students. These are not connected to the managed network.

Computer Viruses

The Virus software on the computers in T2 will automatically pick up any infected files. If you get an infected file the lab technician will be able to clean it for you.

Inputting data into the straight line fitting program

Many laboratory experiments will require you to fit a straight line to your data. Data can be directly entered into the line fitting program, however, when the data has to be manipulated into the correct form, it is usually quicker, particularly with large data sets, to enter the data on an Excel spreadsheet. This can then be saved in a form that the line fitting program will recognise.

Setting up the spreadsheet

In order to write the file of data for the line fitting program correctly, it is essential that the numbers for the fit be entered in four adjacent columns in the order

x	Δx	y	Δy
-----	------------	-----	------------

Each set of reading must be on adjacent rows. The block must contain only numbers if the fitting routine is to read the data correctly.

Writing the file

- Select and copy the block containing the data values by highlighting the data and clicking on copy 
- Open a new spread sheet by clicking on the “New” icon 
- From the Edit menu chose “*Paste Special*”. Check the “*Values*” button. This will paste the values only into the new spreadsheet.
- Save the spread sheet by clicking on “*save as*” in the File menu and changing the file type in the “*save as type*” box to “CSV (Comma delimited) (*.csv)”, then click on “*Save*”. A warning message will appear on the screen click “*OK*” . This will save the values in the

same form as they appear on the screen in a way that the line-fitting program can understand.

- The CSV file in Excel MUST be closed before the line fitting program is used or else the file will be inaccessible for use. To do this click on the “Close Window” icon,  An dialogue box will appear click on “No”. The window should close.

Running the Line fitting program

- The line fitting program is menu driven with options and instructions shown at the bottom of the screen
- Press “L” to load a file and enter the path, file name and extension for the file you want to open e.g. “A:/book1.CSV”.
- If you cannot remember the file name press “F1” and then the drive letter e.g. “A” for the floppy drive. This will then let you search for the file using the cursor keys. Highlight the file and press “Enter” to load the file.
- Check the numbers have appeared in the table correctly and press “F1” to perform a fit. Other options are available and can be found at the bottom of the screen.
- To save the fit data press the “S” key. Chose which option you want to save by pressing the relevant key. Change the default file name to what you want making sure it is of the form “A:/FILE NAME.CSV” or “C:/FILE NAME.CSV” and press “Enter”.
- The fit results can then be opened in Excel and used.

Importing the results of the Line fitting program to a spread sheet

- To import the results back into the spread sheet simply open the *.CSV file. Excel will open it into a new spread sheet. This can then be copied and pasted into your lab write up.
- If you have not included the extension CSV, when you attempt to open the file, EXCEL will recognise it is not a standard Excel file and will display the *Text Import Wizard*. Check the box “Delimited” and click on “Next” button. Check “Comma” as a delimiter and “double quotes” as the Text Qualifier (this should be set already). The window should display the results, with the slope and intercept and text in separate cells. Click on “Finish” to load the spreadsheet.
- The data can then be moved using Copy and Paste.

Safety in Laboratories

The following points supplement the general information about Safety and Fire Precautions in the Department of Physics Undergraduate Student Handbook. It is your responsibility to read these regulations and those below. You should exercise particular care with experiments and take note of any local rules posted near experiments.

- It is your responsibility to work at all times in a way that ensures your own safety and that of other persons in the laboratory
- None of the experiments in the laboratory are dangerous provided that normal practices are followed.
- Due care and procedure must be followed in the handling of radioactive materials.
- The treatment of serious injuries must take precedence over all other actions, including the containment and cleaning up of radioactive materials.
- If you are uncertain about any safety matter for any experiment, you must consult a demonstrator. This includes any health condition which you think may be effected by any of the techniques you are expected to use.
- All accidents must be reported to the laboratory supervisor or technician who will take the necessary action.
- After an accident, a report form, which can be obtained from the technician must be completed and given to the laboratory supervisor.
- Clothing and bags must not be placed on the benches or obstruct the aisles.
- Food and drink must not be consumed within the laboratory.

Risk assessments for each experiment (or groups of experiments) are included at the ends of the sections of this handbook that describe the Block A, Block B and Block C experiments.

Block A

Radiation Detection

Radiation Detection

Aims

- To become familiar with the characteristics of scintillation detectors and the associated instrumentation and software
- To carry out experiments involving radiation detectors that utilise and build upon the exercises

Structure of Experiment

Sessions

- Group exercises measuring characteristics of a scintillation detector: Calibration, Resolution, Efficiency 1 – 2
- Two experiments drawn from a pool of five 3 – 10
- Write up experimental report 11 – 12

Characteristics of Scintillation Detectors

Apparatus

In this exercise you will measure the properties of a NaI(Tl) scintillation detector. Set up the electronics as shown in the block diagram below

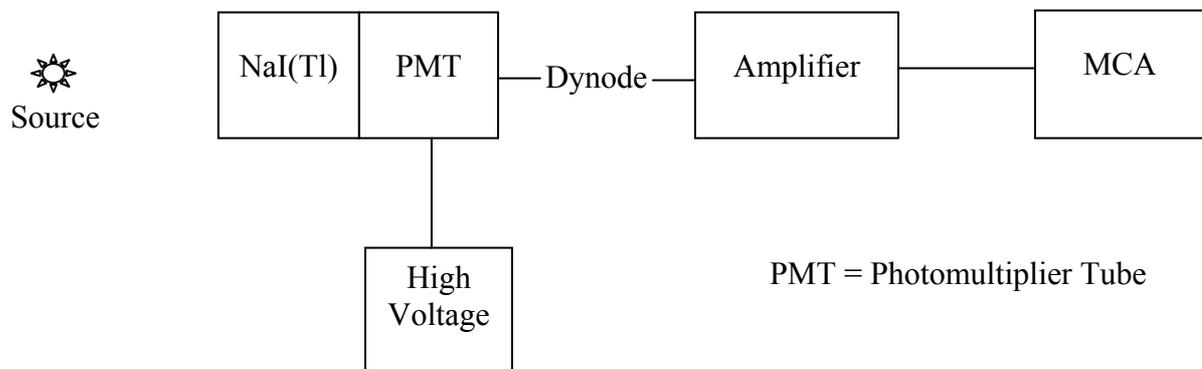


Figure 1: Apparatus layout

Before you switch on the high voltage (HV) get a demonstrator to check the circuit.

Switch on the high voltage.

1. Energy Calibration

To calibrate the spectrum two or more γ -rays of known energy need to be recorded. In the exercise a ^{22}Na , which has two strong γ -rays, will be used. A two-point calibration can then be used.

Connect the output of the amplifier to the multichannel analyser (MCA). Set the gain of the amplifier such that both peaks are seen in the MCA spectrum. Collect a spectrum until there are at least 1000 counts in the highest channel of the high-energy peak. Note down below in the table the values for the energy and channel number.

Energy (keV)	Channel Number

Assuming there is a linear relationship between the channel number and energy calibrate the spectrum using the MCA. Report your results in terms of the slope and intercept, where,

$$\text{gradient} = \frac{E1 - E2}{C1 - C2} \quad \text{[keV/channel]}$$

and

$$\text{Intercept} = E2 - (\text{gradient} \times C2) \quad \text{[keV]}$$

where C1 (C2) is the channel number for energy E1 (E2). Summarise the calibration factors here:

		Units
Gradient		
Intercept		

Radiation Detection Group Work

The purpose of this exercise is to use the calibration to determine the energies of an unknown gamma-ray source and identify the unknown isotope. Obtain the source from your demonstrator and collect a spectrum.

List the energies of the photopeaks observed.

Energy of peaks (keV)

Use the reference provided to identify the source.

The source is:

2. Resolution of a NaI(Tl) detector

For this exercise a ^{137}Cs and a ^{22}Na source will be used. The resolution is defined by the following relationship

$$R = \frac{\Delta E}{E} \times 100 \quad [\%]$$

where

ΔE = Full width of the peak at half its maximum height (FWHM)

E = Energy

Measure the resolution of the peaks in ^{22}Na and ^{137}Cs .

E (keV)	ΔE (keV)	R(%)
511		
1274		
662		

Now make a note of the noise level in the system. That is the point at which the spectrum increases rapidly at low energy.

Noise level =	keV
---------------	-----

3. Efficiency of a NaI (TI) detector

Carefully position a calibrated ^{137}Cs source a fixed known distance from the front of the detector. Collect a spectrum until there are about 50,000 counts in the area of the full energy peak. Complete the table below:

Detector type/size	
Nuclide	
Source Geometry (point/extended?)	
Source Activity (Bq)	
Gamma-ray energy (keV)	
Source to detector distance (mm)	
Net peak area	
Count period (live time)	

Note you will have to calculate the activity of the source on the day of the measurement from the activity of the source when produced, its half-life and age.

Calculate the absolute full energy peak efficiency for the detector for this gamma-ray in this geometry; use the relationship

$$E_{Abs} = \frac{\text{No of counts/sec}}{\text{Source Activity(Bq)} \times \text{Gamma fraction}}$$

For ^{137}Cs the gamma-ray decay branch (fraction) represents 85.1% of all decays. Hence the absolute full energy peak efficiency is:

E_{Abs}	%
Gamma-ray energy	keV
Source-to-detector distance	mm

Radiation Detection Group Work

The absolute efficiency of the detector depends on the source to detector distance. Repeat the absolute efficiency measurement for **three additional** source to detector distances. Put the results in the table

Source to detector distance (mm)	Efficiency (%)

Draw a graph of the result (Excel could be used).

Comment below on the form the graph takes.

Radiation Detection and Neutron Activation Experiments

Aims

In this experiment you will learn how to use a radiation detector for simple spectroscopy measurements and then use the detector to study the radiation emitted by samples that have been activated by a source of thermal neutrons. Five experiments are available (from which two will be assigned to you by a demonstrator) with measurements including: an estimate of the activity of the source; measurement of the half-lives of the activities induced in two different materials; measurements involving the astrophysical s-process cross section; and using a Geiger tube to measure the energy of electrons emitted by a neutron-activated source. Details of Experiments 1–4 can be found in the following pages. Students on the degree programme F350 Physics with Medical Applications may be assigned Experiment 5, which has a medical bias, and the script for this experiment will be provided by a demonstrator.

There is a booklet of supplementary information that will be needed during these experiments — **make sure you get a copy and read it.**

Background

An americium–beryllium neutron source is used to produce a source of fast (high energy) neutrons. The neutrons are products of the reaction between the beryllium and the 5.5 MeV alpha particles emitted by the decaying americium nuclei. These fast neutrons lose energy in the water tank. The water thermalises the neutrons and acts as a radiation shield. Thermal neutrons are low energy neutrons. Thermal neutrons can be absorbed by the samples to produce a source that decays by a combination of β -particles and gamma-ray emission. In these experiments, measurements are made on these sources using both electron and gamma-ray detectors. The data from the detectors are often collected with the aid of a computer so that a large data set is available for analysis. There is information in the laboratory both on the neutron activation process and the data collection programs.

Statistical Uncertainties

In a counting experiment the uncertainty on any count N is $\pm\sqrt{N}$. For example when you collect spectra counts are recorded in a series of channels. If the count in a channel is 156 the uncertainty is ± 12.5 . When calculating peak intensities the computer program will give you an uncertainty.

Question

- (i) What is the reaction used in the americium–beryllium source to produce the fast neutrons? What are the reaction products?
- (ii) What energy (in eV) do thermal neutrons have?

Experiment 1

Thermal Neutron Activity

Use the system in PHA mode, the same as for the introductory exercises. Use a thick sample of indium (NOT a foil) to produce a neutron activated indium source. Place the indium sample in the neutron tank in T7 for about 2-3 hours. Note the position of the sample relative to the neutron source. Transfer the indium source to the gamma-ray detector (quickly) and count a spectrum for a known time.

NOTE: Remember you are measuring the maximum activity of the sample reached during the time in the tank NOT the subsequent decay of the sample. If too long a time is used then corrections must be made for this subsequent decay and hence a short time should be used.

Use the data sheets provided to identify one gamma ray, measure its intensity and use the efficiency calibration (measured earlier) to deduce the gamma activity of the source. This should now be used to estimate the thermal neutron flux in which the sample was placed for activation. The formula is given in the notes provided.

NOTE: Take care, when placing the sample in the tank. Placing it too close is as bad as placing it too far away. The optimum distance is $\sim 4-6$ cm.

Question

Why is this the case? (Hint: Think about thermal neutrons).

Experiment 2

Half-Life Determination

Using the gamma-ray detector (NaI) and the MCS system (see the diagram) you should measure the half-life of the vanadium sample.

The steps are:

1. Activate the sample for about 3 half-lives
2. Use the detection system to measure the count rate as a function of time. Transfer the data to a spreadsheet.
3. Measure the background. Transfer the data to a spreadsheet and subtract the background.
4. Transfer the data to the least squares fit program to determine the half-life.
5. Compare with the tabulated values in the data book. Comment.

NOTE: Take care, when placing the sample in the tank. Placing it too close is as bad as placing it too far away. The optimum distance is $\sim 4\text{--}6$ cm.

Question

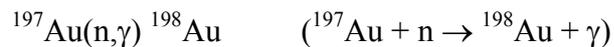
Why is this the case? (Hint: Think about thermal neutrons).

Experiment 3

The Astrophysical S-Process

Introduction

Elements heavier than iron are made by the neutron capture process. In this case a gold sample will be used. The reaction is:



The half-life of ${}^{198}\text{Au}$ is 2.7 days. The decay of ${}^{198}\text{Au}$ can be recognised by a 412 keV gamma-ray.

Method

Use the same set up as in Experiment 1. A gold sample will be activated for about 7–8 days.

Question

Why should the sample be activated for about 7–8 days?

NOTE: These are weak gamma-ray sources. You will need to decide how long to count for. You will also have to consider a method for background subtraction.

Radiation Detection Experiments

The spectrum has one dominant gamma ray. Use this (and the efficiency calibration carried out earlier) to determine the activity of the foil. Use the activation formula in the notes to determine the thermal neutron flux.

The experiment should be carried out by activating a sample in the neutron tank in T7 and repeated by using a sample activated in the carbon stack in T4 (you will need help from a demonstrator or technician for this).

Questions

- (i) For 10^6 neutrons/cm²/s how many ¹⁹⁸Au are made per second for 1 kg of ¹⁹⁷Au?
- (ii) Why might the activation process be different for the two neutron sources?
Think about the different moderators

Experiment 4

Electron Energy

Using the Geiger counter, find the maximum energy of the electrons emitted by the neutron-activated thin indium foil. This can be achieved by measuring the count rate as a function of absorber thickness. You will need to correct your data for the background, and the decay of the source during the measurements. Plan the experiment by deciding (using the references) the best way to measure the range.

From a plot of count rate versus absorber thickness, find the range of the electrons. The information given in the supplementary notes will help you do this. The notes will give you some relationships between the range and the electron energy. Choose an appropriate relationship to allow you to determine the maximum energy of the electrons emitted in the decay of the activated foil.

Notes On Multipurpose Data Collection System

Introduction

You have a PC equipped with a multipurpose data collection system. The system has 8192 channels of memory in which to store information. This is too much for the experiment so it will be divided into 32 blocks of 256 channels each. This is done using the SETUP command followed by MEMORY GROUP, then following the menu. Note that commands can be selected using the mouse. The right-hand button produces an arrow to allow menu commands to be accessed, the left-hand button allows commands to be selected.

Use of the Pulse Height Analysis (PHA) system

The output of the gamma-ray detector is connected to an amplifier that produces a voltage output (0–10 volts) which is energy dependent. This voltage is connected to the data collection system in the computer. The voltage is digitised and a 256 channel spectrum is produced. There is a manual available to help you with the software. Below are some of the most useful commands you will need.

MODE	Allows you to select the PHA/MCS function
	Allows add/subtract to be selected
SETUP/CONVERSION GAIN	Set to 256
CALC/CALIBRATE	Allows the spectrum to be calibrated in keV
FILE/SAVE BINARY FILE	Allows data to be stored on your disc

Most of the other commands you need are displayed on the screen are accessed via function keys. Some examples are:

F1	start/stop data collection
F2	erase the spectrum (CTRL F2 needed as a protection)
F3	allows the data collection time to be preset (0 = ∞)
ESC	accesses the Region Of Interest (ROI) menu
F9	start ROI
F10	end a ROI

Note that more than one ROI can be set on a spectrum. To find the intensity or area of a peak you should with the help of the cursor set a ROI around the peak. The net (background subtracted) peak intensity is then displayed as well as the total area. The time for the count can also be found on the screen.

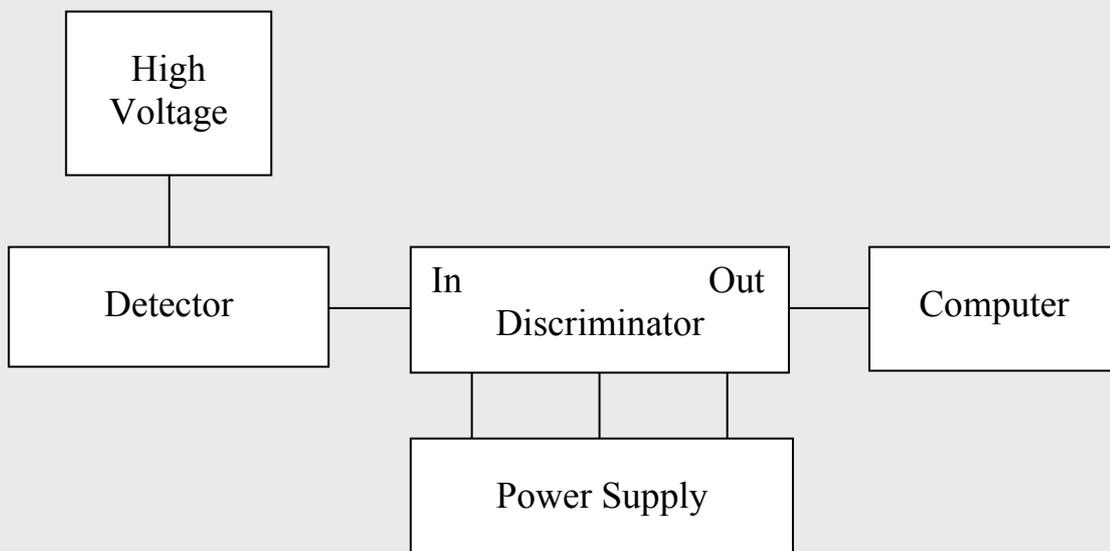
Use of the multichannel scaling system (MCS)

Data is fed from the detector into a discriminator and then to the computer. Now the computer will measure the total number of counts in a given time interval (e.g., 1 s) and store it in one channel of the 256 channel spectrum. The number of counts in the next interval (e.g., 1 s) is stored in the next channel of the spectrum and so on. The user specifies the time interval. This can be used to measure directly the decay curve of a radioactive isotope. You can then read the data from the MCS system into a spreadsheet and least squares fitting program for the detailed analysis.

Some useful commands in addition to those above are:

MODE	allows MCS to be selected. You should select PASSES = 1 and DWELL = a suitable interval for the decay curve you are measuring.
FILE/ASCII FILE SAVE	stores data on disc in a format that can be transferred to a spreadsheet for analysis

Schematic layout for MCS experiments



References

1. Various references supplied in a booklet of supplementary information

2. Table of Isotopes (available in the laboratory)

Browne E and Firestone R B, *Table of Radioactive Isotopes*, Wiley-Interscience

(available in the laboratory)

3. Atomic Data and Nuclear Data Sheets (in the Physics Library)

4. Nuclear Spectroscopy Tables, Wapstra *et al* (Ref. AC33 in the Physics Library)

IMPORTANT NOTICE

The ^{137}Cs source from the laboratory source cupboard has the following characteristics:

Source number	NPRL 172
Gamma-ray energy	662 keV
Source strength	370 kBq ($\pm 3\%$) in February 1978

The ^{22}Na source from the laboratory source cupboard has the following characteristics:

Source number	NPRL 329
Gamma-ray energy	1275 keV and 511keV
Source strength	370 kBq ($\pm 3\%$) in October 1986

Risk Assessment for Block A

In addition to the general safety rules listed on Page 17 of this Laboratory Handbook, the following hazards should be noted:

Electrical Hazards

Various items of instrumentation are powered by mains electricity. No such item should be disassembled or have its plug or fuse(s) replaced. If an electrical item appears to be faulty, consult the laboratory technician.

High voltage power supplies are capable of producing lethal voltages. Pay particular attention to the cables that connect the high voltage to other instrumentation – are they in a good condition, with no obvious damage?

Radiation Hazard

All the Block A experiments, including the group work, require radioactive sources. These must be handled in accordance with the local radiation rules that are displayed on the walls of the laboratory (T7).

Block B

X-Ray Diffraction

X-Ray Diffraction

Aims

- To gain an appreciation of the physics of the diffraction of waves from crystalline materials, using a computer-based model of diffraction applicable to systems based on the simple cubic (SC), body-centred cubic (BCC) and face-centred cubic (FCC) crystal lattices
- To measure the intensity of x-rays diffracted from two unknown materials and hence determine their crystal structures and lattice constants

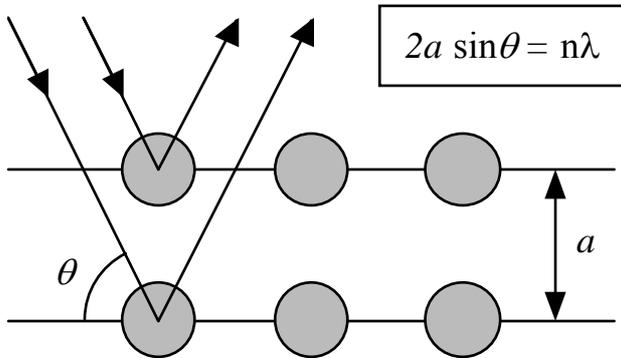
Structure of Experiment

Sessions

- | | |
|---|-------|
| • Introductory tutorial on x-ray diffraction | 1 |
| • Group work on computer-based model of diffraction from BCC and FCC crystals and from compounds based on the SC and FCC lattices | 1–3 |
| • Individual experimental work, involving:
Measurements of intensities of diffracted x-rays from two samples
Comparison of the data with the models to determine the crystal lattices
Analysis of the data to determine the lattice constants
Consideration of diffracted intensities | 4–10 |
| • Write up experimental report | 11–12 |

Principles of X-Ray Diffraction

Theory of Diffraction

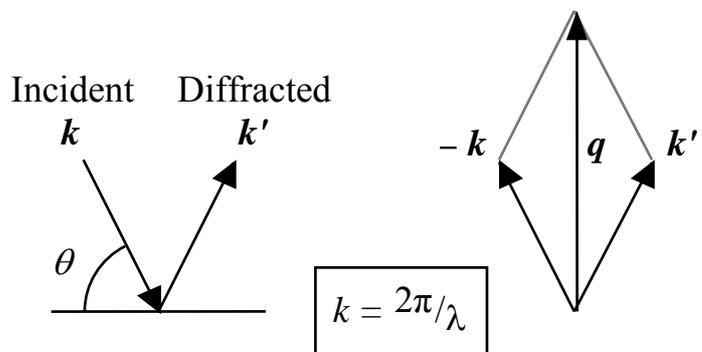


The Bragg Law of diffraction relates the wavelength of the incoming wave to the geometry of the planes of atoms that are scattering the waves. When the Bragg condition is met, constructive interference occurs between the waves scattered by every atom.

We define the quantity

$$\mathbf{q} \equiv \mathbf{k}' - \mathbf{k}$$

which is a vector perpendicular to the plane of atoms diffracting the wave.



Reciprocal Space

In a crystal there are an infinite number of atomic planes that can diffract waves, and so there are an infinite number of \mathbf{q} vectors associated with any crystal.

It can be shown (PHYS363) that the set of \mathbf{q} vectors that represent diffraction from a crystal lattice are the position vectors of points that also lie on a lattice. This lattice exists not in real space but in k space, or q space. It is also called reciprocal space because the magnitudes of the \mathbf{q} vectors are proportional to the reciprocals of distances in real space.

The magnitude of \mathbf{q} is given by

$$q = 2k \sin \theta$$

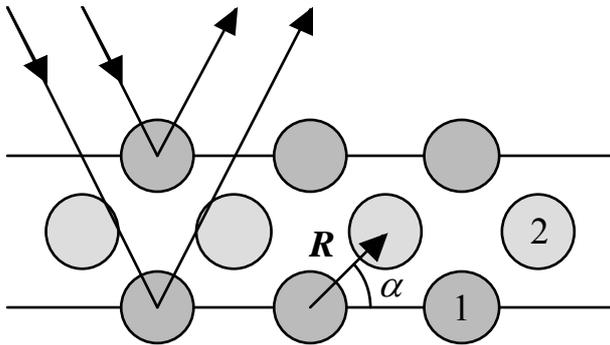
and so the Bragg Law for $n = 1$ can be rewritten

$$2a \sin \theta = \lambda = \frac{2\pi}{k}$$

$$\Rightarrow 2ak \sin \theta = 2\pi$$

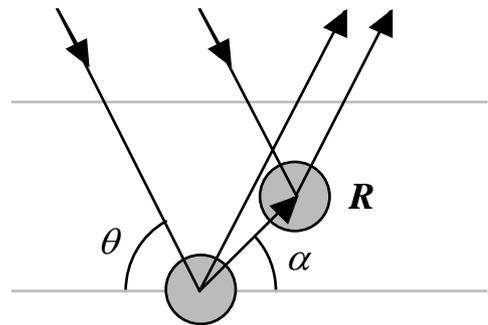
$$\Rightarrow \boxed{q = \frac{2\pi}{a}}$$

What happens when another atom is placed inside the unit cell?



Consider the addition of extra atoms (labelled 2) placed at a position vector \mathbf{R} with respect to each of the atoms in the original crystal structure (labelled 1), as shown on the left. We have created a new crystal.

When the Bragg Law is satisfied for the atoms in the original crystal, the waves scattered from each atom 1 are in phase. Are the waves scattered from atom 2 in phase with those scattered from atom 1? Not necessarily.



We can find the phase difference of waves scattered from atom 1 and atom 2 using the construction shown above right.

The path difference Δr between waves scattered from atoms 1 and 2 is

$$\begin{aligned}\Delta r &= R[\cos(\pi - \theta - \alpha) + \cos(\theta - \alpha)] \\ &= R[-\cos(\theta + \alpha) + \cos(\theta - \alpha)] \\ &= R[-\cos\theta \cos\alpha + \sin\theta \sin\alpha + \cos\theta \cos\alpha + \sin\theta \sin\alpha] \\ &= 2R\sin\theta \sin\alpha\end{aligned}$$

The phase difference $\Delta\phi$ between waves scattered from atoms 1 and 2 is given by

$$\begin{aligned}\Delta\phi &= \frac{\Delta r}{\lambda} 2\pi = k\Delta r \\ &= 2kR\sin\theta \sin\alpha\end{aligned}$$

Look Back at your PHYS258 notes or the textbook "Optics" by Hecht

$$\Delta\phi = \mathbf{q} \cdot \mathbf{R}$$

To calculate the amplitude of the waves scattered from this crystal, we have to add two waves whose phases differ by $\Delta\phi$.

We can write the scattered waves as

$$A_1 \exp(i(\mathbf{k} \cdot \mathbf{r} - \omega t))$$

and

$$A_2 \exp(i(\mathbf{k} \cdot \mathbf{r} - \omega t + \Delta\phi))$$

Look Back at your PHYS258 notes or the textbook “Optics” by Hecht

where the amplitudes A_1 and A_2 depend on how strongly the waves are scattered by atoms 1 and 2, respectively.

Then the sum of the two waves is

$$\begin{aligned} & A_1 \exp(i(\mathbf{k} \cdot \mathbf{r} - \omega t)) + A_2 \exp(i(\mathbf{k} \cdot \mathbf{r} - \omega t + \Delta\phi)) \\ &= [A_1 + A_2 \exp(i\Delta\phi)] \exp(i(\mathbf{k} \cdot \mathbf{r} - \omega t)) \\ &= [A_1 + A_2 \exp(i\mathbf{q} \cdot \mathbf{R})] \exp(i(\mathbf{k} \cdot \mathbf{r} - \omega t)) \end{aligned}$$

and thus the amplitude of the resultant wave is given by

$$A_{sum} = A_1 + A_2 \exp(i\mathbf{q} \cdot \mathbf{R})$$

More generally, if we add n atoms at position \mathbf{R}_i ($i = 1, \dots, n$) then we have

$$A_{sum} = A_1 + A_2 \exp(i\mathbf{q} \cdot \mathbf{R}_2) + A_3 \exp(i\mathbf{q} \cdot \mathbf{R}_3) + A_4 \exp(i\mathbf{q} \cdot \mathbf{R}_4) + \dots$$

or

$$A_{sum} = \sum_i^n A_i \exp(i\mathbf{q} \cdot \mathbf{R}_i)$$

In PHYS363 this is called the Structure Factor

The intensities of x-rays diffracted by a crystal are given by the square of the total amplitude, and depend on:

- q $\left\{ \begin{array}{l} \text{magnitude depends on the x-ray wavelength} \\ \text{direction specifies the plane of atoms} \end{array} \right.$
- \mathbf{R}_i positions of atoms in the unit cell of the crystal
- A_i strength of scattering from individual atoms

Modelling Crystal Diffraction

Using a spreadsheet, the intensities of diffracted x-rays can be calculated from

$$I = |A_{sum}|^2 \quad \text{where} \quad A_{sum} = \sum_i^n A_i \exp(iq \cdot R_i)$$

These values will indicate how x-rays are diffracted in particular directions (defined by the q vectors) from crystals whose structures are defined through the atomic positions (R_i) and atom types (A_i).

We can describe the diffraction from a plane of atoms by specifying the xyz components of the q vector. As the Bragg condition corresponds to $q = 2\pi/a$, it is convenient to express the components of q in units of $2\pi/a$ thus

$$q = \frac{2\pi}{a} (hkl)$$

	A	B	C	D	E	F	G	H
1								
2					Atom 1			
3					x	y	z	
4		h	k	l	0	0	0	
5								
6		0	0	0				
7		0	0	1				
8		0	0	2				
9		0	0	3				
10		0	0	4				
11		0	1	0				
12		0	1	1				
13		0	1	2				
14		0	1	3				
15		0	1	4				
16		0	2	0				

Miller Indices

As the q vector is perpendicular to the plane of atoms diffracting the wave, then the (hkl) indices also specify this plane. The (hkl) are called Miller indices, and will be covered in more detail in PHYS363.

Start by entering in the spreadsheet formulae that will generate all (hkl) indices from (000) to (444) .

X-Ray Diffraction Group Work

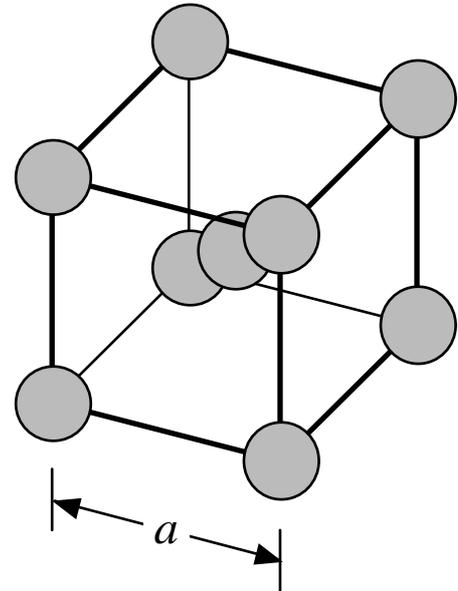
For simplicity, we will be considering only those crystal structures that are based on the simple cubic (SC) lattice. By placing atoms of the same species or of different species in the unit cell, we can construct many different crystal structures.

Body-Centered Cubic (BCC)

- Enter the xyz coordinates (the \mathbf{R}_i , in units of the lattice constant a) of the two atoms in the unit cell of the BCC crystal structure (shown on the right).

Assume that atom 1 is at the origin.

Where is atom 2?



	A	B	C	D	E	F	G	H	I	J	K	L	M	
1														
2					Atom 1			Atom 2			Atom 3			
3						x	y	z	x	y	z	x	y	z
4		h	k	l	0	0	0							
5														
6		0	0	0										
7		0	0	1										
8		0	0	2										
9		0	0	3										
10		0	0	4										
11		1	0											

- For each atom calculate the quantity $\exp(i\mathbf{q} \cdot \mathbf{R}_i)$.
- Taking $A_i \equiv 1$, calculate A_{sum} and hence I .

Questions

For which values of (hkl) does the diffracted x-ray intensity vanish?

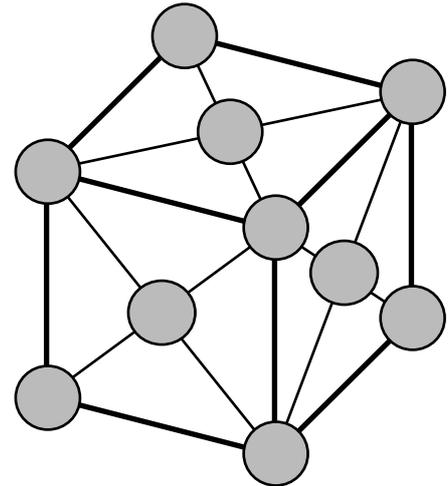
What is the rule that specifies these values of (hkl) ?

Face-Centred Cubic (FCC)

- Enter the xyz coordinates of the four atoms in the unit cell of the FCC crystal structure (shown on the right) and recalculate the diffracted intensities.

Again assume that atom 1 is at the origin.

Where are atoms 2, 3 and 4?



	A	B	C	D	E	F	G	H	I	J	K	L	M	
1														
2						Atom 1	Atom 2	Atom 3						
3						x	y	z	x	y	z	x	y	z
4		h	k	l		0	0	0						
5														
6		0	0	0										
7		0	0	1										
8		0	0	2										
9		0	0	3										
10		0	0	4										
11		1	0	0										

- For each atom calculate the quantity $\exp(i\mathbf{q} \cdot \mathbf{R}_i)$.
- Taking $A_i \equiv 1$, calculate A_{sum} and hence I .

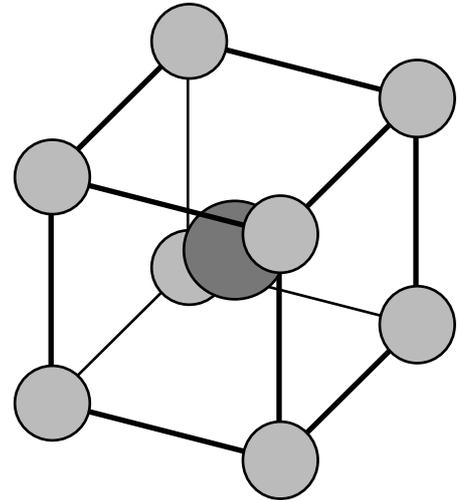
Question

What is the (hkl) rule for FCC?

To model the diffraction from crystal structures comprising atoms of different species, we need to set $A_i \neq A_j$. For x-ray diffraction, an acceptable approximation is to take the *strength of scattering of an atom to be proportional to the number of electrons* in the atom, as it is the electron charge that is interacting with the electromagnetic wave.

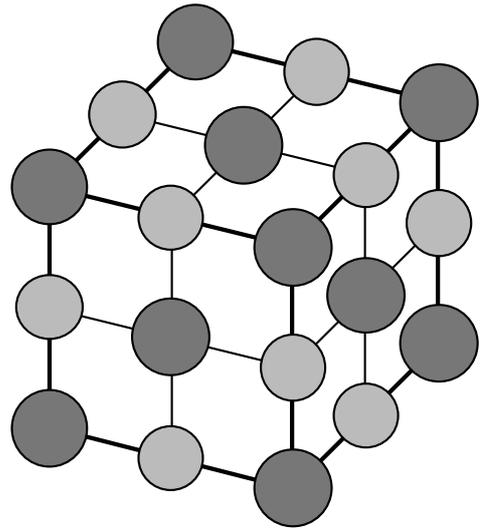
Caesium Chloride (CsCl)

- Use the xyz co-ordinates of the atoms in the BCC crystal structure, but set the scattering strengths of each of the two atoms equal to the number of electrons in the Cs and Cl atom. Note that because the atoms are different, this is not a BCC crystal.
- For each θ , how many atomic planes diffract x-rays at that angle? Hint: How many times does a given θ occur in the θ column? In the spreadsheet, this is labeled the θ multiplicity.
- How many planes with the same θ have we NOT counted by ignoring negative indices? In the spreadsheet, this is labeled the index multiplicity.
- The x-ray intensity at a given diffraction angle θ is proportional to the total number of planes that have that value of θ . Thus, we need to use the product of the θ multiplicity and the index multiplicity.
- For a realistic simulation of experimental intensities we must take into account that scattering of x-rays from electrons is angle dependent. Scattering intensities are given by a ‘polarisation factor’, which is approximately proportional to $1/\theta^2$ (do not worry about the derivation of this – it comes from scattering theory).
- Taking all these factors into account, plot the x-ray intensities as a function of θ to simulate an experimental $I(\theta)$ data set.



Sodium Chloride (NaCl)

- Use the xyz coordinates of the atoms in the FCC crystal structure, and add extra atoms according to the figure on the right. How many atoms are there in the unit cell?
- Calculate and plot $I(\theta)$.
- KCl has the same crystal structure as NaCl. Calculate and plot $I(\theta)$ for KCl.



Questions

What do you notice about the (hkl) rule for KCl?

Why is the rule different from that of NaCl?

References

1. Hecht E, *Optics*, Addison Wesley
2. Kittel C, *Introduction to Solid State Physics*, Wiley
3. Myers H P, *Introductory Solid State Physics*, Taylor and Francis
4. Ibach H and Lüth H, *Solid State Physics*, Springer

Glossary

<i>Lattice</i>	Periodic array of points that has translational symmetry
<i>Lattice Vectors</i>	Vectors that translate the lattice into a copy of itself
<i>Basis</i>	Collection of atoms that is placed at every lattice point to construct a crystal
<i>Crystal</i>	Structure resulting from placing a basis at every lattice point
<i>Unit Cell</i>	Polyhedron whose edges are lattice vectors

X-Ray Diffraction Experiment

Aims

To measure the intensity of x-rays diffracted from two unknown materials and hence determine their crystal structures and lattice constants. All that is known about the materials is that they are of the form M^+X^- , where M is a metal or radical and X is a halogen.

Theory

X-rays incident upon a set of crystal planes of spacing d will diffract radiation of wavelength λ at an angle θ such that,

$$\sin\theta = \frac{\lambda}{2d}$$

Equation 1: Bragg Law

Since the crystalline sample is powdered, all crystal orientations are equally likely therefore, the diffracted beam will lie along the surface of a cone of half angle 2θ whose axis is the incident beam.

If the crystal structure is cubic then d can be expressed in terms of the cube side length and the Miller indices of the plane under construction. Thus by Pythagoras' theorem.

$$d^2 = \frac{a^2}{h^2 + k^2 + l^2}$$

Equation 2

The cube side length a is called the lattice constant.

$$\sin^2\theta = \frac{\lambda^2}{4a^2}(h^2 + k^2 + l^2)$$

Equation 3

$$\sin^2\theta = \frac{\lambda^2}{4a^2}N$$

Equation 4

X-Ray Diffraction Experiment

where $N = h^2 + k^2 + l^2$. The N values corresponding to each angle θ can be calculated (see example below) and hence the lattice constant a can be determined.

Because N is the sum of three squared integers, it cannot assume all values. For example, it is not possible to express 7 or 15 in this manner. These ‘gaps’ can be used to help index the lines in the plot of x-ray intensity I vs. angle θ . If the crystal structure is simple cubic (SC) the hkl values will be a mixture of odd and even numbers. If the structure is face centred cubic (FCC) or body centred cubic (BCC) then some of the hkl values will not produce any diffracted intensity. This was covered in the computer modelling of x-ray diffraction.

Equation (4) can only be used to find the lattice constant a if the correct value of N is assigned to each value of θ . For fixed values of a and λ equation (4) shows that the ratio of $\sin^2(\theta_n)$ for the n 'th angle to $\sin^2(\theta_1)$, where θ_1 is the smallest angle measured, is equal to the ratio of two integers. Thus, once you have obtained your data, you can use a spreadsheet to construct a table of these ratios and by inspection assign the correct N value to each θ . Practice this technique on a spreadsheet using the data in Table 1, where θ is given in degrees.

θ	$\sin \theta$	$\sin^2 \theta$	N	hkl
9.544	0.165805	0.027491	2	(110)
11.72	0.203129	0.041261	3	(111)
13.561	0.23448	0.054981	4	(200)
15.201	0.262206	0.068752	5	(210)
16.701	0.287377	0.082586	6	(211)
19.374	0.331733	0.110047	8	(220)
20.597	0.351793	0.123758	9	(221)(300)
21.771	0.370898	0.137565	10	(310)
37.559	0.609578	0.371585	27	(511)(333)
39.164	0.631542	0.398846	29	(520)(432)
39.966	0.642333	0.412592	30	(521)

Table 1

X-Ray Diffraction Experiment

This method, however does not produce the most precise value for the lattice constant a . To see why, consider the Bragg equation. Differentiating gives:

$$\frac{d\lambda}{d\theta} = 2d \cos \theta$$

Equation 5

$$\frac{d\lambda}{\lambda} = \frac{2d \cos \theta}{2d \sin \theta} d\theta$$

Equation 6

$$d\theta = (\tan \theta) \frac{d\lambda}{\lambda}$$

Equation 7

It then follows that a small change $\delta\theta$ due to a small change $\delta\lambda$ or δd depends upon $\tan\theta$ and so tends to infinity as θ tends to 90° . The K_α X-ray line used in this experiment is a doublet and is thus resolved when θ tends to 90° .

Experimental Procedure

- Prepare a sample according to the instructions on the sheet posted above the sample preparation area.
- With the help of the technician or a demonstrator mount the sample in the x-ray set and acquire an $I(\theta)$ data set. Instructions on the use of the data acquisition software are posted next to the PC.
- Make sure you know whether the x-ray tube is being used with a cobalt or copper anode.
- From the $I(\theta)$ data obtain an approximate value for the lattice constant.
- Investigate how the Nelson-Riley function * can be used to improve the final result for the lattice constant. Explain why the procedure works (see reference 1, section 5.9).
- Identify the material using the table of lattice constants provided.
- Repeat the above procedure for the second sample.
- Compare your results to simulated diffraction data produced by the crystallography software, and comment on the relative intensities of diffraction peaks.*

References

1. Brown P J and Forsyth J B, *The Crystal Structure of Solids*, chapter 5.

(Photocopies are available — please return them when you have finished)

2. Kittel C, *Introduction to Solid State Physics* (chapters 1 and 2) Wiley, New York, 1986.

Risk Assessment for Block B

In addition to the general safety rules listed on Page 17 of this Laboratory Handbook, the following hazards should be noted:

Electrical and Radiation Hazard

The x-ray generator must be switched on and off only by authorised staff or demonstrators. Students can load their samples into the x-ray diffractometer and open/close the x-ray shutters to expose their samples after receiving appropriate instructions from staff or demonstrators. The x-ray set (generator and diffractometer) must be operated in accordance with the local radiation rules that are displayed beside the x-ray set.

Chemical Hazard

The salts that are used to make samples for x-ray analysis may be mildly irritating and should be handled with the gloves and safety glasses provided. The salts should not be ingested.

Block C

Quantum and Waves

Quantum Group Work

Paramagnetic Resonance

Theory

A free radical has an odd number of electrons. The odd electron can be aligned either parallel or anti-parallel to an applied magnetic field, resulting in two different energy states. Applying an electromagnetic field can then induce transitions between these states.

The two states have an energy separation given by $\Delta E = g_E \mu_B B$, where the lower energy state is usually more populated than the higher.

Transitions between the states may be induced by photons of the electromagnetic field of frequency $\nu = \frac{\Delta E}{h}$ whose magnetic component (B) is at right angles to the static magnetic field.

In this experiment, a marginal oscillator is used to supply the radio frequency (r.f.) oscillations to the r.f. coil, which in turn produces the electromagnetic field. The sample is placed in the coil and at electron spin resonance, when the electrons are absorbing energy from the coil, there is a large change in the output from the oscillator that can be detected using an oscilloscope.

For most free radicals the free electron prefers to “couple” with other free radicals, however there are a few stable free radicals that do not display this tendency. The sample provided in his experiment diphenyl-picrly-hydrazyl (DPPH) is an example, and has a measured g_E factor of 2.0036 close to that of the free electron, 2.0023.

Apparatus

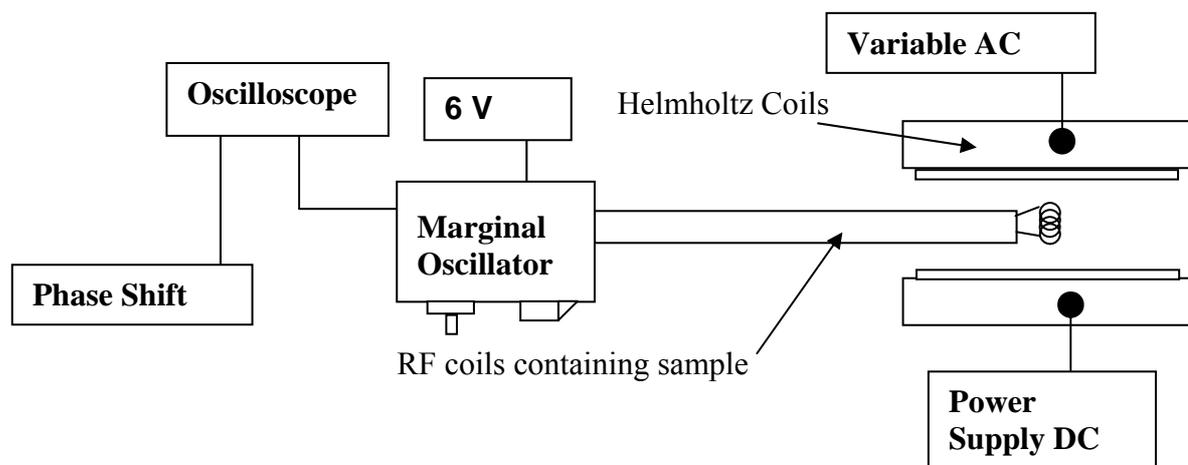


Figure 4: Diagram of Apparatus

Oscilloscope

Connected to the output of the marginal oscillator, it is used to observe the change in oscillator output when transitions occur. The marginal oscillator should be connected to channel 2.

Marginal oscillator

This produces oscillations that are fed to the r.f. coils. The marginal oscillator has both a switch and a knob. The switch enables/disables a noise filter and the knob operates a feedback capacitor which varies the sensitivity of the oscillator. The magnitude of output signal is very sensitive to this control.

Helmholtz coils

These coils provide the magnetic field which splits the energy levels.

R.F. coil

The small coil in which the electromagnetic field is set up (by the marginal oscillator) to produce transitions. The R.F. coil and marginal oscillator act together at a fixed frequency that is independent of any adjustment to the marginal oscillator.

Procedure

To ensure that the energy separation of the two states matches the energy of the photons of the electromagnetic field, the wobble field technique is used (see later). Using a moderate wobble field (equivalent 50V on the supply dial), and a DC field corresponding to 0.35A, and the oscilloscope on a linear time base, adjust the marginal oscillator to give a flat trace. You should find that at some setting of the marginal oscillator the output will saturate, giving a square waveform on the oscilloscope.

Graph A

Sketch the energy separation as a function of time over 2-3 periods of oscillation. For a photon energy equal to the average energy separation, **sketch** the output from the marginal oscillator (on the same timescale) indicating when resonance takes place.

Graphs B and C

Raise and lower the DC component of the coil current by 0.2A, and for each **sketch** the energy separation and output from marginal oscillator.

Graph D

With the DC component of the coil current set at 0.1A and the AC supply at 20V **sketch** the energy separation and output from marginal oscillator.

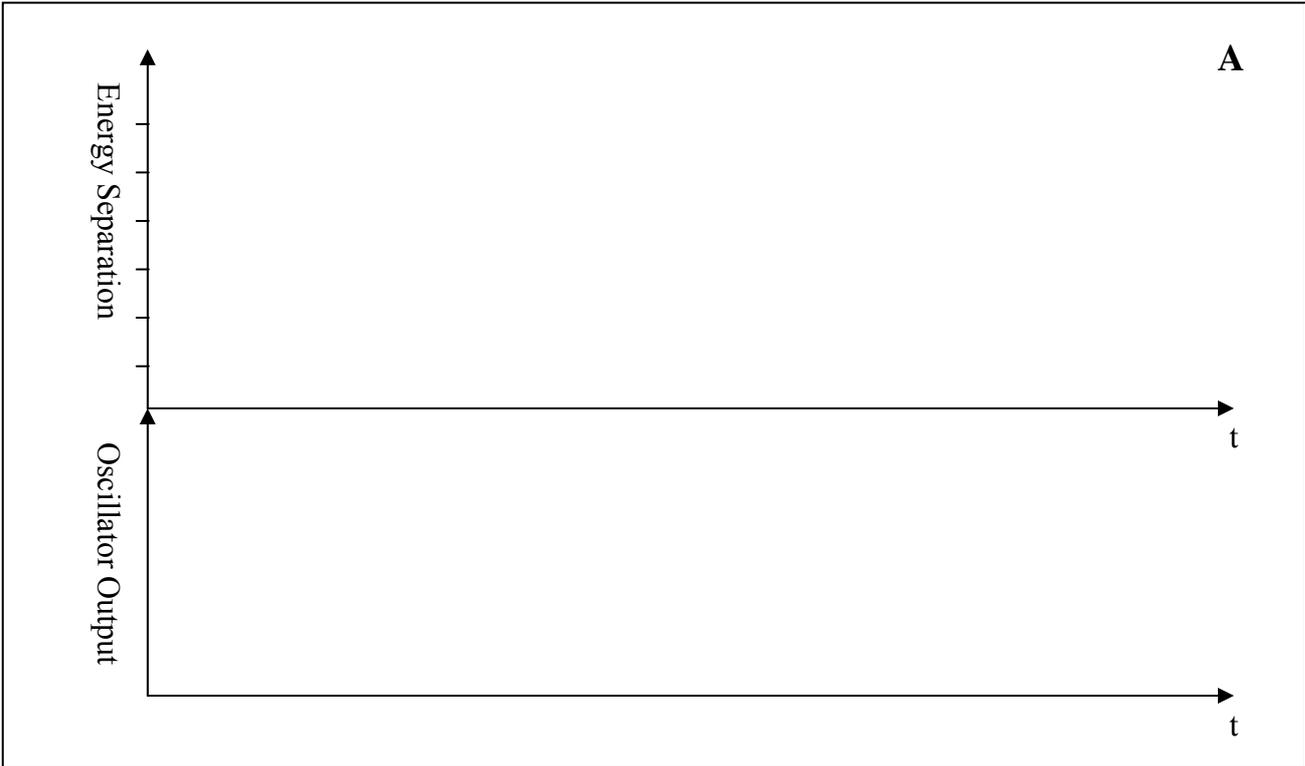
Using a sweep generator connected to channel 1 of the oscilloscope it is possible to get a looped trace with the oscilloscope in x-y mode. The phase of the sweep generator can be adjusted to overlap the peaks, enabling a convenient determination of when the average wobble field meets the condition for resonance. When this occurs, the oscilloscope trace becomes symmetrical.

Find the natural frequency of the r.f coil in use (ask a demonstrator for assistance).

Calculate the energy separation between the two energy states and hence the static component of the magnetic field. Confirm this using a Hall probe to measure the magnetic field intensity.

Include errors in your measurements and calculations.

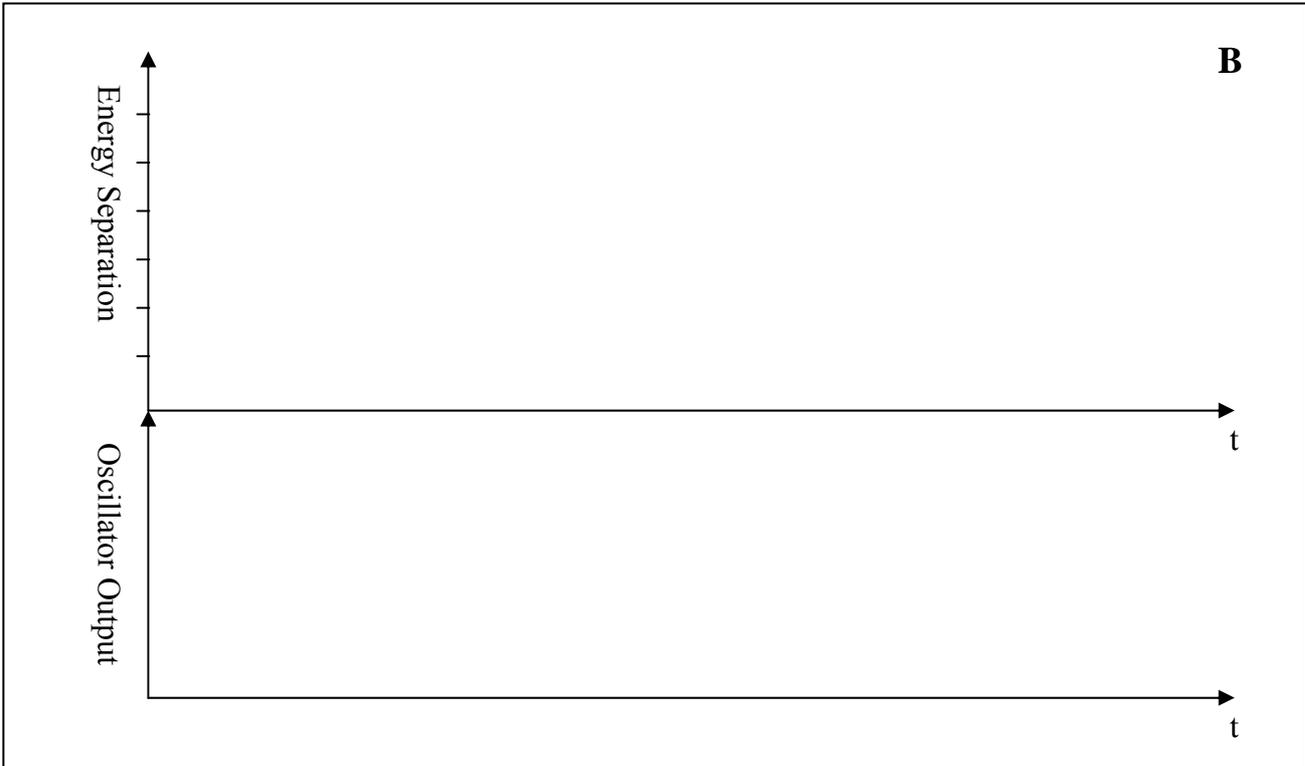
Energy Separation		eV
Calculated B field		mT
Measured B field		mT



Comment:

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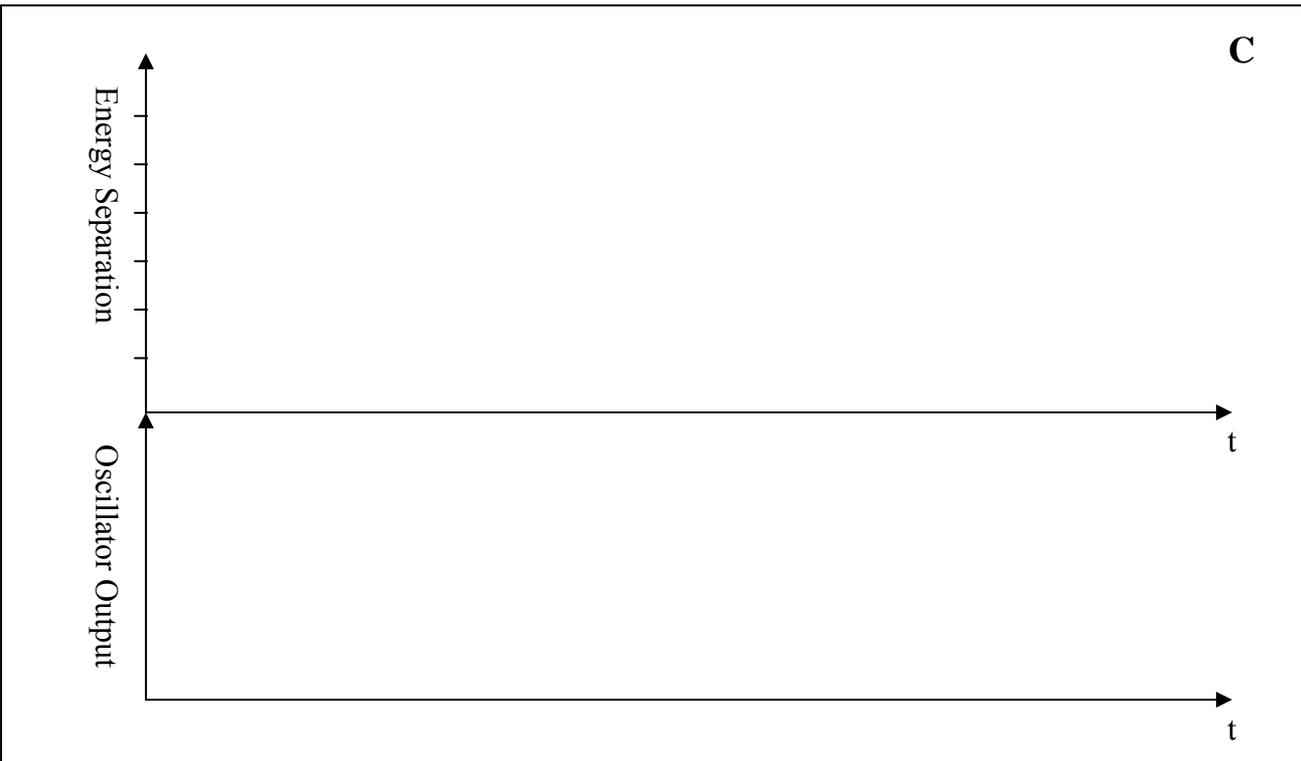
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Comment:

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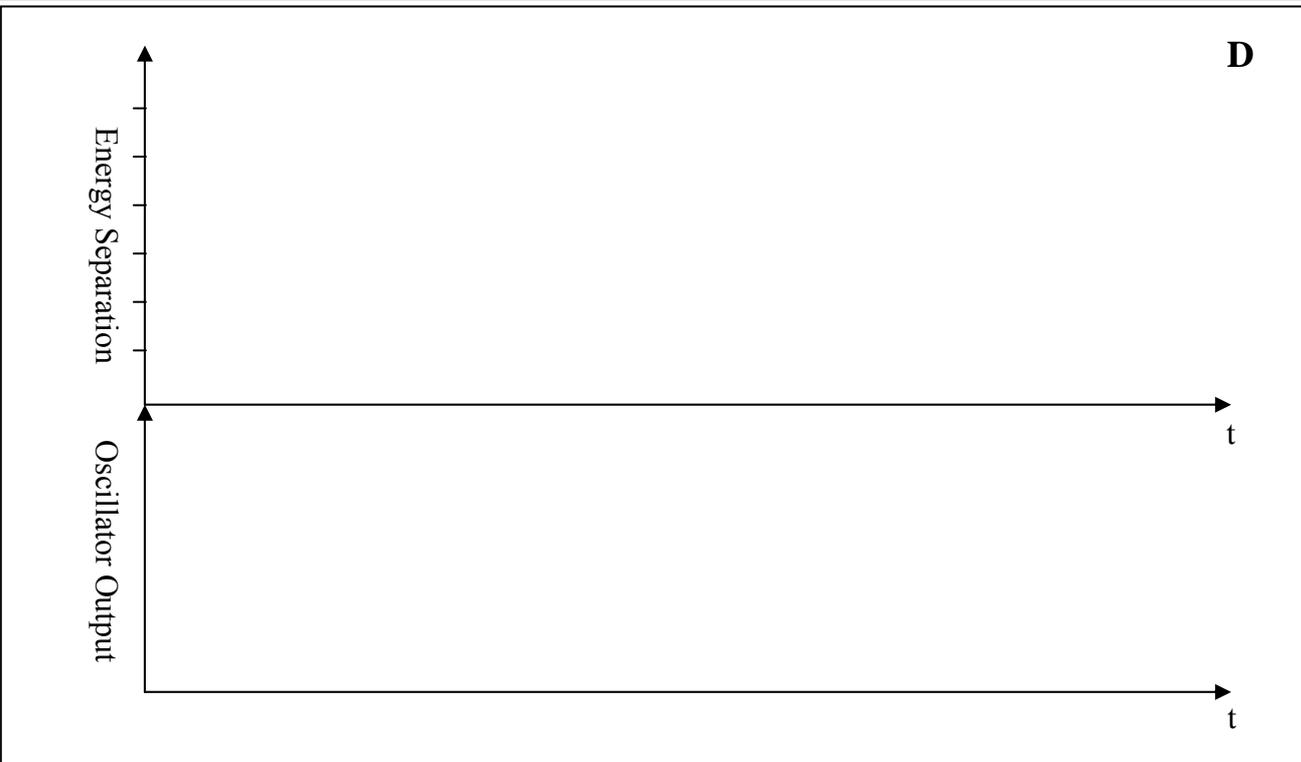
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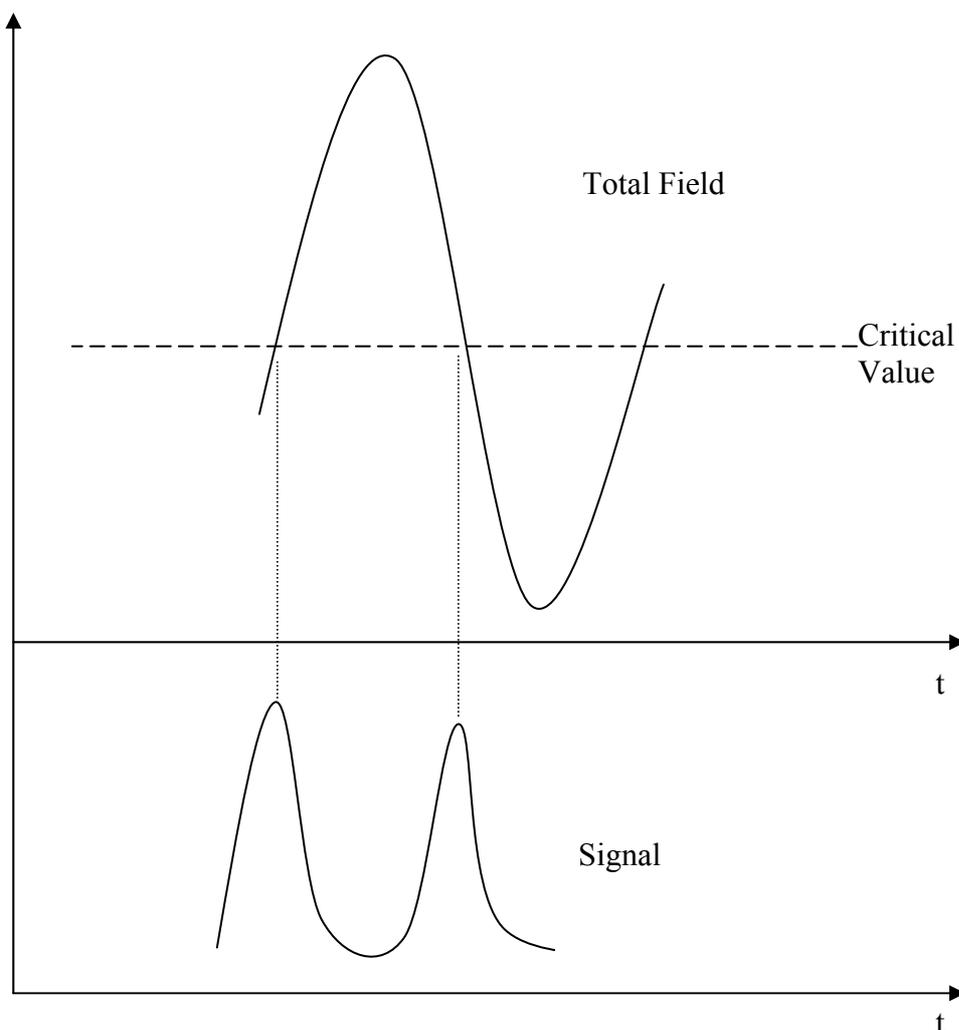
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The Wobble Field Technique

This technique involves perturbing a magnetic field, B by a smaller 50 Hz field, $B_w \sin(\omega t)$.

This technique is useful as there is a field dependent physics process, e.g.: the absorption of a photon, which occurs when the total field ($B + B_w$) reaches a particular critical value. A suitable detector then detects this process.

The output from the detector generates a signal that is fed into the Y input of oscilloscope, the timebase, X input, is fed with a 50 Hz sinusoidal waveform, the phase of which can be altered. The resulting oscilloscope trace then forms an elliptical trace (often with much noise) on which can be seen a pair of peaks, which are caused by the detector responding to the effect that it is measuring, on the *two* occasions per cycle when the total field sweeps through the critical value.



The main problem with this technique is to adjust the primary field such that it equals the critical field. This is achieved in two stages.

1. The phase of the input signal to the timebase (x) is adjusted such that the two peaks on the oscilloscope overlap.
2. The magnitude of the primary field (y) is changed slowly, to cause a translation of the peak to the centre of the scope. Note that the amplitude of the secondary field (the wobble field) is kept constant.

When this has been achieved, the magnitude of the primary field is the same as the required critical field.

To aid in the understanding of this process there is a computer simulation on the Physics department computers. To access this, choose the option **WOBBLE FIELD TUTORIAL** in the **TEACHING** folder on the desktop or from the start menu.

Absorption Spectrum of Iodine

Aims

- To use a calibrated spectrometer to study the absorption spectrum of iodine and identify the transitions observed.
- To use the data to find the values of
 - Classical frequency for infinitesimally small vibrations, ω_e
 - Anharmonicity constant, X_e
 - Dissociation energy, D_e

Theory

A diatomic molecule (eg. O₂, I₂) has atoms that are bound together in a potential well. This gives rise to vibrational energy levels that have quantum numbers v ($v = 1, 2, \dots$). Using Straughan and Walker notation vibrational energy can be written in wave number units (m^{-1}):

$$G(v) = \left(v + \frac{1}{2}\right)\omega_e - \left(v + \frac{1}{2}\right)^2 X_e \omega_e + \dots \quad [\text{m}^{-1}]$$

Equation 1: Straughan & Walker Energy notation

where

ω_e = classical frequency for infinitesimally small vibrations

X_e = anharmonicity constant.

Questions

How would Equation 1 be simplified if the form of the potential were parabolic?

What do you expect to observe when looking at (i) an emission spectrum, (ii) an absorption spectrum?

For the iodine molecule, the absorption spectrum in the visible region is unusually simple and consists of a series of bands that arise from transitions between vibrational energy levels (v'') in the electronic ground state and vibrational energy levels (v') in an electronic excited state (see Figure 1). More information about the absorption spectrum of iodine can be found in reference 1.

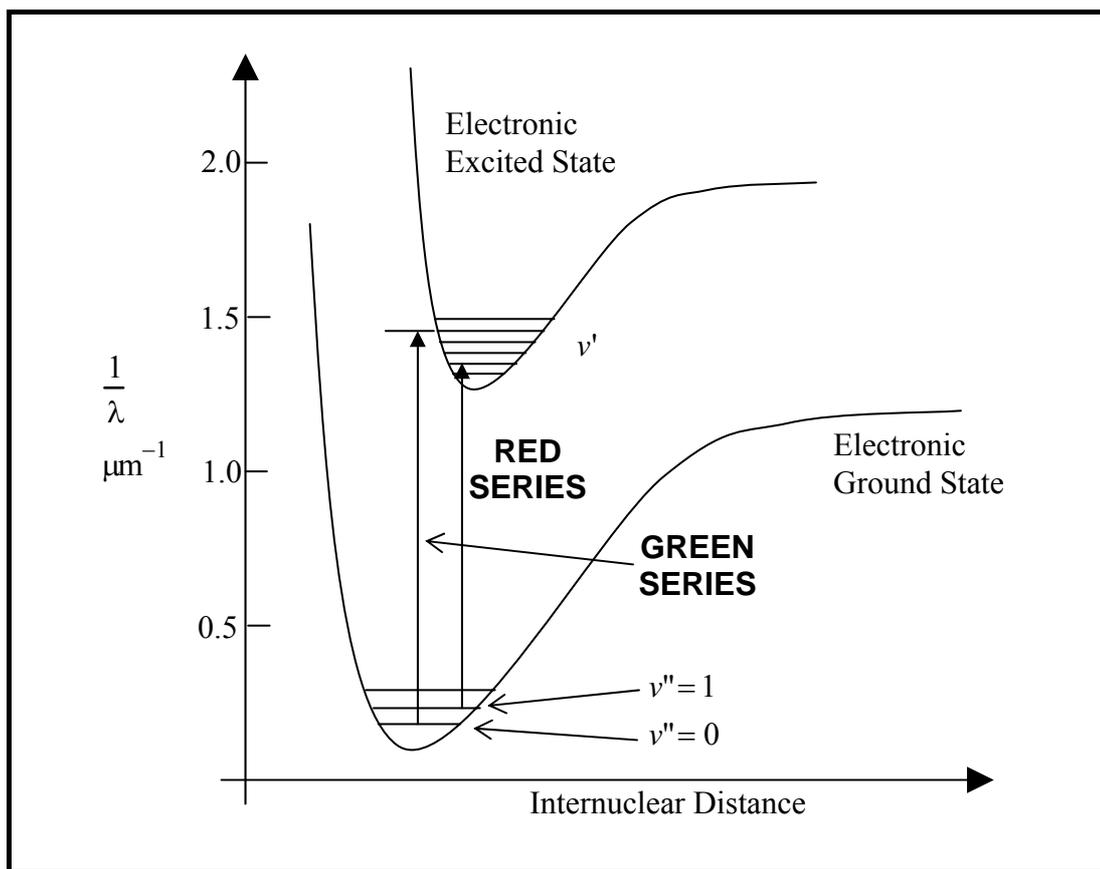


Figure 1: Origin of transitions in the absorption spectrum of Iodine

Calibration

Using the calibration lamp, lines of a known wavelength can be observed. The wavelengths of the lines for mercury (along with other elements) can be looked up.

To Calibrate the Spectrometer

Observe a lamp line. If the wavelength reading is wrong adjust the wavelength drum by unscrewing the knurled locking ring, allowing the drum to rotate freely. This can then be set to the right value and secured by tightening the locking ring.

It is suggested that several different wavelengths are used to calibrate the spectrometer (other lamps are provided in the laboratory) *. The wavelengths of these other lines can be found by consulting appropriate literature.

Data Acquisition

After the equipment has been calibrated, the experiment is carried out using a sealed tube of solid iodine with a white lamp shining through. Heating of the tube will improve the visibility of the bands *.

Record the wavelength (λ) of each of the bands from the red to green end of the spectrum. Assign a 'band number' to each band sequentially from red to green.

Plot $1/\lambda$ against band number. This is a good indicator of the quality of your data.

Identify the transitions and plot a graph of the energy separation of each level in the potential well of the electronic excited state for a wide range of ν' . To identify the transitions the following information is required:

- The line in the green spectrum with a wavelength of 548nm corresponds to a transition to a vibrational level in the electronic state with $\nu' = 25$.
- The energy separation, measured in terms of wave number between $\nu'' = 0$ and $\nu'' = 1$ (Fig. 1) is about 21000 m^{-1} .
- You can only expect to identify transitions for which $\lambda < 600\text{nm}$ with the spectrometer provided.

Data Analysis

1. Determine the energy separation between $\nu'' = 0$ and $\nu'' = 1$
2. Fit each series of bands found to

$$\frac{1}{\lambda} = Av'^2 + Bv' + C$$

Equation 2: Fitting results to a parabola

3. For the electronic excited state determine ω_e and $\omega_e X_e$ in wave number units (m^{-1}) and the dissociation energy D_e' , where

$$D_e' = \frac{\omega_e^2}{4\omega_e X_e}$$

Equation 3: Dissociation energy

Absorption Spectrum of Iodine

4. Calculate the dissociation energy relative to the ground state (Fig. 2).
5. Explain qualitatively the intensity variations across the spectrum in terms of the Frank-Condon principle. *

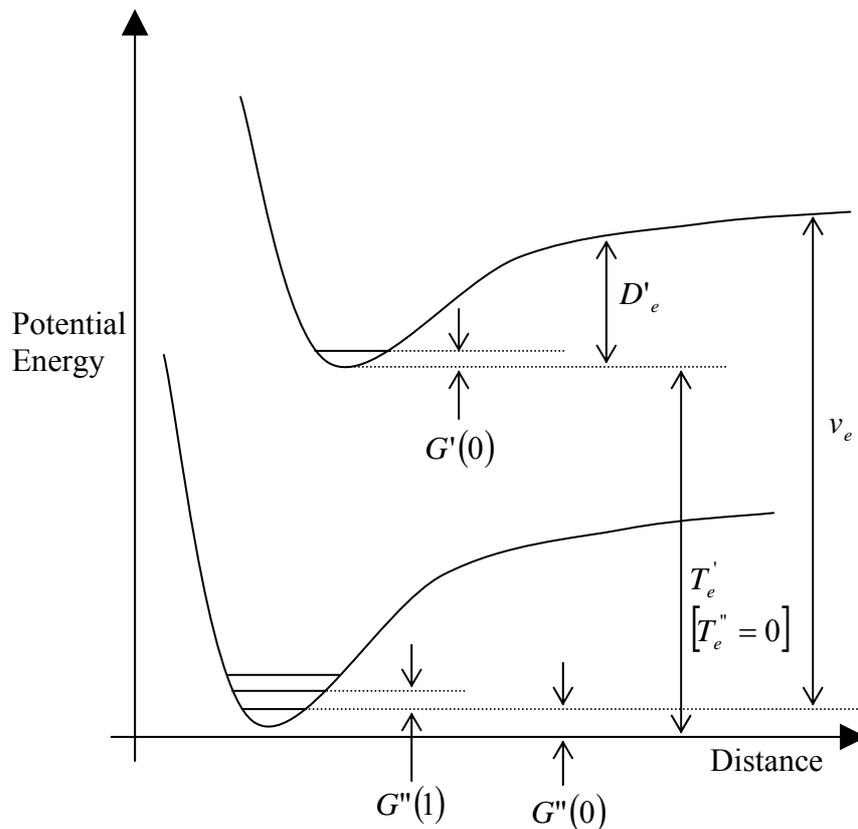


Figure 2: Some energies associated with the absorption spectrum of Iodine

References and Background Reading

1. George S and Krishnamurty N, *Absorption Spectrum of Iodine Vapor – An Experiment*, *Am. J. Phys.*, **57** (1989) 850

Straughan and Walker, *Spectroscopy*, Volume 3

Herzberg, *Spectra of Diatomic Molecules*

Brittain, George and Wells, *Introduction to Molecular Spectroscopy*

Banwell, *Molecular Spectroscopy* (note that the data in Figure 6.5 here does not refer to the transitions studied in this experiment)

Microwave Waveguide

Aims

- To gain familiarity with microwaves and waveguide components
- To measure wavelengths of microwaves in a waveguide and in air and verify the theoretical relationship between them
- To observe destructive interference between waves, to simulate the effect of anti-reflective coatings

Theory

From a communication point of view, electromagnetic waves can be divided into 2 main types:

(a) *Free waves*

Used to broadcast waves in all directions (e.g. dipole radiation)

(b) *Guided waves*

Used to transfer energy from one point to another with little loss (e.g. transmission lines and waveguides)

Guided waves and resonant cavities have many important technological applications. For example microwave ovens, which are resonant microwave cavities. This experiment investigates some of the physical properties of guided waves and resonant cavities. The formal theory of waveguides will be covered in PHYS370, but this is not essential to carry out this experiment. The electromagnetism covered in PHYS254, the wave motion covered in PHYS258 and the references are sufficient to allow you to complete this experiment successfully.

Apparatus

The text below describes the various components used in this experiment with a brief description of their function.

Gunn diode – The microwave power is provided by a Gunn diode oscillator. To a small extent, the DC volts determine the microwave frequency.

The Gunn diode is expensive and the following instructions should be noted:

- (i) Always bring up the volts U_G on the diode slowly from zero.
- (ii) Before turning the power off, reduce U_G to zero to prevent possible damage when the diode is next operated.

Isolator – Allows microwaves to pass in one direction only to isolate the Gunn diode from the load. The Gunn diode and isolator provide a microwave source that is unaffected by what happens in the experimental apparatus, such as reflections of the microwaves.

Attenuator – Attenuates the power transmitted to the load via an absorbing vane protruding into the waveguide.

NOTE: The introduction of the attenuator close to the Gunn diode can alter the frequency of detected microwaves. The position of this device should therefore be noted when the system is calibrated and held constant during the experiment.

Frequency meter – The microwave frequency can be determined using the frequency meter. When the frequency meter cavity is adjusted to resonate at the microwave frequency (achieved by adjusting the micrometer setting), a sharp and characteristic dip in the detected signal will occur (because at resonance, power is absorbed from the system). *

Crystal diode detector – The crystal current should be limited to 500 μA , so for your initial investigations connect the detector to the DC input of the oscilloscope, i.e. a high impedance.

NOTE: This is a “square law” relationship detector, which means that the microwave amplitude is related to the square root of the detected signal.

Standing wave detector – Contains a detector in the form of a vane protruding into the waveguide, which can be moved along a length waveguide.

Slide screw tuner – Contains a vane, whose position along the waveguide and penetration depth into the waveguide can be adjusted, to change the phase and amplitude of reflected microwaves.

Dummy load (or Terminator) – Acts to absorb incident microwaves and therefore reflect as little as possible.

All the waveguides used in this experiment have the same width and height and are of the Electronic Industry Association type WR^b90, with inside dimensions of width 2.286 cm, height 1.016 cm for the X-band between 8 and 12 GHz. **Confirm this (Ref. 1).**

Data Acquisition

Preliminary readings and familiarisation of the apparatus

Connect the Gunn diode, isolator, attenuator, frequency meter, standing wave detector, slide screw tuner and detector in series. Investigate the function of each component.

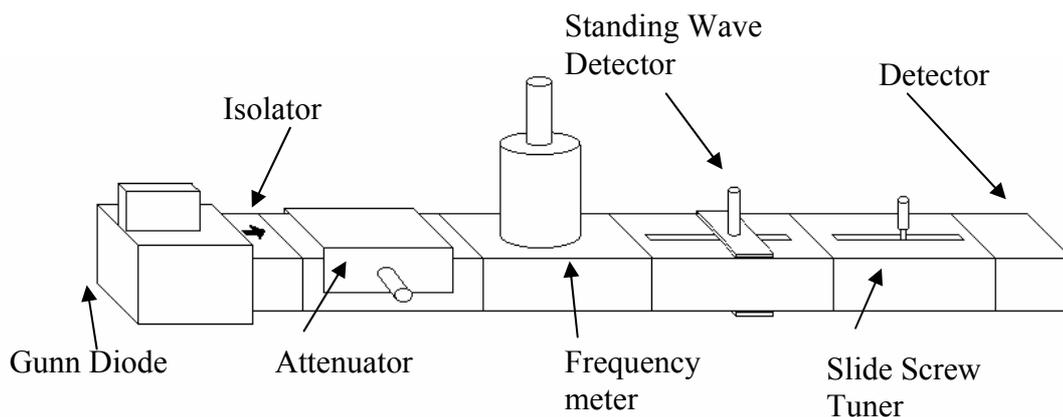


Figure 1: Apparatus assembled in series

Use the calibrated frequency meter to measure the frequency of the Gunn diode. When the frequency meter cavity is adjusted to resonate at the microwave frequency a sharp and characteristic dip in the detected signal will occur. How does this frequency vary with supply voltage U_G ? * After investigating this, set U_G to 10 volts.

Use the relevant equation given in Ref. 1 to calculate the cut-off frequency and then use this to work out the characteristic impedance of the waveguide Z_g at the operational frequency of the cavity. Compare your value with that of free space.

Wavelength in the guide

Remove the detector from the end of the waveguide and withdraw the slide screw tuner so that they do not create reflected waves. Plot out the standing wave with the load connected and with the load removed. Hence measure the wavelength (and therefore the velocity) of waves in the waveguide. Comment on your result.

Wavelength in air

Attach the horn to the end of the waveguide. Using a metal plate and the standing wave detector, measure the wavelength of the microwaves in air. Hence calculate a value for the velocity of microwaves in air. Check the relationship between the wavelength in the waveguide measured with the load removed, the wavelength in air and the dimensions of the waveguide to see how this compares with the relationship predicted by theory.

Properties of the horn

With the load removed from the end of the waveguide, measure the SWR. Fix the horn to the end of the waveguide and again measure the SWR. Compare these results.

Question

What do you conclude about the properties of the horn and why it is used?

Use the references and the information box below as an aid to understanding the SWR.

Standing Waves and the Standing Wave Ratio (SWR)

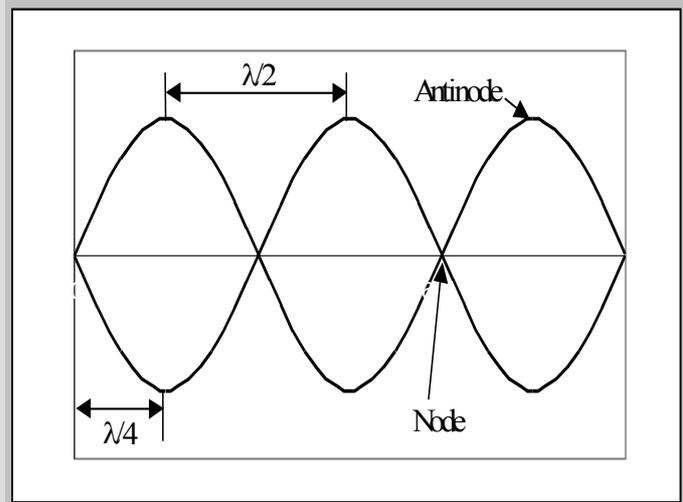
The concept of standing waves is covered in modules PHYS126, PHYS122, PHYS255 and PHYS258. In this experiment standing waves are formed by reflections of microwaves at changes in characteristic impedance, Z . (refer to your notes from PHYS258). The diagram below is included to help you refresh your memory.

The SWR is defined as

$$SWR = \frac{(A + B)}{(A - B)}$$

A = amplitude of incident wave

B = amplitude of reflected wave



Note For complete reflection ($B = A$) $SWR \rightarrow \infty$
i.e. a standing wave with maximum amplitude

For complete absorption ($B = 0$) $SWR \rightarrow 1$
i.e. a travelling wave

[see reference 1, p74–75 for a description of the SWR]

Question

Derive (or look up) the relationship between SWR and the modulus of the reflection coefficient. Hence note that measuring SWR enables this to be calculated. This is sometimes a more useful way of presenting results.

Finding the minimum SWR

Recall that thin coatings on lenses work by interfering waves reflected from the lens/coating interface with those reflected from the coating surface. In this section, SWRs are used to investigate this phenomenon.

Remove the horn so that there is some reflection from the open end of the waveguide. Use the reflections from the end of the waveguide and those from the slide screw tuner to simulate the effect described above. Position the slide screw tuner at a suitable distance from the open end of the waveguide (using the known wavelength in the waveguide). By adjusting the position and depth of the slide screw tuner, find the minimum SWR. Note: the two parameters will need to be varied in a logical manner.

References

1. Liao S Y, *Microwaves Devices and Circuits*, Prentice Hall, 1990
2. Williams T, Wobble Field program (available in T2)
3. Mandl F, *Quantum Mechanics*, Wiley, 1992

Zeeman Effect

Aims

- To gain an appreciation of the relationship between atomic energy levels and transitions between them
- To determine the polarisation states for transitions between energy levels in a magnetic field
- To determine a value for the Bohr magneton

Theory

The Zeeman effect is concerned with transitions in a magnetic field between atomic energy levels with total angular momentum \mathbf{J} , where $\mathbf{J} = \mathbf{L} + \mathbf{S}$ (\mathbf{L} is the orbital angular momentum and \mathbf{S} is the spin angular momentum). The magnetic moment $\boldsymbol{\mu}$ of an atom is related to its total angular momentum (PHYS256). Transitions chosen for this experiment are between the 1D_2 and 1P_1 energy levels, both with total spin $\mathbf{S} = 0$. This simplifies the expression for $\boldsymbol{\mu}$ as only orbital angular momentum is contributing to the magnetic moment (this is known as the *normal Zeeman effect*)

Spectroscopic notation

The energy level of an atom is expressed in terms of the quantum numbers L , S and J . This takes the form $^{2S+1}L_J$ where L is expressed as a letter

$L =$	0,	1,	2...
	S,	P,	D...

$$\boldsymbol{\mu} = -\frac{e}{2m_e} \mathbf{L} = \frac{\mu_B}{\hbar} \mathbf{J}$$

where μ_B is the Bohr magneton. The potential energy of a magnetic dipole in a magnetic field \mathbf{B} is

$$E = -\boldsymbol{\mu} \cdot \mathbf{B}$$

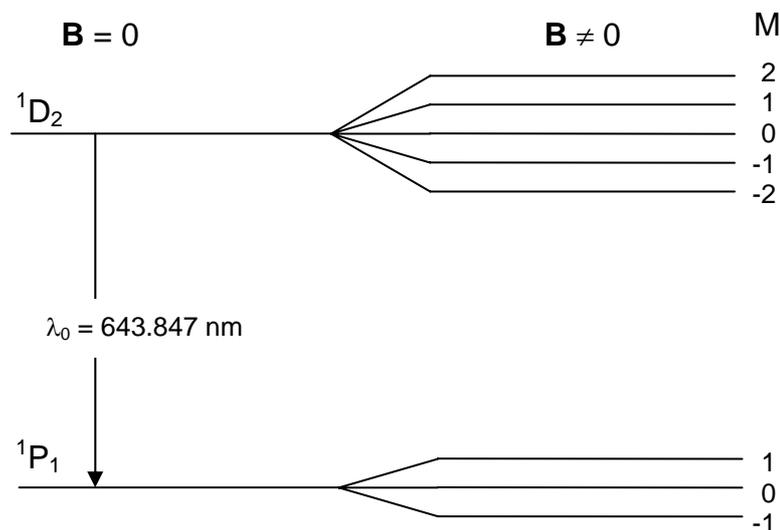
The components of \mathbf{J} in the \mathbf{B} direction are quantised (given by quantum number M) and hence the components of $\boldsymbol{\mu}$ and the values of E are quantised.

Thus, the application of a magnetic field will split an energy level with quantum number J into $2J+1$ separate levels, each equidistant with a spacing between neighbouring levels given by

$$\delta E = \mu_B B$$

If transitions between energy levels with $S \neq 0$ were to take place, the magnetic moment would consist of an orbital component and a spin component. This leads to the Landé g factor (PHYS256 and PHYS361) being included within the expression for energy level splitting. $\delta E = \mu_B g B$

Following this it is possible to draw an energy level diagram showing the splitting due to the Zeeman effect.



Transitions between energy levels are governed by selection rules for M :

$$\Delta M = 0, \pm 1$$

- Using the selection rule for M draw an energy level diagram showing all the possible transitions between the 1D_2 and 1P_1 states.
- Calculate the energy separation between the 1D_2 and 1P_1 states for $B = 0$ and the energy of all transitions for $B = 1\text{T}$. Comment on the difference between these energies and the energy of λ_0 .
- How many distinct transition energies are possible? What is the selection rule corresponding to each transition? (You may need your PHYS361 notes/text book here)

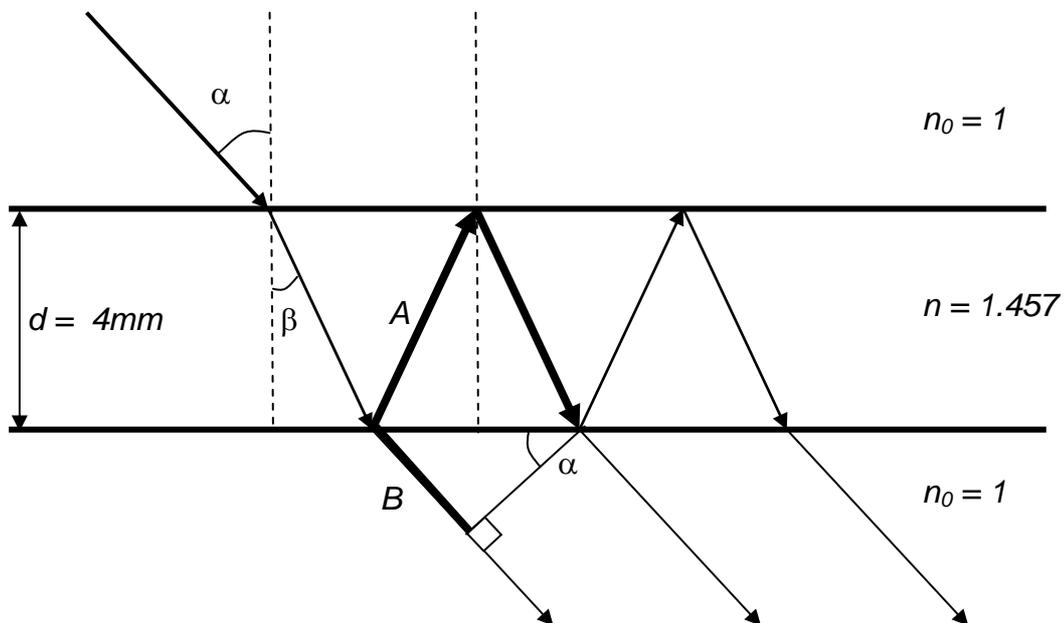
Apparatus

Light Source

A cadmium vapour lamp is placed within the pole pieces of an electromagnet. The pole pieces are hollow to allow light to pass down the axis of the magnetic field. **The cadmium lamp used in this experiment is fragile, please ask a demonstrator for assistance if it requires adjusting.**

Fabry-Perot Etalon

To observe the components of the transition in the red spectral line of cadmium, corresponding to $^1D_2 \rightarrow ^1P_1$, extremely fine shifts in wavelength need to be resolved. A high-resolution spectrometer, such as a Fabry-Perot etalon, is required to distinguish between these components. An etalon is an interferometer consisting of two plane parallel semi-reflective surfaces. A slightly divergent ray enters the etalon and is internally reflected many times, with each reflection a small proportion of the light emerges from the etalon. The emerging light can interfere producing a circular pattern.



Emerging light rays will interfere constructively if the optical path difference is equal to an integer value of the wavelength

$$OPD = k\lambda = 2nA - B$$

- Using the condition for interference, derive an equation relating OPD to α , d and n . Hint: find values for A and B in terms of β and d , and then convert into α . You will need to use Snell's Law. The result is called the etalon equation.
- From this equation calculate values for α , corresponding to $\lambda = \lambda_0$ and k values in the range 18090 to 18110 (best done in a spreadsheet). The circular interference pattern is imaged using a lens of focal length 150mm. What are the radii of the fringes, r_k ? What is the largest value of k for which the condition for interference can be met?
- For the largest value of k , and for values r_k close to that found above (e.g. 0.1mm steps over ± 0.5 mm), calculate values for λ . Express this in the form of $\Delta\lambda/\lambda_0$.

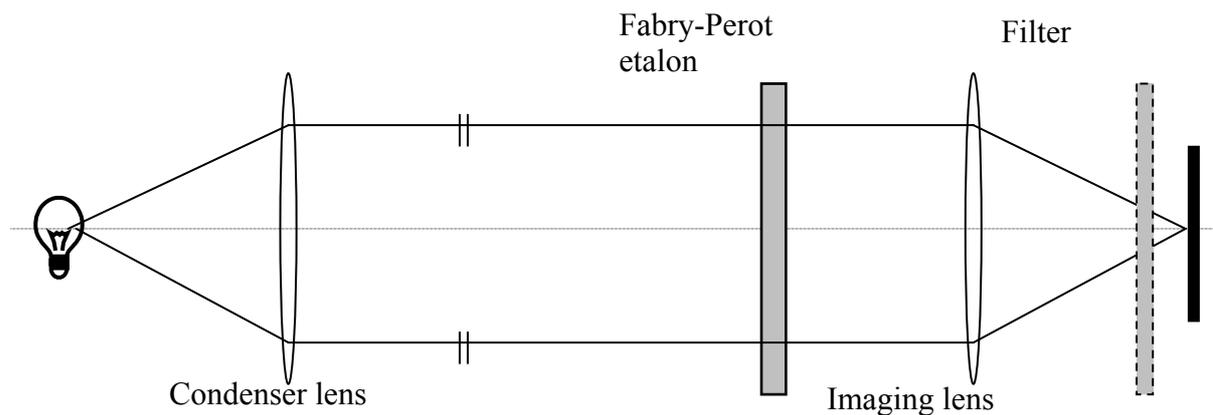
For a 'real' etalon it is impossible to obtain values for d and n of sufficient accuracy to specify k absolutely. A method of finding the shift in λ without involving k can be found by taking differences in the etalon equation.

- Starting from the etalon equation show that $\Delta\lambda/\lambda_0$ is independent of k and d . Confirm the values for $\Delta\lambda/\lambda_0$ found using this equation agree with those calculated above.

Optical Set up

Using a magnet with hollow pole pieces allows the experiment to be conducted normal to the magnetic field (transverse orientation) and parallel to the magnetic field (longitudinal orientation). This is accomplished by rotating the lamp and electromagnet through 90° relative to the optical axis. **Due to the delicate nature of the lamp this should only be done with power to the lamp and magnetic field off and while the apparatus is cool.**

Light from the lamp is made parallel by the condenser lens placed at one focal length away from the source, passes through the etalon and is brought to a focus by the imaging lens. A narrow band pass filter is placed between the imaging lens and the focus to remove contributions from unwanted transitions. The interference pattern is then viewed through an eyepiece or with the CCD camera.



To observe the different polarisations of light a polarisation filter and quarter-wave plate can be added between the condenser lens and the etalon.

Initial Set-up

Before setting up the apparatus the eyepiece may need to be adjusted to suit the individual. To do this, aim at a blank part of a wall and rotate the front half of the barrel until the graduations are clearly visible. Once the ocular has been adjusted, it should be placed at the end of optics bench with the filter next to it.

The etalon should be placed around two thirds of the distance between the cadmium lamp and the focus (with the left edge of the optics rider around the 72 cm position). **The etalon is very delicate, do not touch the optical surfaces.**

The imaging lens then can be adjusted while looking through the eyepiece to produce a sharp bright ring pattern.

With the interference pattern now in focus, the condensing lens and etalon can be adjusted for optimal performance. The condensing lens should be adjusted to give the brightest image possible without obscuring the outer rings. The central position of the pattern can be adjusted by rotating the etalon and fine adjustments are achieved using the three screws.

Data sheets for the Fabry-Perot etalon and the eyepiece are available as part of the supplementary information.

CCD Camera

The CCD camera consists of 2048 pixels in a single line ($\approx 28\text{mm}$ long). Each pixel is $14\ \mu\text{m}$ wide and $200\ \mu\text{m}$ high. It can replace the eyepiece directly without additional lenses, although the imaging lens and etalon may require slight adjustment to ensure the interference pattern is in focus and centered on the CCD strip.

The VideoCom software package is used to read information from the CCD camera. If the camera is switched on and connected to the PC it should be recognised when the software is loaded. If this is not the case, please ask a demonstrator for assistance.

To access the data taken from the CCD the software should be running in the “Intensity I1” mode. During set up the camera can run with a reduced number of pixels to speed up the data acquisition (F8) and for final readings the full number of pixels should be used (F9). The exposure time can also be adjusted, and should be set so that the largest peaks are around 50-70%. A running average is also available to improve the signal to noise ratio of the intensities. All these options are available as buttons in the top tool bar.

A selection of tools for analysis of a peak (such as Zoom and Display Coordinates) is accessed through a mouse right-click. It is suggested that you practice using the VideoCom software before taking your final data and also investigate the error on determining a peak position.

Polarisation States

Light emitted by the lamp can either be plane polarised or circularly polarised. The states observed are dependent on the orientation of the magnetic field relative to the optical axis and also the transitions involved.

Plane Polarisation

The plane containing the **E** vector is called the plane of polarisation. Using a polarising filter it is possible to preferentially absorb or transmit light with particular planes of polarisation.

Circular Polarisation *

Circular-polarised light is a wave with an \mathbf{E} vector (of constant amplitude) which is perpendicular to the direction of propagation and which rotates with angular frequency ω about that direction. A circular-polarised wave can be constructed from two plane-polarised waves, with orthogonal polarisation planes (labelled O and E), that differ in phase by $\pi/2$. Depending on the sign of the phase difference, the circular-polarised wave can be in one of two states — left or right handed. If the vector \mathbf{E} is rotating clockwise, looking in the direction of propagation, the wave is described as right circular-polarised [3].

Polarisation Analysis *

Circular-polarised light can be analysed using birefringent or biaxial materials. These materials are characterised by the way that light with different planes of polarisation is propagated through the material. A suitable thickness of such a material can be used to introduce an $\pi/2$ phase difference between O and E waves. Known as Quarter-Wave Plates, they are used to convert circular-polarised light into plane-polarised light and vice versa [2]. Thus in conjunction with a polarising filter, it is possible to determine the state of circular polarised light.

Investigation of Zeeman components

The angular intensity distribution of light emitted can be explained by drawing classical analogues to dipole oscillators. Covered in more detail in PHYS370 Advanced Electromagnetism, these are summarised in Fig. 3 of Ref. [4].

- Using the apparatus in both transverse and longitudinal orientations determine the polarisation state of each of the Zeeman-split components. Check that this is consistent with Fig. 3 in Ref. [4]. *

Data Acquisition

With experiment set up in the transverse mode, using the eyepiece as the imaging device, turn up the magnetic field until the Zeeman splitting is clear.

- Using the scale on the eyepiece record the diameter of the first clearly visible ring set. Estimate a value for the Bohr magneton from your equation for $\Delta\lambda/\lambda_0$. Assume the magnetic field generated is 100mT/A.

Replace the eyepiece with the CCD camera (ask a demonstrator if you are unsure how to do this).

For each of the transverse and longitudinal orientations:

- Record the diameter of an interference ring set for a range of magnetic fields. For each measurement:
 - Calculate the angle α
 - Calculate $\Delta\lambda/\lambda_0$
 - Calculate ΔE .
- Plot a graph of ΔE versus B and calculate a value for the Bohr magneton. You will need to use the magnetic field calibration data provided in the supplementary information booklet.

References

1. Mandl F, *Quantum Mechanics*, Wiley, 1992
2. Longhurst R S, *Geometrical and Physical Optics*, Longman 1973
3. Grant I S and Philips W R, *Electromagnetism*, Wiley 1990
4. Leybold Didactic, *Physics Leaflet P.6.2.7.3*

Risk Assessment for Block C

In addition to the general safety rules listed on Page 17 of this Laboratory Handbook, the following hazards should be noted:

Electrical Hazards

Various items of instrumentation are powered by mains electricity. No such item should be disassembled or have its plug or fuse(s) replaced. If an electrical item appears to be faulty, consult the laboratory technician.

Chemical Hazard

Some experiments require the use of chemical materials:

Absorption of Iodine: The vessel containing the iodine must remain sealed. If the vessel is damaged, consult the laboratory technician.

Risk Assessment Forms and Local Safety Rules

THE UNIVERSITY *of* LIVERPOOL

RISK ASSESSMENT

Department: Physics	Location: Chadwick Laboratory T7	
Brief description of work/project: PHYS378 Block A Radiation Detection		
Person(s) involved: Undergraduate students	Status (Technician, research student etc.): Undergraduate students	
Main risks of the work/project Various items of instrumentation are powered by mains electricity. High voltage power supplies are capable of producing lethal voltages. All of the Block A experiments, including the group work, require radioactive sources.	Precautions required No such item should be disassembled or have its plug or fuse(s) replaced. If an electrical item appears to be faulty, consult the laboratory technician. Pay particular attention to the cables that connect the high voltage to other instrumentation – are they in a good condition, with no obvious damage? Radioactive sources must be handled in accordance with the Local Radiation Rules that are detailed in this handbook and posted on the walls of the laboratory (T7).	
Relevant Codes of practice or local rules to be followed: 1. Laboratory safety code of practice as detailed in this Handbook and posted in the laboratory 2. Local Radiation Rules as detailed in this Handbook and posted in the laboratory		
<i>For research students:</i> Level of supervision (circle)	A = Work may not be started without direct supervision B = Work may not be started without supervisor's advice or approval C = No specific extra supervision requirements	
Date for review of assessment (maximum period, 5 years) August 2009		
Authorisation: D.S. Martin	Status: PHYS378 Organiser	Date: 19 / 8 / 2006
Acknowledgement by person(s) involved: n/a		

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RISK ASSESSMENT

Department: Physics	Location: Chadwick Laboratory T7
Brief description of work/project: PHYS378 Block B X-Ray Diffraction	
Person(s) involved: Undergraduate students	Status (Technician, research student etc.): Undergraduate students
Main risks of the work/project The x-ray generator produces high-voltages and x-ray radiation. The salts that are used to make the samples for x-ray analysis may be mildly irritating.	Precautions required The x-ray generator must be switched on and off only by authorised staff or demonstrators. Students can load their samples into the x-ray diffractometer and open/close the x-ray shutters to expose their samples after receiving appropriate instructions from staff or demonstrators. The x-ray set (generator and diffractometer) must be operated in accordance with the Local Radiation Rules that are posted beside the x-ray set. The salts should be handled with the gloves and safety glasses provided. The salts should not be ingested.
Relevant Codes of practice or local rules to be followed: 1. Laboratory safety code of practice as detailed in this Handbook and posted in the laboratory 2. Local Radiation Rules as detailed in this Handbook and posted in the laboratory	
<i>For research students:</i> Level of supervision (circle)	A = Work may not be started without direct supervision B = Work may not be started without supervisor's advice or approval C = No specific extra supervision requirements
Date for review of assessment (maximum period, 5 years) August 2009	
Authorisation: D.S. Martin	Status: PHYS378 Organiser Date: 19 / 8 / 2006
Acknowledgement by person(s) involved: n/a	

THE UNIVERSITY *of* LIVERPOOL

RISK ASSESSMENT

Department: Physics	Location: Chadwick Laboratory T7
Brief description of work/project: PHYS378 Block C Absorption Spectrum of Iodine	
Person(s) involved: Undergraduate students	Status (Technician, research student etc.): Undergraduate students
Main risks of the work/project Various items of apparatus are powered by mains electricity. The vessel containing the iodine must remain sealed.	Precautions required No such item should be disassembled or have its plug or fuse(s) replaced. If an electrical item appears to be faulty, consult the laboratory technician. If the vessel is damaged, consult the laboratory technician.
Relevant Codes of practice or local rules to be followed: Laboratory safety code of practice as detailed in this Handbook and posted in the laboratory	
<i>For research students:</i> Level of supervision (circle)	A = Work may not be started without direct supervision B = Work may not be started without supervisor's advice or approval C = No specific extra supervision requirements
Date for review of assessment (maximum period, 5 years) August 2009	
Authorisation: D.S. Martin	Status: PHYS378 Organiser Date: 19 / 8 / 2006
Acknowledgement by person(s) involved: n/a	

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RISK ASSESSMENT

Department: Physics	Location: Chadwick Laboratory T7
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Brief description of work/project: **PHYS378 Block C Microwave Waveguide**

Person(s) involved: Undergraduate students	Status (Technician, research student etc.): Undergraduate students
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Main risks of the work/project

Various items of instrumentation are powered by mains electricity.

Precautions required

No such item should be disassembled or have its plug or fuse(s) replaced. If an electrical item appears to be faulty, consult the laboratory technician.

Relevant Codes of practice or local rules to be followed:
Laboratory safety code of practice as detailed in this Handbook and posted in the laboratory

<i>For research students:</i> Level of supervision (circle)	A = Work may not be started without direct supervision B = Work may not be started without supervisor's advice or approval C = No specific extra supervision requirements
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Date for review of assessment (maximum period, 5 years) August 2009

Authorisation: D.S. Martin Status: PHYS378 Organiser Date: 19 / 8 / 2006

Acknowledgement by person(s) involved: n/a

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RISK ASSESSMENT

Department: Physics	Location: Chadwick Laboratory T7
Brief description of work/project: PHYS378 Block C Zeeman Effect	
Person(s) involved: Undergraduate students	Status (Technician, research student etc.): Undergraduate students
Main risks of the work/project Various items of apparatus are powered by mains electricity. Cadmium Vapour Lamp	Precautions required No such item should be disassembled or have its plug or fuse(s) replaced. If an electrical item appears to be faulty, consult the laboratory technician. The lamp should not be touched, and the surrounding magnetic yoke must not be adjusted by students.
Relevant Codes of practice or local rules to be followed: Laboratory safety code of practice as detailed in this Handbook and posted in the laboratory	
<i>For research students:</i> Level of supervision (circle)	A = Work may not be started without direct supervision B = Work may not be started without supervisor's advice or approval C = No specific extra supervision requirements
Date for review of assessment (maximum period, 5 years) August 2009	
Authorisation: D.S. Martin	Status: PHYS378 Organiser Date: 19 / 8 / 2006
Acknowledgement by person(s) involved: n/a	

UNIVERSITY *of* LIVERPOOL

LOCAL RULES

(Extract)

Department of Physics, Chadwick Laboratory

Revised December 2002

ADMINISTRATIVE ORGANISATION

These Local Rules are issued by the University and are intended to ensure that unsealed radioactive sources and x-ray sets are used safely and in accordance with the requirements of "The Ionising Radiations Regulations 1999" and the associated Approved Code of Practice prepared for the Health and Safety Executive and published by H.M.S.O. They are required in law and must be read, understood and obeyed by all staff. Any user not complying with these Local Rules or with the standard required may have their permit to work revoked and render themselves liable to prosecution.

UNIVERSITY RADIATION PROTECTION ADVISER

The University has appointed Dr P.R. Cole, as University Radiation Protection Adviser, (U.R.P.A.). His duties are to advise as to the observance of the relevant Regulations and as to other Health and Safety matters in connection with ionising and non-ionising radiation.

DEPARTMENTAL RADIATION PROTECTION SUPERVISOR

The University has appointed Dr M.A. Houlden as Departmental Radiation Protection Supervisor (D.R.P.S.) for the Department of Physics. He shall administer the Local Rules, the requirements of the Regulations and Approved Code of Practice and shall report to the Head of Department and the Registrar.

The duties may be summarised as follows:

1. To ensure that the recommendations of the Senate Health & Safety Committee and the rules contained in the Ionising Radiations Regulations 1999 and the associated Codes of Practice are carried out within the department.
2. To keep a register of all radioactive sources within the department and of all disposals of radioactive waste. In this respect it is particularly important that the conditions contained in the Certificates of Registration and Authorisation issued under the Radioactive Substances Act should not be violated.

A monthly return of all sources purchased, current stock and waste disposed of should be made on form RP2A to the University Radiation Protection Adviser (U.R.P.A.). Whenever solid radioactive waste is to be removed, application to the U.R.P.A. should be made on Waste Container Declaration.

3. To be responsible for the issue and collection of any radiation film badges used for monitoring personnel exposure.
4. To inform the U.R.P.A. whenever new radiation workers are about to commence work in the department, and whenever people are intending to leave so that the necessary medical examinations may be carried out. Form RP6 (proposed scheme of work) should be completed and submitted for new workers.
5. To inform the U.R.P.A. in the event of any serious spill of radioactive material or any suspected high radiation exposure.
6. To inform the U.R.P.A. of the purchase of any new items of equipment (X-ray sets, Electron Microscopes, Lasers, U.V. sources etc.) which emit radiations or of any proposed major changes in the use of radioisotopes.

DESIGNATION OF RADIATION AREAS

REGISTERED RADIATION AREA

A laboratory in which part is set aside for low-level radiation work is designated as a Registered Radiation Area. Non-radiation workers are permitted to use other facilities within the laboratory and the part in which radiation work is done must be clearly indicated or demarcated.

Labs T1, T3, T4(1), T7, and T12 are registered radiation areas. (*Note - Areas within T3 and T4(1) are Supervised and/or Controlled Areas which are covered by Local Rules for Neutron Sources*)

Cleaners should have clear, written instructions from the D.R.P.S. when entering a laboratory in which there is a registered radiation area.

REGISTERED RADIATION WORKERS

All personnel, other than undergraduates, routinely working with radioactive sources (henceforth termed "the user") must become registered radiation workers. The D.R.P.S. must be informed before commencing work with radioactive materials. Form RP6 (obtainable from the D.R.P.S. or U.R.P.A.) must be completed and returned to the Radiation Protection Office. Permission will then be given to commence work with radioactive material.

DUTIES OF REGISTERED RADIATION WORKERS

The University of Liverpool is registered under Section 1 of the Radioactive Substances Act 1993 to keep and use a variety of specified radioactive materials and is also authorised to accumulate and dispose of radioactive waste materials arising from its activities. A duty is placed on the University to ensure that the requirements of the Registration and Authorisation are met and it is therefore necessary that permission be obtained from the D.R.P.S. before any radioactive source or material is brought to or taken from the University.

Each user must read and understand the relevant sections of the "Approved Code of Practice" issued by H.M.S.O. and held by the D.R.P.S. Each user must bear in mind that it is his duty to protect himself and others from the hazards associated with unsealed radioactive sources.

ORDERING AND RECEIPT OF RADIOACTIVE MATERIALS

- 1) The user shall not order or obtain any radioactive material without the prior knowledge and consent of the D.R.P.S.
- 2) On the receipt of radioactive material into the department the user shall notify the details of the material to the D.R.P.S. who shall initiate an entry in the record for that material.

Local Rules for the Use of Closed Radioactive Sources

Revised December 2002

USE OF RADIOACTIVE MATERIAL

- 1) The user shall work in accordance with the experimental procedures detailed in “Procedures Applicable to the Chadwick Laboratory” below.
- 2) Work with radioactive materials should not take place outside normal working hours unless at least two people are present in the building and written permission has been obtained from the D.R.P.S.
- 3) Movement of sources into or out of the Source Store must be legibly recorded in the Log book set aside for the purpose in order that the location of sources is adequately identified.
- 4) The minimum quantity of radioactivity compatible with the objectives of the experiment should be used. Work should be carried out carefully and tidily.
- 5) A monthly check of sources shall be made.

DOSE INVESTIGATION LEVEL

The University sets an Investigation Level of 2mSv on an individual's whole body dose accumulated in any calendar year. The Radiation Protection Office routinely reviews doses received by individuals and will conduct an Investigation, in consultation with the Departmental Radiation Protection Supervisor, if a whole body dose of more than 2mSv is accumulated in any calendar year. This Investigation Level is one third of the maximum permissible dose for a non-classified worker (6mSv)

INCIDENT CONTINGENCY

LOSS OF SOURCE

If a source is not accounted for and believed to be missing then the D.R.P.S and U.R.P.A. shall be notified immediately.

ACCIDENTAL EXPOSURE

If any person is believed to have been subjected to an accidental exposure then the apparatus and source shall be left untouched, the area shall be closed for investigation and the D.R.P.S and U.R.P.A. shall be notified immediately. An assessment of the likely exposure will be made by the D.R.P.S and U.R.P.A.

PROCEDURES APPLICABLE TO THE CHADWICK LABORATORY

1. A register of all sources in the Chadwick Laboratory shall be kept up to date by a designated person (Mr P. Rossitter).
2. Personnel are only permitted to work in the registered area provided that they maintain radiation dose rates in occupied parts of the laboratory at less than $2.5\mu\text{Sv/hr}$.
3. Sources must be signed out when in use and returned immediately after use.
4. When not in use in experiments for any period exceeding 2 weeks all sources shall be kept in the metal safes in Rooms T3, T7 and T12. The Sr^{90} sources shall be kept in the β -ray spectrometers in T3. (*Note - The Am/Be neutron sources in labs T7 and T4(1) are covered by separate Local Rules for Neutron Sources*).
5. Handling of all γ -rays reference sources of strength 400kBq ($10\mu\text{Ci}$) or less must be effected with tweezers, forceps or similar unless contained in such a way that the maximum dose rate from any part of the container is $2.5\mu\text{Sv/hr}$.
6. Handling of all other sources must be effected only by a technician or a member of academic staff who is a registered radiation worker. The sources must be held using tweezers, forceps or similar, and rubber gloves and safety glasses must be worn. Sources must be transported to different parts of the Chadwick Laboratory in lead pots so that the external radiation from the source is less than $2.5\mu\text{Sv/hr}$.
7. Experiments using sources must be shielded from all personnel so that the maximum dose rate at a working distance is less than $2.5\mu\text{Sv/hr}$.
8. No food or drink must be consumed in the vicinity of experiments involving sources.

RISK ASSESSMENT

With the exception of three Cs^{137} sources in lab T3 and the neutron sources covered by separate Local Rules, all sources in Chadwick Laboratory are closed sources having individual activity not exceeding 300kBq . The sources are routinely wipe tested and hence the risk of ingestion of radioactive material through contamination is insignificant. The sources are handled for an estimated maximum period of 15 minutes in any week (being transferred from store to experiment and vice versa) therefore the external dose hazard from the sources less than 300kBq is below 20 microSieverts per year. The three Cs^{137} sources in lab T3 are located in lead shielding within the experiment such that the external dose is less than 2 microSieverts per hour to the experimenter. With each experiment having a duration of three hours and being conducted once by each experimenter during any university year, the external dose received by the experimenter is less than 6 microSieverts per year. The installation and removal of Cs^{137} sources is undertaken by technical staff. The handling time of a source being installed or removed is estimated not to exceed 2 minutes in a radiation field not exceeding 120 microSieverts per hour producing a resultant dose of 4 microSieverts per source per operation. Each source is installed and removed once per year resulting in a maximum dose to the handler of 24 microSieverts per year. It is considered to be extremely unlikely that any person will receive a dose exceeding 50 microSieverts in a year

Local Rules for the Use of Am/Be Neutron Sources

Revised October 2002

DESIGNATION OF RADIATION AREAS

CONTROLLED RADIATION AREA

The Controlled Radiation Area is deemed to be anywhere within the water tank.

SUPERVISED RADIATION AREA

The area 25cm surrounding the water tank contains an instantaneous dose rate exceeding 7.5 μSv per hour and is designated a Supervised Radiation Area. The Supervised Radiation Area boundary is demarcated with a yellow line on the floor of T7 around the tank. Warning signs are positioned on the wall next to the tank.

AUTHORISED USERS OF NEUTRON SOURCE FACILITY

The neutron source facility in Lab T7 is used by staff and students of the University of Liverpool and by visiting staff and students undertaking courses or training under the aegis of the University of Liverpool.

Prior to working in the facility each user must be aware of the Local Rules. Each user must also be given a copy of the Risk Assessment and an abbreviated copy of the Local Rules which will include the Operating Procedure to be followed and the Contingency Plan.

Each user must bear in mind that it is his duty to protect himself and others from the hazards associated with neutron sources.

USE OF RADIOACTIVE MATERIAL

- 1) The user shall work in accordance with the “**EXPERIMENTAL PROCEDURES**” detailed below.
- 2) Work should not take place outside normal working hours unless at least two people are present in the neutron source facility.

INCIDENT CONTINGENCY

LOSS OF CONTAINMENT (Water Tank)

The water in the water tank acts also as the moderator which thermalises the neutron flux. In the event of rupture of the tank the moderator effect would be lost. If the presence of water outside the tank indicated the loss of containment then the facility shall be evacuated and secured by locking access door, and the D.R.P.S. and U.R.P.A. shall be notified immediately (see also RISK ASSESSMENT).

The water level in the tank should be monitored at regular intervals and its level recorded at least once per week and the check log (T7_WATERCHECK.DOC) filled in.

LOSS OF SOURCE (Water Tank)

If a source is not accounted for and believed to be missing then the D.R.P.S and U.R.P.A.

ACCIDENTAL EXPOSURE

If any person is believed to have been subjected to an exposure greater than expected then the apparatus and source shall be left untouched, the area shall be closed for investigation and the D.R.P.S and U.R.P.A. shall be notified immediately. An assessment of the likely exposure will be made by the D.R.P.S and U.R.P.A.

DOSE INVESTIGATION LEVEL

The University sets an Investigation Level of 2mSv on an individual's whole body dose accumulated in any calendar year. The Radiation Protection Office routinely reviews doses received by individuals and will conduct an Investigation, in consultation with the Departmental Radiation Protection Supervisor, if a whole body dose of more than 2mSv is accumulated in any calendar year. This Investigation Level is one third of the maximum permissible dose for a non-classified worker (6mSv)

EXPERIMENTAL PROCEDURES

THE NEUTRON SOURCE EXPERIMENT (T7)

The neutron source is Am/Be with an activity of 11.1 GBq on 23/09/02 which is housed in the centre of a water tank. Samples can be put into the tank for irradiation by staff and students. Only samples specified in experimental scripts or samples agreed by the laboratory supervisor can be put into the tank. These samples will normally be hung in the water on supplied metal rods. Samples will be either metal foils or be contained in a waterproof container. Samples can only be put into the tank through the small opening in the metal grid that covers the tank. Once removed the activity of samples should be checked with the radiation monitor supplied and then taken quickly to the appropriate counting apparatus. Staff and students should wear protective plastic gloves while putting samples into the tank or while removing samples. Irradiated samples should only be handled with the tweezers provided. Staff and students should not remain adjacent to the tank containing the neutron source for longer than is necessary to load or remove a sample. Once an irradiated sample is no longer needed for experimental measurements it should be stored in the shielded storage area available. If a sample drops to the bottom of the tank then staff and students should not attempt to retrieve it. This should be reported to the Laboratory technician who will retrieve the sample using an appropriate handling device.

RISK ASSESSMENT

NORMAL USE

The Dose rates in μSv per hour in September 2002 around the source are as follows:-

	Water Tank	
	Neutron	Gamma
Front of tank	9	3.9
Sides of tank	1	0.7
Zero moderation (1m)	5	4.6

All measurements made using NE 0075 neutron monitor (serial number 081) for neutrons and Radalert 1201 (serial number 2635) for gamma

Risk Assessment for normal use – see documents RA_T7_tank.doc (Appendix 1) and T7_doses_0902.doc (Appendix 2).

APPENDIX 1 : RISK ASSESSMENT

Risk Assessment for Routine Use of Neutron Source (NPRL 261) in Lab T7 of Chadwick Building.

Source is Am/Be (≈ 11.1 GBq on 23/09/02). Situated in the water tank in Lab T7.

Hazard	Persons at Risk	Current Control Measures	Actions Required
1. Breach of inner tank.	Staff Students	Outer tank will hold the water. Water level checked and logged weekly by technician. Sudden drop in level will indicate such a breach. Contingency plans for loss of containment in local rules.	
2. Breach of both tanks.	Staff Students General Public.	Very unlikely to occur. Lab used most days and water spill would be noticed. Contingency plans for loss of containment in local rules.	
3. Evaporation of water.	Staff Students	Water level checked and logged weekly by technician. Water level rod indicates level. If water level drops below the lower yellow mark on the rod then it is topped-up back to the higher yellow mark.	
4. Loss or theft of source.	Staff Students General Public.	Lab door is locked when not occupied. Keys kept secure. Source is padlocked into its housing tube. Keys kept secure.	
5. Unauthorised tampering.	Unauthorised user.	Source is padlocked into its housing tube. Keys kept secure. Grill permanently fixed to the top of the tank. Tank clearly labelled with radiation warning signs.	
6. Radiation dose from routine use of the source.	Staff Students General Public	See T7_doses_0902: Neutron and γ Dose Rate Measurements. In a worst case scenario, an individual would spend no longer than 10 minutes at a distance of 10cm from the front of the tank. This would result in a whole body dose of $13/6 = 2.2 \mu\text{Sv}$ per year. Neutron dose rate on the pavement outside was measured to be zero, and the gamma dose rate to be below background.	

Assessor : Peter Cole

Date : 23rd September 2002

Review Date : 23rd September 2003

Monitors used:

RadAlert 1201 (Next calibration due: 01/06/03)

NE Neutron DE Rate 95/0075-1/6 (Next calibration due: 12/12/02).

APPENDIX 2: DOSE RATES AROUND TANK

Measurement of Neutron and Gamma Dose Rates around Am/Be Source (NPRL 261) in Water Tank of Lab T7 of the Teaching Labs of the Chadwick Building.

1. Source Exposed.

Distance (cm)	Gamma Dose Rate ($\mu\text{Sv/hr}$)	Neutron Dose Rate ($\mu\text{Sv/hr}$)
30	60.5	40
50	26.8	20
100	4.6	5
150	3.0	2.5

2. Source in central position in water tank.

Postn of Reading	γ Dose Rate ($\mu\text{Sv/hr}$)	Neutron Dose Rate ($\mu\text{Sv/hr}$)
10cm from tank front (32cm from source)	3.9	9
20cm from tank front (42cm from source)	2.8	5
30cm from tank front (52cm from source)	1.9	3.5
78cm from tank front (100cm from source)	0.6	1
10cm from tank side (66cm from source)	0.7	1
Behind tank on outside wall. Public access.	< background	0

Measurements done by : P.R. Cole
Date : September 2002.
Instruments used : RadAlert 1201 (Next calibration due: 01/06/03)
NE Neutron DE Rate (Next calibration due: 12/12/02).

Local Rules for Work with Enclosed X-Ray Sets

Revised November 2002

DESIGNATION OF RADIATION AREAS

CONTROLLED RADIATION AREA

The area contained within the X-ray enclosure is designated as a Controlled Radiation Area whilst the X-ray set is operating with the shutters open. No person shall have access to the Controlled Area whilst the set is in normal use. Access will be permitted for lining up procedures only.

Lining up procedures may be carried out only by the appointed radiographer and at minimum power settings. The interlocks may only be overridden by key operation during lining up procedures by the appointed radiographer who shall retain possession of the key at all times.

RADIOGRAPHY PROCEDURES

The appointed radiographer is Mr V. Vasey (or Dr S.D. Barrett or Dr D.S. Martin in his absence). The radiographer shall supervise radiographic procedures; in that capacity he shall have authority over all persons present during X-ray production. The correct sequence as listed in the "X-ray Procedures" notice posted on the X-ray enclosure, and given in these Local Rules, should be rigorously followed.

X-RAY SET

The X-ray set may not be modified so as to alter its performance or shielding without the express permission of the D.R.P.S. in consultation with the U.R.P.A. Any malfunction shall be reported immediately to him.

PERSONAL MONITORING

There is no radiation detectable above background outside the defined Controlled Area (ie outside the Diffractometer housing). There is therefore no requirement for personal monitoring

CONTINGENCY PLAN

FAILURE TO TERMINATE

If the x-ray exposure indicator indicates that an exposure has failed to terminate:

1. Press Emergency OFF button on wall adjacent to power supply
2. Notify D.R.P.S. immediately
3. Arrange for maintenance

OVEREXPOSURE

In case of suspected accidental over-exposure to X-radiation :

1. The equipment should be left untouched so that a reconstruction/dose determination can be carried out.
2. Inform the DRPS and URPA
3. Check the area of skin where the beam could have been incident at hourly intervals. With a high absorbed dose (say 10Sv upwards) skin goes a brick red colour in a few hours (similar to sunburn). This may appear up to 12 hours after the incident.
4. In the event of a skin reaction contact Dr N. Wilson (Occupational Health Service).

SERVICING / OUTSIDE USERS

When control of any part of the machine is passed to a person not employed by the University of Liverpool (eg to a servicing engineer) then the following procedure must be adopted. The person shall be informed in writing that responsibility for provision of Local Rules is devolved to his/her employer whilst under their control. An acknowledgement signed by that person shall be obtained for, and retained by, the D.R.P.S.

OPERATING PROCEDURES

PHILIPS PW1729 X-RAY GENERATOR

Alignment, adjustment, repair and exchange of components and X-ray tube must only be performed by qualified personnel.

The following persons are authorised to operate the x-ray generator:

**Dr D S Martin
Vince Vasey
Dr S D Barrett**

**Trained postgraduate demonstrators
(list near apparatus)**

SWITCHING ON

1. Switch on main isolator on wall.
2. Turn on mains water supply valve (red valve) under bench.
3. Switch on pump isolator and press green contactor button to operate water pump.
4. A water pressure of 40 p.s.i. will allow the X-ray generator to be switched on, but use of the darkroom sink taps causes the pressure to drop and the machine to shut down. Check the water pressure on the gauge. It should read 65psi so that when the cold water tap in the darkroom is fully open the pressure still reads 45psi and the generator will still operate. There is a by-pass valve that can be adjusted to produce the required pressure but any major adjustment will mean that the in-line filter needs cleaning.
5. Turn key-switch on rear panel to **ON**
6. Check that kV and mA controls are set to a minimum.
7. Ensure all doors are closed
8. Operate the Generator '**ON**' push button. If the safety circuit is complete, the 'X-Rays On' lamps will light (one on the rear-mounted post and one on the front panel). - two red lamps will also illuminate (outside and above the door) as a warning that X-rays are being generated. The lamp in the push button will also be lit.
If the generator fails to operate this will be due to one of the following :-
 - 1 Low water pressure - filter needs cleaning
 - 2 Failure of 'X-Rays On' bulb in either the front panel or rear-mounted post.
 - 3 A blocked filter in the X-ray tube head (see separate note).
9. Slowly turn up the mA and kV controls to 30mA and 40kV.

SWITCHING OFF

When no more exposures are required :-

- 1 Slowly turn down the mA and kV controls to minimum.
- 2 Press the generator **O** pushbutton (this should put all the '**X-Rays On**' lamps off).
- 3 Turn the key-switch at the rear to **OFF**.
- 4 Switch water pump off.
- 5 Turn off water supply valve.
- 6 Switch **OFF** mains isolator on wall

DIFFRACTOMETER SYSTEM PW1840 OPERATION

A metal housing encloses the complete Diffractometer, giving full protection against stray radiation thereby conforming to safety standards. This metal housing can be removed for certain adjustments and maintenance work, but this must only be done by the Radiographer or other authorised and competent person.

LOADING THE SAMPLE

If open, close the shutter by operating the **SHUTTER 4 O** pushbutton

The sample chamber is accessible when the cover lid is removed by turning it anticlockwise. *(The movement of the extension arm releases the tube shield safety interlock and, if not already closed, the beam shutter will close).*

The sample and holder are placed in position by pushing down the spring under the guide block and sliding the sample holder into place. *(Solid samples or Glass slides can be used instead of the sample holder).*

Replace the cover lid, ensuring that the tube shield interlock button is operated.

OPERATION

Open the shutter window 4 by simultaneously pressing the two shutter pushbuttons **SHUTTERS** and **SHUTTERS 4 I**.

If the shutter fails to open (ie. the shutter lamp fails to light), check that the shutter clock position switch for positions 3 and 4 is in the central position (infinite time).

The goniometer arm can then be operated by computer control.

When the scan is finished, the shutter should be closed by operating the **SHUTTERS 4 O** pushbutton.

MAINTENANCE

CLEANING IN-LINE WATER FILTER

- 1 Switch off pump then turn off water.
- 2 Unscrew base of filter unit, remove dirty filter and either fit new filter or force-wash the old one.
- 3 Reassemble filter unit, turn on water, switch on pump.

REMOVAL OF X-RAY TUBE AND CLEANING THE X-RAY HEAD FILTER

WARNING - Do not remove the X-ray tube if the generator has switched off because of a failure (safety circuit actuated).

- 1 Switch off generator by pressing generator **O** pushbutton.
- 2 Turn off water pump at contactor.
- 3 Turn off water supply.
- 4 Switch off mains isolator.
- 5 Drain water from the set by opening the air inlet valve next to the water inlet and outlet connections at the rear of the machine and wait until air has filled the system. Some water may run from the air inlet valve. This process can be speeded up by pumping the excess water out with a bicycle pump.

- 6 Unscrew the two slotted bolts at opposite corners of the tube head.
- 7 The X-ray tube should be lifted from the tube shield very carefully, handling by the metal base. **Do not touch the beryllium windows.** These windows are thin and can be poisonous to the skin.
- 8 To reach the filter remove the 4 allen bolts in the tube head and remove the head. The filter is under the centre cap which has a slot in its end. Wash the filter and cap and refit re-assembled tube, first checking the condition of the "O" rings for the water tubes.
- 9 Close the air inlet valve, turn on the water supply and check there are no leaks.

The tube will need to be checked for alignment after removal and refitting of an X-ray tube.

RISK ASSESSMENT

NORMAL USE

The metal housing totally enclosing the Diffractometer gives full protection against stray radiation in normal use. Access to the sample chamber is via an interlocked cover that causes the beam shutter to close (if not already closed) when the cover is opened.

MAINTENANCE

The Diffractometer and the tube may only be maintained by authorised persons. Realignment of the tube after removal and refitting should be made at minimum voltage and current settings to minimise dose. Care must be taken by the authorised person to avoid passing any part of the body through the x-ray beam.