實驗六　　X-ray 繞射實驗

原理：電磁波波長可用光柵測量，但 X 光波長極小，光柵不易製作。Sr L. Bragg 認為晶體可作為 X-光之光柵。如圖(1)，建設性干涉時，入射及反射線之夾角 2θ 滿足 Bragg 條件：

$$n\lambda = 2d\sin\theta \qquad n = 1,2,\ldots$$

(Herht: Optics 或 Guenther: Modern Optics)

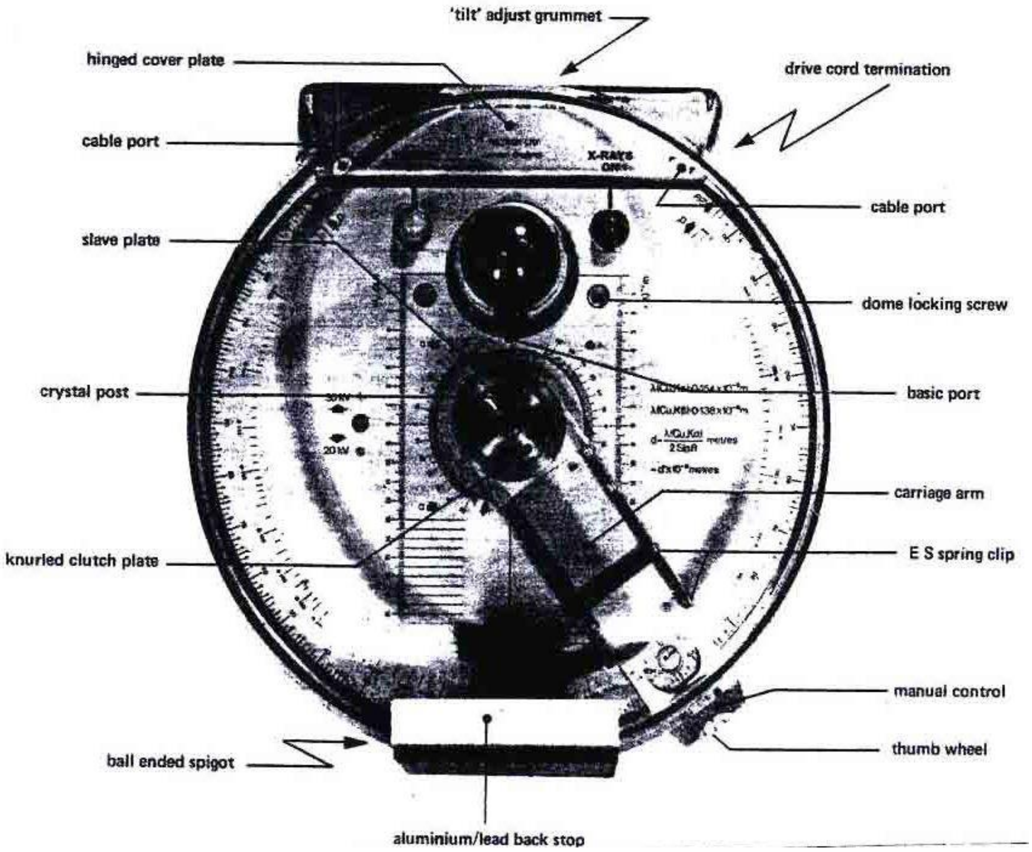
晶體中原子的間距 d 可由其分子量 M 及密度 ρ 計算

如 NaCl　$d = \left(\dfrac{M}{2\rho N}\right)^{1/3} = 2.82\,\overset{0}{A} \qquad N = 6.02\times10^{23}$

典型的繞射分佈如圖(2)，圖中可以看出 Cu 之 K_α 及 K_β 之 1,2,3 級繞射，由 θ 及 d 可算出波長。

注意：晶體極易潮解，不可觸摸、不可對之呼氣。請戴手套、口罩並用鑷子夾取。

實驗結束時，請助教點收晶體。



WAVELENGTH MEASUREMENT: BRAGG METHOD ($1\frac{1}{2}$ HOURS)

Sir Lawrence Bragg presumed that the atoms of a crystal such as Sodium Chloride were arranged in a cubic and regular three-dimensional pattern.

The mass of a molecule of NaCl is M/N kg, where M is the molecular weight (58.46×10^{-3} kg per mode) and N is Avogadro's number (6.02×10^{23} molecules per mode).

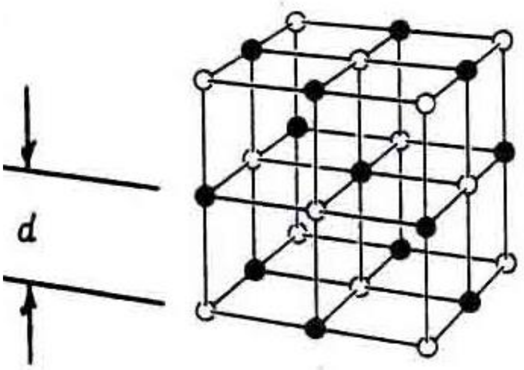
The number of molecules per unit volume is $\rho \Big/ \frac{M}{N}$ molecules per cubic metre, where ρ is the density ($2.16 \times 10^{3} kgm^{-3}$).

Since NaCl is diatomic the number of atoms per unit volume is $\frac{2\rho N}{M}$ atoms per cubic metre.

The distance therefore between adjacent atoms, *d* in the lattice is derived from the equation:

$$d^3 = \frac{1}{2\rho N / M} \quad \text{or} \quad d = \sqrt[3]{M / 2\rho N}$$

and for NaCl, $d = 0.282 nm$.



The first condition for Bragg "reflection" is that the angle of incidence θ equals the reflection – this is as for optical reflection and infers that any detector of the reflected rays must move through an angle 2θ, the 2:1 spectrometer relationship.

The second condition is that reflections from several layers must combine constructively:

$$n\lambda = AB + BC = 2d \sin\theta$$

1. Mount the NaCl crystal, TEL582.004, in the crystal post (see Part 1, para A.9) ensuring that the major face having "flat matt" appearance is in the reflecting position.
2. Locate Primary Beam Collimator 582.001 in the Basic Port with the 1mm slot vertical.
3. Mount Slide Collimator (3mm) 562.016 at E.S.13 and Collimator(1mm) 562.015 at E.S.18.
4. Zero-set and lock the Slave Plate and the Carriage Arm cursor as precisely as possible (see Part1, para A.6).
5. Sight through the collimating slits and observe that the primary beam direction lies in the surface of the crystal.
6. Mount the G/M tube and it's holder at E.S.26.
7. Using a Ratemeter track the Caiirage Arm round from it's minimum setting (about 11^0, 2θ) to maximum setting (about 124^0, 2θ).

   Flot on graph paper the count rate per second at 1^0 (2θ) intervals, allowing time at each reading to estimate the mean of the fluctuations of the needle.

   The Carriage Arm should be indexed to 15^0 (2θ) and the thumb wheel set to zero; when the Scatter Shield is closed, setting from 11^0 to 19^0 can be achieved using only thumb-wheel indications.

   If the ratemeter has a loud speaker, note the random "quantum" nature of the beam of radiation at low count rates.

   Where the count rate appears to peak, plot intervals of only $10'$ arc using the thume-wheel (see Part 1, para A.8); at each peak, measure and record the maximum count rate and the angle 2θ as precisely as possible.

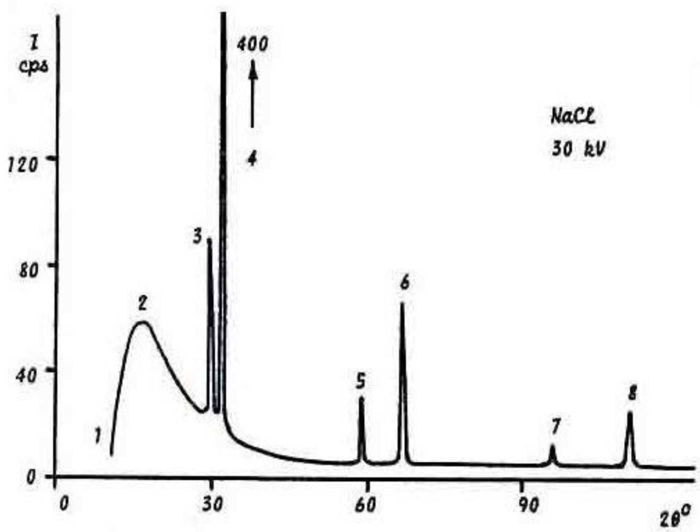
   Observe that the continuous spectra of "white" radiation exhibit peak intensities (feature 2) and intercepts on the 2θ axis (feature 1) which only vary with the voltage setting of the X-ray tube.

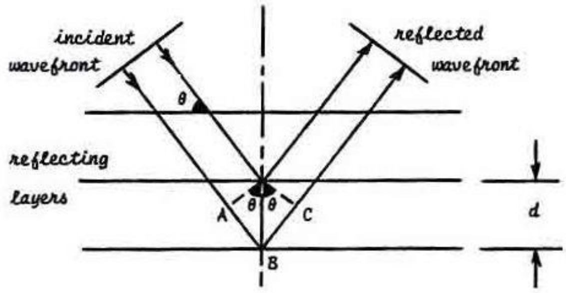
8. The six peaks, features 3 to 8, superimposed on the continuous spectrum do not vary in angle 2θ with voltage setting, but only in amplitude.
9. Tabulate the results from the six superimposed peaks of the graph and calculate λ and n.

Feature	2θ	θ	$\sin\theta$	2d	$n\lambda$	N
3				0.564		
4				0.564		
5				0.564		

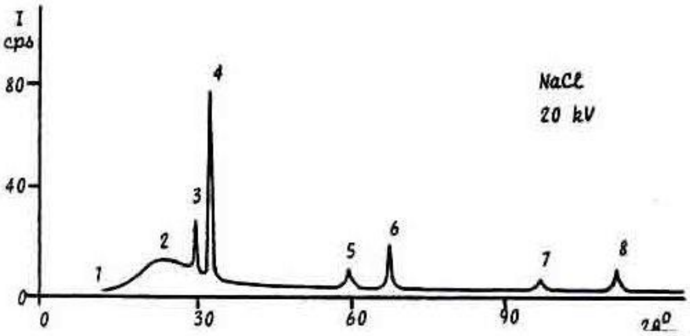
Observe that the sharp peaks are a pair of "emission lines" which re-appear in second and third orders of diffraction.

The more energetic radiation, termed kβ, is successively less intense than the longer wavelength, kα line.





DI4.8 Select 20kV and repeat DI4.7.



Introduction of Equipment

A. EXPERIMENTAL TECHNIQUES

1. **Primary Beam:**

   The X-ray emission from the tube is collimated at the lead glass dome to be a circular beam of 5mm diameter, see Fig.1; this primary beam diverges from the Basic Port to give a useful beam diameter at the Crystal Post of 15mm diameter, at Experimental Station (ES) 13 of 20mm diameter and at Experimental Station (ES) 30 of 38mm diameter.

   Where the primary beam impinges on the lead back-stop it has a diameter of 60mm and a penumbral width of approximately 20mm. With the unit operating at 30kV and 50µA the intensity of radiation in the useful beam at 20cm (ES.28) from the focus is about 2 rads per minute.

2. **Primary Collimators:**

The primary beam can be collimated to a fine circular beam using the 1mm diameter collimator TEL 582.002 and to a ribbon beam using the 1mm Slot collimator, TEL 582.001; each of these collimators is installed by inserting the "O" ring shank into the Basic Port and pushing it home; the "O" ring retains the collimator in position and allows exchange of the collimators even when they become warm.

The collimators should be rotated when they are inserted to ensure that they are securely seated.

The 1mm Slot Collimator can be rotated in position the provide a vertical ribbon of X-rays, a horizontal ribbon or any diametrical ribbon.

3. **Secondary Collimators:**

   There are two secondary collimators, a 1mm Slot Collimator, TEL 562.015 and a 3mm Slot Collimator, TEL 562.016. Each of these collimators is slide mounted and can be positioned in any E.S. on the Spectrometer Arm. Always use a E.S. Spring Clip, see Fig.1, when positioning these collimators (and indeed any of the experimental slides) to ensure that all the slides are centered on the X-ray beam by pressing them against the numbered side of the carriage which acts as a datum.

   The E.S. Spring Clips can be easily repositioned by springing open the toothed jaw on the outside face of the plastics carriage. Six of these E.S. Spring Clips are provided, four on the Spectrometer Arm and two on the Auxiliary Carriage.

4. **Auxiliary Slide Carriage, TEL 582.005:**

   A demountable Auxiliary Slide Carriage is included.

   Using this carriage, Experimental Stations 1 to 4 can be placed in the X-ray beam in a variety of positions. Some of the experiments recommended in this booklet require this carriage to be mounted in two particular modes.

   Mode H (Horizontal)

   The hole in the end face of the Auxiliary Carriage is placed over the Basic Port in the glass dome and then held in that position by one or other of the Primary Collimators. In this mode the axis of the center of each experimental slide is HORIZONRAL and is transcribed by the X-ray beam.

   Note that the Carriage Arm is now restricted to a maximum 2θ angle of 100^0.

   Mode V (Vertical)

   The end face of the Auxiliary Carriage is placed over the minor diameter of the Knurled Clutch Plate screwed over the Crystal Post, see Fig. 1; the crystal mounting jaw and screw should be removed. In this mode the axis of the center of each experimental slide is VERTICAL and co-incident with the pivot axis of the spectrometer table. The X-ray beam now passes through the slides at right angles to the center line axis. The height of the Auxiliary Carriage can be adjusted by screwing the Clutch Plate up or down accordingly.

5. **Knurled Clutch Plate**

   When the Clutch Plate is screwed down it exerts pressure on a spring plate which in turn engages the 2:1 spectrometer drive mechanism. When it is unscrewed it disengages the drive mechanism. This permits the Crystal Post to e manually positioned in any desired orientation with respect to the Carriage Arm.

6. **The 2:1 Spectrometer Mechanism:**

   Open the Scatter Shield and rotate the Carriage Arm until the cursor gives an accurate no-parallax zero reading on the 2θ scale.

Release the drive by unscrewing the Clutch Plate and push the Slave Plate (the inner rotating plate engraved with two datum lines, see Fig. 1) round until the datum lines are accurately opposite the zeros on the θ scale. It may be necessary to "mean out" small zero-reading differences on each side of the θ scale.

Check that the Carriage Arm cursor is still at zero on the 2θ scale and screw in the Clutch Plate to engage the 2:1 drive mechanism.

Now rotate the Carriage Arm through 90^0 (2θ) and note that the crystal Slave Plate moves through 45^0 (θ).

7. Choice of operating side:

Close the Scatter Shield - note that the Shield can be displaced in two directions; always slide the Shield to the same side as that of the Carriage Arm. Note that the gap between the Shield and the spectrometer table, where the Carriage Arm normally freely rotates, is eliminated on one side of the instrument, depending on the direction of placement at the hinge.

To transfer the Carriage Arm to the opposite side, rotate the arm to the smallest angle possible, about 11^0 (2θ); now open the Shield by sliding away from the Carriage Arm and then rotate the arm through the zero axis to the opposite side.

8. Fine and Coarse controls:

The Carriage Arm is terminated outside the Shield with an orange plastic Manual Control; measurements can be made at the cursor to an accuracy of 15minutes of arc.

To achieve fine adjustment of the Carriage Arm, rest the hand on the base flange of the Tel-X-Ometer or on the bench top and using the thumb rotate the knurled aluminum drive wheel protruding from the Manual Control.

To achieve fine measurements line up the cursor exactly on the most convenient 2θ graduation which is central within the region requiring detailed examination; hold the Manual Control rigidly in this position and "slip" the Thumb Wheel against the friction of the drive cord until the zero on the Thumb Wheel scale aligns exactly with the pointer.

Now the Thumb Wheel can be moved $\pm 4^0$ (2θ) about the preset center line by an amount which can be measured at the Thumb Wheel scale to an accuracy of 5 minutes of arc; this amount should be added or subtracted from the selected cursor setting according to the direction of movement of the arm.

9. Mounting of cubic crystals:

Select the LiF crystal (BLUE) from the box of accessories and place one of the short edges onto the step in the Crystal Post; ensure that a long broad face, a (100) cleavage plane, is butting against the chamfered protrusion of the post, see Fig. 2. Screw up the clamp until the crystal is held securely by the rubber jaw.

Ensure that the crystal is vertical and flat on the step.

THE EXPERIMENTAL FACE OF THE CRYSTAL IS THAT FACE WHICH IS COINCIDENT WITH THE CHAMFER ON THE POST.

10. Mounting of Geiger Muller Tube:

Plug the G/M tube MX168 (TEL546) into the Holder, TEL 47.

Place the square flange of the holder in E.S. 26 on the carriage arm without use of an ES Spring Clip, ensuring that the co-axial cable is leading out from the underside and that the end-window of the G/M tube is facing the Crystal Post. It is advisable to leave the plastics guard over the end-window unless very feeble count rates are to be monitored.

The co-axial cable can be trailed out from the Shield beside the Manual Control.

11. **General Facilities:**

    There are two radiation-proof located at each end of the hinge cover plate for introducing large cables, vacuum pipes, etc. into the experimental zone.

    There are four blind holes on the surface of the spectrometer table for the mounting of innovative experiments of the teachers' or pupils' choice; these holes are 4mm diameter, located around the θ Scale and marked A, B, C and D.

B. MORNITORING INSTRUMENTS

During Bragg Diffraction experiments count rates of 800 to 1,000 counts per second are experienced for CuKα reflections from the Lithium Fluoride crystal. Count rates of up to 500 cps are more normal for the other three crystals, NaCl, KCl or RbCl.

Background count rates vary from up to 300 cps at the top of the "whale-back" down to 10 cps at the tail-off.

For crystallography experiments therefore a Ratemeter capable of integrating up to 2000 cps is recommended.

For measurements in the main X-ray beam it should be noted that the normal intensity of radiation will saturate the G/M tube; the MX168 has a dead time of 100 microseconds and hence X-ray tube current must be reduced to about 5 μA to prevent saturation and achieve a count rate of about 7,000 cps. For quantitative experiments in the main beam therefore a Ratemeter capable of integrating up to 7/8000 cps is recommended.

The X-ray tube current should NOT be adjusted without monitoring the current, using the jack-plug provided and an external 100μA or 150μA meter.

NEVER EXCEED 80 MICROAMPS

The polarizing supply for the MX168 G/M tube should be variable from 250 to 450 volts d.c.; the threshold voltage is about 370 volts d.c.

When varying the tube current from 20 to 80μA the EHT will remain constant within 5% of the selected value, 20 or 30kV. Conversely, when varying the EHT from 30 to 20kV the regulation is such that the tube current will remain constant within 5%.

C. Operational Alignment

The Tel-X-Ometer has been carefully aligned during the final stages of factory inspection; but to verify that the pre-set-alignment has not been disturbed during transit it is recommended that the following tests be performed.

1. Place the 1mm Collimator, TEL 582.001 in the Basic Port and the 1mm Collimator Slide, TEL 562. 015 at E.S.30 and ensure that both collimators are vertical.
2. Zero-set and lock the Slave Plate and the Carriage Arm cursor as precisely as possible.
3. Spring a Spindle Clip TEL 567.008 over the chamfered Crystal Post and carefully insert a Glass Fiber TEL 567.004 to be truly vertical, see Fig. 3.
4. With the Scatter Shield open, operate the POWER ON switch and sight through the collimators to view the reflection of the tube filament in the copper target anode.

The Glass Fiber should normally be observed as central in the "viewing column" created by the two collimators, i.e. the direction of the primary beam and it should be possible to obtain an uninterrupted view of the anode past each side of the fiber; the glass dome can be rotated by a small amount if the two dome-locks are unscrewed.

5. Remove the Glass Fiber and the Spindle Clip.

D. EXPERIMENTAL VERIFICATION

Mount the LiF Crystal (Blue) in the Crystal Post.

1. Check that the cursor is at zero reading (2θ) and again sight through the collimators; the direction of the primary beam should lie in the surface of the crystal, as does the axis of rotation.
2. The normal to the reflecting face of the crystal should dissect the angle between the primary beam and the centre line of the Carriage Arm; for Bragg reflection experiments the arm should always be moved to the same side of the crystal as that occupied by the chamfered Crystal Post protrusion.
3. Move Collimator (1mm) 562.015 to E.S.18 and mount Collimator (3mm) 562.016 at E.S.13. Mount the G/M tube and Holder at E.S.26.
   Connect to the Ratemeter and energies the polarizing supply to the G/M tube (400-420 volts d.c.).
4. Check that the voltage selector indicates 30kV.
5. Move the Carriage Arm through at least 15^0 (2θ) in the direction to detect Bragg Reflection.
6. Close and centre the Scatter Shield.
7. Depress the "X-RAYS ON" button; the RED signal lamp should be illuminated.
8. Verify that a strong CuKα reflection (high ratemeter count rate) is evident at a 2θ angle of $45^0 \pm 30'$.
   Record the 2θ angle of the peak reflection.
   IF THE PEAK OF THE REFLECTION LIES OUTSIDE THIS TOLERANCE THEN THE X-RAY TUBE SHOULD BE ADJUSTED FOR "TILT" AS IN PARA.
9. Return the Carriage Arm to about 12^0 (2θ) and open the Shield by displacing it away from the arm.
10. Unscrew the Clutch Plate, rotate the Slave Plate (and thereby the Crystal) through 180^0 and again zero-set the Slave Plate and the Carriage Arm as precisely as possible.
11. Repeat stages D.5 to D.8 and record the 2θ angle of the peak CuKα reflection.
12. The Mean Reading of the reflections recorded at D.8 and D.11 should be $44^0 56' \pm 12'$. WHEN MAKING ACCURATE ANGULAR MEASUREMENTS EXPERIMENTAL ERRORS SHOULD BE MINIMISED BY EMPLOYING THIS TECHNIQUE OF TAKING A MEAN OF THE EQUIVALENT READING AT EACH SIDE OF THE SPECTROMETER TABLE.
13. The Tel-X-Ometer is now commissioned and certified as capable of measuring angular deflections (2θ) to an accuracy of $\pm\frac{1}{2}\%$.

E. "TILT" ADJUSTMENT OF X-RAY TUBE:

Before proceeding with this adjustment carefully repeat both paras C and D to ensure that no alignment errors have been introduced.

1. Remove the upper black rubber grummet located just below the hinge extension.
   The screw-head thus exposed controls the “tilt” of the X-ray tube about a central position.
2. Set the cursor of the Carriage Arm at the exact mean of the reading measured at para. D.8 and the true Bragg angle (2θ) for LiF of $44^0 56^{'}$.
3. Obtain a peak count rate by carefully adjusting ONLY the “tilt-control” without displacing the Carriage Arm.
4. Now seek and record a peak reflection by displacing the Carriage Arm only, without operating the tube tilt-control.
5. Set the cursor at the exact mean of the reading measured at para E.4 and the true Bragg angle $44^0 56^{'}$ (2θ).
6. Iteratively repeat paras E.2, E.3 and E.4 until the cursor peak reading falls within the tolerance $45^0 \pm 30^{'}$.
7. Replace the black rubber grummet and carry out recommendations D.9 to D.12.