A METHOD FOR REMOUNTING A CRYSTAL ALONG A SPECIFIED DIRECTION

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1. Introduction

Once a crystal has been mounted along a crystallographic axis on a Weissenberg goniometer, it is possible, in one setting, to determine the unit cell parameters (Buerger, 1942) and to collect complete three-dimensional intensity data, excepting for the axial reflection corresponding to the rotation axis.

Yet, in some cases, it is necessary or preferable to obtain diffraction data with the crystal mounted along another crystallographic axis. One may like to have the intensity of axial reflections corresponding to the rotaion axis to fix up the space group from systematic extinctions. For example, without the $00l$ reflections it will be difficult to distinguish the space group $P2_12_12_1$ from $P2_12_12$. Intensity data about a second crystallographic axis is useful in reducing the intensities of different layers obtained from the first setting to a common scale—a procedure which is important if a three-dimensional Patterson or electron density synthesis is to be performed. While solving the structure in projection, it may be necessary to obtain diffraction data with the shortest crystallographic axis as the axis of rotation because of the obvious advantage of better resolution in a projection down such an axis.

It is not always that a crystal being investigated is morphologically well developed. Even when it is so, this advantage is lost in the usual practice of grinding the crystal to the form of a cylinder or sphere. It is easy to mount the crystal along the needle axis if the crystal is cylindrical. The mounting of the crystal along second crystallographic axis is usually, by trial and error, a method expensive both of labour and time.

This paper describes an attachment to the Weissenberg goniometer and a simple method by which a crystal, mounted on the goniometer head along any identifiable axis, can be transferred to second goniometer head along any other desired axis. The unit cell parameters and a zero-level Weissenberg photograph are needed for the setting.

2. Principle of the Method

The attachment to the Weissenberg goniometer is merely a second goniometer head G2 (Fig. 1) whose axis is in the plane (called " G2 plane " hereafter) which is perpendicular to the X-ray beam and contains the principal rotation axis of the goniometer. G2 is capable of being bodily rotated about the direction of the X-ray beam.

The crystal is mounted on G1 along any identifiable axis with wax which can be removed easily. From the zero-level Weissenberg photograph it is possible to calculate the angle Φ, through which the goniometer head G1 is to be rotated about the principal rotation axis of the goniometer, to bring the desired axis in the " G2 plane ". G2 has now to be rotated through an angle Ψ about the direction of the X-ray beam so that the axis of G2 coincides with the desired direction. It is shown below how Φ and Ψ can be calculated. At this setting of G2 the crystal is fixed to the glass fibre attached to G2 with " Durofix " or " Araldite ". When the adhesive has set the crystal is detached from G1 by dissolving away the wax.

3. Conventions

The following conventions have been used in this paper. The unit translation vectors are properly chosen to form a right-handed system of co-ordinates (Fig. 2 *a*). The Weissenberg photograph from the crystal in the first setting has been indexed properly in the right-handed system. Figure 2 *b* shows the sequence of axial reflections recorded on the right-handed goniometer. (For details *see* Vaidya and Ramaseshan, 1963, and Manohar and Ramaseshan, 1963.)

The interaxial angles α, β and γ are, by convention, chosen obtuse. A positive sign of Ψ is taken to indicate a clockwise rotation of G2 about the direction of the X-ray beam. In Fig. 1 the angle marked Ψ is actually $-\Psi$.

The crystal is said to be mounted along $\vec{r}$ if $\vec{r}$ points away from the goniometer head.

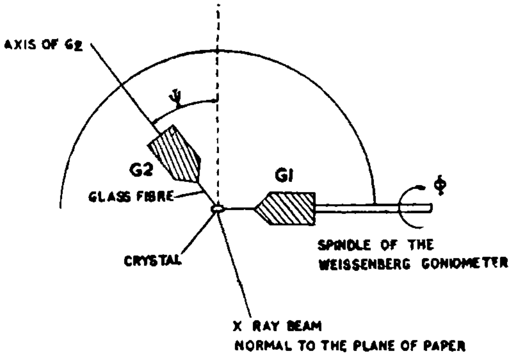


FIG. 1

4. CALCULATION OF Φ AND Ψ

General Case

Let the crystal be mounted on G1 along a rational direction $\vec{r}_0$ defined by

$$\vec{r}_0 = h_0 \vec{a} + k_0 \vec{b} + l_0 \vec{c} \tag{1}$$

where $\vec{a}$, $\vec{b}$ and $\vec{c}$ are unit translation vectors.

It is required to transfer the crystal to G2 along $\vec{r}$ defined by

$$\vec{r} = h\vec{a} + k\vec{b} + l\vec{c} \tag{2}$$

The zero-level Weissenberg photograph taken with crystal mounted along $\vec{r}_0$ will contain central reciprocal lattice rows appearing as straight lines. Let one of such rows be designated by $(h_1 k_1 l_1)$ and the reciprocal vector in this direction be $\vec{\sigma}$ such that

$$\vec{\sigma} = h_1\vec{a}^* + k_1\vec{b}^* + l_1\vec{c}^* \tag{3}$$

where

$$\vec{a}^* = \frac{\lambda\,[\vec{b} \times \vec{c}]}{\vec{a}\,.\,[\vec{b} \times \vec{c}]}$$

and similar expressions for $\vec{b}^*$ and $\vec{c}^*$; λ is wave-length of the radiation employed.

Since $\vec{\sigma}$ appears in zero-level, $\vec{\sigma}.\vec{r}_0 = 0$.

Figure. 2 *c* shows the general appearance of central lattice rows. The direct beam falls at P when the camera is at the extreme end. Let the reading of the graduated drum of the goniometer at this extreme position of the camera be Φ_0. Suppose the row of spots $(h_1k_1l_1)$ intersect the central line at Q (Fig. 2 *c*). Then at the setting

$$\Phi' = (\Phi_0 + \mathrm{PQ} \times \mathrm{C}_2) \tag{3 b}$$

of the drum, $\vec{\sigma}$ is in the G2 plane. Here C_2 is camera constant (2°/mm. for Unicam —S·35).

For this setting relative orientations of $\vec{\sigma}$ and $\vec{r}$ are given in Fig. 3.

Let the angle between $\vec{r}_0$ and $\vec{r}$ be $\triangle$ and that between $\vec{r}_0$ and $\vec{\sigma}$ be ϵ.

Since $\vec{\sigma}$ and AE are parallel, the projections of $\vec{r}$ on $\vec{\sigma}$ and AE are equal. Thus

$$\mathrm{OD} = \mathrm{AB} \tag{4}$$

$$\mathrm{OD} = |\vec{r}| \cos \epsilon \tag{5}$$

$$\begin{aligned} \mathrm{AB} &= \mathrm{AC} \cos \delta \\ &= |\vec{r}| \sin \triangle \cos \delta. \end{aligned} \tag{6}$$

Combining equations (4), (5) and (6) we get,

$$\cos \delta = \frac{\cos \epsilon}{\sin \triangle} \tag{7}$$

The angles ϵ and $\triangle$ can be calculated as follows:

$$\vec{r}.\vec{r}_0 = |\vec{r}|\,|\vec{r}_0| \cos \triangle = [h\vec{a} + k\vec{b} + l\vec{c}].[h_0\vec{a} + k_0\vec{b} + l_0\vec{c}].$$

$$\cos \triangle = \frac{\begin{array}{l}[hh_0\,|\vec{a}|^2 + kk_0\,|\vec{b}|^2 + ll_0\,|\vec{c}|^2 + (k_0l + kl_0)\,|\vec{b}|\,|\vec{c}| \cos \alpha \\ \quad + (h_0l + hl_0)\,|\vec{a}|\,|\vec{c}| \cos \beta + (h_0k + hk_0)\,|\vec{a}|\,|\vec{b}| \cos \gamma]\end{array}}{|\vec{r}|\,|\vec{r}_0|} \tag{8}$$

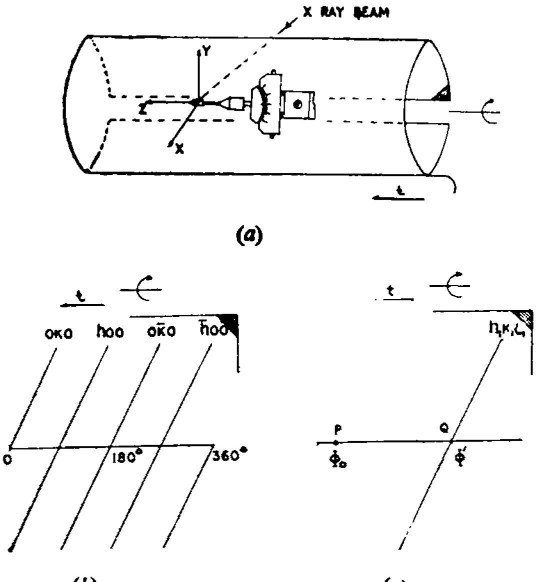
where

$$\begin{aligned} |\vec{r}| = [&h^2\,|\vec{a}|^2 + k^2\,|\vec{b}|^2 + l^2\,|\vec{c}|^2 + 2kl\,|\vec{b}|\,|\vec{c}| \cos \alpha + 2hl\,|\vec{a}|\,|\vec{c}| \cos \beta \\ &+ 2hk\,|\vec{a}|\,|\vec{b}| \cos \gamma]^{\frac{1}{2}} \end{aligned} \tag{9}$$

and

$$|\vec{r}_0| = [h_0^2 |\vec{a}|^2 + k_0^2 |\vec{b}|^2 + l_0^2 |\vec{c}|^2 + 2k_0l_0 |\vec{b}| |\vec{c}| \cos \alpha$$

$$+ 2h_0l_0 |\vec{a}| |\vec{c}| \cos \beta + 2h_0k_0 |\vec{a}| |\vec{b}| \cos \gamma]^{\frac{1}{2}}. \quad (10)$$



(*a*) (*b*) (*c*)

FIG. 2

The angle ϵ is given by

$$\vec{\sigma}.\vec{r} = |\vec{\sigma}| |\vec{r}| \cos \epsilon = [h_1\vec{a}^* + k_1\vec{b}^* + l_1\vec{c}^*].[h\vec{a} + k\vec{b} + l\vec{c}]$$

$$= \lambda [hh_1 + kk_1 + ll_1]$$

$$\cos \epsilon = \frac{\lambda [hh_1 + kk_1 + ll_1]}{|\vec{\sigma}| |\vec{r}|} \quad (11)$$

$|\vec{r}|$ is given by equation (9) and $|\vec{\sigma}|$ is given by

$$|\vec{\sigma}| = [h_1^2 |\vec{a}^*|^2 + k_1^2 |\vec{b}^*|^2 + l_1^2 |\vec{c}^*|^2 + 2k_1l_1 |\vec{b}^*| |\vec{c}^*| \cos \alpha^*$$

$$+ 2h_1l_1 |\vec{a}^*| |\vec{c}^*| \cos \beta^* + 2h_1k_1 |\vec{a}^*| |\vec{b}^*| \cos \gamma^*]^{\frac{1}{2}}. \quad (12)$$

Thus at setting $\Phi = [\Phi' + \delta]$, $\vec{r}$ is in G2 plane, but $\triangle$ as obtained from (8) has two values, $\pm \triangle$.

Equation (7) thus takes the form,

$$\cos \delta = \pm \frac{|\cos \epsilon|}{|\sin \Delta|}. \tag{13}$$

The Equation (13), when solved for δ, has four solutions, $\delta_1 = \pm \delta$ corresponding to $+\Delta$ and $\delta_2 = 180° \pm \delta$ corresponding to $-\Delta$. In general two out of four solutions are correct. The correct solutions can be chosen by considering the geometry of the real and reciprocal cell. This is illustrated by considering some useful particular cases.

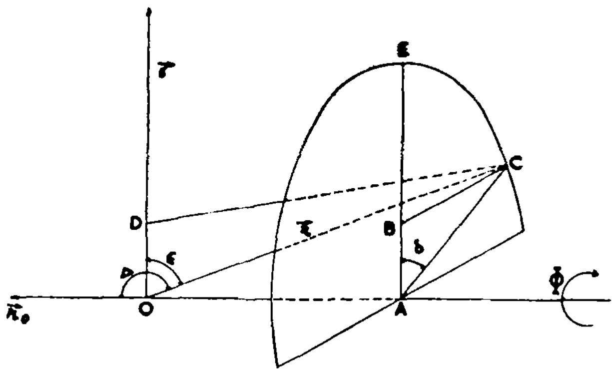


FIG. 3

5. PARTICULAR CASES

1. (*a*) In the case of orthorhombic, tetragonal and cubic crystals, the translation vectors of a real cell coincide, in direction, with the corresponding translation vectors of the reciprocal cell. In an orthorhombic crystal mounted on G1 along *b*-axis, suppose it is desired to transfer it to G2 along the *a*-axis.

Let us choose $\vec{\sigma} \equiv \vec{a}^*$. We have from (8) and (11)

$$\cos \Delta = 0 \quad \text{and} \quad \cos \epsilon = 1.$$

From (13) we have

$$\cos \delta = \pm 1.$$

Therefore,

$$\delta_1 = 0$$
$$\delta_2 = 180°$$

and $\quad \Psi = \Delta - \frac{\pi}{2} = 0 \text{ or } 180°,$

Thus $\Phi_1 = \Phi'$ and $\Psi_1 = 0$ give the setting in which the axis of G2 points along $-\vec{a}$.

The setting $\Phi_2 = [\Phi' + 180°]$ and $\Psi_2 = 180°$ sets the axis of G2 again along $-\vec{a}$.

However, the geometry of the Weissenberg goniometer limits the rotation of G2 to the range $\Psi = +90°$ to $\Psi = -90°$. Therefore, in the above example $\Psi_2 = 180°$ cannot be used. This solution can be conveniently employed to mount the crystal along $+\vec{a}$. At the setting $\Phi_2 = [\Phi' + 180°]$ and $\Psi_2 = 0$ the axis of G2 is along $+\vec{a}$.

In further discussions, if $|\Psi| > 90°$ it will be interpreted to give a mounting along $+\vec{r}$.

(*b*) To illustrate the mounting of the crystal along any direction other than an axial direction, let us consider a cubic crystal mounted along the cube edge $\vec{a}$ and let it be required to transfer the crystal on G2 along [111]. There is no loss of generality in this choice, and the calculations are valid for any direction [*hkl*].

This particular case may be obtained by substituting $k_0 = l_0 = 0$ and $h_0 = 1$ and $h = k = l = 1$ in Equations (1) and (2) respectively. Let us choose $\vec{\sigma} \equiv \vec{a}^*$, so that $h_1 = 1$, $k_1 = l_1 = 0$ in Equation (3).

From Equations (9) and (10) we get,

$$|\vec{r}| = [|\vec{a}|^2 + |\vec{a}|^2 + |\vec{a}|^2]^{\frac{1}{2}} = |\vec{a}|\sqrt{3}$$

$$|\vec{r}_0| = |\vec{a}|$$

$$\cos\triangle = \frac{1}{\sqrt{3}}$$

From Equation (11)

$$\cos\epsilon = \frac{\lambda}{|\vec{a}|\sqrt{3}\,|\vec{a}^*|} = \frac{1}{\sqrt{3}}\,.$$

Thus

$$\cos\delta = \pm\frac{1}{\sqrt{2}}\,.$$

The equation has the following solutions

$$\delta_1 = \pm 45^\circ \quad \text{corresponding to} \quad + |\triangle|$$

and

$$\delta_2 = \pm 135^\circ \quad \text{corresponding to} \quad - |\triangle|.$$

It is clear from Fig. 4 that $\delta_1 = +45^\circ$ brings [111] in the G2 plane. $\Psi_1 = |\triangle| - 90^\circ$ is negative and so anticlockwise. Thus at the setting $\Phi_1 = [\Phi' + 45^\circ]$ and $\Psi_1 = |\triangle| - 90^\circ$ the axis of G2 points in $[\bar{1}\bar{1}\bar{1}]$.

At setting $\Phi_2 = [\Phi' - 135^\circ]$ and $\Psi_2 = 90^\circ - |\triangle|$ the axis of G2 points along [111].

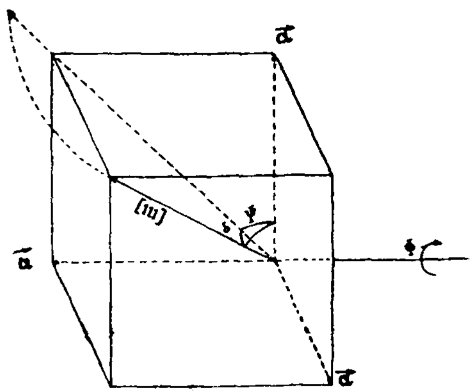


FIG. 4

2. *Monoclinic Case*

(*a*) *Unique axis* (c-*axis*) *mounting*.—Let it be required to transfer the crystal on G2 along *b*-axis (Fig. 5 *a*).

If we choose $\vec{\sigma} \equiv \vec{b}^*$

we have from Equations (1), (2) and (3)

$$\vec{r}_0 = \vec{c}$$

$$\vec{r} = \vec{b}$$

$$\vec{\sigma} = \vec{b}^*$$

$$\cos\Delta = 0, \quad \Delta = \pm 90°$$

$$\cos\epsilon = \frac{\lambda}{|\vec{b}|\,|\vec{b}^*|} = \sin\gamma.$$

Thus

$$\cos\delta = \pm\,|\cos\epsilon|.$$

The four solutions of the equation are $\delta_1 = \pm\,\epsilon$ and $\delta_2 = 180° \pm \epsilon$.

At the setting $\Phi_1 = [\Phi' - \epsilon]$ and $\Psi_1 = 0$ axis of G2 points along $-\vec{b}$ while at setting $\Phi_2 = [\Phi' + (180° - \epsilon)]$ and $\Psi_2 = 0$ axis of G2 points along $\vec{b}$.

It must be mentioned that if the crystal is to be transferred on G2 along $\vec{a}$ and $\vec{a}^*$ is chosen for $\vec{\sigma}$, correct solutions are $\delta_1 = +\,\epsilon$ and $\delta_2 = (180° + \epsilon)$.

(*b*) *Non-unique axis mounting.*—Let the crystal be mounted along $\vec{a}$-axis and let it be required to transfer it on G2 along $\vec{b}$-axis. If we choose $\vec{b}^*$ for $\vec{\sigma}$ we have from Equations (1), (2) and (3),

$$\vec{r}_0 = \vec{a} \qquad \cos\Delta = \cos\gamma$$

$$\vec{r} = \vec{b} \qquad \Delta = \pm\gamma$$

$$\vec{\sigma} = \vec{b}^* \quad \text{and} \quad \cos\epsilon = \frac{\lambda}{|\vec{b}|\,|\vec{b}^*|} = \sin\gamma.$$

Therefore,

$$\cos\delta = \pm\frac{|\cos\epsilon|}{|\sin\Delta|}.$$

The possible solutions are $\delta_1 = \pm\,\delta$ and $\delta_2 = (180° \pm \delta)$.

It is clear from Fig. 5 *b* that the correct solutions are $\delta_1 = -\,\delta$ and $\delta_2 = 180° - \delta$. Thus at the setting $\Phi_1 = [\Phi' - \delta]$ and $\Psi_1 = \gamma - 90°$ axis of G2 points along $-\vec{b}$ while at setting $\Phi_2 = [\Phi' + 180° - \delta]$ and $\Psi_2 = -[\gamma - 90°]$ axis of G2 is along $\vec{b}$.

If the crystal is to be transferred to G2 along unique axis, the situation is same as for an orthorhombic crystal, because $\vec{c}$ is normal to rotation axis and coincides, in direction with $\vec{c}^*$.

c. *Triclinic Case*

Triclinic case is same as when a monoclinic crystal is mounted along non-unique axis and it is desired to transfer the crystal on G2 along non-unique axis.

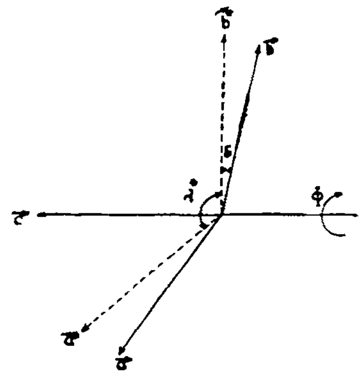


FIG. 5 (*a*)

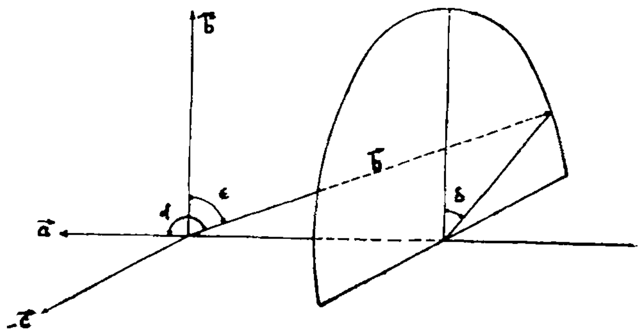


FIG. 5 (*b*)

6. DESCRIPTION OF THE ATTACHMENT

The arrangement shown in Figs. 6 (*a*) and 6 (*b*) is especially designed to suit " Unicam S-35 " goniometer. In any other instrument, all that is necessary is an arrangement that permits G2 to be rotated about the direction of X-ray beam.

The complete assembly can be described in following parts: (*a*) A metal plate has groove on the lower side and can be slipped on the carriage where normally the camera is mounted. (*b*) A rotating arm carries a spindle on which goniometer head G2 is mounted. (*c*) A simple arrangement permits G2 to be raised or lowered by rotating the " adjusting nut " [Fig. 6 (*b*)].

(*d*) Ψ can be read on a graduated scale. (*e*) The crystal can be observod through the microscope when it is being transferred to G2.

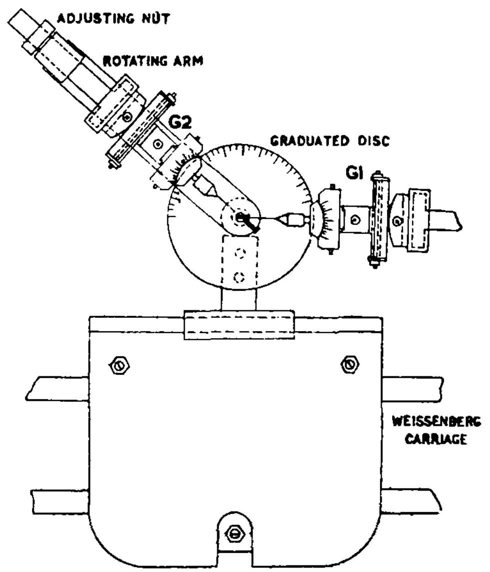


FIG. 6 (*a*)

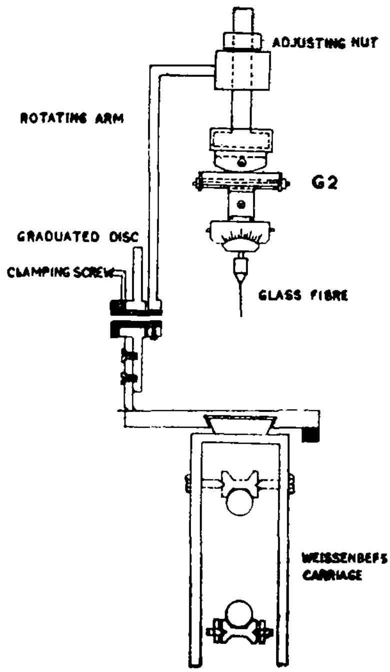


FIG. 6 (*b*)

It must be remarked that the G2 plane need not be normal to the X-ray beam. One may, for example, choose a plane containing principal rotation axis of the goniometer and the X-ray beam as the " G2 plane ". In fact in the first version of the method, the goniometer head G2 was attached to the telemicroscope stand (with telemicroscope removed) so that axis of G2 coincided with direction of the X-ray beam. The goniometer head G1 was rotated through $\bar{\Phi} = [\Phi \pm 90°]$ to bring the required axis in the " G2 plane ". A rotation about " inclination axis " of the Weissenberg goniometer was used to set the axis of G2 along desired direction. This procedure, in spite of its apparent simplicity, had some serious disadvantages. The rotation about " inclination axis " was limited to the range $\pm 40°$. The transfer of the crystal is a delicate operation and consequently it is almost essential to view the crystal through the telemicroscope. It was for these reasons that the more sophisticated attachment was designed.

7. Summary

The paper describes a simple method by which a crystal mounted on a Weissenberg goniometer about an identifiable axis can be remounted on a second goniometer head along any desired direction. The unit cell parameters and a zero-level Weissenberg photograph are needed for the setting.

8. Acknowledgement

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9. References

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