

2. RECIPROCAL SPACE IN CRYSTAL-STRUCTURE DETERMINATION

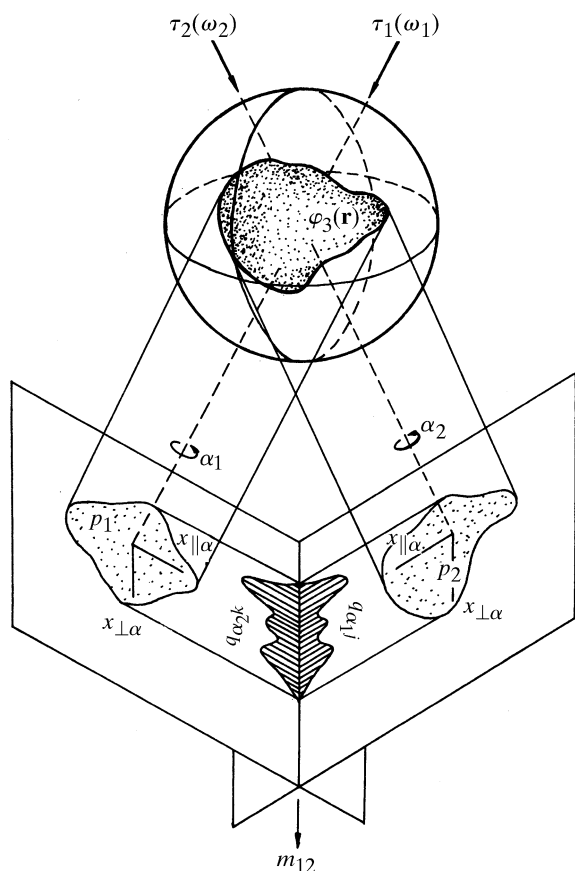


Fig. 2.5.6.6. Relative position of the particle and planes of projection.

accordance with Lemma 1, there exists a one-dimensional projection, common for both  $p_1$  and  $p_2$ , which determines angles  $\alpha_{1j}$  and  $\alpha_{2k}$  along which  $p_1$  and  $p_2$  should be projected for obtaining the identical projection  $q_{12}$  (Fig. 2.5.6.5). Comparing  $q_{1, \alpha_{1j}}$  and  $q_{2, \alpha_{2k}}$  and using the minimizing function

$$D(1,2) = |q_{1, \alpha_{1j}} - q_{2, \alpha_{2k}}|^2 \quad (2.5.6.30)$$

it is possible to find such a common projection  $q_{12}$ . (A similar consideration in Fourier space yields  $Q_{12}$ .)

The mutual spatial orientations of any three non-coplanar projection vectors  $\tau_1, \tau_2, \tau_3$  can be found from three different two-dimensional projections  $p_1, p_2$  and  $p_3$  by comparing the following pairs of projections:  $p_1$  and  $p_2, p_1$  and  $p_3$ , and  $p_2$  and

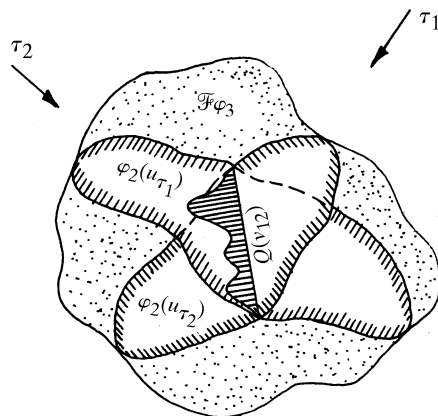


Fig. 2.5.6.7. Section of a three-dimensional Fourier transform of the density of the particles, corresponding to plane projections of this density.

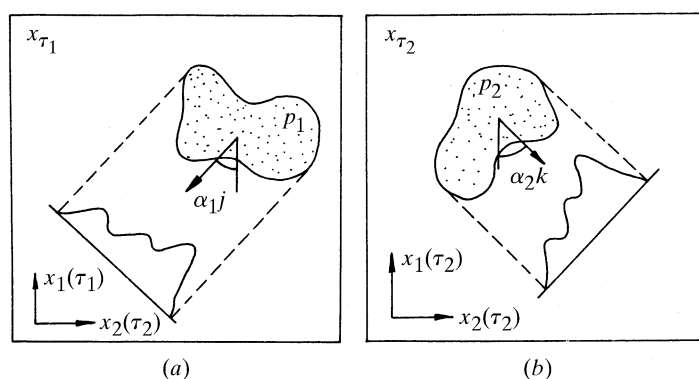


Fig. 2.5.6.8. Plane projections of a three-dimensional body. The systems of coordinates in planes (a) and (b) are chosen independently of one another.

$p_3$ , and by determining the corresponding  $q_{12}, q_{13}$  and  $q_{23}$ . The determination of angles  $\omega_1, \omega_2$  and  $\omega_3$  reduces to the construction of a trihedral angle formed by planes  $h_{12}, h_{13}$  and  $h_{23}$ . Then the projections  $p_i(\omega_i)$  with the known  $\omega_i$  ( $i = 1, 2, 3$ ) can be complemented with other projections ( $i = 4, 5, \dots$ ) and the corresponding values of  $\omega$  can be determined. Having a sufficient number of projections and knowing the orientations  $\omega_i$ , it is possible to carry out the 3D reconstruction of the object [see (2.5.6.27); Orlov, 1975; Vainshtein & Goncharov, 1986a; Goncharov *et al.*, 1987].

2.5.7. Direct phase determination in electron crystallography (D. L. DORSET)

2.5.7.1. Problems with 'traditional' phasing techniques

The concept of using experimental electron-diffraction intensities for quantitative crystal structure analyses has already been presented in Section 2.5.4. Another aspect of quantitative structure analysis, employing high-resolution images, has been presented in Sections 2.5.5 and 2.5.6. That is to say, electron micrographs can be regarded as an independent source of crystallographic phases.

Before direct methods (Chapter 2.2) were developed as the standard technique for structure determination in small-molecule X-ray crystallography, there were two principal approaches to solving the crystallographic phase problem. First, 'trial and error' was used, finding some means to construct a reasonable model for the crystal structure *a priori*, e.g. by matching symmetry properties shared by the point group of the molecule or atomic cluster and the unit-cell space group. Secondly, the autocorrelation function of the crystal, known as the Patterson function (Chapter 2.3), was calculated (by the direct Fourier transform of the available intensity data) to locate salient interatomic vectors within the unit cell.

The same techniques had been used for electron-diffraction structure analysis (nowadays known as *electron crystallography*). In fact, advocacy of the first method persists. Because of the perturbations of diffracted intensities by multiple-beam dynamical scattering (Chapter 5.2), it has often been suggested that trial and error be used to construct the scattering model for the unit crystal in order to test its convergence to observed data after simulation of the scattering events through the crystal. This indirect approach assumes that no information about the crystal structure can be obtained directly from observed intensity data. Under more favourable scattering conditions nearer to the kinematical approximation, *i.e.* for experimental data from thin crystals made up of light atoms, trial and error modelling, simultaneously minimizing an atom-atom nonbonded potential function with the crystal-