experiments at different concentrations to extrapolate to zero concentration (elimination of interparticle interferences).

If the scattering efficiency of the sample is low (low contrast, small particles), it may be necessary to measure the outer part of the scattering function with a larger entrance slit and we will have to merge different parts of the scattering function. The intensity of the instrument (primary beam) should be checked before each measurement. This allows correction (normalization) for instabilities.

It is therefore necessary to have a so-called primary data-handling routine that performs all these preliminary steps like averaging, subtraction, normalization, overlapping, concentration extrapolation, and graphical representation on a graphics terminal or plotter. In addition, it is helpful to have the possibility of calculating the Guinier radius, Porod extrapolation [equations (2.6.1.24)], invariant, *etc*. from the raw data.

When all these preliminary steps have been performed, we have a smeared particle-scattering function $I_{\rm exp}(h)$ with a certain statistical accuracy. From this data set, we want to compute $I(h)$ and $p(r)$ [or $D(R)$] and all our particle parameters. In order to do this, we have to *smooth* and *desmear* our function $I_{\rm exp}(h)$. The smoothing operation is an absolute necessity because the desmearing process is comparable to a differentiation that is impossible for noisy data. Finally, we have to perform a Fourier transform (or other similar transformation) to invert equations (2.6.1.9) or (2.6.1.54), (2.6.1.55). Before we can discuss the desmearing process (collimation error correction) we have to describe the smearing process.

2.6.1.6.2. *Instrumental broadening – smearing*

These effects can be separated into three components: the two-dimensional geometrical effects and the wavelength effect. The geometrical effects can be separated into a slit-length (or slit-height) effect and a slit-width effect. The slit length is perpendicular to the direction of increasing scattering angle; the corresponding weighting function is usually called $P(t)$. The slit width is measured in the direction of increasing scattering angles and the weighting function is called $Q(x)$. If there is a wavelength distribution, we call the weighting function $W(\lambda')$ where $\lambda' = \lambda/\lambda_0$ and λ_0 is the reference wavelength used in equation (2.6.1.2). When a conventional X-ray source is used, it is sufficient in most cases to correct only for the $K\beta$ contribution. Instead of the weighting function $W(\lambda')$ one only needs the ratio between $K\beta$ and $K\alpha$ radiation, which has to be determined experimentally (Zipper, 1969). One or more smearing effects may be negligible, depending on the experimental situation.

Each effect can be described separately by an integral equation (Glatter, 1982*a*). The combined formula reads

$$\bar{I}_{\rm exp}(h) = 2 \int_{-\infty}^{\infty} \int_{0}^{\infty} \int_{0}^{\infty} Q(x)P(t)W(\lambda') \times I\left(\frac{[(m-x)^2 + t^2]^{1/2}}{\lambda'}\right) {\rm d}\lambda'\ {\rm d}t\ {\rm d}x. \qquad (2.6.1.56)$$

This threefold integral equation cannot be solved analytically. Numerical methods must be used for its solution.

2.6.1.6.3. *Smoothing, desmearing, and Fourier transformation*

There are many methods published that offer a solution for this problem. Most are referenced and some are reviewed in the textbooks (Glatter, 1982*a*; Feigin & Svergun, 1987). The *indirect transformation method* in its original version (Glatter, 1977*a*,*b*, 1980*a*,*b*) or in modifications for special applications (Moore, 1980; Feigin & Svergun, 1987) is a well established method used in the majority of laboratories for different applications. This procedure solves the problems of smoothing, desmearing, and Fourier transformation [inversion of equations (2.6.1.9) or (2.6.1.54), (2.6.1.55)] in one step. A short description of this technique is given in the following.

Indirect transformation methods. The *indirect transformation method* combines the following demands: single-step procedure, optimized general-function system, weighted least-squares approximation, minimization of termination effect, error propagation, and consideration of the physical smoothing condition given by the maximum intraparticle distance. This smoothing condition requires an estimate $D_{\rm max}$ as an upper limit for the largest particle dimension:

$$D_{\rm max} \geq D. \qquad (2.6.1.57)$$

For the following, it is not necessary for $D_{\rm max}$ to be a perfect estimate, but it must not be smaller than D.

As $p(r) = 0$ for $r \geq D_{\rm max}$, we can use a function system for the representation of $p(r)$ that is defined only in the subspace $0 \leq r \leq D_{\rm max}$. A linear combination

$$p_A(r) = \sum_{\nu=1}^{N} c_\nu \varphi_\nu(r) \qquad (2.6.1.58)$$

is used as an approximation to the PDDF. Let N be the number of functions and c_ν be the unknowns. The functions $\varphi_\nu(r)$ are chosen as cubic B splines (Greville, 1969; Schelten & Hossfeld, 1971) as they represent smooth curves with a minimum second derivative.

Now we take advantage of two facts. The first is that we know precisely how to calculate a smeared scattering function $\bar{I}(h)$ from $I(h)$ [equation (2.6.1.56)] and how $p(r)$ or $D(R)$ is transformed into $I(h)$ [equations (2.6.1.9) or (2.6.1.54), (2.6.1.55)], but we do not know the inverse transformations. The second fact is that all these transformations are linear, *i.e*. they can be applied to all terms in a sum like that in equation (2.6.1.58) separately. So it is easy to start with our approximation in real space [equation (2.6.1.58)] taking into account the *a priori* information $D_{\rm max}$. The approximation $I_A(h)$ to the ideal (unsmeared) scattering function can be written as

$$I_A(h) = \sum_{\nu=1}^{N} c_\nu \Psi_\nu(h), \qquad (2.6.1.59)$$

where the functions $\Psi_\nu(h)$ are calculated from $\varphi_\nu(r)$ by the transformations (2.6.1.9) or (2.6.1.54), (2.6.1.55), the coefficients c_ν remain unknown. The final fit in the smeared, experimental space is given by a similar series

$$\bar{I}_A(h) = \sum_{\nu=1}^{N} c_\nu \chi_\nu(h), \qquad (2.6.1.60)$$

where the $\chi_\nu(h)$ are functions calculated from $\psi_\nu(h)$ by the transform (2.6.1.56). Equations (2.6.1.58), (2.6.1.59), and (2.6.1.60) are similar because of the linearity of the transforms. We see that the functions $\chi_\nu(h)$ are calculated from $\varphi_\nu(r)$ in the same way as the data $\bar{I}_{\rm exp}(h)$ were produced by the experiment from $p(r)$. Now we can minimize the expression

$$L = \sum_{k=1}^{M} [\bar{I}_{\rm exp}(h_k) - \bar{I}_A(h_k)]^2 / \sigma^2(h_k), \qquad (2.6.1.61)$$

where M is the number of experimental points. Such *least-squares problems* are in most cases *ill conditioned, i.e*. additional stabilization routines are necessary to find the best

references