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Discussion of error in lattice-parameter measurements.

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Any discussion of errors assumes that the systematic errors can be handled independently of the random errors. The two types of error are fundamentally distinct; the possibilities for their correction are completely different (Gauss, 1821).

The *random errors*, which appear as irregular deviations of the observations from each other, can never be completely suppressed, but they can be satisfactorily calculated by means of an averaging method if there is a sufficient number of observations. From all observed values, a, the mean $\bar{a}$ is derived. Usually the root-mean-square error is used as a measure of the random errors $\pm m$ ('standard deviation', 'mittlerer quadratischer Fehler'). The influences of separate errors add quadratically (law of the propagation of errors). In this averaging procedure, based on the Gaussian least-squares method, the systematic errors are not considered, a fact frequently overlooked in the literature.

The *systematic errors* are additional unidirectional deviations of the observations from the true value, and add linearly. In contrast to the random errors, they can be eliminated in principle, though they are more likely to remain undetected. Their elimination or reduction depends only on the test procedure and evaluation of the experiments, but *not* on a high number of observations. The remaining part Δa of the systematic errors is unknown, but can be detected by independent comparison measurements, as discussed below.

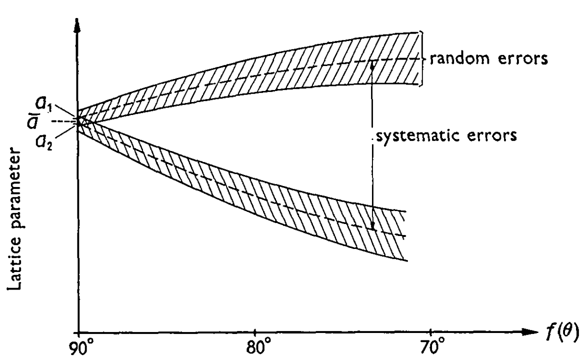

Fig. 1. Random and systematic errors in the extrapolation method (schematic); $\bar{a}$ is the mean value of two exposures with the same measuring method.

For the precise determination of lattice parameters the precision method of Straumanis has led the way (Straumanis & Ieviņš, 1940). By a refined experimental technique the systematic errors could be vastly reduced. But it has become evident that some residual portion Δa of the systematic errors still remains, in spite of careful procedure and evaluation of the experiments. This is to be seen from the fact that for different exposures the positions and the slopes of the extrapolation curves differ slightly (Fig. 1). Subjective errors of observation in measuring line separations are a serious hindrance. This refers to random deviations with the same observer, but especially to the systematic deviations of several observers compared with each other. The difference between the two values of the lattice parameter calculated from the two components of the $K\alpha$ doublet can give an indication of the amount of the observation errors; these cannot, however, be separated with sufficient certainty from the errors caused by the apparatus. The apparatus errors do not all have the same angular dependence; in general, moreover, they appear to an extent that alters from exposure to exposure and is mostly unknown (Parrish & Wilson, 1959; Weyerer, 1957).

The aim of the author's measurements (Weyerer, 1956) was to fix the extrapolation curve in the back-reflexion region as exactly as possible. This was done by multiple irradiation of the same film by two or three X-ray tubes with different target materials.* For measuring the lines a dial-gauge measuring device operating by the coincidence method has proved good.

Though the extrapolated values obtained with these improvements are relatively accurate, there is no guarantee that all systematic errors are really eliminated. That can be proved only by comparing the results of several methods independent of each other (Debye-Scherrer method; back-reflexion methods; focusing methods in cylindrical cameras; diffractometer method) (Weyerer, 1956), all carried out with the same care and experience.

* In accordance with theory, refraction is much less important for powder specimens than for single crystals.

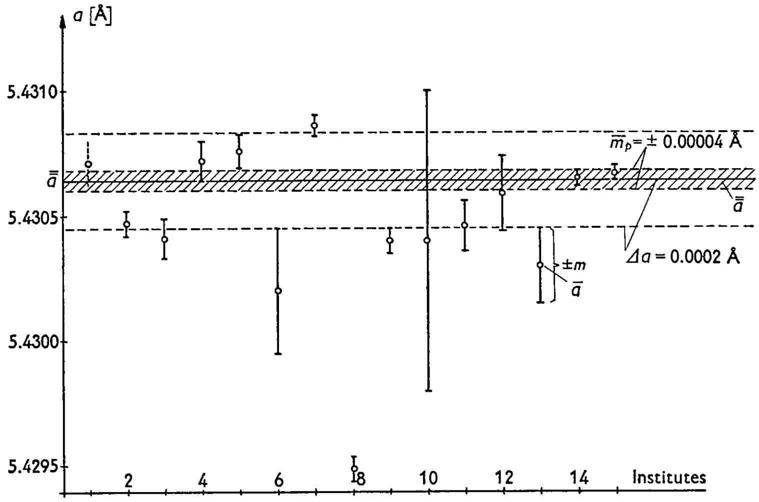

Fig. 2. IUCr comparison measurements on Si (schematic). $\bar{\bar{a}} = 5.43064$ Å. For further explanation see text.
Key to participating institutes;

(1) The Netherlands	(6) U.S.A.	(11) U.S.A.
(2) England	(7) U.S.S.R.	(12) Canada
(3) U.S.A.	(8) Latvia	(13) France
(4) England	(9) U.S.S.R.	(14) Germany
(5) U.S.A.	(10) Spain	(15) Australia

The measurements of the Physikalisch-Technische Bundesanstalt made for the IUCr programme on silicon powder showed differences between the averages ($\bar{a}$) obtained by the three film methods amounting to $\Delta a = \pm 0{\cdot}000\ 04$ Å (residual portion of the systematic error). An arithmetic mean among these methods furnished the final mean $\bar{a}_f = 5{\cdot}430\ 65$ Å, while the random errors could be roughly estimated from the aberrations of the individual lattice-parameter values from their extrapolation curve to be $m = \pm 0{\cdot}000\ 02$ Å.

In international comparison measurements, with several institutes participating, the demand for about an equally high accuracy cannot be fulfilled. The reliability of the results might also depend on the fact that not only one measuring method preponderates. However, each institute can be expected to provide, in addition to other information, a report on the kind and amount of its experimental errors. It is recommended that the final result be reported with a twofold specification of errors in which, besides the measure for the random errors, $\pm m$, a bracket appears containing the assumed residual portion Δa of the systematic error (Weyerer, 1956*a*):

$$\bar{a}_f = \bar{a}\,.\,\{1 \pm (\Delta a/\bar{a})\} \pm m\ .$$

The necessity of this twofold specification of error follows at once from the recognition of existence of the two independent types of error. It should also be retained even if in special cases the systematic errors disappear ($\Delta a = 0$).

From the second report of the IUCr Commission (Parrish, 1960) it is evident that, besides the random errors, there were also systematic errors (Fig. 2). Otherwise the root-mean-square error of all institute values would be the same whether it is calculated with the help of weighting factors $p = 1/m^2$

$$\bar{\bar{m}}_p = \{\Sigma p_i (\bar{a}_i - \bar{\bar{a}})^2 / \Sigma p_i\,.\,(n-1)\}^{\frac{1}{2}} = \pm 0{\cdot}000\ \ 04\ \ \text{Å}$$

Table 1. Error classification

Applied to the error possibilities on lattice-parameter determination

(1) **Random errors** (unavoidable irregular deviations)

- (*a*) *Subjective errors* (random errors in reading and operating)
  - Errors in reading and operating
  - Errors in location (lines, profiles)
- (*b*) *Apparatus errors* (fluctuation of instrument indications, random apparatus defects)
  - Unforeseen changes (adjustment, temperature, electronics, position of film and slits, angular measurement, counter movement, voltage, output of X-ray tube and valves)
  - Counting statistics, film grain
  - Instruments (reading, registration)
- (*c*) *Errors of measuring procedure* (object variations and random influences of surroundings, evaluation errors)
  - Specimen material (preparation, condition, impurities)
  - Inefficiencies of evaluation methods (analytic, graphical)

(2) **Systematic errors** (corrigible in principle, unidirectional deviations)

- (*a*) *Subjective errors* ('personal equation' of observers in reading and operating)
  - Line measurements (line curvature and profile; different position of centre of gravity and maximum; line spottiness)
  - Displacement of neighbouring lines (overlapping profiles; Eberhardt effect)
- (*b*) *Apparatus errors* (wear and ageing of apparatus and instruments, influence of construction and arrangement)
  - Film shrinkage (uniform, non-uniform)
  - Eccentricity of specimen; film radius
  - Focusing circle (specimen, slits, apparatus)

Table 1 (*cont.*)

Equator position, inclination of incident beam
Beam divergence (axial, equatorial); specimen height (interference-cone overlapping)
Angle measurements; counter movement; pulse registration
Beam absorption or transparency of the specimen
Temperature of the specimen
Refraction in the specimen

(*c*) *Errors of measuring procedure* (approximations in methods of measurement and evaluation; errors of calibration; standard comparison)

Incorrect scale (measurement, evaluation)
Angle functions in extrapolation methods
Correction for refraction (dependent on condition of crystal)
Wavelength uncertainty; asymmetry of emission lines
Absolute determination of the X unit

('exterior error') or whether it is obtained without regard to the squares of the deviation as 'interior error'

$$\tilde{m} = \{\Sigma 1/p_i\}^{\frac{1}{2}} = \pm 0{\cdot}000\ 02\ \text{Å};\quad 1/\tilde{m}^2 = \Sigma 1/m_i^2\ .$$

m is the uncertainty of measurement of the final result of each institute calculated on the assumption that m contains only random errors. $\tilde{m}$ turns out smaller than $\bar{m}_p$, and so, in accordance with error theory, it shows the existence of some systematic errors. To estimate their amount, Δa, in the IUCr total result the ordinary root-mean-square error

$$\Delta a \approx \bar{m} = \{\Sigma(\bar{a}_i - \bar{\bar{a}})^2/(n-1)\}^{\frac{1}{2}}$$

can be applied. About two-thirds of all the final results $\bar{a}$ reported by the institutes lie within the limits $\Delta a = \pm 0{\cdot}0002$ Å. This estimate might represent a reasonable criterion for the residual portions of the systematic errors. In it the differences between the institute values are formally treated as random deviations. This is a valid procedure in this case, because the number n of the participating institutes is not too small, and further because their final results are distributed rather regularly around the total result $\bar{\bar{a}}$ of the IUCr. Compared with this residual portion Δa of the mean systematic errors, the random errors of the total result, $\bar{m}_p = 0{\cdot}000\ 04$ Å, are not important.

The final result $\bar{\bar{a}} = 5{\cdot}430\ 64$ Å is obtained from an arithmetic mean with weighting factors p; the results are different depending on whether weighting factors are introduced,

$$\bar{\bar{a}}_p = \Sigma p_i . \bar{a}_i / \Sigma p_i = 5{\cdot}430\ 64\ \text{Å}\ ,$$

or not

$$\bar{\bar{a}}_0 = \Sigma \bar{a}_i / n = 5{\cdot}430\ 54\ \text{Å}\ .$$

Thus as total result for the lattice parameter of the silicon, on the conditions agreed, there results

$$a_{\text{Si}} = (5{\cdot}430\ 64\{1 \pm 3{\cdot}7 \times 10^{-5}\} \pm 0{\cdot}000\ 04)\ \text{Å}\ ,$$

where the brackets contain the estimated residual portion of the mean systematic error, or briefly

$$a_{\text{Si}} = (5{\cdot}430\ 6_4 \pm 0{\cdot}0002)\ \text{Å}\ .$$

Attention may once more be drawn to the—in some respects dubious—assumption underlying this error discussion, namely that the reported uncertainties m contain essentially only the random errors of the institutes. Otherwise, the discussion of error becomes still more difficult or even impossible. This shows the necessity of a precise and detailed specification of the experimental uncertainties of each institute and the advantage of knowing which of the systematic errors were treated in detail in the error elimination. Lastly, an attempt at classifying the many possibilities of errors arising is given in Table 1.

Perhaps the available IUCr comparison measurements cannot yet, strictly, be regarded as ultimate; but in any case they are a very valuable basis for further cooperation.

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The ratio method for absolute measurements of lattice parameters with cylindrical cameras.

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Introduction

The principle of the ratio method for cubic lattices consists in using two diffraction lines for determining the individual values of the lattice parameter. For two-parameter lattices three or four diffraction lines are to be taken. In this way a knowledge of the distance specimen-film or of the camera radius is not needed.

For cubic lattices the method was described in various forms by several authors (Wever & Möller, 1933; Rovinskij, 1940; Černohorský, 1952; Becherer, Brümmer & Ifland, 1955; Rovinskij & Kostiukova, 1958). For two-parameter lattices the method was described also (Matějka, 1956). However, only a flat camera or a cone camera (Kochanovská, 1943) was used. The use of cylindrical cameras has been described recently (Černohorský, 1959*a*).

The present paper shows how to determine *a priori*