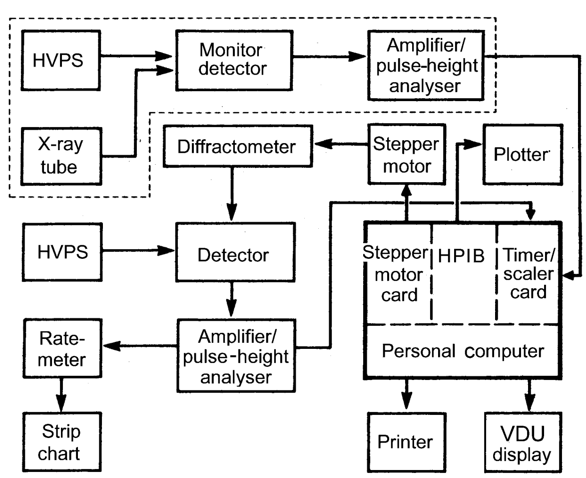
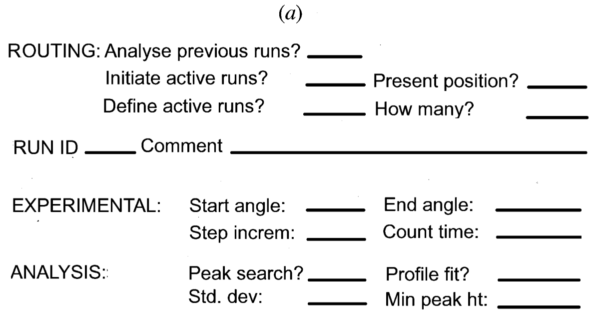
continuous scanning with read-out on the fly, or slewing to selected angles to read particular points. Step scanning is the method most frequently used. It is essential that absolute registration and step tracking be reliably maintained for all experimental conditions.

The step size or angular increment $\Delta 2\theta$ and count time t at each step, and the beginning and ending angles are selectable. For a given total time available for the experiment, it usually makes no difference in the counting statistical accuracy if a combination of small or large $\Delta 2\theta$ and t (within reasonable limits) is used. A minimal number of steps of the order of $\Delta 2\theta \simeq 0.1$ to 0.2 FWHM is required for profile fitting isolated peaks. It is clear that the greater the number of steps, the better the definition of the profile shape. The step size becomes important when using profile fitting to resolve patterns containing overlapped reflections and to detect closely spaced overlaps from the width and small changes in slopes of the profiles. A preliminary fast run to determine the nature of the pattern may be made to select the best run conditions for the final pattern. Will *et al.* (1988) recorded a quartz pattern with 1.28 Å synchrotron X-rays and 0.01° steps to test the step-size role. The profile fitting was done using all points and repeated with the omission of every second, third, and fourth point corresponding to $\Delta 2\theta = 0.02$, 0.03 and 0.04°. The R(Bragg) values were virtually the same (except for 0.04° where it increased), indicating the experimental time could have been reduced by a factor of three with little loss of precision; see also Hill & Madsen (1984). Patterns with more overlapping would require smaller steps. Ideally, the steps could be larger in the background but this also requires a prior knowledge of the pattern and special programming.



(*a*)



(*b*)

Fig. 2.3.3.5. (*a*) Block diagram of typical computer-controlled diffractometer and electronic circuits. The monitor circuit enclosed by the dashed line is optional. HPIB is the interface bus. (*b*) A full-screen menu with some typical entries.

A typical VDU screen menu for diffractometer-operation control is shown in Fig. 2.3.3.5(*b*). A number of runs can be defined with the same or different experimental parameters to run consecutively. The run log number, date, and time are usually automatically entered and together with the comment and parameters are carried forward and recorded on the print-outs and graphics to make certain the runs are completely identified. The menu is designed to prompt the operator to enter all the required information before a run can be started. Error messages appear if omissions or entry mistakes are made. There are, of course, many variations to the one shown.

2.3.3.6. *Counting statistics*

X-ray quanta arrive at the detector at random and varying rates and hence the rules of statistics govern the accuracy of the intensity measurements. The general problems in achieving maximum accuracy in minimum time and in assessing the accuracy are described in books on mathematical statistics. Chapter 7.5 reviews the pertinent theory; see also Wilson (1980). In this section, only the fixed-time method is described because the fixed-count method takes too long for most practical applications.

Let $\bar{N}$ be the average of N, the number of counts in a given time t, over a very large number of determinations. The spread is given by a Poisson probability distribution (if $\bar{N}$ is large) with standard deviation

$$\sigma = \bar{N}^{1/2}. \qquad (2.3.3.7)$$

Any individual determination of N or the corresponding counting rate n $(= N/t)$ will be subject to a proportionate error ε which is also a function of the confidence level, *i.e.* the probability that the result deviates less than a certain percentage from the true value. If Q is the constant determined by the confidence level, then

$$\varepsilon = Q/N^{1/2}, \qquad (2.3.3.8)$$

where $Q = 0.67$ for the probable relative error ε_{50} (50% confidence level) and $Q = 1.64$ and 2.58 for the 90 and 99% confidence levels (ε_{90}, ε_{99}), respectively. For a 1% error, $N = 4500$, 27 000, 67 000 for ε_{50}, ε_{90}, ε_{99}, respectively. Fig. 2.3.3.6 shows various percentage errors as a function of N for several confidence levels.

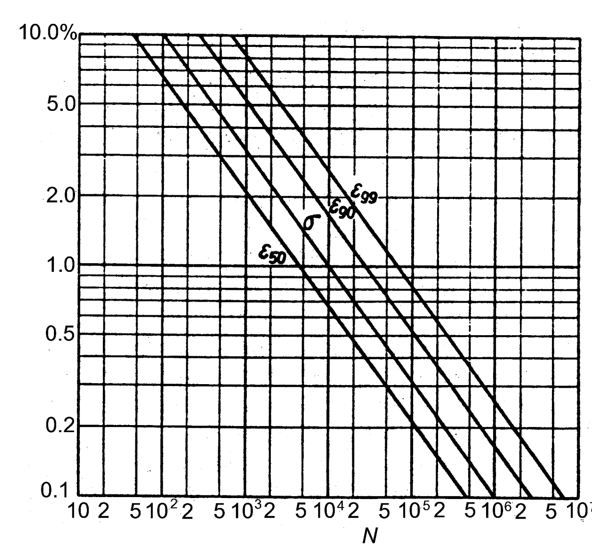


Fig. 2.3.3.6. Percentage error as a function of the total number of counts N for several confidence levels.