In an ideal world where instruments are perfect, the analyst would not need to consider the issues of calibration. However, the realities of economics, the use and hence wear and tear of instruments, occasional design shortcomings and the fact that these instruments are still often at the forefront of their technologies means that the user must be aware of how to calibrate their instrument in order to obtain valid data. Fortunately, helpful activity was started in 1993 in ISO (International Standards Organisation) with a Technical Committee, TC 201 on Surface Chemical Analysis, in order to document, inter alia, calibration procedures to help analysts. Where standards have been completed [1-6], the instructions are well documented. In other cases, the user may need to think through the issues with the guidance given here and in the cited literature. The chapter details these ISO standards, their scientific background and other general issues.

For laboratories operating a quality system, it will be expected that relevant calibrations are made using national or international standards. ISO 17025, General requirements for the competence of testing and calibration laboratories, requires the use of ISO standards where relevant, and the involvement in interlaboratory studies to demonstrate proficiency.

For AES and for XPS, many of the calibration issues are similar since they involve the spectrometer rather than the excitation source. Issues concerning the sources are also covered. The major issues for spectrometers are to ensure that the energy and intensity scales measure correctly. The energy scales need to be correct to an accuracy around 0.2 eV to identify chemical states correctly and to an accuracy around 3 eV for elemental analysis. These values and uncertainties, both here and elsewhere in this chapter unless otherwise stated, are the estimated uncertainties for 95% confidence limits. The intensity scales are not always calibrated but, as a minimum, should be tested for their consistency with the performance they were designed to deliver. Many of the methods used for data reduction claim not to require an intensity calibration although it is difficult to see how this can be true if any theoretical cross sections or processing are used in the data reduction.