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Chemical analysis—Interlaboratory test programs—For determining precision of analytical method(s)—Guide to the planning and conduct

This Australian standard was prepared by a subcommittee of the Association's Committee CH/23, General Methods of Chemical Analysis. It was approved on behalf of the Council of the Standards Association of Australia on 19 April 1986 and published on 4 August 1986.

The following interests are represented on Committee CH/23:

Australian Government Analytical Laboratories
Australian Institute of Food Science and Technology
Australian Mineral Development Laboratories
Bureau of Steel Manufacturers of Australia
Commonwealth Scientific and Industrial Research Organization
Confederation of Australian Industry
National Association of Testing Authorities, Australia
National Health and Medical Research Council
Railways of Australia Committee
Royal Australian Chemical Institute
University of Sydney

The following interests also assisted in the production of this standard:

Kraft Foods Limited
Rural Water Commission of Victoria
SGS Australia Proprietary Limited
State Electricity Commission of Victoria
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PREFACE

This standard was prepared by the Association's Committee on General Methods of Chemical Analysis under the direction of the Chemical Standards Board. It has been prepared to assist persons and organizations involved in the development and testing/validation of methods of chemical analysis.

The standard provides guidance in choosing test materials, laboratories and test procedures to select and validate methods of chemical analysis and includes the subsequent determination of the precision of the test methods. It is based on ISO 5725—Precision of Test Methods—Determination of Repeatability and Reproducibility by Interlaboratory Tests, but has been expanded to include procedures which assist in the validation of test methods, and modified to aid its clarification and application.

It is suggested that the procedures described herein may, with simple but appropriate modification, be applied to many areas of physical testing also, e.g. viscosity determinations, fracture testing, density determinations.

It should be noted that proposals for expanding this standard, particulary with respect to including more details of data evaluation, have already been received by the committee and will be considered for inclusion in the second edition. It was felt that the proposals required far more deliberation and discussion than was considered acceptable for further delaying publication of this workable and much needed standard.

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for

CHEMICAL ANALYSIS—INTERLABORATORY TEST PROGRAMS—FOR DETERMINING PRECISION OF ANALYTICAL METHODS—GUIDE TO THE PLANNING AND CONDUCT

1 SCOPE. This standard sets out procedures for the planning and execution of interlaboratory test programs, including the evaluation of results, to determine the precision of methods of chemical analysis in terms of repeatability, within-laboratory reproducibility and reproducibility.

NOTE: Depending upon the plan selected for the test program (see Clause 8.1), and with due consideration of industrial practice, it may be (more) appropriate, within the context of the statistical treatment of results, that the term 'repeatability (r)' be regarded as being interchangeable with or additional to the term 'within laboratory reproducibility (R_w)' as defined in this standard.

2 APPLICATION. The procedures described are applicable to analytical methods that have been previously developed and validated in terms of concentration range, interfering elements and operating parameters to ensure that technical difficulties and bias have been minimized and methods are rugged and suitable for the purpose for which they have been designed.

The procedures are applicable to participating laboratories which are reasonably experienced in the methods to be evaluated, and to test materials which are homogeneous so that any variability in the material, as a component of the total analytical error, is minimal.

3 REFERENCED DOCUMENTS. The following standards are referred to in this standard:

AS 2667 Chemical Testing—Sampling—Glossary of Terms

AS 2706 Numerical Values—Rounding and Interpretation of Limiting Values

BS 4237 Report on Reproducibility of Methods of Chemical Analysis Used in the Iron and Steel Industry

BS 5497 Precision of Test Methods
Part 1—Guide for the Determination of Repeatability and Reproducibility for a Standard Test Method

ISO 5725 Precision of Test Methods—Determination of Repeatability and Reproducibility by Interlaboratory Tests.

4 DEFINITIONS. For the purpose of this standard, the following definitions apply. For additional definitions refer to AS 2667.

4.1 Accuracy—the closeness of agreement between the true value and the mean of the results.

NOTE: The smaller the systematic component of the experimental errors which affect the results, the more accurate is the procedure.

4.2 Bias—an error which has the same value under a variety of conditions and remains constant from one measurement to another. Such an error is not reduced by taking the average of repeated measurements.

NOTE: Bias may also be termed 'constant error' or 'systematic error'.

4.3 Cell—each combination of a laboratory and a concentration level is called a cell of the precision experiment.

4.4 Critical difference—the value at or below which the absolute difference between two test results, obtained under specified conditions, may be expected to lie with a specified probability.

NOTE: Both repeatability and reproducibility are special types of critical difference.

4.5 Critical value—the value of the test statistic (for a statistical test) above which a given percentage of values for the test statistic may be expected to lie.

4.6 Outlier—an experimental result from which a test statistic that is greater than its 1 percent critical value has been calculated.

4.7 Precision—the closeness of agreement between results obtained by applying the experimental procedure several times under prescribed conditions.

NOTE: The smaller the random part of the errors which affect the result, the more precise is the procedure.

4.8 Range—the absolute difference between the greatest and least values observed.

4.9 Repeatability (r)—the value at or below which the absolute difference between two single test results obtained with the same method on identical test material, under the same conditions (same operator, same apparatus, same laboratory, and the minimum practical time consistent with separate tests), may be expected to lie with a specified probability.

NOTE: In the absence of other specifications, the probability is 95 percent.

4.10 Replicate determinations—one or more additional determinations carried out under repeatability (or within-laboratory reproducibility) conditions on separate test portions of the same test material.

4.11 Reproducibility (R)—the value at or below which the absolute difference between two single test results obtained with the same method on identical material obtained by operators in different laboratories may be expected to lie with a specified probability.

NOTE: In the absence of other specifications, the probability is 95 percent.

4.12 Single test result—the value obtained by applying the standard test method fully once to a single test portion.