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RECOMMENDATIONS FOR PUBLICATION OF PAPERS ON PRECIPITATION METHODS OF GRAVIMETRIC ANALYSIS

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RECOMMENDATION FOR PRESENTATION OF PAPERS ON PRECIPITATION METHODS OF GRAVIMETRIC ANALYSIS

Many papers continue to be published concerned with precipitation methods of gravimetric analysis. These may be devoted to the elucidation of the fundamental processes involved in precipitation or to the development of new precipitants for inorganic or organic species or to the application of complexing agents to extend the possibilities of gravimetry for the separation and determination of metal ions. The role of gravimetric analysis in the provision of referee methods for establishment of the composition of standards used for calibration in instrumental methods of analysis remains important.

The fundamental operation in gravimetric analysis is the quantitative precipitation of the component to be determined in a form that is free from contaminants and easy to separate from the mother liquor. The precipitate must either itself be a stoichiometric compound that is suitable for weighing, i.e. involatile, non-hygroscopic, non-efflorescent, and inert to reaction with air, or one that is easily convertible into such a compound by drying or ignition. Although these requirements seem simple, it is difficult to keep the error by dissolution losses, contamination of the precipitate, etc., below 0.1-0.2% which, in most cases are the upper limits required in practice.

This report is based on a paper by Erdey, Polos and Chalmers (1) and it proposes a standard set of requirements for the development and publication of a gravimetric method and makes recommendations concerning the date which should be incorporated into such a paper.

Information pertaining to the following should be given:

1. Sample and Preparation of Sample for Analysis

(a) Description of chemical composition of sample matrix.
(b) Appropriate sample weight (based on homogeneity of sample and concentration of component to be determined).
(c) Necessary drying conditions for solid samples.
(d) Dissolution procedure for solid samples and evidence of complete dissolution.

2. Precipitant and other reagents

(a) Purity and stability of solid reagents used to form the insoluble species that is to be weighed.
(b) Any procedures required for the purification of solid reagent(s).
(c) The method of preparation, composition, formulae and properties of new reagents in such detail as to allow their production and characterisation by other workers. Source of commercially available reagents.
(d) Required purity and stability of other reagents used (buffers, solvents, wash-liquid(s), standard solutions, etc).
(e) Stability of precipitant in solution under laboratory conditions (i.e. effect of daylight, temperature, oxygen, CO_2).

3. Conditions for Sample Solution

(a) Chemical composition of the solution before precipitation.
(b) Values of permissible range of absolute amount and concentration of the component to be determined.
(c) Values of permissible range of pH, temperature and volume of sample solution before precipitation.

4. Method of Precipitation

(a) pH, volume, temperature and concentration of precipitant reagent solution. Permissible limits for these variables, e.g. pH 5± 0.2.
(b) Order of addition of reagents.

(c) Rate of addition of precipitant (or details of method of conducting precipitation from homogeneous solution).
(d) Final pH, time and temperature of digestion.
(e) Temperature for filtration.
(f) Type of filter.
(g) Composition of wash-liquid, volume and number of washes.
(h) Temperature(s) and duration of drying or ignition.
(i) Any other special technique or precautions required during precipitation, filtration, washing, drying or ignition.
(j) Recommended method for cleaning filtration apparatus.

5. Properties of Precipitate

(a) Statement of expected nature of precipitate (e.g. gelatinous, crystalline, etc.) and estimate of particle size or particle size distribution if applicable.
(b) Values of solubility of precipitate in (i) moth liquor, and (ii) wash liquid, to enable estimate of completeness of precipitation and loss on washing, respectively.
(c) Thermogravimetric data for precipitate if available.

6. Method of Calculation of Analytical Result

The stiochiometric factor (gravimetric conversion factor) should be given for the weighing form. This is expressed as the ratio of the relative atomic (or molecular) mass of the species determined to the relative molecular mass of the weighing form.

7. Selectivity

(a) Any systematic deviations in the analytical result arising from the presence of other components (which thus constitute 'interference') should be measured for the possible interfering species likely to be present in sample matrices at the appropriate concentration levels. Concentrations which cover the full range of likely ratios of interfering species to analyte species should be investigated at high and low analyte concentrations. The existence of systematic deviation (interference) should be defined in relation to the precision of the complete analytical procedure (i.e. when the error is greater than e.g. 2 or 3 times the standard deviation of the analytical result obtained in the absence of the other component).
(b) Limiting permissible concentrations of those elements found to interfere should be reported.
(c) When two or more species, which do not interfere individually, are present together with the component to be determined, checks should be made that there is no slight interference from each which is additive or subtractive.
(d) Recommendations should be given for minimising the effects of interfering species, e.g. by use of masking agents or preliminary separation of either the species to be determined or interfering species.

8. Precision

(a) The reproducibility of the complete analytical procedure under optimum conditions and in separation procedures or other sample pretreatment (e.g. wet or dry ashing) should be reported. The relative standard deviation for the complete method of analysis, expressed as a decimal fraction, should be given at both high and low concentrations of the component determined. The number of measured values from which the relative standard deviation is derived must be stated in each case.

9. Accuracy

(a) The results of tests for losses, recovery and contamination when the method is applied to the analysis of standard or reference samples or simulated sample solutions should be reported.
(b) Results should be compared with those obtained by other methods in analysis of standard or reference samples. Estimate of systematic errors.

10. Applications

The applicability of the recommended procedures for various specified matrices should be described together with results for samples actually examined.

11. Assessment

There should be a realistic assessment of the new method in terms of precision, accuracy, speed, cost, selectivity, simplicity, etc., compared to existing methods.

New methods for the gravimetric determination of simple inorganic anionic and cationic species should be proposed only if they show promise of superiority over the best existing methods. The particular advantages and disadvantages of the method proposed should be mentioned. It is stressed that the above requirements represent the minimum acceptable amount of information which should be included in any report of a new gravimetric method of analysis.

REFERENCES

1. L.Erdey, L.Polos and R.A.Chalmers, Talanta, (1970), 17, 1143.