

Standard operating procedures



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SOPs – Standard Operating Procedures – A General Overview

Aim

This document is not meant to be all encompassing but is designed to give readers an overview as to what should be in a SOP and what a SOP might look like.

Background

It was common in the past for to be photocopied from a book and notes/scribbles to be added to a method to adapt it to a Laboratory's specific needs. This resulted in analysts using different versions of the same method and obtaining different results. It also hindered the communication of improvements that an individual analyst had made to a method, as it was not known how many copies of a method existed and whether they had all been updated to include the improved method.

A way to improve this chaos is to ensure that all methods and procedures shall be documented to the extent necessary to ensure proper implementation and consistency of application. Hence the introduction of the concept of SOPs – Standard Operating Procedures.

It is sometimes not practical for laboratories to use an SOP which is fully documented specifying each type and individual test procedure. For example, analysis of soil samples for polyaromatic hydrocarbons. It is simpler to write an SOP on the preparation of soil extracts for analysis by gas chromatography and another SOP of a more general nature regarding the analysis of extracts by gas chromatography.

Information required for all SOPs

All SOPs should contain the following information

Authorised by (Someone in the organisation needs to approve the SOP before it can be used)

·Copy number (A record of the number of copies of an SOP needs to be kept - each individual copy has its own number - identifies if there are any illicit versions in use)

Page x of y (e. g. Page 1 of 6 – appears at the bottom of each page – identifies if there are pages missing from a method)

Other information may include when the method should be reviewed, individual reference numbers for when an organisation has many SOPs, Issue numbers – to identify new versions of a method from old versions of a method which have been withdrawn.

To help explain some of the terms below, parts of an SOP involving the analysis of nitrite in water have been reproduced in boxes like this. THESE WILL ONLY BE EXAMPLES AND NOT COMPREHENSIVE.

Title

Determination of A analyte(s) or measurand(s)}, in C {matrix} using D {principle}

Determination of nitrite in water by UV/Visible spectrophotometry

Scope

Enables a user to see quickly whether the method is appropriate for a desired application (see also the Annex- method validation and measurement uncertainty).

- the analyte (measurand) which can be determined by the method,
 - the sample matrix from which the analyte (measurand) may be determined,
 - the technique used in the method,
 - the minimum sample size,
 - known interferences which prevent or limit the working of the method,
 - the concentration range of the method.
- The amount of nitrite in an aqueous sample can be determined.
- The detection limit is 0. 01 mg dm⁻³ for a 30 cm³ sample.
- There is a maximum limit of 0. 25 mg dm⁻³ for a 30 cm³ sample. Dilutions can be carried out on the sample to extend the range of analysis.
- Type of sample: Raw and potable waters and sewage effluents.
- Samples that are highly coloured or turbid can cause interference*.
- * If the alkalinity of the sample is high, such that a pH value of 1.9 ± 0.1 is not attained after the addition of the sulphanilamide reagent (....) then the formation of the azo-dye is affected.
- *It may be more appropriate to have a separate section for interferences.

Warning & Safety Precautions

handling the samples

handling or preparing solvents, reagents, standards, or other materials

operation of equipment

requirements for special handling environments, e. g. fume cupboards

- N-1-naphthylethylenediamine dihydrochloride should be regarded as a special hazard. Contact with the solid and solution containing it should be avoided.

- Disposable gloves should be worn when handling effluent or sewage samples.

Normal precautions to avoid skin contact and/or ingestion should be taken in the handling of all reagents.

Definitions

Define any unusual terms, use ISO definitions wherever possible. Quote sources. Include chemical formulae/structures if appropriate.

Principle

Outline the principle by which the analytical technique operates. This section should be written so that a summary of how the method works can be seen quickly.

The method is based on the diazotisation of sulphanilamide by nitrite in the presence of phosphoric acid, at pH 1.9, and the subsequent formation of an azo dye with N-1-naphthylethylenediamine. The absorbance of this dye is measured at 540 nm and is proportional to the nitrite concentration by means of a calibration curve.

Reactions

Where appropriate to clarifying the working of the method or calculations, include details of any relevant chemical reactions. This may be relevant, e.g. where derivatisation is involved, titrimetry etc..

Reagents & Materials

List all of the reagents materials, blanks, QC samples and standards and certified reference materials required for the analytical process, identified by letter or number. List:

- details of any associated hazards including instructions for disposal,
- appropriate grade of reagent/chemical,
- specialist suppliers for rare chemicals,
- need for calibration and QC materials,
- details of preparation, including need to prepare in advance,
- containment and storage requirements,
- shelf life of raw material and prepared reagent,
- required concentration, noting whether w/v, w/w or v/v,
- labelling requirements.

a. All reagents should be of at least analytical reagent grade.

b. Water : In all cases demineralised water shall be used.

c. Nitrite Stock for Standard Solution, 1000 mg dm⁻³ NO₂: Weigh out 1.5000 g ± 0.0002 g sodium nitrite, (previously dried overnight at 105 ± 5° C and stored in a desiccator – desiccant silica gel) and transfer quantitatively to a 1 dm³ Grade A volumetric flask containing approximately 500 cm³ of water (b). Make up to the mark with water (b) and stopper. Shake until all the solid dissolves. Transfer to an appropriately labelled glass

and store in the refrigerator. The solution is stable for 1 month.

d. Orthophosphoric acid 85% w/w:

e. 2 mg dm⁻³ Nitrite Standard: Using a Grade A glass pipette, pipette 2 cm³ of 1000 stock nitrite solution (c) into a 1 dm³ Grade A volumetric flask and make up to volume with water (b). Stopper and shake to mix. Transfer to an appropriately labelled glass bottle with stopper and store in a refrigerator. The solution is stable for 1 week.

f. Nitrite Stock for Quality Control Solution 1000 mg dm³ NO₂: Ideally the sodium nitrite should be obtained from a different source to that used for the standard. Weigh out 1.000 ± 0.0050 g of sodium nitrite...

g. Sulphanilamide Reagent: Using a 100 cm³ measuring cylinder, add 50 cm³ of 85% orthophosphoric acid (d) to a 500 cm³ beaker containing 250 cm³ of water (b). Weigh out 1.000 g ± 0.025 g of sulphanilamide and quantitatively transfer to the beaker. Stir with a glass rod until the solid dissolves. Weigh out 1.000 g ± 0.010 g of N-1-naphthylethylenediamine dihydrochloride and quantitatively transfer to the beaker. Stir with a glass rod until the solid dissolves. Quantitatively transfer the solution to a 500 cm³ volumetric flask and make up to volume with water (b). Stopper the flask and shake to mix. Transfer to an appropriately labelled amber glass bottle and store in the refrigerator. The solution is stable for 1 month. NB Immediately before use, this solution should be brought to room temperature.

Points to note from example:

Each reagent is uniquely identified so as to avoid confusion.

Weigh 20.00 g... Indicates that a balance capable of weighing to 2 decimal places is required.

20.00 g \pm 0.25 g... Tells the analyst that he needs to be between 19.75 g and 20.25 g.

Using a 50 cm³ measuring cylinder... Indicates accuracy required for this procedure.

Quantitatively transfer... Indicates that you need to transfer all the solid with washings to the beaker.

Appropriately labelled... This might include: Name of reagent, Concentration of reagent, Date reagent made, Expiry date of reagent, Name of analyst who prepared the reagent – Most computer systems can be used to produce sheets of labels that suits an individual's and/or organisation's needs.

Apparatus & Equipment

Describe in sufficient detail the individual pieces of equipment and how they are connected to enable unambiguous set-up. For glassware include grade where applicable. Include environmental requirements (fume cupboards etc.).

a. A {Manufacturers name/model number} UV/Visible spectrophotometer with 10 mm cells is used.

b. The instrument should be switched on for a minimum of half an hour before the analysis. Set the wavelength to 540 nm. Details of operation of the instrument are given in the instruction manual for the instrument.

It may be appropriate to reproduce the relevant parts of the instruction manual in the SOP – i. e. how to set up and use the spectrophotometer. This will depend on how easy it is to get access to the manual.

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Sampling & Sample Preparation

Include sufficient detail to describe how the test portion is obtained from the sample as received by the laboratory. All information on the precautions to be taken to minimise the risk of contamination of the samples needs to be included. Include information regarding the storage, conditioning and disposal of samples. Many laboratories will write a separate document about the receipt, labelling, storage and disposal of samples to save having to duplicate this information on individual SOPs.

Environmental Control

List any special environmental conditions and cross reference to safety section, e. g. temperature control.

Calibration

Identify the critical parts of the analytical process. These will have to be controlled by careful operation and calibration. Cross reference to the relevant sections above. It may be easier to incorporate this section with the analytical procedure section.

Include calibration of equipment – what needs to be calibrated, how, with what, and how often?

It is normally easier to write separate documents to cover the calibration of balances, automatic pipettes, thermometers, refrigerators, freezers and spectrophotometers to save having to put all this information into each SOP.

Quality Control

Explain the quality control procedures, frequency of quality control checks during batch analysis, pass/fail criteria, action to take in the event of a

failure. It may be appropriate to incorporate this section in the analytical procedure section.

Analytical Procedure

Describe the analytical procedure, cross referencing previous sections as appropriate including reagents (with the appropriate identifier), apparatus and instrumentation. Indicate at which point in the analytical procedure the quality control, and calibration procedures should be performed.

- Use a 50 cm³ measuring cylinder to transfer 30 cm³ of either sample, blank water standard ... or QC solution... to appropriately labelled test tubes. For each batch of samples one sample should be replicated.
 - For effluent samples or samples which are likely to contain significant levels of nitrite use a glass pipette, pipette 3 cm³ of sample into a 50 cm³ measuring cylinder containing 20 cm³ of water.
 - If any of the samples are highly coloured or turbid, prepare a sample blank by transferring 30 cm³ of the appropriate sample to an appropriately labelled test tube and add 1 cm³ of 10% orthophosphoric acid (...). Treat as an ordinary sample except that water (...) is used instead of sulphanilamide reagent (....).
 - Using an automatic pipette add 0.75 cm³ of sulphanilamide solution (....) to each test tube immediately by swirling, and allow to stand for at least 30 minutes but no longer than 2 hours.
 - Measure the absorbance of the blank (....) and the standard (....)
 - Enter the relevant information in the spectrophotometric logbook* and check that the absorbance of the standard is within the specified range detailed in the log book. If the absorbance falls outside this range, consult a senior officer before proceeding further.
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· Determine the nitrite concentration of the QC solution (...) and the samples. Plot the concentration recorded for the QC solution (...) on the appropriate quality control chart together with the date and your initials. If the point does not fall within the set limits, the senior officer in charge must be informed.

* Any samples that give concentrations greater than 0.25 mg dm³, must be discarded and the analysis repeated using a diluted sample.

Points to note:

If any of the samples are highly coloured or turbid.... Guidelines on how to handle “difficult” samples

Using an automatic pipette... Defines the accuracy that the reagent has to be measured to

For effluent samples or samples which are likely to contain significant levels of nitrite... Guidelines on how to handle samples whose nitrite concentration will probably exceed the range for the method.

Leave the colour to develop for 30 to 60 minutes – This implies that the colour development time is important. You will have needed to carry out some checks to show that these times are satisfactory.

Any samples that give concentrations greater than 0.25 mg dm⁻³, must be discarded and the analysis repeated using a diluted sample... Only concentrations in the working range of the method should be reported.

Consult a senior officer... There will always be times when the analyst encounters problems or unknown situations – the analyst should be encouraged to seek help from an experienced member of staff.

Log Books

Information to be recorded in the log book associated with spectrophotometer might include – serial number, service dates, calibration checks (e. g. wavelength), repairs (e. g. new lamp).

On a daily basis information recorded might include date, name of analyst, analysis (e. g. nitrite), wavelength used, absorbance of blank, concentration and absorbance of standard.

At the front of the log book should be sufficient information regarding the nitrite analysis for the operator to determine if there is a problem, e. g. nitrite analysis, wavelength 540 nm, acceptable absorbances of (a) blank 0.000 ± 0.005 , (b) 0.25 mg dm⁻³ nitrite standard 0.250 ± 0.010 . Repeatability measurement on the same solution ± 0.003 .

Log Books; points to note:

Use of a standard, quality control and blank and a replicate sample and recording the information in a log book means that you can demonstrate with reasonable certainty that (a) the reagents were made up correctly, (b) the standards were made up correctly, (c) there was no contamination present, (d) the spectrophotometer was set up correctly and (e) the analysis can be repeated and give a result to within the expected limit.

Calculation

Lay out the formulae for calculating the results ensuring all terms are clearly defined and derived. Specify requirements for checking, cross reference to QC requirements and indicate how corrections should be dealt with.

Reporting Procedures Including Expression Of Results

Indicate how results should be reported, including; rounding of numbers; final units. It may be appropriate to indicate the level of associated with the method.

- Results are reported to two significant figures as mg dm⁻³ Nitrite (NO₂) except where customer has asked for the results to be reported as mg dm⁻³ Nitrogen in which case analytical results are multiplied by 14/46.

- * Where significant dilutions have to be made then a comment about the dilution used be put on the sheet used to report the results.

Precision , accuracy and detection limits

- Spectrophotometric measurements on the same solution must have an absorbance no greater than ± 0.003 .

- A significant absorbance for a sample is 0.010 (after correction for the blank). A detection limit of 0.01 mg dm⁻³ (equivalent to an absorbance of approximately 0.010) is quoted for a 30 cm³ sample. This becomes 0.1 mg dm⁻³ for an effluent sample where the amount of sample taken is 3 cm³.

- Where typical absorbance readings (as listed in the log book) cannot be obtained for a standard, quality control and the blank, then further investigations must be carried out until the cause of the problem is found.

* The results obtained for replicate analysis should be monitored, any significant difference ($\pm 0.010 \text{ mg dm}^{-3}$) should be investigated

Literature References

References may include operating manuals as well as published methods and standards.

This method is based on Method H in the Department of the Environment Standing Committee of Analysts booklet “ Oxidised Nitrogen in Waters 1981”.

Annex – Method Validation and Measurement Uncertainty

Methods should be supported by experimental data providing information on accuracy, precision and selectivity . The major sources of uncertainty, relating to the method should be identified and the assigned values listed. The overall uncertainty should be listed together with an explanation of how it was derived.

In the case of the nitrite method that was based on a published method, the validation work involved:

- Reproducibility – showing comparable results were obtained.
- Repeatability – showing in the results obtained for a known sample measured five times on the same day.
- Making up a set of 5 standards to show that the calibration was linear for the range 0.01 to $0.25 \text{ mg dm}^{-3} \text{ NO}_2$.
- Checking the detection limit using spiked samples .
- Checking that the time interval quoted for the colour development time was correct and non-critical within the stated range.

We also looked at the method to identify major causes of variation in results – e. g. measuring the sample volume with a measuring cylinder. We found that the biggest variation was in different analysts making up the ‘ same’ standard solutions – hence the need to define an acceptable absorbance range for the standard.