HOW TO WRITE AN SOP

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SOP

- SOP is “Standard Operating Procedure”
- SOP is a written, standardized, operations manual for laboratory procedures
- The SOP should be specific for each la, based on the specific approved method, equipment, reagents used in that lab
- SOP is to ensure that ANY lab tech will perform the method exactly the same way

COMPONENTS FOR AN SOP

- Title
- Scope and Application
- Summary of Method
- Definitions
- Interferences
- Safety
- Equipment and Supplies
- Reagents and Standards
- Sample collection/ preservation/ storage
- Quality Control

Not all components will be used for every analysis

COMPONENTS FOR AN SOP

- Calibration/ standardization
- Procedure
- Data Analysis
- Method Performance
- Pollution Prevention
- Waste Management
- References
- Flow Charts/ graphs/ diagrams
- Appendices

EXAMPLES FOR SET-UP

- I will use Dissolved Reactive Phosphorus (automatic, colorimetric) and Total Dissolved Solids as my examples for this presentation

TITLE

- Title of the SOP should include:
- Analytical Method Name
- Approved methodology you are using
- Date procedure was written/ updated

EXAMPLE TITLES

DRP

- Dissolved Reactive Phosphorus
- US EPA 365.3
- October 2016

TDS

- Total Dissolved Solids
- Residue, filterable
- SM 2540C, 1997
- October 2016

SCOPE AND APPLICATION

- Scope and application should include :
  - What analyte you are looking for
  - What sample type you can analyze
  - The range of the method

SCOPE AND APPLICATIONS

DRP

- This method is used to determine dissolved reactive phosphorus in drinking, surface, and saline waters, and in domestic and industrial wastes

TDS

- This method is suitable for determination of total dissolved solids in potable, surface, and saline waters, as well as domestic and industrial wastes

SUMMARY OF METHOD

- Summary of method should include
  - An explanation of the chemistry behind the methodology
  - What complex forms with which reagents, what color (if colorimetric)
  - If physical science (filtration) then what particles/ size, etc

SUMMARY OF METHOD

DRP

- Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phosphor-molybdate complex

TDS

- A well mixed sample is filtered through a standard glass fiber filter, and the filtrate is evaporated to dryness in a weighed dish and dried to a constant weight at 180 degrees C

DEFINITIONS

- Define any terminology as if the person that will be reading your SOP has no background in the industry

DEFINITIONS

DRP

- Calibration Standard (CAL) – A solution prepared from the primary dilution standard solution or stock standard solution and the internal standard and surrogate analytes. The CAL is used to calibrate the instrument response with respect to analyte concentration

TDS

- "Dissolved Solids" is the portion of solids that passes through a filter when performing a TSS

INTERFERENCES

- List all potential interferences for the analysis here
- How do you deal with the interferences

INTERFERENCES

DRP

- Sample turbidity must be removed by filtration prior to analysis

TDS

- Highly mineralized waters with a considerable calcium, magnesium, chloride, and/or sulfate content may be hygroscopic and require prolonged drying, proper desication, and rapid weighing

SAFETY

- Properly identify any known safety hazards associated with the method/ reagents used for the method that are outside of “normal” laboratory practices

SAFETY

DRP

- No known safety hazards for this method other than normal laboratory practices

TDS

- No known safety hazards for this method other than normal laboratory practices

EQUIPMENT AND SUPPLIES

- Identify all equipment and supplies (non chemical) used for this method
- Include things like the balance, pipets (sizes/ type), graduates, flasks, beakers
- If you have to adjust the pH of a reagent/ samples – identify the meter used
- Any equipment used for filtration/ distillation should be listed
- Any specs/ analyzers used should be listed.
- Include the proper name, model of the instruments used.
- Brand of pipets is NOT necessary

EQUIPMENT AND SUPPLIES

DRP

- Westco SmartChem Discrete Wet Chemistry Analyzer, model 200
- (now Unity Scientific Smartchem 200)

TDS

- Evaporating dishes: dishes of 100 mL capacity made of one of the following materials:
- Porcelain (90 mm diameter)
- Platinum
- High silica glass

REAGENTS AND STANDARDS

- List all reagents used here
- The list should include:
  - Type of water
  - Any pre-made, purchased solutions
  - How to prep all solutions necessary to perform the analysis
  - How frequently the solutions should be prepped: daily, weekly, monthly, etc
  - How should reagents be stored once made?

REAGENTS AND STANDARDS

DRP

- Ascorbic Acid Solution: Ina 250 mL volumetric flask, dissolve 3g of ascorbic acid ($C_6H_8O_6$) in 200 mL deionized water. Dilute to 250 mL with reagent water. Make fresh daily.

TDS

- none

SAMPLE COLLECTION, PRESERVATION, STORAGE

- Identify the type of sample (grab, composite)
- Does sample need filtered first
- What type of bottle can be used
- What preservative can be used (if any)
- Does sample need refrigerated?
- Can sample be frozen?

SAMPLE COLLECTION, PRESERVATION, STORAGE

DRP

- DRP should be a grab sample, and must be filtered within 15 minutes of collection with a 0.45 um filter.
- Sample should be refrigerated to </= 6 degrees C (not frozen) and analyzed within 48 hours

TDS

- TDS should be collected in glass, polyethylene, or fluoropolymer
- Samples should be refrigerated to </= 6 degrees C (not frozen) and held for no longer than 7 days

QUALITY CONTROL

- List all QC requirements necessary by the method, your facility, and your regulating agency
- Explain what parameters you do (blanks, dups, spikes, etc)
- Explain the MINIMUM frequency of doing the QC
- If using an automated system, explain if the QC is manual or programmed

QUALITY CONTROL

DRP

- DRP is run monthly, and must include a blank, a standard, duplicate and spike for each analysis.
- External controls are analyzed once per quarter

TDS

- TDS is done daily, a duplicate and a blank are run when every 10th sample
- Duplicate samples must be within 5% of their average

CALIBRATION AND STANDARDIZATION

- What standards do you use to create the calibration curve?
- Do you use a blank?
- How do you perform the calibration?
- Do you use anything to verify the calibration?

CALIBRATION AND STANDARDIZATION

DRP

- The SmartChem automatically prepares the standard dilutions. Prep your stock solution and insert into the correct spot in the instrument. Place 3 empty cups in the cal standard tray

TDS

- none

PROCEDURE

- List EVERY step you need to complete to perform the analysis here
- Do you have to distill sample? List all steps
- Does sample need digested first? List all steps
- Do you use any electronic equipment? List all steps, including start-up/ warm-up, etc
- Nothing is to be considered “automatic” or “routine” as far listing steps, EVERY step should be listed

PROCEDURE

DRP

- Turn SmartChem on by using toggle switch located at rear of left panel of instrument.
- Open the SmartChem software and allow the system to stabile to 37 degrees C (about 20 minutes)

TDS

- Preparation of evaporating dish: Only dissolved solids are run, so heat clean dish to 180 +/- 2 degrees C for 1 hour in an oven. Store in desicator until needed. Weigh immediately before use.

DATA ANALYSIS AND CALCULATIONS

- Does the equipment automatically calibrate the curve, or is this done manually?
- Do you need to plot the absorbance/ optical density?
- Do you need to multiply for dilutions?
- What is the minimum reporting level? Don't report values below this
- What is the max of the curve?
- Have you done any studies to verify you can exceed the max value of curve?
- Report nothing over the max value
- What units should results be reported in?

DATA ANALYSIS AND CALCULATION

DRP

- The SmartChem has been programmed to dilute and re-analyze any values that exceed the curve maximum.

TDS

- mg TDS/L = ((A-B)*1000)/C
- Where
  - A = weight of dried residue, mg
  - B = weight of dish, mg
  - C = mL of sample used

METHOD PERFORMANCE

- How do you determine if the analysis is working properly?
- For equipment/ instrumentation there is usually a list of things for determination of method performance

METHOD PERFORMANCE

DRP

- DRP on SmartChem:
- Test Range is 0.01 to 0.50 mg/L

TDS

- Method is reliable up to 200 mg residue in evaporating dish

POLLUTION PREVENTION

- Do you use chemicals that may be toxic?
- Does any reagent you use need special disposal?
- What steps have been taken to reduce waste?

POLLUTION PREVENTION

DRP

- Method is done on discrete analyzer to reduce the amount of waste generated

TDS

- none

WASTE MANAGEMENT

- How do you handle wastes generated with this method?
- Make sure your methods are in compliance
- Reference " The Waste Management Manual for Laboratory Personnel" or "Prudent Practices in the Laboratory"

WASTE MANAGEMENT

DRP

- Liquid wastes from this method are disposed of through the laboratory sink, which feeds into an acid pit

TDS

- Residue is washed according to laboratory protocol

REFERENCES

- Did you cite any external material? List it here
- Proper reference listing should be followed

DRP

TDS

REFERENCES

TABLES, DIAGRAMS, FLOWCHARTS

DRP

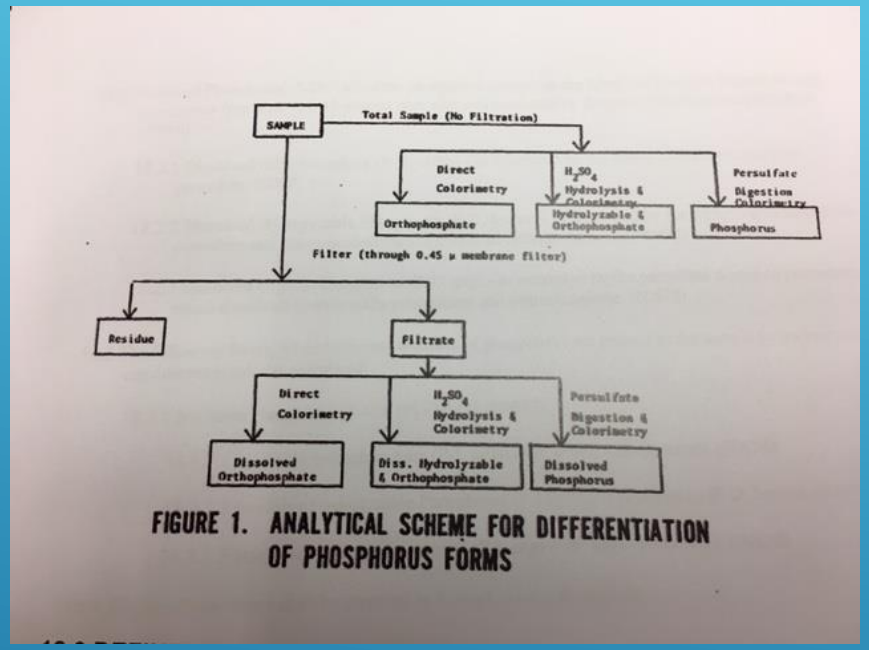

FIGURE 1. ANALYTICAL SCHEME FOR DIFFERENTIATION OF PHOSPHORUS FORMS

TDS

- none

TABLES, DIAGRAMS, FLOWCHARTS

APPENDICES

- Do you have a “quick” version of instructions?
- Something that says: turn on and allow to warm up for x minutes
- Filter sample
- Shortened version of reagent prep

DRP

- Turn equipment on and allow to warm up for 30 minutes
- Set up filtration apparatus before collecting sample to reduce delay

TDS

- Filtrate is collected as part of TSS
- Make sure evaporating dishes have been dried in oven and weights are stable

APPENDICES

Calibrate balance:

Turn balance on: touch and hold on/off

Touch the Adjust int icon on the display screen....balance will enter self calibrate mode

When done, touch ok button. (also on the touch screen)

Calibrate DO:

Place meter in cradle to power up (do NOT turn on first)

Allow meter/ probe to stabilize for at least 30 minutes

To calibrate:

Check barometer for temperature and barometric pressure

Record these values in log book

Calibrate DO:

Press the “sun” key to illuminate the screen for better viewing

Press cal key

Scroll to <barometer> and press enter

Select <in Hg>

Scroll to <enter value>

Scroll through numbers to enter the correct barometer reading, pressing the enter key to select the correct value

- Questions?

- Contact info:
- Denise Seman
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I have an electronic template for SOP

Email me and I will forward you a copy