
	<p style="text-align: center;"><b>Standard Operating Procedure</b></p> 	Effective Date:	Version:
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<b>SOP_Chem_002 Chemical Analysis_ Spectroquant Chemical Oxygen Demand</b>			Page #: <b>1 of 4</b>

## Standard Operation Procedure – Total Chemical Oxygen Demand Closed Reflux, Spectrophotometric Method, Cell Test (Cat. No.

### 1. Scope and Application

- Chemical Oxygen Demand (COD) measures the oxygen equivalent of that portion of the organic matter in a sample that is easily oxidised by a strong chemical oxidant.
- It is an important and common parameter to measure the amount of organic compounds in streams, industrial waste and in operational control of wastewater treatment plants. It is also applicable for measurements on human excreta.
- The procedure described hereafter is applicable to measure the COD, in the range of 25-1500 mg/L..

### 2. Summary

- The COD (chemical oxygen demand) expresses the amount of oxygen originating from potassium dichromate that reacts with the oxidizable substances contained in 1 L of water under the working conditions of the specified procedure.
- 1 mol  $K_2Cr_2O_7$  is equivalent to 1.5 mol  $O_2$  Results are expressed as mg/l COD (= mg/l  $O_2$ ).
- The sample is oxidized with a hot sulphuric solution of potassium dichromate, with silver sulphate as the catalyst. Chloride is masked with mercury sulphate. The concentration of unconsumed yellow  $Cr_2O_7^{2-}$  ions is then determined photo- metrically.

### 3. Apparatus

- Spectroquant, NOVA 60
- Reaction cells
- Thermoreactor
- Pipettes

### 4. Interferences

- Chlorides reacts with silver ion to precipitate silver chloride and thus inhibits the catalytic activity of silver.

## 5. Collection, Preservation and Storage

- Collect samples in 1L plastic buckets.
- Preferably, analyse samples immediately after sampling.
- Store samples at 4 °C or freeze dry samples.
- Preserve wastewater samples by acidifying with concentrated sulphuric acid to pH 2 and faecal samples by freeze drying or freezing.
- Determine COD on well- homogenised samples.

## 6. Safety Precautions

- Handle concentrated sulphuric acid with care.
- Always use safety goggles, gloves and laboratory coat while working in laboratory.
- Wear face shield and protect hands from heat produced when contents of the vessels are mixed.
- After the analysis, clean bottles and beakers with water then dry.
- Dispose used gloves after completion of analysis.
- Clean hands using antiseptic soap.
- Avoid spillage and contact with skin. In the latter case use copious washings with cold water and call for medical attention.

## 7. Sample Preparation –Faecal Sludge

### 25-1500 mg/L COD

1. Weigh out 2.0000g of well-mixed faecal sludge sample.
2. Blend the weighed sample with 500ml of distilled water in a 1L blender for 30 seconds on the highest speed.
3. Add 250ml distilled water and blend on highest speed until the sample is homogenised (this could range from 30 to 60 seconds).
4. Transfer the blended mixture into a 1L volumetric flask.
5. Add 200ml of blender washings into the flask and top up to 1L with distilled water.
6. Transfer the 1L solution to a plastic bottle and store at 4 °C.

## 8. Reagents

- Potassium dichromate
- Silver sulphate
- Mercury sulphate

## 9. Calibration

- Prepare a series of at least three standards, covering the desired range, and a blank by diluting suitable volumes of standard solutions. Prepare a calibration curve by plotting instrument response against standard concentration. Compute sample concentration by comparing sample response with the standard curve. Multiply answer by appropriate dilution factor. Report only those values that fall between the lowest and the highest calibration standards. Dilute and re-analyse samples exceeding the highest standard. Report results in mg/L.

## 10. Procedure

- Pipette 3 ml of pre-treated sample into the reaction cell.
- Heat the cell at 150 °C in the preheated thermoreactor for 2 hours.
- Remove the hot cell from the thermoreactor and allow cooling in a test-tube rack.
- Do not cool with cold water!
- Wait 10 min, swirl the cell, and return to the rack for complete cooling to room temperature (cooling time at least 30 min).
- Measure in the photometer. The bar code on the vial will automatically pick up the wavelength, testing range and testing parameter.
- Notes on the measurement:
- For photometric measurement, the cells must be clean. Wipe, if necessary, with a clean dry cloth.
- Measurement of turbid solutions yields false-low readings.
- The measurement value remains stable for 60min.

### Notes on the measurement:

- Analyse immediately after sampling.
- Reclose the reagent bottles immediately after use.
- For photometric measurement, the cells must be clean. Wipe, if necessary, with a dry paper towel.
- The colour of the measurement solution remains stable for 30 min after the end of the reaction time stated above.
- The measurement value remains stable for 60min.

## 11. Waste Disposal

- Collect waste in a 2.5L bottle for Waste Tech collection.

## 12. Data Quality

Measurement	10 – 150 mg/l COD
Standard Deviation (mg/l COD)	± 1.1
Confidence Interval (mg/l COD)	± 3
Sensitivity (mg/l COD)	2
Accuracy (mg/l COD)	± 5

**13. References**

<http://www.merckmillipore.com/ZA/en/products/analytcs-sample-prep/test-kits-and-photometric-methods/instrumental-test-systems-for-quantitative-analyses/photometric-measurements-spectroquant-system/spectroquant-tests/>

**APPROVAL OF STANDARD OPERATING PROCEDURE**

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