

STATE OF WASHINGTON DEPARTMENT OF ECOLOGY
SOURCE TEST CALIBRATION METHOD V
ECOCAL GENERATOR CALIBRATION

I. Principle

The generator with the Ecocal permeation devices for H₂S and SO₂ are calibrated over the required ranges with gravimetrically calibrated Teflon permeation tubes traceable to NBS and Scott-Marrin certified gas standards. Wet chemical methods are used to provide alternate checks.

II. Equipment

- (1) Ecocal Model 202 calibration unit, with H₂S and/or SO₂ permeation units installed.
- (2) Barton Analyzer with pump, scrubber, and Teflon tubing.
- (3) IOC SO₂ Analyzer.
- (4) Gravimetrically calibrated Teflon permeation tubes and Scott-Marrin certified gases. Constant temperature bath and gas dilution panel.
- (5) Teflon or Saran bags. Gas tight syringes. Calibrated wet test meter.

III. Procedure

The Ecocal calibration unit is set up with the Barton Titrator and IBC Analyzer and connected to the gas dilution panel with teflon tubing and glass-teflon control valves. The constant temperature bath is allowed to come up to the specified temperature as is the Ecocal unit. Permeation tubes and devices are allowed to equilibrate until gas output is steady.

- (1) For H₂S:

The Barton Titrator is used to determine the output of the calibrated permeation tubes over the range of 0 – 60 ppm H₂S. Concentration values of 20, 40, 60 and 80% are sampled through the manifold and recorder values are tabulated with allowances for the instrument blanks between each point.

Using Teflon or Saran bags of 10 liter capacity, aliquots of H₂S (99.5%) are injected into the 10 liter measured amounts of scrubbed air with the gas-tight syringe. After a 15 minute equilibration period and manual mixing in the bag, the bag sample is attached to the Barton Titrator for measurement. Three samples are introduced at 30, 60 and 90% of the 0 – 60 ppm range. Recorder values are tabulated with allowances for blanks. The Scott-Marrin H₂S tank value is also recorded after analysis on the Barton Titrator.

In order to determine the accuracy and precision of the set of calibrated permeation tubes, a determination by the zinc acetate method of the concentration of the 40% value of the permeation tube standard atmosphere will be made. The procedure for the above method is attached as Appendix A.

From the above data, a calibration curve is plotted for the Barton Titrator in the range of 0 – 60 ppm H₂S. The Ecocal calibration unit is then connected to the output manifold of the dilution panel. Using the flowmeter of the Ecocal on the H₂S channel, five points are recorded on the Barton recorder. Blanks are run between each point and an accurate value is determined for five metered flow rates through the H₂S permeation device in the Ecocal unit. Using the following formula, a mean permeation rate is determined for the H₂S permeation device. (Form, Appendix A) ~

$$P = \frac{\text{ppm H}_2\text{S} \times F}{.759}$$

Where P = Permeation rate in ng/min. @ 37 degrees C

F = Flow rate in cc/min. corrected for temperature and pressure.

A calibration curve is plotted for the Ecocal permeation device for the various settings of the calibrator flowmeter.

(2) For SO₂:

The IBC SO₂ analyzer will be used to calibrate the Ecocal SO₂ permeation device in the Tracor calibrator. High-emission Teflon permeation tubes are calibrated gravimetrically and by means of wet chemistry (Appendix C). Four 40 cm high emission SO₂ permeation tubes are stored in a water bath and ventilated container for a period of two weeks, weighed twice weekly on a balance sensitive to 0.1 mg, and calibrated for emission rates by the formula:

$$P = \frac{W_1 - W_2}{T}$$

Where P = Permeation rate in ng/min.

W₁ = Starting weight in ng.

W₂ = Ending weight in ng.

T = Time elapsed in minutes

Concentration of SO₂ in a standard atmosphere can be calculated by the formula:

$$\text{ppm SO}_2 = \frac{.382 \times \text{Permeation rate and tube length}}{\text{Flow rate in cc/min.}}$$

The IBC SO₂ analyzer is connected to the manifold of the permeation tube water bath and dilution panel. Air flows are adjusted to give five concentrations of 20, 40, 60 and 100% of the range from 0 to 1000 ppm SO₂. These points are recorded and care is taken to run duplicates until a linear calibration curve accurate to plus or minus 10 ppm is obtained. A mean permeation rate is determined for the SO₂ device.

The IBC analyzer is now connected to the SO₂ output of the Tracor calibrator. Flows are set on the flowmeter of the calibrator to provide six concentration values over the range of 0 – 1000 ppm. These values are tabulated on the procedure form (Appendix A), and compared with the calculated values.

Wet chemistry determinations are performed on the concentration value of approximately 200 ppm SO₂ from the Tracor calibrator. Duplicate runs are made by means of the Hydrogen Peroxide Method (Appendix C), and must agree within 10 ppm of the values obtained by calculation of the permeation tube values above. A calibration curve is plotted of various settings of the calibrator flowmeter.

IV. Calculations

The procedure form is completed for the two gases and the values recorded from the H₂S and SO₂ analyzers. Two calibration curves are provided for the Tracor calibrator. The curves will show the gas, S/N, and permeation rate of the devices installed in the calibrator, as well as a table of H₂S and SO₂ ppm values for flowmeter settings of the Tracor calibrator flowmeters.

Ecocal Generator – H₂S and SO₂

Procedure Data Form

H₂S

Permeation Tube

Std. Atmospheres

Barton Analyser

Blank

Recorder

_____	_____
_____	_____
_____	_____
_____	_____

H₂S

Bag Standards

Conc.

_____	_____	_____
_____	_____	_____
_____	_____	_____

H₂S

Scott-Marrin Certified

Tank Gas No. _____

Conc. _____

_____	_____
_____	_____

Zinc Acetate H₂S Method

Std. Conc. 1) _____

2) _____

Analysis 1) _____

2) _____

Permeation Rate

Tracor Device s/n _____

_____ ng/min

H₂S Calib. Curve

Flowmeter Reading	Flow Rate	Calculated Concentration	Barton Analyser	
			Blank	Recorder
1) _____	_____	_____	_____	_____
2) _____	_____	_____	_____	_____
3) _____	_____	_____	_____	_____
4) _____	_____	_____	_____	_____
5) _____	_____	_____	_____	_____
6) _____	_____	_____	_____	_____

SO₂ Calib. Curve – IBC Analyser
Bath Temp. _____

Perm. Tub Nos	Perm. Rate	Flow Rate	Calc. Concentration	IBC Recorder
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

SO₂ Tracor Device Calibration Curve

Tracor Calibrator s/n _____

Flowmeter Reading	Flow Rate	Calculated Concentration	IBC Analyser Recorder
1) _____	_____	_____	_____
2) _____	_____	_____	_____
3) _____	_____	_____	_____
4) _____	_____	_____	_____
5) _____	_____	_____	_____
6) _____	_____	_____	_____