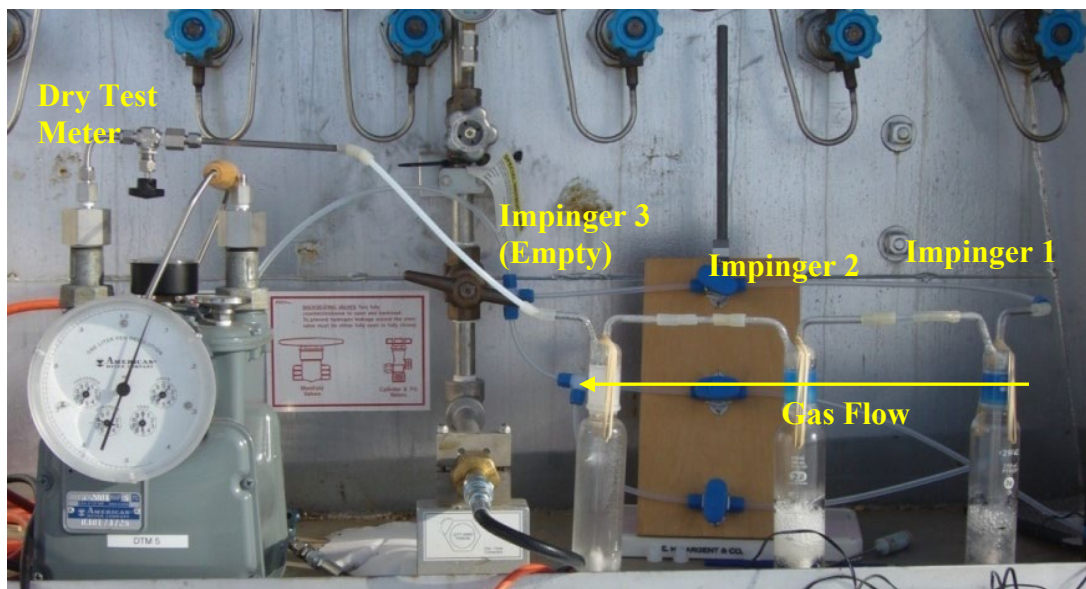


## Trace-metals sample collection procedure

A slightly-modified EPA Method 29 is used in sampling for volatile metals. Based on previous experience, the sampling train will consist of two 250-ml liquid spargers (“bubblers”) in series each fitted with a coarse frit. Each sparger contains 100-ml of a 5% HNO<sub>3</sub> / 10% H<sub>2</sub>O<sub>2</sub> solution. A third empty sparger serves as a spray trap. Double-deionized water, Fisher trace metals grade HNO<sub>3</sub> and J.T. Baker trace metals grade H<sub>2</sub>O<sub>2</sub> (or equivalent) must be used to minimize blank values. Single-pass deionized water is not recommended. The detection limits for the metal compounds will be dependant on the volume of gas sampled. A 4-hour sampling period should result in a detection limit of 1.0 µg/m<sup>3</sup>. Prior to use, each sparger should be cleaned by soaking in dilute nitric acid and rinsing several times using pure nitric acid and double-deionized water. The sampling layout is shown below.



### **The nitric acid/hydrogen peroxide absorbing solution is prepared as follows:**

Add carefully with stirring 50 ml of concentrated HNO<sub>3</sub> to a 1000-ml volumetric flask containing approximately 500 ml of double-deionized water. Then add carefully with stirring 333 ml of 30 percent H<sub>2</sub>O<sub>2</sub>. Dilute to volume with additional double-deionized water. Mix the

resulting solution well. This will provide sufficient absorbing solution for eight scrubber fillings, plus enough left over to be saved and later analyzed as a “field blank” sample. The solution must be prepared fresh for each sampling run.

**The total gas volume passed through the scrubbers must be determined.**

For accurate analytical results, it is critical that the total gas sample volume and sample temperature be accurately measured and recorded for each test run. A dry test meter is the preferred instrument for measuring the total gas volume. If a dry test meter is not available, a calibrated rotameter and stop watch may be used. A sampling duration of four hours at a sample flow rate of one liter per minute will provide sufficient material for analysis.

Any flow or volume measuring devices must be downstream of the scrubbers. Connections to the gas stream should be made with inerted stainless tubing (preferred), Teflon tubing, silicone tubing, or stainless steel tubing. Do not use Tygon or other plastic tubing.

The sample line pressure must be regulated, in-line before the scrubbers, with either a inerted or well-conditioned heated stainless-steel pressure regulator, or by the use of an accurately-controllable inerted or well-conditioned stainless-steel throttling/metering valve. The regulated pressure must be down to a sufficiently low enough level to eliminate any safety risks due to over-pressurizing the glass scrubbers. This initial sample line pressure regulating device is to be used as the primary flow-rate-setting device, in order to minimize back-pressure problems in the scrubbers and sampling train. Minor flow adjustments can be made immediately “downstream” of the pressure-setting device, but at no time should any flow adjustments be made at the outlet end of the sampling train by use of valves on the flow-measuring device (rotameter or dry test meter.)

Foaming will be observed in the scrubbers due to the reaction of the natural gas components with the hydrogen peroxide.



**Caution:** If unsaturated hydrocarbons are suspected to be present in the gas stream, there exists the possibility of epoxide formation. Appropriate safety measures should be taken to shield the scrubbers from breakage and to shield the sampling personnel from glass shards.

### **Collection of solutions**

At the conclusion of each test run, shut off the gas flow at the initial sample line pressure regulating device. Quickly disconnect the scrubber tubing starting at the last scrubber and working towards the first scrubber to avoid back-flow of the liquid. A practice run using bottled nitrogen as the gas stream and deionized water as the absorber is recommended to gain experience if the operator is not familiar with this type of sampling.

The scrubber solutions should be collected into separate glass bottles (with Teflon-lined caps) as follows:

The solution from the first scrubber (closest to the sample source) is to be transferred into one bottle, using sufficient deionized water to rinse the scrubber quantitatively.

The solution from the second scrubber is to be transferred to a second separately-labeled bottle, again using sufficient deionized water to rinse the scrubber quantitatively. The empty third (“spray trap”) scrubber should also be rinsed with a small quantity of deionized water, which should be then combined with the rinsings from the second scrubber.

The sample bottles containing the collected solutions should be left loosely capped overnight to prevent gas-pressure buildup, either from dissolved sample gases or from the residual hydrogen peroxide. Prior to shipment back to the analyzing laboratory, the bottle caps should be securely tightened. Mark the liquid level on the glass bottle using a marker. This mark will be examined in the laboratory to check that there has been no loss of sample.

### **Shipping**

Since these solutions do not have such stringent “holding time” constraints as the gas-cylinder samples, they can be shipped via UPS-Ground or FedEx-Ground delivery service, thereby minimizing concerns over sample outgassing in transit. Each bottle must be securely wrapped in



bubble wrap and placed in a leak-proof plastic bag. It is recommended that the bottle + bag assembly be placed in a paint can type overpack filled with absorbing material. The final packing is in a 4G cardboard box.

The shipment must be declared as hazardous as the solutions contain nitric acid and hydrogen peroxide. Corrosive, oxidizer, and cargo aircraft only stickers should be used on the box exterior. The hazardous declaration that GTI uses is:

UN3093, corrosive liquid, oxidizing, n.o.s. (nitric acid, hydrogen peroxide)

**NOTES:** This sampling procedure is provided only as a guide to sampling gas from a sampling port. It assumes a representative sample flow of the source gas can be obtained. It is the sampler's responsibility to ensure a representative sample. Any historical information regarding the sample would aid us in better analyzing your sample. This would include previous results of laboratory or field screening analyses.

Inerted materials may be obtained from Restek Corporation, [www.restek.com](http://www.restek.com). Their Sulfinert® or Siltek® lines have been successfully used.

It is the sampler's responsibility to ensure sampling is performed in a safe manner. Neither GTI nor any person acting on behalf of GTI assumes any liability with respect to the use of, or for damages resulting from the use of, any information presented in this procedure.