IN-PLACE TESTING OF OFF-GAS IODINE FILTERS

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Prepared for the U.S. Department of Energy
Assistant Secretary for Nuclear Energy
Office of Nuclear Waste Management
June 30, 1983

INTRODUCTION

At the Idaho National Engineering Laboratory, both charcoal and silver zeolite (AgX) filters are used for radioactive iodine off-gas cleanup of reactor systems. These filters are used in facilities which are conducting research in the areas of reactor fuel failure, reactor fuel inspection, and loss of fluids from reactor vessels. Iodine retention efficiency testing of these filters is dictated by prudent safety practices and regulatory guidelines. Current annual testing of AgX iodine off-gas filters involves two different phases. The first phase is a freon mechanical efficiency test. Items that can cause mechanical failure in a filter are voids in the filter media, seal failures, settling of media, etc. The freon test can determine a 99.95% mechanical efficiency. The second phase involves iodine removal efficiency determination of the filter media. The media efficiency is determined in the laboratory by subjecting predetermined aliquots of the filter media to a RDT standard method.[1] The absolute efficiency is usually assigned the lowest efficiency value determined from the two tests. Only annual freon testing is performed on charcoal filters.

A procedure for determining iodine off-gas filter efficiency has been developed and tested on both AgX and charcoal filters. The procedure involves establishing sample points upstream and downstream of the filter to be tested. These sample points are then validated using helium dilution techniques.[2] After the sample points have been proven to be valid, sampling devices are installed at the sample points, and the filter is challenged with ^{131}I labled methyl iodide. The measured activity on the inlet and outlet sampler components is a direct determination of the filter efficiency.

DISCUSSION

The following discussion will detail a step-by-step approach for filter efficiency testing. There are five basic areas that will be addressed: sample point location, sample point validation, release of ^{131}I labeled methyl iodide, the use of the sampling devices, and the determination of the iodine off-gas filter efficiency.

In choosing the location of your sample points, several considerations need to be pointed out. For the best results the location of the sample points needs to be at least eight duct diameters from the last and next perturbation of the duct flow. Examples of perturbations are bends in the duct, filter housing, fans, feeder ducts entering the main duct, valves for ventilation control, heater units, etc. This rule is not always practical however and use of already existing sample points, modification of existing sample points, or else locating the sample points in less desirable locations is mandated. Also an injection point needs to be established upstream of the inlet sample point. This point needs to be far enough upstream to allow for good mixing in the duct. This injection point can be ventilation inlets, hoods, ports on the duct, etc. After these points have been established, the helium measurement can be started to validate these locations, i.e., do they allow for a representative sample of the ventilation gas concentrations.

A DuPont leak Detector, model 24-120D, is used in sample point validation because of its wide range of sensitivity to helium and its durability. A dual pen strip chart recorder with multispan input modules is used to record all responses. Figure 1 shows the filter assembly as prepared for measuring the helium concentrations at the sample points. The following section describes a step procedure for measuring helium concentrations at the various points.

1) Startup the leak detector and allow enough time for the instrument to stabilize.
2) Calibrate the leak detector for the response -vs- system pressure using three or more He standards of different concentrations and using four or more system pressures for each standard. (Note that the system pressure refers to pressure changes made to the leak detector mechanism and not to the environmental or ventilation pressure.) See Figure 2 for a typical response -vs- pressure records for standards.
3) Inject He at the injection point at a rate that will give a ventilation

concentration bounded by the range of standards used for calibration.

4) Measure the ventilation He concentration at the filter inlet and outlet sampling points. The system pressure should be maintained at a level which gives good He response. See Figure 3 for response -vs- pressure records for an actual run.

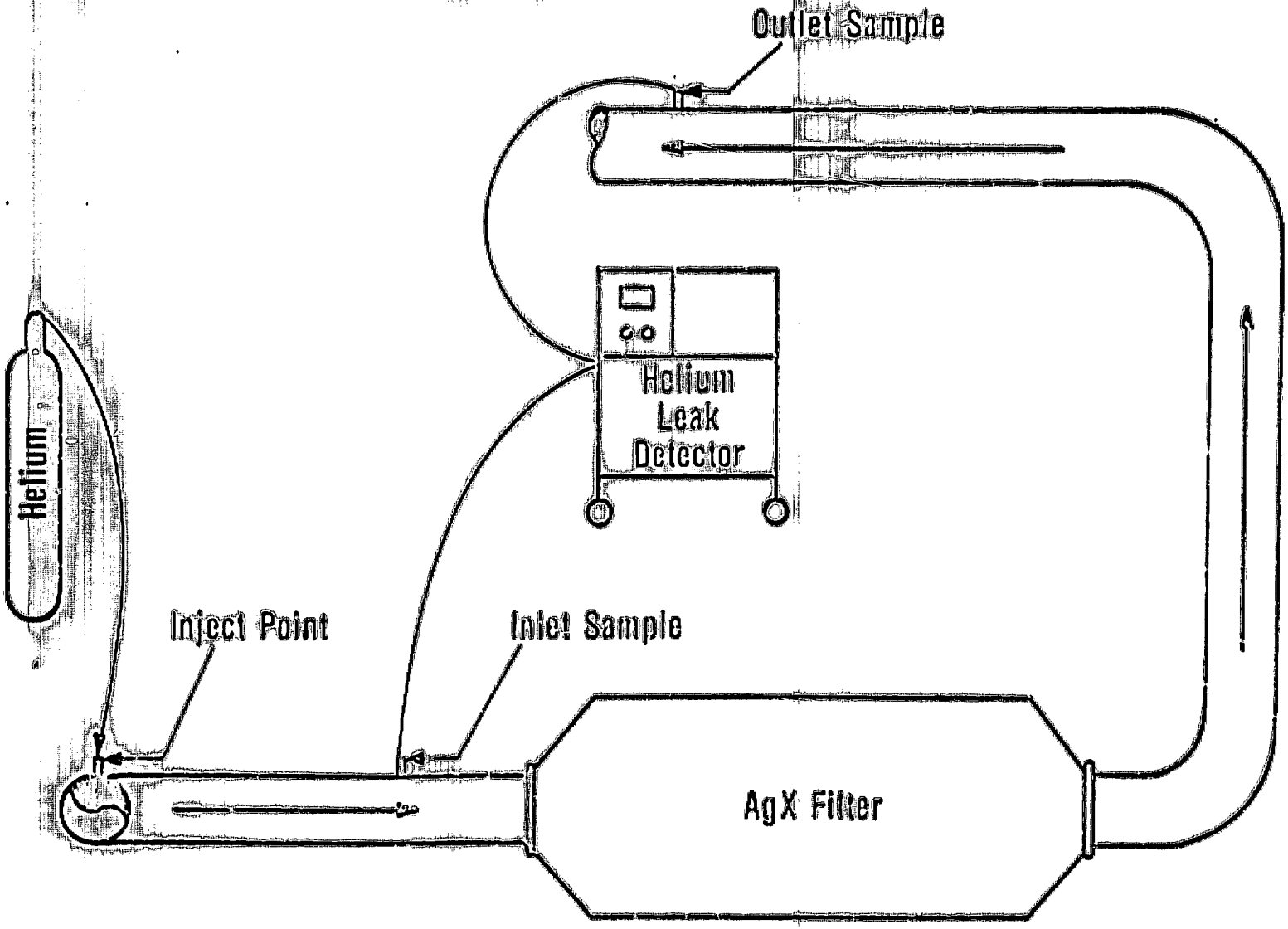
5) Calculate the sample point flowrate at the inlet and outlet using the following equation: Flowrate = He injected / He measured ppm. The measured ppm He is found by plotting pressure -vs- response curves from the standards and then plotting ppm -vs- response for a given pressure. See Figure 4 and 5 for examples of these graphs. The inlet and outlet calculated flows should agree within 10% for valid sampling locations.

After determining that the injection and sample points are valid, proceed with the next phase, make-up of the ^{131}I methyl iodide ($CH_3{}^{131}I$).[3] The amount of ^{131}I methyl iodide that needs to be generated depends upon the concentration desired in the ventilation system to be tested, the expected efficiency of the adsorber filter, and the sensitivity of the samplers and counting equipment used. The methyl iodide is generated by introducing a 0.5N NaOH solution, which contains the $^{131}I^-$, into a reaction vessel containing 1 mL of dimethyl sulfate and 0.5 gms $CaCO_3$ powder. (See Figure 6 for a schematic of the generation device.) A low helium purge (approximately 10-20 cc/min) is used as a carrier gas for transporting the $CH_3{}^{131}I$ out of solution and over to the evacuated transport cylinde . To purify the gas stream a small amount of IpH is used between the reaction vessel and the cylinder. The IpH is an adsorbent media which will selectively adsorb I_2 and HOI forms of iodine that may be generated in the reaction vessel. After the reaction is complete, as determined by readings on a GM instrument, the transport cylinder is isolated from the reaction vessel and is prepared for shipping to the work location.

With the upstream and downstream sampling devices installed, the test of the adsorbent filter can proceed. The transport cylinder containing the radiolabeled methyl iodide is pressurized to around 50 psig, and is then attached to the injection point. The sampling devices are started

FILTER ASSEMBLY

as prepared for He Work



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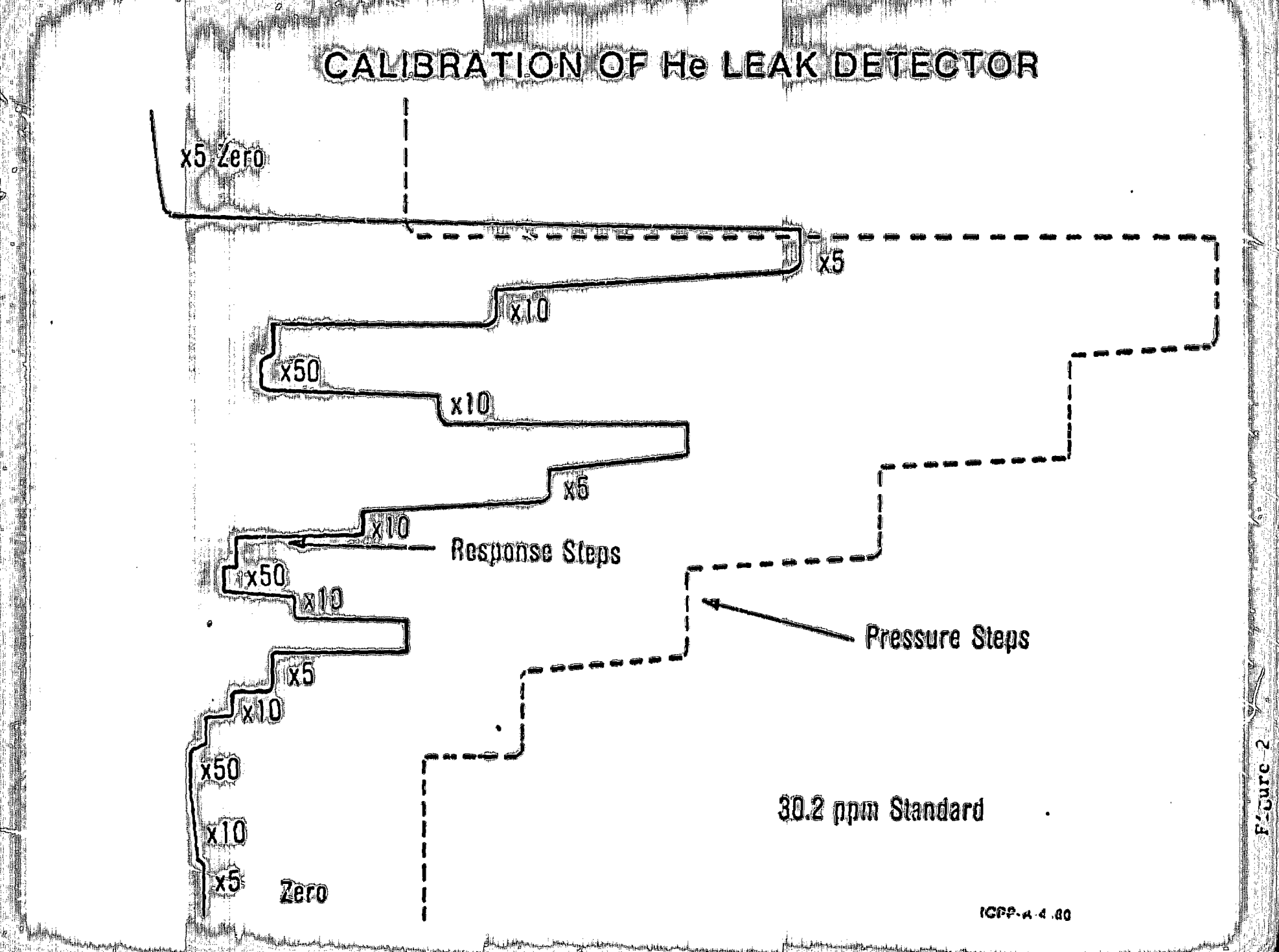
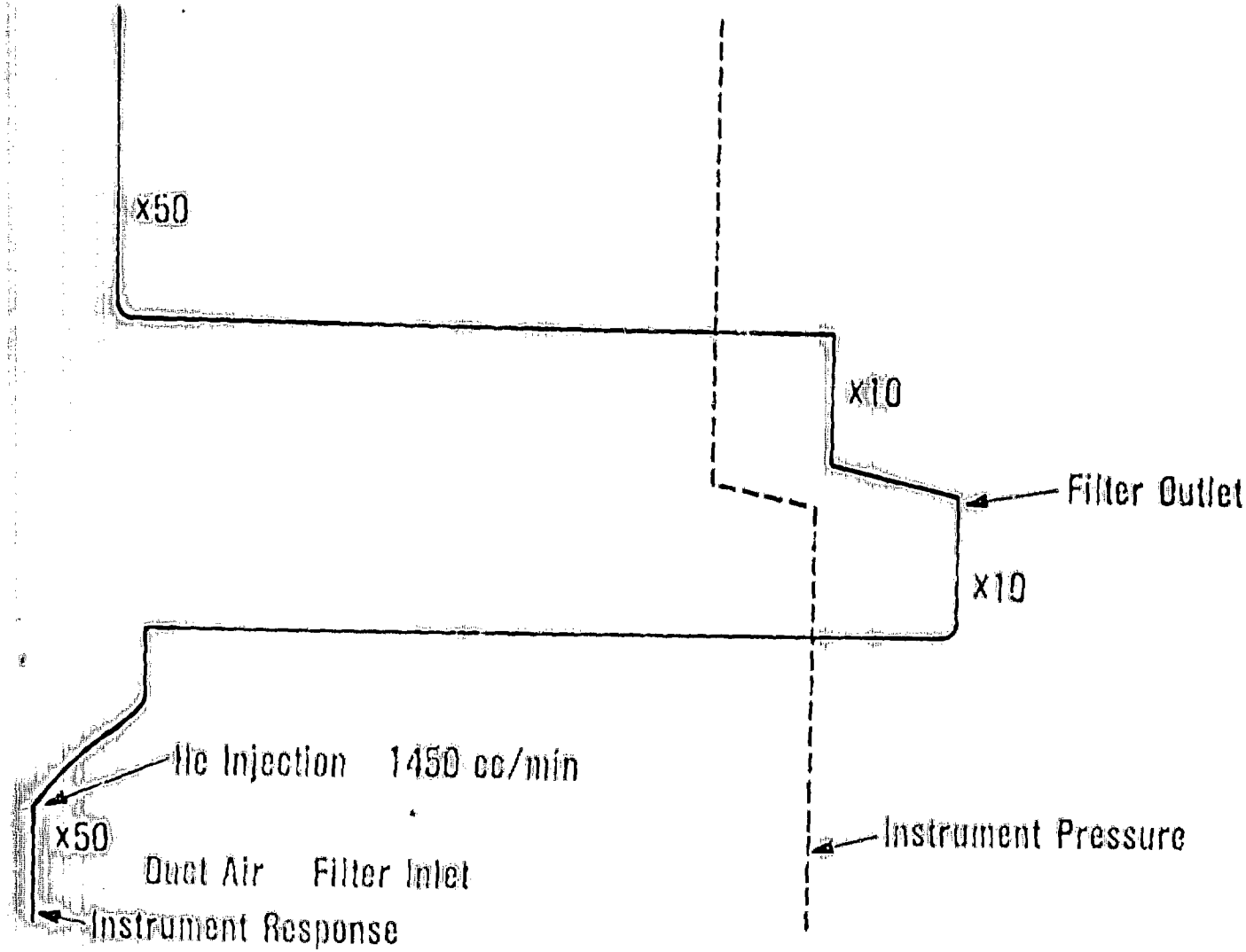

CALIBRATION OF He LEAK DETECTOR
x5 Zero
x5
x10
x50
x10
x5
x10
Response Steps
x50
x10
x5
x10
x50
x10
x5
Zero
Pressure Steps
30.2 ppm Standard

Figure 2

VERIFICATION OF SAMPLE POINTS
FILTER F-12



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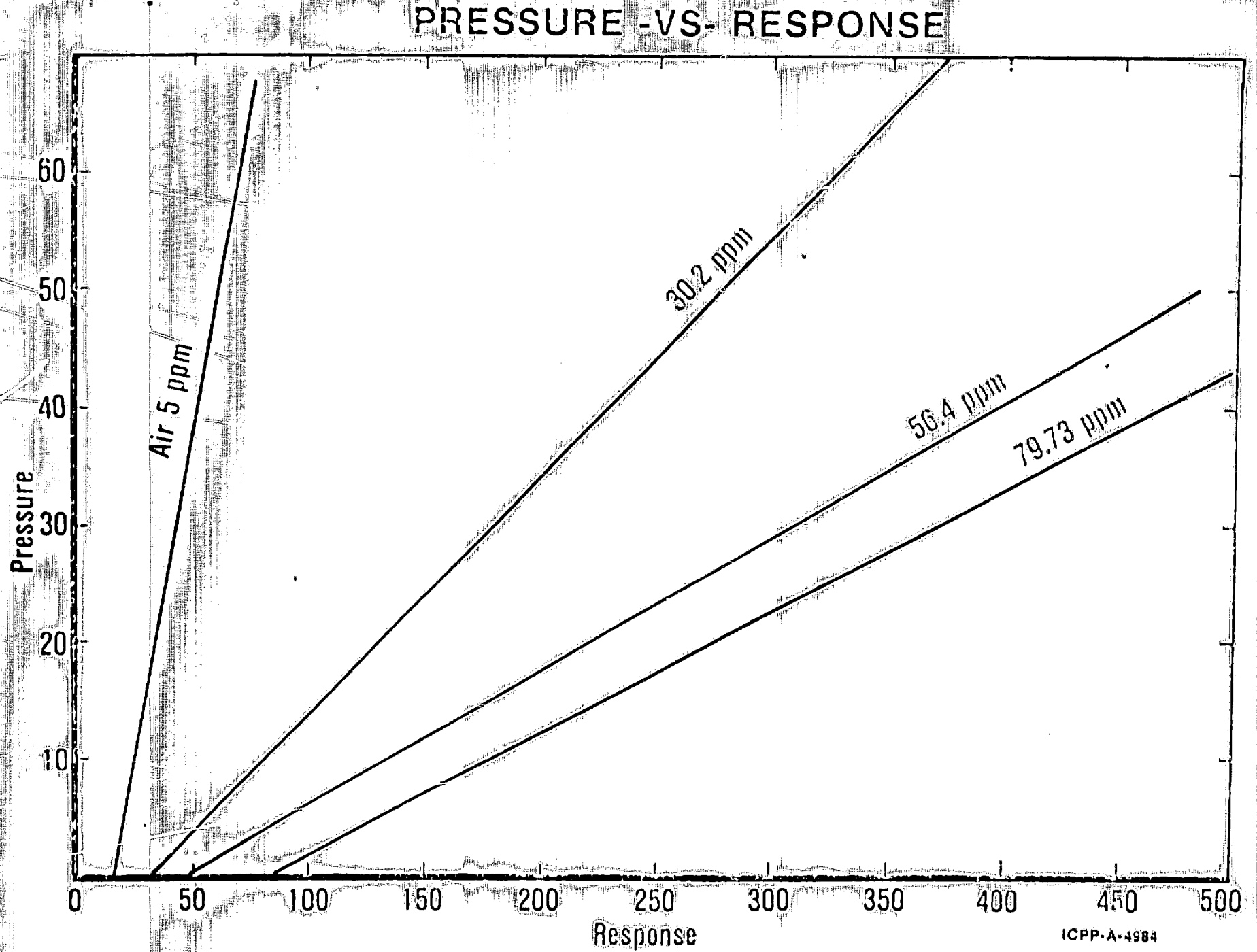

PRESSURE -VS- RESPONSE
Pressure
60
50
40
30
20
10
Air 5 ppm
30.2 ppm
56.4 ppm
79.73 ppm
0
50
100
150
200
250
300
350
400
450
500
Response
ICPP-A-4984

Figure 4

He ppm -VS- RESPONSE
He ppm
0
20
40
60
80
100
Response
0
100
200
300
400
500
600
700
Pressure @ 25
28
30
34
ICPP-AV-4983

Figure 5

and the $CH_3{}^{131}I$ is injected at a flowrate that will allow injection for approximately thirty minutes. At the end of the injection the transport cylinder is isolated and the sampling devices are allowed to run for approximately two hours longer so any breakthrough can be determined. At the end of the sampling time the samples are isolated and taken to a gamma counting facility.

The sampling equipment used is composed of a 5 cfm rotary vane gas pump, two flowmeters, two 0-30 inches water vacuum gauges, and the two sampling devices.[4] Figure 7 shows a schematic of half the total assembly while Figure 8 shows a schematic of the filter assembly as prepared for efficiency testing.

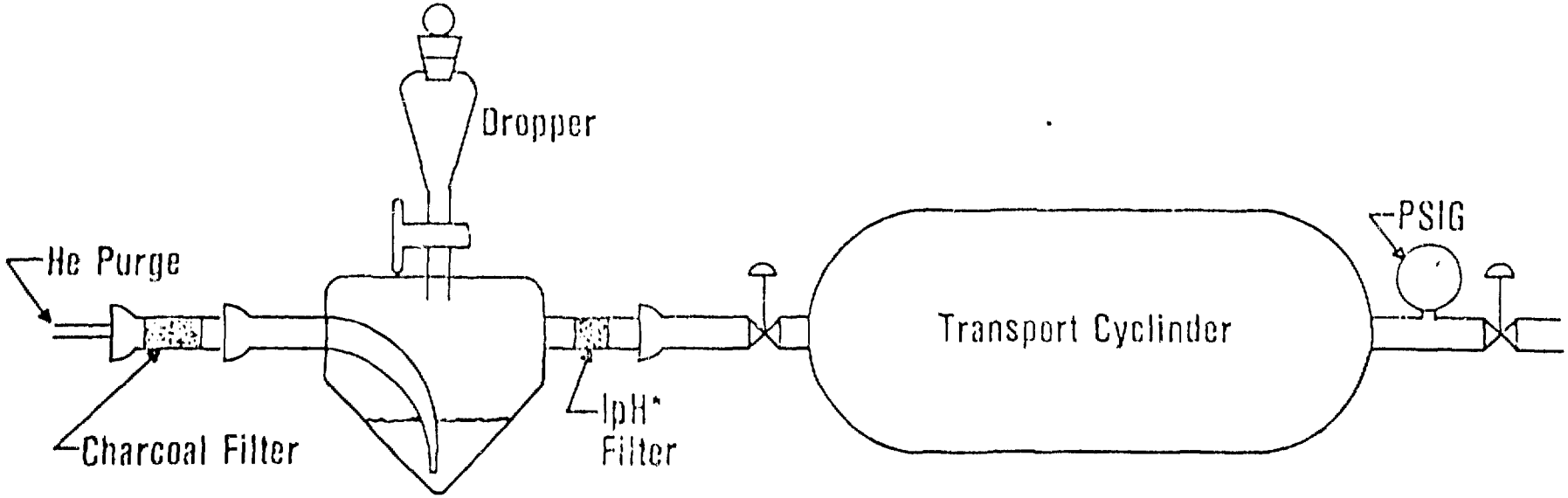
The inlet and outlet sample components are counted on a Ge(Li) detector to determine the μCi of ^{131}I which was adsorbed on each component. The absolute iodine filter efficiency can then be determined by using the following equation:

$$\text{Filter Efficiency} = \left[1 - \frac{\text{filter outlet } \mu\text{Ci}}{\text{filter inlet } \mu\text{Ci}}\right] \times 100$$

If different flowrates are used on the inlet and outlet samplers, it will be necessary to convert μCi to μCi/cc before using the above formula. If this is necessary, use only the flow in cc for the time during injection and not the flow in cc for the total time the samplers were operating as this will give erroneous results.

CONCLUSION

A method for determining iodine adsorption filter efficiencies in-place has been developed and shown to be effective for different filter configurations as well as different flowrates. The range of efficiencies determined by this method has been from 99.98% to as high as 99.99999%. Once helium dilution techniques have shown hard plumbed sample locations to be valid this procedure can be easily repeated on an annual basis. This procedure is economical



$$^{131}I^{-} + CH_3 \bullet \xrightarrow[\Delta]{CaCO_3} CH_3I^{131}$$

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*Iodophenol on Alumina 5% by weight

and can determine actual filter efficiency in-place as compared to current practice of laboratory testing of the filter media, and in-place freon testing of the filter assembly for mechanical failures.

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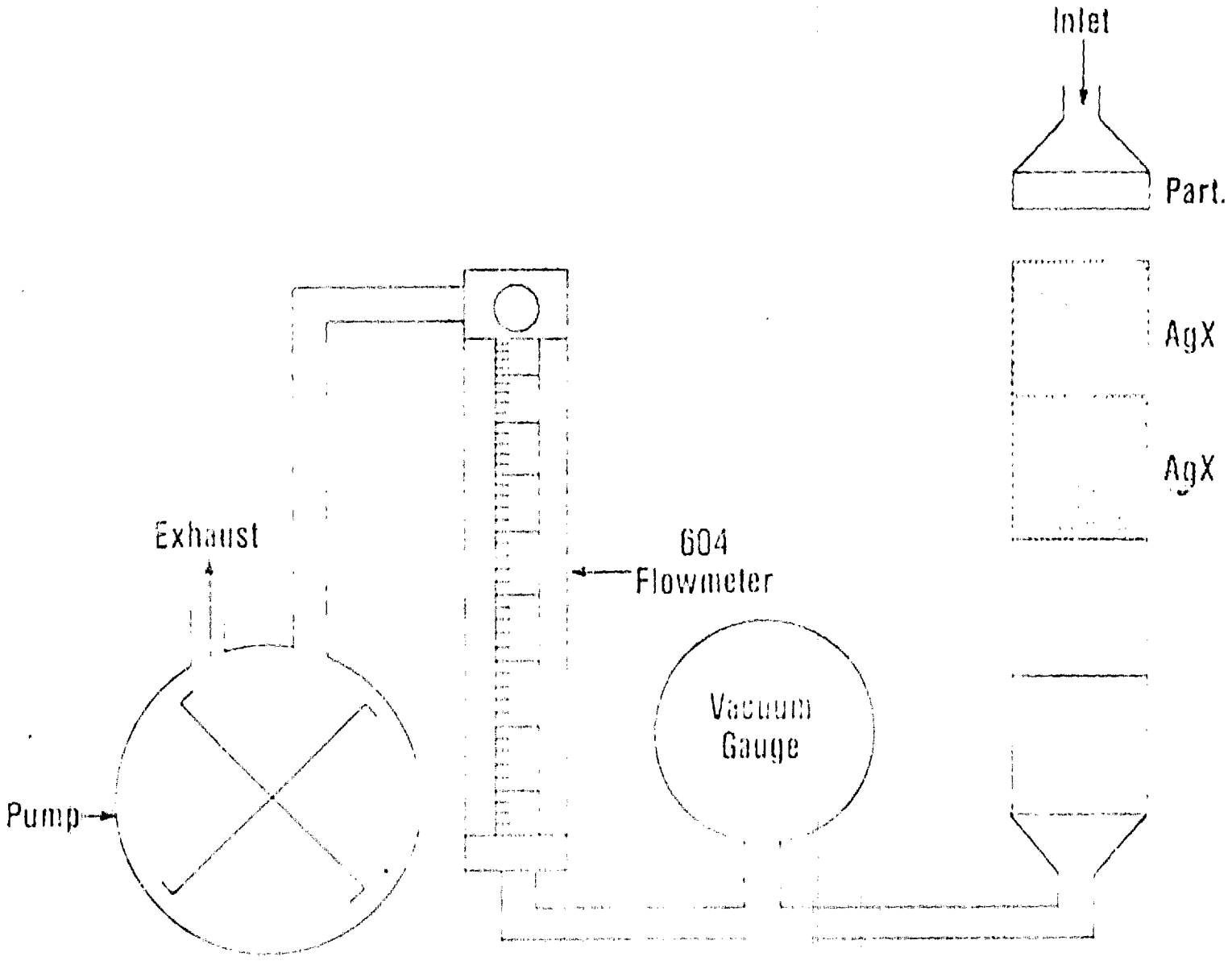

Iodine Sampler System

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FIGURE 7

-12-

FILTER ASSEMBLY
as prepared for Filter Test

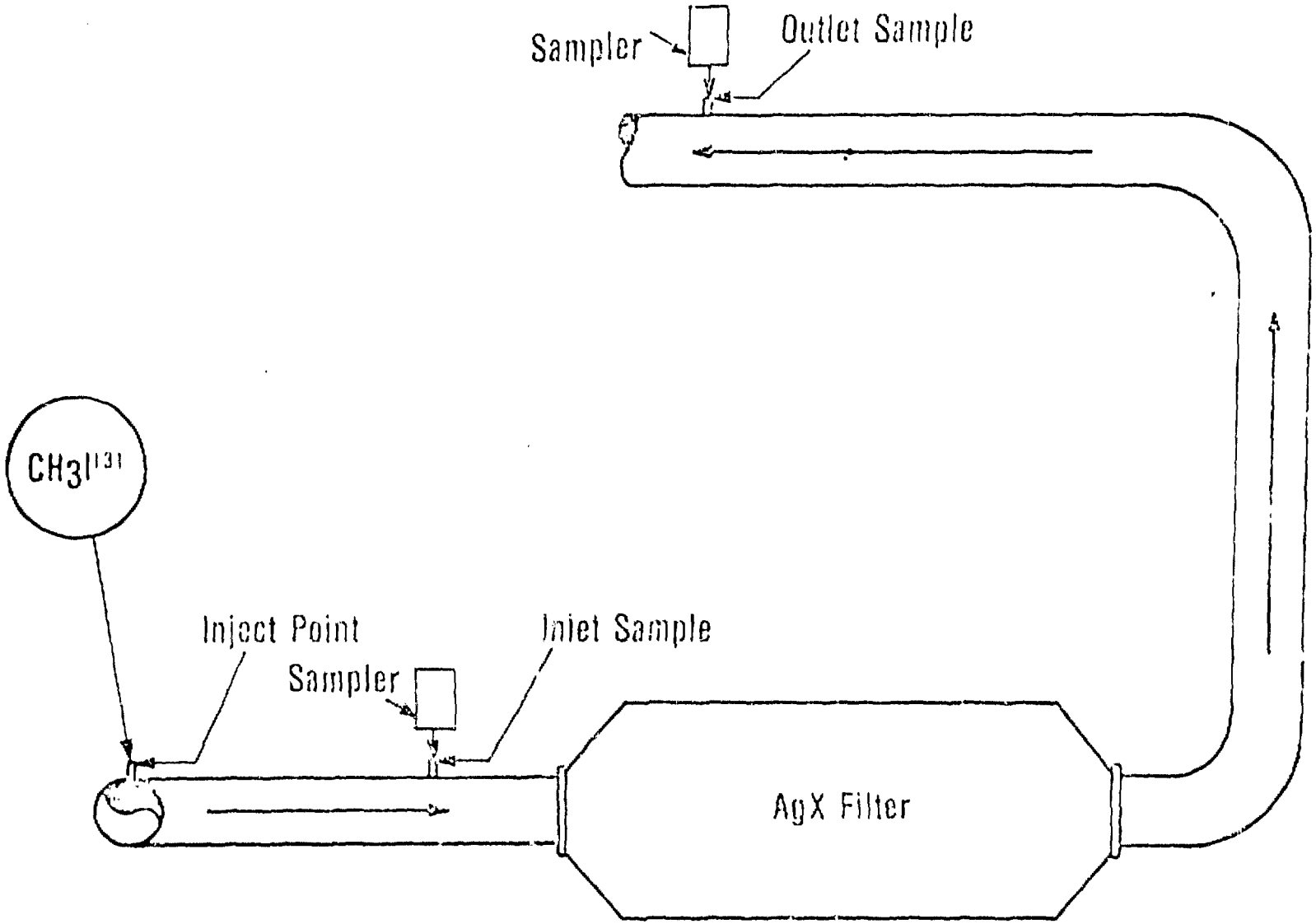

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Figure 5