SEMI F30-0298
START-UP AND VERIFICATION OF PURIFIER PERFORMANCE TESTING FOR TRACE GAS IMPURITIES AND PARTICLES AT AN INSTALLATION SITE

1 Purpose

1.1 The purpose of this procedure is to verify the performance of purifiers by employing analytical instrumentation to measure gas impurities and particles to customer specifications. If specific inlet challenge(s) and/or inlet measurements are required, it should be discussed beforehand with the customer. Inlet impurities must be measured by part-per-million (PPM) or part-per-billion (PPB) analytical equipment. This procedure applies only to large scale bulk purifiers rated at greater than 50 liters-per-minute (LPM) flowrate.

2 Scope

2.1 Verify performance of large scale purifiers in nitrogen, argon, helium, oxygen, and hydrogen service. Verification tests are done at PPB or sub-PPB levels of gaseous impurities and sub-micron sizes of particles measured downstream of any installed filter modules. Tests are done at maximum achievable flow of purifier, and/or customer's specified percentages of maximum flow.

3 Limitations

3.1 PPB and sub-PPB gaseous impurity levels are achievable using atmospheric presssure ionization mass spectrometry (APIMS), which is the preferred method of choice, and the reduction gas detector (RGD)-gas chromatograph. APIMS is currently not available for oxygen service. A partial list of non--APIMS measuring equipment for use in oxygen service is in Appendix 1 for commonly measured impurities.

4 Referenced Documents

4.1 Approved procedures for operation of analytical equipment.

4.2 Approved gas sampling and purifier procedures.

5 Terminology

5.1 None.

6 Summary of Method

6.1 The purifier is started, and the operation is checked. Each purifier bed is regenerated, and analytical tests are done. The analytical results determine if the purified gas meets the customer specifications.

7 Interferences

7.1 The following sources might contribute to misleading or high analytical results. Some identified sources are unpurged, dead-ended piping or isolation valves, leaks in gas distribution system, insufficient purge flow, and insufficient system clean-up time.

8 Apparatus

8.1 Face seal fitting(s) with metal gaskets and stainless steel tubing for sampling.

8.2 Dynamic dilution system for diluting calibration standards to PPB and sub-PPB levels for calibration of analytical equipment.

8.3 *APIMS* — The sample gas, nitrogen, is introduced into an APIMS where a small amount of it is ionized. By collision with ionized nitrogen, impurity molecules are ionized with high efficiency. The mass analyzer, which can be a quadrupole, time-of-flight (TOF), or even a magnetic sector, separates and focusses the ions by their mass-to-charge ratio. An electron multiplier detects and counts each ion fragment and amount.

8.4 *Reduction Gas Detector-Gas Chromatograph (RGD-GC)* — The reduction gas detector is a heated mercuric oxide bed that reacts with reducing gases, such as hydrogen and carbon monoxide. The mercury evolved is detected and displayed as a peak. The GC employs a heated molecular sieve column to separate the H_2 and CO.

8.5 *Ultratrace Analytical Instrumentation Required other than APIMS* — Ultratrace instrumentation is defined as having sufficient sensitivity to measure all impurities of interest at the specified level of the customer at the PPB or sub-PPB.

8.6 Particle counter.

8.7 Data collection and reduction system.

9 Reagents and Materials

9.1 Certified calibration standards.

10 Safety Precautions

10.1 The testing area should have adequate room ventilation and atmospheric monitors.

10.2 Instruments should be exhausted to vent, and if required in Class 1 environments, should be case-purged or in an approved enclosure.

10.3 Testing personnel should be aware of customer alarm and evacuation procedures.

10.4 Designated customer contact required during testing.

11 Sampling

11.1 Test each purifier bed independently to verify performance to customer specifications. Refer to gas supplier's certificate of conformance for inlet impurity levels. Refer to Figure 1 for overall test sequence.

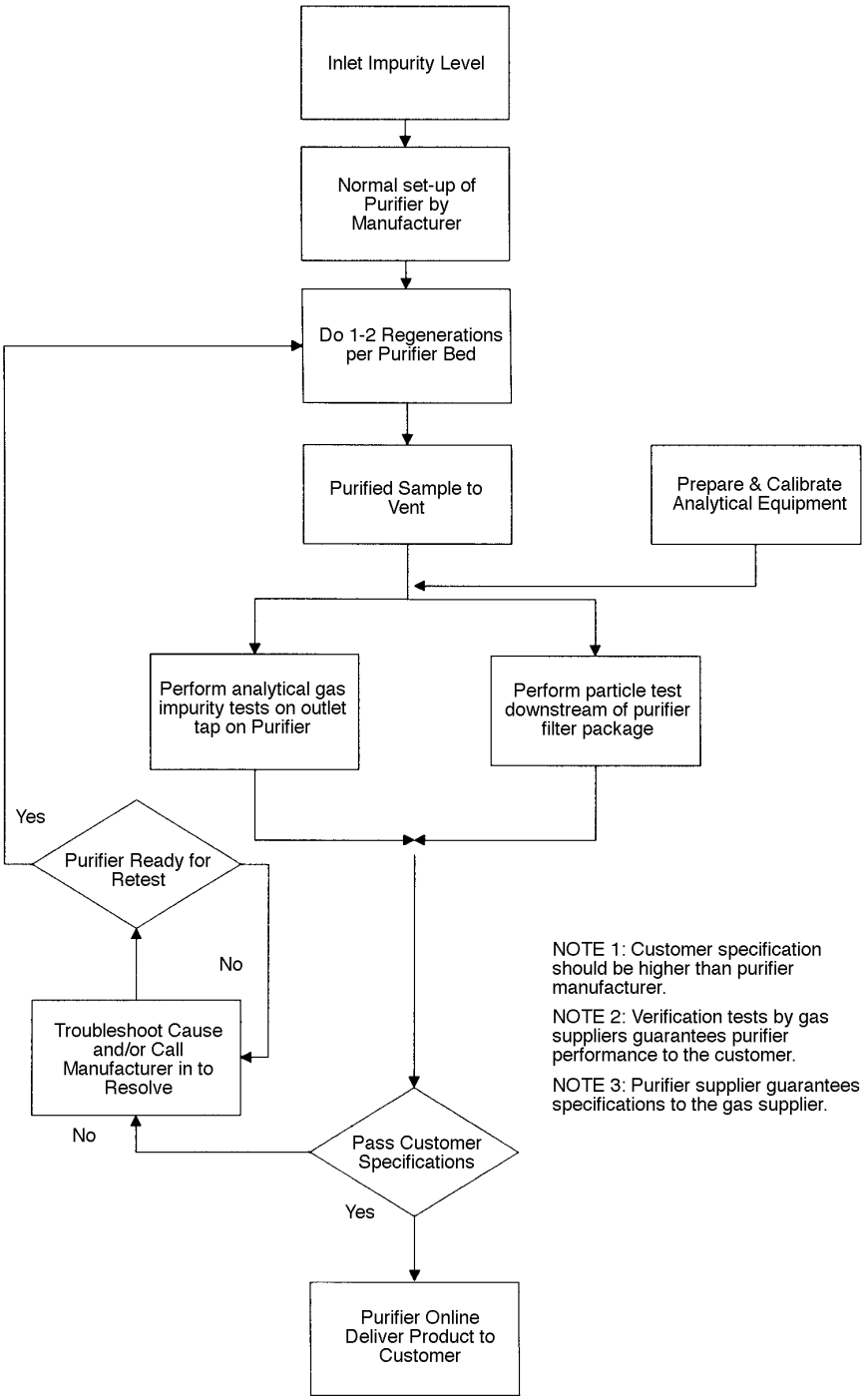

Figure 1
Overall Test Sequence

12 Preparation of Apparatus

12.1 *Sampling System* — Use appropriate clean tubing and fittings, or clean before use. Purge system prior to sampling.

12.2 *Dynamic Dilution System* — Use certified standards and dilute using equation (1) to calculate final concentration (C_F). For moisture, certified moisture permeation devices can be used.

(1) $C_c \times D.F. = C_F$

C_c = PPM- or PPB -certified cylinder standard concentration.

C_F = final concentration.

D.F.= dilution factor which is calculated by taking the flow (in liters) of the certified standard and dividing by total flow (in liters). Dilution factors can be multiplied in series, if diluting more than once.

Example 1: What is the final concentration for dynamically diluting 10 ml of a 100 PPB impurity into 1 liter?

$$100 \text{ PPB} \times [0.010 \text{ liter}/1.010 \text{ liter}] = 0.99 \text{ PPB}$$

12.2.1 Calibration should be done in the region specified by the purifier manufacturer. For example, if the purifier has an outlet impurity guarantee of 1 PPB of each impurity, then the test equipment should be calibrated with levels at approximately 1 PPB, not at 30 PPB and extrapolated down. Also, multipoint calibration data is preferred.

12.3 APIMS, RGD-GC, ultratrace analytical instrumentation. Particle counter.

12.3.1 Start-up and purge the instrumentation. Perform calibration. Determine if calibration is satisfactory. Proceed to sampling section.

12.4 *Data Collection System* — Check for proper signal inputs, range inputs, and sampling intervals.

12.5 *Data Reduction System* — Prepare data as print-outs and/or graphs. Include statistical analysis as required. Generate final report.

13 Calibration and Standardization

13.1 See Section 12, Preparation of Apparatus.

14 Procedure

14.1 *Trace Gas Impurity Measurement*

14.1.1 Connect sample source to analytical equipment.

14.1.2 Start data collection.

14.1.3 Stop sampling. Review preliminary data. If it is within customer specification, disconnect sample source. If it does not meet specifications, investigate cause or refer to manufacturer literature to resolve. Once condition is corrected, repeat tests.

14.1.4 Repeat procedure for next bed or sampling point. If instrumentation is relocated, calibration check is required.

14.2 *Particle Counting*

14.2.1 Select sample location. Ideal location is a permanently installed pitot probe.

14.2.2 Select where the particle tests will be done on the pipe.

14.2.3 Use Reynold's equation to determine the required gas rate for turbulent flow to the particle counter. Use this value or higher for sampling purposes. Reynold's number greater than 2100 are suggested for turbulent flow.

14.2.4 Reduce incoming sample pressure to the particle counter by following manufacturer's recommendation or by best practice.

14.2.5 Test particles with turbulent flow through pipeline, if possible. However, do not exceed the manufacturer's maximum rated flow for the purifier.

14.2.6 Start data collection.

14.2.7 Stop sampling. Review preliminary data. If it is within customer specification, disconnect sample source. If it does not meet specifications, investigate cause or refer to manufacturer's literature to resolve. Once condition is corrected, repeat tests.

14.2.8 Repeat procedure for next bed or sampling point.

15 Calculations or Interpretation of Results

15.1 Results are interpreted by trending analysis, averaging, or steady-state analysis.

16 Reporting Results

16.1 Sample location.

16.2 Operator identification.

16.3 Test parameters and conditions (pressures, flowrates, temperatures, etc.).

16.4 Test date and duration.

16.5 Description of instrumentation.

16.6 Calibration information for analyzer(s).

16.7 Report test results by data table and/or graphs.

16.8 Comments on testing.

16.9 Conclusion.

APPENDIX 1

NOTE: This appendix was approved as an official part of SEMI F30 by full letter ballot procedure.

Partial List of Analytical Equipment for Use in Oxygen Service	
Impurity	Analytical Equipment
H_2O	Dewpoint Detection Electrolytic Peizoelectric Capacitance (aluminum oxide, silicon array) Fourier Transform Infrared (FTIR)
CH_4, Total Hydrocarbons, Nonmethane Hydrocarbons	Flame-Ionization Detector-Gas Chromatograph (FID-GC) Discharge Ionization Detector (DID)-GC
CO	Reduction Gas Detector (RGD)-GC Nondispersive Infrared (NDIR) DID-GC
CO_2	Methanator on FID-GC DID-GC NDIR
H_2	RGD-GC
Particles	Special counters required for O_2 service: for 0.01 µ or greater, condensation nucleus counter for 0.1 µ or greater, laser counter