

SEMI F26-0697 (Reapproved 0303) PARTICLE SPECIFICATION FOR GRADE 10/0.2 TOXIC SPECIALTY GASES

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1 Purpose

1.1 The purpose of this document is to set a maximum permissible particle concentration for 10/0.2 grade toxic specialty gases and to describe a reference method for its verification.

2 Scope

2.1 This document applies only to toxic gases delivered through specialty gas systems at pressures up to 8×10^5 Pa (8 atmospheres). This method is not suitable for direct sampling from high pressure cylinders at pressures above 8×10^5 Pa (8 atmospheres). This document applies only to the following gas:

Ammonia (NH ₃)
Arsine (AsH ₃)
Phosphine (PH ₃)

2.2 This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the users of this standard to establish appropriate safety and health practices and determine the applicability of regulatory or other limitations prior to use.

3 Referenced Standards

3.1 Japanese Industrial Standard¹

JIS B 9921 — Japanese Industrial Standard (1989), “Light Scattering Automatic Particle Counter”

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

4 Terminology

4.1 Variables

V_{Mi}	Volume of the i^{th} sample interval of the system gas
V_{Bi}	Volume of the i^{th} sample interval of the background
X_{Mi}	Concentration of particles observed in the i^{th} sample of

	interval of the system gas
X_{Bi}	Concentration of particles observed in the i^{th} sample of interval of the background
N_M	Number of sample intervals of the system gas
N_B	Number of sample intervals of the background
$\bar{X}_M$	Average observed concentration of counts in the system gas sample
$\bar{X}_B$	Average observed concentration of background counts
$\bar{X}_C$	Calculated concentration of particles in the system gas
S_M	Standard deviation of $\bar{X}_M$
S_B	Standard deviation of $\bar{X}_B$
S_C	Standard deviation of $\bar{X}_C$

4.2 *gas sample volume* (V_{Mi} , V_{Bi}) — The volume of the sample interval, expressed in standard liters at standard conditions, 0°C (32°F) and 1×10^5 Pa (1 atmosphere) pressure. Standard cubic feet (SCF) is defined at 21.1°C (70°F) and 1×10^5 Pa (1 atmosphere) pressure.

4.3 *average observed concentration of counts* ($\bar{X}_M$, $\bar{X}_B$) — The average concentration of counts, i.e.:

$$\bar{X}_M = \frac{\sum X_{Mi}}{N_M} \quad \bar{X}_B = \frac{\sum X_{Bi}}{N_B}$$

4.4 *calculated concentration of particles* ($\bar{X}_C$) — The concentration of particles in the system gas obtained by correcting the observed concentration in the system gas for the observed concentration in the background, i.e.:

$$\bar{X}_C = \bar{X}_M - \bar{X}_B$$

4.5 *standard deviation* (S_M , S_B , S_C) — A statistical measure of the spread of the concentration of the counts or particles. The first two are obtained from the interval and average concentrations and the number of intervals, i.e.:

¹ Japanese Industrial Standards, Available through the Japanese Standards Association, 1-24, Akasaka 4-Chome, Minato-ku, Tokyo 107-8440, Japan. Telephone: 81.3.3583.8005; Fax: 81.3.3586.2014 Website: <http://www.jsa.or.jp>

$$S_M = \sqrt{\frac{\sum (X_{Mi} - \bar{X}_M)^2}{(N_M - 1)}}$$

$$S_B = \sqrt{\frac{\sum (X_{Bi} - \bar{X}_B)^2}{(N_B - 1)}}$$

NOTE 1: The third is obtained from the first two, i.e.:

$$S_C = \sqrt{S_M^2 + S_B^2}$$

5 Requirements

5.1 *Maximum Permissible Particle Concentration* — 10 particles per 25 standard liters as determined by the instrument specified in Section 6.

5.2 The specification will be considered met if the calculated concentration of particles plus 2 times the standard deviation does not exceed 10 particles per 25 standard liters, i.e.:

$$\bar{X}_C + 2 \times S_C \leq 10 \text{ particles/25 standard liters}$$

6 Apparatus

6.1 *Particle Counter* — An instrument suitable for counting particles in compressed toxic gases at a pressure of up to 8×10^5 Pa (8 atmospheres) with a 50% counting efficiency should reach 90% at 0.3 micrometer or smaller. The resolving power of the instrument near 0.2 micrometer should be no worse than 10%. The counting efficiency is determined by a calibration at 1×10^5 Pa (1 atmosphere) pressure using polystyrene latex spheres in an inert gas and a reference particle counter with a proven counting efficiency of not less than 95% at 0.2 micrometer.

NOTE 2: Suitable test methods for determining counting efficiency and resolving power are contained in Japanese Industrial Standard JIS B 9921 (1989), "Light Scattering Automatic Particle Counter". More sensitive particle counters result in a higher measured particle concentration.

7 Test Method

NOTE 3: The details of the sampling configuration, measurement procedure, and instrument calibration procedure and frequency must be agreed upon by the user and supplier, taking into account good engineering practice. Material safety data sheets should be referred to for safe handling of specialty gases.

7.1 Determine the average observed concentration of counts in the background ($\bar{X}_B$) by passing nitrogen, believed to be free of particles 0.2 micrometer or more in diameter, through the instrument and recording the total number of counts. The nitrogen purge assembly

for performing this test, using a filter which removes particles in this size range, is shown in Figure 1. Count a minimum of 3 equal intervals, each of at least 25 standard liters (0.95 SCF), or 30 minutes, whichever is greater. Calculate $\bar{X}_B$ as defined in Section 4. $\bar{X}_B$ must not exceed 2 counts per 25 standard liters.

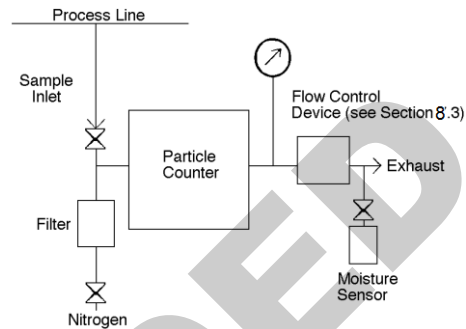


Figure 1
Schematic Diagram of Configuration for Obtaining Particle Samples from Toxic Specialty Gas Systems

7.2 The sampling point should be near the point of use, and sampling lines should be clean and as short as possible.

7.3 The sampling system configuration is shown in Figure 1. The specialty gas system should be connected through a sample valve directly to the particle counter, a pressure gauge, and a flow control device (FCD). When sampling is performed using wall tap sample ports, the aspiration efficiency should be checked using the method described in Related Information 1. The sampling system should contain minimum dead volume and sample tube length. All components in the sampling system should be leak tight and cleaned in accordance with good engineering practice. A leak check of the system should be performed in accordance with normally accepted practice. The FCD may be part of the particle counter. The particle counter should be maintained at the pressure of the gas system. The FCD can be a metering valve and a flow meter or a critical orifice. The exhaust line should be leak tight and piped to an appropriate exhaust system. The exhaust line diameter should be large enough to produce no more than 1×10^4 Pa (1.5 psi) pressure drop during purge and sample flow.

7.4 Using the high purity nitrogen system shown in Figure 1, purge the sampling system and particle counter for at least 5 minutes at the instrument manufacturer's specified flow rate. The exact purge time should take into account the reactivity of the gas and should be sufficient to purge the entire exhaust line. A moisture sensor should be placed at the system exhaust to verify less than 1 ppmv water in the purge