

SEMI C6.7-93 (Reapproved 1102)

PARTICLE SPECIFICATION FOR GRADE 10/0.2 NITROGEN IN HIGH PRESSURE GAS CYLINDERS

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1 Purpose

1.1 The purposes of this document are (1) to set a maximum permissible particle concentration for 10/0.2 grade cylinder nitrogen, and (2) to describe a reference method for its verification.

2 Scope

2.1 This specification applies to nitrogen contained in high pressure gas cylinders; it is not applicable to pipeline gases.

2.2 A nitrogen cylinder consists of three components: (1) a cylinder bottle, (2) compressed nitrogen contained within, and (3) a cylinder valve. Each component can be a particle source. This specification applied to the total number of particles detected in the gas as obtained from the cylinder under a prescribed condition. No consideration is given to the origin of the particles.

2.3 It is known that pressure reduction, if not controlled, can produce a large number of artifact particles through nucleation and condensation. To avoid this complication, this specification adopts particle counters that can be operated at a pressure up to 200 bar (3000 psi), eliminating the need for pressure reduction and its associated problems.

2.4 It is known that particle content in cylinder gases varies with time because particles can be lost to cylinder walls by diffusion or sedimentation, and detached from cylinder walls by flow pulses or mechanical shocks. It is important to measure particle concentration under the worst conditions which represent typical handling of gas cylinders. This specification describes a standard shock test that fulfills the above requirements and provides a procedure to count particles immediately after the shock.

2.5 If this test method is to be used for more than one cylinder, then each cylinder must be tested. It is known that particle contamination in cylinder gases is a strong function of the handling history of the individual cylinders. Cylinders in the same batch of filling are usually returned from various customers after various periods of service. Uniform quality can not be assumed for the same batch of cylinders unless each of them has gone through a dedicated process that erases the

memory of previous history prior to filling. Therefore, batch sampling at 10% or 20% cannot be accepted because of the significant differences among cylinders.

2.6 The requirement of 100% sampling restricts the total amount of gas in each cylinder that can be used for sampling purposes. This restriction, in turn, calls for certain relaxation of the statistical requirement for particle sampling at low concentration levels.

2.7 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 health practices and determine the applicability or regulatory limitations prior to use.

3 Terminology

3.1 Variables

X_{Bi}	Observed particle concentration in the i^{th} sample interval of the background
X_{Mi}	Observed particle concentration in the i^{th} sample interval before shocks
X_{Ni}	Observed particle concentration in the i^{th} sample after shocks
N_B	Number of sample intervals of the background
N_M	Number of sample intervals before shocks
N_N	Number of sample intervals after shocks
$\bar{X}_B$	Averaged observed particle concentration of the background
$\bar{X}_M$	Averaged observed particle concentration before shocks
$\bar{X}_N$	Averaged observed particle concentration after shocks
X_P	Calculated particle concentration before shocks
X_Q	Calculated particle concentration after shocks
S_B	Standard deviation of $\bar{X}_B$
S_M	Standard deviation of $\bar{X}_M$
S_N	Standard deviation of $\bar{X}_N$
SE_P	Standard deviation of X_P
SE_Q	Standard deviation of X_Q

3.2 Averaged Observed Concentration (X_B , X_M , X_N)

$$\bar{X}_B = \text{SUM}(X_{Bi})/N_B$$

$$\bar{X}_M = \text{SUM}(X_{Mi})/N_M$$

$$\bar{X}_N = \text{SUM}(X_{Ni})/N_N$$

3.3 Calculate concentration (X_P , X_Q)

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

$$X_Q = \bar{X}_N - \bar{X}_B$$

3.4 Standard Deviation (S_B , S_M , S_N)

$$S_B = [\text{SUM}(X_{Bi} - \bar{X}_B)^2 / (N_B - 1)]^{1/2}$$

$$S_M = [\text{SUM}(X_{Mi} - \bar{X}_M)^2 / (N_M - 1)]^{1/2}$$

$$S_N = [\text{SUM}(X_{Ni} - \bar{X}_N)^2 / (N_N - 1)]^{1/2}$$

3.5 Standard Error (SE_P , SE_Q)

$$SE_P = [(S_M^2/N_M) + (S_B^2/N_B)]^{1/2}$$

$$SE_Q = [(S_N^2/N_N) + (S_B^2/N_B)]^{1/2}$$

4 Apparatus

4.1 High-Pressure Particle Counter (HPC) — An instrument capable of counting particles in compressed gaseous nitrogen at a pressure up to 200 bar (3000 psi), having a minimum counting efficiency of 50% at 0.2 μm and reaching 100% at 0.25 μm . This value is determined at ambient pressure by the instrument manufacturer using 0.2 μm monodisperse particles and a reference counter with a proven counting efficiency of 100% at 0.2 μm . Note that the nominal flow rate of the HPC is fixed at any pressure but the sample flow rate should be adjusted to within 5% of the manufacturer's specified flow rate, for the pressure and reported its equivalent standard flow rate at ambient pressure (1 bar).

4.2 Impact Shock Device — A device that can reproducibly impart an impact shock of 10^4 m/sec^2 with a 10% tolerance to a gas cylinder. A convenient set up as shown in Figure 1 can be used. A steel ball is attached to a chain with the other end fastened to the test cylinder. The ball is lifted to form a 90° angle with the cylinder, is released to follow a 90° free fall arc, and strikes the test cylinder. The desired ball mass and chain length are to be determined by monitoring the corresponding shock intensity by an accelerometer. For a typical 44 liter cylinder, a ball mass of 160 gm and a chain length of 60 cm can produce the desired impact shock.

5 Test Method

5.1 Determine the averaged background concentration ($\bar{X}_B$) by passing particle-free, compressed nitrogen through the HPC. Count a minimum of 5 sample intervals, each of at least 6 standard liters (0.2 SCF) or taken over a time period of 6 minutes, whichever is greater. A high purity gas filter can be used to remove particles greater than 0.2 μm . Calculate $\bar{X}_B$ as defined in Section 3. $\bar{X}_B$ must not exceed 1 particle per 30 standard liters.

5.2 Set up the experimental apparatus as shown in Figure 2. Directly connect the test cylinder to a High-pressure Particle Counter (HPC), a pressure gauge, and a Flow Control Device (FCD). Note that NO REGULATOR is used before the HPC. The sampling line should be clean and as short as possible. The flow control device can be a metering valve and a flowmeter or a critical orifice.

5.3 Determine $\bar{X}_M$ by opening the cylinder valve and count the particles for at least 5 sample intervals, each of at least 6 standard liters (0.2 SCF) or taken over a time of 6 minutes, whichever is greater. Calculate X_P and SE_P as defined in Section 3.

5.4 Apply 4 impact shocks of approximately 10^4 m/sec^2 each, 10 sec apart, to the bare surface of the test cylinder. Each shock should be applied at the approximate vertical midpoint of the cylinder; the four shocks should be separated from each other by approximately 90° circumferentially.

5.5 Determine $\bar{X}_N$ by counting the particles after the shocks for at least 5 sample intervals, each of at least 6 standard liters (0.2 SCF) or taken over a time of 6 minutes, whichever is greater. The sampling should be completed within 2 hours after the shocks. Calculate X_Q and SE_Q as defined in Section 3.

6 Specification

6.1 Maximum Permissible Particle Concentration — 10 particles per 30 standard liters.

6.2 The specification will be considered met if the calculated concentration of particles plus two standard errors does not exceed 10 particles per 30 standard liters for both measurements before and after the impact shocks, i.e.:

$$X_P + 2SE_P \leq 10 \text{ particles/30 standard liters}$$

and

$$X_Q + 2SE_Q \leq 10 \text{ particles/30 standard liters}$$