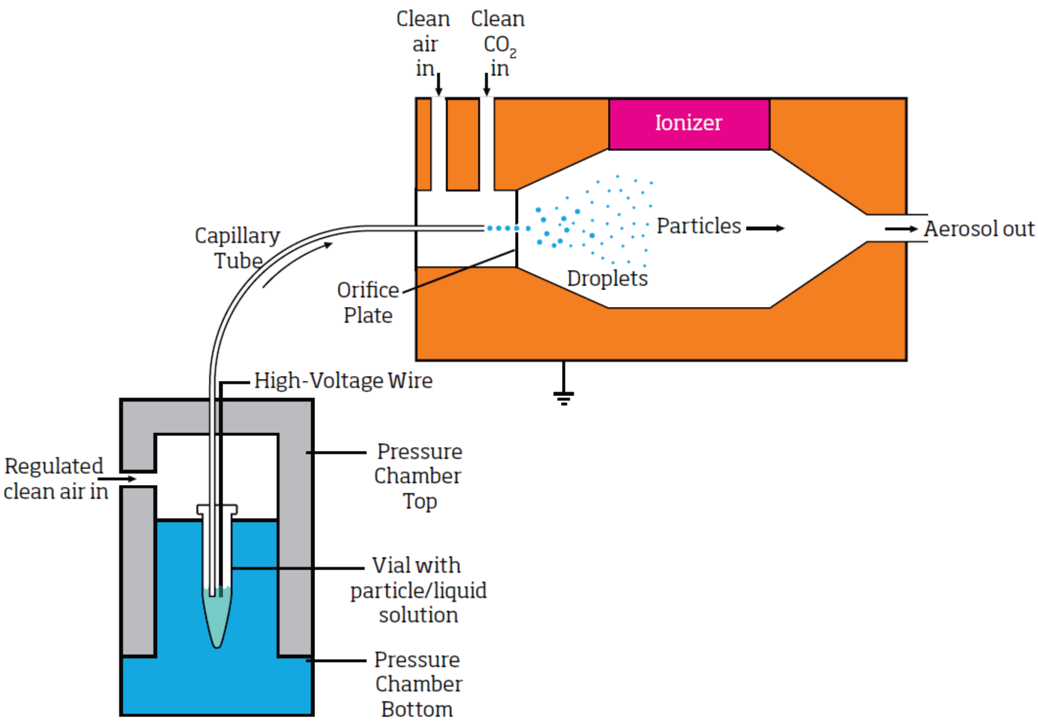
ELECTROSPRAY OPERATION USING NITROGEN IN PLACE OF AIR

APPLICATION NOTE 3480-002

Electrospray Aerosol Generator

The Electrospray Aerosol Generator (EAG) Model 3480 is used to generate monodisperse aerosol particles as small as 3 nm in diameter. The EAG pushes a charged liquid solution or suspension through a capillary tube and exerts an electrical field on the liquid at the capillary tip. Typical EAG operation uses a mixed flow of air and carbon dioxide (CO_2) to evaporate the liquid and transport the dried aerosol particles.



The EAG is used to generate a variety of nanoparticle and macromolecular aerosols for calibration, research, and quality-control applications. The use of air and CO_2 may not be suitable for air-sensitive samples. Therefore, the EAG was operated using nitrogen (N_2) in place of air, and the CO_2 flow was varied, to determine the limits of EAG operation using N_2 and CO_2.

Methods

The EAG was used as part of the LiquiScan-ES (Model 3980), which combines the EAG with a Scanning Mobility Particle Sizer™ Spectrometer (Model 3936) to measure particle size distributions. Dry, filtered air and high-purity (>99.999%) N_2 and CO_2 gases were used. A silicon dioxide (SiO_2) nanoparticle standard material (30 nm ± 5%; TSI, P/N 6005269) was electrosprayed at ~0.05 wt% in 20 mM ammonium acetate; the SiO_2 standard is known to contain dissolved residue. The particle size distribution of the standard was measured using air and N_2 mixed with CO_2.

Results

Table 1 shows the operating conditions of the EAG during stable cone-jet spraying using air and N_2 at 1.5 L/min and different CO_2 flow rates. Operating voltage and current were similar using air with and without CO_2. For EAG operation using N_2, voltage was decreased slightly to maintain a stable current of approximately -230 nA as CO_2 flow decreased from the nominal value of 0.1 L/min. At CO_2 flow rates less than 0.05 L/min, stable operation was not possible using N_2.

Table 1. Electrospray Aerosol Generator Operation Using Air and N_2 Mixed with CO_2

	Air		Nitrogen			
CO_2 flow rate (L/min)	0.1 (nominal)	0	0.1 (nominal)	0.075	0.05	< 0.05
EAG voltage (kV)	2.04	2.04	2.00	1.92	1.80	—
Current (nA)	-230 ± 1	-238 ± 2	-232 ± 1	-231 ± 1	-228 ± 2[1]	—

[1]Infrequent spikes in current were observed, up to -260 nA

For all particle size measurements, a 25-µm capillary was used, and a stable cone-jet spraying mode was achieved. All particle size distributions are the average of three scans. No adjustments were made to the gas viscosity, density, and pressure in Aerosol Instrument Manager® software.

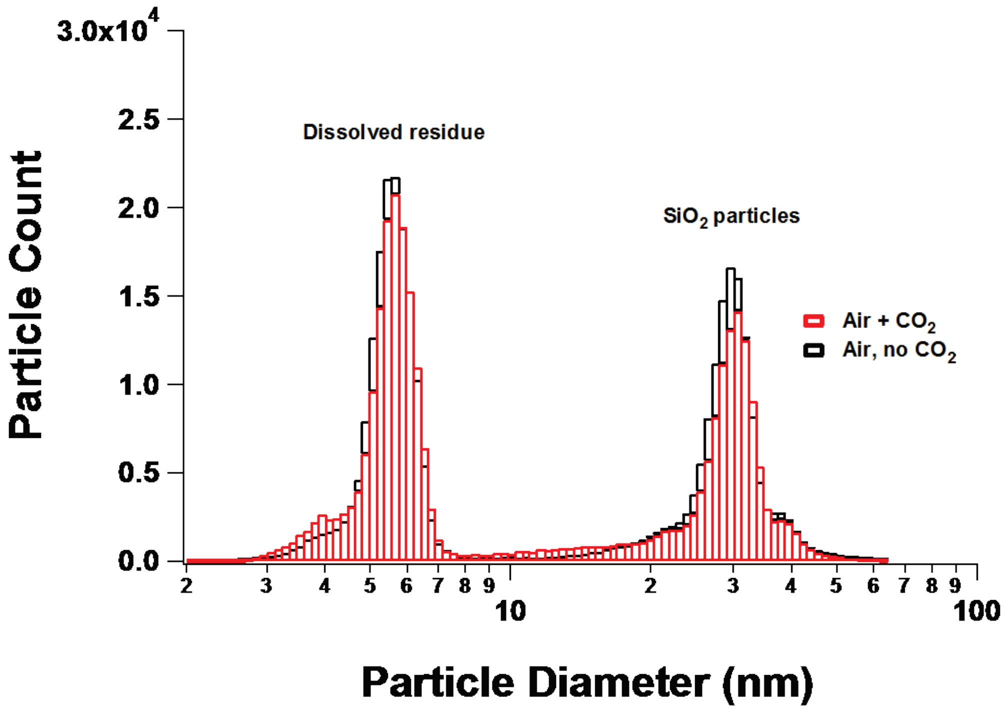


Figure 1. Particle size distribution of SiO_2 standard using air with and without CO_2. The SiO_2 particles were accurately sized at 28.9 nm. A residue peak was observed at 5.5 nm due to soluble impurities. The results with and without CO_2 were similar.
(Air flow = 1.5 L/min; CO_2 flow = 0.1 or 0 L/min).

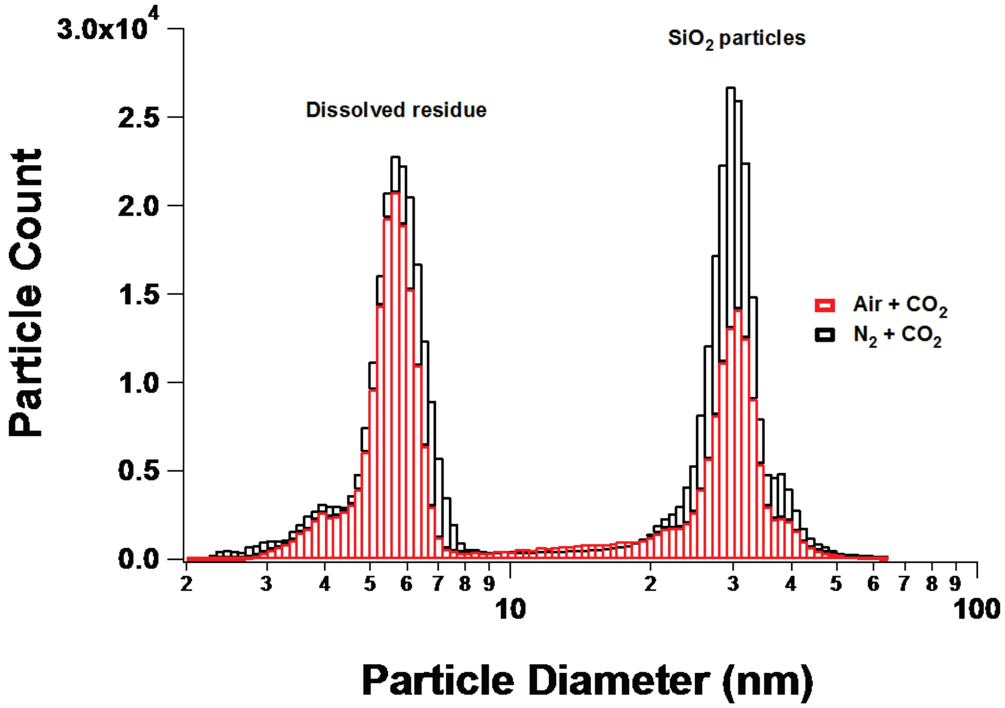


Figure 2. Particle size distribution of SiO_2 standard using air and N_2 with CO_2 at the nominal flow rate. The SiO_2 particles were accurately sized at 28.9 nm in both gases. The size of the residue particles was consistent in both gases. The SiO_2 particle count was higher using N_2, while the particle count for the residue was similar in both gases. (Air flow = N_2 flow = 1.5 L/min; CO_2 flow = 0.1 L/min).

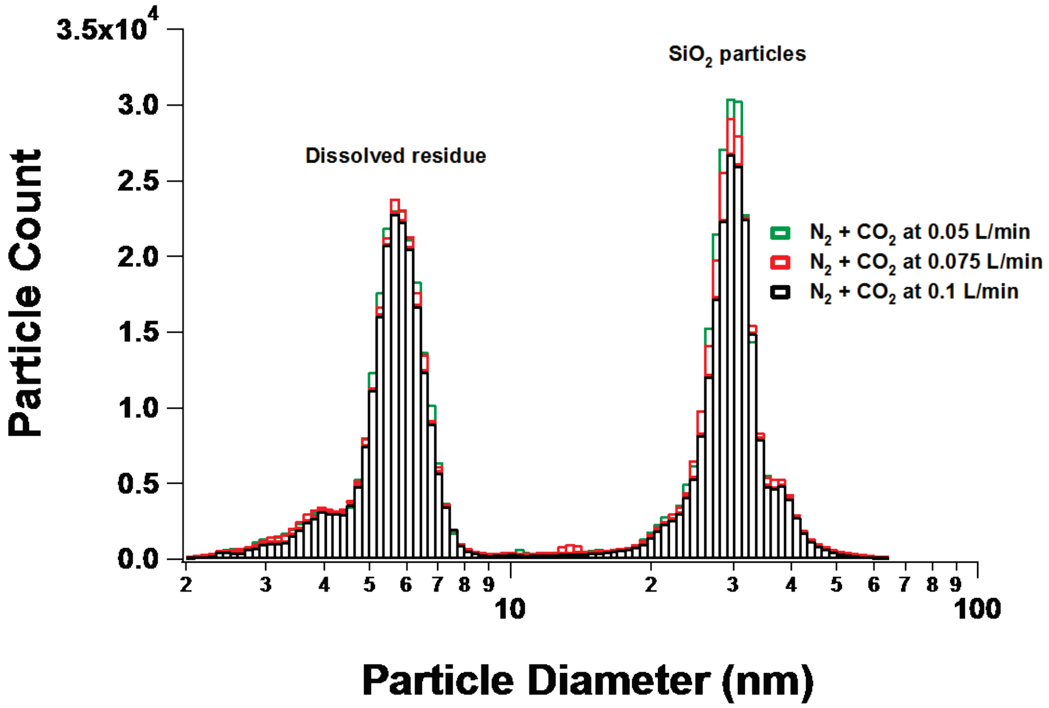


Figure 3. Particle size distribution of SiO_2 standard using N_2 with various CO_2 flow rates. The SiO_2 particles were accurately sized at 28.9 nm at CO_2 flow rates down to 0.05 L/min. The size of the residue particles was consistent in both gases. The SiO_2 particle count increased slightly as CO_2 flow rate decreased, while the residue particle count was similar at all CO_2 flow rates. At CO_2 flow <0.05 L/min, the sample could not be electrosprayed; a droplet was observed at the capillar tip, but the droplet was not drawn away from the tip at any voltage (0–3.5 kV). (N_2 flow = 1.5 L/min).

The gas used in an electrospray must be able to sustain the electric field required to achieve and maintain stable operation. Dielectric strength, an intrinsic property of gases, determines the maximum electric field strength that can be sustained before electrical breakdown occurs. As the voltage and electric field strength increase, the gas can become ionized and release free electrons that are accelerated by the electric field. As the electric field exceeds the dielectric strength of the gas, free electrons collide with molecules in the gas, releasing more electrons and forming a conductive path. This phenomenon is called corona discharge. The collision process can lead to a chain reaction, known as an electron avalanche or electrical breakdown. Stable electrospray operation cannot be sustained at voltages that induce corona discharge/electrical breakdown.

To prevent these phenomena, the gas or gas mixture used in an electrospray must be electrically insulating. That is, the gas molecules can remove electrons freed during ionization by attaching to the electrons during collisions. N_2 is a non-electron-attaching gas, due to its electronic structure, and has a lower electrical breakdown voltage (V_B) than air and CO_2. Thus, N_2 alone cannot sustain stable electrospray operation. On the other hand, CO_2 is an electron-attaching gas. Therefore, adding CO_2 to N_2, even at < 5%, allows stable electrospray operation at the nominal voltage setting of ~ 2 kV. The V_B of air is significantly higher than that of N_2 because O_2 is also an electron-attaching gas, and its V_B is much higher than that of N_2. Air and CO_2 can maintain stable electrospray operation at higher voltages than N_2 because they are able to control the acceleration of free electrons and thereby inhibit an electron avalanche.

Conclusions

The EAG can be operated using N_2 in place of air, as long as CO_2 is supplied at ≥ 0.05 L/min. The sizing of particles using the LiquiScan-ES is not affected by replacing air with N_2. The addition of CO_2 is necessary to prevent electrical breakdown of the carrier gas during EAG operation using N_2.

References

Meek, J.M. and Craggs, J.D. Electrical Breakdown of Gases, John Wiley, New York (1978).

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