DP-MS-80-30

CONSULTANTS REPORT

Particle Mass Concentration and Size Distribution Determination in the TNX Melter Pilot Plant

Submitted by

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Contents

1. Introduction

The purpose of this report is to make recommendations concerning the techniques and instrumentation for measuring particle mass concentration and size distribution in various process streams of the E.I. DuPont TNX melter pilot plant.

It should be recognized at the outset that real-time measurement of particle mass concentrations and size distributions in process streams is currently an art. Although several promising measurement systems are under development, there are presently no tried-and-true instruments to meet the need at hand. In addition, the extraction of a sample and sample transport for particle analyses present orders of magnitude greater difficulty than gas sampling.

The approach in this report was to evaluate alternative instrumentation and to recommend those which are reasonably accurate and which will present fewest maintenance, calibration and sampling problems for a particular application. It should be understood that in recommending a particular instrument, this consultant in no way assumes any liability for the operation or performance of that instrument.

Also, certain problems relating to sample extraction, transport and conditioning were evaluated based on process conditions estimated by DuPont engineers and on assumptions of the range of particle sizes present at the sampling points within the process. Recommendations were made to minimize the effects of these problems on sample representativeness.

2. Summary of Recommendations for Specific Streams

	Mode	Continuous				Manual			
Location	Analysis	Sampling Velocity	Sample Transport	Recommended Instrument	Sample Conditioning	Sampling Velocity	Sample Transport	Recommended Instrument	Sample Conditioning
Calciner Filter Input	Total mass concentration	N.A. (non-extractive)	N.A.	IKOR Model 2710	possibly sample, neutralize & scavenge air ions	high velocity	turbulent 1500-2000 cm/s very short lines, no sharp turns	EPA Method 5 sampling train in-stack alundum thimble or cascade impactor	heat to prevent condensation
Calciner Filter Exhaust	Total mass concentration	N.A. (non-extractive)	N.A.	IKOR Model 2710	possibly sample neutralize & scavenge air ions	isokinetic	laminar short lines, no sharp turns	EPA Mothod 5 sampling train, alundum thimble or cascade impactor outside duct	heat to prevent condensation
Cyclone Inlet & Outlet	Total mass concentration	N.A. (non-extractive)	N.A.	IKOR Model 2710	possible sample neutralize & scavenge air ions	isokinetic	turbulent for inlet; laminar for outlet	EPA Method 5 sampling train, alundum thimble or cascade impactor outside duct	heat to prevent condensation
Deep Bed Scrubber Inlet, Outlet, & Between Stages; Separator Input; Absorber Input; Scrubber Tank Bypass	Size Distribution	approx. isokinetic	laminar	TSI Model 3030 EAA	impaction of large water droplets; dilution	approx. isokinetic	laminar	EPA Method 5 sampling train, filter collection	impaction of large water droplets
Effluent Gas	Total number of particles; (continuous); Total mass concentration (manual)	not critical	laminar	condensation nuclei counter (Environment One or TSI)	impaction of large water droplets if bypass line is used	approx. isokinetic	laminar	EPA Method 5 sampling train, filter collection	impaction of large water droplets if bypass line is used.

3. General Considerations

3.1 Location of Sampling Points-

Ideally sampling points should be located at lease 8 duct diameters downstream and 2 diameters upstream of elbows, entries or other flow disturbances.

3.2 Traverse of Ducts-

Whenever possible, provision should be made for moving sampling probe across the entire I.D. of the duct in two traverses perpendicular to one another, according to standard source sampling practice.

3.3 Control of Instrument Environment-

The instrument recommended for particle size analysis, the Electrical Aerosol Analyzer, is a laboratory instrument. While there have been applications of the EAA to industrial source sampling, the instrument was not designed for this use. It is recommended that each instrument be mounted in an enclosure supplied with air at a moderate temperature and relative humidity. The enclosure should provide for access to and full view of the front panel. Also, care should be taken to reduce the vibration to which the instrument is exposed. If instruments must be shared between locations, these enclosures must be portable.

3.4 Sampling Ports-

Openings must be provided in the ducts for insertion of sampling probes. Many configurations are possible. Such ports should provide support for the probe while allowing it to move across the duct, and ensure that the traverse is normal to the central axis of the duct. Provision must be made for closing the opening when no probe is inserted. The ports should be designed so that both continuous and manual probes can be inserted.

The calciner filter housing already has a flanged opening covered with a plate. Ports for individual probes could be made to fit this flange. Filter exhaust and cyclone inlet and exhaust could be fitted with the same size flange so that probes would be interchangeable. Ports for other sampling locations downstream of the venturi scrubbers should also be designed for interchangeability of the measurement system specified for these locations.

3.5 Sampling Velocity-

When large particles comprise a significant portion of the aerosol to be sampled, then inertial effects must be considered. (For this purpose, large particles are considered to be particles with a diameter greater than 5 um at a density of 1 g/cm^3, or equivalently 2.2 um at a density of 5 g/cm^3). Errors in obtaining a representative sample of gas and entrained particles from a moving gas stream are a function of: (1) the ratio of the velocity of the gas (U_m) to the velocity of gas drawn into the sampling nozzle (U_s), and (2) the Stokes Number (the ratio of particle stop distance to the diameter of the nozzle.) As a general guideline, errors for sampling 50 um-diameter particles

(ρ=1 g/cm^3) can be kept below 10% of the total mass concentration by high velocity sampling ($U_m/U_s<0.03$), or isokinetic sampling ($0.92<U_m/U_s<1.15$) for a 1 to 2 cm nozzle.

In high velocity sampling, considerations of both Stokes number and sedimentation become important. Satisfactory samples under these conditions of up to 100-um particles may be obtained by employed a combination of nozzle size and sampling flowrate given below:

sampling flowrate (cm^3/s)	minimum nozzle radius (cm)
1	0.63
10	1.4
100	2.9
1,000	6.3
10,000	14.0
100,000	29

The sampling nozzle should be oriented to draw air in horizontally to reduce bias from sedimentation.

3.6. Sample Transport-

The sample of gas and particles must be transported from the nozzle to the collection device or measurement instrument. Even if the sample is representative when withdrawn from the bulk gas stream, particles may be removed from the sample by sedimentation (large particles), centrifugal impaction (large and medium particles), diffusion (small particles). These processes can greatly alter the total mass collected and the size distribution observed. In general, sample lines should be kept as short as possible. Walls of lines must be kept dry (by heating if necessary). Metallic lines should be employed when possible and they should be grounded.

There are two different approaches regarding selection of flow regime in sampling lines. If at all possible, contact between the walls and the particles should be reduced. This implies keeping the velocity as high as possible within the limits of laminar flow. If, however, contact cannot be avoided, an alternative is to maintain a high transport velocity, (1500-2000 cm/s), so that particles will be scoured off the walls. This approach is good for total mass measurements, but the particle size distribution may be affected as some agglomeration may result.

4. Recommendations for Specific Process Streams

4.1 Calciner Filter Input

Total mass concentration of particles is desired.

4.1.1 Sampling Velocity-

Calcine analyzed by SRL ACD had a mass median diameter of 34 um and a geometric standard deviation of 1.8. Comparison of particle settling velocities (at calciner conditions) within the range of diameters exhibited by calcine with the bulk gas velocities carrying particles to

the filter indicates that particles of diameter of about 60 um and less may be entrained by a flowrate of 600 ACFM (350°C & 0.963 Atm) and particles of diameter about 85 um and less may be entrained by a flowrate of 1400 ACFM. Thus, particulate mass concentration in the gas input to the filter should be quite high and inertial effects in sample will be quite important.

High velocity of sampling is recommended at this location as opposed to isokinetic sampling for two reasons. H.V. sampling avoids the problem of automatically matching sampling velocity with gas velocity in the filter housing. This would be particularly difficult at these low velocities (30 to 68 cm/s). Also, the direction of gas flow is less important in H.V. sampling. This is important in the filter housing since the flow direction may not be uniform across the housing and thermal eddies may result in rapidly varying gas speed and direction.

The high velocity probe should draw air in horizontally, with a flowrate of at least 6,000 cm^3/s. The nozzle should flare out to a diameter as wide as is practicable, say 3 inches. It is not possible to comply fully with the requirements discussed in Section 3.1 without too great a reduction in nozzle velocity.

4.1.2 Sample Transport-

Because a 4 foot long horizontal probe is required to traverse the filter housing and because large particles are involved, it was determined that contact of a large portion of the particulate mass with the probe walls was unavoidable. Thus, high transport velocity is recommended, say 2,000 cm/s. Change of direction of the sampling train should be avoided. Condensation on sampling line walls should be avoided by insulation, or heating if need be.

4.1.3 Instrumentation-

While an instrument which directly measures the mass of particles on a continuous basis, such as the quartz microbalance system (TEOM) of Rupprecht and Patashnik, is theoretically ideal. There are certain practical drawbacks. The current TEOM will not operate at high temperatures, thus the sample would require dilution. It would be necessary to develop a filtered bypass system so that particles do not load the quartz sensor when data is not required. This would reduce maintenance requirements. Also, the earliest delivery date would be sometime this summer.

Thus, an alternative such as the IKOR Particulate Monitor Model 2710 should be considered, which uses a charge transfer principle. This instrument has the advantage of avoiding the sampling problems discussed above because it works in the stack (non-extractive). A further advantage is that it can withstand the high temperatures of the calciner off-gas, whereas the TEOM would require use of a dilution system for cooling the sample down to an acceptable temperature.

Since the IKOR system is not a primary mass measurement, manual stack tests utilizing filter collection must be made as a means of calibrating the IKOR. Ideally, these manual tests should employ the

recommendations for sampling in section 4.1.1, and an in-stack thimble holder for collecting material in an alumdum filter thimble. If size distribution data is needed at this location, the manual method can be adapted using an in-stack cascade impactor (Andersen Samplers, Inc.).

The sampling train recommended is an EPA Method 5 sampling train. A simpler train would due for this purpose because of the high velocity sampling, however, it will be useful for manual sampling of input to the cyclone.

4.1.4 Sample conditioning-

Initially, the IKORR should be tested with no sample conditioning. It has been noted that the contact electrification sensors respond to existing charge on the aerosol[3], and aerosol created by atomization is expected to have a charge. It may be necessary to extract a sample gas stream and neutralize the aerosol (TSI Kr-85 neutralizer) and scavenge air ions before contact is made with the sensor.

4.2 Calciner Filter Exhaust

The total mass concentration in the gas stream is desired.

4.2.1. Sampling Velocity-

The duct velocity here will range from about 850 cm/s to 2000 cm/s. Particle size distribution is not known, but isokinetic sampling procedures should be followed initially. If it can be shown that no significant portion of particle mass exceeds about 1 um in diameter at this location, then maintenance of isokinetic conditions is unnecessary.

4.2.2. Sample transport-

Inertial deposition is of greatest concern. Flow in probe and lines should be kept laminar. Changes in direction and condensation in lines should be avoided.

4.2.3. Instrumentation-

So that equivalent measurements may be made upstream and downstream of the filter, the IKOR Model 2710 is again recommended. With manual sampling for calibration and verification of results.

4.2.4. Sample Conditioning-

The aerosol may require neutralization as noted in Section 4.1.4.

4.3. Cyclone Inlet and Outlet
(Filter Bypass)

Total mass concentration of particles is desired. Since the cyclone and filter will not be in operation simultaneously, it will be possible to use the same monitoring equipment for both provided sampling ports are compatible with those employed in filter inlet and outlet.

The only difference is that during manual sampling, the cyclone inlet should be sampled isokinetically since the gas velocities will be much higher with less variation here than at the filter inlet.

4.4. Deep Bed Scrubber Inlet, Outlet and Between Stages; Separator Input; Absorber Input; and Scrubber Tank Bypass

Total mass concentration and particle size analysis is desired. These process streams are similar in that they will be at moderate temperatures, saturated with water vapor, and will contain some large water droplets. The particle size at these locations is not expected to exceed 1 um diameter.

4.4.1. Sampling Velocity-

Isokinetic sampling is recommended for manual methods employed in these locations. Continuous monitoring using the Electrical Aerosol Analyzer (EAA) need be only approximately isokinetic since particles greater than 1 um are not sized. Thus, for continuous monitoring, it should be sufficient to set the nozzle velocity equal to the bulk gas velocity (Q/A) for the particular process stream and flowrate condition.

4.4.2. Sample Transport-

Laminar flow conditions are recommended for the sampling line to reduce contact of particles with the wall. It is estimated that a 1 meter length of 1 cm I.D. sampling line at a Reynolds number of 1,000 would cause a 3% loss of 0.01 um diameter particles and 0.2% loss of 0.1 um diameter particles. A 25% loss of 0.1 um diameter particles would not occur until sampling line length reached 2,000 meters, but for 0.01-um-diameter particles only a 26 meter length would produce a 25% diffusional loss. Thus, diffusional loss in very long lines is a possibility but this is not a critical problem for the particles of interest.

It was also estimated that inertial loss of large particles should not be important, assuming that particles are no greater than 1 um in diameter and the conditions of flow described above. Nevertheless, turbulent flow should be avoided as should unnecessary sharp bends.

Walls of sampling tube must be kept dry by heating with heating tape since the gas will be close to saturation.

Following these guidelines, it appears that transport of sample up to 10 meters or so should have negligible effects on the concentration and size distribution.

4.4.3. Instrumentation-

The only instrument which has been used extensively in this range of particle sizes is the TSI Model 3030 Electrical Aerosol Analyzer (EAA). The instrument has sizing capability within the nominal range 0.0032 um to 1.0 um (Actual useable range for most applications is about 0.01 um to 0.562 um).

4.4.4. Sample conditioning-

Since the sample is likely to contain large water droplets (say d>50um), it is recommended that a course impactor be installed in-line close to the duct such as the impactor on the TSI Model 3062 Diffusion Dryer. Liquid collected from this impactor can then be analyzed using a wet particle sizing technique to determine what particle sizes are to be found in these fluidized, coarse droplets. (Note: droplet removal is also recommended for manual sampling at these locations.)

The sample streams will very likely require dilution before entering the EAA to reduce concentration to measureable levels. It may be that a commercial diluter can be adapted such as the Climet CI-294, or one may be constructed as described in the Appendix.

4.5. Effluent Gas

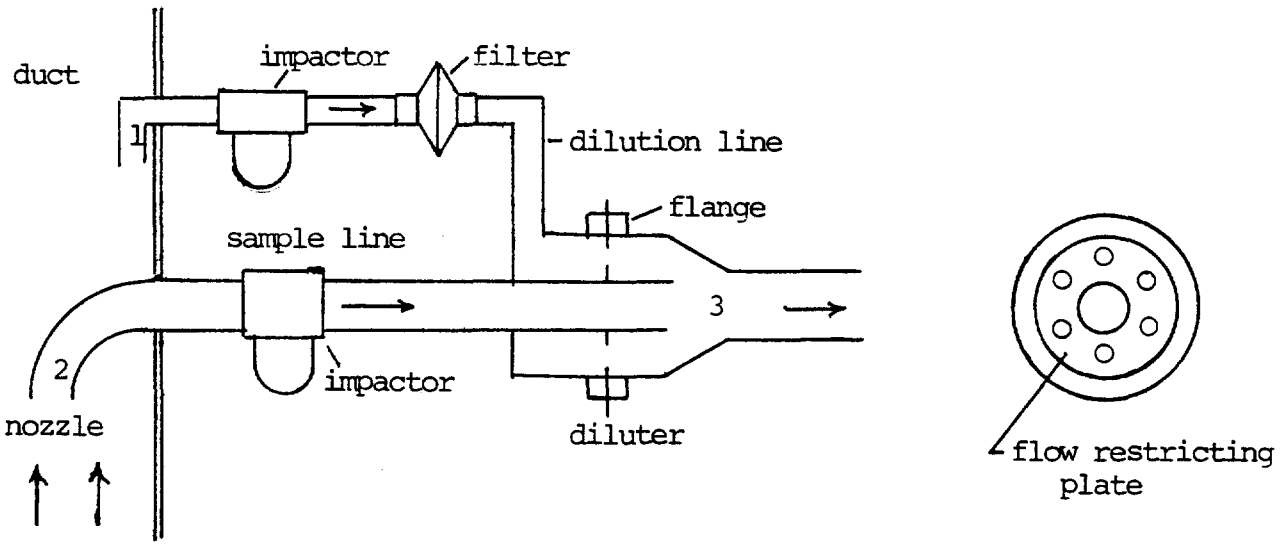
It is adviseable to monitor emissions to the atmosphere to identify any possible problem in the pilot plant stage so that full scale facility may be designed to avoid any problems in meeting emission standards. A continuous monitor is desireable since emissions may be intermittent or correlated with specific process operating conditions.

A condensation nuclei (CN) counter such as the Environment One Model Rich 100 is recommended. A CN counter measures the approximate number concentration of all particles with diameters greater than about 0.0025 um.

If a significant number of particles are observed, it may be necessary to manually sample this stream to obtain size and composition data.

Appendix

Diluter Design



Since the flow through a resistance under varying downstream pressure is generally related to the square root of the pressure drop,

$$Q_1 = (P_1-P_3)^{\frac{1}{2}}/R_{13}$$

$$Q_2 = (P_2-P_3)^{\frac{1}{2}}/R_{23}$$

where Q is flowrate, P is static pressure and R is resistance to flow. We want the ratio Q_1/Q_2 to be the constant for the changes in P_3 required to produce roughly isokinetic conditions. When dilution air is withdrawn from the duct in which sample takes place as shown in Figure 1, $P_1=P_2$ and Q_1/Q_2 is constant for varying P_3.

This rudimentary design may require a few refinements since flow in these branches may not be exactly proportional to the square root of the pressure difference.

References

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2. Cadle, R.D. The Measure of Airborne Particles. John Wiley and Sons, New York (1975). p. 320-324.

3. John, W., G.P. Reischl, and W. Devor, "Aerosol Monitors Based on the Contact Electricity Principle." in Aerosol Measurement, Lundgren, et al. Eds. University Presses of Florida, Gainesville (1979).