



Flame Sampling using the DMS500

The DMS 500, in conjunction with a bespoke sampling system and its standard dilution system, has been used to measure the size-distribution of particles formed within flames. The DMS was particularly suited for such an application, because the low pressure used in the classification system, combined with the high specification turbo pump, allows extremely rapid (supersonic) sampling and dilution of particles from flames, without the requirement to build a complicated sampling system.

This note, based upon work at the Department of Chemical Engineering at the University of Cambridge, details the experiments which were carried out, the minor modifications to the DMS which were made, and the sampling probes which were constructed. It also details the best conditions which were found to obtain reliable results.

Introduction

Sampling particles from a flame is not a simple proposition. In addition to the high temperatures, the chemical environment is frequently very challenging, with high concentrations of radicals and / or highly oxidising or reducing conditions. Furthermore, severe losses of particles can occur during the sampling process:

1. At the orifice at the entrance to the sampling probe
2. Whilst the gases are cooling during their transit through the probe
3. During their transport from the probe to the analyser.

In order to produce quantitative results, all three sources of particle loss must be reduced, together with any agglomeration of particles during sampling or in sample lines. Points (1) and (2) are addressed by careful design of the sampling probe, point (3) is addressed by sampling at reduced pressure into dilution N_2 . The main aim of this research was to investigate the best way to sample from a flame, such that the measured size distribution and total number of particles was that formed within the flame, and was independent of any sampling effects.

Recommended Instrumentation

The DMS500, with the standard dilution system is used, but with the inlet restrictor changed for that used with remote sampling.

Cambustion aided in the design of bespoke sampling probes^{1,2} which were constructed from quartz. These probes were designed to take advantage of the DMS's dilution ability and its ability to sample at reduced pressure. Figure 1 shows a probe sampling from a flame.

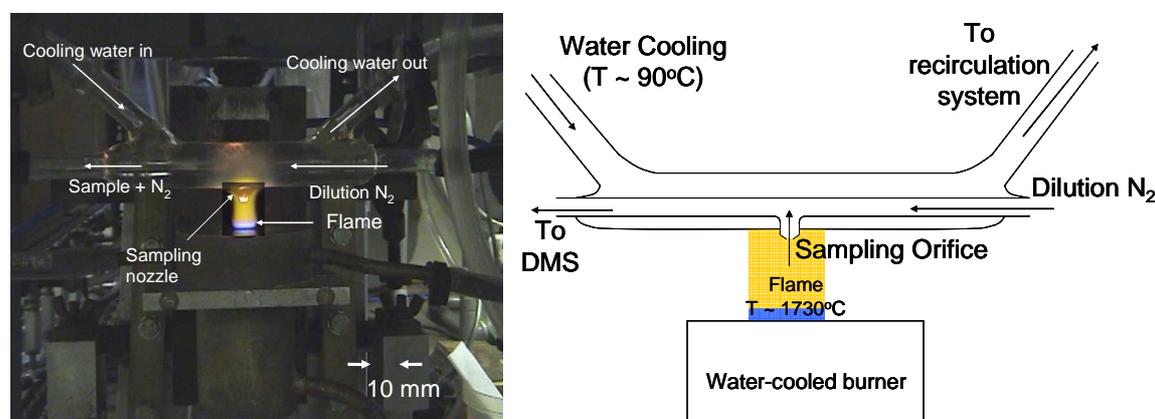


Figure 1. DMS500 sampling from a flame using a quartz sampling probe.

The left hand image is a photograph of the actual sampling system. The flame (which has been made visible by the addition of a small amount of sodium to the feed gas) is stabilised on a movable water-cooled burner.

The sampling orifice, glowing white hot, supersonically samples the flame gases into the sampling system, which is held at ~ 0.3 bara. Because the flow through the orifice is choked, the flowrate of sampled gas does not depend upon the conditions downstream of the orifice. Nitrogen (0.3 bar, supplied and metered by the DMS) rapidly dilutes the sampled gas, preventing agglomeration. The high flowrate within the sampling system effected by the low pressure and the high flow of N₂ from the DMS leads to residence times as low as 25 ms in the sampling lines, with simultaneous dilution by a factor of 10 and expansion of the sampled gas by a factor of ~ 3.3 means that the sampled particles neither agglomerate nor coalesce post sampling. Standard non-conductive sampling hose was used to transport the sampled gas, after cooling and dilution in the probe, to the DMS500

Experimental Conditions

The flame which was sampled was a laminar flame, with a temperature of ~ 1730 ± 30°C. It was extremely fuel lean, and consisted of C₂H₂ / O₂ / N₂ with mole fractions in the unburned gases of 0.054 / 0.380 and 0.570, so that there was ~ 2.8 times more O₂ in the flame than that required for complete combustion. The total flowrate of the unburnt gas was 154 ml / s. A small amount of MgCl₂ was nebulised into the flame, giving an initial mole fraction of ~ 13 ppm of Mg in the gas phase within the reaction zone of the flame. Because of the extremely high oxygen (and O radical) concentration in the reaction zone, particles of MgO formed extremely rapidly in the flame; changing the height at which they were sampled did not appear to vary the measured PSD (particle size distribution) markedly.

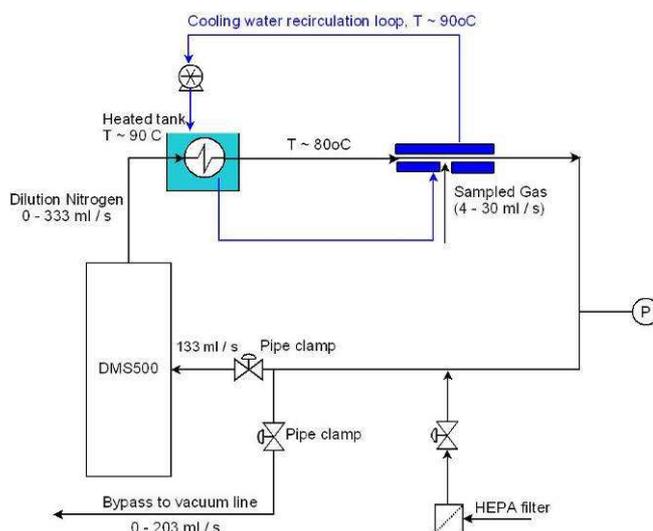


Figure 2. Flowsheet showing the DMS sampling from a flame, with a cooling water recycling loop. All flowrates are the equivalent flow at N.T.P.

Figure 2 shows the DMS500 sampling from the flame, and controlling and metering the dilution nitrogen for the sampling system. The dilution nitrogen at ~ 0.3 bara (filtered by the DMS through a HEPA filter), which was supplied by the DMS500 passed through a coil of copper tube, ~ 3 mm i.d., 3 m long, submerged in a tank of water at ~ 90°C, and was heated to ~ 80°C. The N₂ then passed into the

sampling probe (which also cooled by the water from the tank at 90°C), where it diluted the gas sampled from the flame. The gas then continued to the DMS500, which requires an inlet flowrate of 133 ml / s (N.T.P). Any gas in excess of this amount was spilled to a vacuum line, which bypassed the DMS500 and entered the vacuum pump (supplied with the DMS500) directly. For experiments where the total flowrate of dilution gas + sampled gas was less than 133 ml / s (N.T.P.), extra gas is admitted to the sampling line via a HEPA filter. The pressure in the entire sampling loop is controlled using a pipe clamp on the sampling line at the inlet to the DMS 500, as is the total amount of gas spilled to the vacuum line. Generally, no constriction was applied at the inlet to the DMS – in this case, the pressure in the system is ~ 0.3 bara.

This experimental setup allowed the effects of the extent post-sampling dilution and expansion of the gas from the flame to be measured, allowing measurements to be made of the extent to which sampling of the particles affected the measured size distribution and total mass of particles.

Results

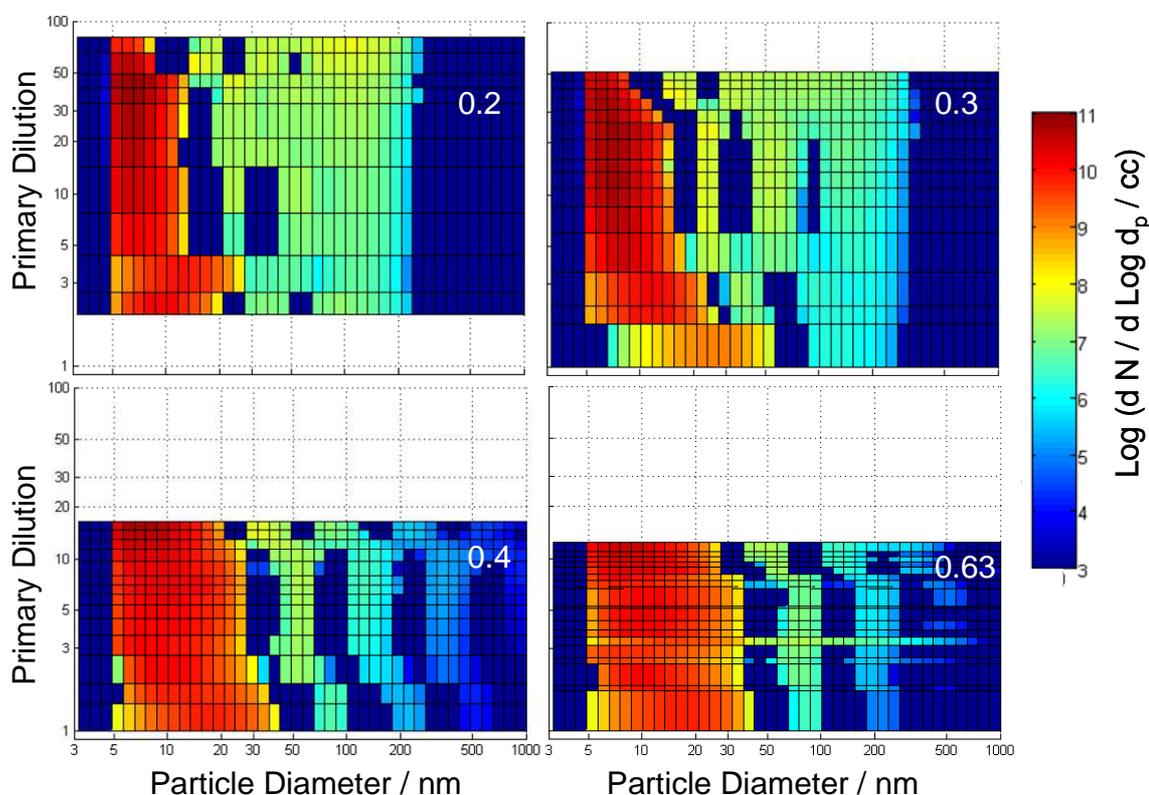


Figure 3. Particle size distributions measured using different diameters of orifice (as shown, all dimensions in mm), plotted against the dilution in the first stage (i.e. that within the sampling probe). The colour corresponds to Log_{10} of the total number of particles sampled.

Figure 3 shows a map of the number of particles (colour) plotted against the particle diameter (x-axis) and the dilution within the sampling probe (y axis), defined as the total flow divided by the sampled flow. It is clear from figure 3 that the measured diameter of the particles shifts from an average of ~ 20 nm for a dilution ratio of unity, to ~ 6 – 8 nm for particles. There is a corresponding increase in the number of particles, so that the total mass sampled by each orifice remains more-or-less constant. This is clear evidence of agglomeration or coalescence of particles post sampling, should they be insufficiently diluted.

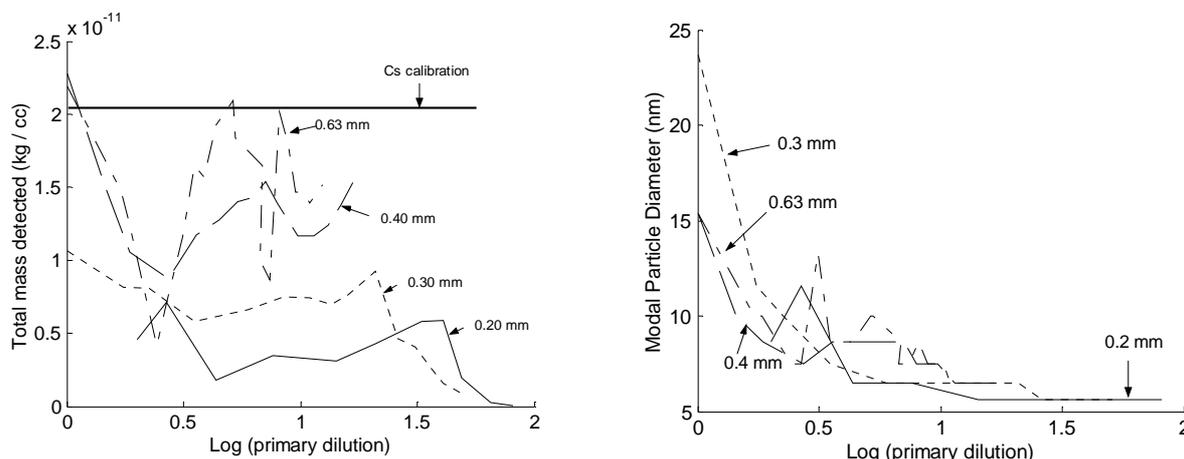


Figure 4. Total mass detected (left) and modal particle diameter (right), plotted against Log_{10} of the dilution ratio.

Figure 4 shows the total mass sampled for each of the four different sample orifices, plotted against the Log_{10} of the dilution.

Sampled Mass of Particles

Disregarding results for dilution ratios < 3 (i.e. 0.5 on a logarithmic basis) where the very hot gases probably damaged the sampling probe, it is clear that the total mass of particles sampled is reasonably consistent, at least for the 0.40 mm and 0.63 mm orifices at dilution ratios of ~ 10 or more (of course, since the mass of particles depends upon their diameter cubed, it is notoriously difficult to obtain reliable measurements of the total mass of particles, hence the errors). It is also clear that the results for the total mass of particles sampled agree to within 25 % with the expected results from an independent calibration of the nebuliser using an independent calibration method (in fact, since this work has been completed, losses in the pipes from such a system leading into the DMS have been much better characterised, so that the observed discrepancy can, at least in theory, be corrected for). The smaller orifices lost a greater proportion of the sampled particles around the relatively cool sampling nozzle. Interestingly, when the flowrate of dilution nitrogen to the smaller nozzles was increased to very high total flowrates, such that the flow within the sampling probe was turbulent, the measured number of particles decreased dramatically; this indicated that turbulence in the sampling system was to be avoided for quantitative results to be obtained. This was checked by inserting an object into the sampling probe to artificially induce turbulence for lower flowrates of dilution gas – as expected, the total mass of particles sampled dropped markedly.

Sampled Particle Diameter

The modal particle diameter is also shown in Fig. 4. It is apparent that the measured particle diameter converges to 6 – 8 nm for all diameters of sampling orifice as the dilution within the system increases, though it is important to bear in mind that the total mass of particles reduces markedly for the very highest dilutions (i.e. these with the smallest sampling orifices).

Conclusions

The DMS500 is an ideal tool for sampling from flames, due to the ease with which it can be converted to rapidly expand and dilute samples. Different designs of sampling probe have been tested to see which ones give the most consistent results in terms of the mass and particle size distribution sampled. It was found that a water-cooled probe, with a conical end and an orifice ~ 0.4 mm in diameter, with a flowrate of dilution N₂ passing through it, diluting the sampled gas by a factor of at least ten was required to produce quantitative results. For a much more in depth description of the sampling system and results obtained, the interested reader is directed to reference [2].

Acknowledgements

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1. Fennell, P. S.; Dennis, J. S.; Hayhurst, A. N., The Size Distributions of Nanoparticles of the Oxides of Mg, Ba and Al in Flames: Their Measurement and Dependence on the Concentrations of Free Radicals in the Flame. *Proceedings of the Combustion Institute* **2007**, 31, (2), 1939-1945.
2. Fennell, P. S.; Dennis, J. S.; Hayhurst, A. N., The Measurement of the Concentrations and Size Distributions of Nanoparticles of MgO formed by Doping an Oxygen-Rich Flame with MgO. *Combustion and Flame (in press)* **2007**.