Project A – Establishing sampling and analytical procedures for potentially harmful components from post-combustion amine based CO_2 capture

Task 1: Design of Sampling Points for Treated Flue Gas

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EXECUTIVE SUMMARY

The atmospheric emissions from amine-based post combustion capture (PCC) systems will be significantly different from those of conventional power plants. However, these emissions are not yet well characterised and detailed analysis of the stack emissions will be required. The first step in the analytical process is taking representative samples of the gas and other material in the stack. Because the composition of flue gas is often not homogeneous, it is essential to design a sampling system that can fully represent the gas flow while at the same time ensuring the integrity of the samples collected.

Since emissions from power plants and other large industrial combustion processes are normally subject to government regulation, stack sampling is a routine operation in most industrialised countries and the general principles are well understood. As a result, there are now numerous standard methods available that provide specific advice for the design of the sampling system. Many of these methods are internationally recognised procedures that are mandated for statutory emissions testing purposes.

In Task 1 of the Project A, the design of sampling points appropriate for large scale amine capture plants was considered. The main thrust of the investigation was the design of the physical layout of the sampling within the gas stream in the stack. Although important to the overall sampling process, the sample collection devices and other components of the sampling train are only briefly discussed in this report. These aspects are to be dealt with in detail in Project A Task 2: Procedures for manual sampling; and Task 3: Online sampling/analysis

The main conclusions from Task 1 are as follow:

- Methodology for selecting sampling points is well established and has been used for regulatory emissions monitoring for many years. In all methods, the preferred location of the sampling plane is in sections of stack with long runs of straight flow (termed ideal flow).

- In principle, the sampling methods provided in the available international standard methods should be suitable for application at amine scrubbing plants, The materials from which the sampling components are fabricated will need to be selected bearing in mind possible incompatibilities with the analytes in the stack gas. Corrosion resistant materials such as stainless steel will probably be used in the sampling system although other materials may be used as further information on the chemistry of the potential emission compounds comes to light.

- If the compounds of interest are gaseous, chemically stable, not soluble in any condensed phase or adsorbed on solid phases, and the flue gas stream is well mixed, sampling should be straightforward, requiring as little as one sample point that can be located anywhere in the gas flow. In these cases, ideal flow is not a requirement for representative sampling. However, many of the components in the exhaust from an amine PCC plant are unlikely to meet these criteria and more elaborate sampling methods will be required.

- Previous experience suggests that solid particulates and entrained liquid aerosols will not be evenly distributed within the stack gas flow and there may be significant spatial variation in concentration. Consequently, representative sampling from these stacks

will require a comprehensive series of sampling points distributed through the sample plane. Sampling for these components should be in ideal flow conditions detailed in the relevant standard methods.

- The sampling probes may significantly upset the flow in small ducts. This could be a problem in pilot scale plants with small diameter stacks but should not be an issue for full scale plants.

- CFD modelling should be used to theoretically identify and validate designs and examine the influence of these upon the particle or aerosols flow. This will reduce the amount of practical work needed on the plant.

- If emission fluxes are to be determined, the volumetric flow rate will need to be measured concurrently with sample collection.

- Sampling and velocity measurements are likely to be made in sections of the stack that are at elevated locations. To ensure that personnel are able to work safely and operate the sampling equipment in accordance with the approved methods, provision must be made for appropriate access ports and work platforms.

- Either USEPA Method 1 or VDI 2066 can be used for locating and experimentally validating the flow conditions at the selected sampling plane. VDI 2066 has a slightly more stringent criterion with respect to the cyclonic flow (<5°) and it is this method that CSIRO recommends for the Mongstad facility.

- While both USEPA Method 1 and VDI 2066 identify the use of flow correcting devices for constraining cyclonic flow to within the acceptability criterion of each respective method, flow correcting devices should only be utilised if absolutely necessary. It is recommended that that at a minimum the USEPA (*2D/0.5D*) criterion be achieved which for the Mongstad facility flow requires a minimum of 6.25m of straight duct.

- It is recommended to use a three-dimensional system for measuring all components of the flow using such as described in USEPA Method 2F: This method is capable of measuring all of the vectors associated with the flow and provides detailed picture of the flow at each sampling point. It is also recommended that spherical type probes be used due to their ability to measure pitch and yaw in real time.

- Round stacks, such as at the proposed Mongstad facility, require a minimum of four access ports located at each end of two perpendicular transverses of the duct. The traverse points are located along these traverses with six points located on each traverse for USEPA Method 1 and ten points located along each traverse for VDI 2066

- VDI 2099 provides the most appropriate design and dimensions for sampling ports for the proposed Mongstad facility. The ports described are of sufficient size to enable the insertion of new designs of “in stack” sampling apparatus that will be required to capture the emissions from the plant. However, it is recommended that the sampling port design enables the sealing plate of the access port to form a smooth fit with the inside surface of exhaust duct resulting in minimal gas stagnation in the region of sampling port

- To reduce the uncertainty of flow measurement, wall effects from the hydraulic friction at the duct surface should be determined using USEPA Method 2H and high resolution spherical 3-dimensional Pitot tube instrumentation.

- Sampling nozzles and all transitions between fittings of different sizes upstream of a particle filter should have a transition angle of 15 degrees.

- Depending upon the aerodynamic aerosol distribution, strict adherence to isokinetic sampling may not be required at the Mongstad facility. While this would simplify stack sampling operations, this approach would require experimental assessment and validation of the aerodynamic particle distribution over the selected sampling plane.

- It is recommended that inert material such as 300 grade stainless steel, quartz and Teflon be used on all wetted surfaces within the sampling train. Heated sampling lines will be required. Galvanised steel, copper and copper bearing alloys are incompatible with many amines and should be avoided.

- CFD modelling should used to theoretically identify and validate designs and examine the influence of these upon the particle or aerosols flow. This will reduce the amount of practical work needed on the plant.

1 INTRODUCTION

Amine scrubbing is currently one of several processes being investigated to reduce atmospheric emissions of CO_2 from fossil-fuelled power stations. In this process, an aqueous alkanolamine solution absorbs CO_2 from the flue gas; with mild heating, the CO_2 is then released for compression and subsequent geological disposal. One of the principal advantages of the amine process is that it is well understood and already widely used to remove CO_2 and other acid gases in gas processing plants throughout the world (Kohl and Nielsen, 1997). Consequently, is considered to be the most technically and economically advanced system available for post combustion capture (PCC), and therefore the closest to deployment on commercial power plants (Rochelle, 2009).

Like conventional power generation utilities, atmospheric emissions from power plants equipped with amine based PCC systems will be subject to public scrutiny and government regulation. In a conventional power plant, stack emissions of environmental concern (apart from CO_2) generally include NO_x, SO_2, particulate matter, and trace quantities of various other compounds, depending on the type of fuel. Emissions from an amine PCC plant, on the other hand, are likely to be substantially different. Along with the obvious reduction in CO_2, the concentrations of acid gases such as NO_x and SO_2 will be lower because they are removed by reacting with the basic amine solvent. Solid particulate matter (e.g. fly ash), too, is likely to be reduced since it will be physically washed out of the gas stream to some extent as it passes through the capture plant (although solid particulate matter emissions from gas-fired plants are generally very low).

Countering this, however, is that some other species not normally associated with power station emissions will be produced. For instance, despite the relatively low volatility of alkanolamines, it is inevitable that some of the amine will escape from the plant. It has been estimated that the flue gas from the proposed PCC plant for the Kårstø power station in Norway would contain up to 4 ppm amine, resulting in the release of about 160 t per year (Knudsen et al., 2008). Other compounds may be formed by thermal degradation of the amines or by chemical transformations that occur within the absorber and stripper columns and these, too, may escape from the plant. Complicating the issue further is that numerous amine compounds can be used for CO_2 capture, including proprietary blends with unknown compositions. Additives such as corrosion inhibitors, foam suppressants and pH buffers may also affect emissions. Clearly, characterising the full scope of the emissions from a large scale amine capture plant will be a considerable analytical challenge. Equally, however, sufficient attention must be given to the design of the sampling regime to ensure that the results of the analyses are representative.

Sampling is a complex procedure in its own right, involving a number of discrete steps; however, the first step in any sampling process is the selection of the points where material is to be extracted for analysis. Because it is obviously not be possible to collect all of the material emitted from the stack, it is necessary to take a sub-sample that is representative of the bulk flow. This sample comprises only a tiny fraction of the total volume of gas emitted, and it is usually necessary in large diameter stacks and ducts to take samples from a number of locations within the gas flow. The sampling regime is also determined by the type of material of interest. Gases, for instance, may be well mixed in the gas flow and relatively few sampling points (even one) may be sufficient to obtain a representative sample. Solid and liquid phase aerosols, however, are frequently unevenly distributed and many sample points are required to adequately characterise the flow. In some cases, the material can partition

between the gas phase and solid and/or liquid phases (Vicard and Fraisse, 1994) so in these cases, sampling must include the gaseous and aerosol components. This may be particularly important for emissions from amine plants where liquid aerosols containing amines and other soluble compounds may be in equilibrium with gas phase components.

Since emissions from the power industry are closely monitored by government agencies to ensure that environmental and public health standards are maintained, methodology for stack sampling from stationary sources is well established. Consequently there are numerous internationally recognised standard methods available that provide detailed guidance on the design of sampling systems for regulatory stack emission monitoring. Most of these methods have been in widespread use throughout the world for many years and are considered to be robust and reliable. As well, the general principles are applicable to any large stationary emissions source and indeed, are routinely applied in a diverse range of industries.

The purpose of the study reported here (Task 1) was to examine how sampling points in treated flue gas from an amine PCC plant should be designed to obtain representative sampling both for manual and online sampling. The sampling methods themselves are not considered here since they are the subjects of two separate studies (Task 2: Procedures for manual sampling; Task 3: Online sampling/analysis).

In this report, general aspects of the selection appropriate sampling points are discussed along with the various relevant international standard methods available. In particular, the following aspects are addressed:

- Points to consider for sampling in pilot and demonstration plants
- Appropriate location of the sample points
- Common errors associated with sampling in large ducts

Most emissions measurements are ultimately used to estimate emission fluxes of particular species, thus we also consider aspects for accurately measuring flow velocities in large diameter stacks. Also materials used in the construction of probes are briefly discussed.

2 SAMPLING POSITION

2.1 Principles of Representative Sampling

The primary objective of all emission sampling and analyses systems is, of course, to accurately quantify the abundance of the target species present in the flowing exhaust. While this process is conceptually simple, the experimental reality is often not straightforward. One of the main factors dictating the sampling regime is the nature of the gas stream. In the simplest case, a well mixed, chemically stable gas can be sampled from single point anywhere within the stack. However, in many instances the gas flow will be multi-phase, containing gaseous, solid particulate and liquid material, often with significant spatial distribution throughout the gas stream. In these cases, the sampling protocol must be designed accordingly, and samples are normally taken at numerous locations across the entire cross-sectional area of the stack or duct. Typically, a grid spacing pattern is used for sampling particulate material from stacks and ducts.

For solid and liquid aerosols, sampling is usually isokinetic, where the velocity of the sample stream into the nozzle of the collection device is equal to the velocity of the bulk gas flow. If the flows are not matched, there may be substantial sampling biases introduced that can severely compromise the quality of the samples obtained. The degree in mass bias is highly dependent upon aerodynamic size range of the target aerosol such that the requirement for isokinetic sampling diminishes as the maximum aerodynamic diameter of the aerosol becomes smaller. This diminishing importance of the isokinetic sampling criteria may simplify the sampling requirements at the proposed Mongstad plant if the aerodynamic aerosol distribution is demonstrated to be of sufficiently small Stokes diameters. The criteria for isokinetic sampling are discussed further in Section 4 of this report. Additionally, it will be dealt with in greater detail in Task 2.

As well as determining the concentration of a particular compound, most stack monitoring procedures involve measuring the volumetric flow of the gas stream because in most cases, emission fluxes of these materials must be reported. Consequently, the velocity of the gas in the stack must be measured. Often the gas velocity is uneven, so representative measurements must be made within the flow according to the grid patterns used for sample collection. Section 3 of this report considers gas flow measurement in greater depth.

Before designing the sampling system, it is crucial to understand the physical and chemical characteristics of the target species. Physical properties of the emission species (i.e. whether the compounds are gaseous, solid particulate material or liquid aerosols) have a strong bearing on the type of methods used for sample collection. The chemical properties may also affect the sampling regime. For instance, the compatibility of the target compound with the materials of the sampling apparatus must be considered. In some cases, target compounds are unstable or react with other species within the flue gas or within the sampling train, and one therefore must be cognisant of these reactions. The difficulty is compounded where the system presents unusual and novel compounds whose thermodynamics and kinetics are unknown. To a large extent, this is the case for emissions from an amine capture plant.

In broad terms, the sampling and analyses process can be divided into four experimental steps:

- Identification of the sampling position most suitable for the accurate capture of the target species.

- Experimental validation of the acceptability of the sampling position. This is especially important when sampling solid or liquid aerosols where “ideal” sampling conditions normally require straight flow (i.e. free from disturbances caused by bends, fans, dampers or other obstructions) within the duct. In some sampling situations, ideal conditions can be validated using fluid dynamics modelling.

- Identification and validation of appropriate sampling techniques and equipment (including material compatibility) to accommodate the physical and chemical properties of the target species. Aerosol systems generally require more diligence than well mixed gaseous systems because aerosols must be sampled isokinetically to ensure that size biases are not introduced. Some aerosols may also exhibit a variation in chemical composition as a function of size, which must be accounted for when designing the sampling system.

- In cases where reactive compounds are to be sampled, it may be necessary to preserve the sample by some form of quenching to prevent loss of the compound during the sampling process. This requires detailed understanding of the chemistry of the material.

The last two points are not covered in detail in this report; they will be dealt with specifically in the reports of Project A Task 2 and Task 3.

To ensure accurate and representative sampling in a large duct or stack, the selection of the sampling position (or positions) must take account of many factors. As discussed above, the nature of the target species has a strong bearing on the sampling protocol (e.g. gaseous, solid, liquid, chemical stability); the position and number of sample points may be different for different species. The geometry of the stack is also an important consideration since bends, fans and other obstructions can disturb the gas flow within the duct, which may affect the distribution of material in the gas stream. It is generally considered that sampling, especially for particulate material should ideally be from relatively straight undisturbed flow. Finally, the velocity, moisture content and temperature of the gas stream in the duct can affect sampling decisions. Sampling at temperatures below the dew point, for example, may lead to unintended sampling errors caused by soluble compounds being removed with condensing moisture. In many industrial cases, however, selecting appropriate sampling points is a compromise between experimental accuracy and practical considerations such as access to the duct and the safety of personnel.

Emissions from power stations and other large stationary sources have been regulated in most countries for decades, and the general principles for sampling from large stacks and ducts are well established. Methods have been developed for testing emissions, including detailed procedures for selecting appropriate sampling positions. Many of these methods have been adopted as national and international standards that have been tested and validated over many years, and are widely accepted throughout the world by governments and industry alike. Since the principles relating to the design of sampling protocols in stacks are universal, it is very likely that they can be applied directly to amine capture plants. Some of the main recognised standard methods for determining sampling positions within stacks and ducts are described in the following sections.

2.2 International Methods

In this section, the main internationally recognised methods regarding large ducts (i.e. > 0.3 m in diameter) are discussed. It is anticipated that these methods will be best suited to the proposed Mongstad plant. Each method provides guidance on how to select suitable sampling positions for measuring flow velocities and/or concentrations of pollutants in waste gas emissions from stacks, flues or ducts. Sampling is performed in a single plane perpendicular to the flow of the gas stream in the stack. All methods are designed to collect samples from flue gas streams where material may be unevenly disturbed throughout the gas flow (especially particulate matter). Consequently, all methods require sampling to be performed at a number of points within the sample plane.

Examination of the methods quickly shows that they are all quite similar and share many common features, although there are some minor differences in the detail between individual methods. The main features common to all methods are:

- Sampling should be performed wherever possible in a steady gas flow parallel with the axis of the stack, and free from the influences of flow disturbances such as bends, obstructions or fans. Accordingly, each method specifies certain minimum distances (in terms of duct diameters) from flow disturbances to ensure straight flow (Figure 2.1). When these conditions are met, sampling is considered “ideal”. However, in some instances the physical layout of the stack may preclude ideal conditions. In these situations, the methods provide for “non-ideal” conditions, usually by specifying a greater number of sampling points within the duct. In general, the direction of the gas flow must be no more than about 15 to 20° from the axis of the stack.

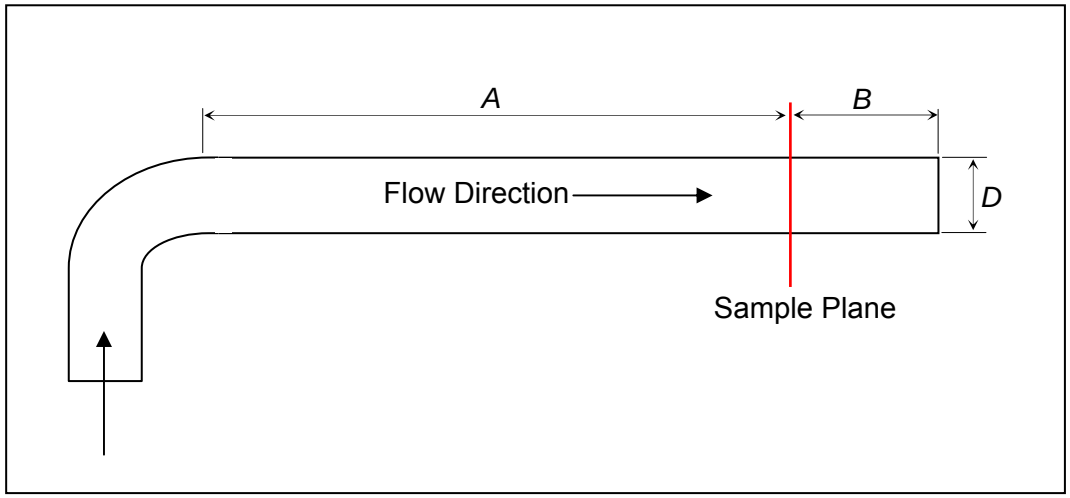


Figure 2.1. Schematic layout of gas flow through a large diameter stack of diameter D. Each method specifies a certain distance downstream (A) and upstream (B) of flow obstruction for ideal sampling conditions. *(Diagram generated by CSIRO)*

The requirement for straight sections of flow means that in vertical stacks, preferred sampling positions may be very high above the ground. This needs to be considered when designing sampling systems to ensure that staff can safely access the ports during sampling operations. Although not mentioned any of these methods, computational fluid dynamics (CFD) simulation may provide additional information on the structure of the flow that may help guide the selection of sampling planes.

- Most methods specify a minimum gas velocity. This is particularly important when measuring the velocity of the gas with Pitot tubes, since measurement uncertainty increases at low velocities.

- The temperature range of the gas is often specified and preferably should be above the dewpoint of the sample gas. There is also usually a maximum permissible range of temperature between adjacent sampling points.

- Gas flows must be in one direction, with no backward flows (i.e. no recirculation within the sampling plane).

- The sampling plane itself is a cross-sectional area perpendicular to the inner walls of the duct that, depending on the size and shape of the duct, is divided into a number of sections of equal area. Samples are taken at the centroid of each section by moving the sample probe and/or Pitot tube across the diameter of the duct (i.e. traversing). Each method specifies the number of traverses and sampling points

necessary for representative sampling, which vary according to the size of the stack. A diagram similar to Figure 2.2 is included in all the standards. This diagram illustrates the “Tangential Rule” for placement of sampling points within the sampling plane of a circular duct. There is no sampling point in the centre of the duct. Some methods also allow a slight variation where an additional point is included in the centre. This is known as the “General Rule”. While the two methods are considered equivalent, the Tangential Rule is useful when the duct diameter is large and it is difficult to reach the centre.

To avoid artefacts induced by disturbances caused by the stack wall, minimum distances from the wall for the sample points are specified. As well, specific information on how many points must be sampled under non-ideal conditions are usually provided in each method. Examples of the sampling plane layout in circular and rectangular section stacks are shown in Figure 2.2.

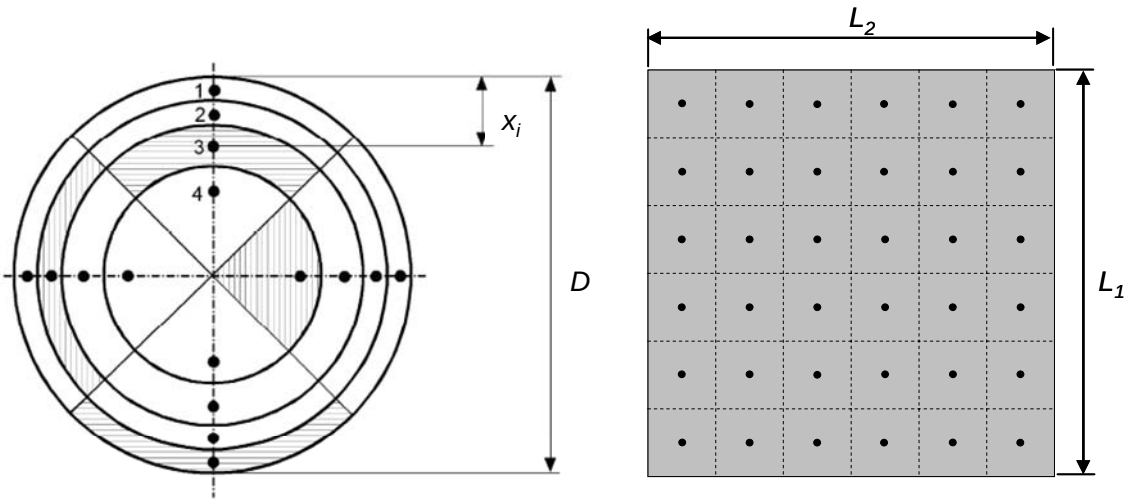


Figure 2.2. Sampling points pattern in a circular duct using Tangential Rule (left) and square duct (right). (Reference VDI 2066)

Some of the more widely used methods are summarised below:

2.2.1 US Standards

USEPA Method 1 – Sample and Velocity Traverses for Stationary Sources

This method has been in use for many years by testing authorities around the world to select sampling ports and traversing points in large diameter stacks and ducts. Method 1 is referenced by many USEPA (and other) methods for testing stack emissions. It is applicable only to large ducts and should not be used for stacks smaller than 0.3 m in diameter or with cross-sectional areas < 0.07 m^2.

In this method, the preferred location of the sample plane is in a straight section of stack or duct at least eight diameters downstream (i.e. dimension *A* in Figure 2.1) and two diameters upstream (dimension *B*) of a disturbance. However, if this is not possible, shorter distances can be accommodated provided that it can be demonstrated that the sample plane is not affected by non-axial (often termed cyclonic) flow. If *A* > *2D* and *B* > *0.5D*, then non-axial flow can be verified according to a ”simplified method” using an S type Pitot tube. However, if *A* <

2D or *B < 0.5D*, the “alternative method” must be used where a directional flow sensing probe is used to measure the gas flow angles across the sample plane (see Section 3). If the gas flow angle is more than 20° off-axis, the sampling location is considered to be unacceptable. This method includes a caveat that flow straightening devices may be used before and/or after the sampling plane to constrain the straightness of the flow across the sampling plane as long as the “alternative method” criterion can be met.

For stacks more than 0.61 m in diameter, the minimum number of sampling points would be 12 when the sample plane is in a preferred section of duct (*A > 8D* and *B > 2D*), but in other locations the number of points is increased. For *A = 2D* and *B = 0.5D*, the minimum would be 24 points.

Method 1 does not nominate a minimum velocity, but other methods designed to measure gas velocity and use Method 1 to define the sample plane do refer to minimum velocities .

VDI 2099 Part 1 - Particulate matter measurement. Dust measurement in flowing gases. Gravimetric determination of dust load

This detailed German standard describes the procedure for gas velocity and measuring particulate matter concentration in stacks and ducts. It also specifies the position of the sampling points. This specification is very similar to the ISO methods, except that a preferred minimum gas velocity is recommended. Like some other methods for determining particulate matter, it is preferred that the sampling plane be located in a vertical section of duct.

Rather than prescribing minimum duct diameter distances before and after a flow disturbance the acceptability of a sampling plane position is determined by the absence of cyclonic flow (average resultant velocity angle over the sampling plane <15°). In this aspect this method is similar to USEPA Method 1 “*Alternative Procedure*” and provides additional flexibility in assessment of a sampling plane. Also, this method includes the introduction of “*suitable structural measures*” or vanes to improve the flow characteristics at the sampling position as is the case with USEPA Method 1.

Californian Air Resources Board Method 1 – Sample and Velocity Traverses for Stationary Sources

This method’s scope and application are the same as those in USEPA Method 1.

ASTM D 3154 -00 Standard Test Method for Average Velocity in a Duct (Pitot Tube Method)

This standard method describes measuring the velocity of a gas stream in a stack, duct or flue. Selection of a suitable sampling plane is also discussed. Like the other two US methods, the ASTM states that the sample plane should be located eight duct diameters downstream and two diameters upstream from any flow disturbance. Other non-ideal locations may be used provided that they are more than two diameters away from any flow disturbances.

The ASTM method requires verification of the absence of non-axial flow, using a procedure similar to the simplified method in USEPA Method 1. However, in this case, the maximum permissible off-axis flow angle is 10° (rather than 20° as used in the USEPA method), before the flow is considered unacceptable.

Uniform gas flows are required through the sampling plane so that 80 to 90 % of the measurements are greater than 10 % of the maximum velocity. As well, the minimum gas flow velocity is 3 m s^{-1}.

While no gas temperature is specified, the method does require that the gas temperature be measured to within ±1°C when the stack gas is suspected of being saturated or containing water droplets.

2.2.2 International Standards

The ISO standards do not specifically relate to sample plane selection. However, detailed criteria are provided in two standards:

ISO 9096 – Stationary source emissions – Manual determination of mass concentration of particulate matter;

ISO 10780 – Stationary source emissions – Measurement of velocity and volume flowrate of gas streams in ducts.

Both of these documents describe the process for selecting sampling planes in large ducts, which is the same in both cases. The requirements specified are generally similar to the US methods, except that the number of duct diameters from flow disturbances is slightly different. Here, a straight length of duct at least seven duct diameters long is preferred, with $A \geq 5D$ and $B \geq 2D$. A further requirement of ideal sampling is that the sample plane be at least 5*D* from the outlet of the duct. Other conditions can be used but in such cases, the stated accuracy for velocity measurements of ±5 % is not guaranteed (in ISO 9096). The maximum allowable deviation from straight flow is 15°. No correction factors are specified for the number of sampling points in a non-ideal sampling plane position.

A minimum velocity is not directly defined in either document although ISO 10780 specifies that the flow must be sufficient to yield a differential pressure across the Pitot tube > 5 Pa. ISO 9096 also requires that the highest to lowest flow velocity ratio should be less than 3:1.

Although the temperature range of the gas is not specified, it suggested that the temperature be monitored at the sampling points in the duct to “provide an indication of the steadiness of the stationary source operations”.

2.2.3 British/European Standards

BS EN 15259:2007 - Air quality. Measurement of stationary source missions. Requirements for measurement sections and sites and for the measurement objective, plan and report.

Requirements for measuring air pollutants in waste gas ducts at industrial plants are provided in this standard. It is suggested that the method is preferred for vertical stacks rather than horizontal duct in recognition of the fact that particulate material has a tendency to concentrate in horizontal flows under the influence of gravity (note that this applies equally to solid and liquid aerosols). The method otherwise is virtually identical to the ISO methods.

In this method, ideal sampling is achieved when the sample plane is located at least 5*D* downstream and 2*D* downstream of a disturbance. Like the ISO methods, the sample plane should also be no less than 5*D* from the top of a stack.

The gas flow needs to be at angle less than 15° with regard to duct axis, with no backward flow in the sampling plane. The minimum flow is defined as sufficient to produce a differential pressure of more than 5 Pa across a Pitot tube. The ratio of the highest to lowest local gas velocities of less than 3:1.

BS EN 13284-1:2002 Stationary source emissions. Determination of low range mass concentration of dust. Part 1: Manual gravimetric method

This method is designed for measuring particulate emissions from waste incinerators, although it can be applied to other facilities. The design of the sampling plane and selection of sampling points are identical to the requirements in BS EN 15259:2007.

NF X 44-052 Stationary source emissions — Determination of high range mass concentration of dust — Manual gravimetric method

This is a French method and is very similar in scope and procedures to the German method VDI 2099 Part 1. The procedure for sampling is the same as the ISO methods.

2.2.4 Australian Standards

In Australia, the preferred method for sampling from stationary sources is:

Australian Standard AS 4323.1 – 1995 Stationary Sources Method 1: Selection of Sampling Positions

This standard is usually prescribed by Australian government regulators. AS 4323.1 sets out a method for selecting sampling positions to obtain representative samples from stacks, ducts or other similar conduits. This method requires six to eight duct diameters downstream and two to three duct diameters upstream from a flow disturbance, depending on the obstruction. For bends, junctions, dampers etc. six and two diameters are considered sufficient to provide straight flow (i.e. ideal conditions) whereas fans are thought to cause a greater effect and hence require longer lengths of duct. Non-ideal sampling is accommodated by increasing the number of sampling points within the sample plane and a series of factors are given to determine the number points required. For example, if the sample plane position is four or more diameters less than ideal, the number of sample points is increased by a factor of 1.2, rounded up to the next whole number.

For circular ducts, the tangential rule is applied when selecting the position of the sampling points, i.e. there is no point in the centre of the duct. For all sampling, the angle of gas flow must be no greater than 15° with regard to duct axis, regardless of whether the conditions are ideal or not.

This standard also requires a minimum gas velocity of 3 m s^{-1} and the ratio of the highest to lowest Pitot tube differential pressure must not exceed 9:1. It is further specified that when conducting isokinetic sampling using impingers, the velocity ratio across the plane should be no more than 1.6:1.

This method suggests that the gas temperature should be above the dewpoint and also specifies that the temperature across the traverse should not vary by more than 10 % of the mean.

AS 4323.1 also has a specific reference to the requirements for sampling gaseous materials. It recognises that the sampling requirements for gases in a well mixed stream are much less stringent that for non-homogeneous components (such as particulates and liquid aerosols). In an appendix to the standard therefore, it suggests that under these conditions, single point sampling from any part of the stack will suffice. However, the point is made that the homogeneity of the system should be first determined before adopting this approach. It should also be remembered that if the gas concentration data are to be used for determine emission flux, the velocity of the gas stream must be measured, usually in accordance with the normal sampling plane setup.

2.2.5 Differences between Methods

It is apparent that the various methods are generally quite similar, although there are some important differences. The key differences are indicated in Table 2.1.

CSIRO recommends either USEPA Method 1 or VDI 2066 for locating and experimentally validating the flow conditions at the selected sampling plane. Both methods are experimentally and scientifically robust and provide a stepwise approach to the assessment process. For the Mongstad facility both methods would provide an equally flexible approach which allows the placement in nearly any duct position as long as it can be experimentally demonstrated that the “straight flow” criteria of each method can be achieved. The VDI 2066 has a slightly more stringent criteria with respect to the cyclonic flow (<5°) and it is this method that CSIRO recommends for the Mongstad facility.

When comparing various methods it can be instructive to use an example to illustrate the differences. Using the design criteria provided to CSIRO regarding the large scale capture plant proposed for the Mongstad facility, the sampling plane was determined according to various methods. The stack in this example is circular and 2.5 m in diameter. We assumed that the sampling plane was located within ideal flow conditions.

The methods considered were USEPA Method1, ISO 9096 and AS 4323.1. The other American methods yield the same results as Method 1 and the other European methods yield results that are equivalent to the ISO result.

Table 2.1. Comparison of the principal characteristics of various international methods (NS = not specified).

	Relative Ranking see note 4	Min Stack Diameter (m)	Dimension A (preferred)	Dimension B (preferred)	Dimension A (minimum)	Dimension B (minimum)	Max Off-Axis Angle (deg)	Min Number of Sampling points (see note 3)	Min Velocity ($m\ s^{-1}$)
USEPA Method 1	1	0.3	8D	2D	See note 1	See note 1	20	12	NS
VDI 2099	1	<0.35			See note 2	See note 2	15	12	Pitot DP > 5 Pa
CARB Method 1	2	0.3	8D	2D	See note 1	See note 1	20	12	NS
ASTM D3154	2	NS	8D	2D	2D	2D	10	20	3
ISO 9096	2	NS	5D	2D	NS	NS	15	17	Pitot DP > 5 Pa
ISO 10780	2	> 0.07 m^2	5D	2D	NS	NS	15	17	Pitot DP > 5 Pa
BS EN 13284	2		5D	2D			15	12	Pitot DP > 5 Pa
BS EN 15259	2		5D	2D			15	12	Pitot DP > 5 Pa
AS 4323.1	2	0.2	6D-8D depending on disturbance	2D-3D depending on disturbance	NS	NS	15	20	3

Note 1: USEPA Method 1 minimum dimensions from obstructions are determined by the absence of cyclonic flow (<20°)

Note 2: VDI 2099 distance from disturbances determined by absence of cyclonic flow (<15°)

Note 3: Minimum number of sampling points respective to the Mongstad facility

Note 4: Ranking based upon the detail of the experimental methodology and the ability to provide scientifically based alternative experimental approached to identify and validate sampling positions in non-ideal positions.

(Table generated by CSIRO)

USEPA Method 1

Since the duct is greater than 0.61 m in diameter and we have assumed ideal conditions with 25m of 'straight flow' (the 8*D*/2*D* criterion applies) a minimum of 12 sampling points is required. The traverse points are located along two perpendicular traverses with six points located on each traverse. The distance from the stack wall for each point on each traverse is shown in Figure 2.3.

Traverse Point	Distance from Wall (m)
1	0.11
2	0.365
3	0.74
4	1.76
5	2.135
6	2.39

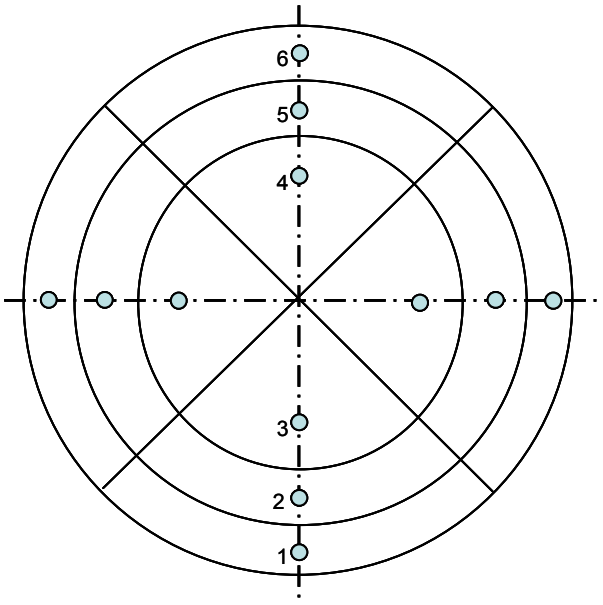


Figure 2.3. Layout for ideal sampling plane in a 2.5 m diameter stack using USEPA Method 1. ***(Table generated by CSIRO)***

Further conditions occur when a traverse points falls within a distance of 2.5cm from the stack wall, however, these additional criteria only applicable to ducts with diameters smaller than that of the Mongstad facility and are not considered.

If the 8*D*/2*D* criterion, that was assumed for above example, is not achievable (i.e. a minimum of 25m of straight flow for the Mongstad facility), then USEPA Method 1 offers two further procedures to assess the suitability of a sampling plane position. The first procedure applies where the disturbance is less than the *8D/2D* criterion but greater than 2 stack diameters downstream and 0.5 stack diameters upstream of a flow disturbance (*2D/0.5D* criterion). For this case the number of sampling positions is increased to at least 24 points. It should be noted that both of the above procedures require the experimental assessment of the sampling position to preclude the existence of cyclonic flow.

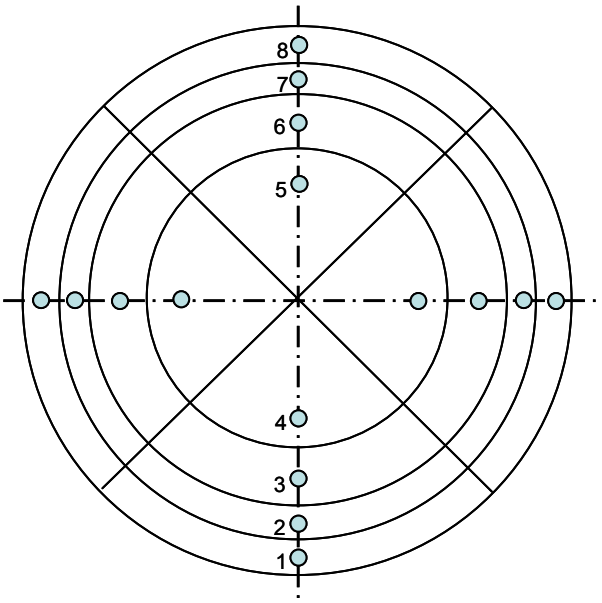
The third procedure applies where the *2D/0.5D* criterion cannot be met and involves the experimental measurement of the velocity vector at 40 or more traverse points across the duct to determine the acceptability of the position. The position is acceptable if the average resultant angle is within 20 degrees and the standard deviation of these measurements is within 10 degrees. This is a unique procedure not found in other international methods. Additionally, it is acceptable to install flow straightening devices before and/or after the sampling plane in situations where the resultant velocity angle over the sampling plane is greater than 20 degrees and/or the standard deviation is greater than 10 degrees. This method has been found useful for CSIRO for validation (or otherwise) of sampling positions

that have not met the *2D/0.5D* criterion. In cases where it has been required to design and install flow straightening devices at sampling locations CFD modelling has been utilised to calculate the performance of the flow strengtheners with respect to the above acceptability criteria.

ISO 9096

With the ISO method, at least two traverses are required for circular ducts more than 2 m in diameter, and each traverse must have at least eight points, i.e. 16 in total. If the General Rule is followed and additional point is taken in the centre of the duct. Details of the sample plane according to the ISO method are shown in Figure 2.4.

Traverse Point	Distance from Wall (m)
1	0.083
2	0.263
3	0.485
4	0.808
5	1.693
6	2.015
7	2.238
8	2.418



(Table generated by CSIRO)

Figure 2.4. Layout for ideal sampling plane in a 2.5 m diameter stack using ISO 9096.

AS 4323.1

The sample plane according to AS 4323.1 is identical to that determined by ISO 9096 shown above. If the flow conditions are non-ideal, additional points are taken, the number of which is determined by applying a factor. If the sample plane is located four or more diameters off ideal, the number of sample points is increased by a factor of 1.2, rounded up to the next whole number. Hence for a 2.5 m diameter stack the minimum number of sample points is 20 rather than 16 specified in ideal flow.

2.2.6 Sampling Plane Summary for the Proposed Mongstad Facility

The following tables provide a summary of the minimum conditions/dimensions required for the proposed Mongstad facility to enable the stack sampling position to comply with international sampling methods. The stack widths used are 2.5m and 7.0m, square or circular cross sections. It should be noted that the international methods shown in Table 2.1 do not differentiate between an absorber plant, pilot plant or a full scale facility. The criteria and experimental methods used to identify and validate a sampling location are based on the fluid dynamics at the sampling plane and not the type of facility or process. Table 2.2 shows

the minimum length of straight duct needed to comply with each of the international methods identified in table 2.1.

Table 2.2. Summary of minimum sampling plane criteria – The 'Ideal' or preferred case *(Simplified criterion for USEPA Method 1).*

	Stack Diameter 2.5 m			Stack Diameter 7 m			
	Dimension A (m)	Dimension B (m)	Minimum Straight Duct length (m)	Dimension A (m)	Dimension B (m)	Minimum Straight Duct length (m)	Min Number of Sampling points
USEPA Method 1 CARB Method 1 ASTM D3154	20	5	25	56	14	70	12
VDI 2099	See note 2	See note 2	See note 2	See note 2	See note 2	See note 2	20
ISO 9096 ISO 10780	12.5	5	13	35	14	49	17
BS EN 13284 BS EN 15259	12.5	5	13	35	14	49	12
AS 4323.1	15-20*	5-7.5*	20 – 27.5*	42 -56	14 -21	56 – 78*	20

* depending on disturbance *(Table generated by CSIRO)*

Note 1: VDI 2099 distance from disturbances determined by absence of cyclonic flow (<15°)

For the situation where it is not feasible to locate sampling planes in positions that comply with the distances shown in table 2.2, USEPA Method 1, VDI 2099 and AS4323.1 provide further criterion that can be used identify a suitable sampling position. The "Alternative" method, (USEPA Method 1) of "Non-ideal" method (AS 4323.1) uses a greater number of sampling points to maintain the accuracy of the measurement. The following table shows the minimum distances of straight duct required at the Mongstad facility for sampling locations that are "non-ideal".

Table 2.3. Summary of minimum sampling plane criteria – The 'Non-ideal' case *(Alternative criterion for USEPA Method 1).*

		Stack Diameter 2.5 m			Stack Diameter 7 m			
	Distance From Disturbance Upstream/downs	Dimension A (m)	Dimension B (m)	Minimum Straight Duct length (m)	Dimension A (m)	Dimension B (m)	Minimum Straight Duct length (m)	Min Number of Sampling points
USEPA Method 1 CARB Method 1	2/0.5 5/1.25 6/1.5	5 12.5 15	1.3 3.2 3.8	6.3 15.7 18.8	14 35 42	3.5 8.8 10.5	17.5 43.8 52.5	24/25[a] 20 16
VDI 2099		See note 2	See note 2	See note 2	See note 2	See note 2	See note 2	20
AS 4323.1	6/1.5 4/.0.5	15 10	3.8 1.3	18.8 11.3	42 28	10.5 3.5	52.5 31.5	27/40** 30/46**

** depending on disturbance (Table generated by CSIRO)*
*** for 2.5m duct/ 7m duct and depends on the relative distance of dimension "A" and "B" and on the disturbance type.*
Note 1: VDI 2099 distance from disturbances determined by absence of cyclonic flow (<15°)

(a) larger number is for rectangular ducts

Table 2.3 shows that the minimum straight duct length required at the proposed Mongstad facility to comply with USEPA Method 1 (*Alternative method)*. These are 5.3m of straight duct for a 2.5m emissions stack and 17.5m for a 7m emissions stack and the sampling resolution of 24 or 25 points for each duct depending on duct geometry. To comply with AS 4323.1 these minimum lengths are 11.3m of straight duct for a 2.5m diameter stack and 31.5m of straight duct for the 7m diameter stack and with a sampling resolution of 30 to 46 points depending on the type of disturbance. For VDI 2099 minimum distances from a disturbance are not prescribed and the conformity of a sampling position is determined by the absence of cyclonic flow (<15°).

Both the USEPA Method 1 and VDI 2099 provide further criteria to evaluate the suitability of non-compliant sampling positions at a facility and these may be applicable to the proposed Mongstad Facility if the above minimum duct lengths are excessive. These criterion do not restrict the sampling position to a minimum distance from a disturbance but evaluate the position experimentally through the absence of cyclonic flow (<15° for VDI 2099 and <20° for USEPA Method 1). These of these criterion also allow the installation of "flow straightening" devices such as vanes to artificially produce a compliant sampling position in an otherwise non-conforming position. The application of these criteria is not simple and depends upon the individual geometry and flow condition and required the application of computational fluid

dynamics. Thus these further criteria must be considered on a case by case basis rather than a generic application of a standard.

2.3 Other Considerations

The general criteria required to select sampling planes for proper representative sampling are covered by any of the standard methods listed above. However, in addition to these requirements are a number of other aspects that can affect the utility of the sampling system. In particular it is essential to experimentally validate the sampling plane to ensure that the flow characteristics are well defined and within the prescribed tolerances. As well, more prosaic aspects such as the design of the access ports, physical layout of the work area where sampling will be performed and staff safety must be addressed when designing the sampling protocol. Some of these aspects, based on CSIRO experience, are discussed below.

2.3.1 Minimum Gas Velocity

When sampling aerosols (both solid and liquid) in horizontal ducts, the gas velocity must be greater than the fall velocity of the largest particles or else the largest particles will collect at the floor of the duct. As a result, some, but not all methods for determining particulate matter note that measurements should be in vertical stacks where possible. This aspect is important for emission sources with a wide range in particle diameters such as coal-fired power stations, but at this stage its relevance to the proposed Mongstad plant is not known.

2.3.2 Experimental Validation of Sampling Plane Positions.

The standard methods discussed above specify certain limits on the deviations from axial flow. It is important therefore to experimentally measure the flow conditions within the sample plane to ensure that they comply with the relevant method. Some methods include procedures for measuring flow (e.g. USEPA Method 1) although most do not. The suggested procedures usually state that the non-axial component of the flow can be determined by the use of Pitot tubes, however, in our experience these are not particularly well suited to the task. Since Pitot tubes only measure in one plane they therefore only determine the yaw component of the velocity vector (indeed, they are designed to operate correctly in straight flow). It is preferable to use a three-dimensional system for measuring all components of the flow such as that described in USEPA Method 2F: Determination of Stack Gas Velocity and Volumetric Flow Rate with Three-Dimensional Probes. This method is capable of measuring all of the vectors associated with the flow and provides a much more detailed picture of the flow at each sampling point.

Usually the flow profile will remain constant if the flow rates are kept within a relatively narrow range and there are no changes to the geometry of the duct (such as altering the position of dampers etc.). Consequently, validation is generally only needed once.

2.3.3 Use of Flow Correcting Devices

Both USEPA Method 1 and VDI 2066 allow flow correcting devices to constrain the flow across a sampling plane. However, these “insertions” as they are called in VDI 2066 “*must not cause any dust deposition and shall not affect the effectiveness of any clean-up stream precipitators*”. Essentially the flow correcting devices must not influence the particle or

aerosol in any way. The correct design and installation of these devices is not a trivial and CSIRO has had experience in this area. CSIRO has utilised USEPA “alternative method” to retrofit sampling planes to existing coal fired electricity generating power stations to produce a sampling plane in a duct position that would otherwise fail the USEPA acceptability criteria. The process involves CFD to theoretically identify and validate designs and examine the influence of these upon the particle or aerosol flow. A suitable flow correcting device is subsequently constructed and installed and the flow condition across the sampling plane validated using the USEPA Method 1 “Alternative procedure”. If the acceptability criteria is not achieved then either a new position is identified or a new design of flow correcting device designed. For the Mongstad facility flow correcting devices should only be utilised if absolutely necessary and as such it is recommended that that at a minimum the USEPA (2D/0.5D) criterion be achieved which for the Mongstad facility flow requires a minimum of 6.25m of straight duct.

2.3.4 Design of Access Ports

Any stack sampling system needs a certain number of access ports so that Pitot tubes, sample lines, filter holders and other equipment can be inserted into the gas flow. These are usually fixed into the stack and fitted with caps or plugs that can be removed during sampling. Most of the standard methods discussed in Section 2.2 specify the minimum number of access ports required for a stack of a particular size. Round stacks, such as at the proposed Mongstad facility, require a minimum of four access ports. The minimum number of sampling ports for square or rectangular stacks depends upon the dimensions of the duct and whether access into the duct is horizontally or vertically. Square or rectangular ducts with vertical aligned access require a minimum of 3 or 4 access ports, depending upon the international method used. Horizontally aligned access positions would require 6 or 8 access ports, depending upon the method used with these access ports placed in opposing positions on each side of the duct.

Some, such as AS 4323.1 and VDI 2099 also provide quite detailed guidance on the design and installation requirements of fixed sample ports. VDI 2099 provides the most appropriate design and dimensions for sampling ports for the proposed Mongstad facility. The ports detailed in VDI 2099 are of sufficient size to enable the insertion of new designs of “in stack” sampling apparatus that will be required to capture the unique emissions from the plant. However, it is recommended that to the sampling port modification discussed in the following section be included in the port design. This modification enables the sealing plate of the access port to form a smooth fit with the inside surface of exhaust duct resulting in minimal of gas stagnation in the region of sampling port.

It is common for sampling flange designs, including those found in AS 4323.1 and VDI 2099, to extend outwards and away from the duct to facilitate the mounting (bolting or clamping) of the probe supporting plate and to penetrate through any thermal insulation covering the duct. However, depending upon the size and design of the flange, a cavity is formed between the surface level of the duct and the probe mounting plate (Figure 2.6). It is our experience that this design can, where a large cavity is formed by the access port, produce gas stagnation with the influence of this gas stagnation extending into the sampling area.

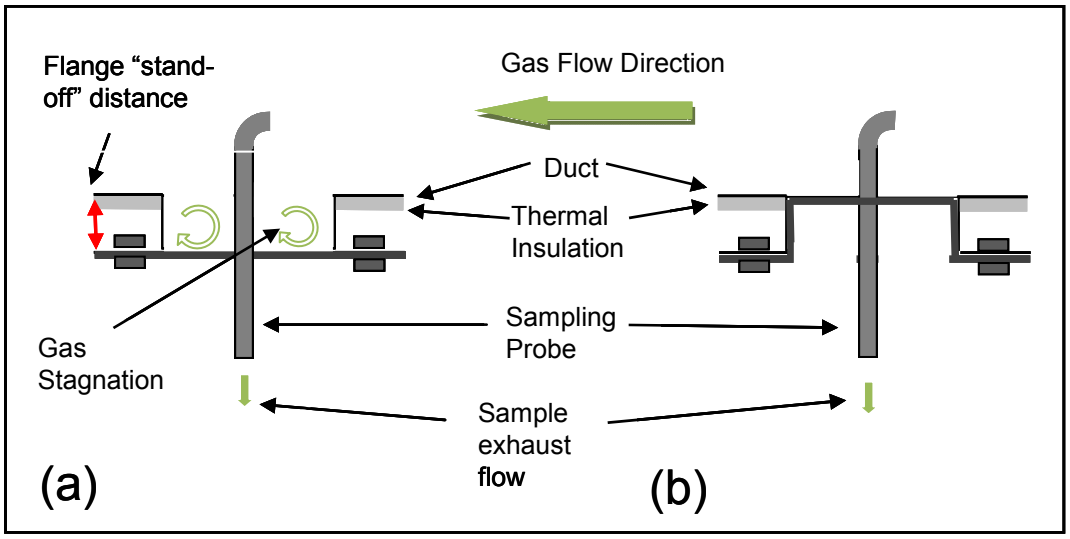


Figure 2.6. Formation of stagnation air with conventional designed sampling flange (a) and a design that minimises gas stagnation (b). *(Diagram prepared by CSIRO*)

This can result in increased errors for manual sampling measurements closest to the duct walls. Ideally, the surface that the manual stack sampling flange presents to the gas flow should be a smooth surface that is flush with the internal surface of the duct. Additionally, gas stagnation can cause deposition or condensation of material that can in some situations corrode the sampling housing. Figure 2.7a shows an example of a conventional sampling flange (as depicted in Figure 2.6a) but in this case two separate sampling assemblies are capturing different analytes.

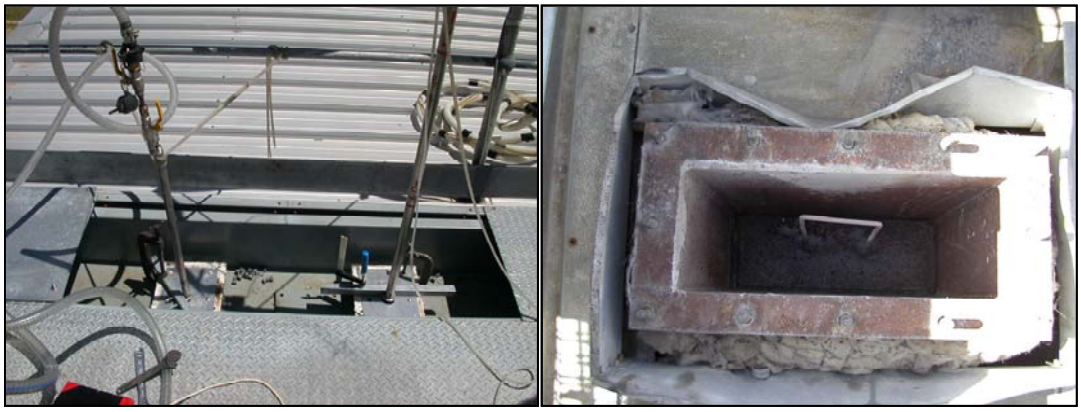


Figure 2.7. Rectangular section sampling ports: (a) sampling probes installed; (b) showing flange fitting to eliminate gas stagnation. (Copyright CSIRO, CCSD 2010)

Figure 2.7a displays a conventional mounting flange with the alignment surface raised ~100 mm above the steel ductwork. Notice that the manual sampling flanges in Figure 2.7a, of which there were four, are recessed within a larger cavity. The cavity bounded by insulation is covered with aluminium sheeting. In this case the sampling point was located after a fabric filter (FF) cleaning device at a coal-fired power station. The FF outlet gas temperature was 110 to 120 °C and thus the exposed steel surfaces of the duct that can be seen in this figure were also at these temperatures. Thus this sampling point design poses

significant occupational health and safety challenges for personnel undertaking the stack sampling operations at this plane.

Figure 2.7b displays a similar sampling flange but in this case it is located at the inlet to the electrostatic precipitator (ESP). However, the design of this sampling flange is non-conventional. The sampling probe and mounting plate in this case is not attached to the outer surface of the flange as shown in Figure 2.7a but instead rests within the flange cavity and seals directly against the steel surface of the exhaust flue. Thus a smooth gas flow along the duct internal surface is maintained at the sampling flange interface.

In summary, it is important to utilise manual sampling point flanges that have minimal influence on the flow characteristics. With good engineering practices, these artefacts and errors can be eliminated.

2.3.5 Dimensions of Manual Sampling Ports

Often sampling in stacks involves inserting the sample collection devices into the gas stream. These in-stack procedures have a number of advantages compared to out-of-stack sampling techniques and are preferred where sample loss due to deposition, surface adsorption or reactions within sample probe are significant, or the analyte is at trace concentration. It is only used in larger ducts where insertion of the collection device has negligible influence on the exhaust flow. However, in-stack sampling requires suitably sized sampling access ports to enable the entire sampling device as well as associated flow and temperature sensors to be inserted entirely within the duct. It has been found that many sampling ports are inadequate for this method due to their small size.

Figure 2.8 shows display examples of two in-stack sampling devices.

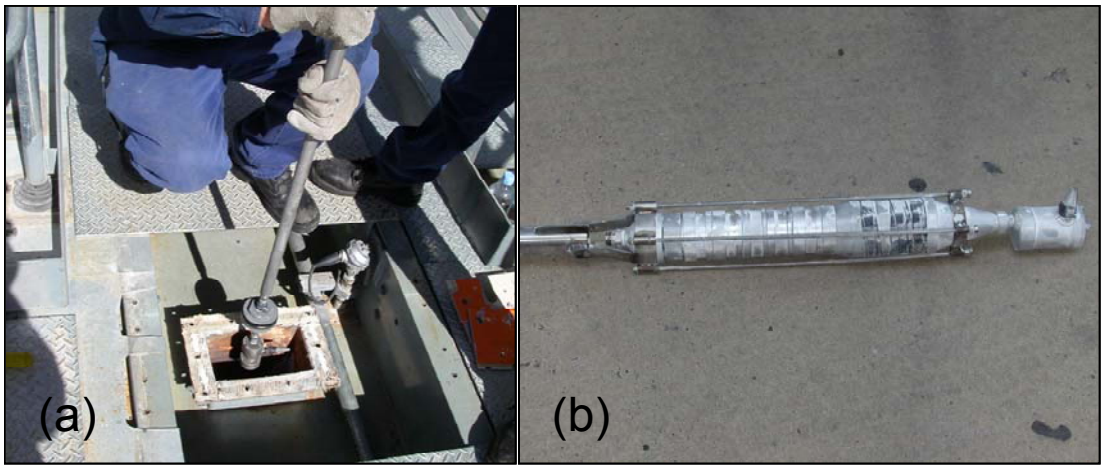


Figure 2.8. Two devices used during experimental in-stack sampling operations.
(Copyright CSIRO, CCSD 2010)

Figure 2.8a additionally displays a manual sampling flange that is large enough to insert sampling equipment such as those shown in these two figures. Clearly, large ports are an advantage for in-stack sampling, and if possible, large ports should be considered when constructing the sampling system such as described in VDI2066.

2.3.6 Access to the Sampling Position

Although the accuracy of a stack sampling is not directly influenced by the ease of access to the sampling point position, it can significantly affect the time required to perform traverses and the safety of personnel. In situations where access is difficult, the extra time needed may prevent short term temporal variations in the flow from being captured.

An example of poor access is shown in Figure 2.9 where temporary scaffolding has been erected next to the sample ports of an exhaust duct. The positioning of the scaffolding restricted access to the sampling points to a degree where partial disassembly of the scaffold was necessary to access some ports. These pictures highlight the difficulty of inserting long probes into a duct where the scaffolding structures used to support personnel place obstructions in the insertion path of long sampling probes.

Figure 2.9. Temporary scaffolding used to support personnel support while sampling: (a) scaffolding treads were removed to enable measurements whereas in (b) horizontal scaffolding structural sections were remove to allow sampling probe insertion.
(Copyright CSIRO 2010)

Another example of improperly designed manual sampling positions is shown in Figure 2.10, where in this case, the insertion of a three-dimensional Pitot probe was obstructed. Figure 2.10a displays a simple oversight of not allowing adequate clearance to enable the probe to penetrate fully across the duct. While this error is obvious from these images, good sampling flange design and positioning is often overlooked during plant design. In this case, only slightly more than 60 % of the duct could be sampled. Figure 2.10b displays a second example of sampling probe obstruction where in this case a safety railing places additionally obstruction to the alignment of the probe.

Figure 2.10. Examples of obstruction interfering with the insertion of sampling probes: (a) too close to the ground; (b) hand rail in wrong position. ***(Copyright CSIRO 2010)***

The two examples above were at ground level; however, sampling points are often located on elevated sections of the stack, and this is likely to be the case at an amine scrubbing plant. In these situations, poor access may have significant safety implications for staff performing the sampling but also for personnel working underneath.

Figure 2.11 displays the importance of installing well designed personnel platforms at manual stack sampling positions.

Figure 2.11. Examples of obstruction from incorrect installation of personnel platforms: (a) work platforms on ductwork; (b) three-dimensional Pitot tube probe inserted in port. *

Figure 2.11a shows the personnel access platforms for sample ports installed on a large duct, while Figure 2.11b shows one of the sampling points with a probe inserted. In Figure 2.12 below, the difficulty in manoeuvring a 6 m probe through the hand railings at this location can be appreciated. In this case three of the ports could not be accessed due to the hand railing obstructing access.

Figure 2.12. Example of poorly designed work platform. *

** CSIRO Acknowledges Malfroy Environmental Strategies P/L as partners in the measurements displayed in figures 2.11 and 2.12. (Copyright CSIRO/ Malfroy Environmental Strategies 2010)*

While Figures 2.9 through to Figure 2.12 illustrate a number of inadequacies in sampling point positions with respect to obstructions, Figures 2.13 and Figure 2.14 display well engineered and sampling ports that have been installed to minimise the uncertainty of stack sampling procedure but also provide good access. In this example, the stack is of annular design with two circular steel flues contained within a concrete supporting structure. The sampling position is in about the middle of the stack about 60 m above the ground (Figure 2.13). It should be noted that the 120 m height of this stack was designed of optimal plume dispersions and not to optimise stack sampling operations. The sampling plane in this facility easily complies with USEPA Method 1 “simplified procedure” which at a minimum required 40 m of straight flow before the sampling plane and 10m of straight flow after the sampling plane.

Figure 2.13. Position of stack sampling points that comply with USEPA simplified procedure. *(Copyright CSIRO, CCSD 2010)*

The arrangement of the sampling points within the exhaust stack is shown in Figure 2.14.

Figure 2.14. An example of the well designed and positioned sampling points within the stack shown in Figure 2.13: (a) sample ports visible; (b) online gas analysers installed in sample plane. ***(Copyright CSIRO, CCSD 2010)***

Figure 2.14a displays four of the eight sampling ports (covered in insulation in this figure). These ports are of conventional design with a small protrusion away from the circular duct to facilitate the bolting of manual sampling locating plates. The work platform around the sampling ports is sufficiently large to allow easy access and utilise long probes that can penetrate fully through the flue duct. The access ports are also positioned at a suitable height above the floor for convenient and safe operation.

Figure 2.14b displays installed online gaseous sampling instrumentation in the sampling ports on the opposite side to those displayed in Figure 2.14a. Placing the sample ports near online systems can be useful since manual sampling methods are often used to verify the results from continuous analysers.

2.3.7 Sampling Requirements at Pilot Scale and Full Scale Plants.

As far as we are aware no special considerations are in existence in stack sampling requirements of pilot scale plants. However, as the stack diameters of pilot scale plants are considerably smaller than full scale plants, the sampling criterion will generally require fewer and usually only a single stack sampling position. VDI 2066 accommodates the sampling of small ducts without the need for additional regulations. Ducts with areas of less than 0.1 m^2 can be sampled using this method as shown in Table 2.4.

In situations where sampling is required in small ducts (<0.5 m) the influence of the sampling apparatus that is inserted into the duct needs to be considered and evaluated. As a rule of thumb and from CSIRO's experience, ducts with diameters larger than ~1 m diameter can be successfully sampled using in-stack sampling methods. Ducts with diameters less than ~1 m require out-of-stack sampling methods as the insertion of the sampling device may alter the flow condition at the sampling position. It is our preference to use "out of stack" sampling methods for ducts less than 1m diameter. Most commercial stack sampling equipment is suitable for ducts down to at least 1m diameter. Very small ducts with diameters of less than ~0.3 m require further careful consideration regarding the influence of the stack sampling nozzle assembly and the associated and attached flow and temperature measuring devices. To fully examine the influence of the sampling device on the duct flow must be considered wind tunnel testing is often required and these tests are also used to validate that the

sampling nozzle has negligible influence upon the flow conditions within the small duct. It is normal for small ducts to remove the flow and temperature sensors that are normally attached to the sampling probe and insert these devices a distance behind the position of the sampling probe. Alternatively, specialised streamlined and/or miniature probes can be manufactured to undertake the sampling in cases where commercial sampling equipment is found to be unsuitable.

Table 2.4. Summary of minimum sampling points relative to sampling plane areas.

Range of sampling plane areas in m^2	Minimum number of sampling points
<0.1	1 (1)
0.1 to 1.0	4
1.1 to 2.0	9
>2	At least 12 and 4 per m^2 (2)

(1) Using only one sampling point may give rise to errors greater than those specified in the guidelines of VDI 2066.
(2) For large ducts, a number of 20 sampling points is generally sufficient.
(Reference VDI method 2066)

3 GAS FLOW MEASUREMENT

Emissions monitoring usually involves the reporting of the flux of particular species. It is therefore necessary to measure accurately both the volumetric flow rate and the concentration of the compounds of interest. International standard sampling methods treat these two measurements separately as the technique used for measuring of the concentration of target species dependents upon characteristics of the such as its phase. This current section considers gas velocity and flux measurements independent of the sampling techniques.

Figure 3.1 shows a gas flow example where the velocity of the gas flow varies significantly across the duct. In this example (which is consistent with the size and flow rate envisaged from an amine plant), the gas velocity within a section of horizontal duct is plotted as a function of position. Over only a short distance the velocity varied between about 20 and 34 m s^{-1}. To accommodate such large variations, velocity measurements must be made by traversing through the gas stream using a sampling pattern as defined by the standards discussed above.

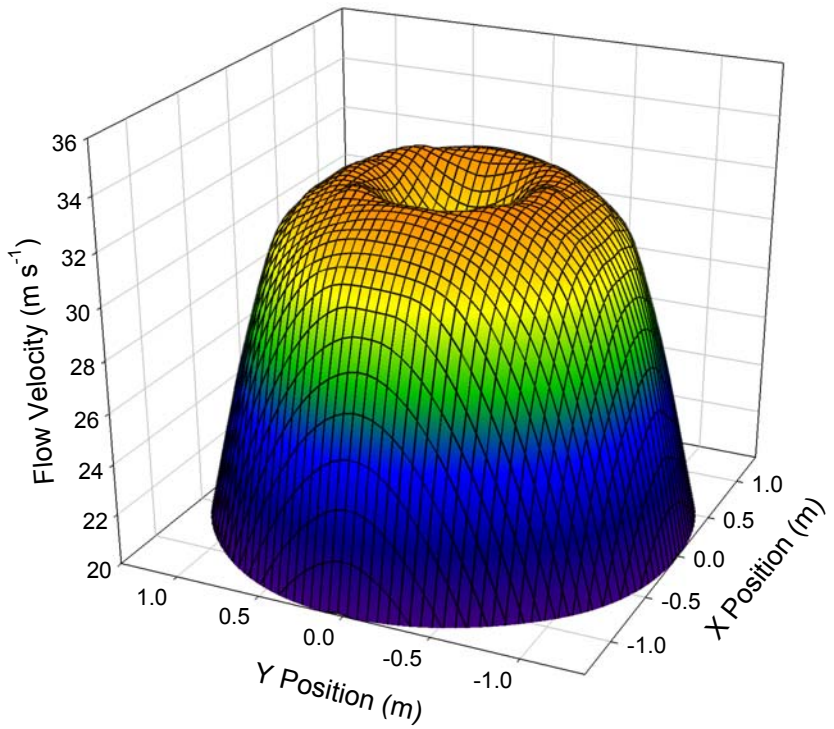


Figure 3.1. Velocity profile measured by CSIRO with a three-dimensional Pitot probe system (according to USEPA Method 2F) in a 3 m diameter duct. ***(Copyright CSIRO 2010)***

A common requirement of most standard methods is that velocity measurements should be made in ideal conditions, i.e. in steady state flow, and with the number and position of the sampling points consistent with those specified for sample collection discussed above. If the sample plane is non-ideal, the same caveats that apply to sampling under non-ideal conditions apply to measuring the velocity.

Some of the more common standard methods for measuring velocity of stack gases are:

- USEPA Method 2 – Determination of stack gas velocity and volumetric flow rate (type S Pitot tube)
- ISO 10780:1994 Stationary source emissions – Measurement of velocity and volume flow rate of gas streams in ducts
- VDI 2066:2006 Particulate matter measurement. Dust measurement in flowing gases. Gravimetric determination of dust load.

The standard methods above rely on the use of ‘standard’ and “S-type” Pitot tubes to measure the flow velocity within the duct. “S-type” Pitot tubes are more commonly used for stack sampling as they are less susceptible to clogging from particles. These methods assume that the gas flow is laminar and orthogonal to the sampling plane with a maximum allowable ‘off-axis’ angular deviation of 20 degrees for USEPA Method 2 and 15 degrees of angle for VDI 2066. However, as these methods measure only a component of axial velocity and as such have increased error when these measurements are used when the resultant

velocity vector approaches the maximum allowable angular deviation. For increased accuracy of flow measurements methods such as: USEPA Method 2F – Determination of Stack Gas Velocity and Volumetric Flow Rate with Three-dimensional probes should be used, as these measurements determine the true axial velocity of the gas flow at the sampling position.

This method employs a three-dimensional Pitot probe to measure the axial flow velocity where the pitch (up and down) and yaw (side-to-side) angles associated with the velocity are taken into account. Based on our experience of measuring gas velocities in large stacks and ducts in industrial applications, we prefer to use three-dimensional probes because they yield improved accuracy over conventional Pitot tubes.

In the following sections, the use of three-dimensional probes is discussed, along with some common problems that have the potential to affect the accuracy of the measurements.

3.1 Experimental Flow Characterisation Methods

The comprehensive experimental validation of sampling locations is an essential component of the acceptance process for all sampling plane positions. This assessment is important even in situations where the proposed sampling plane location and geometry comply with acceptance criteria such as defined in USEPA Method 1 “Simplified method” or other similar international methods. In addition to providing confidence in the acceptability of a sampling plane, and therefore confidence in the robustness of subsequent stack sampling measurements, experimental validation also identifies unusual flow artefacts. Flow artefacts include wall effects that would be otherwise difficult to identify and when accounted for, can produce up to a 3 % improvement in the accuracy of the volumetric flow rate measurement. (refer to the example with the “silencers” in earlier section as showing flow artefacts.)

While a range of scientific and industrial instruments exist for characterising flow direction and velocity, in industrial situations sampling locations are normally validated utilising one of two procedures. The simpler procedure involves a two-dimensional assay of the flow character using an S-type Pitot tube while the more comprehensive, and recommended, procedure involves a three-dimensional assay over the sampling plane. In both methods the flow measurement probe is inserted into the duct and the flow direction and velocity are then determined across the sampling plane at each sampling point. Thus, in addition to assessing the overall acceptability of the sampling location, the acceptability of all individual manual sampling positions is assessed. This detailed assessment is especially useful for identifying unusual flow artefacts such as a poor sampling flange design, ductwork leakage of exhaust or air entrainment, excessive hydraulic friction at the wall surface and identifying flow influences from instrumentation or devices installed upstream of the sampling location.

All two-dimensional assessment standards provide only limited data for assessing the acceptability of a sampling plane as only one component of the flow vector, the yaw angle, is measured at each sampling position. While this method has an advantage of requiring minimal specialist equipment, it falls short in providing a robust assessment of the acceptability of the sampling plane. The experimental procedure involves rotating a correctly inserted S-type Pitot tube into and out of the flow to obtain two null points that are at angles of +90° and -90° to the flow. Through the use of a protractor as well as the identification of the maximum velocity position, the yaw angle can is deduced. As the pitch angle component

of the velocity vector is not determined this method is not recommended except for qualitative assessment sampling positions.

The more comprehensive, and recommended, assessment involves a three-dimensional assay of the flow character over the sampling plane. This assessment uses the more sophisticated three-dimensional Pitot probe to simultaneously measure both pitch and yaw of the gas flow at all proposed sampling positions. A number of commercially available probes are suitable to undertake these measurements and these are generally based on either the "prism" or "spherical" design. Figure 3.2 shows an image of each of these two designs.

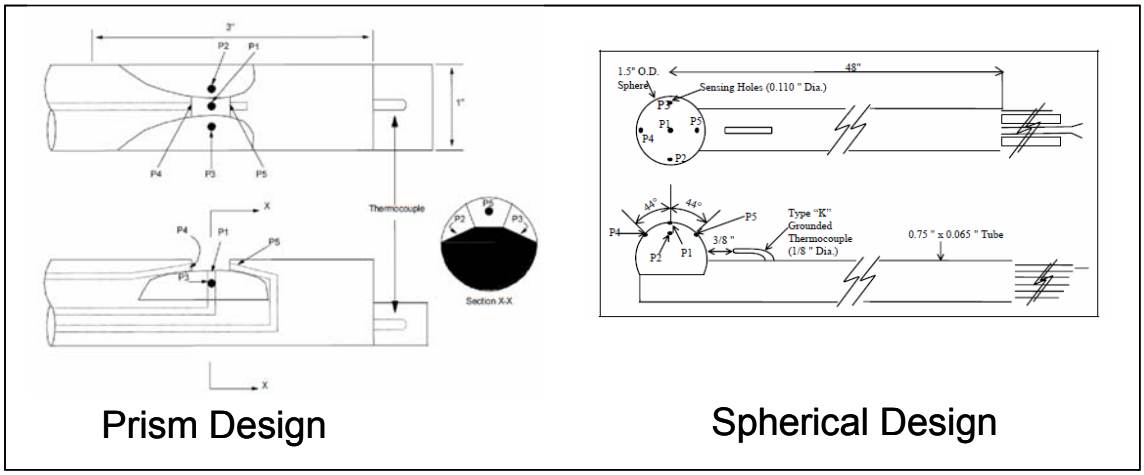


Figure 3.2. Schematic diagram of the three-dimensional Pitot probe use for measuring gas flow velocity according to USEPA Method 2F. ***(Reference USEPA Method 2F)***

Both designs have the ability to determine the resultant velocity vector at a sampling point, although the spherical probe design has a number of advantages for flow characterisation. The spherical probe measures the pitch, yaw and velocity simultaneously by monitoring the pressure differential between P1 and P2, P3, P4 and P5 and comparing these four pressure differentials to a mapping of calibrated velocities, pitch angle and yaw angle. The pressure comparison is usually computer automated so that these flow parameters are determined in "real time". Minimum manual rotation of the probe is required to complete a measurement allowing the probe to rapidly respond to changes in these parameters. This rapid response has enabled this design to find application in aerospace and aerodynamic industries. In addition, the simultaneous measurement of these three parameters without operator interaction is very useful for assessing longer term or diurnal variations of flows in exhausts ducts as well as for quantifying turbulent flow.

A variation of the spherical probe is the "Cobra" system (Shepherd, 1981). Due to the small sizes of the sensor (1.5, 2.6 and 5 mm with pressure tap separation of 0.25 mm) compared to other three-dimensional probes, the "Cobra" probe is well suited for characterising the flow in small ducts and pipes with diameters smaller than 300mm depending upon the measurements sensor used. Its high sensitivity and small measuring head makes it useful for detailed experimental mapping of the hydraulic friction at the surfaces of ducts. Additionally, the Cobra probe has a very high sampling frequency 2000 Hz making it suitable for flow measurements even in turbulent systems.

Prism based probes measure the yaw angle by manually rotating the probe into the gas flow until the pressures of P2 and P3 (Figure 3.2) are equal. Once pressures P2 and P3 are equal

P1 will be aligned axially with the yaw flow component. The pitch is then calculated from the differential pressure between P4 and P5 while the velocity is calculated from the pitch angle, the yaw angle and the pressure differential between P1 and P2 by way of calibration charts. This yaw angle rotation is not an automated process and the balancing of the P2 and P3 pressures is visually assessed by the operator who observes the deflection of an analogue differential pressure gauge located on the instrument. Following the balancing of the P2 and P3 the yaw angle is manually measured using either a protractor assembly or an electronic inclinometer that is attached and aligned to the probe. Thus manual operational requirements of this probe provide limited temporal resolution of the yaw angle component of the gas flow.

For maximum temporal resolution both prism and spherical probe designs require pressure measuring transducers be located as close as possible to the measuring probe. Long connection tubes and large diameter connection tubes between the pressure transducers and the probe not only increases the time required to null the device but also increase the uncertainty in the measurement due to reduced response to flow fluctuations. The dampening of short term transients caused by long lengths of connecting tubing is an important consideration in probe operation as rapid response is essential for the identification of non-laminar flow.

An example of a three-dimensional flow assessment of a sampling plane at a coal-fired power station is shown in Figure 3.3. Here, the complex design of the exhaust ductwork at this power station provided very few positions to install acceptable sampling planes before the exhaust passed into and up the exhaust stack. The best available sampling location was identified downstream of an induced draft fan; the acceptability of this sampling location was experimentally measured using a three-dimensional prism based probe.

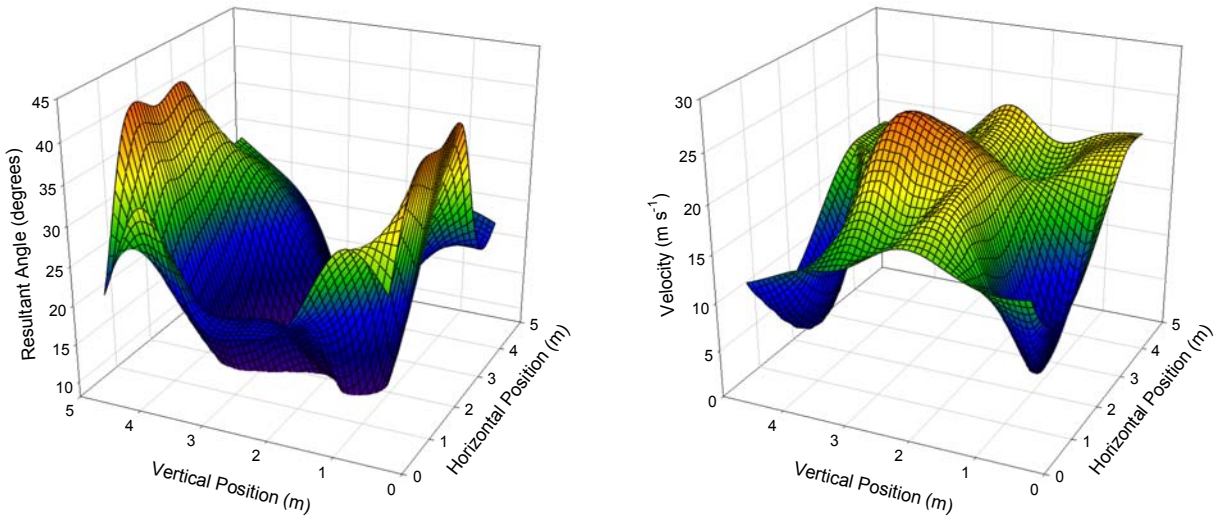


Figure 3.3. Flow characteristics in a 5 m square section duct showing the resultant angle as a function of position (left) and velocity as a function of position (right). ***(Copyright CSIRO 2010)***

The results showed that the sampling position failed to comply with the USEPA acceptability criteria of an average resultant angle of < 20° and a standard deviation of < 10° and as such the sampling position was assessed as unsuitable. It should be noted that an acceptable

sampling position at this facility was obtained after further three-dimensional measurements at a different position and only after the installation of flow straightening devices upstream of the sampling position.

3.2 Probe Misalignment

All international manual sampling methods for aerosols stringently require isokinetic and isoaxial sampling. These methods assume the assembly that supports the probe or sampling flange is accurately aligned in a direction orthogonal to the duct flow direction to facilitate isoaxial sampling. Any misalignment of sampling flange results in an increased experimental uncertainty and reduced data accuracy. Additionally, the magnitude of the misalignment may vary between separate sampling flanges resulting in a further increase in experimental uncertainty. Figure 3.4 below illustrates an example where misalignment of the manual sampling flange has skewed the sampling probe, which in this case is a 5-hole three-dimensional Pitot tube. The red arrows in these images indicate the correct alignment position.

Figure 3.4. An example of misalignment of a sampling port access flange. ***(Copyright CSIRO 2010)***

It can be seen that a number of flanges were incorrectly aligned by up to 5° resulting in an additional ~3 % uncertainty in each measurement during isokinetic sampling (calculated on the basis of reduction in sampling nozzle area presented to the aerosol flow). While it is possible to account for probe misalignment during manual sampling compensation with additional sampling time this it is not normally undertaken commercially. Additionally, this correction can only be completed accurately when both the resultant velocity vector and the resultant misalignment angle are quantified. It is therefore recommended that manual sampling flanges be accurately aligned to be perpendicular to the flow during initial installation.

3.3 Wall Effects

When measuring the flow of a gas stream using a sample pattern defined by one of the sampling methods, the average velocity of the flow for each section is assumed to be equal to that measured at the sampling point. Although this is generally true for the bulk flow, it may not apply for those areas closest to the stack wall because hydraulic friction tends to reduce

the velocity near the wall. Consequently, the results from velocity traverse may overestimate the actual flow if wall effects are not taken into account.

Recognising this problem, the USEPA introduced Method 2H - Determination of Stack Gas Velocity Taking into Account Velocity Decay Near the Stack Wall, which prescribes a standard approach to accommodating surface artefacts in circular exhaust ducts with diameters approaching 1 m diameter. The wall roughness is determined either by applying a factor (0.9900 for brick and mortar and 0.9950 for other surfaces) or by experimentally measuring the gas velocity boundary condition. The latter is the preferred approach. While USEPA Method 2H is specified only for circular ducts, this experimental approach may be applied to rectangular geometries. However, the application of USEPA Method 2H to rectangular geometry ducts requires a significant understanding of fluid dynamics as well as detailed individual measurements of the hydraulic friction at all geometrically different sampling positions within the duct. Recently, a Conditional Test Method 41 (CTM-041) has been developed to deal with rectangular ducts (Norfleet, 2005).

In small ducts, it is essential to utilise small velocity probes so that the influence of the probe head itself is minimised. The “Cobra” probe is ideal in this situation and is recommended due to the miniature size (that allows it to approach to a few millimetres of a surface with minimal influence), fast response time and ability to measure the resultant velocity flow vector. Figure 3.5 shows an example of a measured velocity profile in an exhaust duct that exhibits surface boundary effects. In this figure wall artefacts can be clearly observed as a slowing of the gas flow near the outer edge of the duct.

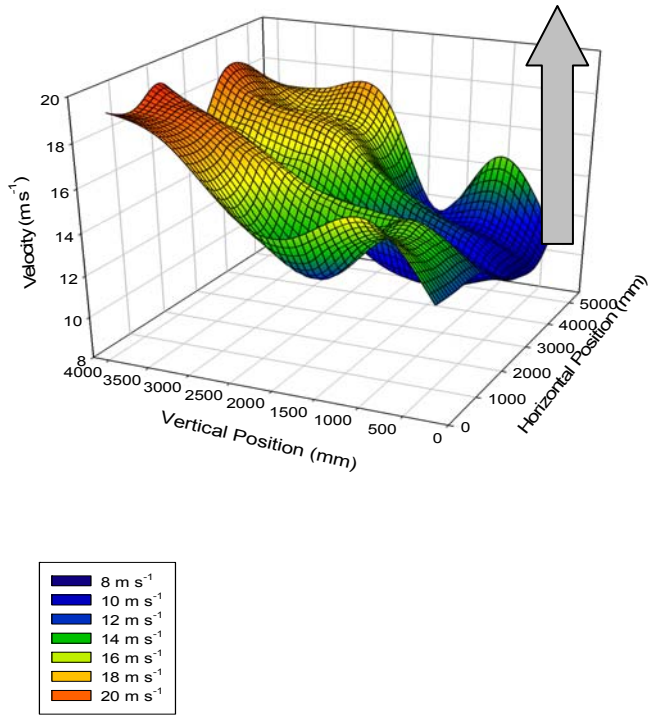


Figure 3.5. Three-dimensional plot of the air velocity in the ventilation duct as a function of the horizontal and vertical position of the Pitot probe. The arrow shows the direction of flow. ***(Copyright CSIRO 2010)***

3.4 Continuous Flow Measurement

While Pitot tube methods (and their variants) can yield accurate flow rates, they are more suited to periodic measurements, especially if many points must be sampled within the stack. In our experience, an experienced team of technicians may take several hours to conduct a single traverse on a large duct. Hence, these methods are not usually suited to frequent monitoring. Rather they are used for periodic stack testing to satisfy statutory requirements or for verifying the results of online systems.

Continuous monitoring such as may be required for statutory emissions reporting generally rely on automated online instrumentation. Various instruments for continuous flow measurement are commercially available and as with the Pitot tube methods, there are international standard methods applying to automated flow measuring systems: e.g.

ISO 14164:1999 Stationary source emissions – Determination of the volume flow rate of gas streams in ducts – Automated method

The automated method described in ISO14164:1999 refers to the use of commercial systems which are based on principles of measuring differential pressure, thermal properties or the speed of sound within the gas stream. These systems have sensors that are normally fixed to the stack wall and integrate over the full width of the stack. They do not provide information on the velocity profile within the gas flow, thus multiple sensors may be required to provide a satisfactory average velocity reading if the flow is subject to significant asymmetry.

The location of the sensors is subject to similar requirements of being sufficiently far away from obstructions to avoid erroneous readings. It is also noted in this standard that provision should be made to allow for comparative measurements with another method, such as Pitot tubes, to validate the accuracy of the automated method.

4 SAMPLING EFFICIENCY

In an ideal sampling system the target species, whether it is a gas phase, a suspended liquid or a suspended solid is captured from the moving carrier gas stream, transported through the sampling probe and collected in a stable form that is ready for analytical quantification and with no loss or change in the sample species The reality is significantly different with sample losses occurring at all stages of the sampling and analytical process. The detailed study of fluid dynamics of sampling and sampling efficiency is outside the scope of this brief and can be found in numerous monographs such as Hinds, 1999 or Baron and Willeke, 2005. The following provides a brief summary of the important aspects of sample losses discussed in the above monographs; an aspect will be more comprehensively examined in Task 2.

At the proposed Mongstad facility many of the components of interest will be present as aerosols. It is likely that sampling will need to be undertaken isokinetically. Hence the sample probe will be fitted with a internally polished tapered nozzle that ensures a smooth hydrodynamic flow over the probe and representative capture of the in-flight particles. Current USEPA methods relating to isokinetic sampling stipulate that nozzles must be of a non-reactive material with a sharp tapered leading edge and taper angle < 30°. In our experience, we have found that < 30° is acceptable for particle sampling, although we prefer to use an initial leading edge taper of 15°, which provides a cleaner “cut” with less

disturbance of sampled air at the nozzle leading edge. Nozzle diameters usually range from about 4 mm to about 15 mm, depending on the gas velocity and sample concentration in the gas stream. However, we have found that the smallest diameter nozzles are prone to blockages especially in moist, heavily laden gas streams and larger diameters are generally favoured where possible.

4.1 Inlet Efficiency

The efficiency of an inlet of a sampling system to capture a target species is dependent upon the flow conditions at the sampling inlet, the design of the sampling inlet as well as the inertial properties of the particles. International stack sampling methods, such as those listed below, have been prepared by government agencies for generic application to a wide range of emission sources ranging from coal fired electricity generating power stations to brick kilns and industrial incinerators.

USEPA Method 5 - Determination of Particulate Matter Emissions from Stationary Sources

USEPA Method 17 - Determination of Particulate Emissions from Stationary Sources

VDI 2099 Part 1 - *Particulate matter measurement. Dust measurement in flowing gases. Gravimetric determination of dust load*

As such these methods assume that the particle distribution will be wide ranging and extending from nanometre sized particles up to a hundred or more micrometres. Since the Stokes diameter for these larger particles will also be large, these methods demand that the particle sample be extracted isokinetically to reduce sampling mass biases. Sampling mass biases arise because the inertia of these larger particles is sufficiently large that they do no easily follow the gas flow lines at the nozzle entry to the sampling system. This can result in one of three conditions depending upon the sampling velocity at the entrance to the sampling nozzle.

For the isokinetic condition, the gas velocity at the sampling nozzle is identical to gas velocity local to the probe. In this case both the coarse and fine particles follow the gas flow lines that extend straight into the nozzle. In this case the efficiency of aspirating all diameter particles into the sampling tube approaches unity and hence a representative sample is captured at the sampling nozzle. This is the condition prescribed by international stack sampling methods and required when the target aerosol exists over a wide range of Stokes diameters.

For the super-isokinetic condition, the sample velocity at the sampling nozzle is greater than the gas velocity local to the probe. In this case a greater volume of gas is aspirated into the nozzle than would normally be the case for the isokinetic condition and hence gas flow lines bulge outwards to accommodate this increased gas volume. In this case the finer particles will follow the gas flow lines up to the limiting streamline and are entrained into the nozzle. Aerosols of larger Stokes numbers, that is aerosols with large inertia, will not follow the gas flow lines and will penetrate through the limiting streamline and bypass the sampling nozzle. The result is that these larger Stokes diameter aerosols will be underrepresented in the final mass balance. The collection efficiency is therefore a combination of the ratio of velocity mismatch as well as a function of the ability for the particle to follow the gas flow lines, or Stokes diameter.

For the sub-isokinetic condition, the sample velocity at the sampling nozzle is less than the gas velocity local to the probe. Hence, a reduced volume of gas is sampled into the nozzle than would be the case for the isokinetic condition. For this case the gas flow lines converge inwards towards the centre axis of the sampling nozzle as the reduced gas volume is aspirated into the nozzle. The result is that particles with larger Stokes diameters and thus greater inertia penetrate across the gas flow lines and into the sampling nozzle. The result is that the coarse particles will be overrepresented in the final mass balance. The collection efficiency of the subisokinetic condition is similar to that of the superisokinetic condition and is a combination of the degree of velocity mismatch as well as a function of the ability for the particle to follow the gas flow lines, or Stokes diameter.

It is essential that samples containing large Stokes diameter aerosols be aspirated into the sampling nozzle isokinetically. However, at small Stokes diameters the requirement to isokinetically aspirate an aerosol to achieve a representative sample diminishes as the aerosol sample behaves more like a gas than large inertial particles. Correspondingly, the quality of aspirated sample as well as the errors associated with the sample become independent of the isokinetic sampling criterion as the Stokes diameter of the aerosol reduces. However, it should be highlighted that this does not necessarily mean that the resolution of sampling over the sampling plane is also relaxed as sample inhomogeneity may still exist over the sampling plane.

The need or otherwise of isokinetic conditions during stack sampling at the proposed Mongstad facility is dependent upon the aerosol size distribution leaving the absorber. If it can be demonstrated that the aerosol is comprised of entirely small Stokes diameter particles then the importance of the isokinetic criterion diminishes and stack sampling operations somewhat simplified. However, this condition would require detailed experimental assessment and validation. The aerodynamic particle distribution over the selected sampling plane would need to be determined under all operating conditions before deviations from the isokinetic sampling criterion could be accommodated into revised stack sampling methods.

It should also be noted that the requirement for isoaxial alignment of the sampling probe into the gas flow also diminishes as the magnitude of the Stokes diameter of the aerosol diminishes. As is the case for the isokinetic criterion, this aspect of stack sampling is also explained in by the differences in the hydrodynamic behaviour of particles of different Stokes diameters. For the case where the sampling nozzle is non-isoaxially aligned with the gas flow direction, aerosols of smaller Stokes diameters tend to be aspirated into the sampling nozzle independent of the misalignment since these aerosols follow the gas flow lines into the sampling nozzle. The coarser particles, however, as a result of their increased inertia tend to follow a straighter trajectory and may not be aspirated into the sampling nozzle resulting in reduced representation of these aerosols in the aspirated sample.

4.2 Transmission Efficiency

In addition to the sample losses that occur during sample aspiration, losses may occur during transmission of the sample into and through the sampling train. These losses are well documented in numerous monographs such as Hinds, 1999 or Baron and Willeke, 2005. Transmission losses can arise from wide range of aerosol removal processes including, diffusion losses, impaction losses, losses due to aerosol charging, surface losses due to both

chemical and physical absorption of target species to the sampling system. The minimisation of transmission losses within a sampling apparatus is important for the representative collection of a sample, however, these losses can be minimised by optimising the design of the sampling system.

5 SYSTEM COMPONENTS AND MATERIALS

Systems for stack sampling (excluding the sample train for collecting that material) consist of a probe that can be inserted into the gas flow through the sample ports. Normally, the sample probe comprises a metal tube up to about 3 m long and 25 to 50 mm in diameter, with associated smaller diameter tubing to connect the probe head to pumps and/or analysers and sample trains. In some cases, in-stack sample collection devices will be attached to the head of the probe (usually a filter or impactor for particulate/aerosol collection). Depending on the species being sampled, the tubing may need to be heated. It is essential therefore to ensure that the sampling ports are sized appropriately to accommodate all of this equipment.

Because of the nature of manual sampling probes and associated gear (including Pitot tubes), they are not amenable to being left in place when not in use. In the aggressive environment of the stack, tubing and Pitot tubes quickly block with particulates and in the off-gas from an amine plant, heat stable salts may also deposit on probes left in situ. They may also be subject to excessive corrosion. Hence this type of system is deployed only during the sampling campaign. Online systems, on the other hand, will be fixed in the gas stream and it will be necessary to ensure that these systems are made from materials that are resistant to corrosion and are designed to avoid blockages (with purging systems, for example).

Probes and ancillary equipment are generally made from stainless steel, quartz or other non-corrosive materials. Aluminium is also frequently used to manufacture nozzles and filter holders used for isokinetic sampling, however, it is important that the environment be non-corrosive to the aluminium surfaces. Although these traditional sampling probes are likely to be suitable for collecting samples of some of the exhaust components from amine plant stacks, there may be others where this is not the case. Some of the compounds anticipated in amine plant exhaust are unstable (e.g. nitrosamines) or may react on metal surfaces. Galvanised steel, copper and copper bearing alloys are incompatible with many amines including alkyl alkanolamines, ethylemeamines and ethanolamines. As such, brass, bronze and copper based collars and sleeves that have traditionally been used to eliminate galling of stainless steel fittings and probes during stack sampling tasks will need to be replaced with an alternative more compatible material. GRAFOIL^{TM}, an amine compatible material recommended by DOW Chemicals, that can be formed into gaskets and friction reducing bushes.

The probe itself will almost certainly be fabricated from a 300 grade stainless steel tube to have the required mechanical strength to comply with normal minimum deflection requirements of sampling probes (typically a maximum of 2° of probe sag). However, the internal tubing may be made from other materials with non-reactive characteristics if necessary. Teflon and quartz, for instance, are recommended for reactive species. However, Teflon it is not suitable for particulate material since Teflon tends to collect particles, preventing proper sampling.

Sharp transitions from the internal nozzle diameter to the sampling probe diameter should be avoided since they can produce areas of stagnation where material can accumulate during sampling. This is particularly important issue for very small aerosols as these aerosols relatively easily removed by diffusional processes. This can be a problem with small diameter nozzles where it may be difficult to avoid ridges around the point of nozzle transition to the sampling tube connection. Larger nozzles also need careful attention in this regard when a larger nozzle diameter is reduced to the diameter of the probe liner. It is recommended that all transitions within the sample prove be smooth, polished and have taper angles of less than 30 degrees, however, we favour smoother transitions of 15 degrees where possible. In addition, direction changes or bends in the sampling system must be carefully constructed to maintain proper flow but also to avoid aerosol deposition and impaction within the collection device.

The time required to collect sufficient sample from the gas stream is dependent on the concentration of the material in the gas and the sample stream flow rate. Most commercially available pumps for isokinetic sampling are capable of flow rates between about 10 to 50 L min^{-1} and depending on the concentration, samples may be collected over periods of minutes to several hours. In amine capture plants, the concentration of the components is not yet known with any confidence, however, it is likely that some compounds will be at very low concentrations and will require a large volume of stack gas to be withdrawn to collect sufficient sample for analysis. In such situations, significantly higher pumping rates than available for commercially available sampling apparatus may be required to collect sufficient sample material within a reasonable time. These flow rates can pose challenges for the sampling apparatus as large volumes of moisture will need to be accommodated in the design. Alternatively novel sampling designs may be required to overcome this moisture issue without diluting the quality of the sample.

The brief discussion above highlights the complexity of stack sampling and the numerous issues that need to be addressed when designing the overall sampling system. It is apparent that no single sampling system will be suitable for all of the compounds likely to be present in the stack gas; rather a number of separate parallel sample trains that are optimised for particular species will be required. The topic of sampling equipment and the general design of the sampling system are extremely important aspects and accordingly, they will be discussed further in Task 2 which deals with manual sampling from stacks.

6 RECOMMENDATIONS FOR DESIGN

Methodology for selecting sampling points is well established and has been used for regulatory emissions monitoring for many years. Broadly speaking, all of the standard methods for determining the location of sampling points are very similar. Any differences are generally in minor detail. In all methods, the preferred location of the sampling plane is in sections of stack with long runs of straight flow (termed ideal flow). However, the methods can also accommodate sampling in shorter runs provided that off-axis flow is within certain limits (generally less than 20°).

In principle, the methodology provided in the available international standard methods should be suitable for application at amine scrubbing plants, regardless of the type of material under investigation. However, the materials from which the sampling components are fabricated will need to be selected bearing in mind possible incompatibilities with the compounds in the stack gas. Corrosion resistant materials such as stainless steel will probably be used in the

sampling system although other materials may be used as further information on the chemistry of the potential emission compounds comes to light.

If the compounds of interest are gaseous, chemically stable, not soluble in any condensed phase or adsorbed on solid phases, and the flue gas stream is well mixed, sampling should be straightforward, requiring as little as one sample point that can be located anywhere in the gas flow. In these cases, ideal flow is not a requirement for representative sampling. However, many of the components in the exhaust from an amine PCC plant are unlikely to meet these criteria and more elaborate sampling methods will be required.

Previous experience suggests that solid particulates and entrained liquid aerosols will not be evenly distributed within the stack gas flow and there may be significant spatial variation in concentration. Liquid aerosols, in particular, are likely to be a significant component of amine capture plant stack gases. It is likely that numerous target species will also be in a soluble gas/liquid aerosol phase equilibrium requiring the simultaneous sampling of both gaseous and aerosol species to maintain the stability of the phase equilibrium until sample capture. Consequently, representative sampling from these stacks will require a comprehensive series of sampling points distributed through the sample plane. It is recommended that as far as possible, sampling for these components should be in ideal flow conditions detailed in the relevant standard methods.

With small ducts the sampling probes may significantly upset the flow. This could be a problem with pilot scale plants with small diameter stacks but probably will not be an issue for full scale plants.

If emission fluxes are to be determined, the volumetric flow rate will need to be measured concurrently with sample collection. Velocity may be measured by conducting Pitot tube traverses at locations in the flow according to the standard sampling methods, although the use of three-dimensional Pitot probes is preferred since they yield increased accuracy compared to S-type or standard Pitot tubes.

Sampling and velocity measurements are likely to be made in sections of the stack that are at elevated locations. To ensure that personnel are able to work safely and operate the sampling equipment in accordance with the approved methods, provision must be made for appropriate access ports and work platforms. It is suggested that where possible, these factors should be considered at an early stage of the plant design, rather than trying to adapt unsuitable situations to suit.

CFD modelling should be used to theoretically identify and validate designs and examine the influence of these upon the particle or aerosols flow. In addition the stated modelling would provide information about the structure of the flow along the duct where the steady state conditions can be determined. This will reduce the amount of practical work needed on the plant.

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