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Principles of gas sampling: TOS with critical challenges

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Introduction

Sampling and analysis of materials in the gas phase is not covered in general sampling standards and guides, due to the complex nature of the subject matter. Most gas-phase materials exist in the region from ambient temperatures (~300 K) to combustion temperatures typically around ~1200 K. Common to both temperature range margins, though predominantly for hot gases, is the fact that continuous reactions often take place in material that is moving at high speeds, presenting less than trivial challenges to conventional Theory of Sampling (TOS). The purposes of representative gas sampling are many, but three standard cases are presented, covering the most dominating scenarios met with in science, technology and industry.

Volume sampling

Sampling of a nominal gas volume, contained in a bespoke container, suitable with respect to processing and analysis at a different location and/or or at a different time.

In situ sampling

This case has the aim of monitoring a specific gas component under a set of fixed conditions (usually specified by a set of physical parameters). This sampling modus is relevant for gas components or gas-phase phenomena, which are changing or unstable over time, i.e. gas processes that will change, oxidise, adsorb, react or shift balance due to irreversible chemical reactions.

On-line gas analysis

This involves, for instance, securing a representative gas sample in a gas cell, cuvette, chamber or other analytical device, ensuring a representative basis for process monitoring and control.

It will be clear that gas sampling to a very large degree must be addressed on a case-by-case basis. Very often gas samplers must be designed and implemented as built-in sampling valves/fixed piping, often with considerable sample conditioning hardware. Still the TOS must rule supreme.

Classical gas sampling cases

Most gas sampling can be classified as either a “hot” or a “cold” case, which distinction refers to the need to keep the gas temperature above a certain threshold to *avoid condensation*, primarily of water vapour, but other species may also add significantly to the complex dew point issue. Another argument to keep temperature high is the fact that various components only exist above an elevated temperature—a good example being SO_3, which will dissociate an oxygen atom, thus becoming SO_2, below a few hundred degrees Celsius. Thus, the analytical temperature may quickly become a critical issue depending on the gas mix composition.

The most often met with cases of gas sampling are for the following analysis purposes:

- Outdoor air quality (cold)
- Indoor air quality (cold)
- Stack emission monitoring (hot)
- Process gas characterisation or monitoring (hot and cold)
- Combustion gas characterisation or monitoring (hot, very hot)

In the descriptions below annotations in [square brackets] refer to the complement of the TOS sampling errors [SE] vs the total analytical error [TAE]. Focus will naturally be on the so-called “Incorrect Sampling Errors” [ISE], which cause a significant, sometimes fatal *sampling bias.*

There are four [ISE], viz. the Incorrect Delineation Error [IDE], the Incorrect Extraction Error [IEE], the Incorrect Preparation Error [IPE] and the Incorrect Weighing Error [IWE]. The TOS describes how [ISE] that have not been eliminated will give rise to a *sampling bias* which is inconstant, and which, therefore, cannot be corrected for (however desirable). Occasionally there *may* also be sampling challenges caused by the two so-called Correct Sampling Errors [CSE]. These are a function of material heterogeneity and thus [Fundamental Sampling Error (FSE) and Grouping and Segregation Error (GSE)] *may* also influence the total sampling error [TSE], although it is generally assumed (with good reason) that gas mixtures are universally *well mixed*. A special case very far removed from the well-mixed gas paradigm is analysis near a burner in a furnace, in which case pockets of high concentration can be present in semi-stable scenarios presenting a [GSE] challenge when placing a sampling point near such burners.

Delineation of specific gas *analytical errors* is also contained in the text below.

Physical manifestation of gas mixtures

A gas mixture, a gas matrix, consists of several types of chemical species, all under the influence of a set of physical parameters that need special attention to overcome unwanted effects and non-representative sampling [ISE, CSE]. A gas mixture is always comprised by one or more components of the classes below:

- Major gas components (usually present at % levels)
- Trace element gas components (necessarily present at ppm, or ppb, levels)
- Mechanical dust, of three types:
  - Organic dust (bio effluents, pollen, *unburned* organic matter)
  - Inorganic dust (ash, salt, sand)
  - Metal particles
- Aerosols (sub-µm droplets); a case made highly relevant in the era of the coronavirus pandemic.

Typical major gas components would be “classical species”, e.g. Nitrogen, Oxygen, Carbon dioxide, Argon, water vapour. The class of major components can be subdivided into *active* and *passive* components. Whereas Nitrogen and Argon, in almost all cases, will act passively, species like oxygen and water vapour may affect, and sometimes react readily with, other major or trace gas components. If this takes place after sampling, significant [IPE] effects will be unavoidable.

Trace components gases are typically present from a maximum of 10,000 ppm (1 %) to as low as ppb (or even ppt) levels. While the list of major components is fairly quickly exhausted, the list of variously interesting trace components is in the hundreds or higher. Trace components can further be subdivided into the subclasses of inorganic and organic species.

Mechanical dust is present in almost all gas mixtures from ng Nm^{-3} to higher levels in the g Nm^{-3} range and may give rise to severe challenges. Besides the purely mechanical effects of blocking, clogging, wear and tear of the sampling tool [IDE, IEE], sub-µm particles may function as "seeds" for forming aerosols/droplets, e.g. causing catalytic effects, thereby *changing* the composition of the gas mixture, both physically as well as chemically [IPE]. Most extractive systems try to avoid the different dust species by *filtering* the gas mixture prior to extraction, i.e. a problem-specific fractionation, which may work well, but careful consideration should be given to a range of potential unwanted effects in the filter (selection and manufacturing of the filter material) as this may cause changes to the sample as well [IEE, IPE].

Aerosols exist in more or less all gas mixtures when a matrix is in a state *below* a complex dew point temperature regimen (actually a series of dew point temperatures), which must be calculated specifically *for each case and specific hardware configuration*. This is especially true when sub-µm dust particles form a seeding basis for aerosol formation. Aerosols may consist of relatively harmless matter, e.g. water, but since many trace components are soluble in water, the aerosols may also easily take on roles much different from harmless [IDE, IEE, IPE] and will then generally be of an acidic nature, causing damages to pipelining, sampling equipment and optics if spectroscopic analytical devices are used.

In general, therefore, a gas matrix is characterised by (at least) the following important features:

- Temperature
- Dew point(s)
- Matrix effects

Passive vs active components

Passive compounds

A gas component that can be analysed *in situ* or extracted, *without* reacting with other gas components or parts of the extraction and analysis system (filter, pipe, cell, cover glass etc.). In practice, this means that the gas sampling and analysis system has been designed in a TOS-informed way, such that the quality criterion is that *no* [ISE, CSE] can occur.

Examples: Nitrogen; Argon (other inert gases)

Active components

A gas component that in general will have a non-neglectable *tendency* to react with *other* gas components to form new compounds [IEE, IPE]. The degree to which the sampling is compromised may vary, and will have to be evaluated by problem-specific consideration or by a pilot experiment (replication experiments or perhaps a variographic characterisation).[1–3]

Examples: Oxygen, various nitrous gases N_xO_y, water vapour, SO_2, SO_3

Aggressive components

A gas component that *always* displays a significant propensity to react with the sampling and analysis systems. This will always cause an *indirect* quantitative effect by reducing the activity of the analyte gas in the sample volume, thereby causing [IEE, IPE].

Examples: HCl, HF, NH_3

Three most common sampling strategies

In situ sampling

In situ sampling and analysis is conducted directly in the gas matrix, for example in a pipeline, in a gas chamber or in open air. *In situ* sensor sampling [for example in the Process Analytical Technologies (PAT) regimen] is based on transmission of a continuous beam of energy across the analytical volume with an illumination source power appropriate to achieve a representative spectroscopic response from the gas component(s) analysed for. Typical optical sources operate from the ultraviolet (UV) to the infrared (IR), i.e. wavelengths from 200 nm to several µm. The analysed gas volume is the volume of the *optical cone* formed by the geometry of the source and detection system. Over time, the risk of sampling volume changes *may* develop, e.g. by condensation, scaling or precipitation, which will cause [IDE, IEE]. Special attention should also be on the fact that it cannot be guaranteed that concentrations are constant at all positions in the "observation cone" giving rise to potential [GSE].

This heterogeneity issue may sometimes make quantitative calculations difficult, arising for two reasons:

i) Lambert–Beer's law states that the optical pathlength, as well as the specific molecular concentration, serve an equally important role with respect to the *total absorption* of the radiation emitted and detected.

ii) Energy absorption is almost always non-linearly related to the concentration, which in practice means that a localised high concentration "pocket" will not necessarily get weighted properly in the cumulative absorption signal recorded at the detector.

The inferred total concentration may then show up as being *lower* than in reality. Such effects are usually considered sufficiently countered by acquiring many (very many) signals over time, strongly relying on a beneficial mixing and averaging effect. N.B. this may, or *may not*, be justified in specific cases, however.

An upside regarding *in situ* sampling is the absence of typical mechanical effects, and abundant "real-time" opportunities. Once this type of sampling has been proved to function according to the desired specifications, the road is open to reliable, representative on-line (in-line) process monitoring and control (PAT). Here the use of variographic analysis will be especially relevant and welcome, see, for example, References 1–3.

Extractive sampling

The objective of extractive sampling is to facilitate removal of a representative sample that is reliably contained and ready for at-line analysis, or which can be transported for analysis in the laboratory or similar. Extractive analysis is widely used, and highly efficient empirical solutions for many sampling systems have been developed for many standard applications, but a series of complex effects must nevertheless still be carefully considered and counteracted where appropriate. Such effects are due to:

- gas physics
- oxidation
- catalysation

- adsorption
- substitution

The most important issue to address regarding extractive gas sampling and analysis systems is related to the governing gas physics, i.e. to the ideal gas equation:

$$P \times V = nRT$$

This famous equation links pressure (*P*), volume (*V*) and temperature (*T*) to the number of molecules (*n*) in a simple linear relationship, i.e. any one parameter cannot be changed without a *resulting* change in one of more of the others. To secure a representative sample in an extractive container for transport or analysis, the "*n*" signifies the number of molecules extracted. This should "obviously" be the same number in a volume of the original gas matrix equal to that of the sample container. This may seem trivial, but if, for example, the pressure drops in the gas matrix, without *concomitant* volume and temperature changes, the number of molecules will actually change as some molecules will drop out of the gas phase and become liquid (condensation) and in this way change the concentration of the analytical aliquot [IDE, IEE, IPE].

Other ways of "losing" molecules are oxidation of a species, NO becoming NO_2 (or reversibly). Such oxidation or oxygen-balance reactions may be catalysed along the walls of the sampling system, for which reason due attention should also be given to the choice of container material. Stainless steel tubes and PTFE hoses are predominantly used in most gas systems, both have distinct advantages and disadvantages, which must be known and considered in the design of a gas sampling and analysis systems.

Various gas components are keen to adsorb and thus form a monolayer on many surfaces [IDE, IEE, IPE]. A building up of immobilised molecules in the sampling system may then contribute to a gradual lowering of the analytical response time, adding a [TAE], and may in the long term even cause harm to the sampling system. Components like HCl, and especially ammonia, are known readily to form such monolayers, *unless* the sampling system is heated and a prescribed level of water vapour keeps the adsorbing species in the gas phase; note that these are active counter measures to ISE and TAE effects. For water vapour, significant hydrogen bonding capability and water molecule electro-negativity effects will typically result in adsorption to the sampling/analysis system walls, blocking out adsorption from other species characterised by less attraction force. If a monolayer of, for example, HCl is present in a sampling system and water vapour is introduced (unintentionally), very high levels of HCl can be released in very short time spans (seconds) giving rise to a large concentration spike, which is *not* a truthful signal pertaining to the matrix under analysis, however.

Finally, an important potential effect is *substitution* and *reaction* with the sampling system materials. At high concentrations, various gas species may *swap* molecules with those of the sampling system. Examples are high-level HCl flowing in PTFE (poly-tetrafluoroethylene) conduits in which Fluorine and Chlorine atoms can be substituted, leading to detectable levels of HF in the analytical system. HF is a very aggressive component and may thus be especially harmful to analytical gas cells made from various glass types and has even been reported to form complex new species with silica.

From the above it is clear that designing a gas sampling and analysis system can be fraught with a range of problem-specific difficulties, ranging from the easy to the extremely difficult, very often requiring considerable in-depth knowledge of gas and mixture physics and chemistry, as well as a professional command of the TOS.

The extraction system

The principal major elements involved in a gas extraction system are shown in Figure 1.

Probe

The extraction probe consists of a series of elements, not all of which are used in all cases. The main component is the probe head, which is typically placed outside the process containment. Inside the probe head, a filter can be placed if and when appropriate. The filter can be metallic, ceramic or made from other suitable materials, which must be considered from case to case to avoid in-probe reactions. To avoid condensation, the filter can be heated, which is needed for all above-ambient temperature sampling scenarios. To reach into the process, to be able to sample the process, a probe inlet pipe is connected to the head. The material and size/shape of the probe pipe is subject to identical problem-dependent considerations as outlined above. The probe head also works as the necessary link to gas sampling transportation lines. One needs to pay special attention to so-called *cold spots*, which constitute potential locations where unwanted condensation *may* occur [IDE, IEE, IPE]. A gas sampling probe may have the option of a "blow back" facility, for example by mean of a series of solenoid valves. Dust blow back creates a series of bursts of pressurised air with the intention to clean the filter. Another option is an arrangement of valves allowing introduction of a pilot and calibration gas(es) from certified reference gas cylinders with known gas concentration.

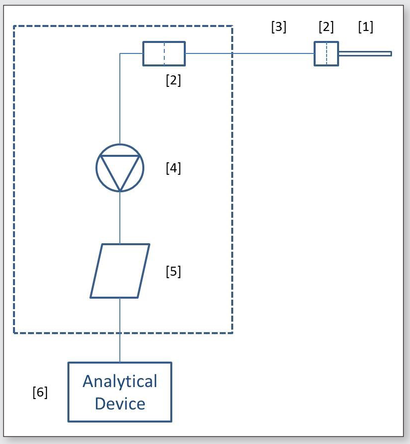

Figure 1. Schematic illustration of a generic gas sampling system. [1] Probe; [2] Filter(s); [3] Transportation lines; [4] Pump; [5] Conditioning system; [6] Analytical device (or storage container).

In many situations it is necessary to arrange for *iso-kinetic gas extraction* in order to avoid various *influx segregation* phenomena; this is especially critical for two-phase systems like gas–dust, gas mixture–dust, gas–droplets systems etc. This is a sampling aspect which is governed by "classical" TOS considerations, many of which are presented in Reference 2.

Filters (extra)

Wherever appropriate additional filters can, if necessary, be inserted before the analytical equipment. Considerations follow the same principles as outlined above.

Transportation lines

All gas sampling systems need suitable tubes and pipes connecting the probe head to the gas conditioning and the analytical equipment. The most important considerations concern allowing for appropriate temperature conditions and the choice of piping material; other issues concern the diameter and flow regimen necessary to eliminate the kind of [ISE] described above. In general, the best performance of a gas sampling system is achieved when the tube/pipeline diameters, and the gas flow, are kept *constant* throughout the entire transportation length, since a sudden diameter change with a fixed flow may generate unwanted pressure fluctuations, again with the potential of unwanted condensation.

Pump

The pump, or the device to facilitate a pumping effect in case of a venturi pipe, is a very important component in any gas sampling system. The process pressure is often different from standard atmospheric conditions; the pump must, therefore, be able overcome all over- and under-pressure issues, while being able to yield a steady flow through the analytical system before releasing the gas sample into ambient or back into the process. The pump can be inserted either before or after the analytical device, creating a small over or under pressure in the analytical device. Some analytical technologies will be sensitive to pressure changes, since it changes the number of molecules in a given volume.

Conditioning system

The gas conditioning system serves the critical role of setting up, or changing, the physical state parameters of the gas flow, or the gas volume intended for analysis, so that the gas aliquot corresponds with the stipulated requirements of the analytical method/analytical equipment. There may also be a need for conditioning gas samples before extensive transport and/or storage before analysis, as described above.

Analytical device, or gas container

When gas is collected for analysis (or processing) at another location, or time, one is obliged only to use appropriate gas sample containers, thoroughly pretested and validated with regard to the suitability of the materials, which must be chemically non-reactive.

Eventually, if, and if after, *all* the above issues regarding proper sampling, handling, transport, conditioning etc. have been properly dealt with, the representative gas sample arrives at the analytical device. The analytical technology must be *appropriate* for the gas quantification demands at hand. A large number of technologies are available.

Gas homogeneity in time and space

In order to be able to sample representatively in time and space, the fundamental issue is *how to* sample without the debilitating effects from the sampling process itself [ISE], i.e. the TOS' "sampling correctness" imperative, or effects due to the material heterogeneity [FSE, GSE]. There are simple rules that always must be observed even for relatively easy mixable multi-component gas systems.

In the full dynamic context of a combustion process, say, species like oxygen and carbon monoxide are far from evenly distributed in space and time, but will tend to form steady-state spatial *patterns* like that shown in Figure 2, in which is illustrated that fuel injection point(s) will cause a variable oxygen depletion in the core of the flame. Similarly, nitrous gases are also known to form at specific locations, but subsequently they will be reduced in concentration due to oxidation, an example of how the *sampling location* may critically determine the validity of the gas analysis. Likewise, for outdoor or indoor air quality monitoring, odour (or even poisonous bursts of gases from process doors opening and closing) may contribute to create a time-dynamic inhomogeneous gas matrix, inhomogeneous in both time and space.

"Temporal homogeneity" here means the ability to sample/detect *flash* or *spike* phenomena existing for only a short time, but which may well contribute to significant concentration variations with a high impact on hourly average values, for example. It is obvious that temporal homogeneity will be operatively dependent upon a pre-specified time-resolution threshold. When temporal heterogeneity is significant, fast, repeated sampling is needed to map out such variation with accuracy and precision in time. Variographic characterisation is particularly well advised for this type of time-dynamic systems. A variographic pilot experiment may be essential.

"Spatial homogeneity" means the ability to sample at one specific point location, or across a given cross-section, and be able to ascertain that such sampling reliably represents the point location intended or the entire cross-section of a gas stream with respect to the relevant components in the mix. Since this is not often the case, for example close to a combustion chamber, in a process reaction vessel or at a specific point location in a factory building, the sampler/analyst must often choose between *point analysis* (in both time and space) or make use of integrated *composite sampling* (which may also be in both time and space, although most often only in time). N.B. it is often *assumed*, quite often without sufficient justification, that gas systems are always "well mixed". Another way out of such heterogeneity problems may be to increase the distance to the source and rely on the inherent mixing in containers and/or pipelines etc. to provide for a less heterogeneous gas matrix. There are also

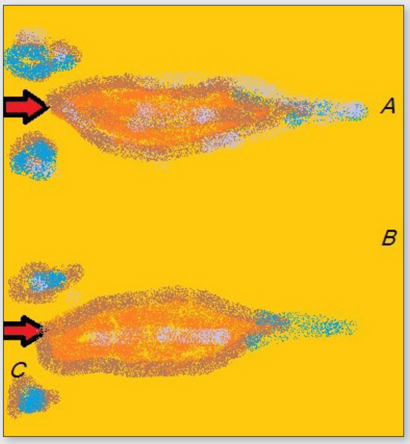

Figure 2. Illustration of alternative deployments of a gas sampling probe inlet. The analytical result from locations A, B or C will not be identical (even though it is commonly assumed that gases are always well mixed).

Gas conditioning system. Photo credit: Q-Interline often issues related to non-contained gas matrices or to specific outdoor situations—common for these contexts are the very real danger of *unwanted dilution*.

Analytical methods, criteria

Many analytical technologies exist for single gases, gas mixtures and for other components in gas matrices. Common to all of them is a set of attributes that must be taken into consideration in order to select an *appropriate* analytical method.

An analytical device is characterised, and should be validated by the following criteria:

- **Specific sensitivity**, i.e. is the analytical tool sensitive *only* to a specific component, or to a general group or type (e.g. hydrocarbons are often reported as total C_nH_m in Methane equivalents).
- **Cross-sensitivity**, normally specified in % of range, is the documented and validated analytical error introduced from *other*, specified gas components.
- **General sensitivity** is the ability to detect *small* changes in concentration.
- **Limit of detection (LOD)** is the smallest *detectable* concentration.
- **Limit of measurement (LOM)** is the smallest *measurable* concentration.
- **Response time T90** is the time for the analytical equipment to reach 90% deflection when subjected to a high-gradient concentration change.
- **Linearity** is the specification within a concentration range, of how linear the dose–response relationship will be.

Some analysers are designed for single component analysis whereas others are multi-component systems, but the same set of validation criteria apply to all.

The most common quantitative gas analytical technologies are:

Mass spectrometry (MS) is based on the principle of fragmenting and ionising the gas components, after which the sample is introduced to the analytical chamber, in which different molecular fragment masses are accelerated towards the detector. A magnetic field induces differential deflection, resulting in a mass spectrum, allowing for interpretation and quantification. MS may suffer from cross-sensitivity, but can importantly be made sensitive to ppt levels.

Gas chromatography (GC) is based on a separation principle. Introducing a small volume of gas into a very thin pipe, constructed so that different gas components will travel with different velocities, thus coming through the terminal end of the fibre effectively separated from each other. Chromatographic separation may prove useful for complex mixtures of very similar components (e.g. hydrocarbons).

Spectrometry (Vis/UV/IR/FT-IR/Laser) are all technologies making use of the different absorption of energy/light by various molecules. The technologies may be single component non-dispersive infrared-based analysers or multi-component systems based on Fourier transform infrared spectroscopy.[5]

Several other gas analytical technologies exist; the list above is merely to indicate examples, and is in no way exhaustive. The general Process Analytical Technology introduction[4] also treats gas analysis for on-line and in-line purposes, but the full arsenal of potential analysis techniques for use in science, technology and industry is much larger.

N.B. A critical success factor for many gas analytical systems is the common challenge to able to deliver exactly the same volume to the inlet of the analytical device. This challenge has been met with a variety of specific solutions all focused on avoiding potentially debilitating [IWE] effects. Solutions are typically mechanical, or consist of data analytical normalisation, e.g. row-summing of multivariate spectral data in chromatography.

Theory of Sampling: governing principles

The dominating issue in gas sampling, based on the synoptic summary above, is to eliminate (or at least reduce the effects as much as possible) the so-called Incorrect Sampling Errors [ISE], i.e. IDE (Incorrect Delineation Error), IEE (Incorrect Extraction Error) and IPE (Incorrect Preparation Error). There may, or may not be, a fourth incorrect sampling error, IWE (Incorrect Weighing Error), which in the case of gas sampling is equivalent to an error committed when not being able to secure exactly the same gas volume for each sample. It is comparatively rare, however, that this is an important issue with modern gas sampling and analytical equipment. The governing principles for "correct sampling", i.e. non-biased sampling, are described in the general TOS literature; see references below (and many additional references therein).[1–4]

Benchtop gas analytical setup: a modified IR instrument equipped with the necessary pre-analysis hardware. Photo credit: Q-Interline.

Variographic characterisation of a process gas sampling/analysis system

All gas sampling and analysis systems intended to contribute towards reliable process monitoring and control, must be evaluated and assessed by a *variographic analysis*, the essential features of which are described in References 2 and 3.

Conclusions

Gas sampling is an application field that to a very large degree must be attended to on a case-by-case basis. Very often gas samplers must be designed and implemented as fixed, *built-in* sampling valves/fixed piping, often with dedicated sample conditioning hardware as well. Thus, many of the complex gas sampling issues are very specific and would at first sight appear not to be part of the *standard* TOS curriculum. However, this overview has shown that the *sampling process* for gases, gas mixtures and two-phase systems (g,s) or (g, l) nevertheless always must be in strict compliance with the principles outlined in the TOS, lest several, quite typical sampling errors are equally unavoidable as for all other solid material heterogeneous systems, especially regarding IDE, IEE, IPE, IWE. No application area escapes the demands for representativeness, i.e. TOS will reign supreme also for gas sampling.

But, as has been shown above, there is quite an array of special issues within this field, which only have been introduced here. We would, therefore, like to guide you to the book: *Industrial Sampling Systems: Reliable Design & Maintenance for Process Analyzers* by Tony Waters[6] in which can

continued on page 47

continued from page 45

be found a plethora of design, implementation, operational and other aspects also pertaining to gas sampling, substantiated by a vast catalogue of practical examples and experiences. We strongly recommend this book as a powerful complement to the full set of principles for representative sampling (TOS). An overview of the book can be found in Reference 7.

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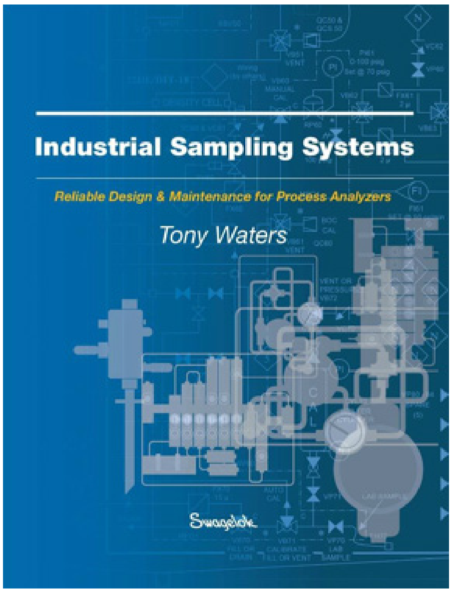

Tony Waters and Swagelok have published *Industrial Sampling Systems*, a comprehensive book covering the engineering involved with designing and operating a sampling system for a process analyser. The book has 750+ pages of full-colour technical content in a hardbound cover, organised into twelve chapters with three detailed appendices and 1000+ photos, tables and drawings.

The book covers:

- Introduction to sampling
- The core principles of sample system design
- Evaluation and design of sample transport lines
- Location and design of process sampling taps
- Preconditioning the process sample
- Controlling sample flow
- Controlling sample pressure
- Sample temperature control
- Change of state
- Sample conditioning and disposal
- Sample isolation and switching systems
- The future of process sampling

This book focuses on important design characteristics of sampling systems for process analytical measurements. Design principles that are essential to the success of any process analyser installation are discussed in full, as are the consequences of neglecting those principles. The application of sampling principles to homogeneous and heterogeneous process streams is considered in detail, as are the calibration and validation procedures, the maintenance concerns and the necessary safety precautions.