

## Paper 9.1

# Online Gas Chromatography: A Technical and Historical Overview – Design and Maintenance Advices to Achieve An Accurate End Result

**Steinar Fosse**  
Norwegian Petroleum Directorate

**Reidar Sakariassen**  
Metropartner

**Viktor Hauge**  
Fiskal Teknikk



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## **1 INTRODUCTION**

Gas chromatography was invented over 100 years ago by a Russian scientist. In the late 1940ies and early 1950ies the gas chromatograph principle was further developed by German and American scientists into an important practical analytical instrument. It was then used as a laboratory piece of equipment and the laboratory engineer/technician would have the full responsibility and control of the operation of the equipment.

Our business the offshore oil and gas business came a bit later. Our needs were connected to online sampling and analysis of natural gas. This was traditionally done by automatic sampling.

The main task for the Gas Chromatograph (GC) will then be to produce a correct component split for the gas analyzed. From the component split is then Gross Calorific Value (GCV) and density calculated. The GC can also be used for H<sub>2</sub>S measurement, but that will have to be a dedicated unit.

## **2 HISTORY IN THE NORTH SEA AREA**

The development in Norwegian and UK waters have been very much the same. The first offshore online gas chromatograph I saw installed was on 2/4 S platform where the Statpipe gas was tied into the Ekofisk complex. This device was ready for operation in 1984, but it was not much of a success story. The operation was so poor that the automatic grab sampling system was absolutely not challenged by this device. Also the Statfjord platforms that were delivering rich gas into the Statpipe system upstream of Kårstø were equipped with online gas chromatographs from Combustion Engineering (later taken over by ABB). Credit to the U.S. oil companies, Mobil and Phillips (now ExxonMobil and ConocoPhillips). They were pioneers to introduce this technology. The Statfjord online GC case already was presented at this conference in 1988, by Endre Jacobsen. The challenge on Statfjord as on most rich gas installations was to prove that the C<sub>6</sub>+ components are presented in the GC within an acceptable uncertainty. As this was rich gas and the sample handling systems were not of today's standard, it was shown that it was an impossible task to reproduce the C<sub>6</sub>+ component in the GC. The gas chromatographs on Statfjord therefore just served as pipeline monitoring device for 20 years.

The pipeline monitoring requirements are, however, also important and it was soon laid down as a requirement from the engineers responsible for the gas transport systems that all entry points into the gas pipelines should be equipped with online gas chromatographs.

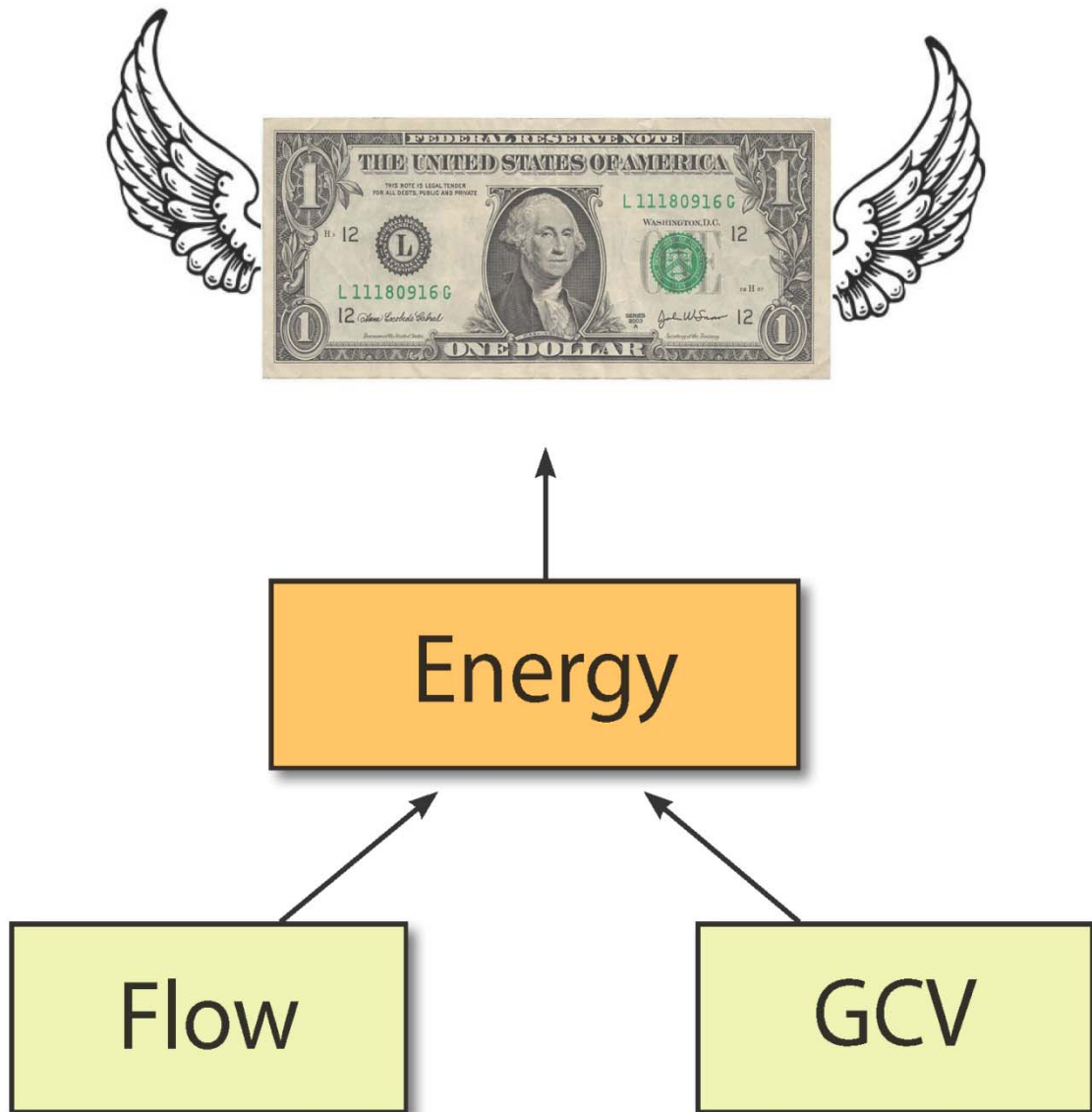
In 1993, we as Norwegian regulators were challenged at this NSF MW conference, then arranged at Solstrand near Bergen, Norway. The statement from the Elf (TOTAL) metering responsible was that the authorities were blocking the way for taking the online gas chromatographs in use for fiscal purposes. The response from the Norwegian Petroleum Directorate (NPD) was quite firm and easy. We had at that time not received any proposals for using the GC's for fiscal purposes.

Later in the 1990ies things changed and the GC's were gradually taken in service for fiscal purposes. First for dry gas systems, but then gradually also for rich gas systems.

It was several reasons for this:

- Sample handling systems with better equipment and design.
- More robust design of the GC itself.
- Easy to see all the benefits with GC compared to an automatic sample system.
- The GC`s were already in place for pipeline monitoring purposes.

**Fig. 1. Illustrates the importance of the online GC in a gas metering system**



The uncertainty of the analyzes from an online GC is of utmost importance to the companies that uses these figures in energy calculations, as that forms the basis for the economical transactions between seller and buyer. An online GC is, no matter how sophisticated it is, fully dependant on that a representative sample is delivered by the sample handling system.

3. A BRIEF DESCRIPTION OF A TYPICAL SAMPLE HANDLING SYSTEM

Fig 2. Typical sample handling system

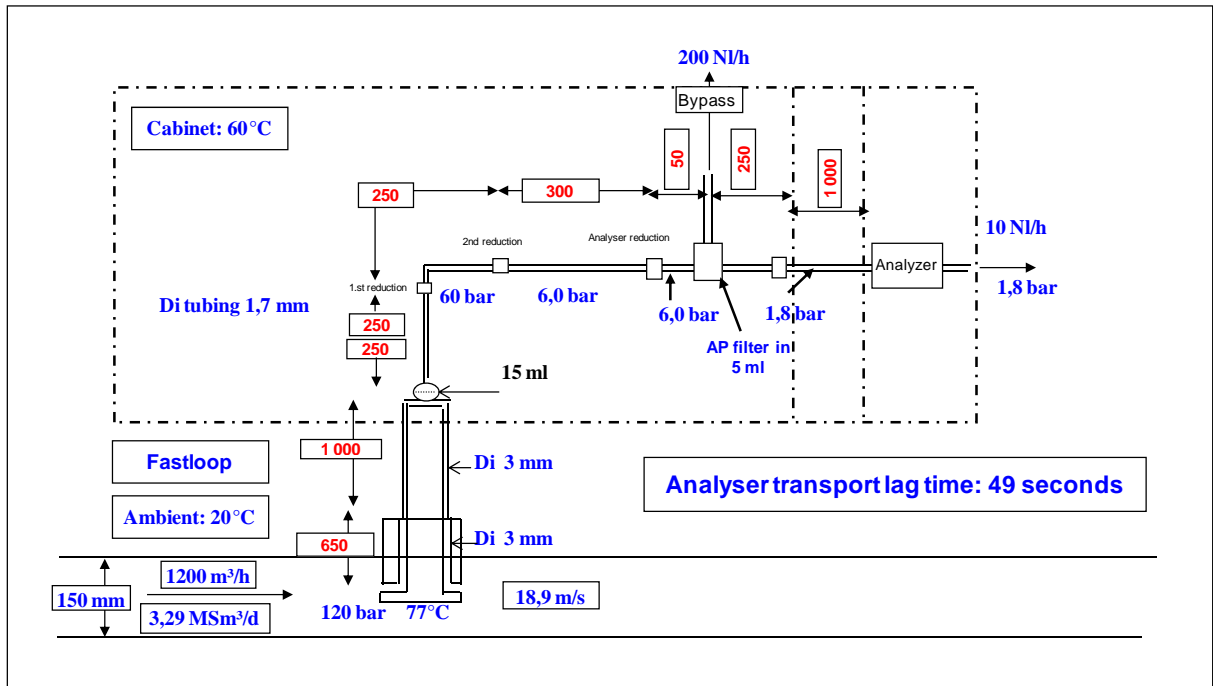
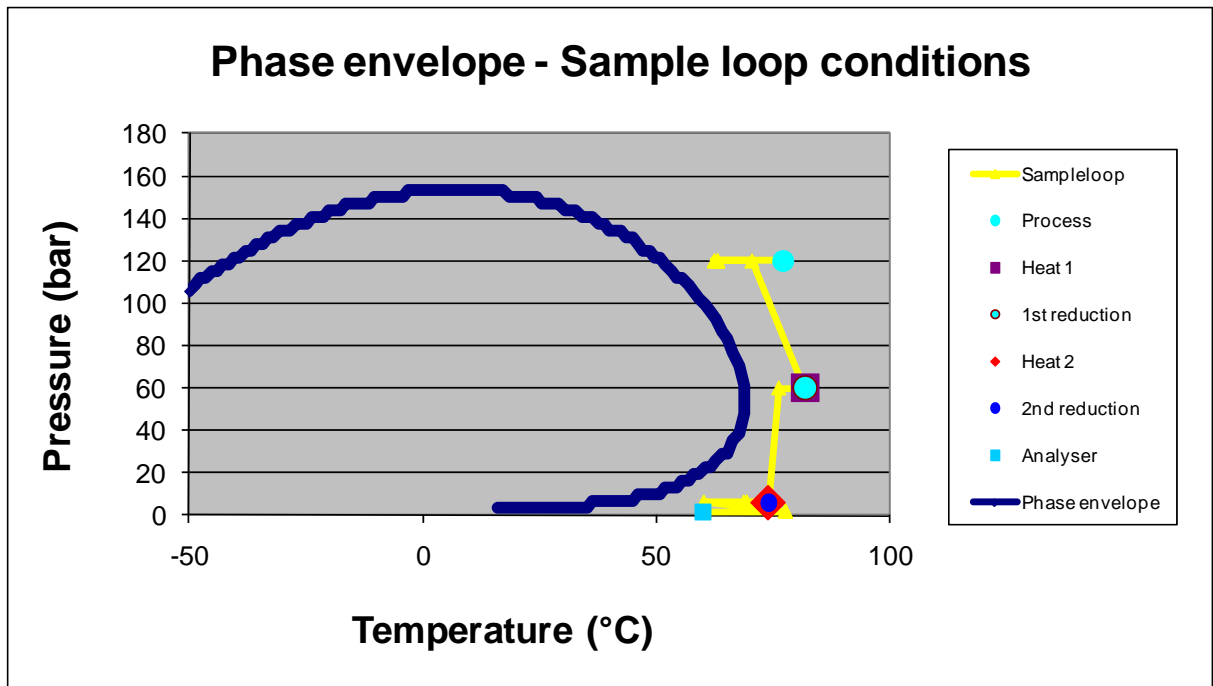


Fig 3. An example of the phase envelope calculation



### **a) Overview**

The phase envelope is given in figure 3. It is the ultimate calculation which always has to be done to ensure that the gas always stays in gas phase during the transport through the sample handling system. A two step pressure reduction is shown on figure. A one step pressure reduction is, however, often possible and should be aimed.

The elements which the sample handling system consists of: sample conditioning, probes, regulators, filters, heat tracing, tubing. The principle sketch is given in figure 2.

A sample analyzer must have a sample that is representative of the process stream reliable and accurately delivered to its injection valve. The sample should be unaffected by ambient conditions with a minimum of time delay between the sample point and the analyzer. When a sampling system for an analyzer is designed, a thorough knowledge of the process fluid is necessary including limitation requirements for pressure, temperature, solids, liquid contamination and corrosiveness that could affect the sample. For process analyzers, the speed and quality of the sample loop are important factors. Regulators, filters, tubing size, and fittings all contribute to the time lag between the sample point and the injection valve of the analyzer. Regardless of how sophisticated the analyzer is, and how much it costs, the proper sample handling is imperative.

Some attention should also be paid to the material selection of hoses, tubing and couplings. Robust materials should be selected to avoid any assumptions of molecular diffusion from test gas or reference gas into the instrument lines.

### **b) Some design considerations**

The pressure is taken from the high pipeline pressure and down to analyser pressure. The various calculations to be performed in connection with this sample handling system are extremely important:

- The gas conditioning calculation as it ensures that the gas is always on the right side of the phase envelope.
- The transport time calculation as it ensures that the sample is really representative for the time frame set. It also fulfil regulatory or international standard requirements for transport lag time.
- Pressure and temperature measurement on various places for monitoring of the status of the sample loop and eventually take action if some faulty indication comes up. This is important as it is here any problems to stay on the right side of the phase envelope will be detected.
- A flow indicator should be mounted in the loop so that it can be followed up that the GC is getting the right sample amount at all times.
- A pressure reduction valve with heated body shall always be selected.

The P& ID drawing will normally give you more details but the complexity of this type of drawings make it not useful for such a presentation as this. The principle sketch is given in fig. 2, as illustration.

### **c) Some key installation issues**

The probe should be located in an area where there is positive velocity with minimum turbulence. In areas of high turbulence, the contaminations in the gas stream that normally travel along the bottom of the pipe or close to the pipe walls will be put in flight. While in flight, they may be ingested into the probe and become a part of the sample. If the contaminations are in the gas phase, they are a part of the gas stream. If, however, they are in flight because of turbulence, they are not part of the gas stream and should not be analyzed. The probe should be installed in an as long pipe as possible (20 D minimum). Stay away from headers, blow down stacks, dead end lines, meter manifolds and reduced bore valves.

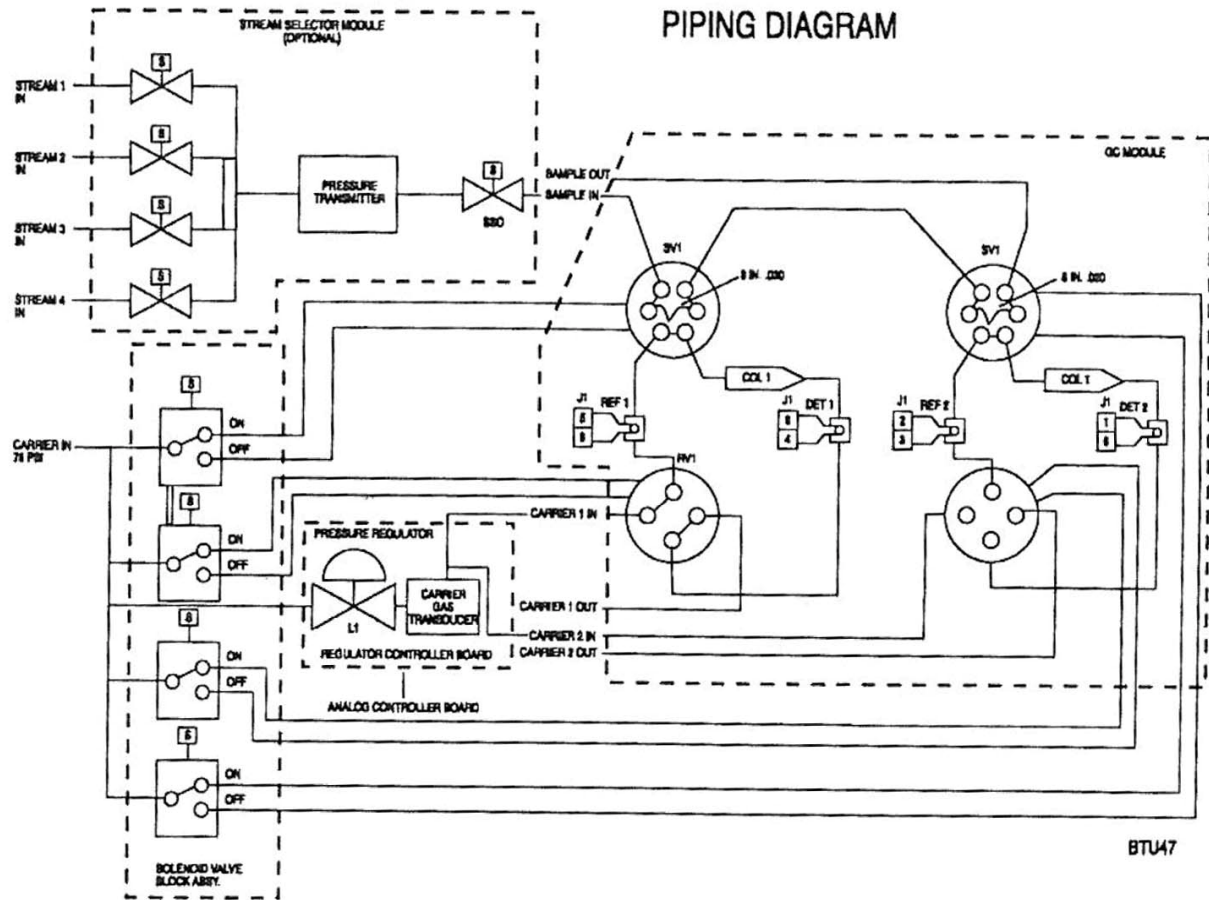
Install the probe on the top of the pipe with the probe 1/3 D into the pipe.

Install shut off valves on the outlet. The valves should be full bore valves which will not affect the flow of gas in open position.

By using a double pitot probe, a self driven fast loop can be made with outlet and return to the probe. Filter and regulator should then be the first equipment of the sample handling system. On the outlet side of the regulator a relief valve will have to be installed to protect the downstream system.

#### 4. A BRIEF DESCRIPTION OF A TYPICAL GC UNIT

Fig 4, A drawing from the AAI data book showing valves, columns and detectors.



#### GC Module Flow Diagram

The Gas Chromatograph module could consist of the following components as shown in fig 4:

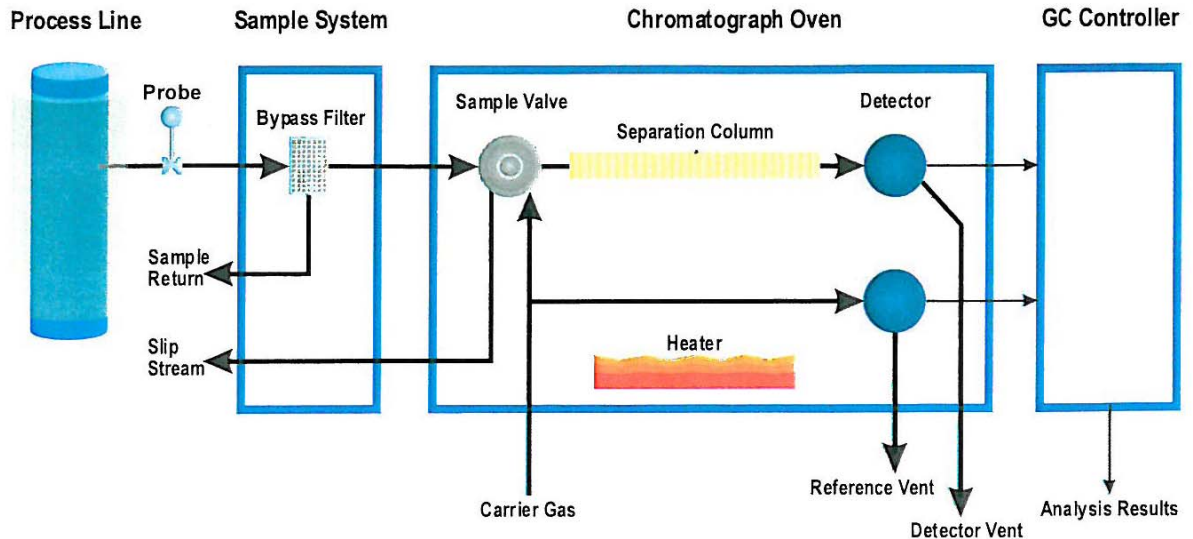
- Two sample valves, SV-1 and SV-2
- Two reverse valves, RV-1 and RV-2
- Two chromatograph stainless steel packed columns, COL-1 and COL-2
- Two dual element thermal conductivity 30k ohm matched thermistor detectors, DET-1 and DET-2
- Two reference detectors REF-1 and REF-2

The temperature within the GC module should be 60 deg C ( $\pm 0,3$  deg C).

The time for one sequence of a sample going through a column could be 60 seconds but the total time between two total cycles could be 180 seconds (3 minutes).

It is quite clear that when we speak of these small sample volumes and relative complex valves 6 port for sample valves and 4 port for the reverse valves, it is crucial important that all equipment operate according to their design to not disturb the analyzing result in any way.

**Fig 5, A drawing from the Emerson Daniel documentation which shows the principles of the GC and the gas flow through the system.**



Gas chromatography is in principle the separation of a mixture of chemical compounds due to their different migration rates through a separation column. This separates the compounds by boiling point difference, polarity differences or differences in molecular sizes.

The separated compounds then flow across a suitable detector (TCD) that determines the concentration of each component in the overall sample.

Knowing the concentration of the individual compounds makes it possible to calculate certain physical properties as Gross Calorific Value (GCV) or density based on standard equations from ISO 6976 or AGA 8.

## 5. AUTHORITY REQUIREMENTS

Natural gas for sales or allocation purposes has been under the jurisdiction of National metering authorities since the start up of the activity

The Department of Energy and Climate Change (DECC) in UK has in their Guideline section 4.3.5 to 4.3.8 some advices to avoid condensation problems when designing and during the operation of gas chromatographs.

The Norwegian Petroleum Directorate (NPD) has in their regulation section 8,11 and 17 with additional comments some requirements to Gas Chromatographs, in addition it refers to the NORSOK standard I-104 (2005), Fiscal measurement systems for hydrocarbon gas. This standard has in section 9 and Annex B several requirements for online GCs. It is setting maximum allowed requirements for uncertainty of various parts of the GC system and for linearity testing of the GC. The linearity check is required to be done with 3 gases. A 48 hours stability check during the FAT is also required.

Some relevant ISO/ASTM standards are also referred to in the NORSOK document, ASTM 1945, ISO 6974 part 1 and 3, ISO 6976, ISO 10715, as well as AGA report no 8.

The general NPD requirements for calculation check of software (section 8) and to maintain the equipment to the standard it is when installed (section 23) are also applicable for the online Gas Chromatographs.

It has often been so that the international standards serve as a good tool for maintaining a good quality. Not everything has however been mandatory. Parts could have been left out based on a judgement of the practicality of the requirements.

Recently a new authority has entered the scene. The European Union has through the climate quota trading scheme imposed requirements on natural gas used for fuel and if possible also for flare that base the quality requirements on ISO 10723, which require linearity tests on GCs to be done with 7 test gases and repeated at regular intervals. National agencies responsible for climate and pollution control handle the measurement challenges in connection to the CO<sub>2</sub> climate quota trading scheme. This has led to that on places where authority requirements already have been in place as in Norway, the requirements from a new agency comes on top and they are also sometimes more tight and stringent than the requirements that have been in place also for sales gas applications. This is in some way not logical, as the main money steam is still the sales gas. This has been a frustrating situation for the metering personnel in the oil/gas companies, and of course ourselves.

#### **Uncertainty calculation**

The overall uncertainty of the Gross Calorific Value (GCV) is in the NPD regulation (section 8) required to be less than 0,30%. The following components will have to be included on a component basis: Calibration gas uncertainty ( $U_{cxi}$ ) + the linearity of the GC (from linearity test) ( $U_{Lxi}$ ) + the repeatability of the GC (from linearity test) ( $U_{Rxi}$ ). The sum will then have to be root squared.

In addition the basic uncertainty of the values in the standard for GCV and molar mass adds up to the total uncertainty. This is in the existing ISO 6976, described in section 9.2. The accumulated uncertainty contribution from these effects could be estimated to 0,052%. In the standard it is mentioned that this uncertainty contribution may be neglected when compared to the uncertainty in the analytical data from the analysis.

The new ISO 6976 is dealing with this aspect in a more complex way in section 11 and 12, but it is no reason to believe that the end result would differ very much.

## **6. INTERNATIONAL STANDARDS**

### **ASTM 1945-03 (Reapproved in 2010)**

Standard test method for Analysis of Natural Gas by Gas Chromatography. It gives a good explanation of the method. It describe the apparatus and define how various parameters should be calculated. It also give some guidance on accuracy for linearity and repeatability as well as for test gases.

### ISO 6974 part 1,2,3,4,5 and 6 (2001)

The standard consist of 6 parts and gives a good guidance on the working principle as well as handling of data from the GC. The standard is now due for revision and the committee draft (CD) has recently been out for last comments/voting. It will then move into the Draft International Standard (DIS) phase, before it finally becomes an international standard.

### ISO 6976 (1995) (reprinted and corrected 1996) (GPA 2172, used in US)

The standard contains the calculations for Calorific Value, Density, Relative Density and Wobbe Index from composition. Also this standard is now due for update and the CD has recently been out for last comments/voting. It will then move into the Draft International Standard (DIS) phase, before it finally becomes an international standard.

### ISO 10715 (1997)



Natural Gas - Sampling guidelines. This standard will be updated in the near future and ISO is seeking experts for the working groups.

#### ISO 10723 (1995)

Natural Gas – Performance evaluation for on-line analytical systems. This standard is also now due for update and the CD has recently been out for last comments/voting.

It will then move into the Draft International Standard (DIS) phase, before it finally becomes an international standard.

It could seem that ISO structure related to the standards ISO 6974 and ISO 10723 cover very much of the same areas. They are also under the same committee, ISO /TC193. But now when all standards are due for new release it is unlikely that a simplification will happen. This could lead to situations where requirements are not similar and the logical question is what shall we then use. It is as far as we see it important to use all standards with the right objectivity so that equipment and testing are not over specified (or under specified) in any way. This requires a certain competence level in the oil/gas company to be able to make technical evaluations and make correct decisions.

The use of 7 calibration gases have been a discussion topic in relation to ISO 10723. Now it could be deducted that the standard is more open for flexible solutions in this area, That means use of 3 test gases as has been standard for FATs on Norwegian systems (as specified by NORSOK I-104, 2005). The precaution is that the area of each chromatogram for each component( C1 to C6) in relation to the measured amount (mol %) is presented by a polynomial function of the first order. Most vendors seems to use this method. (ref new ISO 10723 ch. 5.2.2 and 5.4.2).

## 7. SELECTION OF THE RIGHT GC EQUIPMENT AND SAMPLE HANDLING SYSTEM

Items to be aware when specifying a GC system:

- Components present
- Components to be measured
- Ranges of measurement
- Gas Conditions
- Standards to be met/used
- Legal requirements
- Sample handling
- Calibration method
- Carrier Gas
- Input/Outputs
- Alarms required
- Interface/communication links to onshore/vendor
- Duplication of various functions

The last item which mention duplication is something which is more and more used but we have to remember that to achieve a fully redundant system we have to duplicate also the sample handling system and the sampling probe and that is more seldom seen.

### Detectors

As an example a typical thing which need consideration is the type of detector the GC should be equipped with.

The Thermal Conductivity detector (TCD) is the most commonly used but also the Flame Ionization Detector (FID) is used. They both have their advantages and disadvantages. In some applications they might be combined. Also other detectors for more special applications might be considered, but all this will have to be based on vendor recommendation. It is therefore so crucial important that the process information the vendor receives is correct.

A brief explanation of the two detector types are given underneath.

## **TCD**

Since all compounds, organic and inorganic, have a thermal conductivity, all compounds can be detected by this detector. The TCD is often called a universal detector because it responds to all compounds, it will respond similarly to similar concentrations of analyte.

The TCD is a good general purpose detector. The TCD is less sensitive than the flame ionization detector and has a larger dead volume it will not provide as good resolution as the FID. However, in combination with thick film columns and correspondingly larger sample volumes, the overall detection limit can be improved. The TCD is not as sensitive as other detectors but it is detecting all components and is non-destructive.

The TCD is also used in the analysis of permanent gases (argon, oxygen, nitrogen, carbon dioxide) because it responds to all these pure substances unlike the FID which cannot detect compounds which do not contain carbon-hydrogen bonds.

## **FID (not allowed on many offshore installation for safety reasons)**

FID's are best for detecting hydrocarbons and other easily flammable components. They are very sensitive to these components, and the response tends to be linear across a wide range of concentrations.

However, a FID destroys most - if not all - of the components it is detecting. Contrarily, with a TCD the components can continue on to another detector after passing through the TCD; thus it is considered a non-destructive detector. However, with an FID, most components are destroyed and no further detection is possible.

The current measured, between the electrodes corresponds roughly to the proportion of reduced carbon atoms in the flame. The response of the detector is determined by the number of carbon atoms (ions) hitting the detector per unit time. This makes the detector sensitive to the mass rather than the concentration, which is useful because the response of the detector is not greatly affected by changes in the carrier gas flow rate.

FIDs are insensitive to H<sub>2</sub>O, CO<sub>2</sub>, CS<sub>2</sub>, SO<sub>2</sub>, CO, NO<sub>x</sub>, and noble gases because they are not able to be oxidized/ionized by the flame.

The Methanizer enables the Flame Ionization Detector to detect low levels of CO and CO<sub>2</sub>. The Methanizer is packed with a nickel catalyst powder. During analysis, the Methanizer is heated to 380 °C. When the column effluent mixes with the FID hydrogen supply and passes through the Methanizer, CO and CO<sub>2</sub> are converted to methane.

## **Location of equipment**

The probe shall be located at a place where it is possible to achieve a representative sample.

The distances between the sample probe and the GC shall be as short as possible and similarly the reference gas and carrier gas should be conveniently located. We have over the years seen a large number of rather inconvenient installations. (For example GC and Gas bottle cabinet in a high location where it is very difficult to bring large bottles).

The distance between the sample probe and the GC is in many installations unnecessary long.

## **8. REFERENCE GAS**

ISO 6142 (2001) – Gas analysis – Preparation of calibration gas mixtures – Weighing methods

ISO 6143 (2001) – Gas analysis – Determination of composition of calibration gas mixtures – comparison methods.

It is important that the reference gas is specified and produced to the highest standard at the production facility. It is, however, a long and winding road from the production facility to the place where it shall be used. The transport should therefore be done in a controlled way and precautions should be implemented when arriving at the place where it shall be used, so that the reference gas is in good shape when it is connected to the GC.

The reference gas is delivered with a traceable certificate where all critical data as components ordered and delivered as well as uncertainty will be stated. The normal validity of the gas is 3 years. Also the minimum storage temperature which often is 10 deg C will be stated, as well as the minimum bottle pressure. If some of this information is missing then the vendor shall immediately be contacted.

If the bottle has been exposed to temperatures below the preset limit, then retrograde condensation would have taken place. The bottles should then be kept in a heated cabinet for some time before they are connected so they are brought back to their original condition. Eventually rolling of the bottle should also be considered if necessary to bring bottle back to the original condition.

When bottles are changed then the system should be flushed and the regulator valves should be checked for any failure. This to ensure proper operation when the system is set back in service.

Flexible hoses should not be used as connecting lines between the reference gas and the GC as diffusion can take place. O<sub>2</sub> can enter into all materials except copper and stainless steel.

The CO<sub>2</sub> quota trading regime has imposed a requirement for accreditation of reference gas deliveries. This has created some difficulties as very few vendors had an accreditation certificate.

## **9. HOW TO MONITOR THE ONLINE GC`s DURING THE DAILY OPERATION**

Vent system influence the raw data of the GC.

The vent system should therefore be designed to minimize these effects, by having the vent duct in a sheltered area. At least it should not be directly exposed to the weather as we have seen examples of.

It is primarily in the "raw" values we see this effect. Any calibration activity against the GC should therefore not be launched by a shift in the "raw" values alone. By using normalized values, the weather effect is masked.

a)

Various temperature and pressure and flow elements in the sample loop shall be verified to ensure the healthiness of the sample system at all times. Limits for the operation and relevant action shall be defined. All pressure reduction valves should be equipped with pressure and temperature monitoring equipment. Rich gas systems are obviously more exposed for difficulties in the sample handling system. If the gas composition changes then a verification of the sample handling system should take place to ensure that the gas is still outside the phase envelope.

Other items to be checked regularly in the sample loop:

- Filters to be checked or replaced at regular intervals
- Check of carrier gas consumption
- Check of calibration gas heater
- Check of calibration gas flow rate
- Check of carrier gas pressure

b)

**Fig 6, the Formula 1 cars are indicating the various components.**



We have hopefully all seen the chromatograms which the chromatographs gives out. The peaks are coming with a fixed shape and preset plan. If this is upset in any way then something is wrong. Just as Formula 1, therefore the illustration.(At least as people who are not fans of that sport could see it).

The chromatograms should be regularly examined to detect any weaknesses with the GC. The shape and position of the peaks should be examined. Some examples:

- Column failure will give valve timings not longer appropriate.
- If some small components misses then it can be a sign that the flow of carrier gas is too low.
- The shape of the peaks of the largest being too flat, indicate too high flow of carrier gas.

c)

The test against the reference gas (benchmark) is a standard test which is done regularly on all systems. It is here important that calibration activity is not launched as soon as we see a shift. Reasonable limits should be set for critical parameters and when some of these are over run then a calibration activity should take place. It should, however, not necessarily take place immediately. The weather effect as mentioned in item a) is influencing the injected volume. Therefore it should be set a band where it is acceptable to do calibration. If we are outside this band then a calibration should not be done. A calibration should then preferably be followed by a benchmark to verify that the situation has improved.

Changes in injected sample volume are affecting the small components heavily, therefore the control limits on these will have to be wider or evaluated by statistic.

The ASTM 1945 -03, has a requirement which says that the calibration gas components should be close to the real gas (limits are set). This can be hard to meet for the small

components as they can vary significantly. It should, however, not be focused too much on this as it easily can be demonstrated that the impact on the overall uncertainty is small.

Calibration shall be done when it is necessary, but it should be as seldom as possible. The reason for this is that it changes the reference point and that reduces the ability to trace wear and tear in the columns, detectors and valves.

d)

If the gas chromatograph is duplicated as they often are (NPD requirement (section 11) on gas sales stations) then the two units should be compared against each other. Realistic deviation limits should be set and proper action should be taken if these are over run. It is a requirement in these situations that you should use unit 1, as long as both units are defined as healthy. An average value from both units is also possible to use and then switch to the healthy unit if one fails.

Some gas chromatographs now have double set of equipment within the same unit. Then same type of philosophy as above should be applied.

e)

**Response factor** = Area response from a detector for one component related to a reference

**The retention time** = The time it takes for a single component to pass through the column

The response factor and the retention time will have to be monitored regularly.

Realistic alarm limits will have to be set for both the Response factor and the Retention time. Trending should be performed. Both these parameters will if they exceed preset limits indicate column failure.

f)

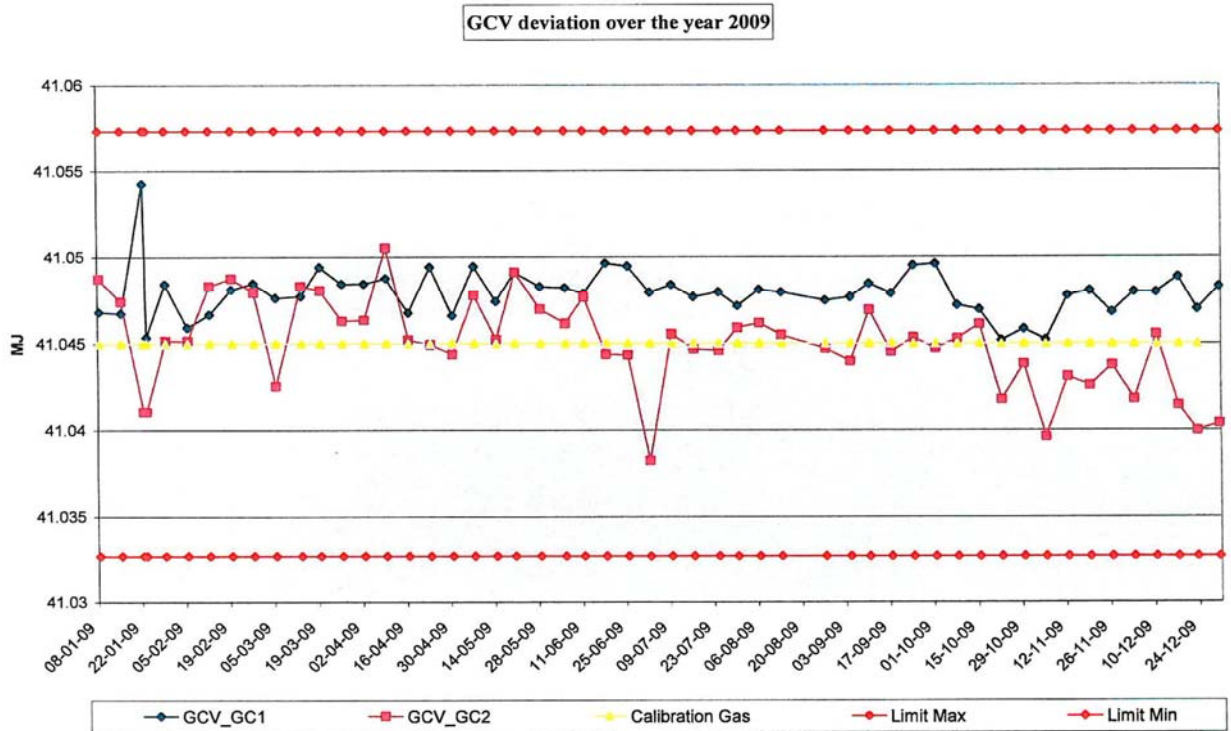
Regular linearity checks are done some places (land terminals), with seven test gases according to present ISO 10723. Some places 3 test gases are used. If the Gas Chromatograph is used at a stable GCV as it normally is, regular linearity checks should not be necessary. The regular benchmark and calibration should take care of the quality follow up together with other monitoring and maintenance activities on the equipment which is described in this paper.

If it is suspected any error or just to confirm the performance, a test with one reference gas with other data than the permanent reference gas can be done.

The updated standard ISO 10723, open for an evaluation of number of points to be used based on what will be the normal operating area of the GC, and that is positive.

g)

Trending of the final outcome of the GC, the GCV is an important monitoring tool if two GC's are in place. Figure 7, is giving an example of this and the limits in use. Be aware of the alarm limit  $\pm 0,03\%$ .



h)

Regular maintenance support visits by GC specialist (vendor/independent) to verify the status of the chromatograph and also with the aim to transfer competence to maintenance personnel could be a part of the quality follow up strategy on the online gas chromatographs.

## 10. SUMMARY

The speed of the Gas Chromatographs have the recent years increased. 3 minutes or may be even also faster than that is now the cycle time on new units.

Some wish to specify as low cycle time as possible. We have however seen that this is not always a good idea. The unit need some time to be able to separate the components properly,

And that is of course the most important job for the chromatograph. If you therefore for one or another reason need high speed you better install two units and space them in time.

The Online Gas Chromatograph technology including sample handling now seems to have reached a level where it is fully capable of delivering high quality data to the public.

The human factor may always have a high potential for causing errors. The rich gas systems on the offshore installations are mostly exposed, due to heavier gas, less redundancy and more changing of people. The focus on staff, training, maintenance and quality control can also here never be underestimated. The management focus is also a part of this. To really get management acceptance that the GC is an important equipment for the cash flow in the company. (See figure 1 of this paper).

The Gas Chromatograph is a nice and sophisticated piece of technology which really need tenderly care to perform to its specification.

A GC maintenance strategy document to be developed. Alarm limits on different parameters should be set. Trends on different parameters should be logged and followed up. It should be clearly defined what work shall be done on the installation and what shall be done from coordinating engineering office or from vendor.

Recruitment and training is always important. This will have to be paid special attention as the tenderly care for the GC`s will have to be taken care of. Time is of course also important. The allocation of necessary time for the follow up job must be done.

Best judgement should be steering. Our aim should be to do technical actions based on that they improve quality and not because they are written in some documents for quite different applications. To be able to act in this manner we need competence both on the authority side and on the oil/gas company side, as well as with vendors and other contractors involved.

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