SGC Rapport 2013:291

# Best practice for controlling content of oil, water and sulphur in CNG at refuelling station level

## (Bästa förfarande för begränsning av innehåll av olja, vatten och svavel i fordonsgas på tankstationer)

**Dino Novosel**

*"Catalyzing energygas development*
*for sustainable solutions"*

SGC Svenskt Gastekniskt Center AB

**Best practice for controlling content of oil, water and sulfur in CNG at refuelling station level**
(Bästa förfarande för begränsning av innehåll av olja, vatten och svavel i fordonsgas på tankstationer)

Dino Novosel

Denna studie har finansierats av/This study has been funded by:
Energimyndigheten/Swedish Energy Agency
E.ON Gas Sverige AB
Kraftringen Produktion AB
FordonsGas Sverige AB
Svensk Växtkraft AB
Ballast Nedam IPM
Bohlen & Doyen Bauunternehmung GmbH
Idro Meccanica srl
WEH GmbH Gas Technology



*Postadress och Besöksadress*
Nordenskiöldsgatan 6 4tr
211 19 MALMÖ

*Telefonväxel*
040-680 07 60

*Telefax*
0735-279104

*E-post*
info@sgc.se

*Hemsida*
www.sgc.se

# Svenskt Gastekniskt Center AB, SGC

SGC är ett spjutspetsföretag inom hållbar utveckling med ett nationellt uppdrag. Vi arbetar under devisen *"Catalyzing energygas development for sustainable solutions"*. Vi samordnar branschgemensam utveckling kring framställning, distribution och användning av energigaser och sprider kunskap om energigaser. Fokus ligger på förnybara gaser från rötning och förgasning. Tillsammans med företag och med Energimyndigheten och dess *Samverkansprogram Energiteknik* utvecklar vi nya möjligheter för energigaserna att bidra till ett hållbart samhälle. Tillsammans med våra fokusgrupper inom *Rötning*, *Förgasning och bränslesyntes*, *Lagring och transport*, *Industri och hushåll* och *Gasformiga drivmedel* identifierar vi frågeställningar av branschgemensamt intresse att genomföra forsknings-, utvecklings och/eller demonstrationsprojekt kring. Som medlem i den europeiska gasforskningsorganisationen GERG fångar SGC också upp internationella perspektiv på utvecklingen inom energigasområdet.

Resultaten från projekt drivna av SGC publiceras i en särskild rapportserie – *SGC Rapport*. Rapporterna kan laddas ned från hemsidan – www.sgc.se. Det är också möjligt att prenumerera på de tryckta rapporterna. SGC svarar för utgivningen av rapporterna medan rapportförfattarna svarar för rapporternas innehåll.

SGC ger också ut faktabroschyrer kring olika aspekter av energigasers framställning, distribution och användning. Broschyrer kan köpas via SGC:s kansli.

SGC har sedan starten 1990 sitt säte i Malmö. Vi ägs av E.ON Gas Sverige AB, Energigas Sverige, Swedegas AB, Göteborg Energi AB, Lunds Energikoncernen AB (publ) och Öresundskraft AB.

Malmö 2013

Martin Ragnar
*Verkställande direktör*

# Swedish Gas Technology Centre, SGC

SGC is a leading-edge company within the field of sustainable development having a national Swedish assignment. We work under the vision of *"Catalyzing energygas development for sustainable solutions"*. We co-ordinate industry-wide technical development on the production, distribution and utilization of energygases and disseminate knowledge on energygases. Focus is on renewable gases from anaerobic digestion and gasification. Together with private companies and with the Swedish Energy Agency and its frame program *Co-operational program in Energygas technology* we develop new solutions where energygases could provide benefits for a sustainable society. Together with our focus groups on *Anaerobic digestion*, *Gasification and fuel synthesis*, *Storage and transportation*, *Industry and household* and *Gaseous fuels* we identify issues of common interest in the industry to conduct joint research, development and/or demonstrations projects on. As a member of the European gas research organization GERG, SGC provides an international perspective to the development within the Swedish energygas sector.

Results from the SGC projects are published in a report series – *SGC Rapport*. The reports can be downloaded free of charge from our website – www.sgc.se. It is also possible to subscribe to the printed reports. SGC is responsible for the publishing of the reports, whereas the authors of the report are responsible for the content of the reports.

SGC also publishes fact brochures and the results from our research projects in the report series *SGC Rapport*. Brochures can be purchased via the website.

SGC is since the start in 1990 located in Malmö. We are owned by E.ON Gas Sverige AB, Energigas Sverige, Swedegas AB, Göteborg Energi AB, Lunds Energikoncernen AB (publ) and Öresundskraft AB.

Malmö, Sweden 2013

Martin Ragnar
*Chief Executive Officer*

# Author's foreword

The goal of the study has been the creation of an informative document for the elaboration of best practices collection when controlling the content of oil, water and sulfur in CNG at refuelling station level. The final purpose of the study has been to create recommendations on how to deal with the above mentioned concerns that apply at refuelling station level. The study has been done with a system perspective, taking into account the opinions/recommendations of every stakeholder involved in the process (vehicle/engine manufacturers, component manufacturers, refuelling station builders, final users, fleet operators, etc.). The consultant was requested to use the Swedish report on oil in gas[1] as a major source of information concerning oil in gas that may be used as a template regarding the structure of the new study. Regarding sulfur, the German developments on the issue were used as the main source of information, with German actors such as DVGW and Erdgas Mobil as main points of reference. Machine translations of the following Swedish reports[2] have been useful as further sources of information.

Dealing with the different types of oil used for NG compression has also been in focus, with regard to end-user complications such as component non-compatibility with the oil itself or any of its additives. An exhaustive survey from as many different European markets as possible has been carried out.

According to the Project definition, the following deliverables were to be addressed:

- General information on oil, water and sulfur in relation to natural gas distribution, CNG and NGV’s.
- Surveying the current situation among operators and market actors in Europe regarding operational disturbances and malfunctions attributed to oil and water, and their possible solutions, through questionnaires and telephone follow-ups. An exhaustive study is not necessary, but the major actor or actors in the most prominent markets should be covered, with at least the following countries included: The Netherlands, Sweden, Germany, Italy, Austria, Switzerland, France, Spain, Portugal, Czech Republic and Poland. In addition, all vehicle manufacturers selling NGVs on the European market should be approached, such as Volvo, Scania, Mercedes, MAN, IVECO, Volkswagen, Fiat and Opel, to find out if they have recommendations on critical levels of oil, water and sulfur for current and future vehicles, engines and aftertreatment technologies
- Survey of the market state-of-the-art regarding removal technology for oil, water and sulfur.
- Survey of the NG compressor oil market, and if possible make recommendations regarding best choice of oil. The issue of component non-compatibility regarding the compressor oil or any of its additives should be addressed.
- Survey of the current situation regarding sampling and analysis of oil, water and sulfur. What is the situation regarding validation of test methods which are or can be referred to in national and international standards?
- Recommendations for best practice control measures of oil, water and sulfur from the viewpoint of optimal instalment and configuration of removal equipment

[1] Clementsson M, Held J (2007). “Olja i fordonsgas – regelverk, mätmetoder och filter” non-public SGC Report, 34 p.
[2] Svensson M (2011). ”Utvärdering av svensk biogasstandard - underlag för en framtida revision” SGC Rapport229 http://www.sgc.se/ckfinder/userfiles/files/SGC229.pdf Norén C, Thunell J (2002). “Svavelfri och svavelfattig odorisering av gasol och naturgas” SGC Rapport A26 http://www.sgc.se/ckfinder/userfiles/files/SGCA26.pdf

# Summary

The purpose of this report is to provide a general overview of the CNG market in Europe, with special attention to contaminants in CNG at refuelling station level, such as oil, sulfur and water, which are affecting the quality of the CNG delivered. The report was created from a system perspective, where all the key groups of stakeholders, including Refuelling Station Operators, CNG Equipment Producers, NGV OEMs and NGV Fleet Operators/Users have been interviewed and their practices in dealing with contaminants in the CNG were analysed and benchmarked against the identified key success factors (KSF) that are important for ensuring a premium quality of the CNG in the broader European marketplace.

The ultimate goal of the report is to serve as a collection of best practices for all the addressed groups of the stakeholders, in defining and implementing their strategies toward zero contamination CNG. The document is thus structured in a logical framework that consists of the three major parts: theoretical, practical/experienced and recommendations.

In the first theoretical part (chapter 1-7), a presentation is made to convey a general understanding of the subject matter and the mechanisms of contamination of CNG at refuelling station level, how to monitor these contaminants and finally how to remove/minimize them. Special attention has been given to the available state-of-the-art technologies that may facilitate the work of improving the CNG quality, which eventually means lower levels of oil, sulfur and water in the fuel and increased customer satisfaction. Since real-world measurement of the aforementioned contaminants is quite challenging, detailed case studies from market leaders such as Sweden, Germany and USA have been thoroughly evaluated so as to serve as best practice for emerging markets.

The second part of the best practices collection has been based on the practices and experiences that were collected from market players. In this respect, 115 stakeholders in total from 17 EU member states have been approached with tailor made questionnaires (followed up by telephone interviews) to gather their experiences. The resulting feedback is presented in Chapter 8.

Based on the significant results of the market investigation and the presented theoretical / Industrial knowledge, a list has been drawn up of the key success factors (KSF) which affect the final quality of the CNG delivered at refuelling station level. It is presented in the last section, with recommendations and suggestions for best practices (Chapter 9). In total, 29 KSFs were identified. These have been evaluated in relation to the experiences reported by the two key stakeholders, Refuelling Station Operators and CNG Equipment Producers. This has then been presented in the form of a detailed gap analysis, together with a KSF priority list.

For oil lubricated compressors two effective methods of oil minimization have been identified. The first method is implemented when using a polyglycol type of oil. At least two highefficiency coalescing filters to remove oil aerosols should be installed in series, one close to the compressor, one further away to allow for maximum cooling and precipitation of dissolved/vapourised oil. The second method applies if mineral oil is used; then a final adsorption filter to remove oil in the vapor phase should also be installed.

Oil consumption in the compressor should be monitored continuously, partly to assess the proper functioning of the compressor since increased consumption indicates a need for service, partly since the amount of oil added gives a rough estimate on how much oil is contained within the CNG delivered. In addition to this control, follow-up measurements of the gas are also required. The problem today is that there is no standardized method for measuring oil in the CNG, neither for the oil aerosols or the vapour phase oil. New work in Germany and Sweden are showing great promise; the Swedish method from SP will probably be used as a reference in the on-going CEN work on standardization of CNG/biomethane.

# Sammanfattning på svenska

Syftet med denna rapport är att ge en allmän översikt över fordonsgasmarknaden i Europa, med särskild fokus på föroreningar i fordonsgas på tankstationsnivå, till exempel olja, svavel och vatten, vilka påverkar kvaliteten på levererad fordonsgas. Rapporten skapades ur ett systemperspektiv, där alla de viktigaste grupperna av intressenter, (Tankstationsoperatörer, Fordonsgaskomponenttillverkare, Fordonstillverkare och Gasfordonsoperatörer har intervjuats och deras rutiner i hanteringen av föroreningar i fordonsgas har analyserats och jämförts med de identifierade nyckelframgångsfaktorerna (KSF) som är viktiga för att upprätthålla en hög kvalitet på fordonsgas på den europeiska marknaden.

Det yttersta målet med rapporten är att fungera som en samling av bästa förfarande för alla berörda grupper av intressenter när de utformar och genomför sin strategi för noll föroreningar i fordonsgas. Dokumentet är således uppbyggd på ett logiskt ramverk som består av de tre stora delar: teoretisk, praktisk/erfarenhetsbaserad och rekommendationer.

I den första teoretiska delen (kapitel 1-7), presenteras området för att ge en allmän förståelse om ämnet, beskriva mekanismerna till förorening av fordonsgas på tankstationsnivå, hur man kan övervaka dem och slutligen hur de kan tas bort/minimeras. Särskild uppmärksamhet har ägnats åt att beskriva bästa tillgängliga teknik som kan underlätta arbetet med att förbättra fordonsgaskvaliteten, vilket på sikt innebär lägre nivåer av olja, svavel och vatten i bränslet och ökad kundnöjdhet. Eftersom verklig mätning av dessa föroreningar ännu är ganska utmanande, så har detaljerade fallstudier från marknadsledande länder som Sverige, Tyskland och USA noggrant utvärderats för att fungera som bästa förfarande för tillväxtmarknader.

Andra delen av insamlingen av bästa förfarande har baserats på metoder och erfarenheter insamlade från marknadsaktörer. I detta avseende har totalt 115 aktörer från 17 EU-medlemsstater fått sig tillskickat skräddarsydda enkäter (uppföljt av telefonintervjuer) med frågor om deras erfarenheter. Den resulterande återkopplingen presenteras i kapitel 8.

Grundat på de omfattande resultaten från marknadsundersökningen och den presenterade teoretiska/industriella kunskapen så har en lista tagits fram över de viktigaste framgångsfaktorerna (KSF) som påverkar den slutliga kvaliteten på fordonsgas levererad på tankstationsnivå. Den presenteras i den sista delen, med rekommendationer och förslag på bästa praxis (kapitel 9). Totalt har 29 KSFs identifierats. Dessa har utvärderats i förhållande till erfarenheterna inrapporterade av de två nyckelaktörerna, Tankstationsoperatörer och Fordonsgaskomponenttillverkare. Detta har presenterats i form av en detaljerad gapanalys, tillsammans med en prioriteringslista för de olika nyckelframgångsfaktorerna (KSF).

För oljesmorda kompressorer har två metoder identifierats för effektiv oljeminimering. Den första metoden tillämpas vid användning av en polyglykol-baserad olja. Minst två högeffektiva coalescing-filter för att ta bort oljeaerosoler bör installeras i serie, en just efter kompressorn, en längre bort för att möjliggöra maximal kylning och utfällning av upplöst/förångad olja. Den andra metoden gäller om mineralolja används; då bör ett avslutande adsorptionsfilter installeras för att avlägsna olja i ångfasen.

Oljekonsumtionen i kompressorn bör övervakas kontinuerligt, dels för att bedöma kompressorns funktion, eftersom ökad konsumtion indikerar ett behov av service, dels eftersom mängden tillsatt olja ger en grov uppskattning av hur mycket olja som finns i fordonsgasen som levereras. Utöver denna kontroll krävs också uppföljande mätningar av gasen. Problemet i dag att det inte finns någon standardiserad metod för mätning av olja i fordonsgasen, varken för oljeaerosoler eller för oljan i ångfas. Nytt arbete i Tyskland och Sverige är hoppingivande; den svenska metoden från SP kommer troligen att användas som referens i det pågående CEN standardiseringsarbete av CNG/biometan.

# CONTENTS

# 1. BACKGROUND

The Natural Gas Vehicle (NGV) market is still young in most of the European countries, and thus still lacking many of the regulations and standards that may inform and guide the actions of the different market actors involved. An example of this can be found in the erection and management of NG refuelling stations, which currently has no international standards.

The practical work regarding gas quality control is in most cases not an issue at this level, since this is done upstream. However, the issue of oil and water content is important to address at refuelling station level.

Water is monitored and controlled throughout the natural gas distribution networks, to minimize unwanted corrosion. Nevertheless, at refuelling station level the issue of drivability makes it necessary to remove water to a further extent, in order to avoid hydrocarbon hydrate formation in the vehicle pressure regulator during decompression.

The possibility of compressor oil being entrained into the final product, together with the fact that these oil slips going into NGV engines could cause operational problems with pressure regulators and gas injectors, means that oil levels need to be monitored and controlled through oil removal downstream of the compressor. Another important aspect is to manage any vehicle incompatibility issues regarding the oil in itself or any of its additives.

With increasingly stricter emission regulations, the optimal performance of the aftertreatment system becomes more and more important. With this in mind, minimizing the sulfur levels of the fuel is crucial to maximize its performance and durability. It's well known that NG is currently used for many different applications, but since no other gas utilization has these demands, it may be a solution to handle this at refuelling station level. So far, this has only been proposed in Germany, where some sulfur rich natural gas qualities are in use. For most other gases, the sulfur content is dominated by the contribution from the odorant addition.

# 2. INTRODUCTION – GENERAL INFORMATION ON OIL, WATER AND SULFUR IN RELATION TO NATURAL GAS DISTRIBUTION, CNG AND NGV'S

If nothing else is noted, this chapter is based on excerpts from the published report: Clementsson M, Held J (2007). “Olja i fordonsgas – regelverk, mätmetoder och filter” non-public SGC Report, 34 p.

## 2.1. Oil

Vehicle gas, namely biomethane or natural gas used as vehicle fuel, is compressed to about 200 bar prior to charging to the vehicle's tanks. Small amounts of oil used to lubricate the compressor can accompany the gas. Oil removal in the compressor and in the filters after the compressor stages reduces the oil content in the gas. The small amount of oil that comes with the gas (ppm level) can be incinerated with CNG.

Even though the refuelling station compressors are the main source, the oil contained in CNG may come from more sources. Not only can it come from the compressors in the gas network, but also from air compressors in refuelling stations.

Oil can be used for the lubrication of both the cylinder and the drive unit in the compressors.

Depending on the lubrication, compressors can be divided into three types:

- compressor with oil lubrication of cylinder and drive unit
- compressor with oil lubrication of drivers, but with oil-free cylinder
- compressor with oil free cylinders and drive unit

The latter two variants are known as oil-free or dry compressors. Both oil lubricated and dry compressors are used in Europe. The more oil that is handled in the compressor, the greater the risk is that oil is entrained with the gas. Oil lubricated compressors thus have, compared to dry compressors, a greater need for effective and properly maintained oil filters.

In dry reciprocating compressors there is a pressurized ventilated space between the oil lubricated crankcase and the gas side. Dry hydraulic compressors also have a pressurized ventilated space between the gas cylinder and the driving hydraulic oil cylinder, to prevent oil leakage.

The lubricating oil has a number of functions in the compressor. First, it separates and reduces the friction between the parts that slide against each other, and the oil dissipate heat, improves piston seal at compression and carries away dirt particles.

There are two main types of lubricating oil. One is mineral oil, which is produced by distilling and refining of oil. The other is synthetic oil, which also can be produced from crude oil, but normally produced by a chemical process synthesis.

The oil that comes with CNG is partly in the form of aerosols, i.e. small liquid droplets formed by compression movements of the cylinder (mechanical shearing), partly in the form of oil vapour that forms on hot surfaces. At high pressure oil can also dissolve into CNG[3].

Aerosols are formed in the compressor during the compression, and after the final cooling, aerosols in the gas have a size of 0.1 to 0.8 microns. Mineral oils can be evapourated and absorbed in the vehicle at the pressure and the temperature of the compressor. When the temperature and/or pressure drop some of the oil in the vapour phase condenses to small aerosols with a size from about 0.01 to 0.8 microns.

[3] DVGW Jahresrevue, Erdgastankstellen: Öleintrag ins komprimierte Erdgas gewinnt zunehmenden Beachtung, sid 2-4, Energie/Wasser-Praxis, nr 12, 2006.

Overall, this means that aerosols of oil contained in CNG after the final cooling usually have a size of 0.01 to 0.8 microns[4].

All mineral oils can be dissolved in CNG. At pressures, density and temperatures above the critical pressure and temperature, under so-called super-critical condition, more oil can dissolve into CNG. This behaviour is demonstrated in next Figure. The figure clearly shows that with increasing pressure and temperature, more mineral oil may be in equilibrium with the gas. It should be noted that the values in the figure are theoretical and are calculated with a model of steady state, which means that such high saturation levels in practice are never attained.

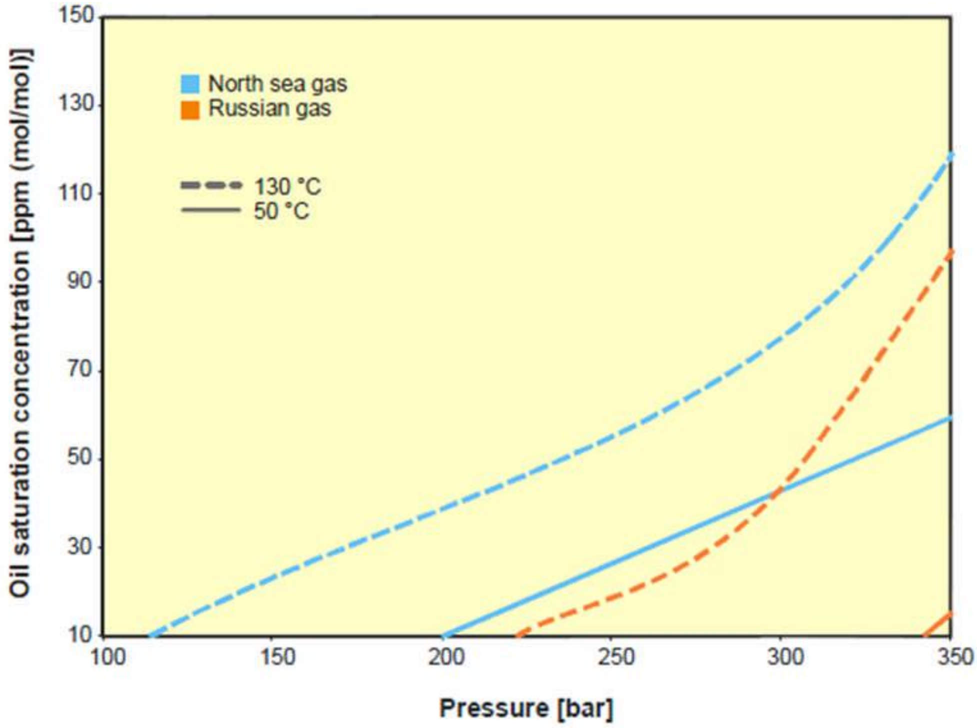



*Figure 1 Mineral oil saturation concentrations of different gas compositions at 50 and 130 °C as calculated by Aspen Hysys using the Peng-Robinson equation of state*[5]

For pure methane the critical pressure is 46.4 bars and the critical temperature is -82.5 °C. The solubility decreases with increasing molecular weight, which is the case with a higher share of heavier hydrocarbons in the gas. Also, the absolute values of oil solubility vary depending on gas composition; naturally occurring higher hydrocarbons (e.g. $C_5 – C_7$) have a distinct solubilizing effect on mineral oil.[6] The oil solubility in CNG becomes notable only above approx. 120 bars. Thus, the final compressor stage is the most critical one. Dissolved oil will fall out when the gas is expanded in the pressure reducer of a CNG vehicle. When condensation occurs, it is to be expected that the mineral oil will be accompanied by higher hydrocarbons which have been present in the gas by nature. Typically, such a condensate will be composed of 50 % oil and 50 % higher hydrocarbons[7].

*There are a number of factors that affect how much oil is entrained with the gas at compression; a majority of these are described here.*

The higher the temperature the more the oil is in vapour form or dissolved in the gas. At higher pressures (above the critical pressure of about 50 bar) the saturation point for dissolved oil in the gas increases with increasing pressures.

[4] M Czachorski et al, NGV Fueling Station Compressor Oil Carryover Measurement and Control, GRI-95/0483, Final Report, Institute of Gas Technology, February 1996

[5] R. Forster, Verfahrenstechnische Aspekte zum störungsfreien Betrieb von Erdgastankstellen, gwf-Gas/Erdgas 149 [2008], 2, pp. 100-105

[6] Gas Quality Survey of German Natural Gas Fuelling Stations, Rüdiger Forster, International Gas Union research Conference, Paris 2008

[7] R. Forster, Verfahrenstechnische Aspekte zum störungsfreien Betrieb von Erdgastankstellen, gwf-Gas/Erdgas 149 [2008], 2, pp. 100-105

The solubility of smaller oil aerosols depends on the residence time in the gas. Theoretically, they can dissolve up to saturation of the supercritical gas. This means that the quicker and more efficient the aerosols are removed, the less time there is for aerosols to dissolve in the gas, which means less dissolved oil in the gas.

High flow rate results in high turbulence which contributes to the formation of smaller aerosols. The smaller the aerosols are, the easier they transition to vapour or dissolve in the gas. Large pressure pulsations can also give rise to similar effect.

Oil type has a major impact on the amount of oil available as vapour and how much is dissolved in the gas. Mineral oil and synthetic oil of hydrocarbon base with an affinity for CNG has a high saturation limit in the supercritical CNG. Synthetic oils such as polyglycol or polyalkylene glycol (PAG) based ones, have very low vapour pressure and dissolves only in smaller amounts in the supercritical gas.

Oil in aerosol form can be separated and removed in coalescing filters. The oil dissolved in the gas or available as vapour cannot be removed with such filters, thus gas temperature or pressures must first drop, so that the oil precipitates as aerosol. To finally remove oil absorbed or dissolved in the gas adsorption filters are needed.

## 2.2. Water

This chapter is based on excerpts from the published article: *Moisture Measurement in Natural Gas*, Rolf Kolass, Michell Instruments GmbH, Friedrichsdorf, Germany; Chris Parker, Michell Instruments Ltd, Cambridge, UK.

Natural gas extracted from underground sources is saturated with liquid water and heavier molecular weight hydrocarbon components. In order to meet the requirements for a clean and dry gaseous fuel suitable for transmission and distribution through pipelines and finally utilized for burning by end users, the gas must go through several stages of processing, including the removal of entrained liquids from the gas, followed by drying to reduce water vapour content. The dehydration of natural gas is critical to the successful operation of the production facility and the whole distribution chain through to the end user. The presence of water vapour in concentrations above a few 10s of parts per million has potentially disastrous consequences. The lifetime of a pipeline is governed by the rate at which corrosion occurs which is directly linked to the available moisture in the gas which promotes oxidation. In addition, the formation of hydrates can reduce pipeline flow capacities, even leading to blockages, and potential damage to process filters, valves and compressors. Such hydrates are the combination of excessive water vapour with liquid hydrocarbons, which may precipitate during transmission, forming emulsions that, under process pressure conditions, become solid masses. Furthermore, in the processing of gas prior to transmission, a cold temperature separator is most often used to extract the heavier molecular components to avoid the formation of such hydrocarbon liquid condensates at prevailing pipeline operating temperatures that change with climate. The drying of natural gas to a dew point lower than the operating temperature of the chiller plant is of obvious importance to prevent freeze up problems, causing flow restriction, with resulting consequences in terms of plant operating efficiency.

For these reasons it is standard practice at natural gas production facilities, both on- and offshore, to measure the moisture content in natural gas on a continuous, on-line basis at critical points to ensure successful processing and efficient, reliable plant operation. The successful design, installation and operation of industrial hygrometers for such applications require special consideration to be given to the particular nature and composition of the gas being measured and the processing techniques being utilised.

As mentioned above, direct interaction of water with other substances, such as carbon dioxide and hydrogen sulfide, may cause strong acids formation. Additionally, vehicle-related standardization (for its separate components, systems and the complete vehicle) has a primary focus on safety and not on operability/drivability. The ISO 15403 standard gives priority to the regulation of water content, since it is the most important liquid contaminant in the natural gas. In addition to corrosion risk, presence of water can also cause physical damage to the compressors. Water content in natural gas which is equal or less than 30 $mg/m^3$ is supposed to be satisfactory at the ordinary pressures and temperatures. The Swedish standard SS 15 54 38 declares a maximum content of 32 water $mg/m^3$, which at 200 bars corresponds to a dew point of -9 °C. At lower temperatures SS 15 54 38 stipulates that pressurized water dew point must be kept 5 degrees lower than the minimum monthly average daily temperature.

Scandinavian experience, however, shows that a much higher degree of drying is necessary to ensure drivability at lower temperatures. The actors in Scandinavia dry gas down to a dew point as low as -80 °C at 4bar, corresponding to approximately $1mg/Nm^3$. In practical experiments conducted by Stockholm Water Company, showed that the limit for winter drivability at low temperatures is about -25 °C (200 bar), and completely disappears at a water dew point of -30 °C, corresponding to approximately 2-3 $mg/m^3$.

## 2.3. Sulfur

This chapter is based on a translated excerpt from the published report: Svensson M (2011), ”SGC Rapport 229 Utvärdering av svensk biogasstandard - underlag för en framtida revision”.

Total amount of sulfur is the sum of all sulfur containing compounds in the gas, and recalculated to the amount of elemental sulfur contribution. Sulfur is corrosive even in the absence of water. Hydrogen sulfide is the sulfur compound that predominates in untreated biogas, and is generated from anaerobic microbial decomposition of sulphates and sulfur-containing organic compounds which occur to a greater extent in products of animal origin.

The second most common group of sulfur compound are mercaptans, which may also be added to achieve sufficient odorisation of the gas (natural gas smellable at 1% concentration in air). The biogenic mercaptans are often of lower molecular weight, such as methyl mercaptan. These may be at levels higher than 1 ppmV significantly impairing the quality of odorisation (GTI 2009). For instance, in a leaflet from the Stockholm Water Company it is mentioned that the sulfur content, including THT (Tetrahydrothiophene, $C_4H_8S$), is in the range 4-7 $mg/Nm^3$, and sulfur content excluding THT is typically less than 0.5 $mg/Nm^3$.[8]

The sulfur compounds in sulfur based odorants are typically a mixture of different mercaptans or sulfides. The substances typically present in an odorant are:
IPM Isopropylmercaptan, DMS Dimethylsulfide, NPM Normal propyl mercaptan, MES Methylethylsulfide, TBM Tertiary butylmercaptan, THT tetrahydrothiophene and EM ethyl mercaptan.

The sulfur odorant provides a characteristic odour that becomes associated with gas leakage. In some countries gas companies send out so called "sniff cards" to its customers to be sure that they recognize the smell.

Example of country that mainly uses THT is Germany, Belgium, Italy and the Netherlands. The U.S.A. favour mercaptans, especially IPM and TBM, while France uses both THT and mercaptans. UK utilizes an odorant designated as BE, a mixture of diethyl sulfide (DES). As a rule, THT produces a higher total sulfur concentration compared to the others in order to reach the same level of odorization.

There exist sulfur free odorant alternatives. The sulfur-free mixture S-FREE, composed of methyl acrylate, ethyl acrylate and 2-ethyl-3-methylpyrazine, is commercially available and has been tested with successful results in Germany, but suffer from the disadvantage that it

[8] Stockholm Vatten 2007: “Varuinformation Biogas”

is allergenic and potentially carcinogenic. The smell is also uncharacteristic, requiring information campaigns if introduced.

Odorants are normally added in Measurement and Regulation stations, i.e. the input point of distribution. Central odorisation is cost-advantageous compared to odorisation in several points downstream but obviously means less flexibility. One disadvantage of the central odorisation is that some industrial consumers, who use natural gas in their processes, must remove the odorant before the natural gas can be used. Examples of such industries are industries where natural gas is used as feedstock for chemicals or fuels. Such is the case if catalysts are used in the processes, which would be poisoned by sulfur.

In addition to the increased risk of corrosion, sulfur may also affect on-board vehicle aftertreatment equipment by catalyst poisoning, which mainly affects the methane oxidizing capacity of palladium. Sulfur levels are not greater than 5.5mg S/Nm$^3$ with addition of 15mg THT/Nm$^3$. The lower temperature of the exhaust of gas-powered lean-burn engines makes its aftertreatment the most sensitive. Stoichiometric gas engines have higher exhaust gas temperature, and are therefore less sensitive to sulfur based deactivation.

A content of sulfur dioxide in the exhaust gas as low as 1ppmM, can lead to inhibitory activity (Ly 2002). Even motor oil can be a source of sulfur dioxide in the exhaust, especially at idling mode. Lampert and Farrauto (1997) produced data on how much the sulfur content of natural gas and engine oil contributed to the sulfur content in the exhaust gases of a natural gas-powered lean-burn engine.

*Table 1 Share of sulfur (ppmM) in the exhaust gases from natural gas and engine oil (0.4% v / v) at various operating conditions and sulfur contents in lean-burn engines (Lampert and Farrauto 1997). Assumed density of natural gas: 0.755 kg/Nm$^3$*

| *Compound* | *Max S level (ppmM)* | *The average S level (ppmM)* |
|---|---|---|
| *In the CNG* | *30 (22.65 mg S/Nm3)* | *12 (9.06 mg S/Nm3)* |
| *The exhaust at idle:* | *2.6* | *1.6* |
| *CNG* | *1.7 (65%)* | *0.7 (44%)* |
| *Engine oil* | *0.9 (35%)* | *0.9 (56%)* |
| *The exhaust at cruising speed:* | *1.3* | *0.6* |
| *CNG* | *1.2 (92%)* | *0.5 (83%)* |
| *Engine oil* | *0.1 (8%)* | *0.1 (7%)* |

The data in the table show that max content of 23 mg S/Nm$^3$ used in many standards leads to inhibitory levels of $SO_2$ at all operating conditions, while the level reported by the Stockholm Water Company would lead to inhibition only at idle. Use of a more low-sulfur lubricating oil could reduce the aging effect at idling below the inhibitory level of 1 ppmM.

### 2.3.1. German experiences with sulfur

This subchapter is an excerpt from: Entschwefelung in Erdgastankstellen, Seminar „Erdgastankstellen“, 17. Juni 2010 in Celle, Dipl.-Ing Hans-Jürgen Schollmeyer, E.ON Ruhrgas AG

Quality requirements for natural gas as fuel in Germany are regulated under the umbrella of the Tenth Ordinance of the Federal Pollution Control Act (as amended on 27 January 2009). The new limits on the sulfur level apply to the fuel at the transfer point on the vehicle to be refuelled, according to DIN 51624.

Total allowed sulfur content in Germany until 31$^{st}$ December 2008 was 20 mg/m$^3$, compared to G260 where the limit was set to 30 mg/m$^3$, while since 1$^{st}$ of January 2009 DIN 51624 standard proscribed limitation to the content of sulfur in natural gas at 10 mg/m$^3$.

Requirements of the DIN standard are clear – sulfur levels in natural gas have to be aligned with those of liquid fuels.

According to the regulations, from January 1st 2009, petrol and diesel fuels may be in trade and sold to the consumer, if their sulfur content does not exceed 10 milligrams per kilogram. There is no room for negotiation for the gas industry in this regard.

The bulk of the sulfur content of natural gas is made up of the following sulfur compounds:

- Carbon oxide sulfide (COS)
- Hydrogen sulfide (H2S)
- Mercaptans (traces)

} The total sum according to G 260 is limited to 30 mg/N $m^3$ or about 40 mg/kg

On the final distribution level, natural gas must be odorized with mainly sulfur-containing substances. The important odorants are:

- Tetrahydrothiophene (THT)
- Mercaptane (BuSH, PrSH)
- Gasodor S-Free

Currently in Germany natural gas is odorized to 70% with THT and about 25% with S-Free.

*Table 2 Overview of natural gas odorants*

| ***Odorant*** | ***Minimum concentration (DVGW G 280)*** | ***Practice values odorant in mg / m³*** | ***S content in%*** | ***S-content odorants in Gas in mg (S) / m³*** |
|---|---|---|---|---|
| *THT* | *10 mg/m3* | *15 - 18* | *36,4* | *5,5 – 6,5 (7-8 mg S/kg)* |
| *Mercaptane* | *3,8 mg/m3* | *5 - 8* | *36 - 37* | *1,8 – 3,0 (2,5-4 mg S/kg)* |
| *Gasodor™ S-Free™* | | *13 - 16* | *0* | *0* |

The predominant sulfur content in CNG comes in most supply areas from the odorants.

From the perspective of the producers, the problem of too high sulfur content in certain domestic sources of natural gas is perceived as being of a transient nature.

However, development of commercially feasible technical measures for on-site desulfurization at refuelling station level has proven to be more difficult than expected.

Even though DIN 51624 proscribes a maximum amount of sulfur in natural gas, the results from the filed test show that it is almost impossible to ensure this compliance at every CNG station in Germany.

From the German experiences, several measures for sulfur content removal at CNG stations are being set in practice:

- development and use of sulfur filters at natural gas stations
- changes in the odorant application (i.e., reduction of odorant content, and use of low sulfur or sulfur free odorant at the gas station where possible)

As the final consequence for non-compliance with DIN 51624, Germany is considering closure of certain CNG stations.

## 2.4. European natural gas quality standardization: Who plays a role?

The material for this chapter is provided by the courtesy of Jaime Alamo form NGVA Europe and his presentation "NG/Biomethane Fuel", UNECE GFV Informal Group, Brussels, 12th September 2013.

Even if the European market has been using natural gas for many years and for many different applications, there is still a lack of harmonization regarding a vital aspect: natural gas quality. Here we have to make a distinction between the natural gas grid specification, and the potential specifications for specific applications like the use as CNG.

For many years the only requirements on natural gas quality/composition have been set for the natural gas grids and at national levels in order to assure its proper use for different applications such as home heating or power generation.

Those requirements mainly comprised parameters such as:

- Wobbe Index
- Heating Value
- Relative Density
- Oxygen
- Hydrogen
- Carbon Dioxide
- Water
- Total Sulfur
- Hydrogen Sulfide (+ Carbonyl Sulfide)
- Dust and particles

Regarding its use as a fuel, only a few countries developed their own national standards setting specifications for NG and biomethane when used as vehicle fuels. All in all the requirements where similar to those for the grid but slightly more detailed/restrictive when it came to certain components such as heating value, water content, methane number, hydrogen, dust and particles, and compressor oil.

Due to the lack of harmonization on the specifications reported above this line, the European Commission recently addressed the need to cover those gaps, and addressed two separate Mandates to the European Committee for Standardization (CEN) to:

Mandate M/400: after a certain interoperability project, the CEN should develop a European Standard(s) for H-gas qualities → taken on-board by CEN/TC 234/WG 11

Mandate M/475: CEN should develop:

- A European Standard for a quality specification for natural gas and biomethane to be used as fuels for vehicle engines → taken on-board by CEN/TC 408/EG 2
- A Technical Specification or European Norm for a quality specification for biomethane to be injected into natural gas pipelines CEN/TC 408/EG 3

CEN's task force has the straight forward mission; to clarify to what extent can the grid and fuel specifications differ from each other?

In order to properly address this mission, several key parameters are currently under discussion as follows.

**Water content/ dew point:** both parameters are important for safety (corrosion) and driveability (potential water condensation). As these two parameters are correlated (ISO 18453), limiting one of those should be enough.

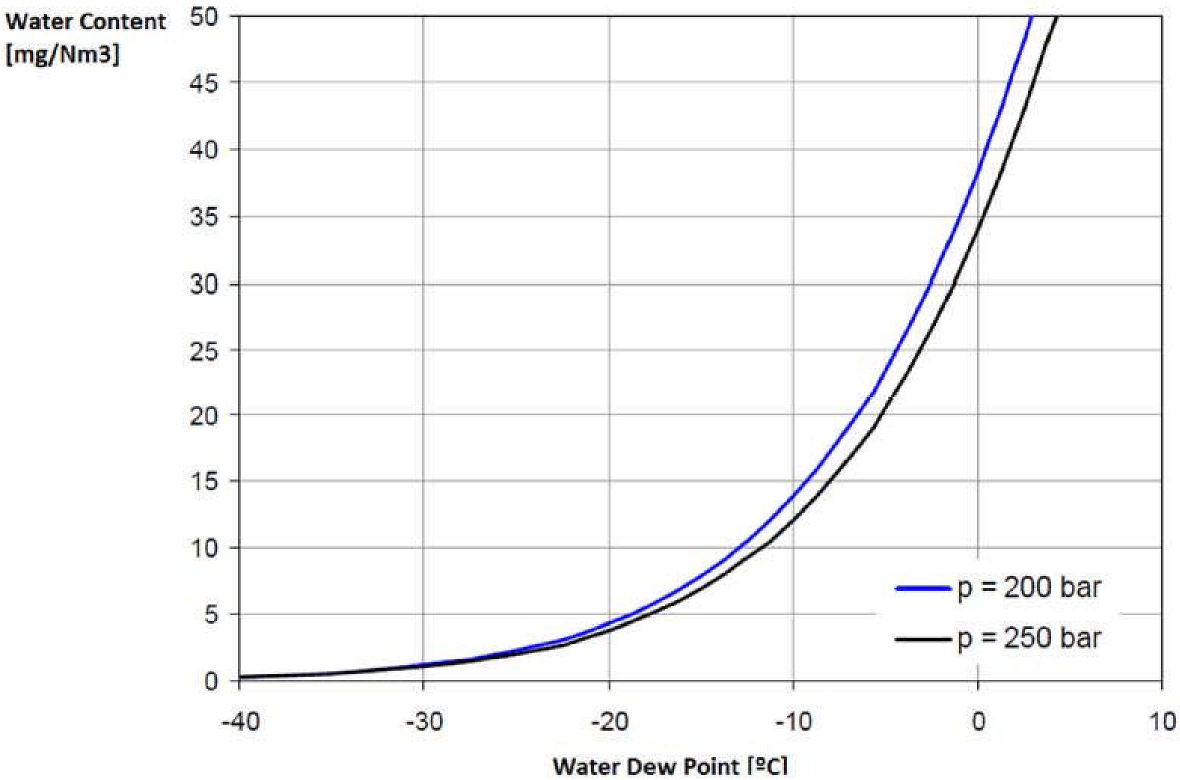



*Figure 2 Water content vs dew point characteristics in natural gas*

ECE Regulation 110 sets a 32 mg/m$^3$ limit just for safety. It has proven to not be enough for cold climates. Thus a variable limit for different climatic zones is being proposed:

- Zone A: -10°C at 200 bar
- Zone B: -20°C at 200 bar
- Zone C: -30°C at 200 bar

This implies that, for many stations, there will have to be drying equipment embedded on to facilities.

**Hydrogen Sulfide + Carbonyl Sulfide:** associated corrosive issues and combustion by-products sticking engine valves.

ECE Regulation 110 sets a limit of 23 mg/m$^3$ for safety/ corrosion. Several documents have been shared by different experts. **A proposed limitation of 5 mg/Nm$^3$** is currently being proposed, which is in line with the gas grid requirement being developed by CEN/TC 234/WG 11.

*Figure 3 Corrosive effects of Hydrogen Sulfide and Carbonyl Sulfide on metal pieces of CNG equipment*

**Sulfur:** poisoning effect on after-treatment systems. Problem is that vehicle manufacturers request 10 mg/m$^3$ contaminant cap value that can't be assured nowadays by the gas industry.

The main reason lays on the current odorisation practices, which are typically based on the addition of sulfur-based components to the pipeline, like THT and mercaptanes.

Different requests can be found between different manufacturers as the engine technology and thus the after-treatment used have a great impact on the sulfur sensitivity.

At the same time, reports from the NG industry reflect that typical levels are generally below 30 mg/Nm$^3$, so the limitation they are proposing is:

- 20 mg/Nm$^3$ for non-odorised NG
- 30 mg/Nm$^3$ for odorised NG (indicative value; national rules apply)

# 3. DEFINITION OF VALUE CHAIN CONTRIBUTING TO THE INTRODUCTION OF CONTAMINANTS IN CNG

Starting point when assessing the elements that are affecting the quality of CNG is to get a better understanding of the CNG Value Chain, through which natural gas is transmitted, compressed, treated and delivered to the NGVs, and also how particular contaminants (oil, water and sulfur) find their way into the gas at each stage.

The CNG value chain is made up of five consecutive blocks:

1. Distribution network
2. CNG Compressor
3. CNG Treatment Facility
4. Dispenser
5. Customer (NGV users)

Aforementioned blocks of the value chain are grouped in the three stakeholder groups:

1. Supplier (of natural gas)
2. CNG Refuelling Station Operator
3. Customer (NGV user, NGV fleet operator)

In the focus of a CNG quality assessment is certainly the final user (NGV user in this case), the party establishing the requirements needing to be met. Apart from the listed three groups of stakeholders, an additional two have been analysed in this study: CNG Equipment Producers and NGV OEMs.

As it was described in the first chapter of this study, there are three major contaminants that are affecting the quality of CNG at refuelling station level: oil, water and sulfur. The levels of every single contaminant in the CNG Value Chain depend on the addition of them in each particular block. The largest changes in the content of contaminants in natural gas occur in CNG Compressors and CNG Treatment Facilities.

It has to be stressed that the greatest responsibility for the ultimate CNG quality is in the hands of CNG Refuelling Station Operators. Consequently, this stakeholder group will be further analysed in detail.

The possibilities of changing the quality of natural gas that is entering from the connecting distribution system into the process units of the CNG Refuelling Station Operator are quite limited but still very important.

Dynamics of oil introduction in the CNG Value Chain

It has to be stressed that once natural gas is processed in a CNG refuelling station compressor, its initial level of oil will increase as oil and aerosols released from the compressor's lubrication fluid is added. Thus, the CNG Treatment Facility at refuelling station level plays a significant role in controlling the oil content of CNG. After CNG is treated and delivered to the dispenser, it is filled into NGV storage tanks. Generally it is additionally purified by on-board CNG filters, with regards to oil and particles.

Dynamics of sulfur introduction in the CNG Value Chain

In contrast to oil, the sulfur level of the gas is, besides the addition of odorant, independent of the distribution system. Thus, the level of sulfur is defined by the natural gas quality, and the amount of odorization. The level of sulfur can be decreased at the CNG refuelling station if it is appropriately equipped, but this may also lead to the loss of odorization, making reodorization necessary. The costs of introducing such measures have as of yet been prohibitive.

Dynamics of water introduction in the CNG Value Chain

The dynamics of water introduction in the CNG Value Chain is similar to that of sulfur, thus a clear understanding of the mechanisms that affects these levels are of vital importance for effective water removal in CNG.

**Figure 4** qualitatively depicts the dynamics of the contaminants, Illustrations intentionally exaggerated.

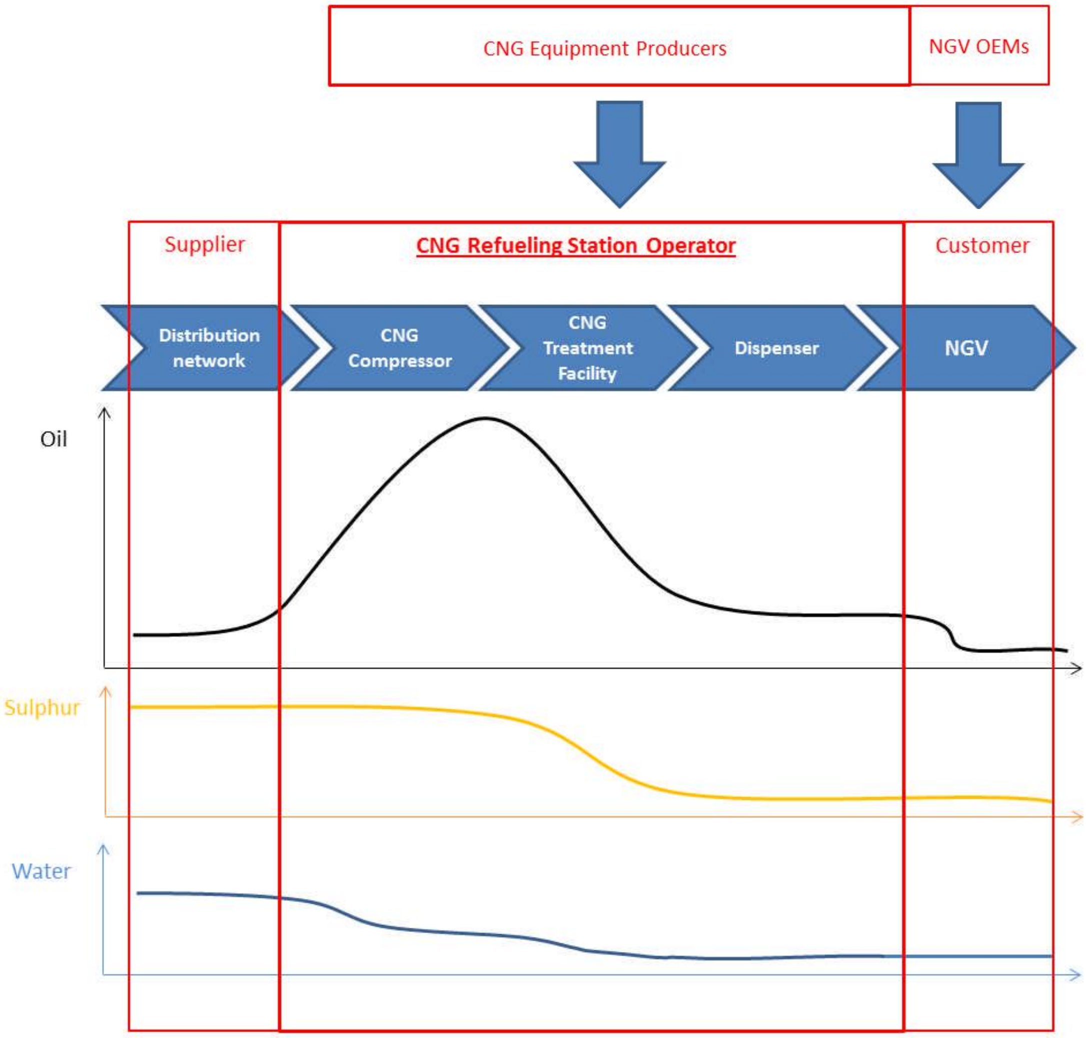



*Figure 4 Contaminants appearance through the CNG Value Chain at the refuelling station level*

# 4. REQUIREMENTS ON THE CONTENT OF OIL IN CNG

This section summarizes the requirements imposed on CNG regarding its content of oil, and according to the international regulatory framework and among vehicle manufacturer requirements. It is based in its entirety on excerpts from the published report: Clementsson M, Held J (2007). “Olja i fordonsgas – regelverk, mätmetoder och filter” non-public SGC Report, 34 p.

## 4.1. International and national standardization

*ISO 15403: Natural gas – Natural gas for use as a compressed fuel for vehicles*
*Part 1: Designation of the quality (061015), Part 2: Specification of the quality (060815).*

The ISO standard 15403 defines quality of the compressed natural gas used in vehicles. Part one of the standard states that the oil content shall not affect the safe operation of the vehicle. To ensure this, it states that it may be necessary to use on board filters or other equipment for the separation of oil, while simultaneously underlining that a small amount of oil can be useful for the protection of tanks and lubrication of injectors.

The standard also provides test methods that can be used to verify that the requirements of the gas composition are met. For oil and particles, there are no test methods.

In part two of the standard, the technical report, and quantitative data on gas quality are provided. As in part one, it is indicated that it has been documented in studies that some oil content in the gas is advantageous, if not essential, to lubricate the fuel system. It is further stated that high concentrations of oil in the gas can be detrimental because parts of the fuel system can be blocked and oil precipitated in the storage tank.

The technical report does not specify a minimum or maximum limit for the oil content of the gas. However, it is stated in the technical report that long-term OEM experience suggests a level of 70-200 $ppm_v$.

According to the technical report, the operators of refuelling stations should check the consumption of oil in the compressor. It is also important that the operator regularly maintains the filters for cleaning and drying, which may accumulate both water and oil.

*UN-ECE R110*

The standard does not specify any restrictions on the oil content in the gas. Under the section gas composition it is stated that if so-called wet gas (> 23 mg water vapour per $m^3$) is used in the vehicle, it is necessary to have at least 1 mg per kg of gas oil (1 $ppm_m$) to protect the CNG cylinders.

*SAE J1616 Recommended Practice for Compressed Natural Gas Vehicle Fuel*

In this recommended practice from SAE, Society of Automotive Engineers International highlighted the problem of oil in the compressed natural gas. It is reported that lubricating oil from the compressors in the gas grid or from compressors at gas stations often occurs as a trace compound in the gas. It is stated that high concentrations of oil in the gas in form of aerosols or vapour phase is not acceptable, and that the oil can condense and cause operational problems in the vehicle, for example by precipitation in the regulator. Furthermore, more information is requested on normal levels of oil, in order to establish reasonable levels of an acceptable content of oil in the gas and standardized testing to determine the level of oil in the gas. In order to ensure that the oil content in the gas is not too high, careful monitoring of the compressors and the use of coalescing filters after the compressor is highly recommended.

*Draft SAE J1616 – (6/1/04)*
The draft revision of SAE J1616 gives recommendation on the level of oil in the gas and how this should be handled. The final draft is from January 2004, after that the work on the new version was interrupted, and to this date the work has never been reopened.

The additions to the previous version are that it states that the oil burned is the largest source of particulate emissions from gas engines. It is further stated that some fuel injectors require a certain amount of oil to prevent wear. Against this background, it is recommended an oil content of 10-80 $ppm_m$ in the gas. Oil content in gas can be monitored with the method set out in two reports from the Gas Technology Institute, *NGV Fueling Station Compressor Oil Carryover Measurement and Control* and *Validation Testing of a Gravimetric Method of Measure CNG Compressor Oil Carryover*.

Appendix A of the draft states that the normal method to reduce the oil content in the gas is inserting coalescing filters after the compressor. It is important to manage the emptying and maintenance of the filters. Moreover, tests indicate that the carryover of oil is lower when using synthetic lubricating oil, especially polyalkylene glycol (PAG). It is stressed that mineral oils and PAG do not mix, which need to be considered in the case of compressor oil changes.

*DIN 51624 Automotive fuels - natural gas - Requirements and test methods*

The German Institute for Standardization, DIN, adopted in 2008 a standard for compressed natural gas as a fuel for vehicles. The standard gives specification and testing methods to meet these requirements. The standard states that natural gas may be contaminated with oil in particular during compression at refuelling stations. It is stated that:

> *Solid particles exceeding 10 µm in size shall be removed effectively from the gas, e.g. by means of the respective fine filters provided at- the natural gas refuelling station.*
>
> *Compressor oil is not contained in natural gas a priori; it can enter the gas, however, in the form of contaminants, particularly when the gas is compressed, as is the case in refuelling stations, for example. Minimizing the contents of compressor oil and suspended solids is classified as very important by both users and suppliers. This minimization has to be ensured by applying the appropriate measures. Thus, until a suitable test method for establishing a limit value is available, the following principle shall be followed: "The less the better".*

In the draft further information could be found: it was stated that the supplier shall, by adopting various measures at refuelling station, as choosing oil-free compressors, integrating filters and performing regular cleaning and maintenance ensure a low level of oil in the gas. A maximum limit of 5-50 ppmm was suggested as appropriate.

## 4.2. Requirements of vehicle manufacturers

Daimler Chrysler Cars (Mercedes-Benz) indicates that they have no limit on the maximum concentration of oil in the vehicle, but that they require that the gas does not contain any oil at all. Daimler Chrysler has had problems with vehicles such as Mercedes-Benz E200 NGT in Sweden. It has been found that oil has blocked the regulators so they stopped working, or that they shed noise. However, no test has been made of the oil found in the vehicles and so it has not been established whether the oil comes from CNG or not, but this is suspected.

Volvo Car Corporation of Sweden stated that CNG must not contain more than 8 ppm of oil per $Nm^3$. This value is calculated based on gas filter capacity and the current service interval. The corporation has no official list of the problems that oil can result in the gas vehicle. It is however stated that if the gas filter becomes saturated with oil, oil may pass through the filter and affect the gas regulator and the gas manifold, giving drivability problems. Problems with the Volvo V70 may also have been due to an outdated regulator and bad software. Trials have been conducted in which oil sprayed into the manifold and regulator, which nevertheless still worked well.

Scania buses and coaches say they have no recommendation on oil content in vehicle fuel. Furthermore, they consider that they have not had any problems with the vehicles associated with oil in CNG.

Volkswagen reports that they do not have specified limits for maximum/minimum oil content of CNG. Neither Volvo buses have a fixed limit on the content of oil in CNG.

According to the German Industry Association for Gas and Water, DVGW, the German automotive manufacturers and component manufacturers, in connection with the preparation of the standard DIN 51624, has demanded an oil content of vehicle between 5 and 10 ppmM. Examples of German automotive and component manufacturers are Daimler Chrysler, MAN, Volkswagen, Opel and Bosch (injectors).

Most vehicle manufacturers state that they have no specified conditions on the levels of oil in CNG, which is a natural consequence of that there is no clear requirement in the standardization. For those vehicle manufacturers who state requirements, such as Volvo and most of the German players, the requirement lies between 5 and 10 ppmM.

# 5. SAMPLING AND ANALYSIS OF OIL IN NATURAL GAS

This chapter elaborates finding related to methodologies for sampling and analysis of oil water and sulfur in natural gas that is being used as a motion fuel in transportation. Given the fact that there is no standardized method for determination of oil level in natural gas, discussion includes various methods used to determine the level of oil in CNG, both through monitoring of the oil consumption in the compressor and direct measurements of the gas. Since Sweden and Germany are the most advanced countries in this regard, their experiences will be elaborated more in details.

The source material of this chapter is, if not otherwise indicated, based on excerpts from the published report: Clementsson M, Held J (2007). “Olja i fordonsgas – regelverk, mätmetoder och filter” non-public SGC Report, 34 p.

## 5.1. Standard analytical methods for oil content determination pressurized air

There is no standardized method for measuring oil in CNG, but there are standardized methods for oil in air. First, the standard for determining level of oil aerosols in compressed air (ISO 8573-2:2007, Compressed air - part 2: Test methods for oil aerosol content) and the standard for determining oil mist and organic solvents in air (ISO 8573 -5, Compressed air - Part 5: Test methods for oil vapour and organic solvents).

ISO 8573:2-2007 describes both the sampling method, and the quantification of oil aerosols in compressed air. Determination of the compressed content of aerosols can be done by two methods, A or B.

Method A is based on two highly efficient coalescing filters that can be used if contamination is between 10-40 mg /m$^3$ (about 0.8 to 33 ppmM, the air density of 1.2 kg/m$^3$). The oil separated in the first filter is collected, weighed and related to the flow that has passed during the collection period, from which the oil content in milligrams per cubic meter of air can be calculated. The second coalescing filter serves as back-up and verifies that the first filter is working properly. If the second filter contains oil the first filter needs to be replaced. The coalescing filters should both be able to separate the oil to a level downstream of them to a level of 0.01 mg/m$^3$ or less. This method allows you to let the entire flow of air pass through the filter set. Other equipment used in the tests includes temperature gauges, flow meters and pressure gauges to check that the pressure does not go beyond the recommended pressure for the coalescing filters.

Method B should be used if contamination is from 0.001 to 10 mg/m$^3$. Here, three highly efficient membranes of micro-fibre glass are placed in series in a membrane holder. After the air has passed through the membrane during a certain time, the membrane holder is taken out. The oil that has accumulated in the membranes is dissolved with a solvent. The amount of oil is then determined by infrared spectrometry. The method may either be designed so that all air, or just a part flow, crosses the membranes. Next table shows the most important parameters for measuring oil content in aerosols in compressed air.

*Table 3 Methods for measurement of oil aerosol content in pressurized air (Source: ISO 8573-2:2007)*

| Parameter | Method A Full flow | Method B Full flow/part flow |
|---|---|---|
| Level of contamination | 1-40 mg/m$^3$ | 0,001-10 mg/m$^3$ |
| Max. velocity through filter | According to filter manufacturer | 1 m/s |
| Sensitivity | 0,25 mg/m$^3$ | 0,001 mg/m$^3$ |
| Accuracy | +/- 10% of mean | +/- 10% of mean |
| Max. temperature | 100 °C | 40 °C |
| Sample time (typical) | 50 h to 200 h | 2 min to 10 h |
| Filter type | Coalescing filter | Membrane in three layers |

Oil found in vapour phase in air is analysed through the collection in an adsorption material, with subsequent analysis of the gas chromatograph.

## 5.2. Follow-up of oil consumption in the compressor

The most common method used to determine oil levels in the gas of refuelling stations is to monitor the oil consumption in the compressor. Supplied amount of oil to the compressor is measured and removed amount of oil in for example coalescing filters is measured. The oil supplied and the oil removed may be measured as a volume, or be weighed. By taking the difference between input and removed amount of oil, one can calculate the amount of oil entrained in the gas. If you know the volume of CNG (Nm3) that has been processed by the station, the amount of oil per Nm$^3$ of CNG can be calculated.

For better accuracy the added amount of oil and the removed amount of oil may be monitored over a prolonged time period.

An advantage of this method is that oil consumption in the compressor is at any rate necessary to monitor in order to check the compressor. Increased consumption by oil indicates that there is a need for maintenance. The amount of removed oil, waste oil, may need to be documented in the context of the environmental reporting.

Disadvantages of this method are that it has low accuracy, and that it only measures the mean between two sample points. The method will not be able to detect, for example, a larger amount of oil entrained in the CNG at a certain peak event.

## 5.3. Analysis of oil in natural gas – Swedish experiences from 2007

This chapter describes the methods that have been used in Sweden to determine the level of oil in CNG. The material of the chapter reflects the content reported in the Swedish study from 2007.

*A rough estimate of how much oil that comes with the gas can be obtained by taking the difference between supplied oil to the compressor and removed amount of oil in the filter divided by the volume of gas passed.*

*Better accuracy is provided by direct measuring of the oil content in the gas. Of the methods identified in Sweden, the method of precipitation by solvents and coalescing filters is deemed as the most interesting.*

*Samples can also be taken on the gas and sent away for analysis. One of the drawbacks of doing like this is that only small volumes can be collected.*

*Determination of the presence by oil mist or organic solvents in the air can be done either with GC or with a chemical indicator tube. The latter method should be used only as a preliminary method to see if there is oil present, accurate determination is then done with the* *gas chromatograph when the compressed air is containing oil in the level of 0.001 to 10 mg/m$^3$.*

### 5.3.1. Collection of gas samples

SGS Sweden AB has taken out gas samples at refuelling stations for analysis of presence of oil in the gas. Samples were collected at the refuelling area, using the refuelling nozzle and were then sent to Germany or Switzerland for analysis. Two pressure vessels were filled with gas at each refuelling location. In the autumn of 2006, SGS was collecting samples in this manner at refuelling stations from Gothenburg in the north and from Malmö in the south. Which method was used to analyse the samples has not been possible to find out.

### 5.3.2. Measurement of oil content by precipitation with solvents

Saybolt Sweden AB has developed a method to make measurements of oil content in CNG. The method is based on the gas bubbling through a solvent (e.g. pentane, $C_5H_{12}$). The oil content is then determined by gas chromatograph (GC) or mass analytical method. GC is used to characterize what type of oil there is in the gas, but the method can also be used to measure the content of oil. A gravimetric method is used to determine the level of oil in the gas. The solvent is evapourated and the residue can then be determined by weight, this is similar to the method used for determining residual oil in the gas, ISO 13757, 1996.

Saybolt has developed an equipment to collect samples on site at the refuelling station. At the refuelling nozzle a device is connected that can lead out a part flow of gas at a reduced pressure through the solvent and then into the air. Volume measurement is also used to determine how much gas that has crossed the solvent. A test takes about 5-20 minutes. The oil content is then analysed in the laboratory. Analysis personnel from Saybolt go out and conduct the test. The accuracy of this method is not evaluated, but it is believed to be good and it is estimated that oil in the vapour phase is collected.

Also SP Technical Research Institute of Sweden (SP) has conducted tests where in the same manner gas was bubbled through washing bottles with solvents such as pentane or dichloromethane. The oil content was then determined using GC or infrared spectrometry. The pressure of the gas has been reduced to approximately atmospheric pressure before being introduced into one or more washing bottles.

Volumes of gas that has passed the bottles were determined with a gas meter. The suspected risk of this method is that during the lowering of pressure oil may precipitate and not be available for sampling. One advantage of this method is that oil in the vapour phase also should be included.

To get around the problem of oil condensation at pressure reduction, it would be interesting to be able to do the sampling at higher pressures. Swedegas AB, formerly Nova Gas AB, has a device that can measure the presence of heavier hydrocarbon gases. The method is based on a fixed amount of gas going through three washing bottles in series. The gas is entering the bottom of the bottles and goes out in the top of them. The bottles are made of steel. Only after the gas has passed the bottles the pressure is reduced in a regulator for the measurement of gas flow with a bellow based gas meter. Methanol has been used as washing liquid in the bottles. After collection, the bottles are sent for analysis to a laboratory in Norway, SINTEF, along with a blank test of pure methanol. The laboratory determines the presence and concentrations of heavier hydrocarbons. The equipment has been developed by Statoil. Swedegas AB took is using the method for measurements at the Swedish gas storage “Skallen”. The method is considered to be accurate and will measure both oil in aerosol and the vapour phase. The disadvantage of this method is that it is relatively expensive and that it is complex to use. Swedegas AB has only used the method at the pressure of 60 bars in the transmission network in Sweden. But the bottles are rated at 200 bars, so they are also suited for measuring in CNG.

### 5.3.3. Measurement of oil content with coalescing filters

Processkontroll AB delivers equipment with measuring filter cartridge for measuring oil content in the gas at refuelling stations. The filter used is a coalescing filter, Balston EU 37/25. The filter is weighed before it is installed and after it has been in place for some time (several weeks or longer). Weight gain indicates how much oil has been collected from the gas. If the amount of gas that crossed the plant during the same time is known, a ratio can be calculated on the amount of oil contained in the gas. A disadvantage of this method is that it only captures the oil vapours. The filter should be collecting 99.97% of the oil in aerosol form.

### 5.3.4. Measurement by oil content through collection on glass fibre filters

SP has also conducted tests in which the pressure of the gas is lowered through a vent, so the gas can then be passed through a fiberglass filter. The filter can then be weighed or alternatively a solvent can be used to extract the oil that has accumulated for further analysis by GC or IR. Disadvantage of this method is that it only collects oil aerosols, no oil in the vapour phase.

### 5.3.5. Mirror instruments

In connection to the measurements performed at the gas storage “Skallen” by Swedegas AB, known at the time as Nova Naturgas, the Danish company Energinet was there and measured with a mirror based instrument. A chilled mirror is used to determine the hydrocarbon dew point in the gas[9].

Chandler Instruments sells such instruments; they are called hydrocarbon dew point analysers. Instruments for both on-line measurement, as well as instruments for manual measurements are available[10]. The gas is transmitted through a mirror that is cooled, for example with carbon dioxide.

Based on the information that has been found in the method, it is considered that it does determine the hydrocarbon dew point of the gas, but not inclusive of the content of heavier hydrocarbons, so the method is not directly relevant to be used for determination of oil in CNG. The heavier the hydrocarbons, the higher the hydrocarbon dew point.

### 5.3.6. Indicator tubes

Draeger tubes are designed for short-term measurement and analysis of various substances. The tubes are also developed for the analysis of oil. There are two types of tubes for oil, one for the measuring range from 0.1 to 1 mg/Nm$^3$ (Oil 10/a-P) and one for the range of 1-10 mg/Nm$^3$ (Oil Mist 1/a)[11].

[9] The hydrocarbon dew point is the temperature when hydrocarbons in a hydrocarbon-rich gas begin to condense, at a given pressure. In addition to pressure the hydrocarbon dew point also depends on the gas composition.
[10] Hydrocarbon Dew Point Analyzers, www.chandlerengineering.com
[11] www.draeger.se, Dräger Short Team Tubes in alphabetical order

Measurement with the first type of these tubes was done at a refuelling station[12]. A part flow of the gas was led through a volume meter and then through the Draeger tube. The tube should change colour in the presence of oil, but gave no response. A check-up was made with Draeger and it turned out that the tubes are not recommended for measurement of oil in methane. No other tube was recommended as a replacement.

*Table 4 Qualitative evaluation of different oil measurement techniques*

| ***Oil measuring technique*** | ***Identification oil in aerosol*** | ***Identification oil in vapour*** | ***Ease of implementation*** | ***Continuous measurement*** |
|---|---|---|---|---|
| *Precipitation with solvents* | + | + | + | - |
| *Coalescing filters* | - | + | + | + |
| *Glass fibre filters* | + | - | + | + |
| *Mirror instruments* | +/- | +/- | + | + |
| *Indicator tubes* | - | - | + | - |

[12] Carl-Magnus Pettersson, Svensk Växtkraft AB, telephone conversation 10 July, 2007, telefon 021 - 4480530.

## 5.4. Analysis of contaminants in natural gas – Experiences from the USA

Institute of Gas Technology conducted in 1998 an evaluation of a **gravimetric method** for measuring the amount of oil in the compressed natural gas[13]. The idea of the method was that it would be practical and cost-effective to use in the field. The material of the chapter reflects the content reported in the Swedish study from 2007.

The method was checked against the method previously developed by GTI with the separation of liquid and gas through a cryogenic trap[14] (Figure 5). The method of a cryogenic trap was in the report specified to be too expensive and complicated to be used in the field. However, the cryogenic trap tests showed that coalescing filters effectively removed all the oil in aerosol form, with only oil in the vapour phase remaining, not affected by the coalescing filters. To also test the function of adsorption filtration, when mineral oil was used as a lubricant, such a filter was installed after the two coalescing filters. The filter was from Pneumatech and consisted of aluminium oxide. The filter reduced the total oil content from 37 to 3 ppm, of this oil in vapour phase was reduced from 23 to 2 ppm. **The tests indicate that adsorption filtration together with coalescing filters can effectively reduce the level of mineral oil in the compressed natural gas.**

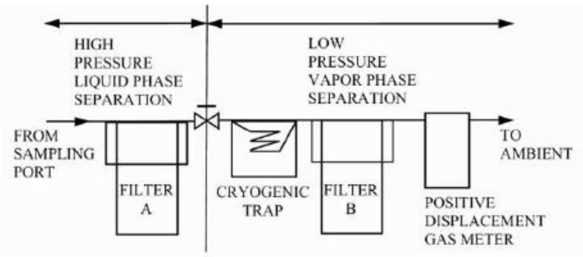



*Figure 5 The collection method used by GTI for the collection of oil in vehicle both in the form of aerosols and vapour phase.*[15]

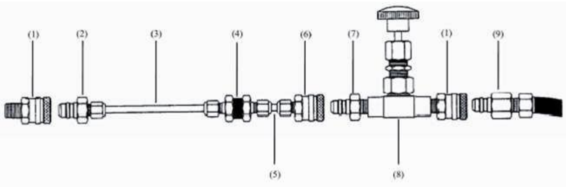
The gravimetric method equipment consists of a stainless steel tube packed with adsorption material. The material used in the experiments was the brand Chromasorb ® P NAW and this is available through suppliers of products to gas chromatographs. A schematic diagram of the equipment is shown in Figure 6.

During capture the collection device is connected to a high pressure pipe with gas and a known volume of gas is passed through the tube. Water and oil are collected by the adsorption material in the tube. The difference in tube weight before and after the collection is equal to the amount of water and oil collected. By heating the material the water can be separated out, and the dry weight gain related to the collected amount of oil.

In the experiments tested how well the weight of analytical method measured the presence of oil in the gas, compared with measurements with GTI-method. Trials were conducted with both mineral oil and synthetic oil. The samples were taken at the gas downstream of the second coalescing filter. The quantity that passed the gravimetric equipment was about 0.3 $Nm^3$ and about 0.5 to 1.5 $m^3$ with the GTI method.

Data collection time was about 30 and 45 minutes, respectively. In each instance, three samples were taken.

[13] M. Czachorski, R. Kina, Validation Testing of a Gravimetric Method to Measure CNG Compressor Oil Carryover, GRI-98/0228, Institute of Gas Technology, June 1998.
[14] M Czachorski et al, NGV Fueling Station Compressor Oil Carryover Measurement and Control, GRI-95/0483, Final Report, Institute of Gas Technology, February 1996.
[15] Filters A and B are highly efficient coalescing filters (Ref: GRI-95/0483)




| Number | Swagelok®Number | Description |
|---|---|---|
| (1) | SS-QF4-B-4PM | 1/4" MPT Quick Connect Body (2) |
| (2) | SS-QF4-S-400 | 1/4" Swagelok® Quick Connect Stem |
| (3) | | 6" long x 1/4" O.D. x 0.035" wall Seamless Tube |
| (4) | SS-4FWS-0.5 | In-line Filter, 0.5 micron |
| (5) | SS-401-PC | 1/4" Port Connector |
| (6) | SS-QF4-B-400 | 1/4" Swagelok® Quick Connect Body |
| (7) | SS-QF4-S-4PM | 1/4" MPT Quick Connect Stem |
| (8) | SS-31RF4 | 1/4" FPT Metering Valve |
| (9) | SS-QF4-S-4PF | 1/4" FPT Quick Connect Stem |

*Figure 6 Gravimetric collection device for oil (Ref. GRI-98/0228)*

With the synthetic lubricating oil is detected an oil content of 12.6 to 16.1 ppmM with the weight of analytical method, the mean was 14.9 ppmM. This was significantly higher than with the GTI, which indicated 2 to 3.1 ppmM oil in the gas with an average of 2.7 ppmM. The difference between the methods of about 12 ppmM may be due to other substances from natural gas, as heavier hydrocarbons or moisture, accumulated in the pipe of the gravimetric method. If there was still moisture in the tube, it shows that the heating process for removing water did not work correctly.

For mineral oil, the gravimetric method detected 8-32 ppmM oil in the gas, with a mean of 19 ppmM. The method of the GTI got here 27, 25 and 166 ppmM. The last high value is not reasonable and indicates that the sample is contaminated during the collection process, even though the outlet for gas cleaned before the test. The high value is believed to be due to a small drop of oil separating from any surface. Since this problem can arise, it is important that, when the samples do several successive measurements.

For mineral oil, the mean of 19 ppm with the gravimetric method is lower than the mean of 31 ppm with GTI-method. That the weight of analytical method shows a lower value of mineral oil is believed to be due to some of the oil in the vapour phase and the GTI cryogenic trap method is more efficient to capture this than adsorption material in the gravimetric method.

The conclusion of the experiments was that the gravimetric method is relatively practical and cost-effective for testing in the field. The method can be used when there is no need to distinguish if the oil is present as aerosols or in the vapour phase. The method is suitable mainly for use in suspected high levels of oil in the gas. If you are requested to measure differences of a few ppm of oil in the gas, the gravimetric method is less suitable.

The aim of the trials was to see if the gravimetric method could be used as a standard test method. Here, the answer was no. The draft revision of SAE J1616 from 2004 choose to mention the method from GTI with the removal of both aerosols and vapours with a cryogenic trap as a method to monitor the oil content in the gas, although the method because of its complexity and cost is not suitable for field trials.

In an evaluation of refuelling stations in California in 1999 about 40 owners of refuelling stations were contacted[16].

In a report from California it was indicated, just as the report from the Gas Technology Institute did, that properly chosen, well-maintained compressor and synthetic lubricating oils with effective coalescing filters solve the problems related to oil in the compressed gas.

[16] Evaluation of Compressed Natural Gas (CNG) Fueling Systems, Consultant report, California Energy Comission, October 1999

## 5.5. Analysis of oil in natural gas – Development of new sampling and test method in Sweden

*Results presented her reflect interim results. For final results, please consult "SGC Rapport 290 Development and validation of methods for test of CNG quality inclusive of oil carryover (Utveckling och validering av testmetoder för test av fordonsgaskvalitet, inklusive oljeförekomst)" Karine Arrhenius, Haleh Yaghooby, Per Klockar 2013*

During 2012 and 2013 SP Technical Research Institute of Sweden has conducted a project that has opted to develop and test a device to sample CNG so that reliable results can be obtained for defining the gas quality in terms of oil carryover, concentrations of water vapour, hydrogen sulfide, carbon dioxide, oxygen, nitrogen, siloxanes, heavier hydrocarbons in the gas phase, other sulfur substances etc.

*Figure 7 CNG Sampling Device*

The project was implemented in eight steps:
1) Oil Composition. Inventory of oils and possibly other liquids, which may come in contact with the gas (their composition and physical properties). Selection of a few typical oils for the rest of the project
2) Examine the possibility to produce a gas mixture (mainly consisting of methane) containing known amount of oil under a specific pressure
3) Control if the proposed sample media are suitable. Determine the flow rate and sampling time to achieve an appropriate detection limit.
4) Modify the existing mobile equipment to include a collection device for oil at high pressure
5) Develop a method to clean the device before and after sampling in order to avoid carryover from one sample to the next sample.
6) Evaluate the determination of water vapour, hydrogen sulfide and ammonia with laser spectroscopy
7) Test the device on site
8) Summarize the results and provide recommendations

Samples of the most common oils were collected by contacting operators of refuelling stations. An analytical method to quantify the amount of oil present in a solvent was developed. The method allows the determination of a specific oil type or of several oils.

In the next step, two sample media (Chromosorb, an adsorbent, and coalescing filters) were studied in order to control that the oil background on the media was negligible and in order to control that a spiked amount of oil could quantitatively be recovered by following the extraction procedure and the analysis.

Before the sampling methods were tested on site, requirements were listed to ensure a quantitative sampling: two sample media shall be used in series, the amount of oil on the second media (called backup) shall not exceed 10% of the amount of oil on the first media and sampling shall be easy to reproduce under the same conditions from station to station.

The principle of the sampling is that the pressure is reduced on the sample media, resulting in the simultaneous decrease of the temperature and adsorption of oil on the sample media.

Three sampling lines with coalescing filters have been tested. The first sampling line has a manual valve in order to reduce the flow, followed by the two filters and the gas was released in atmosphere. The results obtained with this sampling line were promising. In several tests, oil on the backup filter was found to be less than less than 10% of the oil recovered on the first filter. As high as 68 ppm oil carryover were then measured. But the sampling was difficult to perform as the dispenser stopped several times to test for leak. Because of the sampling line, the condition in term of pressure for these tests (the pressure drops to less than 250 bars) was not fulfilled. Moreover, the sampling was different from place to place as dispensers have different electronics.

To overcome these problems, the sampling line was completed at the end by an empty tank (in order to facilitate the leak tests). But in all the tests, more than 10% (often reaching up to 50-70%) of the oil was found on the backup filter. One of the possible explanations for these results was that the flow across the filters was too high to allow the oil to absorb.

The sampling line was then equipped with a part having a hole of 0.8 mm resp. 0.68 mm diameter instead of the manual valve. This allows the flow to be reduced and the sampling to be controlled. But in this case, the amount of oil on the second filter was also found to be more than 10%.

Tests with Chromosorb adsorbent have shown that the adsorbent is packed to a plug by the high pressure.

Two main obstacles have been identified that must be solved for the next testing round:

1. The flow across the filters should be reduced even more than it has been done to date. There is a lowest flow allowed by the dispenser (that may be different from one dispenser to the other) that may not be low enough for the oil to be efficiently absorbed on the filters
2. The leak tests that are performed in the beginning of the refuelling stop the delivery of the gas which create problem for the sampling.

The next step is consequently to sample the gas for oil carryover determination before the dispenser in order to avoid the problem with the limited lowest flow and leak test of the dispenser equipment.

Different flows and sampling times (consequently different sampled volume) shall be tested in order to define the most appropriate conditions to fulfil the requirements for a quantitative determination of oil carryover. A flow measuring device shall be used to set up different flows.

Chromosorb adsorbent shall also been tested again, but the sampling line to test this setup needs to be equipped with a part reducing the flow and the pressure through the adsorbent.

Once the method is developed in term of flows, sampling times and volume, new tests shall be carried out at the dispenser if there is a need for it, and it is possible to rule out any effects of the leak tests performed.

## 5.6. Analysis of oil in natural gas – German experiences

German experiences are here presented based on courtesy of authors of the following report: "*Monitoring CNG Quality In Germany", Frank Graf, Jörg Riedl, Kerstin Kröger, and Rainer Reimert from DVGW-Research Station at Engler-Bunte-Institut, Universität Karlsruhe (TH) and Jörg Meyer from Institut für Mechanische Verfahrenstechnik und Mechanik, Universität Karlsruhe (TH), WGC 2008.*

In Germany, a standard for CNG as motor fuel was elaborated and published as DIN 51624 in 2008[17]. An extract of the most important parameters are presented in the next table. Beside the composition of the main components, especially the methane number, sulfurous components and moisture are limited. According to the 10 BImSchV (Tenth Ordinance for the Implementation of the Federal Emission Control Act (2009)) the CNG quality has to be checked by public authorities. For the sampling of CNG directly from the injection nozzle the DVGW standard G 264 "CNG - Sampling and analysis" was introduced.

Up to now, no limit for the oil and particles content has been fixed in DIN 51624 as no analysis standard is yet available.

To provide a basis for including figures for oil and dust content in the standard for CNG the German Association for Gas and Water (DVGW) initiated two research projects. First, a suitable method for the gravimetric analysis of oil and particle traces in CNG was developed, and second, CNG refuelling stations are monitored Germany-wide. The second project was co-funded by Initiativkreis Erdgas als Kraftstoff (IEK). Both the projects are executed by the DVGW Research Center at Engler-Bunte-Institut in cooperation with Institut für Mechanische Verfahrenstechnik und Mechanik, Universität Karlsruhe (TH).

*Table 5 CNG specification (DIN 51624 (2008))*

| Parameter | Unit | min. value | max. value |
|---|---|---|---|
| $CH_4$ | Mol.-% | 80 | - |
| $C_2$ | Mol.-% | - | 12 |
| $C_3H_8$ | Mol.-% | - | 6 |
| $C_4H_{10}$ | Mol.-% | - | 2 |
| $C_5H_{12}$ | Mol.-% | - | 1 |
| $C_6$ | Mol.-% | - | 0.5 |
| $CO_2 + N_2$ | Mol.-% | - | 15 |
| total sulfur (inclusive odorization) | mg/kg | - | 10 |
| moisture | mg/kg | - | 40 |
| methane number | - | 70 | - |

### 5.6.1. Requirements on the sampling system

To determine the content of liquid hydrocarbons and solid particulates originating from oil carry over and abrasion, a new sampling method based on filtration and gravimetric analysis was developed based on the following requirements:

1. The duration of the sampling should not exceed 30 min
2. The sampling should be accomplishable by one person
3. The sample should be taken integrally over a complete refuelling procedure
4. The interference of the sampling (e.g. pressure gradient) with the refuelling procedure should be minimal

[17] Normenausschuss Materialprüfung (NMP) im DIN, Fachausschuss Mineralöl- und Brennstoffnormung (FAM) des NMP, DIN 51624 Kraftstoffe für Kraftfahrzeuge – Erdgas - Anforderungen und Prüfverfahren, Beuth Verlag, Berlin, Feb. 2008

5. Natural gas born gaseous hydrocarbons should not deposit on the filter

It has to be taken into account that no gaseous hydrocarbons can be analysed with this method thus only liquid and solid matter are captured.

### 5.6.2. Sampling system

The sampling system is modular and consists of the filter system (Module 1, Figure 8) and a tank system (Module 2, Figure 9) which can be used optionally if no CNG car is available when taking the sample.

Module 1 is directly connected to the injection nozzle. To avoid memory effects (e.g. for oil droplets, sulfurous components) the receptacle, which typically consists of a filter element [18] and a check valve, was bored through. Gas samples can be taken upstream and downstream the filter with high pressure gas cylinders. For the sampling of sulfurous components sulfinert®-treated gas cylinders are used.

The duration of sampling averages 30 min, inclusive installing and uninstalling of the apparatus. During the sampling the temperature, the system pressure and the pressure drop in the filter are measured and registered continuously The run of the different parameters depends significantly on the type of CNG refuelling station (compressors, banks etc.). The pressure drop in the filter, that is an important parameter for the filtration efficiency, reaches during the sampling moderate values below 100 mbar. The first results were promising and enable the use of a smaller filter element. Thus the sampling apparatus could be designed compactly (Figure 10).

After the sampling the filter element is removed from the filter housing and weighed in an air conditioned laboratory (20 °C, 40 % relative humidity). For this the filter elements are transported in baggies and stored 24 h in the laboratory before weighing. To quantify the content of gaseous higher hydrocarbons ($C_9$ - $C_{40}$) the gas samples are analysed by GC-FID and GC-HCD. Before the analysis of higher hydrocarbons enrichment in cyclohexane is necessary. Furthermore, the total sulfur content is determined with a high-resolution UV-detector. The quantitative analysis of selected sulfurous components is undertaken with a pulsed flame photometric detector (PFPD). The moisture content is measured with a mobile device (Michell Cermax EX) at the CNG refuelling station.

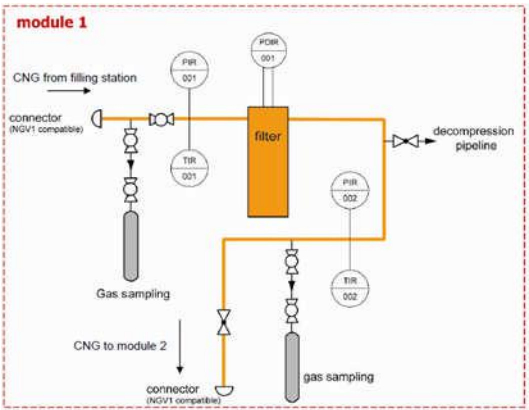



*Figure 8 Sampling system (module 1)*

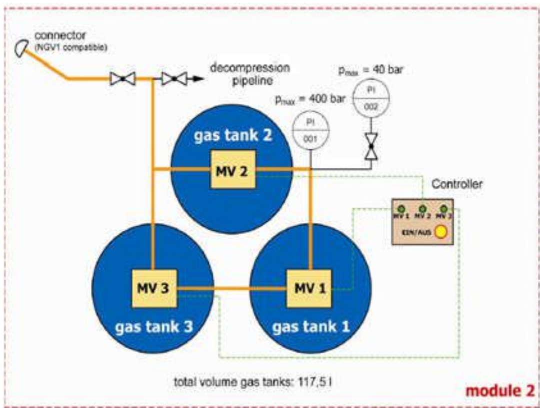



*Figure **9** Gas tanks (module 2)*

[18] (Zander TB 20 CE) was chosen

*Figure 10 Modified sampling system*

### 5.6.3. Results of the monitoring program

With the new sampling system various CNG fuelling stations were monitored from December 2008 to July 2009 to evaluate CNG-quality in Germany. In total approximately 40 CNG-fuelling stations were analysed. Various different types of fuelling stations and operating parameters were regarded (e.g. compressor type, filter media, natural gas quality, maintenance rates).

Moisture was no problem within the monitoring program. The limit of 40 mg/kg was not reached by far. Typical dew points lie between -40 and -25 °C, corresponding to 0.44 to 1.96 mg/kg.

Regarding particles and oil loading, a wide range was observed. With total values for regular samplings ranging from 0.67 to 21.29 mg/kg the content of solid and fluid particles exceeds the favoured limits (5 - 10 mg/kg) of the automotive industry considerably in two cases. The average particle content lies at 8.36 mg/kg for the considered samples. The lowest value was measured at a CNG refuelling station with an oil-free compressor system. In one case an extremely high value of 137.08 mg/kg was analysed. In the corresponding gas analysis a content of higher hydrocarbons of 1,070 mg/kg was measured. The analysis of the higher hydrocarbons was critical in some cases as droplets accumulated in the pipeline system of the sampling apparatus. These droplets lead to a memory effect. To avoid this phenomena the system should be cleaned or reinstalled previously to each sampling which is not feasible from the practical and economical point of view. Furthermore, sharp edges or internal components should be avoided.

## 5.7. Experiences from the surveyed stakeholders

This subchapter is original material, written by the author.

From the answers that were obtained from the CNG station operators, it seems that the vast majority of the CNG station operators practice weekly controls of the oil level in compressors. On the upstream side (inlet of natural gas in CNG station), nobody from the surveyed operators reported the practice of checking the oil content in natural gas and no serious oil content measurements in CNG have been performed to date.

Techniques that were mainly used for measuring oil level in CNG are: oil consumption control method, and method with coalescing filters.

Unlike the relatively positive experiences with the oil measurements, survey has shown that almost none of the CNG station operators conduct sulfur measurement activities. However, additional market research has shown that in Germany there were performed couple of sulfur measurement campaigns in recent history. This will be elaborated in details in chapter 8.3.

With moisture measurement is a bit different story, because some of the operators reported that they have installed dew point sensors at the compressors, which allows them to monitor moisture level in CNG.

# 6. SAMPLING AND ANALYSIS OF WATER IN NATURAL GAS

The measurement of moisture content in natural gas is extremely important, from a technical perspective and in order to ensure conformance to contractual specifications.

Typically, this measurement is one of the most difficult to perform successfully. Moisture removal is a key stage of natural gas processing prior to sale and its efficiency is important in order to satisfy contractual obligations, to ensure that the gas has the right properties for subsequent use.

## 6.1. Analytical methods for water determination

This subchapter is an excerpt from SGC Rapport 290 Development and validation of methods for test of CNG quality inclusive of oil carryover, Karine Arrhenius, Haleh Yaghooby, Per Klockar 2013, see chapter 5.5

.

Water is known by different names in different states. It can be measured in many ways[19]. In a gas mixture, the total pressure of the gas is the sum of all the individual pressures of its gas components. The partial pressures also represent the proportion by volume of the various gases:

$$H_2O \text{ \%-vol} = \frac{pw\ H2O}{pt}$$

The water vapour saturation pressure (pws $H_2O$) is the maximum partial water pressure (pw $H_2O$) in a gas which is dictated by temperature.

The relative humidity RH (%) expresses the relation between the amount of water vapour present and the maximum amount that is physically possible at that temperature.

$$\text{\% RH} = 100\% * \frac{pw\ H2O}{Pws\ H2O}$$

Dew point temperature is the temperature where condensation begins or where the relative humidity would be 100% if the gas was cooled.

The water vapour saturation pressure is a known variable so the dew point can be calculated from the relative humidity and temperature.

If the dew point temperature is below the freezing point, the term frost point is sometimes used.

Typically, the water content / dew point is determined using portable instruments. Mitchell Instruments CERMAX, used for example by the German association for gas and water (DVGW), is equipped with a ceramic humidity sensor. The measurement principle of these sensors is the adsorption / desorption of water molecules on the hygroscopic layer between two conductive plates causes electrical changes in the dielectric constant between the plates, which is a function of the number of water molecules adsorbed. There are other methods, more or less complicated to measure the moisture content as the Karl-Fischer titration (as recommended in SS 15 54 38[20]) or the use of Dräger detector tubes providing direct impact in mg / L but the last ones are not enough sensitive to measure the level of water that is recommended in SS 15 54 38 standard.

[19] Relative humidity, dewpoint, mixing ratio, the many faces of water, J. Häkkänen, P. Laitinen, http://www.vaisala.com. Another interesting source or further reading is "Herring Jack, *Determination of Hydrocarbon Dew Point Measurement in Natural Gas*, Paper ID: 2008058."

[20] SIS (1999). "SS 15 54 38 Motorbränslen - Biogas som bränsle till snabbgående ottomotorer (Motor fuels – Biogas as fuel for high-speed otto engines)"

# 7. SAMPLING AND ANALYSIS OF SULFUR IN NATURAL GAS

This chapter is based on excerpts from the published report: *Determination of Sulfur Components in Natural Gas: A Review,* Hai Pham Tuan, Hans-Gerd Janssen*, and Carel A. Cramers, Eindhoven University of Technology, Laboratory for Instrumental Analysis, P.O. Box 51 3, 5600 MB Eindhoven, The Netherlands.

The presence of sulfur components in natural gas constitutes a source of concern because of the corrosive nature of these components as well as their potential hazards for human health and for the natural environment[21]. Additionally, if natural gas is used as a reagent in chemical processes, sulfur species present in the gas may adversely affect the performance and life-time of catalysts involved in the reaction.

For the reasons outlined above, the accurate determination of sulfur components in natural gas is of utmost importance. The group of components that should be analysed includes hydrogen sulfide, carbonyl sulfide, the $C_1$ to $C_4$ mercaptans, lower sulfides and odorants, such as for example tetrahydrothiophene (THT), added to the gas to impart a characteristic smell for safety -purposes. The concentrations of these components differ, depending on the origin of the gas. In general, the concentrations of sulfur components in natural gas for domestic and industrial use range typically from a few to several tens of parts per million on a volume basis. An exception is $H_2S$ which can be present at concentration levels up to one per cent.

Standardized methods for the determination of sulfur species in natural gas have been published by the International Organisation for Standardization (ISO). They can be classified as either conventional techniques *(Wickbold, Lingener)* or modern instrumental techniques (GC-based). IS0 standard 4260 describes the *Wickbold* combustion method, a method for the determination of the total sulfur content of natural - and other gases. In the *Wickbold* method the natural gas sample is supplied to the burner of an oxy-hydrogen flame, where the sulfur compounds are burnt with a considerable excess of oxygen. The resulting sulfur oxides are converted into sulfur acid by absorption in a hydrogen peroxide solution. Depending on the sulfur content of the sample, the sulphate ions in the absorption solution are determined by colorimetric nephelometric, turbidimetric or conductometric titrations.

More recently, IS0 standard 6326-5 was published. This standard procedure describes the use of the *Lingener* combustion method. In the Lingener method a given volume of natural gas is burnt with air at atmospheric pressure in an enclosed combustion apparatus. The resulting sulfur oxides are oxidized to sulfuric acid by absorption in a hydrogen peroxide solution and afterwards titrated with a barium chloride solution. The total sulfur content which can be determined with this measurement method is 10 - 1000 mg $S/m^3$ [22].

As opposed to the *Lingener* and the *Wickbold* method, which both measure the total sulfur content, IS0 method 6326-3 describes a potentiometric method for sulfur determination that only responds to hydrogen sulfide, mercaptans and carbonyl sulfide. In this method hydrogen sulfide and mercaptans are absorbed in a 40% ( m/m ) potassium hydroxide solution, carbonyl sulfide is absorbed downstream in a 5% (m/m) alcoholic mono ethanolamine solution and afterwards titration of the absorbed hydrogen sulfide, mercaptans and carbonyl sulfide is performed with a silver nitrate solution. The concentration range of sulfur compounds which can be determined with this measurement method is 1 - 10 mg $S/m^3$ for $H_2S$, 1 - 20 mg $S/m^3$ for mercaptans and 1 - 30 mg $S/m^3$ for COS.

---

[21] Determination of Sulfur Components in Natural Gas: A Review, Hai Pham Tuan, Hans-Gerd Janssen*, and Carel A. Cramers, Eindhoven University of Technology, Laboratory for Instrumental Analysis, P.O. Box 51 3, 5600 MB Eindhoven, The Netherlands

[22] IS0 6326-1 (E) International Standard "Natural Gas-Determination of Sulfur Compounds" Part 1 "General Introduction", Geneve, Switzerland, 1989

Apart from one common advantage, i.e. no calibration procedure is needed as the three methods described above are absolute measurement techniques; they also suffer from a number of common disadvantages. The measurements are time-consuming, complex and are, due to the complexity of the experimental steps involved, difficult to automate. Moreover, the accuracy and the detection limits of especially the Lingener method do not meet the required limits. It is evident that because the methods measure the total sulfur content (IS0 4260 and IS0 6326-5) or the concentration of different classes of sulfur compounds (IS0 6326-3), no information on the concentrations of the individual sulfur species is obtained. This disadvantage can be overcome by using GC-based methods for sulfur determination.

The IS0 standards 6326-2 and 6326-4 describe gas chromatographic methods for separation and detection of individual sulfur components in natural gas. In IS0 6326-2 hydrogen sulfide, methyl to butyl mercaptans and tetrahydrothiophene (THT) are separated on a gas chromatographic system equipped with a separation column containing 30% (m/m) silicone oil and 30% (m/m) dinonyl phthalate on Chromosorb W. The sulfur compounds are subsequently detected with an electrochemical cell, in which they are oxidized by a chromium oxide solution and at the same time the potential difference over the platinum electrodes is measured. The IS0 6236-2 method suffers from two serious disadvantages.

Firstly, it is not applicable for the determination of carbonyl sulfide. Furthermore, the chromatographic conditions specified in the method only enable hydrogen sulfide and methylmercaptan to be determined if the ratio of the concentration of the former to the concentration of the latter is less than 10. The same applies for the quantification of two thiols eluted consecutively.

A few of the major limitations of the IS0 6326-2 method were eliminated in IS0 6326-4. The chromatographic separation of the sulfur components was optimized, resulting in a system that enabled quantification of all major sulfur species in natural gas samples. The components are separated using a temperature programmed 1.2 m x 2 mm column packed with styrene/divinylbenzene porous polymer beads (80 - 100 mesh) and measured with a sulfur-selective flame photometric detector (FPD). The detection limit is approximately 0.1 mg $S/m^3$, which is comparable to the detection limits obtainable by the IS0 standard method 6326-2.

The IS0 methods 6326-2 and 6326-4 yield the concentrations of the individual sulfur components in the gas. The total S concentration can then be obtained by summing the equivalent S-weights of the individual components. The chromatographic separation procedure incorporated in IS0 6326-4 provides sufficient separation of all sulfur components. Unfortunately, however, it still has a number of problems, most of which originate from the use of flame photometric detection. The selectivity of the flame photometric detector (FPD) is limited and, although fairly selective, this detector still responds to high concentrations of non-sulfur components. Moreover, high concentrations of hydrocarbons coeluting with a sulfur containing component can quench the sulfur signal. Finally, the response of the FPD is inherently non-linear and often also compound dependent. For these reasons, the concentrations of the sulfur species to be determined are limited to the range of 0.1-30 mg $S/m^3$.

For most present applications, the detection limits and the reliability of the analytical results achievable by GC with FPD detection are within the desired range. More stringent environmental regulations as well as higher demands currently being posed on the purity of natural feed-stocks for chemical processes, however, force analytical chemists to develop new analytical methods that allow the accurate and reliable determination of sulfur in natural gas at concentrations well below the limits currently achievable. The complexity of the natural gas matrix and the extremely low detection limits required render this task extremely challenging. Analytical methods for analyses at trace levels in complex and interfering matrices often require the use of selective preconcentration/enrichment techniques. Only if this step and the subsequent separation and detection are fully optimized, it is possible to meet the required sensitivity limits with an acceptable level of reliability. In the vast majority of applications, the demands posed on each of these three steps are determined by the performance of the other two. If, for example, a universal detector is employed, the requirements imposed on the sample pretreatment and separation is much more stringent than in the case of the use of a truly specific detector.

Up till now, virtually no attention has been paid in literature to the use of preconcentration techniques in natural gas analysis. On the other hand, various methods for preconcentration of sulfur in samples of environmental or medical origin have been published. In general, the principles of these methods are also applicable for trace analysis of sulfur in natural gas. Irrespective of the matrix, the strong tendency of sulfur components to adsorb on various types of surfaces seriously complicates preconcentration and analysis of these compounds. In literature, both packed and open-tubular columns have been employed for the separation of sulfur species in a wide variety of samples.

The determination of sulfur containing components in natural gas is a typical example of trace analysis in a complex and interfering matrix. Gas chromatography has proven to be an extremely useful technique for this difficult analytical problem. In order to obtain the maximum possible performance, each of the three steps of the analytical procedure, I. e. sample preparation separation, and detection has to be carefully optimized and fine-tuned to meet the requirements of the other two steps of the procedure. In this respect, the choice of the detector plays a key role. Despite tremendous progress in the field of especially flame photometric and sulfur chemiluminescence detection, at present no detector is available that provides the selectivity and sensitivity required to keep pace with the ever more stringent demands currently being imposed on sulfur detection limits and analytical accuracy. Optimized chromatographic separations have to be incorporated in the analytical procedure. Due to their high resolving power capillary columns are to be preferred over the classical packed columns. The sensitivity of the analytical system can be improved by preconcentration of the sulfur components on a suitable solid adsorbent prior to transfer of the sample on to the chromatographic column. Polar adsorbents appear highly promising as these materials could provide selective enrichment of the components of interest.

For calibration of GC based analytical techniques for sulfur determination in natural gas, dynamically generated calibration standards appear to be more reliable than statically prepared standards. Adsorption losses which are unavoidable in static calibration are significantly lower or often even fully absent in dynamic system such as permeation or diffusion devices.

# 8. STATE-OF-THE-ART COMMERCIALLY AVAILABLE REMOVAL TECHNOLOGIES FOR OIL, WATER AND SULFUR

## 8.1. Oil removal technologies

This subchapter is an excerpt from the report: *Oil in vehicle gas – regulatory frameworks, test methods and filters*, Swedish Gas Centre 2007.

To reduce the level of oil in CNG, coalescing filters are mainly used. These filters remove only oil in aerosol form. Another type of filter is adsorption filters, although mainly used to dry the gas. Adsorption also removes oil contained in the vapour phase.

### 8.1.1. Coalescing filters

The principle of a coalescing filter is that the gas passes through a tubular shaped wall consisting of for e.g. thin glass fibre. The gas passes through but aerosols of oil merge into larger droplets which flow downwards inside the filter due to gravity. The oil collects in an oil sump, which needs to be emptied regularly, see Figure 11.

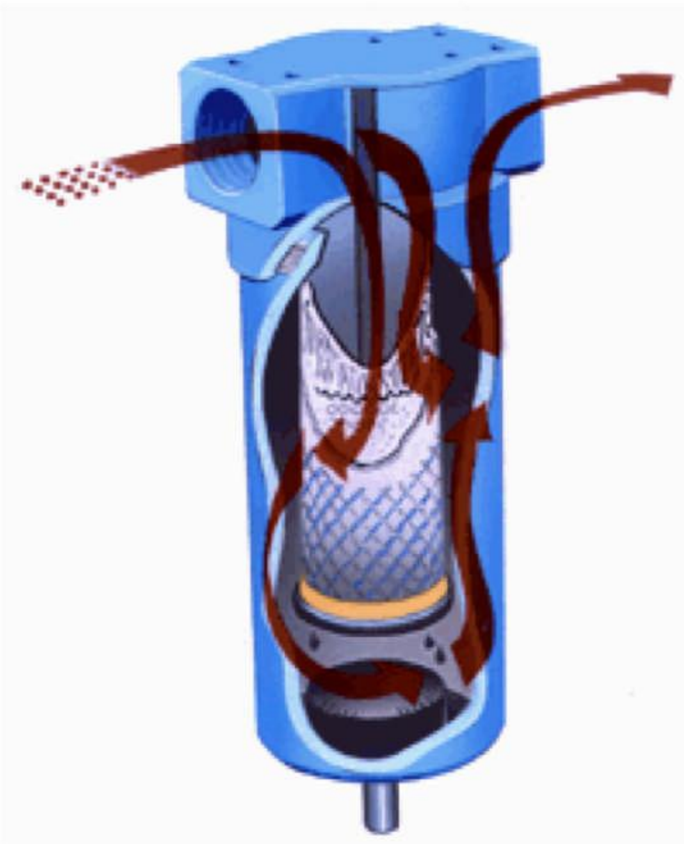


*Figure 11 Sketch of a coalescing filter*

There are several suppliers of filters that can be used for vehicle fuel. Here are the filters from one particular supplier described. What pressure levels they can handle and the size of the particles separated out and the effectiveness with which the particles are separated.

Parker offers Finite ® Filter for natural gas applications. The filters can withstand a maximum pressure of 350 bars and 177 ° C. Commonly used in automotive gas stations are filter elements of grade 4 and 10. Finite ® Grade 4 removes 99.995% of aerosols in the size 0.3 till 0.6 microns, while the 10-degree filter, which removes 95%, used as a pre-filter. Filter housings are those of their J-series that is adapted for filtering at high pressure. Recommended use is a filter of 10 degrees followed by one with grade 4 after the compressor and before the gas storage and then one with grade 4 after the gas storage but before the dispenser[23].

There are several things to consider when choosing coalescing filters. An important factor is how small aerosols the filter can capture and its effectiveness. According to the study from GRI occurring aerosols from air compressors in the range of 0.01 to 0.8 microns. This means that the filter needs to be capable of capturing very small aerosols. It is stated for each filter type what their effectiveness is when separating aerosols of a certain size. It should be emphasized that the filters do not always reach those efficiency values in the real world, where values are produced in standard conditions which are not entirely consistent with conditions at a refuelling station[24]39.

For the oil to be separated properly from the gas a sufficiently large filter surface area is required, or gas will pass at an excessive speed through the filter. This can be achieved either by using a larger filter or by putting more filters in series, the latter being the most common approach.

With each coalescing filter there is also an oil sump, where the oil accumulates. It is important it is selected properly so that the oil does not go up in the filter section.

The oil sump needs to be emptied periodically. To facilitate the maintenance of the filters a large oil sump can be selected, another option is automatic discharge systems, which is activated when the compressor is turned off.

The gas temperature controls the amount of oil contained in the vapour phase and how much is available as aerosols. Cooler gas leads to more oil aerosols, and it implies that it is appropriate to install coalescing filters as far from the compressor as possible, so the gas can cool. At the same this is contradicted by the fact that long residence time of aerosols in the gas means that more oil may transfer into the vapour phase. This suggests the best approach to be two filters in series, where one is placed near the compressor, and the second one as far away as possible from the compressor.

### 8.1.2. Adsorption filters

Coalescing filter cannot separate the oil in the vapour phase, but that is possible to do with adsorption filters. The principle of adsorption filters is based on the water / oil adsorbing (sticking) on the filter material which may consist of activated carbon or alumina. The filter material becomes saturated after a while and need to either be regenerated or replaced.

Adsorption is mainly used to dry the gas from water. Aluminum oxide is a powerful desiccant. The adsorption medium can be regenerated from the water by heating, which is done for example in upgrading facilities for biogas. However, regeneration of oil is more difficult and the material is therefore usually replaced rather than regenerated.

There are several suppliers by filters that can be used for vehicle fuel. This filter is described by the supplier, the pressure levels they can handle, and the size of the particles separates and the effectiveness with which the separate particles.

[23] www.parker.com Bulletin 1300-200/NA, J-series, High Pressure Compressed Gas Filters

[24] M Czachorski et al., NGV Fueling Station Compressor Oil Carryover Measurement and Control, GRI-95/0483, Final Report, Institute of Gas Technology, Februari 1996.

Zander offers adsorption, HDAM series, which can handle flows of 60-600 Nm3 /h and pressures up to 350 bar. The filter is a layer of activated carbon to remove oil from gas. Different filter elements have different efficiency, with the XP model removing aerosols of 0.01 micron to 99.99999% and the model ZP removes aerosols of one micron to 99.9999%. The filter can be installed with a pre-filter and/or post-filter configuration[25].

Adsorption should always be installed after effective coalescing filters so that they are only used to separate oil and water as vapour from the gas. The adsorption medium may be sensitive to large pressure changes, which may affect the structure and reducing its adsorption capacity. Therefore, adsorption filters are installed after the backflow preventer.

## 8.2. Water removal technologies

This subchapter is based on excerpts from: *Moisture measurement in natural gas*, Rolf Kolass, Michell Instruments GmbH, Friedrichsdorf, Germany Chris Parker, Michell Instruments Ltd, Cambridge, UK**.**

### 8.2.1. Dehydration Process

The most common processing technique for drying natural gas is that of simple mechanical separator[26], to divide the gas from the liquids of the two phase flow coming from the gas field, followed by glycol dehydration. The scale and cost of this technology makes it more suited for upstream, centralised treatment, rather than downstream at refuelling station level. Here a riser tower has an array of spray nozzles around it's circumference through which glycol is injected, as a liquid desiccant, into the gas stream flowing up through the tower. The adsorption process results in moisture-laden glycol that coalesces into globules that are naturally forced, through flow dynamics, outward towards the wall of the tower. The liquid glycol is collected in trays, piped out of the tower and is regenerated by heating to evapourate the absorbed moisture prior to re-injection in a continuous operating, re-circulating loop. Such glycol contactors, as they are termed, are specified to achieve a moisture content of less than 48 mg/Nm3 (3 Lb./MMSCF (pounds of moisture per million standard cubic feet of gas)) under normal operating conditions.

The high flow velocity of gas through the contactor leads to possible carry through of glycol mist. Consideration must be given to this characteristic if the application of a hygrometer is to be successful in monitoring the performance of the dehydration process. Contamination of the moisture sensor or sample handling system results in a serious deadening in response for the analyser due to the moisture adsorption/desorption capacity of the glycol. A conventional coalescing filter with fibre element positioned at the front end of the sampling system can effectively protect the moisture sensor from contamination but will not solve response problems unless any collected liquids are flushed out of the filter housing by a continuous flow from the drain port (Figure 12). A membrane type filter offers the best protection in such glycol applications but is restricted to a maximum operating pressure of 10 MPa. These filters also work on a bypass flow arrangement but use a micro-porous membrane of fluorocarbon material to offer superior protection.

[25] www.zander.de CNG-Adsorber for NGV Fuelling Stations
[26]

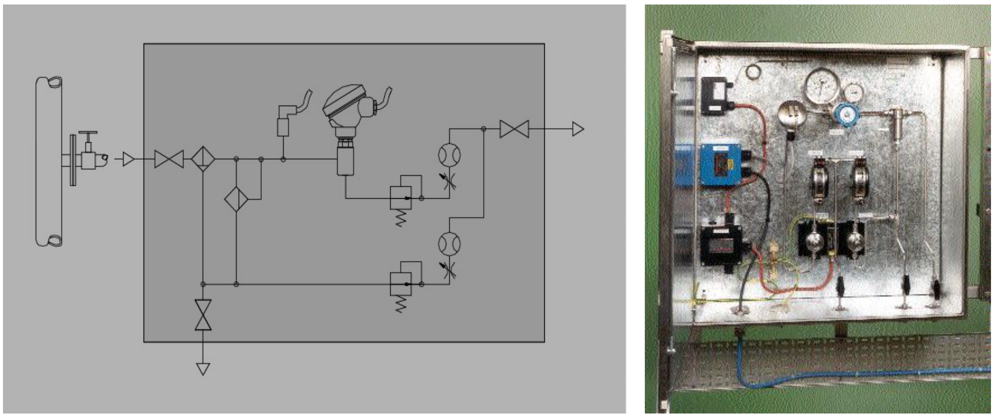


*Figure 12 Schematic Diagram and Photograph of a Typical Moisture Analyser for Natural Gas Dehydration Plant*

### 8.2.2. Natural gas dryers

Natural gas dryers remove water vapour prior to storing or using natural gas for a vehicle fuel. High water content can cause blockage of CNG in refuelling systems and can contribute to corrosion in storage vessels and piping.

The need for a dryer is determined by the water content of the incoming gas and the minimum ambient temperature. In cooler climates using standard pipeline natural gas, a dryer is likely required. A dryer may also be required in warmer climates.

The low pressure dryer uses molecular sieve absorbent desiccants to remove water vapour to extremely low levels. After operating for a period of time, the dryer has removed so much water that it requires regeneration or replacement of the desiccant. The dryers may include a hygrometer which confirms the gas has been sufficiently dried.

*Figure 13 An example of low pressure dryer (ANGI Energy Systems)*

On the other hand side, high pressure dryers for compressed natural gas are the economical and logical choice for drying natural gas for NGV refuelling.

Installed immediately after the compressor, these dependable, yet simplistic systems ensure gas dew points in accordance the latest published standards that pertain to the quality of natural gas for use as a vehicle fuel and optimum gas quality to storage and to NGVs. These systems use replaceable cartridges filled with high capacity molecular sieve tailored for drying natural gas without removing the odorant. New cartridges are inexpensive and can be quickly and easily replaced at your convenience and without interruption to the station. There are no moving parts to the systems and they do not require power for operation. An electronic dew point monitor with LCD display is available for indicating gas dew point in choice of engineering units. The moisture sensor is installed downstream of the gas processing system.

*Figure 14 An example of high pressure dryer (Bauer Compressors INC)*

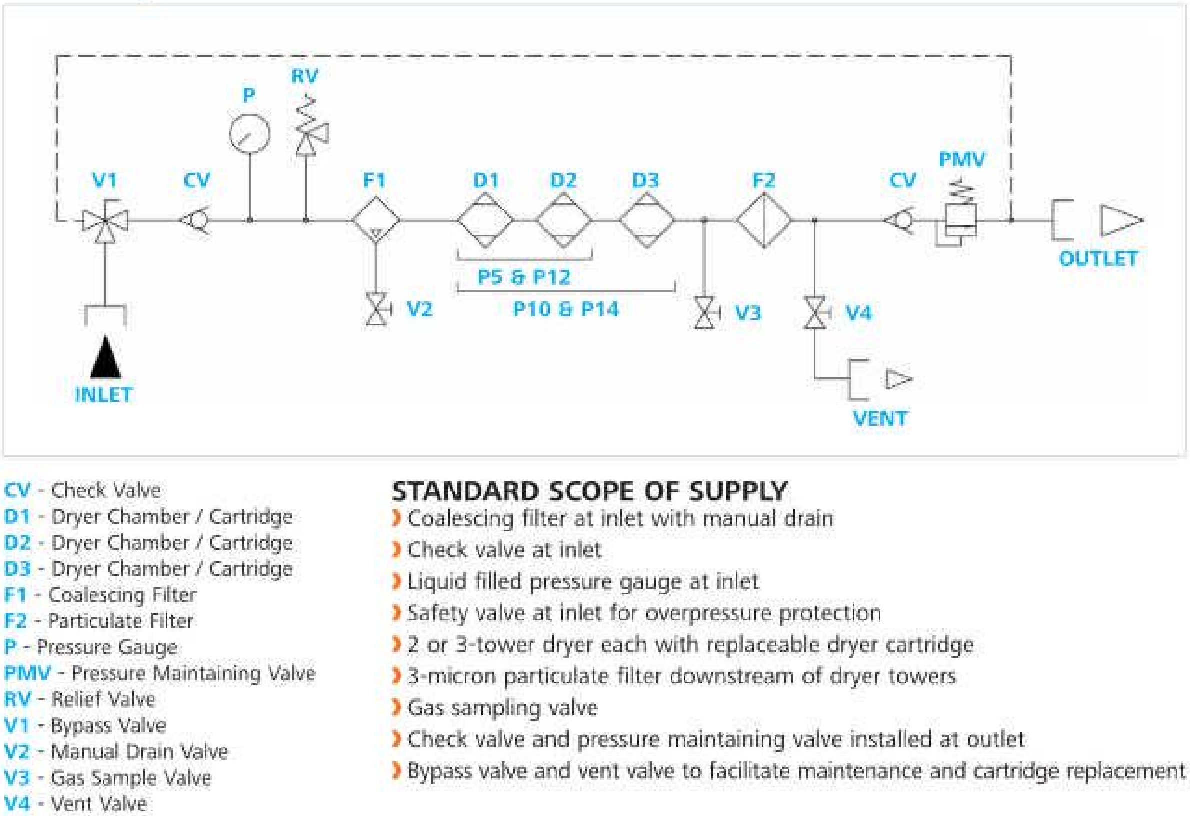


*Figure 15 Operational scheme of high pressure dryer*

## 8.3. Sulfur removal technologies

### 8.3.1. Adsorber filters

For the purpose of elaborating the quality of adsorber filters in a process of sulfur level minimization in CNG, following reports were used:

- *Desulfurization of natural gas in gas stations*, Seminar „Erdgastankstellen“, 17 June 2010 in Celle, Dipl.-Ing Hans-Jürgen Schollmeyer, E.ON Ruhrgas AG
- CNG Refuelling Stations In The L-Gas Market Area, 24 May 2012, DVGW

E.ON has undertaken several field tests with adsorber filters with principal goal to reduce sulfur content in natural gas that has been odorized with regular odorizer.

In principal, two approaches have been applied:

- full desulfurization on the low pressure side and after odorisation in gas station
- full desulfurization on subset of a natural gas at the high pressure side

In addition to E.ON, WINGAS, RWE and BASF has also played the role in a practical desulfurization test as project partners.
Field test has been practiced at CNG station in Gehrden, since October 2008.
The concept of a desulfurisation has been done with bypass gas flow at the low pressure side of CNG station, with following features:

- bypass flow variable, max 14 Nm3 / h

- filter housing dimensions 130 mm x 1000 mm
- Electrically heated adsorber
- Extensive measurement technology, continuous S measurement, remote data transmission

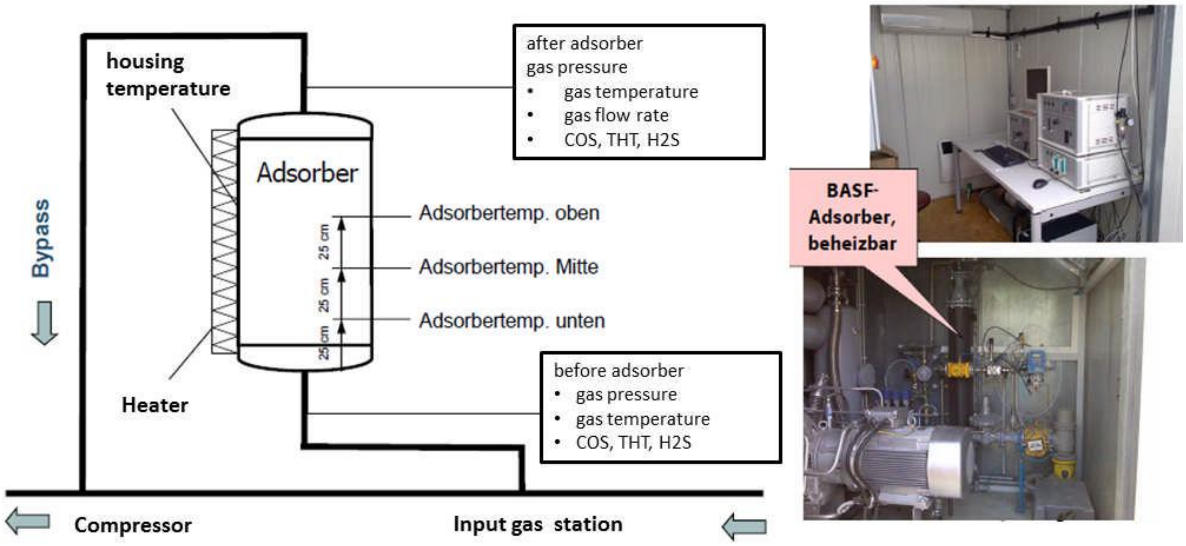



*Figure 16 Experimental setup schematically*

Another interesting field testing was done at Hanover CNG station, located Hildesheimer Straße. The testing was done in two tracks, with the features as follows:

***Test Track 1: Fully demineral desulfurization of natural gas partial stream in High pressure range (S-filter from Bauer, designed for short test)***

- Part flow freely adjustable
- Functional Test / determine the optimal subsets of natural gas
- Test Period 10/2009 - 12/2009

***Test Track 2: Partial demineral desulfurization of natural gas in Low-pressure region (BASF technique)***

- Electrical heating S-adsorber possible
- Discontinuous S measurement
- Functional test
- Determination of the optimum filter materials
- Long-term test
- Started in February 2010


*Figure 17 An overview of the filed test at CNG station Hannover*

Results of field testing CNG Hannover in Hildesheimer Straße, are depicted in the following figures.

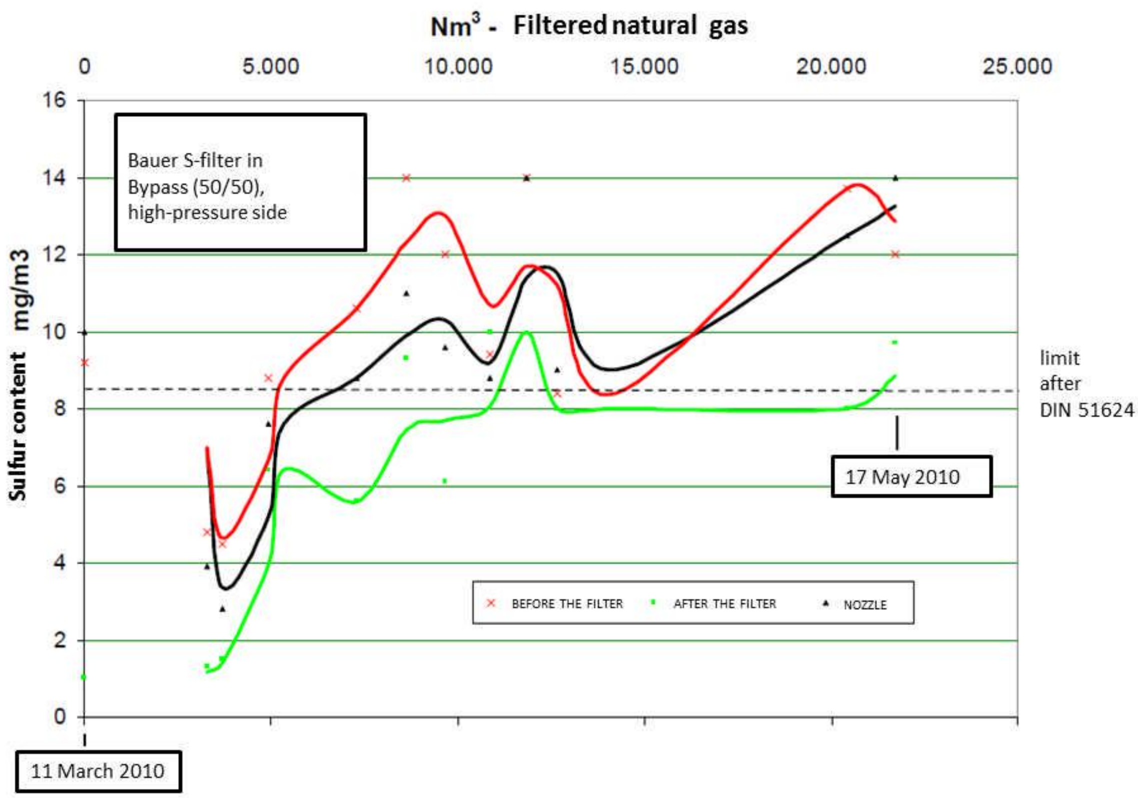



*Figure 18 Evolution of sulfur content at Hannover CNG station*

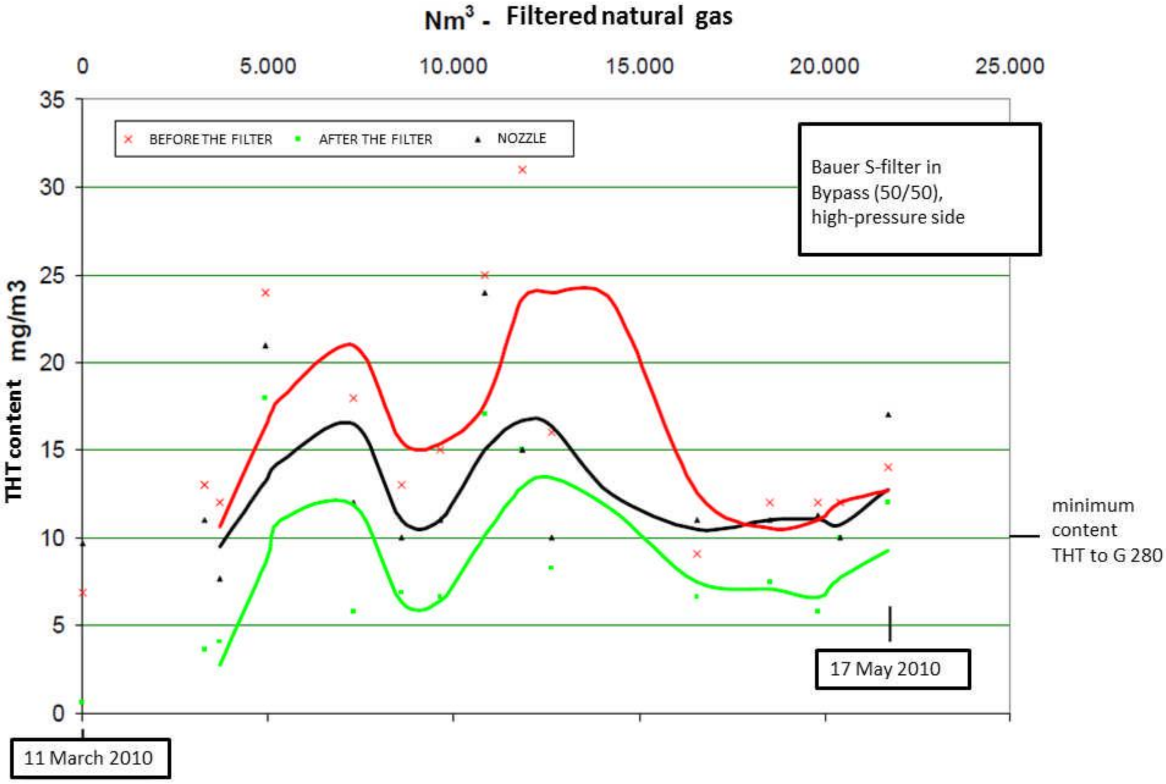



*Figure 19 Evolution of THT content at Hannover CNG station*

Results of field testing with BASF adsorber at a glance:

- Development of desulfurisation is much more complex than expected (due to different boundary conditions)
- For gases, sulfur load is determined by $H_2S$ and already with available technology, a sufficient selective desulfurization can be achieved.
- The reduction of COS is possible only selectively. However, it do not succeed in all conditions, e.g. at low temperatures.
- The use of sulfur-free odorants can be problematic (S-Free is retained in the filter at low temperatures, while THT is unproblematic).
- A total desulfurization is in principle possible, but with appropriate materials (after odorization in the gas station is required)
- Technical solutions were fully adjusted by the beginning of 2011
- Commercially available solution as of mid-2011
- Total investment need for desulfurization on the low pressure side starts at 20,000 €/CNG station while in the case of desulfurization on the high pressure side it is equal to 10,000 €/CNG station

### 8.3.2. The impact of odorants on sulfur content

This chapter is based on translated excerpts from a Swedish report titled “SGC Rapport A26 Svavelfri och svavelfattig odorisering av gasol och naturgas” (Sulfur and sulfur-poor odorization of LPG and natural gas) Norén C, Thunell J (2002).

Sulfides and mercaptans are the dominant odorants today.
Table 6 lists some basic data and characteristics of the mercaptans and sulfur commonly used to odorise energy gases and process gases. It may be mentioned that a number of the

reported odorant have the same chemical formula but different names, depending on the appearance of the molecule, for example (C4HNS) is a cyclic molecule.

Of the reported odorant TBM has far superior qualities, but with one major limitation. The freezing point is -0.5 °C, which means that TBM must be mixed up with other substances to be used. Otherwise, TBM has several desirable properties:

- Ability to penetrate the soil without the smell disappears
- Low sulfur content of all the substances used in the sulfur-containing odorants
- Very good chemical stability and oxidation resistance
- Very low detection ability

THT's main advantages over TBM's better chemical stability in oxidizing environments and lower freezing point. The better chemical stability, however, is of minor importance in the gas distribution in the B-lines. Mercaptans usually have better odour properties than IT so they can be added in smaller amounts than and thus better environmental and corrosion properties.

*Table 6 Facts about various components included in the odorants*

| Name | Tetrahydrotiophene | Dimethylsulfide | Tert-butylmercaptan | Methyl-ethylsulfide | N-propylmercaptan | Isopropylmercaptan | Ethylmercaptan |
|---|---|---|---|---|---|---|---|
| Short name | THT | DMS | TBM | MES | NPM | IPM | EM |
| Chemical Formula | $C_4H_8S$ | $C_2H_6S$ | $C_4H_9SH$ | $C_3H_8S$ | $C_3H_7SH$ | $C_3H_7SH$ | $C_2H_5SH$ |
| Belongs to group | Cyclic sulfides | Sulfide | Mercaptan | Sulfide | Mercaptan | Mercaptan | Mercaptan |
| Is used for | NG/LPG | NG/LPG | NG/LPG | NG | NG/LPG | NG/LPG | NG |
| Form | Liquid | Liquid | Liquid | Liquid | Liquid | Liquid | Liquid |
| Density | 0,999 $kg/m^3$ | 0,848 $kg/m^3$ | 0,81 $kg/m^3$ | 0,842 $kg/m^3$ | 0,842 $kg/m^3$ | 0,814 $kg/m^3$ | 0,839 $kg/m^3$ |
| Boiling point | 121°C | 36°C | 64°C | 66°C | 67°C | 57-60°C | 35°C |
| Freezing point | -96°C | -98°C | -0.5°C | -106°C | -121°C | -131°C | -148°C |
| Sulfur | 36,4% | 51,6% | 35,6% | 42,1% | 42,1% | 42,1% | 51,6% |
| Solubility in water | 3,7 g/l | 22 g/l | 2 g/l | 6,4 g/l | 1,9 g/l | 4,8 g/l | 15,6 g/l |
| hygienic limit | No | 1 ppm (NGV) | No | No | No | No | No |

Concerns about the need for diminishing content of sulfur in natural gas has led to both: development of a completely sulfur free odorant in Germany and production of mercaptans and sulfur mixtures that has so distinctive odour properties to odorant concentration can be kept at a lower level than before.

A completely sulfur free odorant S-FREE has been lately developed in Germany. There have been conducted laboratory and a field test that all have shown that odorant meets the basic requirements for setting of an odorant for natural gas. S-FREE in the current situation can only be used to odorise natural gas, but the adaptation of the S-FREE to other gases is in progress. The main reason for switching to S-FREE is pretty obvious:

Odorant is completely free of sulfur, which means reduced environmental impact and improved opportunities to use natural gas as a raw material for chemicals and fuels.
The main limitations of S-FREE could be stressed as follows:

- Ethyl acrylate is classified as potentially carcinogenic by several cancer research organisation and these organizations also believe that there is no margin for exposure to ethyl acrylate. However, there is not enough scientific evidence to classify ethyl acrylate as carcinogenic.
- Ethyl acrylate is listed on the National Chemicals Inspectorate Observation List with allergenic properties. It is important to emphasize that explanation of a substance in the Observation List does not involve a sale or use prohibition of ethyl acrylate.
- S-FREE is not a "typical smell of gas" which means that a broad set of adaptations should be done before exchange with classical odorants can be made.

Contacts, patent and literature search shows that other development work on completely sulfur-free odorants does not seem to be going on.

One tendency, at least in Europe, is a transition from THI to odorants based on mercaptans. Better odour properties of mercaptans makes dosage level and thus the sulfur content of the gas can be kept lower than the THI.

Field trials in Germany and Denmark show that Scentinel E is an odorant in many respects with substantially better properties than the typical odorants.
Besides Scentinel E, there are other alternatives to THT. Italgas for example, tried a mixture of IBM and MES with very good results.

### 8.3.3. Other sulfur removal concepts

This chapter is based on excerpts from the publicly available presentation: *CNG filling stations in the L-gas market area*, 24th May 2012, DVGW

In addition to the measuring campaigns conveyed in 2009 and 2010, DVGW has in 2012 organized exhaustive measuring campaigns in two German regions: North Rhine-Westphalia and Lower Saxony.

Specificities of those measuring campaigns can be found in continuation.
*Real measuring data from North Rhine-Westphalia*

Implementation of campaigns has taken place in 36 locations spread along the NRW region.

1st part of the measuring campaign was organized between 8 and 9 February 2012 in 14 cities while 2nd part of the measuring campaign took place between 21 - 23 and 26 - 28 Mar 2012 in 22 sites.

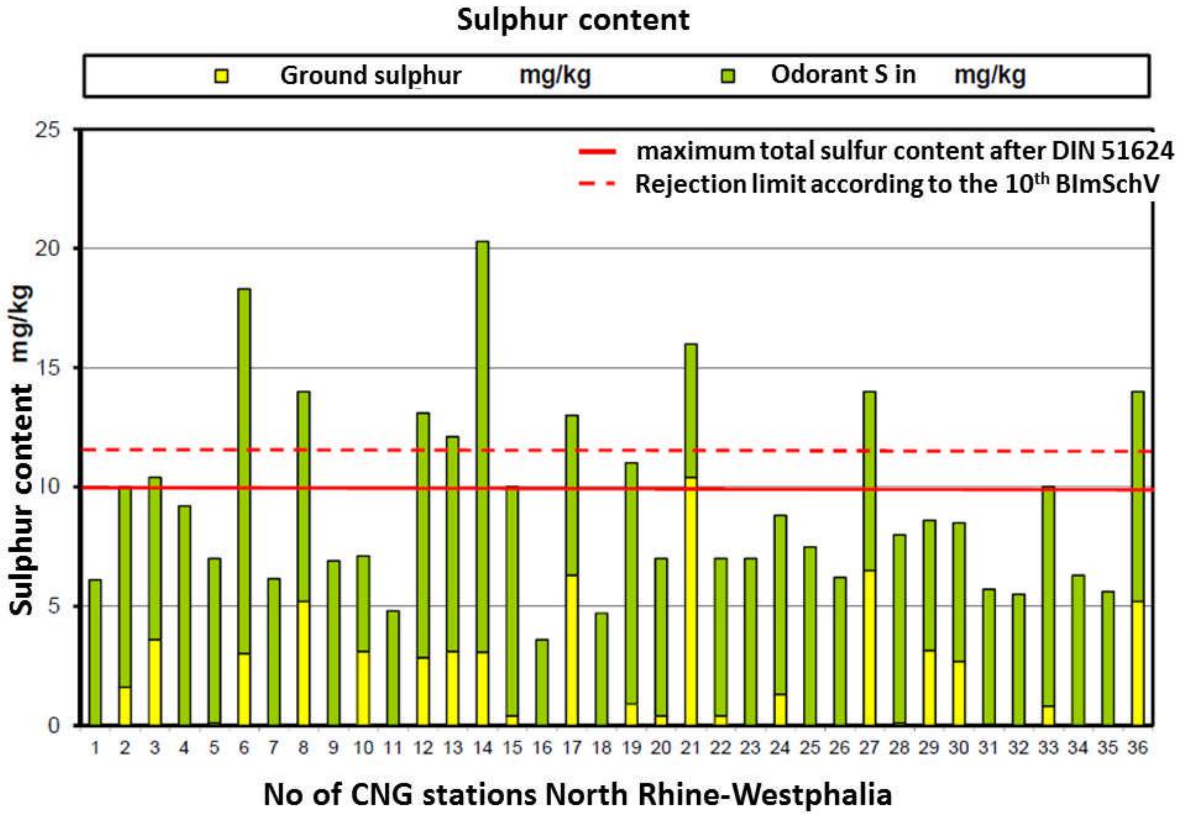



*Figure 20 Results of sulfur content measuring campaign in North Rhine Westphalia*

*Real measuring data from Lower Saxony*

In Lower Saxony implementation of measuring campaigns was conveyed in 42 locations. 1st part of the measuring campaign was done in the period between 2 – 3 and 13-14 February 2012 at 15 sites, while 2nd part was conveyed between 21 - 23 and 26 - 28 Mar 2012 at 27 sites.

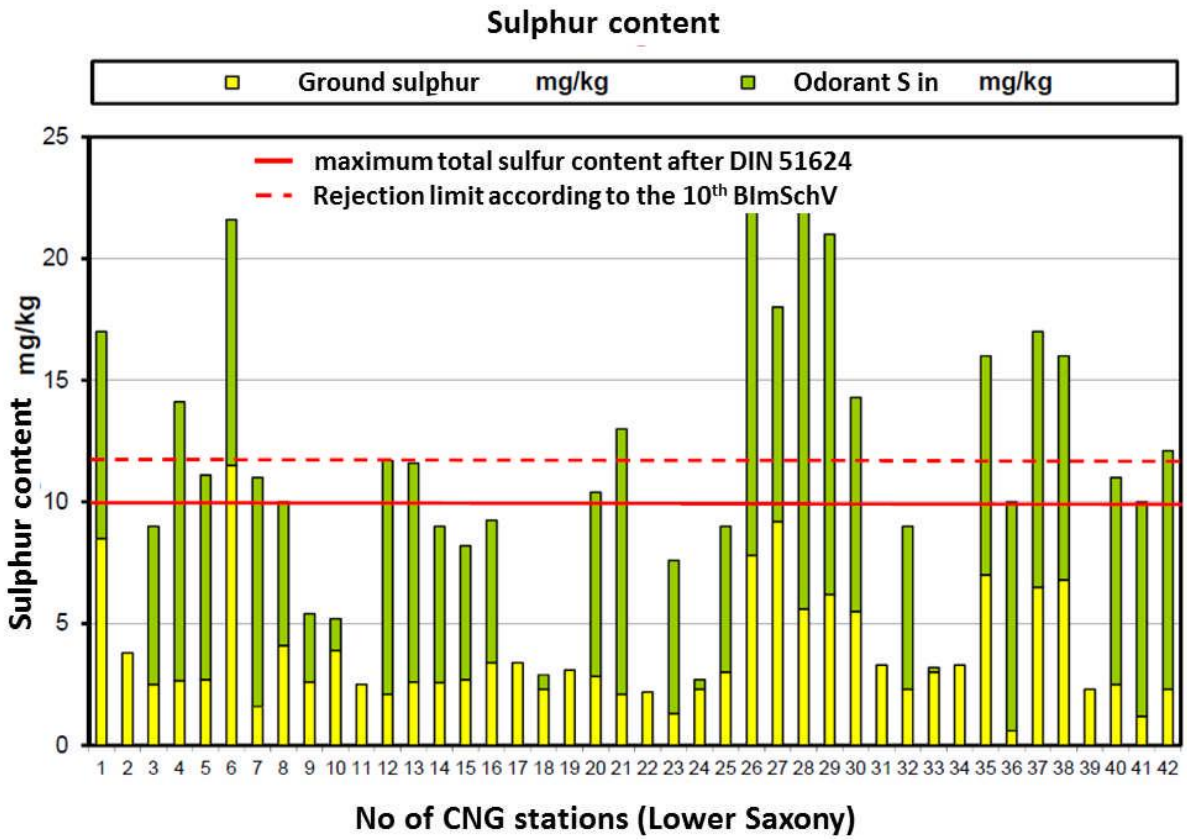



*Figure 21 Results of sulfur content measuring campaign in Lower Saxony*

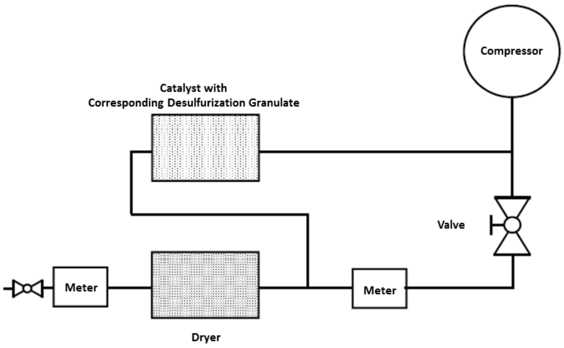
From the measuring campaigns results obtained and depicted in previous figures, it is obvious that principal carrier of sulfur in CNG comes from the odorants, while sulfur that comes together with natural gas, that is being extracted from the ground, represents only minor share and it is always below maximum total sulfur content, as defined by DIN 51624.

After successful execution of the measuring campaigns, under the steering of DVGW, there were introduced measures to reduce the emissions of sulfur at the same CNG refuelling stations.

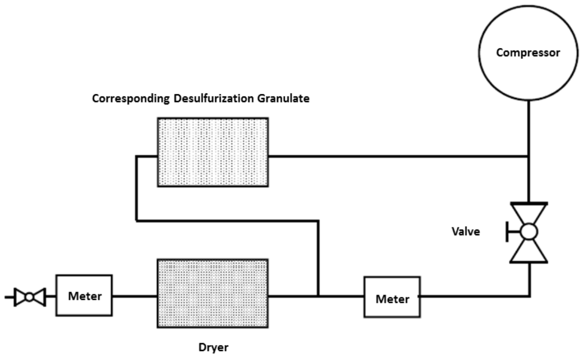
Several actors, such as RWE, E.ON and Enercity were engaged and different measures were applied. Following table and figures elaborate in details the results.

*Table 7 Locations and typical measures applied to reduce emissions at the CNG refuelling station level*

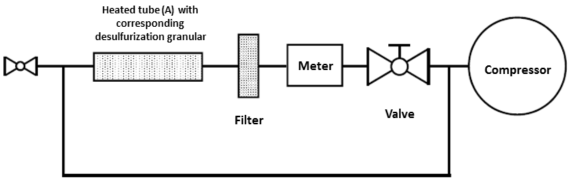
| **Location** | **Project management** | **Process features** | | | | | | **Process scheme** |
|---|---|---|---|---|---|---|---|---|
| | | | *Full desulfurisation* | *After odorisation* | *Low pressure side* | *High pressure page* | *Filter material* | |
| lle | RWE | x | | | x | | BASF | A |
| Gehrden | E.ON | | x | | x | | BASF | B |
| Hannover 1 | Enercity/ E.ON | | x | | x | | | |
| Arnsberg | RWE | x | | | x | | BASF | C |
| Hannover 2 | Enercity/ E.ON | x | | | | x | | |
| Wedemark | E.ON | x | | | | x | Carbo Tech | D |
| Schloß-Holte | RWE | x | | | | x | Carbo Tech | D |
| Borgholzhausen | RWE | | x | x | x | | BASF | E |
| Rheda-Wiedenbrüc | RWE | | x | x | | x | BASF | F |



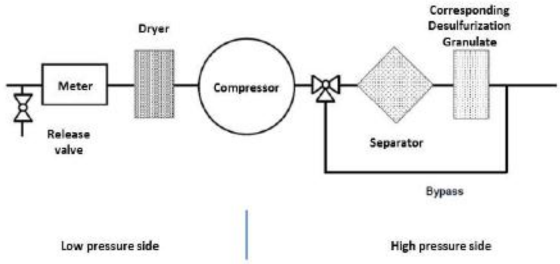

Scheme A - Partial desulfurization at low pressure side



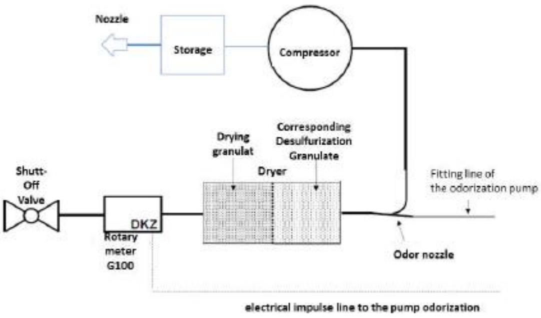

Scheme B - Full demineral desulfurization at low pressure side



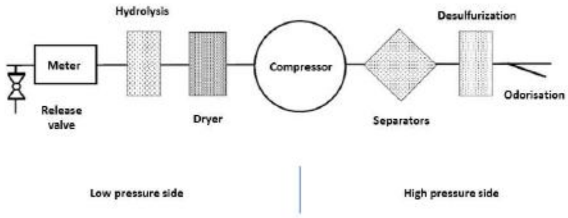

Scheme C - Full desulfurization at low pressure side




Scheme D - Partial desulfurization at high pressure side




Scheme E - Fully Demineral Desulfurization and After Odorisation at Low Pressure Side




Scheme F - Conversion of COS to H2S on low pressure side, desulfurization and after odorisation on high pressure side

*Figure 22 Desulfurisation process schemes*

# 9. CURRENT SITUATION ON THE MARKET REGARDING APPEARANCE OF CONTAMINANTS IN CNG – A MULTI STAKEHOLDER PERSPECTIVE

In order to get a better picture about current state of affairs at the European market when it comes to the presence of contaminants in the CNG, a broad scale market research has been conducted with the ultimate goal to assess the underlying issues from a system perspective. In that sense, comprehensive surveys for 4 groups of stakeholders were designed and deployed across the various EU market players.

The first group of stakeholders that were addressed was CNG station operators. 52 operators from 13 countries, including: Austria, Belgium, Czech Republic, Finland, France, Germany, Italy, Netherlands, Poland, Portugal, Spain, Sweden and Switzerland were surveyed.

Second group of the stakeholders that were interviewed was OEMs of the NGVs. 20 players from 5 countries were surveyed, including: Germany, Italy, Poland, Spain and Sweden.

Out of the many producers of CNG equipment, particularly compressors, 17 players from 6 countries responded to the survey, including: Canada, Germany, Ireland, Italy, Switzerland and Sweden.

Finally, 26 NGV fleet operators from 10 countries were addressed, including: Croatia, Finland, France, Germany, Italy, The Netherlands, Portugal, Spain, UK and Sweden.

**In total, 115 stakeholders from the European market have been invited to share their best practices in relation to appearance and mitigation of contaminants in CNG, while 25 of them answered the questionnaires.**

## 9.1. CNG station operators

CNG station operators interviewed have from 1 – 140 operating CNG stations in their portfolios. The vast majority of them have their businesses mainly nationally present without much international experiences. Average facility age span of the identified CNG station operators was between 3 – 12 years.

Supply of the hydrocarbons at the CNG refuelling stations level is predominately reserved for natural gas, while biomethane distribution is reported in Sweden, Germany and France, but usually far below 5% of the total CNG sales.

From the conducted research it is evident that reciprocating compressors, that are lubricating both the engine and the drive units, enjoy greater presence among the CNG operators in the market place. Only one CNG station operator from Slovakia reported that they have predominantly compressors equipped with oil lubrication of the drivers, but with oil-free cylinders.

From the collected answers, it is clear that, in some countries, CNG station operators have their refuelling stations equipped with high pressure storages that are providing fast refuelling options, but in some countries, like Portugal, only a part of the stations is equipped with high pressure storages.

Compressor brand that have been identified at the refuelling stations of the interviewed operators were:

Ariel, Atlas Copco, Bauer Compressors, Compair, Cirrus, Dresser Wayne, Galileo, Greenfield, Idro Meccanica, Nuovo Pignone and SAFE among others.

### 9.1.1. Oil

As for the frequency of oil level control in compressors, CNG operators are used to practice it on the weekly basis and regular maintenance of the CNG equipment is done between 2000 – 4000 of operating hours on average. Here are some of the answers that were collected:

- *"Oil Level is controlled every week"*
- *"Once a week - operating hours depending on compressor capacity and number of customers – on average something about 5000-7000 Nm$^3$"*
- *"Our maintenance is based on time. We have a big maintenance every year and smaller operations twice a year. We also have remote monitoring and control system."*
- *"Once a year full service (full maintenance) and once a year partial maintenance"*
- *"1 time per month, preventive maintenance (different capacities of compressor = different working hours per station) 4000 working hours scheduled maintenance"*

Lubricating oils that were identified at the level of CNG operators were SAE 40-Galp, Shell Corona AP 100, Bauer trade mark, Mobil SHC 1025, Shell Corona P 100/P 150, BP Energol, RC 150 and other special oils from the manufacturers.

***The interesting finding about the requirements that had been asked at the purchasing stage is the fact the almost none of the CNG operators requested from vendors the ceiling content of oil in CNG.*** *Only two operators answered following: "Yes, 8 ppm (Bauer Kompressoren) and 5 ppm from non-specified vendor."*

Oil removal filters are by default bolted on the majority of CNG compressors and dominant identified brands/models were:

- Coalescing filters – Parker
- Zander-Parker: TGH110/350CF-BA
- Etha Filter: E4520RVF10, E24RVF10, E24100SMA,
- Bauer compressors/Coalescer system.

Number of filters that are mounted on the CNG refuelling stations in most cases varies between 1 and 2, according to the survey findings.

*"At Bauer compressors, the oil removal is installed after every stage and at hydraulic compressors we have 2 oil removal filters."*

Given the answers that were collected, it is evident that recorded distance of the last filter, being installed away from compressors, is in the span between 3 -10 meters, while average one was 5 meters.

The answers that were obtained from the questionnaire addresses the fact that some CNG compressors are not equipped with the adsorption vapour filters, that are meant to serve as facilitators for removing oil in vapour phase, while those that are equipped, have been advised in advance by the CNG compressor vendors, at the commissioning stage.

Almost none of the interviewees responded to the question about the minimum mesh size of the oil filters that were bolted on the outlet stage of compressors.

While coping with oil filters clogging, operators have adopted various methodologies, such as: replacement of the filter, for instance every 1500 operating hours or manual purge every week and opening once a year.

"*We change new filters at every regular maintenance procedure",* was one of the answers that were obtained.

Room for the improvement of CNG station operations could be found in attaching a heat exchanger (Concentric pipe (water/gas)) to the outlets of the compressors in order to ensure a proper temperature of CNG. This practice has been identified in the operations of one French CNG station operator.

One of German CNG station operators provided following answer:

*"We do have installed heat exchangers after every compression stage, which ensure us gas outlet temperature at about 50°- 60 °C max".*

*"In our system we use inter stage intercooler UniTerm",* responded one Slovakian CNG station operators.

The CNG operator's practice has shown that the level of oil in CNG at a pre compression stage has not been monitored by anyone, while at the outlet stage, few operators measure the level of oil (some of them responded that they monitor every 8,000 operational hours). Use of this practice at all CNG stations would probably improve CNG quality.

Out of six methodologies for measuring the level of oil in CNG, such as:

1. Oil consumption control method With precipitation solvents
2. With coalescing filters
3. With glass fibre filters
4. With hydrocarbon dew point analysers
5. With Indicator tubes

, few operators mentioned that they practice fairly simple methodology such as sampling, while minority reported application of the coalescing filters methodology.

For those that are regularly implementing measurements for oil detection in CNG (post-compression stage), according to the records, a span of oil content was between 2 – 10 $mg/m^3$.

Only two operators reported that they had complaints from their customers that excessive level of oil caused a problem to their NGVs.

*"2 major complaints since the beginning of operations",* reported Portuguese operator.

*"4 in 10 years", reported French operator.*

*"We have only few official complaints about the quality of natural gas since 1996.",* reported one Finish operator.

Addressed complaints were mainly coming from the users of buses and trucks. German operator reported that they had some problems with personal cars, like Opel.

### 9.1.2. Water

Some of the CNG station operators use dryers, but they don't measure the moisture content at the outlet stage. Those that use dryers, change them once a year, up to the once each 2 years.

*"We use Bauer brand adsorption dryer. Dew point after dryer is less than -30°C at 200 bar."*

*"We have condensate separators and gas dryers and we dry natural gas down to the dew point below -25°C (200 bar)"*

*"We have dryer on suction line, own construction - Neat NGD", was the answer from one Slovak CNG operator.*

From the answers that have been collected, only few of the operators declared that they measure a level of moisture in CNG (mg/kg). For instance, one Finnish operator reported that moisture is measured with on board installed Bauer dew point sensors.

*"-50°C at 200 bar",* was the answer on the average level of moisture in CNG (mg/kg).

### 9.1.3. Sulfur

Unlike to more or less successful experiences with measuring and handling presence of oil in CNG, presence of sulfur is barely measured. Sulfur measurement is definitely one of the drawbacks in ensuring the top performing CNG quality.

Only one operator reported following:

*"We have regular measurements at one station, every month (ca. 12.000 Nm$^3$), and while at the other stations, once a year."* In this case, sulfur is occasionally measured with gas chromatography methodology.

To the question what is an average level of Sulfur in CNG, few operators replied following:

*"Before odorizing sulfur level is less than 1mg/m$^3$" reported one Finish CNG station operator.*

*"Between 5 and 10 mg/kg and sometimes even more", answered one German CNG station operator.*

### 9.1.4. Other CNG quality aspects

The question about the temperature at which CNG has been delivered to the station has been also raised and few of the stakeholders provided almost the same answer: *"10°C on average above the ambient temperature".*

The aspect that is certainly important when it comes to providing a premium quality CNG is related to the maintenance procedures. The answers that have been received out of three possible options, such as: in – house dedicated experts, outsourced service partner liaised with the vendor and outsourced independent service partner, were pretty unified. Operators most frequently use their in-house specialised experts, while only one reported that they practice outsourcing of the maintenance staff that was recommended by the vendor.

The number of vehicles that is typically supplied with the CNG, at the level of individually interviewed operator, is up to 5,000 a year.

When it comes to the measures that were carried out in order to improve the quality of the CNG, some of the interviewed stakeholders reported that following measures have been applied:

- Reducing the time span between regular control of the oil level in compressor (number of controls/number of refuellings)
- Changing the brand and type of oil for lubrication
- More frequent maintenance of the compressor (frequency of the maintenance/number of refuellings)
- Instalment/replacement of oil removal filters
- More frequent maintenance of the dryer (frequency of the maintenance/number of refuellings)
- Substitution of maintenance service partner

***Out of all listed measures, instalment and replacement of oil removal filters seems to be the most applied one among all the interviewed CNG operators.***

Some of the operators shared their particular experiences in dealing with improvement of the quality of CNG.

*"Yes we practiced adding additional filters up to the moment when the oil origin was not coming from the refuelling station anymore",* reported French operator.

*"Yes, we added oil removal system."*

Some of the CNG station operators reported to which brand/type of oil they switched:

*"Other qualities of Shell Corena and BP energol, partially special oils of manufacturer of station",* stated one German operator.

*"At this time we're conducting a trial with SAE 50 oil",* reported Portuguese operator.

As for the practices of changing the oil removal filters, couple of operators answered following:

*"We used to keep the same brand, but we have intensified oil filters replacement form 4,000 to 1,500 hours"*

*"No switch, added Zander-Parker: TGH110/350CF-BA"*

***For those operators that have applied some of the aforementioned measures, results on the improved CNG quality have been immediately anticipated from their customers.***

***However, none of the CNG Operators reported that they've been conducting a quantitative measurement of CNG quality improvement.***

For the purpose of addressing the economic impact of the applied measures for reducing the level of contaminants in CNG in the questionnaire, several dedicated questions have been posed. The ultimate purpose of those questions was to help to identify the increase of operational costs, stated in EUR/Nm$^3$ of CNG sold.

Unfortunately, none of the interviewed CNG refuelling station operators answered these questions. Addressed issue, could be considered as the room for further improvement.

## 9.2. CNG equipment producers

Even though initial interest in the questionnaire designed for the CNG equipment producers was rather significant, only few of them answered to the questionnaire eventually. Most keen on providing their experiences were stakeholders from Germany.

The number of CNG compressors that are being sold and installed all over the Europe, from the stakeholders that provided the answers, is more than hundred and those compressors were sold mainly in Germany and Switzerland. Average age of the compressors that are being delivered to the market is 5 years and the most common type of the compressor is reciprocating, equipped with oil free cylinders and the drive unit.

### 9.2.1. Oil

***According to the maintenance book, CNG station operators should perform maintenance of the CNG equipment after 2,000 operating hours, in case if the non-oil lubricated cylinders are applied.***

From the answers obtained, lubricating oil for the compressors that is recommended to the operators of CNG is usually the one that is being produced by the CNG compressor producers, whenever there is an option for that.

***None of the interviewed CNG compressors producers have reported that they've been asked by the CNG station owners/operators for the maximum allowed content of oil at the CNG refuelling station nozzle level before commissioning.***

An interesting observation comes out the survey, which is the fact that usually CNG equipment producers offer CNG stations with standard features which doesn't include oil removal filters by default.

In case if customer order originally equipped station with the oil removal filters, than usually following brands are being bolted on: MF, SMF, AK, at least in the case of stakeholders that provided the answers.

Similarly to the practice of not including the oil removal filters in the standard edition of CNG compressors that are being sold, in case of adsorption vapour filters, same habit has been evidenced from the CNG equipment producers' side.

Out of all filter cleaning techniques that have been recommended to the users of CNG compressors, regular (and automatic) drain of oil collection prevailed.

While asking CNG equipment producers how frequently they recommend to their direct customers to perform oil filter cleaning and/or replacing procedures, some of the following answer have been collected:

*"Every 6'000 h or one year for SMF cartridge"*

*"Every 3'000 h or 6 months for AK cartridge"*

As for the heat exchangers, the producers also responded that compressor interstate stage heat exchangers are being designed and installed accordingly.

From the market research, it is evident that almost none of the CNG equipment producers equip their compressors with the on-board oil content identification devices: not at the inlet stage, nor at the outlet stage.

When it comes to making further recommendation to their customers on how they could advance in the oil level content assessment, none of the possible following techniques have been recommended by CNG equipment producers:

- Sampling
- Using precipitation solvents
- Using coalescing filters
- Using glass fibre filters
- Using hydrocarbon dew point analysers
- Using indicator tubes

One of the questions that were posed to the CNG Equipment Producers was addressing the maximum level of oil in CNG that producers declare to their customers if equipment is being properly used. Here is the example of one answer that has been obtained.

*"40 mg/Nm$^3$ max for oil-lub compressors, without filter down up to 0.2 ppm(w) with filter combination"*

### 9.2.2. Water

Water removal units and dryers are usually not installed as standard equipment feature, but if they are, then producers reported that they provide their own designed water removal unit/adsorption dryers.

Dew point that is declared by the CNG equipment producer if the equipment is being properly used is equal to -20°C at 250 bars, according to the one stakeholder's response.

### 9.2.3. Sulfur

***Even though CNG equipment producers admitted that they don't equip their compressors with sulfur measurement devices, they suggest to their customers that equipment should not be exposed to the natural gas that contains more that 5mg/Nm$^3$.***

### 9.2.4. Other CNG quality aspects

As for declared maximum level of CNG temperature that is delivered into vehicle CNG storage at the outlet nozzle of compressor, some of the producers declared that it is equal to +10°C delta to atmospheric temperature.

Interviewed CNG equipment producers have not declared that they had any complaints from their customers regarding appearance of the excessive level of contaminants in CNG.

CNG equipment producers have the power to influence their customers to improve the ultimate quality of the CNG by advising them to undertake whole bulk of tested measures, such as:

- Reducing the time span between regular control of the oil level in compressor (number of controls/number of refuellings)
- Changing the brand and type of oil for lubrication
- More frequent maintenance of the compressor (frequency of the maintenance/number of refuellings)
- Instalment/replacement of oil removal filters

- More frequent maintenance of the dryer (frequency of the maintenance/number of refuellings)

However, from the answers obtained, it is evident that the most common measure that has been promoted and anticipated by the CNG Equipment Producers is offering to the customers an option of adding oil removal filters.

On the side of lubricating oil that has been recommended by CNG compressors users, several brands were identified: Atlas Copco, Hiperfluid VG100 or VG150.

## 9.3. NGV OEMs

The most comprehensive outlook of the experiences regarding the presence of contaminants in the CNG from the NGV OEMs perspective was collected from one Scandinavian Bus OEM. Presence of the buses from the portfolio of interviewed buses maker is evident mainly on the Scandinavian market.

The bus OEM claimed that it doesn't have any preferred CNG equipment vendors which they recommend to their direct customers and/or to CNG station operators. Same goes to the potential recommendation of CNG compressors and lubrication oil producers. The only generic feature that the buses maker recommends to the customer is to use, whenever possible, compressors with the oil free cylinders and drive unit.

Since unfortunately there are no formal standards to lean on, regarding the content of oil limitation levels, sulfur and water in CNG beyond which NGV maker would not be obliged to accept engine warranty terms, only internal limitations to the quality of CNG and biogas have been settled.

The only recommendation that has been given was the one that reflected type and brands of the oil removal filters that are suggested to be installed on-board the vehicles in order to ensure a successful abatement of oil contamination in CNG.

When the question on previous experiences related to presence of contaminants and handling with the appearance of oil, water or sulfur in NGVs was posed, the following answer was received:

*"Yes, we had a problem with excessive level of contaminants in NGVs 10 years ago with biogas in Sweden. Particularly, fuel injectors were clogged and oil in gas tanks has been found. Luckily, problems of that kind has not been recorder ever since."*

Out of several possible measures to reduce the presence of oil in CNG, buses maker recommended to their direct customers to occasionally change the oil filter, being bolted on the vehicle, with improved and advanced one.

## 9.4. NGV fleet operators

In order to get more detailed perspective on the addressed CNG quality issue, dedicated questionnaire for the NGV fleet operators has been designed and launched.

Though only couple of stakeholders responded to the questionnaire, some interesting inputs have been received. Experiences have been collected from the Scandinavian and Adriatic representatives. Both representative fleets have on average 60 CNG buses in their operations.

In first case, average age of the fleet was 7.5 years, while in the second case only 3 years.

Buses that have been in operations were mainly following brands: MAN Lions City and Volvo B10BLE in the case of Scandinavian fleet and IVECO CITELIS 1.2 CNG, IVECO CITELIS 1.8 CNG in the case of Adriatic fleet.

Analysed fleet of NGV buses in Scandinavia is supplied over they own public refuelling station, even though compressors are owned by the gas companies (Gasum), while in Case of Adriatic NGV fleet, the one is supplied over the public refuelling station (including compressors) that is owned by the Municipal Gas Works Company.

*"In the start of the operation with the MAN-fleet (registered 2005-2009) we noticed excessive oil in the gas filter, but it was most likely from the production (pressure-test of the bottles)."*

While posing the question on how frequently those failures were recorded (number of failures per refuellings), Scandinavian partner answered following:

*"The exchange-interval of the filter had to be reduced from 30 000 km to 15 000 km."*

NGV fleet operators were asked whether they have maybe addressed and/or suggested some of the measures to the CNG station operators, but from the answers obtained, ***it seems that NGV fleet operators did not try to influence the CNG station operator in order to improve the quality of CNG that is being delivered at the refuelling station level.***

# 10. ANALYSIS OF THE OVERALL KEY SUCCESS FACTORS AMONG VARIOUS GROUPS OF STAKEHOLDERS

Based on the desktop research of expert literature and a comprehensive market research conveyed among following four different stakeholders group: CNG station operators, CNG Equipment Producers, NGV OEMs and NGV Fleet Operators, a thorough analysis of the **Key Success Factors (KSF),** on improving the quality of the CNG distributed has been developed and applied.

29 KSFs have been identified, which were furthermore benchmarked against two groups of the stakeholders, which are directly responsible for the quality of CNG: **CNG Station Operators** and **CNG Equipment Producers**.

Every particular KSF was evaluated based on following two parameters:

1. Impact on CNG quality
2. Ease of implementation (blend of the time necessary for the implementation and investment/cost that are likely to be incurred for the proper implementation)

Direct assessment of the level of application of the KSFs has been provided for the two groups of stakeholders, based on their *historical performance record*, for the CNG Operators and *action recommended* by the CNG Equipment Producer. The level of applications is then obtained as a relative performance against *Ease of implementation* criteria (meaning that, ease of implementation actually represents a maximum value for every particular KSF). Performance of every KSF has been weighted with the numbers in the range of 1 – 4, where 4 represent the best performance of the observed KSF.

Weights of the KSF performances have been obtained based on the number of responses from the surveyed group of stakeholders, which is elaborated in details in previous chapter. For instance, if only one quarter of the responders has declared that one of the observed measures has been occasionally applied while conveying their CNG contaminants mitigation activities, then this KSF has been valuated with 1 point (25% = ¼).

Red colours in the table represent a room for improvement, with regards to the level of application of each particular KSF.

Following table represents a summary of the evaluated KSF for both relevant groups of the stakeholders.

*Table 8 Key Success Factors influencing CNG quality and their application among relevant stakeholders groups*

| No | Key Success Factor | Impact on CNG Quality | Ease of implem. | Performance | Action recommended by the stakeholder |
|---|---|---|---|---|---|
| | | | | CNG Station Operators | CNG Equipment Producers |
| 1 | Setting the maximum level of oil at the commissioning stage | 4 | 4 | 1 | 2 |
| 2 | Monitoring and managing complaints regarding excessive content of oil, water or sulfur in NGVs coming from customers | 4 | 4 | 3 | 1 |
| 3 | Intensification of different maintenance measures [27] | 4 | 3 | 2 | 2 |
| 4 | Monitoring of the improvement of CNG quality after some of the measures have been adopted | 4 | 3 | 2 | 2 |
| 5 | Application of the several oil removal filters in sequence | 4 | 2 | 2 | 1 |
| 6 | Application of heat exchangers | 4 | 2 | 2 | 2 |
| 7 | Intensity of the oil level control in compressors | 3 | 4 | 3 | 2 |
| 8 | Measuring of the temperature level in CNG | 3 | 4 | 3 | 3 |
| 9 | Application of the oil removal filters | 3 | 3 | 3 | 2 |
| 10 | Application of the specific oil filter cleaning techniques | 3 | 3 | 2 | 2 |
| 11 | Frequency of oil filter cleaning and/or replacing procedures | 3 | 3 | 2 | 2 |
| 12 | Application of the advanced types of lubricating oils | 3 | 3 | 2 | 2 |
| 13 | Application of various techniques for maintenance of CNG equipment | 3 | 3 | 2 | 1 |
| 14 | Habit of changing the brand/type of oil | 3 | 3 | 2 | 2 |
| 15 | Choosing the right type of compressor | 3 | 2 | 2 | 2 |
| 16 | Intensity of the CNG compressor maintenance | 3 | 2 | 2 | 2 |
| 17 | Application of advanced types of oil removal filters | 3 | 2 | 2 | 2 |
| 18 | Application of the specific adsorption vapouring filters | 3 | 2 | 1 | 1 |
| 19 | Habit of changing the brand/type of oil removal filters | 3 | 2 | 2 | 1 |
| 20 | Measuring economic impact on business operations after some of the measures have been implemented | 3 | 2 | 1 | 1 |
| 21 | Understanding of various techniques for measurement of oil level in CNG | 2 | 4 | 2 | 1 |
| 22 | Application of the oil content measurements in CNG (at the compression outlet stage) | 2 | 3 | 2 | 1 |
| 23 | Measuring of moisture level in CNG | 2 | 3 | 2 | 2 |

[27] See Table 10 for a further breakdown of this composite KSF

*Table 8 (continued) Key Success Factors influencing CNG quality and their application among relevant stakeholders groups*

| No | Key Success Factor | Impact on CNG Quality | Ease of implem. | Performance | Action recommended by the stakeholder |
|---|---|---|---|---|---|
| | | | | CNG Station Operators | CNG Equipment Producers |
| 24 | Application of the water removal unit dryer | 2 | 2 | 2 | 2 |
| 25 | Measuring of sulfur level in CNG | 2 | 2 | 1 | 1 |
| 26 | Frequency of moisture level measuring in CNG | 2 | 2 | 1 | 1 |
| 27 | Application of the oil content measurements in natural gas (at the compression inlet stage) | 1 | 3 | 1 | 1 |
| 28 | Frequency of sulfur level measuring in CNG | 1 | 2 | 1 | 1 |
| 29 | Application of various techniques for measurement of sulfur level in CNG | 1 | 2 | 1 | 1 |
| | | Threshold | 79 | 55 | 46 |

A general conclusion could be drawn that the overall performance of CNG station operators, as a group of stakeholder responsible for CNG quality, is much better in taking the care about the CNG quality, comparing to CNG Equipment producers, based on the total threshold[28] of the evaluation points.

Out of 79 points for the *maximum theoretical performance*[29] (sum of all *Ease of Implementation* points), 55 points has collected CNG station operators (sum of all *Performance* points), while only 46 collected CNG Equipment producers (sum of all *Action recommended* points).

To better understand the significance of the different KSFs, four groups of priority KSFs have been identified and grouped in accordance to their impact on CNG quality, from level ++ for 1st priority down to level -- for the last, 4th priority. Based on the KSFs evaluation points from

[28] Treshold determines the overall CNG quality level and it is defined as a sum of performance points for every single KSF, within observed group of stakeholder.
[29] Maximum Theoretical Performance stands for hypotetical scenario in which the stakeholder has adopted all the KSFs at maximum performance

Table **8**, a Key Success factor Matrix has been designed, in order to facilitate a better understanding on the prioritization of the KSFs implementation.

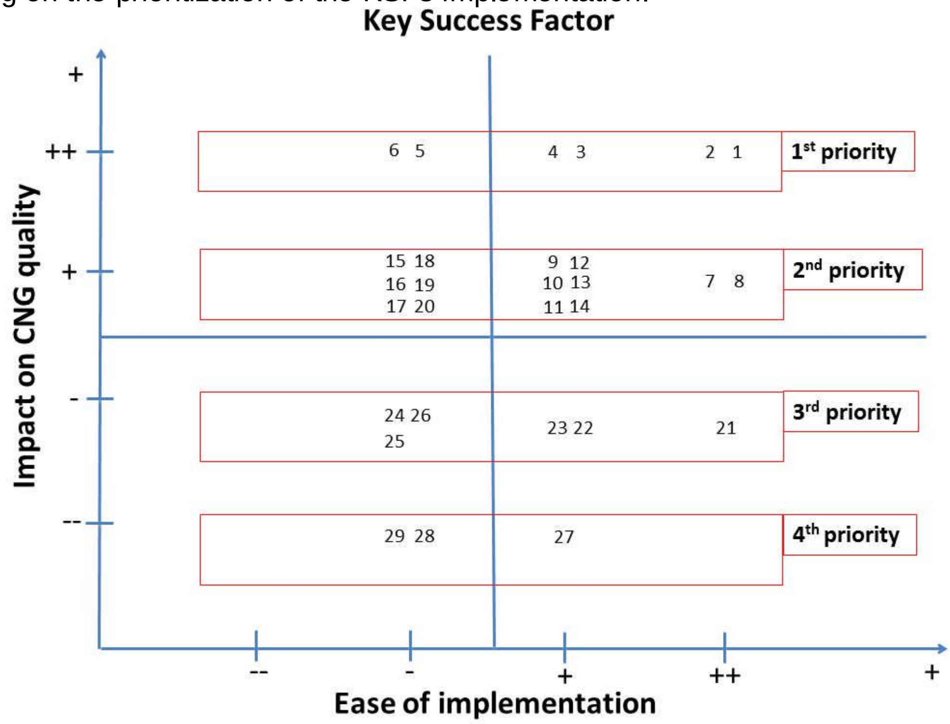



*Figure 23 Key Success Factors Matrix*

Further breakdown of the 3rd KSF *Intensification of different maintenance measures* is presented in the following table:

*Table 9 Intensification of different maintenance measures*

| No | 3 Intensification of different maintenance measures | Impact on CNG Quality | Ease of implem. | Performance | Action recommended by the stakeholder |
|---|---|---|---|---|---|
| | | | | CNG Station Operators | CNG Equipment Producers |
| 1 | Reducing the time span between regular control of the oil level in compressor (number of controls/number of refuellings) | 3 | 3 | 2 | 1 |
| 2 | Changing the brand and type of oil for lubrication | 3 | 3 | 3 | 2 |
| 3 | More frequent maintenance of the compressor (frequency of the maintenance/number of refuellings) | 2 | 2 | 1 | 2 |
| 4 | Instalment/replacement of oil removal filters | 3 | 3 | 3 | 2 |
| 5 | More frequent maintenance of the dryer (frequency of the maintenance/number of refuellings) | 2 | 3 | 2 | 2 |
| 6 | Substitution of maintenance service partner | 3 | 4 | 2 | 2 |

Previous table shows that though some of the measures in question are quite easy to implement and have rather significant impact on the CNG quality, in practice they have not been practiced by all actors. Pure evidence on that show following measures that could be applied/advised by particular stakeholder group:

- Reducing the time span between regular control of the oil level in compressor
- More frequent maintenance of the compressor (frequency of the maintenance/number of refuellings)

*Table 10 Understanding of various techniques for measurement of oil level in CNG*

| No | 21 Understanding of various techniques for measurement of oil level in CNG | Impact on CNG Quality | Ease of implem. | Performance | Action recommended by the stakeholder |
|---|---|---|---|---|---|
| | | | | CNG Station Operators | CNG Equip ment Produ cers |
| 1 | Oil consumption control method | 4 | 4 | 3 | 2 |
| 2 | With precipitation solvents | 4 | 4 | 1 | 1 |
| 3 | With coalescing filters | 4 | 3 | 3 | 2 |
| 4 | With glass fibre filters | 3 | 2 | 1 | 1 |
| 5 | With hydrocarbon dew point analysers | 3 | 2 | 1 | 1 |
| 6 | With Indicator tubes | 2 | 2 | 1 | 1 |

# 11. AN OUTLOOK ON THE CNG COMPRESSOR AND OIL PRODUCERS MARKET – EXPERIENCES FROM THE CUSTOMERS

As a special digest from the KSFs analysis, a reflection on the problems reported by the users in narrow correlation with the type of compressors and oil that has been applied will be elaborated in this chapter[30].

However, it has to be emphasized that there are many other elements that are influencing the quality of CNG. Thus, presented cases have to be perceived as insulated cases, and no general conclusions on the preferred compressors and/or oil types could be made.

Next table summarizes information about specific oil qualities that have caused problems in the vehicles in Sweden and some other EU countries. Table 11 and Table 12 compile information on compressors, oil qualities and filters used in refuelling stations today, and for facilities where customers previously had problems, and any action taken, and the Plants that had problems with oil in the vehicle.

*Table 11 Oil qualities causing problems*

| Oil | Problem | Measure | Country | Reference source |
|---|---|---|---|---|
| Mobil Glygoyle | City buses. Clogging of fuel system, such as valves and regulators. Probable cause: an additive (antioxidation agent) in the oil | Shifting to Mobil Gas-compressor Oil (oil still found in filters, but not as gooey. After that shift to Mobil SHC 527) | Sweden | Clementsson 2007 |
| Mobil Rarus 22 | City buses. Clogging (light brownish, rubbery) was in the injectors and the fuel filter. Depended on the additives in the oil, dioctyldiphenylamine | Shifting to Mobil SHC 527, problems disappeared immediately | Sweden | Clementsson 2007 |
| Mobil Glygoyle, viscosity from 420 down to 22 | City buses. Gooey mass in injectors and the manifold of injectors | Shifting to Mobil SHC 629, mass disappeared and buses are running well | Sweden | Clementsson 2007 |
| SAE 40 - Galp | Presence of the excessive amount of oil. 2 major complaints from the buses operator since the beginning of operations. | Changing the brand and type of oil for lubrication to SAE 50 oil.<br><br>Instalment/replacement of oil removal filters-Same brand but increased oil filters replacement form 4000 to 1500 hours. | Portugal | Market research 2013 |

[30] As a basis for comparison the following reference was used: Clementsson M, Held J (2007). "Olja i fordonsgas – regelverk, mätmetoder och filter" non-public SGC Report, 34 p.(Oil in vehicle gas – regulatory frameworks, test methods and filters)

*Table 11 (continued) Oil qualities causing problems*

| Oil | Problem | Measure | Country | Reference source |
|---|---|---|---|---|
| Shell Corona AP100 | Only 4 problems in 10 years with buses and trucks | Reducing the time span between regular control of the oil level in compressor, More frequent maintenance of the compressor, Instalment/replacement of oil removal filters - no switch added Zander-Parker: TGH110/350CF-BA | France | Market research 2013 |
| Bauer brand and Mobil SHC 1025 | Only few official complains about the quality of natural gas since 1996, mainly with buses. | Adding oil removal system and replacing Schandl's regenerating unit with Bauer dryer units. | Germany | Market research 2013 |
| Shell corena P 100/P 150 | Reported presence of oil: about 4 years ago (Opel) about 3 years ago (DB) | Changing to other qualities of Shell Corena and BP energol, partially special oils of manufacturer of station, Instalment/replacement of oil removal filters | Slovakia | Market research 2013 |

*Table 12 Summary of compressors, lubrication oil and filters used in refuelling stations today – Problems reported from customers and measures taken to mitigate the problem*

| Actor | Compressor | Lubricating oil | Measure taken: Filter |
|---|---|---|---|
| 1 | Ariel JGQ/2, oil lubricated piston compressor, in operation since October 2004 | Synthetic, Mobil SHC 527 | Four coalescing filter in series- 1 Special with Parker insert - 2 Parker - 1 Zaner Smallest mesh size 0,01 µm |
| 2 | Ariel, oil lubricated piston compressor | Mobil SHC 629 | Four coalescing filter in series Modell Zander G3 with high pressure filter Smallest mesh size 0,01 µm |
| 3 | Bauer, oil lubricated piston compressor | | Coalescing filter Adsorption drier/filter |
| 4 | Ariel, oil lubricated piston compressor | Mobil SHC 527 | Coalescing filter |
| 5 | Ariel, oil lubricated piston compressor | Mobil SHC 629 | Coalescing filter |
| 6 | Nuovo Pignone Cubogas 100 2 BVT, oil lubricated piston compressor, in operation since 1996 | Mineral oil, Turbine oil Texaco Regal EP 100 | Coalescing filter, Hahneman. Smallest mesh size 0,1 µm. Will be complemented with drier and additional filters for oil removal |
| 9 | LMF V 17 G, oil lubricated piston compressor, in operation since 2006 | Mineral oil, Shell Corena P 100 | Ultrafilter |

*Table 13 Summary of compressors, lubrication oil and filters used in refuelling stations today - No problems reported from customers*

| Actor | Compressor | Lubricating oil | Filter |
|---|---|---|---|
| 6 | Idro Meccanica TDE 20 Twin, oil greased hydraulic compressor, in operation since 2002 | Hydraulic oil for outdoor use, Texaco Rando HDZ 46, 68, 100 | Coalescing filter Smallest mesh size 0,2 µm Drier Particulate filter 50 µm, in the dispenser |
| 6 | Two Bauer CFS 28.21-75 DUO II, oil lubricated piston compressor, in operation since 2006/2007 | Synthetic oil, Hochleistungs Kompressoröl N26303-2 | Filter with combined oil cyclon Coalescing filter Smallest mesh size 0,1 µm |
| 8 | Bauer CFS 15.4-13 DUO II, oil lubricated piston compressor, in operation since 2006 | | |
| 9 | Nuovo Pignone, Type 2 AVTN/4, oil lubricated piston compressor, in operation since 2002 | Synthetic, Mobil Pegasus 1 | Oil trap Adsorption filter |
| 10 | Rix Model 4VX4BG-23.3, dry piston compressor, in operation since 1978 | Mineral, Q8 Schubert 68 | |

As regards monitoring of oil consumption in the compressor, there are different methods.

Distributors say they have no follow-up of oil consumption. What they do is they drain coalescing filter frequently, which is an important part of the maintenance.

Two participants state that they measure and note the added amount of oil to the compressor. If this increases, it may indicate a need for action. Another operator states that the monitor oil consumption by dipstick in the compressor.

Six of the twelve players say they follow up the oil consumption of the compressor by measuring the incorporation rate and quantity of oil which is separated by filters. An actor is weighing the removed oil, all other actors use volume measurement. Two players do this every week and an actor twice a week. A rough estimate of the concentration of oil in the gas can be made by taking the difference between input and removed amount of oil, and divide this by the amount of gas that crossed the plant. Distributors indicate that this method found that the oil content of gas is between 1-2 ppm.

Distributors say they consider removed amount of oil in coalescing filter and then calculates a ratio of removed quantities of oil per Nm3 of gas passed the plant for some time. The operator removes oil from each coalescing filter, after the compressor, separately. The oil is collected continuously and weighed with an accuracy of <0.5 g two to four times per month. Based on the supplied quantity of gas is determined in the separated oil mg/Nm$^3$. This is now around 1 mg / Nm$^3$ at the end of the filter, i.e. about 1 ppm separated oil in the gas. The accuracy of this method is considered to be 0.01 mg / Nm$^3$.

Another operator has installed a special measuring filter, Balston filter EU 37/25. It is weighed and then installed at the service stations. It is a coalescing filter. The filter is a long time (several weeks or longer) before it is taken out and weighed. The increased importance arises to recovered oil from the gas and a ratio calculated from the amount of gas passed through the facility. The operator indicates that this method found that they are at <1 ppm of oil in the gas.

# 12. RECOMMENDATIONS

## 12.1. Methods to minimize oil transfer to CNG

This subchapter is based on excerpts from the published report: Clementsson M, Held J (2007). "Olja i fordonsgas – regelverk, mätmetoder och filter" non-public SGC Report, 34 p.

There are several different methods to minimize the content of oil from the compressors to vehicle. The components that mainly affect the size of the amount of oil entrained with the gas at compression are:

- Compressor
- Oil Quality
- Filter

The best solution is, of course, if the issue of oil transfer from the compressor to the gas is included as an important point already in the procurement of the refuelling station. The client should make clear requirements on how high the maximum oil levels entrained by the compression may be, and the supplier should specify the method by which this is maintained. A reasonable requirement could be that the facility will be sized and equipped with filters so that the gas after the oil filters contains no more than 5 ppm of oil in aerosol form or lower. This is a level that can be achieved with effective coalescing filters.

That the refuelling station is properly built and designed is the first important part in order to keep a low level of concentration of oil that is entrained with the compressed gas. The second but equally important piece is the operation and maintenance, in particular compressor maintenance and frequent emptying of coalescing filters.

Dry compressors means that less oil is entrained with the gas compared to oil-lubricated ones. But even dry compressors have some oil leaks. The mechanical status of the compressor has a major impact on how much oil is entrained with the gas. It is important to follow the compressor manufacturer's instructions and recommended service intervals, regarding e.g. valves and piston rings, to minimize the amount of oil that can be entrained in the gas.

Either mineral oil or synthetic oils is used as lubricant or hydraulic oil in the compressor. Compressors should only use oils that are recommended or approved by the manufacturer. One way to reduce the amount of oil that comes with the gas is to use synthetic oil of polyglycol type. This type of oil leads to little or no oil in the vapour phase, and it is, as mentioned, oil in the vapour phase that is most difficult to remove. With mineral oil, a large proportion of the entrained oil will be present in the vapour phase. According to the trials in the U.S., the proportion of oil in the vapour phase corresponded to 81% of the total at 75 °C and 56% of the total at 37 °C. It should be noted that Mobile Glygoyle 22 that caused problems with clogging of the buses in Sweden is an oil of polyglycol type. It is important to note that mineral oil and oil of polyglycol type do not mix. In the case of an oil change, the system must be rinsed out thoroughly with oil that is compatible with both oil types.

The oil that is entrained with the gas is partly in the form of aerosols, partly in the form of vapour. At high pressures, oil is absorbed/dissolved by the gas. Aerosols may be removed by coalescing filters and oil in the vapour phase may be removed by adsorption filters. Aerosol oil after the compressor is in the range of 0.01 to 0.8 µm.

For oil lubricated compressor two effective ways to minimize the concentration of oil in the vehicle has been identified. One is to use a polyglycol type and a series of at least two highefficiency coalescing filters. The second is to use mineral oil, with at least two high-efficiency coalescing filters in series and then an adsorption filter to remove the oil in the vapour phase.

Oil consumption in the compressor should be monitored continuously. Above all in order to monitor the functioning of the compressor, since increased consumption indicates a need for service, but also because monitoring the input and removal of oil gives a rough estimate of how much oil is entrained with the gas. By dividing the amount of gas through-put, a value is obtained regarding the amount of oil per normal cubic meters of gas. The oil can either be weighed or measured as a volume in the follow-up.

It is not possible to conclude, based on the information collected in this study, how often a follow-up measurement of the oil content should be made. This is something that every refuelling station owner / operator needs to self-assess according to their refuelling station and age, load and possible remarks/complaints from customers about problems with oil in the gas.

## 12.2. Recommendations for CNG Station Operators

The CNG station operators play the most significant role in ensuring the uncompromised quality of the CNG, among other groups of the stakeholders. Market research that was conducted for the purpose of this best practices collection, shows that even though CNG station operators are by far the best contributor for the CNG quality, there is still a room for improvement in case of many addressed Key Success Factors (KSF). Recommendations on the practical steps in descending order, based on the impact and gap identified, are presented in continuation.

*Setting the maximum level of oil at the commissioning stage (KSF1)* is one of the most trivial measures that can be applied in defining the CNG quality, but it seems that it has not been practiced as it could have. The underlying reason for such a modest application of this measure might be found in the fact that level of understanding among CNG station operators at the time of CNG facility commissioning was not so high, while on the other hand side; there were no clear guidelines how to ensure the minimum level of oil in CNG. Argue that goes in favour of this measure is fact that it is quite easy for the new CNG station operators to request the maximum level of oil in CNG from vendors. However, trade off with the price premium for such equipment should be taken into account. For the existing users, this measure seems not to be relevant.

While conducting the market research, an interesting finding on the *monitoring and managing complaints regarding excessive content of oil water and sulfur in NGVs (KSF2)*, has been found. This measure is certainly one of the easiest that might be applied from the list, but it turned out that it has not been applied as it could have. For the CNG station operators it is important to establish a systematic approach in collecting and managing complaints from their customers in order to maintain and eventually raise the quality of their services.

In usual daily operation of the CNG station operators, clear *understanding of the different types of the practical measures (KSF3)* is of vital importance to the CNG quality. In that sense, six different KSFs have been identified, as follows:

1. Reducing the time span between regular controls of the oil level in compressor
2. Changing the brand and type of oil for lubrication if necessary
3. More frequent maintenance of the compressor
4. Additional instalment/replacement of oil removal filters
5. More frequent maintenance of the dryer
6. Substitution of maintenance service partner

It seems that a respective group of the stakeholders is very much familiar with the notion and impact of the addressed group of measures on the CNG quality. However, additional attention could be stressed to the 1st, 3rd, 5th and 6th group. Thus CNG station operators are invited to refer to the chapter 10 in order to get more details on this subject matter.

One of the KSFs from the 1st priority group that is vital for ensuring the perpetual quality of the CNG is certainly a *monitoring of the improvement of CNG quality*, *after some of the measures have been adopted (KSF4)*. From the market research, a clear evidence on the potential room for improvement with this measure has been recorded.

Aforementioned KSFs can be easily implemented and have rather high impact on the overall CNG quality, thus all the CNG station operators that have not paid attention to them yet are encouraged to consider adoption of this measures in their future operations.

Additional measures that also make the most significant impact on CNG quality, such as *application of several oil removal filters in sequence (KSF5)* and *application of heat exchangers (KSF6)* have been appropriately applied by the CNG station operators.

Best practices shows that the *optimal number of the oil removal filter* should be at least equal or greater to the number of compression stages, which is in practice equal to 2-3. As for the *maximum distance between compressor outlet valve and very last filter* being installed in sequence, it is recommended that it does not exceed couple of meter meters, because aerosols have a tendency to turn into vapour state after that distance, under the condition that the gas has cooled down to at least 30 degrees at that point. Market research findings show that this distance was not greater than 5 meters on average.

Regarding *heat exchangers (KSF6)*, it seems that CNG stations are well equipped with them, mainly after the every compression stage.

Further improvement could be made with the measure related to *intensity of the oil level control in compressors (KSF7)*. The results from the market research show that CNG station operators practice this measure on a weekly basis. However, very few of them reported that they put this practice in correlation with the volume of CNG sales and/or compressor's working hours.

*Measuring of the CNG temperature (KSF8)* is also vital to maintain the premium quality of the CNG. Best practices shows that temperature of the CNG should not exceed 10 - 15 centigrade of the surrounding temperature.

*Application of the oil removal filters (KSF9)* has been anticipated and practiced satisfactory among the CNG station operators. Dominantly, following brands/types of the filters are being used:

Coalescing Filters – Parker, Zander-Parker: TGH110/350CF-BA, Etha Filter: E4520RVF10, E24RVF10, E24100SMA, Etc.

Though the CNG station operator practices additional instalment of the oil filters, they are not familiar with the *technical specifications of the filters*, such as mesh size, which is actually of vital importance. Favourable mesh size of the filters should comply with the span between 0,01-0,1 µm.

*For the appropriate use of the filters, two parameters are important.* First one is application of the various oil filter cleaning techniques *(KSF10)*, and second one is frequency of the oil filter cleaning/replacing procedures *(KSF11)*. More specifically, there are three techniques that define appropriate filter cleaning: manual purge, condensate removal and replacement. Frequency of the oil filter cleaning depends on the local quality of the natural gas and overall quality of the local CNG refuelling station. As a general rule, it is highly recommended that CNG station operators perform quality control/cleaning of the filters at least every 250 hours, while after 1,000 operating hours it highly recommended to consider replacement of the filers.

In case that CNG station operator records higher level of oil contaminates in the CNG compared to the allowed/expected, one of the measures that could improve the processes is certainly a *consideration on lubrication oil substitution (KSF12)*. Some experiences on the various performances of different types of lubricating oils have been presented in the chapter No 11.

The key success factor that has been barely recognised among CNG station operators as a potential driver for the CNG quality improvement is a *practice of the CNG equipment* *maintenance performance (KSF13)*. In that sense, there are three general options that might be considered relevant. First one is to have a specialised in-house dedicated expert that is being appointed as a maintenance service provider. This option could be appropriate if CNG station operator has several refuelling stations in their portfolio and demand for having an in-house expert is justified. Two other options are to have contracted outsourced partners, both that are liaised/recommended by the vendor and/or independent one.

For the CNG station operator it is very important that complies with the *regular service intervals* that have been proscribed by the CNG compressor vendor.

A significant gap has been recorder in actual *application of the specific adsorption filters (KSF18)* at the compressors. Thus, CNG station operators are encouraged to refer to the chapter 8.1.2 where this subject is profoundly elaborated.

The research among various groups of EU stakeholders, particularly among the leading one, has shown that *understanding of the various techniques for the measurement of oil level in CNG (KSF21)* could be further improved. In that sense, the CNG station operators were asked which of the techniques, among possible one (oil consumption control method, with precipitation solvents, with coalescing filters, with glass fibre filters, with hydrocarbon dew point analysers and with indicator tubes) they have applied. Almost all of them responded that sampling technique was applied, while just few of them referred to the coalescing filters. This could be further improved and the CNG station operators are cordially encourage to study and apply other options that are presented in details in chapter 5.3

At the same time, response rate on the actual *application of the oil content measurements in CNG (KSF22)* was much better.

As for the *measuring and treating the moisture in CNG (KSF23)*, findings are rather satisfactory; meaning that CNG station operators really perform moisture level measuring in CNG and application of water removal unit dryers is rather high.

Major concern in ensuring the CNG quality regarding minimization of the contaminants might be found in sulfur measurement and treatment. In that sense, survey results show that *understanding of the various sulfur level measuring techniques, measuring of the sulfur level in CNG as well as frequency of the sulfur measurement (KSF25) (KSF26)* has been rather non satisfactory. The reason for such a low performance of CNG station operators derives from the fact that sulfur itself is brought to the CNG station operator through the natural gas and currently there are no many sulfur removal technologies “off the shelf” available on the market. Also, sulfur has not been, until recently, an issue addressed by the end-users outside of Germany. This topic is reflected in the standards – only the German DIN 51624 standard required a level low enough to make it an issue among refuelling stations.

CNG station operators are not directly responsible for the level of sulfur in CNG and any additional purification will put an extra financial burden to the operator, which is not justified if there are no regulatory or customer satisfaction incentives in relatively short period of time, which the operator could feel in a short business terms (positive cash flow).

However, in the chapter 8.3 of this best practices report, some possible techniques of sulfur purifications are described, so CNG station operators are encouraged to consider them and apply accordingly.

## 12.3. Recommendations for CNG Equipment Producers

Similarly to the previous group of the stakeholders that has the most important role in ensuring a premium quality of the CNG, the CNG Equipment Producers are next to the first group of stakeholders that can play an important role in CNG quality provisioning. Even though they are not directly presented on the spot while customers are being served with CNG that has no standardised quality yet, by anticipating and putting in practice following two pillars of contribution: design and coaching, they can directly influence the quality of the CNG.

Design of the equipment of the right quality is quite a generic term and it does not mean a lot if this equipment is not appropriately exploited eventually. In this context, the collection of best practices is not meant to bring any advice to the manufacturer of CNG compressors, for instance, but instead, it should navigate CNG equipment designers and marketers in better understanding of identified gaps on the market which causes exploitation of lower quality CNG. If anticipated from another perspective, these gaps might serve as a baseline for readjusting current value propositions that are being offered on the market for the better of the CNG quality.

Based on the market research and gap identified, here are some recommendations on the overall design of CNG refuelling stations.

Maximum level of oil in CNG that vendor declares for the ones CNG equipment/compressors is certainly one of the features that sales the equipment. However, from the CNG station operators obtained was feedback that they were not requesting any limit on the maximum oil level in CNG. Thus, it is the CNG equipment producer's responsibility and it should be an interest to address this feature clear to the customer and to commit the resources for the achievement during the commissioning.

Integration of several oil removal filters in sequence could be considered as a standard offer to the new customers, and highly recommended to the existing customers at the time of regular maintenance.

Considering offering an adsorption vapouring filters as standard equipment would certainly help CNG station operators in making a right decision in dealing with CNG quality.

Coaching represents a significant pillar and kind of intangible value which might significantly contribute in increasing the ultimate quality of the CNG. With coaching, CNG equipment producer offers an advice to the CNG Station Operators at ordering, commissioning and after sales stage with a clear goal to maximize the quality of CNG.

In order to make this coaching/mentoring more effective, it is highly recommended that CNG Equipment Producers invest into sort of guidelines/manual for their customers in order to maintain a high degree of the CNG quality along the exploitation period, aside from the regular key success factor, which is actually a quality of the equipment itself.

The CNG Equipment Producers should find an interest to advise and encourage CNG station operators to establish a regular system of monitoring and managing complaints regarding excessive appearance of oil, water or sulfur in NGVs, which are reported from the end users (NGV users).

Part of the recommended guidelines should clearly address and advise on different type of the measures for improvement of the CNG quality that are presented in the

Table **8**, and particularly:

- Reducing the time span between regular control of the oil level in compressor
- Changing the brand and type of oil for lubrication if necessary
- More frequent maintenance of the compressor
- Instalment/replacement of oil removal filters
- More frequent maintenance of the dryer

One of the best practices that are certainly valuable to be mentioned is a recommendation to intensify an oil level control in compressor.

Special advice on filter cleaning techniques, as well as necessary frequency for mentioned undertaking would be appreciated. Some advice on preferable lubricating oils would help CNG station operators in raising the quality of CNG.

Some simulations of the economic impact on business operations would certainly help CNG station operators in making the right decisions on the CNG treatment.

Market research has shown that the CNG station operators are not adequately informed about the various oil level measurements techniques for CNG. Examples of techniques are described in chapter 5.3.

Similarly to the oil measurements techniques, very limited knowledge has been present on the market, among the CNG Station Operators on the sulfur and moisture. Thus, special part of the best practices is dealing with this as described in Chapters 6 and 7.

# 13. CONCLUSIONS

Even if the European market has been using natural gas as an automotive fuel for many years, there is still a lack of harmonization regarding a vital aspect: natural gas quality, particularly when it comes to presence of oil, water and sulfur in it. However, the latest developments on EU regulatory level indicate that in coming future the CNG industry can expect following contaminants limitations: oil at 10-20mg/m$^3$, water at 1-30 mg/m$^3$ [31] and sulfur at 20 mg/Nm$^3$ for non-odorised.

There are four major groups of stakeholders that are directly responsible for the overall CNG quality: CNG station Equipment Manufacturers, CNG Station Operators, NGV OEMs and NGV Fleet Operators/Owners/Users, whereof the largest responsibility and impact is with the CNG Station Operators.

Even though control technologies exist for each particular contaminant, it turned out that the level of market application is still unsatisfactory. Two reasons for this can be identified: Lacking regulations regarding gas quality and construction of refuelling stations, and prohibitive costs (especially regarding sulfur removal).

For the purposes of this best practices collection, improvement of the CNG quality has been broken down into 29 key success factors. The gap analysis has shown that there is significant room for improvement in the existing business operations and that most of the measures are easy and not too costly to implement.

Sweden and Germany are leaders in Europe, both in terms of CNG dispensing technologies and application of gas quality control measures, providing a benchmark for the rest of the business. It has to be stressed that further market expansion relies heavily on maintaining good CNG quality at refuelling station level, and operators and regulators are encouraged to do their utmost.

[31] Corresponding to nationally adopted limits of a water dew point at 10, 20 or 30 °C at 200 bar

# 14. LIST OF REFERENCES


1. **American Society for Testing and Materials:** ASTM designation D 1142-95, “Standard Test Method for Water Vapour Content of Gaseous Fuels by Measurement of Dew-Point Temperature”., Philadelphia, USA, 1995
2. **Arrhenius Karin, Rosell Lars, Andersson Anders and Eriksson Hans:** Validation of test methods for renewable CNG: Development of measuring equipment for monitoring of purity and energy content of CNG, including determination of oil and water content, SP Technical Research Institute of Sweden, 2012
3. **Bukacek, R.F.:** “Equilibrium Moisture Content of Natural Gases”, Research Bulletin 8. Institute of Gas Technology, Chicago, USA, 1955
4. **California Energy Commission:** Evaluation of Compressed Natural Gas (CNG) Fueling Systems, Consultant report,, October 1999
5. **Clementsson M, Held J:** Olja i fordonsgas – regelverk, mätmetoder och filter, non-public SGC Report,, Swedish Gas Technology Centre 2007
6. **Czachorski M et al:** NGV Fueling Station Compressor Oil Carryover Measurement and Control, GRI-95/0483, Final Report, Institute of Gas Technology, February 1996
7. **DVGW Jahresrevue:** Erdgastankstellen: Öleintrag ins komprimierte Erdgas gewinnt zunehmenden Beachtung, sid 2-4, Energie/Wasser-Praxis, nr 12, 2006
8. **Forster Rüdiger:** Gas Quality Survey of German Natural Gas Fuelling Stations, International Gas Union research Conference, Paris 2008
9. **Forster Rüdiger:** Verfahrenstechnische Aspekte zum störungsfreien Betrieb von Erdgastankstellen, gwf-Gas/Erdgas 149 [2008], 2, pp. 100-105
10. **Forster Rüdiger:** GRI-Report GRI-95/0483: NGV Fueling Station Compressors Oil Carryover Measurement and Control, 1996; GRI-Report GRI-98/0228: Validation Testing of Gravimetric Method to Measure CNG Compressor Oil Carryover, 1998; Gas Quality Survey of German Natural Gas Fuelling Stations, Proceedings IGRC 2008, Paris
11. **George D. L., Ph.D., Burkey R. C.:** “Tests of Instruments for Measuring Hydrocarbon Dew Points in Natural Gas Streams, Phase 1”, Prepared for the Measurement Technical Committee of Pipeline Research Council International, Inc., Southwest Research Institute
12. **GERG Technical Monograph:** TM 14. Fortschritt-Berichte VDI, Nr 679, 2002
13. **Graf Frank, Riedl Jörg, Kerstin Kröger, Rainer Reimert, Jörg Meyer**, MONITORING CNG QUALITY IN GERMANY, WGC 2008
14. GTI (2009) “Pipeline Quality Biomethane: North American Guidance Document for Introduction of Dairy Waste Derived Biomethane into Existing Natural Gas Networks” (http://www.gastechnology.org/webroot/app/xn/xd.aspx?it=enweb&xd=1research cap/1_4environsci/dairywastebiomethane.xml)
15. **Herring Jack,** Determination of Hydrocarbon Dew Point Measurement in Natural Gas, Paper ID: 2008058,
16. **IS0 6326-1 (E) International Standard:** "Natural Gas-Determination of Sulfur Compounds" Part 1 "General Introduction", Genève, Switzerland, 1989
17. **Kolass Rolf:** MOSITURE MEASUREMENT IN NATURAL GAS, Michell Instruments GmbH, Friedrichsdorf, Germany; Chris Parker, Michell Instruments Ltd, Cambridge, UK
18. **Lampert JK, Farrauto RJ** (1997). “Catalyst development for methane emissions abatement from lean burn natural gas engines” Gas Research Institute Report GRI-97/0212
19. **Ly, H** (2002). “Effects of Natural Gas Composition Variations on the Operation, Performance and Exhaust Emissions of Natural Gas - Powered Vehicles”
20. **Novosel Dino:** Multi stakeholder on-line market research 2012, Questionnaire for the CNG Station Operators, Questionnaire for the NGVs OEMs, Questionnaire for the CNG Equipment Producers, Questionnaire for the fleet operators

21. **Oellrich, L.R., Althaus, K.:** “Relationship Between Water Content and Water Dew Point Keeping in Consideration the Gas Composition in the Field of Natural Gas”
22. **Pham Tuan Hai, Janssen Hans-Gerd, and Cramers Carel A.:** Determination of Sulfur Components in Natural Gas: A Review, Eindhoven University of Technology, Laboratory for Instrumental Analysis, P.O. Box 51 3, 5600 MB Eindhoven, The Netherlands
23. **Rolf Kolass,** Moisture measurement in natural gas, Michell Instruments GmbH, Friedrichsdorf, Germany Chris Parker, Michell Instruments Ltd, Cambridge, UK
24. **Seiff Hank,** Clean Vehicle Education Foundation, hseiff@cleanvehicle.ord, email 5 December, 2006.
25. **Svensson M (2011),** ”Utvärdering av svensk biogasstandard - underlag för en framtida revision” SGC Rapport 229 http://www.sgc.se/ckfinder/userfiles/files/SGC229.pdf (http://www.sgc.se/display.asp?ID=74&Typ=Rapport&Menu=Rapporter)
26. **Norén C, Thunell J (2002)**, “Svavelfri och svavelfattig odorisering av gasol och naturgas” SGC Rapport A26 http://www.sgc.se/ckfinder/userfiles/files/SGCA26.pdf
27. **Thompson Eric D.:** “Chilled Mirror Device For Water & Hydrocarbon Dew Point Determination”, Chandler Engineering, AGMSC 2003
28. **Verlag Beuth,** Normenausschuss Materialprüfung (NMP) im DIN, Fachausschuss Mineralöl- und Brennstoffnormung (FAM) des NMP, DIN 51624 Kraftstoffe für Kraftfahrzeuge – Erdgas - Anforderungen und Prüfverfahren, Berlin, Feb. 2008
29. **www.chandlerengineering.com:** Hydrocarbon Dew Point Analyzers
30. **www.draeger.se,** Dräger Short Team Tubes in alphabetical order
31. **www.parker.com**, Bulletin 1300-200/NA, J-series, High Pressure Compressed Gas Filters
32. **www.zander.de**, CNG-Adsorber for NGV Fuelling Stations

# APPENDIX 1: LIST OF CONTACTED STAKEHOLDERS

## CNG station operators

| CNG STATION OPERATORS | | |
|---|---|---|
| No | Country | Stakeholder |
| 1 | Austria | Salzburg AG |
| 2 | Austria | OMV Gas & Power GmbH |
| 3 | Austria | IQ Card Vertriebs GmbH |
| 4 | Belgium | Drive Systems |
| 5 | Czech Republic | čerpací stanice Benzina, Hviezdoslavova |
| 6 | Czech Republic | Konopeova ul., areál ČSAD |
| 7 | Czech Republic | Vrbenská 2, České Budějovice |
| 8 | Czech Republic | Havířov Suchá |
| 9 | Czech Republic | Hrušová |
| 10 | Czech Republic | Milovice |
| 11 | Czech Republic | Olomouc |
| 12 | Czech Republic | Ostrava - Ruská ul. |
| 13 | Czech Republic | Pardubice |
| 14 | Czech Republic | Praha 3 |
| 15 | Czech Republic | Praha 5 Jinonice |
| 16 | Czech Republic | Tábor |
| 17 | Czech Republic | Terezín |
| 18 | Czech Republic | Třebíč |
| 19 | Czech Republic | Znojmo - Dobšická ul. |
| 20 | Finland | GASUM |
| 21 | France | French Natural Gas Vehicles Association |
| 22 | France | GNVERT |
| 23 | Germany | Air-LNG |
| 24 | Germany | Enercity Contacting |
| 25 | Germany | erdgas mobil GmbH |
| 26 | Germany | Schandl GmbH |
| 27 | Italy | Assogasmetano |
| 28 | Italy | Eni |
| 29 | Netherlands | OrangeGas |
| 30 | Netherlands | CNG Net |
| 31 | Poland | PGNiG SA |
| 32 | Poland | ORFEMET |
| 33 | Poland | Biogaz Inwestor Sp. z o.o. |
| 34 | Poland | Górnośląski Zakład Obsługi Gazownictwa |
| 35 | Portugal | Cloud Energy |
| 36 | Portugal | Transportes Urbanos de Braga |
| 37 | Portugal | Galp Energia |

| | | |
|---|---|---|
| 38 | Slovakia | |
| 39 | Spain | Gas Natural Fenosa |
| 40 | Spain | Ham Criogénica |
| 41 | Sweden | E.ON Gas Sverige AB |
| 42 | Sweden | FordonsGas Sverige AB |
| 43 | Sweden | Swedish Biogas International AB |
| 44 | Sweden | AGA Gas AB |
| 45 | Sweden | E.ON Gas Sverige AB |
| 46 | Sweden | FordonsGas Sverige AB |
| 47 | Sweden | Öresundskraft |
| 48 | Sweden | Lunds Energi |
| 49 | Sweden | Svensk Växtkraft |
| 50 | Switzerland | Gasmobil AG |
| 51 | Switzerland | EWB |
| 52 | Switzerland | Cluttergasmobil AG |

# NGV fleet operators

| NGV FLEET OPERATORS | | |
|---|---|---|
| No | Country | Stakeholder |
| 1 | Croatia | Public Transport Company ZET |
| 2 | Finland | HELB |
| 3 | France | Trace-Colmar |
| 4 | France | Vitalis |
| 5 | France | TBC |
| 6 | Germany | RBA Regionalbus Augsburg GmbH |
| 7 | Germany | Saarbahn |
| 8 | Germany | Bayer |
| 9 | Germany | BSR (Berlin Waste Management) |
| 10 | Italy | ATAC Roma |
| 11 | Italy | ATAF |
| 12 | Netherlands | Rotra Forwarding Logistics |
| 13 | Netherlands | Simon Loos logistics |
| 14 | Portugal | Transportes Urbanos de Braga |
| 15 | Spain | TMB |
| 16 | Spain | EMT Madrid |
| 17 | UK | GEODRUID |
| 18 | UK | Coca Cola |
| 19 | Sweden | Keolis Sverige AB |
| 20 | Sweden | Veolia Transport Sverige AB |
| 21 | Sweden | Nobina Sverige AB |
| 22 | Sweden | Arriva Sverige AB |
| 23 | Sweden | Skånetrafiken |
| 24 | Sweden | Västtrafik |
| 25 | Sweden | Östgötatrafiken |
| 26 | Sweden | AB Storstockholm lokaltrafik (SL) |

# CNG equipment producers

| CNG EQUIPMENT PRODUCERS | | |
|---|---|---|
| No | Country | Stakeholder |
| 1 | Canada | IMW |
| 2 | Germany | Schandl |
| 3 | Germany | Bauer Kompressoren GmbH |
| 4 | Ireland | EMERALD GAS |
| 5 | Italy | SAFE |
| 6 | Italy | FORNOVOGAS |
| 7 | Italy | Idromeccanica |
| 8 | Italy | DRESSER Italia |
| 9 | Italy | BRC |
| 10 | Italy | SICOM |
| 11 | Italy | The Natural Gas Srl |
| 12 | Italy | ASPRO |
| 13 | Switzerland | GreenField AG |
| 14 | Sweden | Ariel |
| 15 | Sweden | Processkontroll AB |
| 16 | Sweden | NPS Service AB |
| 17 | Sweden | Dresser Wayne |

# NGV OEMs

| NGV OEMs | | |
|---|---|---|
| No | Country | Stakeholder |
| 1 | Germany | Mercedes-Benz Special Trucks |
| 2 | Germany | MAN |
| 3 | Germany | DaimlerChrysler AG |
| 4 | Germany | MAN Nutzfahrzeuge AG |
| 5 | Italy | Fiat Group Automobiles S.p.A. |
| 6 | Poland | Solbus Bus Factory |
| 7 | Spain | IVECO SpA |
| 8 | Sweden | Volvo Cars |
| 9 | Sweden | Volvo AB |
| 10 | Sweden | Volvo AB |
| 11 | Sweden | Volvo Bus Corporation |
| 12 | Sweden | Scania |
| 13 | Sweden | Neoplan |
| 14 | Sweden | Volkswagen |
| 15 | Sweden | IVECO SpA |
| 16 | Sweden | Volvo Cars |
| 17 | Sweden | Volvo AB |
| 18 | Sweden | AFV Alternative Fuel Vehicle |
| 19 | Sweden | Scania |
| 20 | Sweden | MAN |

# APPENDIX 2: QUESTIONNAIRES

## CNG Equipment Producers

1. What's the name of your brand and how many CNG stations equipped with your equipment are currently in operation on the European market?

2. What is the geographical breakdown of CNG stations that are equipped with your equipment (number of stations in 10 largest EU markets per Country)?

3. What is the average age of your CNG stations on the EU market?

4. What percentage of CNG stations (out of all) are delivering both CNG and Bio Methane with your equipment?

5. What type of compressors do you mostly sell?
   - o Reciprocating
   - o Hydraulic

6. What type of compressors regarding lubrication are you mostly offering to the market?
   - o Oil lubrication of both the engine and drive unit
   - o Oil lubrication of the drivers, but oil-free cylinder
   - o Oil free cylinders and drive unit

7. According to the maintenance book, how frequently should CNG station operators perform maintenance of the CNG equipment (after how many operating hours or Nm3 of CNG sold)?

8. What brand and type of oil do you recommend to the users for lubrication of compressors?

9. Have you ever received a request from customer in terms of a ceiling content of oil in CNG (what was the level in mg/m3)?

10. Do you equip your CNG compressors with some kind of oil removal filters?

11. What brand and types of oil removal filters are bolted on your CNG compressors?

12. What is the number of oil removal filters that are originally installed into sequence?

13. How far from the compressor (outlet valve) is the very last filter (m) mounted?

14. Do you equip your CNG compressors with adsorption vapour filters (for removing oil in vapour phase) and what brand/type do you use/recommend?

15. What is the minimum mesh size of the oil filters (in mm)?

16. What techniques of filter cleaning do you recommend to your customers, if any?

17. How frequently do you recommend to your customers to perform oil filter cleaning and/or replacing procedures (after how many operating hours or Nm3 of CNG sold)?

18. Do you equip CNG compressors with water removal unit/dryer (please specify the brand and the type)?

19. Do you equip CNG compressors with heat exchangers (please specify the brand and the type)?

20. Do you equip CNG compressors with oil measurement devices in order to allow determination of the oil content in natural gas (at the compression inlet stage)?

21. Do you equip CNG compressors with oil measurement devices in order to allow determination of the oil content in CNG (at the compression outlet stage)?

22. What additional type of oil techniques do you recommend to your customers?
- Sampling
- Using precipitation solvents
- Using coalescing filters
- Using glass fibre filters
- Using hydrocarbon dew point analysers
- Using indicator tubes

23. What is the maximum level of oil in CNG in mg/m3 that you are declaring to the customers with your equipment if properly used?

24. Do you set any limitations to the maximum level of sulphur in CNG (mg/kg) that should not be exceeded?

25. Do you equip CNG compressors with sulphur measurement devices in order to allow determination of the sulphur content in CNG (at the compression outlet stage) and what type of devices?
- We don't equip
- High resolution of UV detector (PFPD)
- Gas cromatography (Agilent 6890)
- Sulphur – specific detector (Model 355 sulphur chemiluminescent detector)
- Other

26. What is the maximum level of moisture in CNG (mg/kg) that you are declaring to the customers with your equipment if properly used?

27. Do you equip CNG compressors with moisture measurement devices in order to allow determination of the moisture content in CNG (at the compression outlet stage) and what type of devices?
- Mobile device (like Michell Cermax EX)
- Other

28. What is a declared maximum level of CNG temperature that is delivered into vehicle CNG storage at the outlet nozzle of compressor (°C)?

30. How frequent were those customers complaining (number of complaints/per filling, Country)?

31. What type of vehicles customers of your customers were using (personal cars, trucks, buses, others)?

32. Did you recommend any measures to improve the quality of CNG to those customers?

33. What kinds of measures have you addressed?

- Reducing the time span between regular control of the oil level in compressor (number of controls/number of fillings)
- Changing the brand and type of oil for lubrication
- More frequent maintenance of the compressor (frequency of the maintenance/number of fillings)
- Instalment/replacement of oil removal filters
- More frequent maintenance of the dryer (frequency of the maintenance/number of fillings)
- Other

34. Which brand and type of oil have you directed your customers to switch to(brand name, oil type: synthetic (poly-alpha-olefines), synthetic ester oils, mineral and semi-synthetic oils, hydraulic)?

35. Which brand and type of oil removal filters have you directed your customers to switch to (brand name, type: coalescing, etc)?

36. Do you have any evidence on improvement of CNG quality after your customers had undertaken some of the above mentioned measures?

37. What was the average reduction of oil in CNG in mg/m3 after implementation of those measures?

38. What is the average increase of operating costs on a yearly basis that you declare per undertaken measure of improvement?

| | EUR/(NM3 of CNG sold) |
|---|---|
| Reducing the time span between regular control of the oil level in compressor | |
| Changing the brand and type of oil for lubrication | |
| More frequent maintenance of the compressor | |
| Instalment/replacement of oil removal filters | |
| More frequent maintenance of the dryer | |
| Other | |

## CNG Station Operators

1. How many CNG stations in operation do you have in your portfolio?

2. Are those stations operated only nationally or do you operate internationally as well (please declare in percentages share of the internationally operated CNG stations, as well as country of your business origin)?

3. What is the average age of your CNG stations?

4. Do you supply Compressed Natural Gas, Compressed Bio Methane or both (please declare percentage of Bio Methane sold)?

5. What type of compressors do you mainly use?
    - Reciprocating
    - Hydraulic

6. What is the type of compressors regarding lubrication that are mainly installed at your CNG stations?
    - Oil lubrication of both the engine and drive unit
    - Oil lubrication of the drivers, but oil-free cylinder
    - Oil free cylinders and drive unit

7. What is the percentage of CNG stations that are equipped with high pressure storages that are providing fast filling options?

8. What is the brand name and model of compressors that are mostly used at your CNG station/s?

9. How frequently do you control level of the oil in compressors (after how many operating hours or Nm3 of CNG sold)?

10. How frequently do you perform maintenance of the CNG equipment (after how many operating hours or Nm3 of CNG sold)?

11. What brand and type of oil do you use for lubrication of compressors?

12. Did you request at purchasing stage from vendor the ceiling content of oil in CNG (for instance 5 mg/m3 which is manageable by filters)?

13. Are your CNG compressors equipped with any kind of oil removal filters?

14. What types of oil removal filter are bolted on your CNG compressors (please specify brand and type)?

15. What is the number of oil removal filters that are installed into sequence?

16. How far from compressor (outlet valve) is mounted the very last filter (in meters)?

17. Are your CNG compressors equipped with adsorption vaporing filters (for removing oil in vapour phase, please specify brand and type)?

18. What is the minimum mesh size of the oil filters that are bolted at the outlet stage of CNG compressors (in mm)?

19. What technique of oil filters cleaning have you adopted?

20. How frequently do you practice oil filter cleaning and/or replacing procedures (after how many operating hours or Nm3 of CNG sold)?

21. Is your CNG station equipped with water removal unit/ dryer (please specify brand and type) and the approximate final values reached like i.e. 30 mg/kg water content?

22. Is your compressor equipped with a heat exchanger (please specify brand and type)?

23. What is the average level of oil in natural gas in mg/m3 (pre - compression stage)?

24. Do you measure content of oil in natural gas (at the compression inlet stage)?

25. Do you measure content of oil in CNG (post-compression stage) and how frequently (after how many operating hours or Nm3 of CNG sold)?

26. How do you measure an oil level in CNG?
   - Oil consumption control method
   - With precipitation solvents
   - With coalescing filters
   - With glass fibre filters
   - With hydrocarbon dew point analysers
   - With Indicator tubes
   - Other

27. What is the average level of oil in CNG in mg/m3 (post-compression stage)?

28. Do you measure a level of sulphur in CNG and how frequently (after how many operating hours or Nm3 of CNG sold)?

29. How do you measure a level of sulphur in CNG?
   - With high resolution of UV detector
   - Gas cromatography
   - Sulphur – specific detector
   - Other

30. What is the average level of Sulphur in CNG (mg/kg)?

31. Do you measure level of moisture in CNG and how frequently (after how many operating hours or Nm3 of CNG sold)?
   - With mobile device (like Michell Cermax EX)
   - Other

32. What is the average level of moisture in CNG (mg/kg)?

33. What is the average temperature of CNG that is delivered into vehicle CNG storage at the outlet nozzle of compressor (°C)?

34. How do you convey maintenance activities of your CNG equipment?
   - With in-house dedicated experts
   - With outsourced service partner liaised with the vendor
   - With outsourced independent service partner

35. How many vehicles a year do you supply with the CNG (please include referent data for the last three years)?

36. What are your yearly sales of CNG in Nm3 in the last three years?

37. Have you ever had complaints regarding excessive content of oil, water or sulphur in NGVs coming from your customers?

38. How frequent those customers were complaining (number of complaints per fillings or number of complaints per Nm3 of CNG sold)?

39. What type of vehicles those customers were using (personal cars, trucks, buses, others)?

40. Did you undertake any measures to improve the quality of CNG?

41. What kind of measures have you undertaken?

- Reducing the time span between regular control of the oil level in compressor (number of controls/number of fillings)
- Changing the brand and type of oil for lubrication
- More frequent maintenance of the compressor (frequency of the maintenance/number of fillings)
- Instalment/replacement of oil removal filters
- More frequent maintenance of the dryer (frequency of the maintenance/number of fillings)
- Substitution of maintenance service partner
- Other

42. To which brand and type of oil had you switched (please declare brand name, oil type: synthetic (poly-alpha-olefines), synthetic ester oils, mineral and semi-synthetic oils, hydraulic)?

43. To which brand and type of oil removal filters had you switched (please declare brand name, type: coalescing, etc)?

44. Do you have any evidence on improvement of CNG quality after you had undertaken some of the above mentioned measures?

45. What was the level of oil in CNG in mg/m3 after implementation of above mentioned measures?

46. What was the average increase of operating costs on yearly basis per undertaken measure of improvement?

| | EUR/(NM3 of CNG sold) |
|---|---|
| Reducing the time span between regular control of the oil level in compressor | |
| Changing the brand and type of oil for lubrication | |
| More frequent maintenance of the compressor | |
| Instalment/replacement of oil removal filters | |
| More frequent maintenance of the dryer | |
| Substitution of maintenance service partner | |
| Other | |

If you are interested in receiving the final study report, please include your e-mail here:

## NGV OEMs

1. What's the name of your NGV brand and since when are you selling NGVs?

2. How many NGV models do you currently have in your OEM product portfolio?

3. In which European countries do you regularly sell NGVs from your product portfolio?

4. What are the dominant markets for your NGVs in terms of absolute number of sales (first five European countries)?

5. Do you have any preferred CNG equipment vendors which you are recommending to your customers and/or to CNG station operators?

6. Which brand/type of compressors do you usually recommend to the CNG station operators?
   - o Reciprocating
   - o Hydraulic

7. What type of compressors regarding lubrication do you recommend to your customers and/or CNG station operators?
   - o Oil lubrication of both the engine and drive unit
   - o Oil lubrication of the drivers, but oil-free cylinder
   - o Oil free cylinders and drive unit

8. Did you set up limitation levels for the content of oil, sulphur and water in CNG beyond which you don't accept engine warranty terms?

9. What brand and type of oil do you recommend for the lubrication of compressors to the CNG station operators?

10. What brand and type of oil removal to be installed on-board the vehicles have you found as most successful in the abatement of oil contamination in CNG? Which range of oil content are they able to handle?

11. Have you ever had complaints regarding excessive content of oil, water or sulphur NGVs coming from your customers? What have been the mechanical consequences/failures for the vehicles, if any?

12. How frequently were those customers complaining (number of complaints per fillings)?

13. What type of vehicles were those customers using (personal cars, trucks, buses, others)?

14. From which country have you received the most complaints?

15. What are the names of CNG station operators that caused troubles to your NGV customers regarding excessive appearance of contaminants (please specify: oil and/or sulphur and /or water in CNG)?

16. What brand/type/model of compressors were these CNG station operators using?

17. What brand/type/model of lubricating oil were these CNG station operators using?

18. What brand/type/model of oil filters were these CNG station operators using?

19. Do you have any clear mechanism to force CNG operators to improve their quality of CNG?

20. Did you undertake any measures to inspire CNG operators to improve their quality of CNG?

21. What kinds of measures have you addressed?
- Reducing the time span between regular control of the oil level in compressor
- Changing the brand and type of oil for lubrication
- More frequent maintenance of the compressor
- Instalment/replacement of oil removal filters
- Instalment of and/or more frequent service of the on board coalescing filters
- More frequent maintenance of the dryer
- Other

22. Do you have any evidence that some of CNG operators have implemented some of the recommended measures?

23. What was the average level of oil in CNG in mg/m3 after implementation of the above mentioned measures?

## NGV FLEET OPERATORS

1. What is the name of your Company?

2. In which country is your business located?

3. Do you operate nationally or internationally as well?

4. How many NGVs do you have in your fleet?
Buses
Trucks
Vans
Cars

5. What is the average age of your NGV fleet?

6. What is a predominant brand/model of NGV that you have in your fleet?

7. Do you have your own CNG refuelling station or do you use public CNG stations?

8. If you have your own CNG refuelling station, what brand, type and model of compressor is used and how old is the station?

9. Have you ever recorded excessive content of oil, water and/or sulphur in your NGV fleet that caused the problems in vehicle operations? What have been the mechanical consequences/failures for the vehicles, if any?

10. How frequently those failures were recorded (number of failures per fillings)?

11. What particular model of vehicles had the problem?

12. What brand/type/model of lubricating oil was used in CNG compressor before you had undertaken any action?

13. What brand/type/model of oil filters was used at CNG station before you had undertaken any action?

14. Did you undertake any measures to improve the quality of CNG or in case that you were not owning CNG refuelling station, did you try to influence your CNG provider?

15. What kind of measures have you practiced/addressed?

- Reducing the time span between regular control of the oil level in compressor
- Changing the brand and type of oil for lubrication
- More frequent maintenance of the compressor
- Instalment/replacement of oil removal filters
- Instalment of and/or more frequent service controls of the on board coalescing filters
- More frequent maintenance of the dryer
- Other