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Conversion of a natural gas pipeline to hydrogen transport and the effects of impurities on the hydrogen quality

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1 ABSTRACT

DNV supported N.V. Nederlandse Gasunie with the conversion of an existing natural gas pipeline to hydrogen transport in the Netherlands. This 12 km long 16-inch pipeline has been in operation for hydrogen transport since 2018. This paper will focus on the behaviour of contaminants found in the mentioned natural gas pipeline during the pipeline's transition from natural gas to hydrogen and their effect on the hydrogen quality. How the diverse contaminants are measured will also be part of this paper.

The contaminations found in natural gas pipelines are very diverse. A distinction is made between solids, liquids, and volatile/gaseous components. To avoid or limit the effect of natural gas impurities on the hydrogen transported through the repurposed pipeline, specific consideration will be given how the pipeline can be cleaned. An overview will be given on the steps that were undertaken to clean the pipeline before the actual transition took place. The effect of these measures is shown by presenting the concentration of various contaminants during and after the pipeline conversion from natural gas to hydrogen.

The protocol used for the transition from natural gas to hydrogen comprises five steps:

- 1. Pre-clean using cleaning pigs to remove loose dirt and liquids from the pipeline with natural gas.
- 2. Displace natural gas with nitrogen using a pig run to separate the natural gas from the nitrogen and preserve the pipeline under a low-pressure nitrogen atmosphere.
- 3. Carry out the necessary adaptions and/or replacements on the pipeline and performing necessary maintenance during a period in which the pipeline is maintained at low pressure filled with nitrogen.
- 4. Pig run cleaning under nitrogen atmosphere. Monitoring contaminants in nitrogen to test if the criteria to switch over to hydrogen transmission are fulfilled.
- 5. Displacement of the nitrogen by hydrogen using a pig to separate the nitrogen from the hydrogen.

Based on the gathered contaminants during the cleaning of this pipeline, some preliminary criteria that can be used during the cleaning process are:

- Liquids/solids/sludge; maximum 1 litre of material for pipe diameters up to 12 inches and up to 2 litres of material for pipe diameters lager than 12 inches (regardless of pipeline length).
- Hydrocarbons up to 1000 ppm.
- Water dewpoint < -8 °C @ 70 bar.

The results of the measurements carried out during the transition and after the transition show that the levels of contaminants are very low.

2 INTRODUCTION

Hydrogen as a general substitute for natural gas for heating, chemical feedstock and for power generation is new for the Netherlands. Completely new chains will have to be set up in which the supply of hydrogen is linked to the market - initially the industries that need hydrogen for high-temperature processes and as a 'green raw material' for artificial fertilizer, for example. In order to establish this new chain, a robust transport network must be in place in good time, as the 'backbone' for this new chain. A large part of this backbone will exist of former natural gas pipelines, see figure 1.

Hynetwork Services (part of Gasunie) has currently proposed a hydrogen specification for feeding into the hydrogen backbone, see table 1. This hydrogen quality specification is based on the input of stakeholders (market, producers, consumers) provided at the public consultation in 2020. During this consultation, three draft quality specifications were presented by Gasunie and were consulted by stakeholders. For this final specification the (sometimes) opposing interests of all stakeholders was taken into account. The Ministery of EZK in the Netherlands will be advised by Gasunie to adopt this specification and implement it into Dutch legislation and preferably align neighbouring countries in the pentalateral consultation. Similar hydrogen quality specifications have been proposed by the European Association for the Streamlining of Energy Exchange – gas, EASEE-gas [1].



Figure 1. Hydrogen backbone in the Netherlands (Hynetwork Services).

The first experience with the conversation of a natural gas pipeline to hydrogen transport was done with a relatively short natural gas pipeline between two chemical plants in the Netherlands. This 12 km long 16-inch natural gas pipeline was converted in 2018. This report describes the steps that were undertaken to clean the natural gas pipeline before the actual transition took place. Besides this, an overview is given for the behaviour of contaminants found in this natural gas pipeline during the pipeline's transition from natural gas to hydrogen and the effect on the hydrogen quality.

 Table 1. Indicative quality specification hydrogen backbone.

Component	Symbol	Unit	Minimum	Maximum
Hydrogen	H ₂	mole %	98	
Total sum of hydrocarbons including methane	C _x H _y	mole %		1.5
Total sum of inerts (nitrogen, argon and helium)	N ₂ , Ar, He	mole %		2.0
Oxygen	O ₂	ppm		10
Carbon dioxide	CO ₂	ppm		20
Carbon monoxide	СО	ppm		20
Total sulphur including H ₂ S	S	ppm		5
Formic acid	CH ₃ OOH	ppm		10
Formaldehyde	CH₂O	ppm		10
Ammonia	NH₃	ppm		10
Halogenated compounds		ppm		0.05
Water dewpoint	H ₂ O	°C @ 70 bara		-8

Source: Hynetwork Services, <u>https://www.hynetwork.nl/downloads</u>.

3 PIPELINE PREPARATIONS

3.1 BIDI cleaning pig

The existing 16-inch section of the natural gas pipeline was examined using internal Magnetic Flux Leakage (MFL) inspection in 2017. This is a widely used non-destructive testing method for the detection of (internal) corrosion and pitting in steel structures. Before this inspection the natural gas pipeline was cleaned by means of a bi-directional (BIDI) cleaning pig. Approximately 5 litres of sludge (not analysed) came along at that time (see figure 2). Sludge found in natural gas pipelines normally consists of a mixture of natural gas condensates, lube oil and glycol.

The cleaning with the BIDI-pig was performed with a nitrogen displacement to remove natural gas and successively with the MFL inspection, also with nitrogen displacement. After inspection the pipeline was placed under a nitrogen atmosphere of 2 bara. All parties involved were asked whether any problems were expected with regard to gas transport and pipeline integrity as a result of any residual substances present in the pipeline. Substances such as odorant, free water, black powder or aromatics. The expected small quantities will not lead to problems with respect to gas transport and pipeline integrity.

Figure 2. BIDI cleaning pig in receiver.

3.2 SEM-EDX analysis debris

DNV analysed relatively "dry" and "clean" debris that was found in some parts of the pipeline using a Scanning Electron Microscope (SEM). In a SEM an electron beam scans the surface of the material in a grid wise pattern. The electrons reflected or released by secondary emission are detected and captured point by point to form an integral image. Magnifications of 100,000 times are possible with a resolution on the order of one nanometre.

A representative portion of the debris was used to create a SEM sample (see figure 3). Recordings were made of the sample by means of so-called Energy-dispersive X-ray spectroscopy (EDX) with an accelerating voltage of 20 kV. EDX analysis provides images that give an idea of the chemical compositions of the areas. Dark areas contain many light elements of the periodic table, such as carbon and oxygen, while light areas contain heavier elements such as silicon and mercury. The magnifications used were between 200 and 500, depending on the type of sample and the nature of the contamination. In these images, numbers indicate the locations of point and plane analyses performed on the sample.

The detection limit depends on the element to be measured, the composition of the surrounding matrix and the accelerating voltage used. In general, it can be said that the detection limit is 0.1 to 0.5 mass %. The accuracy of the results also depends on element and matrix and is a few tenths of a percent to a few percent. EDX analysis is performed on a relatively small area. This means that local differences in chemical composition can have a relatively large influence on the analysis result due to inhomogeneity present in the sample. Through EDX the carbon content cannot be approximated with somewhat reasonable accuracy. Carbon contents are therefore estimated from experience, based on the relative size of the carbon peak in the EDX spectrum, or indicated with terms such as "much" or "little".

The debris was found to consist mainly of particles in the range of 100 μ m to several millimetres. The analyses show that the particles differ greatly from one another, see SEM-image in figure 4. Based on the composition, a number of categories can be indicated:

- Minerals: materials consisting mainly of relatively large amounts of oxygen (O) with silicon (Si) and possibly with aluminium (AI), calcium (Ca) and iron (Fe).
- Rust particles: recognizable as orange particles. Possibly of natural origin (ochre) or from oxidized steel.
- Iron oxide balls: these are formed during operations on steel that involve high temperatures such as welding and grinding. In the process, iron particles oxidize to iron oxide and solidify in the air to form round spheres.
- Chips of low alloy steel: mainly iron with small amounts of manganese and silicon. Sometimes the surface is slightly oxidized (up to a few dozen percent oxygen). Short particles curled about the longitudinal axis may be saw chips. Particles that spiral around the longitudinal axis are possibly drill chips.

No mercury (Hg) was found in the sample and the amount of sulphur (S) is low. Table 2 gives an overview of the average elemental composition of the debris.

Figure 4. SEM-image debris.

Table 2. Elemental composition debris.

Element	Symbol	Eleme	ental composition in mass%			
		Average	Minimum	Maximum		
Carbon	С	3.6	0.0	39.0		
Oxygen	0	26.7	5.3	50.0		
Sodium	Na	0.2	0.2 0.0			
Magnesium	Mg	0.5	0.5 0.0			
Aluminium	AI	2.9 0.0		47.0		
Silicon	Si	8.4 0.4		50.4		
Sulphur	S	1.6 0.0		18.3		
Potassium	К	0.5 0.0		5.0		
Calcium	Са	4.5 0.0		35.8		
Titanium	Ti	1.7 0.0		17.1		
Manganese	Mn	1.2 0.0		12.3		
Iron	Fe	48.0	1.8	91.7		

3.3 Gas analysis

In June 2018, the pipeline was already placed under a nitrogen atmosphere for several months, gas samples were taken in Tedlar sample bags and evacuated cylinders. These samples were analysed using a Thermo Scientific Trace 1300 Gas Chromatograph (GC). This GC is equipped with Mass Spectrometer (MS, type ISQ, single quadrupole) in parallel with a Flame Ionization Detector (FID). This analytical system was used to screen the samples for various organic and inorganic components (mass range 20 – 400 amu).

Screening of the gas samples revealed mostly hydrocarbons derived from natural gas condensates. Figure 5 provides an overview of a FID chromatogram. Relatively high concentrations of aromatics and cycloalkanes were found. The most important components are shown in table 4 including the concentrations in ppm (on a molar basis).

Figure 5. FID-chromatogram of hydrocarbons detected in nitrogen sample.

Table 4	BTEX	and	cycloalkanes	in	nitrogen samples.	
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Component	Symbol	Retention time (min)	Concentration (ppm)
Benzene	C ₆ H ₆	18.2	43.7
Cyclohexane	C ₆ H ₈	18.8	3.5
Methylcyclohexane	C7H14	22.8	5.1
Toluene	C7H8	24.9	17.2
Ethylbenzene	C8H10	30.6	3.3
p/m-Xylene	C8H10	31.0	6.9
o-Xylene	C ₈ H ₁₀	32.4	4.3

In order to perform a trace sulphur analysis, Single Ion Mode (SIM) of the mass spectrometer was used. The SIM analysis is based on 9 common sulphur components in natural gas. Table 4 gives an overview of the sulphur components and SIM analysis. Detection limits are in the single digit ppb range. The sulphur analysis revealed only the positive identification of the odorant tetrahydrothiophene (THT). This component was found in very high concentrations and well above the upper detection limit. So, an additional analysis for THT took place with an Agilent 490 micro-GC. A concentration of 15 mg/Nm³ (3.8 ppm) was found. This THT concentration is almost identical to the nominal concentration of THT in odorized natural gas, being 18 mg/Nm³.

Table 5 gives an overview of all substances present in the nitrogen the pipeline was filled with during several months at a pressure of 2 bara.

Component	Symbol	SIM mass (amu)	Retention time (min)
Hydrogen sulfide	H ₂ S	34	3.99
Carbonyl sulfide	COS	60	4.27
Methyl mercaptan	CH₄S	47	6.00
Ethyl mercaptan	C ₂ H ₆ S	62	8.95
Dimethyl sulfide (DMS)	C ₂ H ₆ S	62	9.73
Carbon disulfide	CS ₂	76	10.88
n-Propyl mercaptan	C₃H ₈ S	76	14.65
n-Buthyl mercaptan	C ₄ H ₁₀ S	56	21.25
Tetrahydrothiphene (THT)	C₄H ₈ S	60	27.11

Table 4. Sulphur components and SIM settings (RTX-1 5µm 60m x 0.53 mm analytical column).

Table 5. Components present in the nitrogen.

Component	DNV result	Unit
Carbon monoxide	<0.1	ppm
Carbon dioxide	1200	ppm
Ethane	0.6	ppm
Cyclohexane	3.4	ppm
BTEX	75.4	ppm
Other saturated hydrocarbons	40	ppm
Chlorine and organochlorides	*	ppm
Fluoride and organofluorides	*	ppm
Total sulphur (inorganic and organic)	5.5	mg S/Nm ³
Total silicon (including siloxanes)	<0.06	mg Si/Nm ³

*no organic chloride and -fluoride components detected.

4 NITROGEN PURGE

4.1 Introduction

Following an investigation into the residual products in the converted pipeline, which will eventually transport hydrogen, it was found that a number of components have high values, e.g. THT and carbon dioxide. These substances had been present in the nitrogen for some time at a pressure of approximately 2 bara. It was decided to purge with nitrogen at one side and to vent it on the other side of the pipeline. DNV was asked to monitor this process. For this purpose, on-line measurements were performed in July 2018.

4.2 Displacement

In preparation for displacing the pipeline content, a DN50 connection was made for injecting nitrogen (see figure 6). In addition, a DN100 vent stack approximately 4 meters high was installed at the other side of the pipeline for venting (see figure 7). This vent stack was equipped with a sample point. On this sample point, a water dew point sensor (Michell Easidew, measuring range -100 to +20 degrees Celsius) was mounted. The outlet of the sensor was equipped with a Teflon sample line (6 mm OD) to the measuring equipment. This equipment consists of a flame ionization detector (Thermo FID, model TG from the company Mess- & Analysentechnik GmbH) and an Agilent Technologies 490 Pro micro-GC. The water dew point sensor and the FID (measuring range 0 - 1000 ppm C_xH_y) were used to monitor the displacement process in real time. The FID was calibrated with a known concentration of propane (C_3H_8) in nitrogen; therefore, the hydrocarbons are presented as propane equivalents.

The measurement results were recorded with a Datataker DT80 (scan time 30 seconds). The micro-GC was equipped with two analytical modules for the measurement of C1 - C8 hydrocarbons and the odorant tetrahydrothiophene (THT, C₄H₈S), respectively. The micro-GC had an analysis time of three minutes. The detection limit is approximately 1 - 5 ppm (depending on the component).

To inject nitrogen, a nitrogen unit and a Coriolis flow meter from Well Service Group (WSG) was used. The WSG nitrogen unit has a maximum capacity of 1500 m3/h. In principle, it was advised not to let the nitrogen flow exceed 300 m³/h because of vibrations/pulsations and the expected noise level. The calculated theoretical velocity at the entry point DN50 will be around 40 m/s. At the exit point with DN100 diameter it will be around 10 m/s. The maximum stock of the nitrogen unit is 7800 kg of liquid nitrogen (-196 degrees Celsius @ 101.325 kPa). After evaporation, this is a maximum supply of approximately 6250 m³. With this, approximately four times the volume of the pipeline can be displaced at atmospheric pressure.

Figure 6. Nitrogen injection DN50, Coriolis flowmeter on the background.

Figure 7. Vent stack DN100.

4.3 Measurement results

In July 2018 the actual displacement started on a morning at 08:15 hours with a flow of approximately 500 m³/h of nitrogen (minimum flow WSG nitrogen unit). At 08:20 hours that morning, a valve at the exit point was opened to blow off the pipeline. The noise level at that time was about 75 dB. Over time, the pressure in the pipeline decreased and so did the noise level. The nitrogen flow was therefore gradually increased to 1000 m³/h and then to a maximum of 1500 m³/h within half an hour. The noise level remained very acceptable at the exit point at a level of approximately 65 dB. The noise level at the injection was well above 80 dB (whistling noise).

The measurement results are shown graphically in figure 8 and 9. At the start of the measurements, the water dew point (-20 °C), hydrocarbons (300 ppm) and THT content (15 mg/m³) were stable. From 08:55 hours these values suddenly increased. Around 9:40 hours the total content of the pipeline was purged with new nitrogen. This was very clearly noticeable because of the rapid decrease in the hydrocarbons and the THT concentration. The hydrocarbon concentration dropped from over 600 ppm to approximately 10 ppm in a very short time. The THT concentration decreased from approximately 30 mg/m³ to below the detection limit of the micro-GC. Around 10:40 hours, 3000 m³ of nitrogen had been consumed and at 11:30 hours the nitrogen supply was exhausted. The hydrocarbon content at that time was 3.2 ppm and the water dew point was -27 degrees Celsius (511 ppm).

Figure 8. Total hydrocarbons and water dewpoint at exit point.

Figure 9. Micro GC results at exit point.

In consultation with Gasunie it was decided to have an extra supply of liquid nitrogen for a second displacement action. This second displacement started at around 14:30 (note: this meant that the fresh content of the pipeline was stationary for about 3 hours!). Figure 10 shows the hydrocarbon concentration and water dew point of the second displacement. No hydrocarbons were detected with the micro-GC (below detection limit). At the start of displacement, a small peak of hydrocarbons of 20 ppm was observed after which the concentration stabilized around a value of 7 ppm (water dew point -24 °C). At 15:09, approximately 1500 m³ of nitrogen had been injected. Then at about 15:13 a second peak was observed in the hydrocarbon content, after which the content slowly decreased to a concentration of approximately 3 ppm (water dew point -26 °C). At 15:27 a Tedlar sample bag was filled with a gas sample for analysis by GC/MS in the laboratory of DNV in Groningen. Immediately afterwards, the valve at the exit point was closed to pressurize the pipeline. At approximately 16:10 the second supply of nitrogen was exhausted, and the pipeline was stored with an overpressure of 0.9 bar nitrogen.

Table 6 summarizes the GC/MS results of the nitrogen sample after the second displacement. Here, a screening (mass range 20 - 400 amu) and a so-called SIM method for common sulphur compounds in natural gas was performed in the low ppb range.

Figure 10. Total hydrocarbons and water dewpoint at exit point (continued).

Table 6. Concentrations after (second) nitrogen displacement.

Component	Symbol	Concentration in ppb
Hexanes	C ₆ H ₁₄	100
Benzene	C ₆ H ₆	200
Xylenes	C8H10	300
Octanes	C8H18	100
THT	C4H8S	17.5

4.4 Commissioning

In October 2018 the former natural gas pipeline was commissioned. The purpose was to deliver an oxygen free and clean pipeline. From the exit point nitrogen was injected using the WSG facility. During the modification of the pipeline two foam pigs were placed at the entry and exit points in order to minimize the entry of air and moisture. These foam pigs were removed during the first nitrogen purge. The foam pig from the exit point travelled through the entire pipeline during this purge to the entry point. This foam pig was clean and dry, see figure 11. The pipeline was filled with nitrogen to a pressure of 4 bara and subsequently blown off. This was repeated two times. The residual oxygen concentration was below 1 ppm. A total of approximately 16,000 m³ nitrogen was used during commissioning.

Figure 11. Foam pig, on the left front side and on the right back side.

4.5 Observations 2018 – 2022

At the end of 2019, a unique situation presented itself whereby the hydrogen supply would be shut down for two weeks due to maintenance work in the period from November to December 2019. This former natural gas pipeline had been commissioned over a year earlier to transport hydrogen. Upon completion of this pipeline in October 2018, it was determined that (minimal) contaminants such as THT and aromatics were present. However, this does not lead to problems under flowing conditions. To check the situation after more than a year of operation, it was requested by Gasunie to take samples under flowing (entry and exit) and non-flowing conditions (exit).

Samples were analysed in the same way as described in paragraph 3.3. The first sampling was conducted together with UK's national metrology institute, National Physical Laboratory (NPL) to gain knowledge from converted natural gas pipelines. This sampling was performed in December 2019 at the exit point with no flow in the pipeline. The second sampling (under flowing conditions) was performed in May 2020. Thereby, a first sampling was performed at the entry point and subsequently at the exit point. In April 2022 sampling at the entry and exit point was repeated under no flow conditions and low pressure in the pipeline. The results are presented in table 6 and 7.

Table 6. Results DNV 2018 – 2022.

Component	2018	2019	2020	2020	2022	2022	Unit
	Exit	Exit	Exit	Entry	Exit	Entry	
	no flow	flow	flow	flow	no flow	no flow	
Carbon dioxide	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	ppm
Oxygen	-	<1.0	8	<1.0	3	<1	ppm
Nitrogen	-	895	1444	1423	601	804	ppm
Ethane	<0.1	28	27	28	35	27	ppm
Cyclohexane	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	ppm
BTEX	0.5	<0.1	<0.1	<0.1	<0.1	<0.1	ppm
Other saturated hydrocarbons	0.2	<0.1	<0.1	<0.1	<0.1	0.2	ppm
Chlorine and organochlorides	*	*	*	*	*	*	ppm
Fluoride and organofluorides	*	*	*	*	*	*	ppm
Total sulphur (inorganic and organic)	0.03	<0.01	<0.01	<0.01	<0.01	<0.01	mg S/Nm³
Total silicon (including siloxanes)	<0.06	<0.06	<0.06	<0.06	<0.06	<0.06	mg Si/Nm³

*no organic chloride and -fluoride components detected.

Table 7. Results NPL December 2019 (no flow conditions).

Analyte	Concentration [µmol/mol]
Nitrogen	1116
Helium	<36
Oxygen	6.5
Argon	3.1
Carbon dioxide	<1.2
total Sulphur	<0.004
Halogenated compounds	<0.1

5 CLEANING PROTOCOL

5.1 Introduction

There are no references or experiences available in the literature for cleaning natural gas pipelines that have been converted to transport hydrogen. A project in Germany is investigating the possibility of reusing a natural gas transmission pipeline on the Lingen - Gelsenkirchen route [2]. It is to expect that cleaning will be limited to several pig-runs.

To avoid or limit contamination with pollutions from the current natural gas pipeline on the hydrogen quality, specific consideration has been given to how the pipelines can be cleaned. The basic idea is what already can be removed from a natural gas pipeline limits the contamination potential.

Based on potentially hydrogen-polluting components present in a natural gas pipeline, a method has been worked out on how a natural gas pipeline can be cleaned so that it can be used in hydrogen service. For this proposal, requirements as formulated in EIGA - IGC doc 121/14 Hydrogen pipeline systems [3] were considered. Chapter 6 of this document describes a series of pigging operations and the drying of a hydrogen pipeline. However, the EIGA mainly describes the case of a new hydrogen pipeline, although requalification is discussed, no new requirements are set. This means that the pipe must be dry and free of flash rust, welding beads and other materials.

Most natural gas pipelines are using flow coating, except for the welds. So, hardly any surface rust is to be expected from these pipelines. However, there may be contaminates like black powder, welding deposits, lubricating grease, sand, and liquids such as seal oil, glycol, and natural gas condensates. In general, it can be stated that the pipeline must be dry and free of materials (EIGA 121/14). This can be achieved with several pig-runs. The criterium for the cleanliness is the degree of contamination found on the pig. EIGA describes an assessment based on the penetration depth of the colouring in a foam pig. In addition to pigging, EIGA describes "mechanical scraping" and "high velocity gas purge" to clean pipelines. Due to damage, this is not recommended for pipelines with a flow coating and/or large diameters.

The contaminations found in natural gas pipelines are very diverse. A distinction is made between solids, liquids, and volatile/gaseous components. This last group of components will be largely removed when purged with nitrogen, before switching to hydrogen. The focus is therefore on solids and liquids (volatile or not). The solids consisting of sand, black powder, coating particles, lubricating grease, etcetera, which may or may not have become smeared with oil, glycol, and natural gas condensates. The inner pipeline walls, valves and other equipment are partly contaminated with this "greasy" mass. The amount of pollution present per pipeline section is a question mark depending on the period used for natural gas transmission. In addition, a quantity of liquid can accumulate in sinkers. To be able to estimate the degree of pollution present in a natural gas transmission pipeline, it is necessary to assume quantities and qualitative descriptions as found after performing a pig-run. It should be noted that materials trapped in dead ends and branches are not being removed with a pig-run.

5.2 Criteria for cleaning

Based on experience with pigging operations and nitrogen purge of natural gas pipelines, the proposal is to use the following criteria for cleaning natural gas pipelines:

- Liquids/solids/sludge; maximum 1 litre of material for pipe diameters up to 12 inches and up to 2 litres of material for pipe diameters larger than 12 inches (regardless of pipeline length).
- Hydrocarbons up to 1000 ppm.
- Water dewpoint < -8°C @ 70 bar.

If these criteria are met after cleaning, an existing natural gas pipeline can be converted to hydrogen transport.

Figure 12. Examples of contaminants found in existing natural gas pipelines.

5.3 Cleaning method

This section describes a cleaning method for converting a natural gas pipeline to hydrogen transport. Consideration has been given to questions such as how the network operator guarantees that possible impurities from the transport system can be controlled and limited to a minimum. Starting points for the method are:

- The pipeline route must be mapped regarding dimensions, branches and valve locations. This to identify pipe sections that cannot or can only be cleaned to a limited extent, so-called "dead ends".
- The pipeline must be physically completely separated from the natural gas system to prevent crosscontamination.
- To apply the described cleaning method, the pipeline must be pigable over the intended route.
- The integrity of the pipeline must be such that hydrogen transport is possible fulfilling all (safety)requirements.

In order to achieve a cost-effective cleaning method, experience has been included in the conversion of pipelines in the Netherlands. Based on these findings, five steps can be distinguished when converting an existing natural gas pipeline to hydrogen transport. The main steps (1 - 5) are explained in more detail in the following sub-sections:

- 1. Pre-clean using cleaning pigs to remove loose dirt and liquids from the pipeline with natural gas.
- 2. Displace natural gas to nitrogen using a pig-run to separate the natural gas from the nitrogen and preserve the pipeline under a low-pressure nitrogen atmosphere.
- 3. Perform maintenance/replacement of valves. Placing caps on branches that are no longer operational, etc. Monitoring concentrations of volatile components (including hydrocarbons, odorant and mercury) in nitrogen atmosphere as a result of desorption from pipeline parts and components.
- 4. Pig-run cleaning under nitrogen atmosphere. Monitoring contaminants in nitrogen (including hydrocarbons and mercury) and performing tests to see if the criteria to switch over to hydrogen transmission are fulfilled. If criteria are not met, an additional purge with nitrogen is carried out. Since normally no compressor station is available, liquid nitrogen in combination with a vaporizer will be used to generate pressure for pig-runs.
- 5. Displacement from nitrogen to hydrogen using a pig-run to separate the nitrogen from the hydrogen.

Before starting to clean the natural gas pipeline, several matters must be inventoried (step 0).

- The route must be mapped with regard to branches, bypasses, siphons, etc. in order to determine which parts can be cleaned with pig runs or where alternative cleaning must take place.
- The valve positions have been fully mapped (body drain has been performed before start pigging).
- Sinkers and siphons have been fully mapped, the siphons have been drained before and after the pigruns.
- Facilities are available or can be temporarily installed to enable pigging of the pipeline section (pig launcher/receiver).
- Sufficient space and possibility at pigging locations to use nitrogen to displace natural gas and are facilities available to vent nitrogen. To achieve the required cleanliness of N₂ for inertization, The advice is to use nitrogen with a purity 99.9% to minimize oxygen content.
- Monitoring tools are available for analysis of nitrogen for released components (including hydrocarbons, mercury, etc.) from the pipeline during a low-pressure purge.
- Facilities are available to collect debris released during a pig run.

Cleaning can be started when the inventory is completed, and no holds have been raised regarding the starting points.

5.3.1 Pre-cleaning

Pre-cleaning is the first step of the cleaning operation. The natural gas pipeline in question is still in natural gas service and is equipped with facilities for pigging. Pig the intended route during natural gas transport service with a bi-directional (BiDi) pig, monitor the contamination included both the amount of liquids and the amount of solid. Repeat this step several times until the contamination found is less than 1-2 liter solid/liquid (depending on the pipe diameter: 1 liter for pipelines up to 12 inches and 2 liters for pipelines langer than 12 inches). The reason for use of BiDi pig is, if the pig for some reason gets stuck in the pipeline, there is the possibility to divert.

Figure 13. Opening pig receiver.

5.3.2 Natural gas displacement with nitrogen

The second step of the cleaning operation is to displace natural gas with nitrogen using a separation pig. Store the pipeline under a nitrogen pressure of 2 - 3 bar overpressure. Monitor the hydrocarbon concentrations in the nitrogen at several sampling points along the pipeline for a two-week period (indicates to what extent there are still volatile components in the pipeline that are released at low pressure). An additional nitrogen purge must be performed in the event of continuously increasing concentrations of hydrocarbons to above 0.1 volume %. Also check the mercury levels during this phase (if detectable at ng/m³ level).

5.3.3 Modifications

The third step of the cleaning operation includes all maintenance and modifications to convert the pipeline to hydrogen transport.

5.3.4 Cleaning

The fourth step is to perform a cleaning pig-run using a BiDi-pig under nitrogen atmosphere and monitor the contamination found. Depending on the contamination found (welding pellets etc. or whether liquids have also been introduced), perform an extra pig-run if necessary, until the pig is found dry and clean in the receiver. Wait before transferring the system to hydrogen until an affected pipeline section can become part of an operational hydrogen network (i.e., allow residual volatile components from the natural gas operation phase to evaporate as long as possible in the nitrogen monitoring phase, thus preventing them from contaminating the hydrogen in the initial phase).

Figure 14. Dirty tool after pigging a natural gas pipeline.

5.3.5 Displacement from nitrogen to hydrogen

The last step of the cleaning operation is to bring hydrogen into the network with a BiDi-pig as a separation between the gases (if desired, consider installing a second pig (50 - 100 m distance between the pigs) to limit potential backmixing (this also reduces the influence of taps that are dead ends). Displace the nitrogen at a hydrogen pressure of approximately 5 - 10 bar. After full displacement, increase the hydrogen pressure to the operational value and continue operating the network. It should be advised to keep the network under flowing conditions in the beginning to minimize high concentrations of impurities due to desorption of natural gas related contaminations from the inner pipeline wall.

6 DISCUSSION AND CONCLUSIONS

The cleaning method consist of five steps. Initially the natural gas in the pipeline was displaced with nitrogen. After an additional purge, the internal volume of the 12 km 16-inch former natural gas pipeline of approximately 1500 m³ was fully displaced with pure nitrogen, the concentration of contaminants decreased very rapidly from 600 ppm C_xH_y to 3 ppm C_xH_y . The initially measured high concentrations of contaminants (aromatics and THT) originated from pores in the inner wall (flow coating) of the former natural gas pipeline. Because the pipeline had been stored under nitrogen for several weeks, the nitrogen was slowly contaminated because of desorption. This same effect could be observed on a shorter time scale after the initial nitrogen displacement. Indeed, the concentration of hydrocarbons increased from a concentration of 3 ppm to 7 ppm over the entire length of the pipeline within three hours.

After about five displacements with pure nitrogen in July 2018, the concentrations of almost all contaminants are well below the Hynetwork hydrogen specification.

The displacement occurred at low pressure conditions (just above atmospheric). The mechanism of desorption is mainly dependent on temperature as a first order process. Therefore, when the pressure is increased, dilution will occur. In addition, the absolute quantity decreases over time because there is no longer a supply of natural gas components. After commissioning, the pipeline will be deployed with hydrogen at a higher pressure and flow. Under these conditions, the content of all natural gas components will be diluted. It is therefore advised to maintain the pipeline under hydrogen flowing conditions for some time after commissioning.

Based on the measurement results in the period 2018 - 2022, it can be concluded that no contaminants/components were found that can be originally related to the former natural gas transport. This applies to both flowing and stationary conditions of the pipeline in question. After all, after a period of standstill, possibly adsorbed components in the inner pipe wall can cause a (temporary) increase in the gas phase.

The setup of the described cleaning protocol for the use of natural gas pipelines in the hydrogen backbone is based on the experiences gathered during the commissioning of the pipeline described in this paper.

7 REFERENCES

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