Project Acronym and Title:

M4ShaleGas - Measuring, monitoring, mitigating and managing the environmental impact of shale gas

Composition and management of waste from shale gas operations in Europe

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Public introduction

M4ShaleGas stands for *Measuring, monitoring, mitigating and managing the environmental impact of shale gas* and is funded by the *European Union's Horizon 2020 Research and Innovation Programme*. The main goal of the M4ShaleGas project is to study and evaluate potential risks and impacts of shale gas exploration and exploitation. The focus lies on four main areas of potential impact: the subsurface, the surface, the atmosphere, and social impacts.

The European Commission's Energy Roadmap 2050 identifies gas as a critical fuel for the transformation of the energy system in the direction of lower CO_2 emissions and more renewable energy. Shale gas may contribute to this transformation.

Shale gas is – by definition – a natural gas found trapped in shale, a fine grained sedimentary rock composed of mud. There are several concerns related to shale gas exploration and production, many of them being associated with hydraulic fracturing operations that are performed to stimulate gas flow in the shales. Potential risks and concerns include for example the fate of chemical compounds in the used hydraulic fracturing and drilling fluids and their potential impact on shallow ground water. The fracturing process may also induce small magnitude earthquakes. There is also an ongoing debate on greenhouse gas emissions of shale gas (CO_2 and methane) and its energy efficiency compared to other energy sources

There is a strong need for a better European knowledge base on shale gas operations and their environmental impacts particularly, if shale gas shall play a role in Europe's energy mix in the coming decennia. M4ShaleGas' main goal is to build such a knowledge base, including an inventory of best practices that minimise risks and impacts of shale gas exploration and production in Europe, as well as best practices for public engagement.

The M4ShaleGas project is carried out by 18 European research institutions and is coordinated by TNO-Netherlands Organization for Applied Scientific Research.

Executive Report Summary

Proper management of drilling wastes generated during shale gas operations is a crucial issue while considering safety of natural environment, mainly due to the fact that they are new, hardly recognized waste in Europe.

In Europe there are currently no explicit requirements and legal regulations concerning the management of waste from exploration and exploitation of unconventional hydrocarbons deposits, at both the EU level and the individual Member State level. Those countries rely mainly on the general mining and environmental legislation transposing the EU legislation and related permitting procedures to regulate such activities, as they do for conventional gas extraction.

Waste generated during exploration and exploitation of unconventional hydrocarbon deposits is usually not inert waste and often indicates (depending on concentrations of individual substances and chemicals contained in them) toxic or very toxic properties to aquatic organisms and may cause long-term adverse effects in the aquatic environment. Flowback water is characterized by a slightly acidic pH index, varying amounts of suspended solids and organic compounds, significant amounts of salt and other components associated with the geological structure of the rock.

Correct classification of waste (flowback and produced water) is very important for proper further waste management. However, there are currently no uniform guidelines for classifying this type of waste. In Poland, flowback and produced water has so far been classified under code 01 05 99, i. e. *as wastes not otherwise specified*, and in the UK as waste under code 01 01 02, i. e. *wastes from mineral non-metalliferous excavation*. Of course, if flowback or produced water contains hazardous substances or components, then they should be classified as hazardous waste.

Recommended, based on the results of this project, the scope of the determinations for testing flowback water for potential environmental impact should include: pH, conductivity, total dissolved solids (TDS), total suspended solids (TSS), dry residue, total organic carbon (TOC), chemical oxygen demand (COD), biochemical oxygen demand (BOD), metals (including heavy metals), inorganic anions, hydrocarbons (including mono- and polycyclic aromatic hydrocarbons), phenol index, ammonia nitrogen or total nitrogen, anionic surfactants, petroleum ether extract or chloroform extract and alcohols. In addition, when analysing samples of flowback water, it is also important to use an analytical method, which should have a properly defined limit of quantification and uncertainty, which allows correct comparison of obtained test results with e. g. regulatory criteria and results from different laboratories. The realized project has also shown that further testing is needed to develop a flowback water sampling procedure for laboratory testing and to develop guidance on analytical methods dedicated to the determination of individual substances and components in these fluids with very complex matrixes.

There are several possibilities for managing waste generated during the exploration and exploitation of unconventional hydrocarbons deposits. Reuse of flowback water on site in the next hydraulic fracturing process is a preferable one. Only the proper treatment/disposal of these fluids will guarantee safety for people and the environment.

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1 INTRODUCTION

1.1 Context of M4ShaleGas

Shale gas source rocks are widely distributed around the world and many countries have now started to investigate their shale gas potential. Some argue that shale gas has already proved to be a game changer in the U.S. energy market (EIA 2015[1]). The European Commission's Energy Roadmap 2050 identifies gas as a critical energy source for the transformation of the energy system to a system with lower CO_2 emissions that combines gas with increasing contributions of renewable energy and increasing energy efficiency. It may be argued that in Europe, natural gas replacing coal and oil will contribute to emissions reduction on the short and medium terms.

There are, however, several concerns related to shale gas exploration and production, many of them being associated with the process of hydraulic fracturing. There is also a debate on the greenhouse gas emissions of shale gas (CO_2 and methane) and its energy return on investment compared to other energy sources. Questions are raised about the specific environmental footprint of shale gas in Europe as a whole as well as in individual Member States. Shale gas basins are unevenly distributed among the European Member States and are not restricted within national borders, which makes close cooperation between the involved Member States essential. There is relatively little knowledge on the footprint in regions with a variety of geological and geopolitical settings as are present in Europe. Concerns and risks are clustered in the following four areas: subsurface, surface, atmosphere and society. As the European continent is densely populated, it is most certainly of vital importance to understand public perceptions of shale gas and for European publics to be fully engaged in the debate about its potential development.

Accordingly, Europe has a strong need for a comprehensive knowledge base on potential environmental, societal and economic consequences of shale gas exploration and exploitation. Knowledge needs to be science-based, needs to be developed by research institutes with a strong track record in shale gas studies, and needs to cover the different attitudes and approaches to shale gas exploration and exploitation in Europe. The M4ShaleGas project is seeking to provide such a scientific knowledge base, integrating the scientific outcome of 18 research institutes across Europe. It addresses the issues raised in the Horizon 2020 call LCE 16 – 2014 on *Understanding, preventing and mitigating the potential environmental risks and impacts of shale gas exploration and exploitation.*

[1] EIA (2015). Annual Energy Outlook 2015 with projections to 2040. U.S. Energy Information Administration (*www.eia.gov*).

1.1 Study objectives for this report

Summary of existing European legal regulations on the management of extractive waste, including waste from exploration for and exploitation of unconventional hydrocarbon deposits. Summary of data (literature and laboratory) on the quality characteristics of shale gas operations waste (solid and liquid). Presentation of guidelines for environmentally safe management of liquid waste resulting from the exploration and exploitation of unconventional hydrocarbon deposits.

1.2 Aims of this report

The aim of this report is to present the results of analyses and studies obtained during the WP10 (*Composition and management of different types of waste*) task in the M4ShaleGas project. The report collects the results of research on the qualitative composition of waste generated during exploration and exploitation of unconventional hydrocarbon deposits, as well as guidelines for the proper management of this type of waste. The results obtained should contribute to the minimization of environmental impact and avoid potential hazards associated with the generation of waste during shale gas operations.

2 LEGAL REQUIREMENTS CONNECTED WITH WASTE GENERATED DURING SHALE GAS OPERATIONS

At European Union level the extractive waste management is regulated by Directive 2006/21/EC of the European Parliament and of the Council of 15 March 2006 on the management of waste from extractive industries and amending Directive 2004/35/EC (*Directive 2006/21/EC*), which entered into force on 1 May 2006. Directive 2006/21/EC provides for measures, procedures and guidance to prevent or reduce as far as possible any adverse effects on the environment, in particular water, air, soil, fauna and flora and landscape, and any resultant risks to human health, brought about as a result of the management of waste from the extractive industries. Directive covers the management of waste resulting from the prospecting, extraction, treatment and storage of mineral resources and the working of quarries. The following, however, were excluded from the scope of this Directive:

- waste which is generated by the prospecting, extraction and treatment of mineral resources and the working of quarries, but which does not directly result from those operations,
- waste resulting from the offshore prospecting, extraction and treatment of mineral resources,
- injection of water and re-injection of pumped groundwater.

Without prejudice to other Community legislation, waste which falls within the scope of this Directive shall not be subject to Council Directive 1999/31/EC of 26 April 1999 on the landfill of waste (*Council Directive 1999/31/EC*).

The Directive also defines the requirements for:

- waste management plan drawn up for the minimization, treatment, recovery and disposal of extractive waste, taking account of the principle of sustainable development. The objectives of the waste management plan shall be to prevent or reduce waste production and its harmfulness, to encourage the recovery of extractive waste by means of recycling, reusing or reclaiming such waste, where this is environmentally sound in accordance with existing environmental standards at Community level and with the requirements of this Directive where relevant and to ensure short and long-term safe disposal of the extractive waste;
- informing about major accidents and preventing them – Member States shall ensure that major-accident hazards are identified and that the necessary features are incorporated into the design, construction, operation and maintenance, closure and after-closure of the waste facility in order to prevent such accidents and to limit their adverse consequences for human health and/or the environment, including any transboundary impacts;
- filling excavation voids – Member States shall ensure that the operator, when placing extractive waste back into the excavation voids for rehabilitation and construction purposes, whether created through surface or underground extraction, takes appropriate measures in order to secure the stability of the extractive waste, prevent the pollution of soil, surface water and groundwater, ensure the monitoring of the extractive waste and the excavation void;

- waste facilities with regard to their construction and management, as well as their closure and post-closure procedures - facility operator should ensure that the waste facility is suitably located, suitably constructed, managed and maintained, there are suitable plans and arrangements for regular monitoring and inspection by competent persons and for taking action in the event of results indicating instability or water or soil contamination. Suitable arrangements are made for the rehabilitation of the land and the closure and after-closure phase of the waste facility.

Directive 2006/21/EC is supplemented by five published decisions that clarify its provisions:

- *Commission Decision 2009/335/EC of 20 April 2009 on technical guidelines for the establishment of the financial guarantee in accordance with Directive 2006/21/EC of the European Parliament and of the Council concerning the management of waste from extractive industries* (*Commission Decision 2009/335/EC*),
- *Commission Decision 2009/337/EC of 20 April 2009 on the definition of the criteria for the classification of waste facilities in accordance with Annex III of Directive 2006/21/EC of the European Parliament and of the Council concerning the management of waste from extractive industries* (*Commission Decision 2009/337/EC*),
- *Commission Decision 2009/358/EC of 29 April 2009 on the harmonisation, the regular transmission of the information and the questionnaire referred to Articles 22(1)(a) and 18 of Directive 2006/21/EC of the European Parliament and of the Council on the management of waste from extractive industries* (*Commission Decision 2009/358/EC*),
- *Commission Decision 2009/359/EC of 30 April 2009 completing the definition of inert waste in implementation of Article 22(1)(f) of Directive 2006/21/EC of the European Parliament and the Council concerning the management of waste from extractive industries* (*Commission Decision 2009/359/EC*),
- *Commission Decision 2009/360/EC of 30 April 2009 completing the technical requirements for waste characterisation laid down by Directive 2006/21/EC of the European Parliament and of the Council on the management of waste from extractive industries* (*Commission Decision 2009/360/EC*).

Currently, the majority of Member States of the European Union rely mainly on the general mining and environmental legislation transposing the EU legislation and related permitting procedures to regulate such activities, as they do for gas extraction from conventional hydrocarbons deposits. None of the countries assessed provide specific requirements for the management of waste derived from hydraulic fracturing. They rely mainly on the national legislation transposing the EU waste from extractive industries legislation. However, the selected Member States do not have a common view on the applicability of the Directive 2006/21/EC to this type of waste. There are major differences between Member States and uncertainties as to the legislation and requirements applicable to the management of waste resulting from shale gas operations. Selected EU Member States have different approaches to the injection of liquid wastes resulting from shale gas operations, underground disposal or reuse in subsequent fracturing operations. These countries do not have explicit requirements concerning surface storage of liquid wastes from shale gas operations and treatment and discharge to surface waters this type of waste.

The current requirements for the management of waste from exploration for and exploitation of unconventional hydrocarbon deposits in selected EU member states are described in more detail in the D10.1 Report (Chapter 5.2) (*Kukulska-Zając et al., 2015*). The same report also describes the current and applied in Poland requirements for the management of extractive waste, including waste from shale gas operations (*Kukulska-Zając et al., 2015*).

It should be stressed, however, that despite the lack of legal regulations dedicated to waste from shale gas operations, this type of waste (as flowback or produced water) has been included in the updated version of the Best Available Techniques Reference Document for the Management of Waste from the Extractive Industries (*MWEI BREF Draft*, *2016*).

3 COMPOSITION OF WASTE FROM SHALE GAS OPERATIONS – LITERATURE AND LABORATORY DATA

Exploration and exploitation of unconventional hydrocarbon deposits are associated with the generation of waste, the harmfulness of which to the natural environment varies. Part of this generated waste is a typical drilling wastes, i.e. drilling cuttings and used drilling mud, which are well known and characterized. Such waste typically have a semi-solid or solid consistency. A new kind of waste is associated with hydraulic fracturing (flowback and produced water). This type of waste is not, however, well characterized in terms of quality. Of course, apart from waste connected directly with the drilling operations and subsequent reservoir tests (e.g. extractive wastes), waste from drilling well pad operation, delivery of services, presence of employees on the drill site etc. is also generated.

The qualitative and quantitative composition of waste generated during shale gas operations changes widely over time, depending on numerous factors such as, for example, type of drilling fluid used during drilling, depth and construction of the hole, type of drilled rock formations, chemical reactions between the rock and the fluid, the time fluid remains in the borehole and chemicals used in the technological process.

The exact determination of quality of waste generated during exploration and exploitation of unconventional hydrocarbon deposits is very important. Knowledge of the content of particular chemical substances in this type of wastes, especially hazardous substances, allows further sound management of such wastes and contributes to minimizing their negative impact on the environment and human health. It should be noted that regardless of the type of generated mining waste, a further way of their management should be done in accordance with the applicable laws and issued decisions.

Little information is publicly available about the qualitative composition of waste generated during exploration of unconventional hydrocarbon deposits. Characterization of waste connected with shale gas operations has been reported in the literature in various degrees of detail. In the further part of this chapter, data (both literature and laboratory) on the quality characteristics of shale gas operations waste were collected from the project.

3.1 Drilling wastes

Table 1 shows the results of physicochemical parameter designations for drilling waste samples from exploration of unconventional hydrocarbon deposits in Poland (data from Lubocino, Stare Miasto, Wysin, Gapowo, Syczyn and Zawada). The table shows the results obtained for the solid phase of drilling waste (total content of a given component in the waste is designated) and the leaching tests results (the amount of ingredient, which may enter the environment as a result of washing out of the waste, is designated).

Table 1. Summary of the most frequently designated physicochemical parameters in drilling waste samples from Poland (based on *Środowisko i prace rozpoznawcze dotyczące gazu z łupków; wyniki badań środowiska gruntowo-wodnego, powietrza, klimatu akustycznego, płynów technologicznych i odpadów [The environment and shale gas exploration. Results of studies on the soil-water environment, ambient air, acoustic climate, process fluids and wastes], 2015, Kukulska-Zając et al., 2016*).

No.	Determined parameter	Range of changes of designated parameter	
		total content	leachable form
		[mg/kg DW]	
1.	antimony (Sb)	0.03-10.0	$2.90 \cdot 10^{-2}$-0.11
2.	arsenic (As)	2.10-14.7	$1.28 \cdot 10^{-2}$-0.13
3.	bar (Ba)	455-2,836	$1.40 \cdot 10^{-2}$-0.76
4.	beryllium (Be)	0.40-2.80	$2.80 \cdot 10^{-4}$-$3.09 \cdot 10^{-3}$
5.	boron (B)	0.50-33.1	$1.97 \cdot 10^{-2}$-$6.20 \cdot 10^{-2}$
6.	chromium (Cr)	31.8-95.4	$7.60 \cdot 10^{-4}$-$5.33 \cdot 10^{-3}$
7.	tin (Sn)	2.50-24.7	$2.56 \cdot 10^{-3}$-$6.63 \cdot 10^{-2}$
8.	zinc (Zn)	53.4-313	$2.56 \cdot 10^{-2}$-$8.70 \cdot 10^{-2}$
9.	aluminium (Al)	18,370-48,113	$5.85 \cdot 10^{-3}$-1.12
10.	cadmium (Cd)	0.10-5.30	$8.60 \cdot 10^{-4}$-$5.28 \cdot 10^{-2}$
11.	cobalt (Co)	6.40-19.3	$2.80 \cdot 10^{-3}$-$1.20 \cdot 10^{-2}$
12.	lithium (Li)	18.6-31.5	-
13.	magnesium (Mg)	8,432-29,969	-
14.	manganese (Mn)	363-1,448	$1.39 \cdot 10^{-3}$-0.34
15.	copper (Cu)	27.6-106	$1.15 \cdot 10^{-4}$-$1.80 \cdot 10^{-2}$
16.	molybdenum (Mo)	0.20-10.9	$4.10 \cdot 10^{-3}$-$8.20 \cdot 10^{-2}$
17.	nickel (Ni)	27.5-71.3	$1.20 \cdot 10^{-3}$-$1.20 \cdot 10^{-2}$
18.	lead (Pb)	5.80-91.5	$5.50 \cdot 10^{-3}$-$3.84 \cdot 10^{-2}$
19.	potassium (K)	6,794-22,248	3.20-17,400
20.	mercury (Hg)	0.02-0.30	$<10^{-6}$-$4.60 \cdot 10^{-2}$
21.	selenium (Se)	0.10-1.80	$8.70 \cdot 10^{-2}$-2.05
22.	sodium (Na)	1,948-44,987	365-23,585
23.	silver (Ag)	0.00-0.60	$2.81 \cdot 10^{-4}$-$7.90 \cdot 10^{-3}$
24.	strontium (Sr)	87.7-582	$2.41 \cdot 10^{-2}$-0.39
25.	thallium (Tl)	0.10-2.50	$1.81 \cdot 10^{-2}$-0.11
26.	titanium (Ti)	4.70-115	$1.10 \cdot 10^{-4}$-$3.60 \cdot 10^{-3}$
27.	vanadium (V)	47.4-231	$3.20 \cdot 10^{-3}$-$5.02 \cdot 10^{-2}$
28.	calcium (Ca)	20,209-62,543	200-3,206
29.	iron (Fe)	20,812-66,476	$7.00 \cdot 10^{-3}$-0.15
30.	ammonium nitrogen (NNH_4^+)	37.6-267	<0.25-55.0
31.	bromine, bromides (Br)	-	2.20-509
32.	chlorides (Cl^-)	-	1,418-44,676
33.	fluorides (F^-)	29-698	10.1-264
34.	sulphates (SO_4^{2-})	-	18.6-466
35.	hydrogencarbonates (HCO_3^-)	-	610-6,834

No.	Determined parameter	Range of changes of designated parameter	
		total content	leachable form
		[mg/kg DW]	
36.	total dissolved solids (TDS)	-	5,306-78,921
37.	phenol index	<0.5-7.00	<0.01-1.70
38.	total organic carbon (TOC)	3,058-40,650	510-9,380
39.	dissolved organic carbon (DOC)	1,113-10,190	430-7,280
40.	surfactants (anionic)	9.00-64.6	<0.20-8.40
41.	chemical oxygen demand (COD)	7,950-110,229	1,080-24,400
42.	gasoline (total)	3.25-211	1.94-29.4
43.	mineral oils (total)	99.8-1,541	4.08-236
44.	aliphatic hydrocarbons	212-1,591	6.15-242
45.	aromatic hydrocarbons	1.08-70.2	0.65-9.80
46.	polycyclic aromatic hydrocarbons (PAHs)	<0.001-0.446	$<10^{-6}$-2.40
47.	pH reaction	pH	
		7.49-9.65	
48.	specific conductance	[mS/cm]	
		7.68-5,600	
49.	acid-neutralizing capacity (ANC)	[mg $CaCO_3$/kg s.m.]	
		10.0-5,600	

< indicates results below the limit of quantification
- indicates no available data/no designation
DW - Dry Weight (Dry Basis)

An analysis of the data collected in Table 1 shows that the content of individual organic and inorganic components in the drilling wastes (total content and amount of substance leached from waste) and the values of the measured parameters can vary in a wide range. Significant differences in scope are related to the type of drilling mud used and the prevailing deposit conditions. Drilling wastes are characterized by a fairly high pH, high content of chloride ions and high content of metals such as Al, Mg, K, Na, Ca and Fe. The content of total dissolved solids (TDS) is also high in this type of waste, as well as content of total and dissolved organic carbon (TOC and DOC) and hydrocarbons. A detailed analysis of the composition of drilling wastes is presented in Report D10.2 (Chapter 3.2) (*Kukulska-Zając et al., 2016*).

3.2 Flowback water

Table 2, on the other hand, presents the results of laboratory tests of samples of flowback water (from one of the drilling rigs in Poland) obtained as part of this project with the results of tests for this type of waste available in literature (data from Europe, mainly Poland).

Table 2. Comparison of results of laboratory samples of flowback water with results for this type of waste available in literature (based on *Olsson et al.; 2013, Środowisko i prace rozpoznawcze dotyczące gazu z łupków; wyniki badań środowiska gruntowo-wodnego, powietrza, klimatu akustycznego, płynów technologicznych i odpadów [The environment and shale gas exploration. Results of studies on the soil-water environment, ambient air, acoustic climate, process fluids and wastes], 2015; Granops et al., 2013; Klimkiewicz and Korczak, 2012; Starzycka, 2012; Starzycka, 2014; Kukulska-Zając et al., 2016; Kukulska-Zając et al., 2017*).

Lp.	Determined parameter	Range of changes of designated parameter	
		Laboratory data	Literature data
1	density [kg/l]	1.067 – 1.089	-
2	pH	6.4 – 6.8	5.7-7.4
3	specific conductance [mS/cm]	115 – 151	11.9-123
4	dry residue [mg/l]	107,300 – 166,210	-
5	total dissolved solids (TDS) [mg/l]	103,780 – 161,820	-
6	total suspended solids (TSS) [mg/l]	112 – 279	168
7	chemical oxygen demand (COD) [mgO_2/l]	536 – 1,402	307-6,230
8	biochemical oxygen demand (BOD) [mgO_2/l]	2,904 – 3,712	2,416
9	petroleum ether extract [mg/l]	<10.0 – 87.0	-
10	chloroform extract [mg/l]	<2.00 – 54.0	-
11	total organic carbon (TOC) [mg/l]	170 – 350	11.0-1,680
12	dissolved organic carbon (DOC) [mg/l]	170 – 350	99.0-1,193
13	hydrocarbons C_1-C_5 [mg/l]	0.010 – 1.28	-
14	hydrocarbons C_6-C_{12} [mg/l]	<1.00 – 46.4	-
15	hydrocarbons C_{12}-C_{35} [mg/l]	<10.0 – 1,560	-
16	monocyclic aromatic hydrocarbons [mg/l]	<0.40	-
17	polycyclic aromatic hydrocarbons [mg/l]	$1.50 \cdot 10^{-4}$-$2.10 \cdot 10^{-3}$	<$5.00 \cdot 10^{-6}$-$3.82 \cdot 10^{-4}$
18	methanol [mg/l]	<10.0	-
19	ethanol [mg/l]	<10.0 – 21.0	-
20	isopropanol [mg/l]	<10.0 – 49.2	-
21	aluminium (Al) [mg/l]	<1.00	$3.00 \cdot 10^{-3}$-$3.06 \cdot 10^{-2}$
22	antimony (Sb) [mg/l]	<1.00	-
23	arsenic (As) [mg/l]	<0.30	$5.52 \cdot 10^{-3}$-1.10
24	barium (Ba) [mg/l]	200 – 350	0.00-593
25	cadmium (Cd) [mg/l]	<0.01	$1.90 \cdot 10^{-4}$-$1.20 \cdot 10^{-2}$
26	calcium (Ca) [mg/l]	4,850 – 8,040	0.23-22,000
27	chromium (Cr) [mg/l]	<0.10 – 0.16	0.30
28	cobalt (Co) [mg/l]	<0.01	$6.16 \cdot 10^{-4}$-$3.01 \cdot 10^{-3}$
29	copper (Cu)[mg/l]	<0.01	-
30	iron (Fe) [mg/l]	36.0 – 64.0	$5.27 \cdot 10^{-3}$-500
31	lead (Pb) [mg/l]	<0.10 – 0.23	0.30-55.0
32	magnesium (Mg) [mg/l]	613 – 1,080	0.93-2,170
33	manganese (Mn) [mg/l]	7.30 – 11.0	1.00-38.0
34	mercury (Hg) [mg/l]	<0.0005 – 0.0011	-
35	molybdenum (Mo) [mg/l]	<0.10 – 0.41	-

Lp.	Determined parameter	Range of changes of designated parameter	
		Laboratory data	Literature data
36	nickel (Ni) [mg/l]	<0.10 – 0.40	0.30-1.00
37	potassium (K) [mg/l]	582 - 993	1.67-7,510
38	selenium (Se) [mg/l]	<1.00	-
39	sodium (Na) [mg/l]	27,740 – 70,690	0.84-44,800
40	strontium (Sr) [mg/l]	870 – 2,000	$8.80 \cdot 10^{-4}$-1,720
41	tin (Sn) [mg/l]	<0.20 – 1.10	-
42	vanadium (V) [mg/l]	<0.30	-
43	zinc (Zn) [mg/l]	<0.10 – 0.85	$6.79 \cdot 10^{-4}$-290
44	bromides (Br^-) [mg/l]	624 – 967	25.0-500
45	carbonates (CO_3^{2-}) [mg/l]	<6.00	-
46	chlorides (Cl^-) [mg/l]	55,122 – 81,456	3,800-115,140
47	fluorides (F^-) [mg/l]	2.30– 21.7	0.50-6.10
48	hydrogencarbonates (HCO_3^-) [mg/l]	137 – 326	166-509
49	nitrates(V) (NO_3^-) [mg/l]	<10.0	-
50	phosphates (PO_4^{3-}) [mg/l]	<10.0	-
51	sulphates (SO_4^{2-}) [mg/l]	26.0 – 49.0	4.00-1,100
52	sulphide [mg/l]	<0.10	-
53	butanethiol [mg/l]	<0.001 – 0.62	-
54	carbon disulphide [mg/l]	<0.001 – 0.11	-
55	carbonyl sulphide [mg/l]	<0.001 – 0.13	-
56	diethyl sulphide [mg/l]	<0.001	-
57	dimethyl disulphide [mg/l]	<0.001 – 0.22	-
58	dimethyl sulphide [mg/l]	<0.001 – 0.23	-
59	ethanethiol [mg/l]	<0.001	-
60	hydrogen sulphide [mg/l]	<0.001 – 0.03	-
61	methanethiol [mg/l]	<0.001 – 0.07	-
62	methylthioethane [mg/l]	<0.001	-
63	propanethiol [mg/l]	<0.001	-
64	sulphur dioxide [mg/l]	<0.001 – 0.16	-
65	phenol index [mg/l]	<0.008 – 0.02	-
66	anionic surfactants [mg/l]	0.13 – 0.39	<0.50-31.0
67	ammonia nitrogen [mg/l]	80.0 – 104	9.00-159
68	isodrin [µg/l]	<0.03 – <0.06	-

- indicates no available data/no designation

The analysis of the data presented in Table 2 shows that the chemical composition of flowback water is characterised by a high variability in qualitative terms. Of course, not only the composition of the fracturing fluid, but also rock formations into which the fracturing fluid is injected, have a big influence on the content of individual components in the fluid. Flowback water has a pH in the range of 5.7-7.4 and contains significant amounts of chloride ions as well as metals such as Ba, Ca, Mg, K and Na. The total dissolved solids (TDS) content in characterized fluids is also high. A detailed analysis of the results of laboratory test results of flowback water samples was presented broadly in Report D10.3 (*Kukulska-Zając et al.,* 2017).

The results presented in Table 2 also show that so far, the physical parameters (pH and specific conductance) and some organic and inorganic components were determined in the samples of flowback water. Among the inorganic components identified in this type of waste, there are mainly metals (including heavy metals) and anions (bromides, chlorides, fluorides, hydrogencarbonates and sulphates). The organic components determined in the samples of flowback water are hydrocarbons, total and dissolved organic carbon (TOC and DOC), as well as chemical oxygen demand (COD), biochemical oxygen demand (BOD) and anionic surfactants. As shown in the table, these are not all components that may be present in this type of samples. In addition, as shown by the research carried out as part of this project, samples of flowback water may also contain alcohols, sulphur compounds and a broader spectrum of metals than those already determined. However, it should be noted that laboratory tests of flowback water samples are largely in line with literature reports and differences such as e. g. as regards the determination of the content of chemical oxygen demand (COD), total organic carbon (TOC), dissolved organic carbon (DOC), Ca, Fe, Mg, K, Zn, sulphates and anionic surfactants may be associated with various quality of hydraulic fracturing fluids and the type of rock formations to be drilled. These differences may also result from various ways of sampling and sample preparation before testing, as well as differences in analytical methods (see Report D10.3) (*Kukulska-Zając et al., 2017*).

4 MANAGEMENT OF LIQUID WASTE FROM SHALE GAS OPERATION

The issue of rational waste management, including drilling wastes, has been at the centre of the European Union's environmental policy for many years. The primary objective of the Union's policy in this area is to prevent waste from arising first and, in the case of waste that cannot be prevented from arising, reuse as far as possible, recycling and recovery, but only ultimately landfilling.

The common feature of exploration for and exploitation of hydrocarbons from conventional and unconventional deposits is the drilling of a vertical well and the production of the same type of drilling wastes, the main components of which are drilling cuttings and used drilling mud. The management of such waste does not pose any significant problems, as it is a waste that is well known and the companies conducting drilling have many years of experience in this field.

The challenge is the proper management of liquid waste (flowback and produced water) associated with hydraulic fracturing during shale gas operations. Firstly, it results from the fact that large amounts of waste are generated during such operations. Secondly, it is related to the characteristics of generated waste, primarily with their complex and varied chemical composition, ecotoxicity and state of consistency (liquid or semi-liquid). Thirdly, the environmental impact of shale gas operations waste, including soil, surface and groundwater, is still poorly recognised in Europe. Proper management of waste generated during exploration and exploitation of unconventional hydrocarbon deposits, requires a reliable data on the quantity and quality of this waste. Having such information will contribute to increasing environmental safety, e. g. as a result of accidental release, as well as an improperly planned and conducted process of shale gas operations waste treatment or storage.

Flowback water is produced when the hydraulic fracturing procedure is completed and pressure is released. Composition of flowback water is related to the composition of the initial fracturing fluid, the composition of the natural formation water of the shale and the possible interactions between fracturing fluid and shale system over time at the in-situ conditions. Initially this water, called flowback, is mostly fracturing fluid, but with time it becomes more similar to the natural formation water, e. g. there is an increase in salinity, and decrease in DOC (*Cluff et al., 2014*). This later stage water is also called a produced water. However, it should be added here that there is no clear line between flowback and produced water. In addition, it is important to be aware that the produced water from shale gas operations is different from the typical deposit water generated during the exploration and exploitation of conventional hydrocarbon deposits.

4.1 Flowback and produced water classification

Bearing in mind that the waste associated with hydraulic fracturing during shale gas operations is a new type of waste in Europe and its correct classification is very important, as it will ensure proper handling of this type of waste. In the European Union, waste is classified according to the Commission Decision of 18 December 2014 amending Decision 2000/532/EC on the list of waste pursuant to Directive 2008/98/EC of the European Parliament and of the Council (*Commission Decision 2014/955/EU*). The Annex to that Decision contains a List of Waste, allowing for the classification of different types of waste.

The different types of waste in the list are fully defined by the six-digit code for the waste and the respective two-digit and four-digit chapter headings. According to the List of Waste, extractive waste should be classified as chapter 01, i. e. *waste resulting from exploration, mining, quarrying, and physical and chemical treatment of minerals*. On the other hand, drilling wastes and drilling muds for chapter 01 05, i. e. *drilling muds and other drilling waste*, obviously taking into account their properties.

Unfortunately, however, there is no specific code in the current List of Waste that can be arbitrarily assigned to shale gas operations waste, i. e. flowback and produced water. Therefore, correct classification of this type of waste can be problematic as it is based only on the opinion of the waste producer. Until now, waste such as flowback and produced water has been classified in Poland most often as waste of code 01 05 99, i. e. *wastes not otherwise specified*, in chapter 01 05, i. e. *drilling muds and other drilling wastes* (*Środowisko i prace rozpoznawcze dotyczące gazu z łupków; wyniki badań środowiska gruntowo-wodnego, powietrza, klimatu akustycznego, płynów technologicznych i odpadów [The environment and shale gas exploration. Results of studies on the soil-water environment, ambient air, acoustic climate, process fluids and wastes], 2015*), while in United Kingdom as waste with code 01 01 02, i. e. *wastes from mineral non-metalliferous excavation* (*Environment Agency, 2016*).

However, it should be mentioned that if flowback or produced water contains hazardous substances or components, waste should be classified as hazardous waste depending on the concentration of these substances or components in the fluid. Criteria for the classification of waste as hazardous waste according to its characteristics are set out in Annex III to Directive 2008/98/EC of the European Parliament and of the Council of 19 November 2008 on waste and repealing certain Directives (*Directive 2008/98/EC*). Wastes should be regarded as hazardous if they have at least one of the following characteristics: explosive, oxidizing, highly flammable, flammable, irritant, harmful, toxic, carcinogenic, corrosive, infectious, toxic for reproduction, mutagenic, sensitizing, ecotoxic. Hazardous properties are assigned on the basis of the criteria in Annex VI to CLP Regulation, i. e. Regulation (EC) No 1272/2008 of the European Parliament and of the Council of 16 December 2008 on classification, labelling and packaging of substances and mixtures, amending and repealing Directives 67/548/EEC and 1999/45/EC, and amending Regulation (EC) No 1907/2006 (*Regulation (EC) No 1272/2008*).

In Poland, in accordance with the guidelines of the Ministry of the Environment, flowback water is not considered as waste until it leaves the process line. Where flowback water is treated on site, the residue from its treatment shall be considered as extractive waste and the water fraction resulting from the treatment of flowback water suitable for reprocessing in the fracturing process, even if transferred to another holder, shall not be considered as waste. The untreated or not for further use flowback water is considered as extractive waste (*Możliwości postępowania z płynem zwrotnym powstałym podczas procesu szczelinowania w kontekście przepisów z zakresu gospodarki odpadami [Possible handling of flowback water generated during the fracturing process in the context of waste management regulations], 2014*).

4.2 Flowback water quality tests

Flowback water generated during hydraulic fracturing processes should be tested to determine its characteristics and potential risks to the environment in the event of uncontrolled leakage, both the waste itself and the products after recovery/disposal processes. The necessity of the research results both from the fact that fracturing fluids are produced using different chemical substances and from geochemical differences of the drilled rock layers. The results of laboratory analyses of this type of waste will determine whether the examined waste should be classified as hazardous or non-hazardous and thus indicate potential ways of further management, safe for the natural environment.

As has already been mentioned many times before, there are no guidelines for the scope of testing the quality of a new type of waste such as flowback water. As a result, as part of the project implementation, extensive laboratory tests were carried out on flowback water samples coming from one of the drilling rigs in Poland. The results of laboratory tests allowed verification of the initial basic scope of research proposed in Report D10.2 (*Kukulska-Zając et al., 2016*) dedicated to waste generated during exploration and exploitation of unconventional hydrocarbon deposits.

The range of physicochemical parameters, which should be determined in flowback water samples, verified during the project implementation is presented in Table 3. The presented set of tests includes typical indicators of environmental pollution, parameters defined by law for waste in general and parameters, which should be additionally monitored in wastes such as flowback water due to their specific nature.

Table 3. Range of determinations recommended for the flowback water quality test to assess potential environmental impact.

No.	Parameter	Unit
	General parameters	
1.	pH index	-
2.	specific conductance	mS/cm
3.	total dissolved solids (TDS)	mg/l
4.	total suspended solids (TSS)	mg/l
5.	dry residue	mg/l
6.	chemical oxygen demand (COD)	mgO_2/l
7.	biochemical oxygen demand (BOD)	mgO_2/l
8.	total organic carbon (TOC)	mg/l
	Alkali or alkaline-earth metals and others	
9.	sodium (Na), potassium (K), calcium (Ca), magnesium (Mg), strontium (Sr)	mg/l
10.	aluminium (Al), iron (Fe)	mg/l
	Heavy metals	
11.	barium (Ba), cadmium (Cd), lead (Pb), molybdenum (Mo), antimony (Sb), total chromium (Cr), zinc (Zn), copper (Cu), nickel (Ni), vanadium (V), arsenic (As), cobalt (Co), selenium (Se), mercury (Hg), tin (Sn)	mg/l
	Anions	
12.	chlorides (Cl^-), bromides (Br^-), fluorides (F^-)	mg/l
13.	sulphates (SO_4^{2-}), hydrogencarbonates (HCO_3^-)	mg/l
	Aliphatic hydrocarbons	
14.	hydrocarbons C_6-C_{12}	mg/l
15.	hydrocarbons C_{12}-C_{35}	mg/l
	Aromatic hydrocarbons	
16.	monocyclic aromatic hydrocarbons (BTEX)	mg/l
17.	polycyclic aromatic hydrocarbons (PAHs)	mg/l

No.	Parameter	Unit
	Other organic and inorganic parameters	
18.	ammonia nitrogen or total nitrogen	mg/l
19.	phenol index	mg/l
20.	anionic surfactants	mg/l
21.	alcohols (depending on the composition of the fracturing fluid)	mg/l
22.	petroleum ether extract or chloroform extract	mg/l

In addition, such wastes should be subjected to toxicological and ecotoxicological tests, as it was found that flowback water may pose a threat (in case of improper management) to living organisms after they have leaked uncontrolled into the environment (*Środowisko i prace rozpoznawcze dotyczące gazu z łupków; wyniki badań środowiska gruntowo-wodnego, powietrza, klimatu akustycznego, płynów technologicznych i odpadów [The environment and shale gas exploration. Results of studies on the soil-water environment, ambient air, acoustic climate, process fluids and wastes], 2015*).

The range of determinations recommended for flowback water testing for potential environmental impact presented in Table 3 is basically the same as that initially proposed in Report D10.2 (*Kukulska-Zając et al., 2016*). The determination of sulphur compounds was omitted in the revised scope, as the laboratory testing of these compounds in flowback water has shown that the laboratory content of these compounds is low. The recommended range was supplemented by determination of petroleum substances extracting by various types of organic solvents.

It is worth mentioning that the recommended range includes the determination of parameters which are used as good indicators in environmental monitoring studies. These parameters include mainly specific conductance and chlorides content. The latter, due to its very high concentrations in flowback water and its easy and rapid migration into the environment, is an ideal indicator of the appearance of a first wave of potential pollution in the environment. The scope of research presented in Table 3 also includes indications for determination of the substances or components of flowback water, on the basis of which it is possible to assess the usefulness of particular methods of initial treatment of such fluids. Thus, the suitability of sedimentation or filtration as a pre-treatment method can be determined on the basis of total suspended solids (TSS) content, while the content of petroleum substances extracting by various types of organic solvents allows for an appropriate selection of fat separators to protect the elements of the process line during treatment (*Granops et al., 2013*).

The studies conducted as part of this project (see Report D10.3, Chapter 4) (*Kukulska-Zając et al., 2017*) have also shown that an equally important aspect as shale gas operations waste scope of research is also the appropriate way of sampling for testing, sample preparation for testing and selection of the applied analytical method. Laboratories' analytical methods shall:

- have a defined uncertainty of measurement, as the uncertainty of measurement is the only parameter that allows a fully objective comparison of the results obtained with the results of other laboratories or regulatory limits. For the determination of impurities in environmental samples with a complex matrix, this uncertainty shall not exceed 30%;
- have a correctly defined limit of quantification, which is particularly important when determining the traces in flowback water samples. Correct definition of the limit of

quantification also ensures that presence of an analyte above the limit of quantification with an adequate acceptable uncertainty;
- in case of determination of metals content of flowback water samples have a specific procedure for preparation of the sample for testing, allowing correct determination of total metals content in the sample.

The assessment of shale gas operations wastes in terms of environmental impact should be conducted both on the basis of the results of recommended tests of physicochemical parameters and on the basis of the analysis of the fluid composition used in the hydraulic fracturing process. Knowledge of the composition of the fracturing fluid is important, as it will allow us to supplement the recommended scope of research (Table 3) with possible additional parameters, which may have a potentially negative impact on the environment.

4.3 Further management of waste

Volume and composition of flowback and produced water varies over the lifetime of the well. Having information about quality and quantity of generated waste it is possible to select one of the possible options of their further proper management. The main feature that makes the further use of this type of waste difficult is high water content and the resulting consistency of the waste (mostly liquid or semi-liquid). Another difficulty is that chemical composition of the waste (e.g. high and variable contents of heavy metals and salts of some alkali elements in the form of chlorides, sulphates or hydrogencarbonates) is highly variable and difficult to predict.

In the USA, waste such as flowback water or produced water can be disposed in one of the following ways:

- disposal via surface discharge, underground injection or land application,
- reuse/recycle flowback water in subsequent fracturing works,
- transfer flowback water to a Centralized Waste Treatment (CWT) facility,
- transfer flowback water to a Publicly Owned Treatment Works (POTW) facility,
- reuse produced water for beneficial purposes,
- in selected areas, operators also use evaporation ponds (impoundments) for disposal of produced water.

Disposal via underground injection is the most widespread method in the USA, but operators increasingly try to implement reuse/recycle options for this type of waste management.

In Europe, disposal of flowback water by deep well injection is not used, although such a method of use is permitted under European law. It may be possible where it is re-injected into formations from which hydrocarbons have been extracted and will have no impact on the status of water bodies or pose any risk to groundwater. Nor is this method recommended as Best Available Technique (BAT) due to the high risk of pollution of the environment, especially groundwater (*Grant and Chisholm, 2016; Gomez et al., 2016*). Other methods of liquid waste management generated during shale gas operations in Europe are (*Grant and Chisholm, 2016; Gomez et al., 2016; Granops et al., 2013*):

- reuse on-site for the next hydraulic fracturing process. Reuse of flowback and produced water arguably represents the most sustainable process in terms of water resource use. However, this treatment requires pre-treatment (mechanical filtration, neutralisation, oxidation, coagulation and sedimentation) of flowback water. However, it should be added that on-site treatment processes also reduce the risks associated with transporting waste. Flowback water that is not suitable for reuse is classed as a waste and must be sent to an appropriate permitted waste facility for treatment or disposal;

- transfer, treated or not treated on site, to wastewater treatment facilities or other waste recovery or disposal facilities. Such installations should have permission to treatment the appropriate mining waste code for flowback water;
- transfer to a disposal facility for extractive waste.

In the latter two cases, waste must be transported. Spills or leaks could potentially occur during the transportation. Preventative measures should be included in the waste management plan.

In order to ensure optimal environmental safety, information should be collected not only on the quantity and quality composition of flowback water, but also on the treatment and further management of waste. In Poland, there is no obligation to collect and disclose complete information on the exact history of waste, there is no obligation to obtain information on the final location and method of waste recovery/disposal. Only qualitative and quantitative data on each type of waste generated is mandatory. This is waste holders and waste producers responsibility. However, obtaining and collecting data on the final management of waste would be valuable information for local communities, obviously interested in their own safety and the environment in their area, and would also facilitate control in the correct application of procedures for the further management of waste authorised for this control body.

5 RECAPITULATION AND CONCLUSIONS

Proper management of shale gas operations wastes is crucial, mainly due to the fact that they are new, hardly recognized waste, and in addition flowback water is a very complex object for testing, treatment or storage, due to the complexity and changeability of the matrix.

In Europe, waste generated during exploration and exploitation of gas from unconventional hydrocarbon deposits are bounded by the same legal regulations as waste generated during exploration and extraction of conventional hydrocarbon deposits. Therefore, for the waste from exploration and exploitation of gas from shale formations there are no uniform requirements regarding the scope of research on quality of this type of waste, guidelines for their treatment, transportation, disposal and storage and need to disclose the results of research on their quality. Therefore, it is necessary to supplement the existing legislation with requirements on how to handle waste such as flowback and produced water, including uniform requirements for quality testing of this waste.

Correct classification of waste (flowback and produced water) is very important for proper further waste management. However, there are currently no uniform guidelines for classifying this type of waste. However, it is important not to classify flowback water as a wastewater, since it cannot be reused in this form in accordance with current legislation for hydraulic fracturing. In Poland, flowback and produced water has so far been classified under code 01 05 99, i. e. *as wastes not otherwise specified*, and in the UK as waste under code 01 01 02, i. e. *wastes from mineral non-metalliferous excavation*. Of course, if flowback or produced water contains hazardous substances or components, then they should be classified as hazardous waste. In view of the above, it should be concluded that there is a real need to supplement the List of Waste with a new type of waste (i. e. flowback and produced water) with a code that is suitable for both hazardous and non-hazardous waste.

Waste generated during exploration and exploitation of unconventional hydrocarbon deposits is usually not inert waste and often indicates (depending on concentrations of individual substances and chemicals contained in them) toxic or very toxic properties to aquatic organisms and may cause long-term adverse effects in the aquatic environment. The amount of publicly available detailed information on the scope and results of shale gas operations waste in Europe is low and selective, as is the amount of data on substances and chemical compounds used in the preparation of process fluids. There is also no information available on the amount of waste resulting from such works. Therefore, it is suggested that, more broadly than before, data on the composition of the process fluids used in the drilling operations and information on the composition and properties of the waste generated, as well as information on how to further manage the waste, should be made public. Storage of this type of information will allow us to make a clear statement whether possible environmental pollution occurring even after time is related to shale gas operations.

Recommended, based on the results of this project, the scope of the determinations for testing quality of flowback water for potential environmental impact should include: pH, specific conductance, total dissolved solids (TDS), total suspended solids (TSS), dry residue, total organic carbon (TOC), chemical oxygen demand (COD), biochemical oxygen demand (BOD), metals (including heavy metals), inorganic anions, hydrocarbons (including mono- and polycyclic aromatic hydrocarbons), phenol index, ammonia nitrogen or total nitrogen, anionic surfactants, petroleum ether extract or chloroform extract and alcohols. The realized project has also shown that not less important than the proper scope of research is also:

- The method of sampling and preparing the sample for the tests, as it is this initial stage of the study that has a significant influence on the obtained analysis results. The method of sample preparation for the tests is particularly important during the determination of metals content.
- Selection of an appropriate analytical method, which should have an appropriately defined limit of quantification and uncertainty, allowing for a correct comparison of the obtained test results, e. g. with the criteria set out in the legal regulations and the results obtained by different laboratories.

Therefore, further testing is needed to develop a flowback water sampling procedure for laboratory testing (including the purpose and location of sampling before, after separator or from the container) and to develop guidance on analytical methods dedicated to the determination of individual substances and components in these fluids with very complex matrix. Testing of such samples should not involve methods routinely used for testing water quality.

There are several possibilities for managing waste generated during the exploration and exploitation of unconventional hydrocarbon deposits. The shale gas operations flowback water produced can be reused on-site in the next hydraulic fracturing process. However, this treatment requires pre-treatment (mechanical filtration, neutralisation, oxidation, coagulation and sedimentation) of the flowback water. It can also be transferred (treated or not treated on-site) to wastewater treatment facilities or other waste recovery or disposal facilities, or transferred to a mining waste disposal facility. Disposal of flowback water by deep well injection is not used, although such a method of disposal is permitted (under certain conditions) in accordance with European law.

To sum up, it should be added that waste connected with shale gas operations should not enter in an untreated form into the environment, even unintentionally, e. g. as a result of failure. Flowback water should be used on site for further treatments whereas the transport of such waste to other locations for reuse or to mining waste disposal facilities should be in accordance with waste transport procedures. Only the proper treatment/disposal of these fluids will guarantee safety for people and the environment.

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