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Executive Summary

This deliverable is an update of D2.3 and D2.6, which discussed the possible standards and regulations relevant for the Hydroptics platform. Many of the identified regulations were intended to guarantee a safe operation of the device, meaning that neither other devices are affected, nor persons get injured. The measures taken to ensure safe operation of the Hydroptics platforms will be presented as well as the findings to improve the alpha version.

The second topic of this deliverable relates to the possibilities to convert the implemented lab methods into an international standard. Here, the options, requirements and recommendations are presented, and the updated situation is explained.

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1. Introduction

This report is an update of the deliverables D2.3, D2.6 and stems from task T2.3. Here, the regulations and standards that are relevant for the Hydroptics project are identified and evaluated.

In the first section of this deliverable, we give an update on the legal requirements, that apply to the assembled alpha, beta, and final versions of Hydroptics platform. The focus is on the relevant European directives that are implemented in the local laws of the individual countries. We describe which directives apply to the Hydroptics platform and which measures were taken to comply with them.

Then, the status regarding the certification of the individual measurement methods is discussed. The identified options are presented. Based on the observations and the development progress, we will give a recommendation for standardization route.

2. Directives and Regulations

Before a product or service can be introduced on the market, the person or company must ensure that it fulfills the local laws and regulations. To simplify this process, the European Union releases directives that must be implemented in the local laws and harmonizes the legal requirements. Subsequently, the administrative tasks to check for the legal requirements is significantly reduced and simplifies the introduction of new services and products.

As one aim of Hydroptics is to increase the TRL level from laboratory setups (TRL 3-4) towards sellable products (aim: TRL 6-7), it is of utmost importance to consider the applicable regulations and laws as soon as possible. Identifying the various requirements at an early stage of the project helps avoid potential issues that could require major revisions and can reduce the time to bring a new product to the market.

During the planning and assembly of the alpha version of the Hydroptics platform, a series of directives relevant for this project have been identified and were taken into consideration when drafting the specifications and developing Hydroptics tools. The list has been extended as the project progressed towards its end, in November 2023. They shall be discussed in further detail and examples for their applicability will be presented as well.

Directive 2014/35/EU – Low Voltage Directive (LVD)

This directive covers electrical equipment designed for use with a voltage between 75 and 1500 V direct current (DC) or 50 and 1000 V alternating current (AC). It should be pointed out, that this limit addresses only the supply voltages and not the voltages within the device. Moreover, the directive does not cover individual components and evaluation kits destined for professionals at research and development facilities are excluded as well.

The complexity of the Hydroptics platform requires an input voltage of 230 V AC and an expected power consumption of approx. 1.5 kW. This directive was not relevant for the alpha version of the Hydroptics platform, which was tested only under lab conditions and operated by experts. The final versions, however, fulfilled this directive and this has verified independently by the project end-users (OMV and TUPRAS) prior to demonstration of the tools, as reported in D8.2 and D8.3. The strict internal protocols implemented by the end users in their facilities caused an unforseen short delay for the demo activities. Good engineering practices followed by the experts developing and assembling the individual modules minimize any potential risks and ensure safe operation of the Hydroptics platforms in compliance with this directive.

Directive 2014/30/EU – Electromagnetic Compatibility (EMC)

The term “electromagnetic compatibility” means, according to the European Commission, the ability to function satisfactorily in its electromagnetic environment without introducing intolerable electromagnetic disturbances to other equipment in that environment, such as other sensing and communication devices and actuators installed in the refinery/oil industry facilities. Possible sources of disturbances are incorrect installation of electrical motors, wires acting as antennas, or insufficient shielding.

Within Hydroptics, the motors (especially brushed ones) used for liquid handling, as well as insufficiently shielded wires were identified as possible sources of electromagnetic radiation. To minimize the influence on other equipment, adequate shielding, good engineering practices, as well as high-quality power supplies have been used. Moreover, it should be pointed out that this directive, like the Low Voltage Directive, does not apply to evaluation kits (prototypes) in research facilities. Therefore, it is only mandatory for the beta version of the Hydroptics platform.

Directive 2014/42/EU – Machinery Directive

This directive focuses on the requirements a new machine/device launched on the market must fullfil, whereby the term "machine" stands for an assembly of components that are joined together for a specific application. It applies to any device fitted with a drive system driven by other than human or animal effort. It discusses the minimum saftey measures and its documentation. It also includes the consideration of ergonomical aspects, a detailed risk assessment of the machine, risks caused by various hazards, and requirements for servicing, inspections and cleaning thereof. Again, devices for short term research work in laboratories are excluded thereof.

Both, the alpha and the beta version of the Hydroptics platform are equipped with moveable parts, some of them driven by electrical motors, for example cooling fans, peristaltic pumps, the mixer for the liquid/liquid extraction or the centrifugal separator. Partners undertook the following measures to guarantee the safe operation of the Hydroptics platform: a suited cover installed around around the moving parts constituted a mechanical protection, the doors of housing could be only opened by authorized personnal.

Ergonomical issues have been addressed within Hydroptics as well. The measurement device's software can be operated by users, either through a remote connection from the office, or with a bright user-friendly touch-screen. This touch-screen is set at a height of approx. 170 cm above the ground, i.e., at eye level, therefore making it ergonomical. The platform was also made transportable. Equiping the sensor platform with wheels made its transport from the laboratory to the intended measurement sites easy. Once installed, two of the wheels could be blocked with brakes, ensuring mechanical stability. Finally, the overall dimensions (height, width and depth) and weight of the platform have been constrained so that only two persons are needed to move it.

Directive 2014/34/EU – ATEX Directive (Explosive Protection)

This directive harmonises the laws relating to equipment and protective systems intended for use in potentially explosive atmospheres. Aside defining the obligations of manufacturers, the definition of notification bodies and the declaration of conformity, the directive lists safety requirements and describes measures intended at preventing explosions. Reducing the risk of ignition by avoiding electrostatic discharge, preventing heating of mechanical parts, selecting adequate materials, as well as good engineering practice for safe design and construction, are among these measures.

The brushed motors installed on the alpha version of the Hydroptics analysers can cause sparks during their operation. As the beta version was installed in an ATEX Zone 2 - Group IIA/B classified industrial environment, all motors were replaced by brushless equivalents, subsequently minimizing the risk of sparks able to ignite an explosive atmosphere. Moreover, there is room to install temperature sensors at different locations in the housing to prevent over-heating.

Directive 2014/32/EU – Measuring Instruments Directive (MID)

Measurement devices operating in the European Union are regulated by this directive. It lists the obligations of manufacturers, importers and distributors who sell/trade such devices. Products that fulfill the requirements are to be marked with the capital letter "M" and the year of its affixing, surrounded by a rectangle (aside the CE marking).

The requirements defined by this directive apply for water meters, thermal energy meters, automatic weighing instruments and others. Devices classified as "particle counters" or "oil meters" are not listed within. Therefore, this directive does not apply to the Hydroptics platform. However, several recommendations could be

implemented. The device documentation includes detailed description of measurement ranges, rated operation conditions, maintenance operations, repairing procedures, as well as the compatility conditions with other interfaces.

Directive 2014/68/EU – Pressure Equipment Directive (PED)

The Pressure Equipment Directive sets the standards for the design and fabrication of pressure equipment. This includes, for instance, steam boilers, pressure vessels, piping and safety valves. It should be pointed out that their volume is generally over one litre and their maximum pressure is higher than 0.5 bar. The directive also covers topics such as essential safety requirements, conformity assessment procedures and the declaration of conformity.

After review of the components installed in the Hydroptics platform, it appeared that none of the parts would build up a > 0.5 bar pressure under correct operation. Therefore, this directive does not apply to the assembled prototypes.

Directive 2011/65/EU – Restriction of the use of certain hazardous substances (RoHS)

Hazardous substances in electronic products are nowadays a major issue. Considering the high complexity of electronic devices, it is a priority to limit the use of toxic substances commonly found in the industry. This directive addresses this topic in the European Union by restricting the use thereof to protect the human health and the environment. The use of lead, mercury, cadmium and hexavalent chromium (and some other substances) is forbidden, with a few exceptions. Like with other directives, equipment specifically designed for research and development are excluded.

Relying on commercially available components and electronics modules makes it easy to comply with this directive. The alpha version was almost free of hazardous substances. Potentially problematic components, like mercury-cadmium-lead (MCT or HgCdTe) based infrared detectors, are excluded from the directive, and hand-made, lead-soldered, circuit boards are acceptable due to the research nature of the project. However, for a later commercialization of the Hydroptics platform, it is strongly recommended to replace the hand-made circuit boards with industrial printed circuit boards and lead-free solders to comply with this directive.

The final HYDROPTICS prototypes did not contained lead-based solder. Mercury-cadmium-lead based infrared detectors, which are a standard for mid-infrared applications, are not in themsleves RoHS-compatible. However, such detectors are typically used in, non-mass markets, highly-demanding applications, such as military, space and medical applications, due to their high performances. As there are currently no viable alternatives for these detectors, Annex IV (“Applications exempted from the restriction in Article 4(1) specific to medical devices and monitoring and control instruments”) of the 2011/65/EU directive allows MCT detectors for use in “Sensors, detectors and electrodes”. However, the list of exemptions is regularly reviewed, and subject to change, should alternative technologies appear.

REACH Regulation (EC 1907/2006) - (Registration, Evaluation, Authorisation, and Restriction of Chemicals)

The REACH regulation of the European Union was adopted to improve human health and environment the protection against the risks caused by chemicals, while enhancing the competitiveness of the EU chemicals industry. It also promotes alternative methods for the hazard assessment of substances to reduce the number of tests on animals. REACH applies to all chemical substances; those used not only in industrial processes, but also in daily life, for example in cleaning products, paints, as well as in articles such as clothes, furniture and electrical appliances.

The avoidance of reagents or consumables during measurements belong to the main benefits of the spectroscopy-based technologies implemented by Hydroptics. This significantly lowers the analysers’ cost (including maintenance cost) and makes the technology green. The use of chemicals is limited to small quantities of solvents used for cleaning and regeneration purposes, including alcohols and other organic solvents, that are disposed using typical protocols used in the oil industry. The analysers include bins to collect such wastes, which are either disposed using existing infrastructure, or are manually transferred when filled for processing.

3. Standardization

In contrast to directives, that are converted into local laws, standards are voluntary guidelines that provide technical specifications for products, processes, or services. They are often initiated by companies, who see the need for a specific product or process to be described in more details to achieve its objective/intention. Other interested parties, such as regulators and consumers, can participate as well to help improve the quality of a standard.

Depending on the topic and the regional importance of a standard, different standardization bodies are involved. They can be regional (e.g., CEN, CENELEC, ETSI), national (e.g., DIN, ANSI), or international bodies (e.g., ISO, ASTM).

Since the submission of deliverables D2.3 and D2.6, we have identified three different possibilities to implement the standardization topic within Hydroptics. The potential routes and the currently recommended approaches are the topic of the following sub-sections.

3.1. Option A: Comply with existing standards

As the topic of measuring Oil-in-Water is not new, the industry has standardized several measurement methods over time. They differ in their achievable precision, sample preparation steps, instrumental requirements and expected measurement time. Table 1. gives a short overview of the most important standards relating to the Hydroptics project.

Standard	Measurement method	Precision	Time effort	Comments
EPA 1664	Gravimetric	-	--	Extraction, evaporation of solvent
ISO 9377-2	Gas Chromatography	++	-	
ISO 16703	Gas Chromatography	++	-	
DIN 38409	Infrared (FTIR)	+	+	Requires the use of CFC (1,1,2-Trichlortrifluorethane)
ASTM D7678	Infrared (Laser)	++	+	CFC-free (Cyclohexane)
ASTM D8193	IR (Filter)	-	+	CFC-free (Cyclohexane)
ASTM D7066	IR (FTRI)	+	+	Extraction (hexane or pentane)

Table 1: Selection of standards, relevant for Oil-in-Water measurements.

Standards defining measurement of Total Suspended Solids have been identified as well and are listed in Table 2. Most of them rely on filtration and subsequent weighing of the solids. Optical methods are also included. Due to their nature, it is difficult to distinguish particles from oil droplets with latter methods, leading to unprecise results.

Standard	Measurement method	Precision	Time effort	Sample preparation
ASTM D5907	Gravimetric	+	--	Filtration, drying of the sample
EPA 160	Gravimetric	+	--	Filtration, drying of the sample
Standard Method 2540D	Gravimetric	+	--	Filtration, drying of the sample

ASTM D3977	Gravimetric	+	--	Evaporation, filtration
ASTM D1889	Turbidity	-	++	Optical method

Table 2: Selection of standards, applicable for particles suspended in water.

A first option in the standardization strategy for the Hydroptics platform is to comply with one of the standards highlighted by the coarse overview presented above. Although seemly simple and doable, this is not the case. Indeed, it is not sufficient to implement a standardized measurement method in a working device – it is mandatory to fully comply with it. This implies that the whole sample preparation, device qualification process, the subsequent data evaluation, and, if available, the statistics must be performed as described in the standard.

Let us take the case of Oil-in-Water measurements. The developed IR absorption-based dual-DFB-QCL setup could fulfill the technical requirements as described in the ASTM D7678 standard. However, this document also describes sample preparation and handling, requesting manual handling of samples, extraction of oil with cyclohexane for a certain amount of time, as well as performing measurements on quality control samples at well-defined time intervals.

Since sample preparation in the alpha version of the Hydroptics platform is fully automated (it is equipped with an automated batch-wise extraction step), it is not possible to comply with the manual waste/process water' sampling-related requirements described in the ASTM D7678 standard. In addition, none of the conditions on the specified samples volumes, required materials, or mixing times, are fulfilled. Subsequently, as the new Hydroptics developments move well beyond the existing standards, it is inherently not possible to comply with the ASTM D7678 standard.

As with the infrared-based measuring setup, no standard, with which the newly developed Hyperspectral Imaging setup could comply, was found.

Consequently, this first option is not suitable for the standardization of the Hydroptics platform, as it is typical with the development of beyond state-of-the-art technologies.

Furthermore, there are several standards covering the design of equipment operating in the demanding field of the oil industry. These standards overlap with the directives described in the previous section.

Standard	Purpose	Comments
IEC 60529	Degrees of protection provided by enclosures or the ingress protection rating of the sensor, for harsh environments.	Partially fulfilled
IEC 60079	Explosive Atmospheres. Essential for equipment to be used in potentially explosive atmospheres	Partially fulfilled
IEC 60529	Degrees of protection provided by enclosures (IP Code). Ensures the sensor is adequately protected against environmental factors in industrial settings.	Partially fulfilled

Table 3: Selection of standards, relevant for analysers operating in demanding industrial settings.

3.2. Option B: Initiate a CEN/CENELEC Workshop Agreement

The second option to standardize the methods developed in the frame of Hydroptics is to initiate a CEN/CENELEC Workshop Agreement (CWA). This standardization route is strongly driven by the outcomes of Horizon 2020 and Horizon Europe projects and relies on a strong interest of the industry regarding research and technological innovation. For innovative technologies and products, it is sometimes more important to build an early standard

although the method is not yet fully established. It circumvents the situation where established companies try to prevent a new standard that could interfere with their already established ones.

The way to file a CWA is, in short, as follows (further details can be found in D2.3 and the CEN-CENELEC Guide 29):

- Submission of proposal form
- Draft of project plan
- Announcement on the CEN/CENELEC website
- Launching the Workshop
- Commenting phase
- Approval and Availability
- If possible/requested: adoption as European standard

The updated list of technologies developed in the frame Hydroptics is as follows:

- Online oil-in-water extraction using cyclohexane and analysis with a Dual-Frequency Comb Spectrometer
- Online extraction and separation using a mixing reactor and a centrifugal separator
- Enhanced absorption measurements based on Mach-Zehnder Interferometry
- Advanced particle identification and characterization using a Hyperspectral Imaging System (HIS)
- Data analytics based on machine learning for monitoring and predicting critical process parameters

Above are the outcome of the results of demonstration activities (field tests) of the project, as reported in deliverables D8.2 and D8.3. The field tests were concluded at M48 of the project with most of the Hydroptics' technologies reaching TRL7. Nevertheless, the suitable CWA was not initiated during the project and will be considered in the future. The main obstacle into driving the procedure was the departure of the Hydroptics partner QuantaRed Technologies. As such, the third option, described below, is the option of choice for Hydroptics.

3.3. Option C: No standardization during the project

The third option of choice regarding standardization is to delay the process and to perform this task after the Hydroptics project ends. This was needed due to the delays induced during the project by technical challenges, and, importantly, the departure of partner QuantaRed Technologies (initially the leader of the present deliverable D2.7). Although this could be seen as a drastic step, a standardization afterwards has several advantages too. The possible strategy could be as follows.

The time allocated to the project was used to advertise the project in the industry, including the organization of the Hydroptics online Workshop. Based on the promising results obtained during the field tests/demonstration activities, further communication of the project results is foreseen during the next period. Thereby, awareness for online Oil-in-Water measurements has been created. This step includes different tasks, like presenting the results of the field tests at scientific conferences, promoting the installed technology at trade shows, and advertising the need for online Oil-in-Water measurements at technical workshops.

Via these activities, representatives of other oil producing companies and refineries will understand the importance of the developed technology and raise the question whether the technology is already standardized or not. Again, to fulfill such a purpose, it is important to use the outcome of the demonstration activities at high TRL (TRL 7), as people from the industry would be more interested to see the analysers operating in real environment, rather than results from characterisation of the instruments in laboratory settings.

Asking for a standard would be already the perfect feedback because of two reasons. First, they are interested in continuously monitoring water quality and therefore potential project partners for further testing. Secondly, they may be familiar with the standardization processes and, as experts in their fields, could have valuable contacts to other companies who face similar challenges. They could help with their knowledge and to create a consortium/work group that would push the technology and method development of online Oil-in-Water measurements even further.

Raising the necessary awareness and creating the demand for a standard is time consuming and will take longer than 12 months. With the identified importance of the technology, it will be possible to assign a consortium that will take the route for standardization. As the development of its standard is now being driven by the industry and not solely by the experts of the measurement device, the chances for acceptance of setting up a standard is significantly higher.

An example of a successful product without having a standard is the Oil-in-Water analyzer “InfraCal 2 ATR-SP” from Spectro Scientific. This analyzer is based on Attenuated Total Reflection (ATR) technology and measures, after extraction using n-Hexane, the oil content. Although its measurement technique is not yet standardized, this robust and user-friendly device is a successful product that is installed in many laboratories.

3.4. Current and long-term strategy after the project Hydroptics

During the last months of the project, the Hydroptics analysers were successfully demonstrated at TRL7 at the facilities of the end-users. A successful standardization is only possible if there is a clear demand from the stakeholders. These can be companies from the industry itself, customers, who care about the quality of a product or a service, or even public bodies. Since the Hydroptics platform high-TRL demonstration and technology assessment has just been completed, further steps for standardisation can only be taken after the project, given the developers of the platform show a clear interest on standardization. The latest is a challenging task due to the departure and seize of existence of partner QuantaRed Technologies. Nevertheless, partners have clearly expressed their commitment to fully exploit the new technologies, which implies actions towards standardisation in the mid-term.

Based on the field demonstration and assessment of the Hydroptics platform, further successful post-project dissemination and communication will lead to a demand for its standardization. As soon as the industry is convinced of the benefits of the Hydroptics platform, they will support this task and promote either a CEN/CENELEC workgroup or join an ASTM work group.

Conclusions

In this deliverable we have reviewed the current implementation of the directives, that were identified in D2.3 and D2.6. Directives followed by the final version of the Hydroptics platform have been identified and implemented to indicate the significant increase of the TRL level. Fulfilling the required directives and local laws begun at an early stage of the project, aiming to increase the acceptance of the novel measurement setup by the industry and were fully implemented for the successful TRL7 demonstration activities/field tests.

In the second part of this deliverable, three options regarding standardization have been reviewed. Among the three options, the first one, i.e., complying with existing standards, has been excluded due to the novelty of the new developments. The option two, i.e., applying for a new standard during the project, has been excluded, due to lack of time, as well as to technical and other challenges faced during the project execution. Based on the promising results obtained during the demonstration activities (reported in D8.2 and D8.3), the Hydroptics consortium is considering applying for standardisation in the mid-term, as this strongly depends on the demand from the industry, whether they agree that it is necessary to standardize our developed methods at all. Again, it should be pointed out that a successful product does not require/fulfill a standard at all (although it can be a powerful asset).

Appendix

List of Abbreviations

CEN	Comité Européen de Normalisation
CENELEC	Comité Européen de Normalisation Électrotechnique
CWA	CEN/CENELEC Workshop Agreement
ATR	Attenuated Total Reflection
CA	Consortium Agreement
ASTM	American Society for Testing and Materials
ISO	International Organization for Standardization
TRL	Technology Readiness Level