



**EC COOPERATIVE RESEARCH PROJECT  
COOP-CT-2005-017970**

**SAFELUBE**

**A Novel Method of Continuous On-line Conditioning & Safe Management to  
Enable the use of Fire Retardant Lubricants in Gas & Steam Power  
Generation Turbines**

Cooperative Research  
Horizontal Research Activities Involving SMEs

**Full Project Activity Report**  
Date of issue of this report: August 2009

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Duration: 17 Months

**CO-ORDINATOR:** Pera Innovation Ltd

**SME CONTRACTORS:**

Kelman Ltd  
Alpes Lasers  
Macq Electronique  
MinerWa Umwelttechnik GmbH  
Safibra s.r.o.

**OTHER CONTRACTORS:**

Great Lakes  
MAN Turbomaschinen

**RTD PERFORMER CONTRACTORS:**

Pera Innovation Ltd  
Fraunhofer Institut Umwelt, Sicherheits, Energietechnik UMSICHT

## CONTENTS

	<b>Page</b>
<b>PROJECT INFORMATION</b>	<b>3</b>
<b>PUBLISHABLE EXECUTIVE SUMMARY</b>	<b>4</b>
<b>SECTION 1: SUMMARY OF PERIOD OBJECTIVES AND ACHIEVEMENTS</b>	<b>5</b>
<b>SECTION 2: WORKPACKAGE PROGRESS REVIEW FOR REPORTING PERIOD 2</b>	<b>6</b>
<b>Workpackage 1; Including D1, D2 &amp; D3</b>	<b>7</b>
<b>Workpackage 2; including D4, D5, D6, D7 &amp; D8</b>	<b>22</b>
<b>Workpackage 3; including D9, D10 &amp; D11</b>	<b>27</b>
<b>Workpackage 4; including D12, D13, D14 &amp; D15</b>	<b>49</b>
<b>Workpackage 5; including D16 &amp; D17</b>	<b>59</b>
<b>Workpackage 6; including D18, D19, &amp; D20</b>	<b>59</b>
<b>Workpackage 7</b>	<b>67</b>
<b>SECTION 3: CONSORTIUM MANAGEMENT</b>	<b>67</b>
<b>Workpackage 8; including D21 &amp; D22</b>	<b>67</b>
<b>SECTION 4: CONCLUSIONS</b>	<b>72</b>
<b>APPENDIX 1</b>	<b>73</b>

## PROJECT INFORMATION

**PROJECT NO:** FP6-017970

**CONTRACT NO:** COOP-CT-2005-017970

**TITLE OF PROJECT:** **A Novel Method of Continuous On-line Conditioning & Safe Management to Enable the use of Fire Retardant Lubricants in Gas & steam Power Generation Turbines.**

**COORDINATOR:** Pera Innovation Ltd

**SME EXPLOITATION MANAGER:** Kelman Ltd

### ***SME CONTRACTORS:***

Kelman Ltd  
Alpes Lasers  
Macq Electronique  
MinerWa Umwelttechnik GmbH  
Safibra s.r.o.

### ***OTHER CONTRACTORS:***

Great Lakes  
MAN Turbomaschinen

### ***RTD PERFORMER CONTRACTORS:***

Pera Innovation Ltd  
Fraunhofer Institut Umwelt, Sicherheits, Energietechnik UMSICHT

## **PUBLISHABLE EXECUTIVE SUMMARY**

Since 1991 in Europe, there have been in excess of 2500 power generation gas turbine related incidents, including some 700 fires and 14 explosions, and in the renewable sector, 61 wind turbine fires in the last 10 years alone. Clearly there is a need, both economically and socially, for more effective fire prevention measures in the power generation industry to improve the security of energy supply.

Current mineral turbine lubricants only last about 5 years, the cost of replacement is substantial, even before factoring in their disposal and labour. During the replacement of lubricant oils there is also always the risk of introducing contaminants into the machinery. A “fill for life” lubricant requiring minimal maintenance has long been an industrial need.

In comparison phosphate ester (PE) lubricants have superior oxidative stability and potentially last the lifetime of the turbine. Significantly, they have a fire retardant, non-combustible nature. However, despite their availability, there is a limited take up of these in industry as they need regular monitoring and maintenance and they can become unstable in the presence of water. The primary degradation by-products of phosphate esters are acids, which must be removed to prevent corrosion to the turbine. Current oil conditioning programmes for phosphate ester lubricants require intermittent manual sampling and analytical techniques followed by the removal of water and conditioning depending on the analysis results. This process is time consuming, costly and prone to human error.

As such, a European Consortium of SME companies and Research Performers, led by Kelman Limited, have just participated in a two year European Research project, to develop a novel method of continuous on-line conditioning & safe management to enable the use of fire retardant lubricants in gas & steam power generation turbines, for the power generation sector called SAFELUBE.

SAFELUBE addresses the issues relating to the use of PE's by developing a remote sensing and conditioning system. This makes cost effective use of the fire retardant PE's as a turbine lubricant, by extending lubricant life to match the life of the turbine. This is achieved with the development of a system of solid state laser diode sensors to monitor and quantify the water and TAN levels in the phosphate ester lubricants, together with an expert process control system to manage acid and water removal. The integration of these elements into a single turbine lubricant monitoring and condition system is SAFELUBE. The intention of SAFELUBE being that it operates continuously on-line to measure and condition the PE's, to ensure optimum use of resource, reduce production costs and maximise operational life. The SAFELUBE consortium would then promote the expanded use of PE lubricants and create new markets.

### **The Consortium Consisted of:**

Kelman Ltd (UK)  
Alpes Lasers (CH)  
Macq Electronique (BE)  
MinerWa Umwelttechnik GmbH (AT)  
Safibra s.r.o.(CZ)  
Fraunhofer Institut UMSICHT (DE)  
Pera Innovation (UK)

## Objectives

To achieve the aims and objectives of the SAFELUBE project consortium, an ambitious programme of research and development was undertaken, under the EC Framework 6 Programme.

Scientific research work was required to meet the following targets:

- **Characterization of vital properties of moisture levels and TAN.**
- **Sensor development.**
- **Water and acid removal.**
- **Water and acid management & communications protocol.**
- **Prototype integration and validation.**

The Framework 6 programme offered the majority of the SME partners access for the first time to the world class research expertise of two European Research Teams in Germany and the United Kingdom.

## CONCLUSION

In conclusion, SAFELUBE has been subject to the restriction of a limited project time. Key consortium members withdrew from the project and their replacement proved too difficult to fulfil within the project lifetime. Therefore the limited development and results generated are still subject to Intellectual Property Rights (IPR) protection. When the consortium are reformed and in a position to take this technology to its next developmental stage then the benefits and knowledge of the SAFELUBE project can then be further disseminated.

[www.safelube.pera.com](http://www.safelube.pera.com)

## SECTION 1:

### Summary of period objectives and achievements

This report is prepared to inform the Partners and the EC of progress to date and is a summary of period one (P1) and period two (P2), of the Safelube project. The project aim is to address the cost effective and safe operation of steam and gas turbines by developing a remote sensing and conditioning system to support the cost effective use of fire retardant turbine lubricants by extending lubricant life to the life of the turbine. There is a need for more effective fire prevention measures in the power generation industry to improve the security of energy supply, a key objective of the EC. Currently, fire retardant lubricants, which could greatly reduce fire risk, are rarely used in Europe, largely due to the increased lubricant management requirements of fire-retardant formulations. Developing an on-line oil condition monitoring system to reinforce the uptake of fire-retardant lubricant formulations is a key goal of the Safelube project.

In P1 a Kick-off meeting was held at Kelman Ltd, at Lisburn, Northern Ireland, on 25<sup>th</sup> January 2007 which established the relationship between the project partners, reviewed the project goals and agreed management procedures. A review of the state-of-the-art was also presented alongside a technical work plan.

A Month 12 meeting did not take place due to the non-accession to the SAFELUBE contract of two key project partners providing knowledge of the lubricant industry (Great Lakes) and power generation turbines (Man Turbo). This proved to be a considerable stumbling block

for the SAFELUBE consortium. On 10<sup>th</sup> March 2008, the consortium requested a six month suspension of the project in order that potential replacements be put in place.

During the period of suspension, Kelman, the project coordinator, was acquired by GE Energy during the first months of Period 2. Following the take over, Kelman's focus was aimed more specifically at transformer monitoring and so they decided to withdraw from the SAFELUBE project following the suspension on 10<sup>th</sup> March 2008. It is at this point that Pera Innovation Ltd, the lead RTD contractor, also became the project Coordinator.

In P2, there was a sustained effort to recruit replacement partners, however due to the nature and limitations of the turbine technology, this was not ultimately successful and the project was terminated in Month 17 by the European Commission.

The project website found at (<http://safelube.pera.com>) was set-up in P1, and is being accessed by the partners. Project progress, meeting minutes, travel arrangements, meeting dates, partner reports and presentations are among the items disseminated on the website.

In summary, the execution of the project work has been delayed significantly from the start due to the non-accession of two key partners. Replacement partners were sought to take over the commitments of Great Lakes and MAN Turbo during P1 but despite the substantial effort targeted at this task, this was not achieved and it was then agreed to delay the decisions relating to the direction of the project work, for example the identification of which lubricants and resins to focus on. The reasoning behind this decision had been the recognition that it was important to minimise the risk that activities within any updated work packages, and subsequent knock-on tasks (in the remainder of the project), may not have been aligned with the needs of the replacement partner. It was therefore necessary to extend the duration of the tasks designed to characterise the properties of the lubricating oils in Work package 1, into P2. Formulations of lubricants to be investigated were planned to be delivered in P2, this was to check that they were aligned with those employed currently, or in the future, by the end-user within the consortium. To allow for the full execution of the technical programme, a formal request for the extension of the project had been planned once the consortium was fully formed.

## Section 2. Workpackage Progress

### Overview

The progress of the project has been significantly impacted by changes within the make up of the consortium. At the start of P2 it was unclear if the emphasis of the work schedule would focus on phosphate ester lubricant formulations as was initially expected, or whether the scope of the turbine lubricant characterisation phase of Work package 1, would be significantly broadened to encompass a number of other lubricant formulation types. Several additional fire-retardant fluid formulations were identified that could be preferred by turbine users, as the uptake of phosphate ester (PE), formulations within the industry was observed low. In order to maximise the potential impact of the Safelube system within the power generating industry, it was decided by the consortium to broaden the scope of the project in terms of the type of lubricant formulations that were investigated. These were summarised in the P1 Activity Report from detail provided by Houghton Plc.

Activities on the development of laser diode sensors within Work package 2 commenced and some refinement of focus was proposed by the consortium. The aim of expanding the industry standard concept of Total Acid Number (TAN) by resolving weak and strong acid types is considered possible using this technology. As the very nature of the organic acid species to be investigated by this sensor element is dictated by the type of lubricant under examination, putting in place the firm specifications of the laser diode system was delayed, although a possible configuration is proposed.

Activities on the development of membrane regeneration technologies in Work package 3 commenced. Significant effort has been targeted at setting up membrane analysis systems and these are now being employed to assess possible candidates. However, the focus of these activities would change dependant upon the formulation of turbine lubricant that is to be the centre of the Safelube project. The nature of this must be fixed to allow work to progress beyond the initial test stage. Therefore, certain decisions were delayed until the consortium was able to go forward, fully formed.

With the delays encountered in activities in the development of the sensor and filtration systems, knock-on delays resulted with the progress in Work package 4. As access to turbine systems was not possible due to the non-accession of Man Turbo, it was felt by the consortium that commencing the design of control systems, that may require high levels of technical specification dependent upon the nature of the turbine systems upon which they will be implemented, would not be appropriate at this stage. It was decided by the consortium that these tasks within WP4 would be commenced when the consortium was fully formed and technical insight into specific turbine systems could be provided to the consortium.

## **WORKPACKAGE 1 – CHARACTERISATION OF VITAL PROPERTIES**

Objectives: To generate new scientific knowledge and understanding of how moisture levels, total acid number and chlorine levels affects the “condition” and “performance” of the PE’s. To characterize other factors that may affect the performance of the lubricant such as temperature fluctuations, etc. Identification of target moisture and acid content and chlorine levels in the PE’s to deliver maximum life. Data gathering to confirm the specification and concept design of the system. To provide the foundation to of future work programmes and to pave the way forward for the study.

Deliverables D1, D2 and D3 have been produced to meet these objectives

### **Deliverable D1; Relationship between water and TAN content & condition/state of PE’s**

#### **The Use of Phosphate Ester Lubricants**

A literature review has been performed to understand the chemistry related to phosphate ester additives in turbine lubricants.

Phosphate ester-based hydraulic and lubricating fluids have been employed throughout industry for over 50 years as a means of reducing fire-related incidents. Extensively used in steel mills and foundries, natural gas compressors and aircraft hydraulic control systems as well as in the power generation industry, it has been commonly reported that the performance of phosphate ester based fluids depends critically upon the quality of maintenance. Corrosion, valve sticking and unsatisfactory system performance can be expected when phosphate esters are deprived of proper fluid maintenance<sup>1</sup>.

Phosphate ester-based hydraulic fluids are the most common type employed in the electrohydraulic control (EHC) systems responsible for governing steam delivery to the turbines that drive power generators. Triaryl phosphate esters are self-extinguishing – their

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<sup>1</sup> Ken Brown, Utility Service Associates, "Managing the Health of Fire Resistant Steam Turbine Electrohydraulic (EHC) Control Oils". Machinery Lubrication Magazine. May 2001

combustion creates an energy debt that means that fires in phosphate ester based fluid formulations are not readily sustained. Typical species used within formulations include; Trixylene phosphate ester (TXP), Butylated phenol phosphate ester (TBPP) and Isopropyl phenol phosphate ester (IPPP). However, the trend for increasingly elevated temperatures (>500°C) and pressures (>10 bar) of operation within modern power generation mean that these types of fluid formulations need to be observed and managed increasingly closely.

### Problems Associated with Maintenance of Phosphate Ester Systems

Phosphate esters can degrade as a result of thermal, oxidative and hydrolytic breakdown, illustrated below in Figure 1.

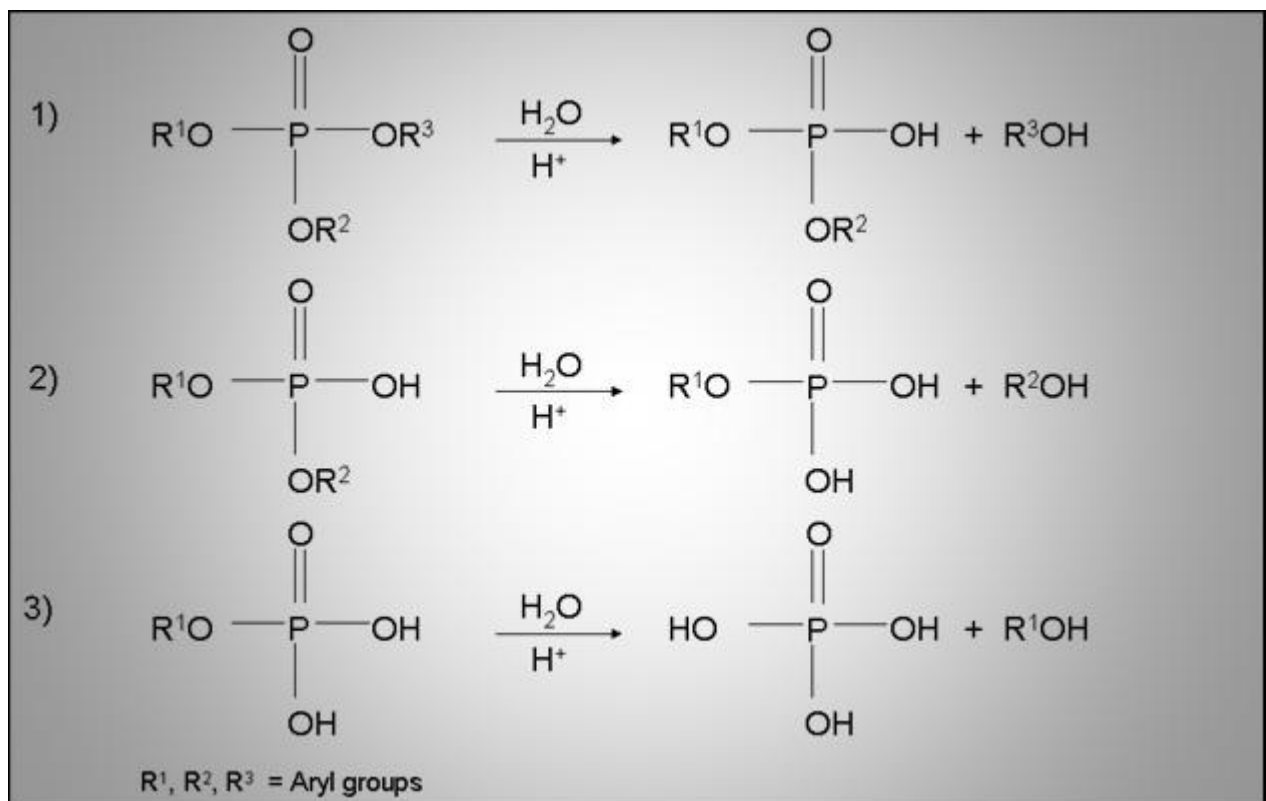


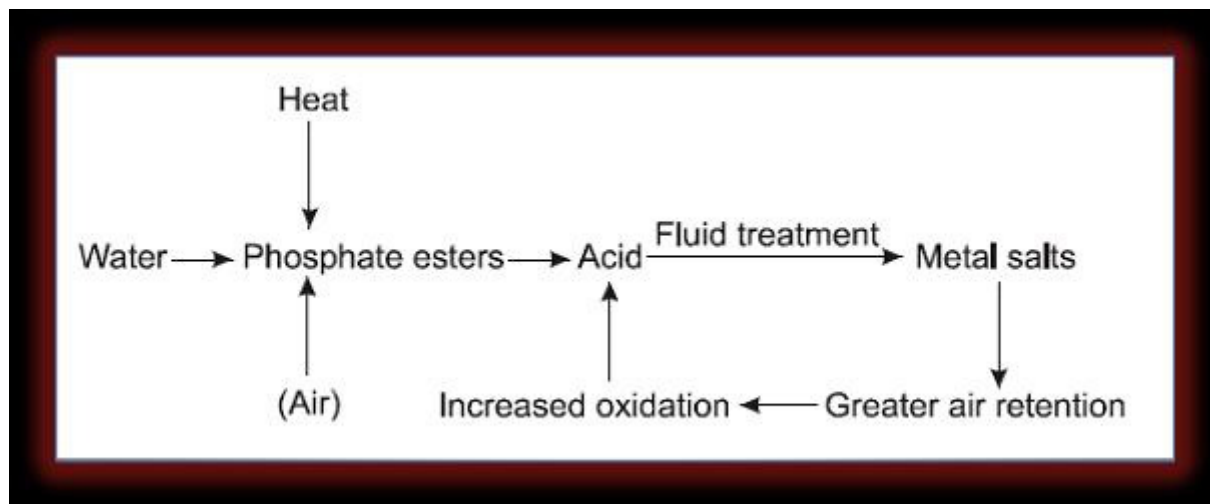
Figure 1: Hydrolysis of triaryl phosphates

### Problems

- The final product of the reaction chain is phosphoric acid. Acids will catalyse the hydrolysis of esters. So this acid has to be removed, to keep the fluid in an acceptable condition.
- Other by-products can occur, too. For example the reaction of two partial esters from step 1 (Fig. 1) would lead to additional complications in the use of phosphate esters.

- Furthermore, partial esters can react with metal ions which are present in the fluid. The reaction of these products will form soaps; these are gelatinous materials which are insoluble in their original fluid. This will also cause problems keeping the fluid system in an undisturbed operation.

Below in Figure 2 is an illustration of the degradation cycle of the phosphate esters within hydraulic oils.



**Figure 2: Degradation cycle of phosphate esters in hydraulic systems**

This cycle can be directly influenced by fluid treatments such as Fuller`s Earth and activated alumina (the current industry standards), which contain small amounts of calcium and magnesium carbonates; basic species that react well with the strongly acidic organo-phosphate species to form soaps.

Recent studies have focussed on a process referred to as dieseling – the high pressure and temperature thermal and oxidative degradation of phosphate esters that results in the rapid darkening of fluids and the formation of a thin layer of deposits or varnish on the surfaces of components<sup>2</sup>.

### **Causes of Decomposition of Phosphate Esters**

There are three main paths for degradation of PEs: thermal degradation, oxidation and hydrolysis (Fig. 3). Each path can become significant at different environmental conditions to which the phosphate ester is exposed:

- All three paths produce acid phosphates (phosphoric acid derivatives) as the main harmful degradation product.
- All three degradation mechanisms progress at increasing rates, as temperature increases.
- Oxidation and hydrolysis are also catalysed by metals such as iron and copper

<sup>2</sup> Phillips, W.D.; The high-temperature degradation of hydraulic oils and fluids; Journal of synthetic lubrication, 23, pp. 39-70, 2006

- Thermal degradation of phosphate esters becomes significant at very high temperatures (above 150°C). This degradation path becomes significant in cases of equipment malfunction or in some special situations such as brake system cylinders where the fluid is not sufficiently insulated from high temperatures

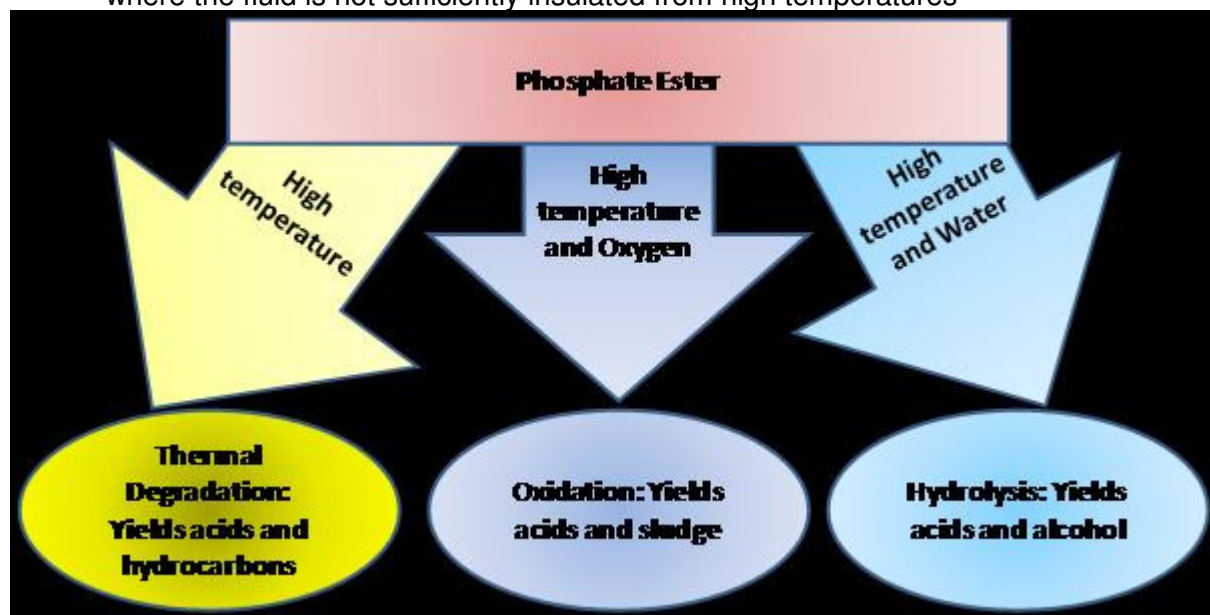


Fig. 3. Decomposition pathways for phosphate ester lubricants

### ***Thermal degradation***

During thermal degradation, alkyl groups of the phosphate esters break up from the molecule to form unsaturated hydrocarbons (such as butane) and leave behind a phosphoric acid derivative.

**Table 1. Thermal stability of phosphate esters under nitrogen by thermal gravimetric analysis**

Weight loss (%)	Temperature (°C)					
	TPP	IPPP	TBPP	TCP	TBP	TOP
10	261	274	301	278	154	208
20	281	292	320	298	173	231
30	294	304	333	310	183	242
50	310	320	350	325	196	257
75	323	334	365	325	207	268

Key: TPP – Triphenyl phosphate; IPPP – Isopropylphenyl phenyl phosphate; TBPP – tert-butylphenyl phenyl phosphate; TCP – Tricresyl phosphate; TBP –Tri-n-butyl phosphate; TOP – Trioctyl phosphate.

Source: Rudnick LR, and Shubkin RL (editor). *Synthetic lubricants and high-performance functional fluids*. Marcel Dekker. Inc, NY, 2005

Table 1 shows the thermal stability of phosphate esters. The data in Table 1 show that significant loss does not begin to occur until well above 150°C.

### ***Oxidative stability***

The oxidative stability of phosphate esters has proven to be quite high and has not been a significant deterrent to their commercial use. Table 2 shows oxidative stability of some phosphate esters.

**Table 2. Oxidative stability of phosphate esters**

Phosphate ester	Time (min)	Temperature (°C)	Percent of original product		
			Non-oxidized	Oxidized	Evaporated
TBP	5	225	13	6	51
	10	225	19	8	73
TCP	30	225	85	1	14
	60	225	67	2	31
TCP	30	250	57	3	40
TXP	30	250	65	4	31
TCP	15	270	60	5	35
TXP	15	270	55	6	39
DBPP	360	250	77	<1	22
	180	270	81	<1	15

Key: TBP - Tri-n-butyl phosphate; TCP - Tricresyl phosphate; TXP – Trixylenyl phosphate; DBPP – Dibutyl phenyl phosphate.

Source: Rudnick LR, and Shubkin RL (editors). *Synthetic lubricants and high-performance functional fluids*. Marcel Dekker, Inc, NY, 2005.

The results show that oxidative degradation of phosphate esters is dependent on the structure of the alcohol from which the phosphate ester was derived. The higher molecular weight products of oxidation are condensation products of the oxidized hydrocarbons.

### **Hydrolytic stability**

Phosphate esters can be considered as to the reaction products of an organic alcohol and inorganic phosphoric acid. Like all esterification reactions, the preparative reaction can be reversed in the presence of water. This means that hydrolysis occurs under proper conditions. **Hydrolysis is the most important consideration in commercial application of phosphate esters. Hydrolysis is very important in evaluating both product and system performance.**

As in thermal and oxidative degradation processes, hydrolysis reaction will produce acidic products, since it is the –O–C– bond that is attacked as illustrated below:



This hydrolysis reaction proceeds stepwise, first yielding the starting alcohol, phenol or alkyl phenol and a substituted phosphoric acid ester.

If the reaction is driven to completion, phosphoric acid and the starting alcohol/phenol will be generated, although lubricant operating conditions are not harsh enough to allow this to occur to any significant extent. Hydrolysis reactions are acid catalysed. This means that acid products of hydrolysis further catalyse the decomposition of phosphate esters. Tables 3 and 4 show the hydrolytic stability of commercial phosphate esters. The data show that higher

molecular weight alkyl chains are more stable than chains of lower molecular weight. The data also show that branched chains are more stable than straight chains, and that alkyl dicresyl esters are more stable than the corresponding alkyl diphenyl esters. While esters of all three types have been successfully used as fluid base stocks, only the TBPP and IPPP esters are important today in industrial uses. The useful operating range of phosphate ester families can be extended by blending chemically or physically.

**Table 3. Hydrolytic stability of some commercial phosphate esters hydraulic fluids and base stocks**

Phosphate ester fluid	Increase in fluid acidity (mg KOH/g)	Increase in water acidity (mg KOH/g)	Copper weight loss (mg/cm <sup>2</sup> )
TCP	0.095	9.1	0.30
TXP	Nil	Nil	0.03
TBPP 46	0.045	6.17	0.18
TBPP 68	0.09	8.98	0.34
TBPP 100	0.20	11.2	0.45
TBPP 32 Gas turbine oil	0.0	4.21	0.0
TBPP 46 MIL H 19457d	0.03	2.71	0.05
IPPP 46 Industrial fluid	0.05	1.4	0.05
IPPP 100 Compressor oil	0.12	1.3	0.04
<b>Standard criteria</b>	<b>0.2 maximum</b>	<b>5.0 maximum</b>	<b>0.3 maximum</b>

Key: TCP – Tricresyl phosphate; TXP – Trixylenyl phosphate; TBPP – tert-butylphenyl phenyl phosphate; IPPP – Isopropylphenyl phenyl phosphate;

Source: Rudnick LR, and Shubkin RL (editors). *Synthetic lubricants and high-performance functional fluids*. Marcel Dekker, Inc, NY, 2005.

**Table 4. Hydrolytic stability of some commercial phosphate esters**

Phosphate ester	Average water acidity (mg KOH/g)	Copper weight loss (mg/cm <sup>2</sup> )
Unformulated esters		
TCP	12	1.1
TXP	7	0.5
IPPP 22	37	12
IPPP 32	24	2
IPPP 46	14	0.4
IPPP 68	9	0.1
TBPP 22	25	0.7
TBPP 46	10	0.6
TBPP 100	3	0.5
TBEP	250	0.3
TBP	85	1.0
TOP	8	>0.1
Formulated esters		
TXP	1	0.1
TBPP 32	8	0.1
TBPP 46	3	0.1
<b>Standard criteria</b>	<b>5.0 maximum</b>	<b>0.3 maximum</b>

Key: TCP – Tricresyl phosphate; TXP – Trixylenyl phosphate; TBPP – tert-butylphenyl phenyl phosphate; IPPP - Isopropylphenyl phenyl phosphate.

Source: Rudnick LR, and Shubkin RL (editors). *Synthetic lubricants and high-performance functional fluids*. Marcel Dekker, Inc, NY, 2005.

## Performance of Phosphate Ester Systems

Phosphate esters are generally not corrosive to common metals used in hydraulic systems. This reliability is best illustrated by performance of a number of commercial fluids in the corrosion/oxidation test (Table 5). Performance data of several phosphate ester products are shown in Tables 5 and 6. As previously indicated the decomposition products of thermal or hydrolytic reactions are acidic and can be corrosive to metals. Copper and copper-bearing alloys will typically be the first metals attacked in such conditions, and nitrogen-containing, metal-passivating compounds.

**Table 5. Performance of industrial phosphate ester hydraulic fluids in corrosion/oxidation test <sup>4</sup>**

Properties	Limits	IPPP 32 Gas turbine oil	IPPP 46 Industrial hydraulic fluid	TBPP 46 MIL H 19457d fluid	IPPP 100 Compressor oil	Low temperature hydraulic fluid
<b>Conditions</b>						
- Hours		72	72	72	168	72
- °C		175	175	175	150	175
TAN increase, mg KOH/g	2.0 max.	0.4	0.16	1.8	1.0	0.14
Viscosity change, %	-5% to +10%	2.3	1.7	13	8.1	7.4
Metal weight change, mg/cm <sup>2</sup>						
- Copper	±0.4	-0.007	0.015	0.051	0.01	-0.015
- Steel	±0.2	0.029	-0.036	0.007	Nil	-0.007
- Magnesium	±0.2	0.015	-0.029	0.015	0.02	Nil
- Aluminium	±0.2	0.015	-0.029	Nil	-0.02	Nil
- Silver	±0.2	Nil	-0.022	-0.095	0.02	-0.007
- Pass/fail		Pass	Pass	Pass	Pass	Pass

Key: TBPP - tert-butylphenyl phenyl phosphate; IPPP - Isopropylphenyl phenyl phosphate; TAN – total acid number

Source: Rudnick LR, and Shubkin RL (editors). *Synthetic lubricants and high-performance functional fluids*. Marcel Dekker, Inc, NY, 2005.

**Table 6. Performance of phosphate ester fluids (Vane pump wear test – ASTM D-2882)**

Phosphate ester fluid	Duration (h)	Temperature (°C)	Weight loss (mg)		
			Ring	Vane	Total
IPPP 22	100	53	3.6	14.5	18.1
	250	53	6.5	15.6	22.1
IPPP 32	100	60	2.5	34.8	37.3
	250	60	2.6	36.7	39.3
IPPP 46	250	66	8.2	8.5	16.7
IPPP 68	100	72	3.7	2.0	5.7
	250	72	5.4	3.3	8.7
TBPP 32	100	66	2.0	5.4	7.4
TBPP 46	100	66	4.3	8.5	12.8

Key: TBPP - tert-butylphenyl phenyl phosphate; IPPP - Isopropylphenyl phenyl phosphate.

Source: Rudnick LR, and Shubkin RL (editors). *Synthetic lubricants and high-performance functional fluids*. Marcel Dekker, Inc, NY, 2005.

## Measures to Minimise Degradation of Phosphate Esters

A number of measures are recommended to minimise this process which are largely focussed on reducing air bubble formation within the fluid, and increasingly, more intensive fluid management approaches are being recommended throughout industry literature as the best way to extend fluid lifespan<sup>3</sup>.

A number of fundamental conditions have been highlighted as major contributors to PE-based fluid degradation, many of which could be easily excluded with more exacting fluid management:

- Purification media not changed often enough or changed incorrectly.
- Purification system flow rate too high or too low and/or not in-service continuously.
- Use of inferior or dated parts and/or bad procedures
- Use of wrong type of phosphate ester.
- Lack of suitable condition-monitoring devices (e.g. pressure gauges).
- Non-application of correct OEM maintenance and operating procedures.
- Use of inappropriate OEM documentation or procedures.

## Monitoring and Control Phosphate Ester systems

Addressing the degradation cycle with the use of an intelligent conditioning and monitoring system would deliver significant benefit. As a basis for this control system a number of regular tests for phosphate ester based fluid formulations are established that constitute good indicators of performance, Table 1 contains data that will be accounted for in the development of the monitoring system within the SAFELUBE prototype:

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<sup>3</sup> Ken Brown, Utility Service Associates, "Condition-Monitoring of Phosphate Ester Hydraulic Fluids". Machinery Lubrication Magazine. November 2002

**Table 7. Routine test program for condition monitoring**

	ASTM NO.	2-Month Sample <sup>(1)</sup>	6-Month Sample	12-Month Sample
ASTM Color Value	D1500	X	X	X
Appearance	-	X	X	X
Viscosity at 40 °C, mm <sup>2</sup> /s	D445	X	X	X
Acid Number, mg KOH/g	D974	X	X	X
Chlorine Content, mg/kg <sup>(2)</sup>	-	X	X	X
Water Content, mg/kg	D6304-98	X	X	X
Particle Count	<sup>(4)</sup>	X	X	X
Mineral Oil Content, ml/1	-	X	X	X
Resistivity at 20 °C, giga-ohm cm <sup>(2)</sup>	D1169	X	X	X
Elemental Spectroscopy, mg/kg	D5185 or D6595		X	X
Flash Point, °C	D92 or D93			X
Relative Density at 15 °C	D1298 or D4052			X
Foaming (Tendency and Stability), ml	D892			X
Air Release at 50 °C, Minutes	D3427			X
Other <sup>(3)</sup>	-			X

<sup>(1)</sup> Some tests can be performed weekly, monthly or quarterly based on history and/or OEM recommendations. It is recommended to take a sample as soon as possible after resuming operations following an outage.

<sup>(2)</sup> Not normally necessary on systems without servo valves. Typically tested to GE method E50A345.

<sup>(3)</sup> Could include DGA (dissolved gas analysis), FTIR, heptane patch test or heptane insolubles, blue soap test. Hatch number high shear foam test and/or tests required for specific problems.

<sup>(4)</sup> ISO 11500 for optical (laser) particle counters.

The key parameter in considering the degradation of PE-based fluids is Total Acid Number (TAN), and it is this parameter that is specifically highlighted in the SAFELUBE project. A strategy has been agreed between project partners where a library of existing oil samples of varying age and condition will be accessed and a series of analytical parameters examined in order to identify the key amongst them with regards to fluid condition monitoring.

**The focus of the characterisation of turbine oils within the SAFELUBE project is to be specifically targeted at the following parameters which will provide vital information**

on the condition of the oil itself and also provide indications of any turbine wear associated with poor lubricant performance.

- Particulate size analysis (5-50 micron)
- Elemental analysis (e.g. Zn, K, Ca, Ba, S, P and Cl)
- TAN & analysis of individual acid species
- Water content

To get an applicable fluid it is necessary to remove water and acids from phosphate esters. **The use of ion exchange technology will be the correct way to handle the acid content, because traditional adsorption technology for acids causes other problems.** Particles should be removed as well; particles will enhance the abrasion of the hardware that is in contact with the fluid. Furthermore, excess particles could stick to servo valves in a steam turbine and this could lead to a loss of electricity.

The following tables (Tables 8 and 9) summarise some specifications that will help to define appropriate targets for the treatment activities in Work Package 3.

**Table 8. Maximum acceptable water and TAN values in EHC systems**

Turbine OEM	Water Content[ppm]	Acid Number [mg KOH/g <sub>Fluid</sub> ]
Westinhouse	1000	0,25
General Electric	2000	0,20
ABB	1000	0,15

**Table 9 Summary of OEM recommended limits for different EHC systems**

Parameter	OEM #1 recommended limit	OEM #2 recommended limit
Water Concentration	0,20 %	0,10 %
Total Acid Number	0,20mg KOH/g fluid	0,25 KOH/g fluid
Particle Count		
5-10 µm	24000/100 mL	9700/100 mL
10-25 µm	5360/100 mL	2680/100 mL
25-50 µm	780/100 mL	380/100 mL
50-100 µm	110/100 mL	56/100 mL
>100 µm	11/100 mL	5/100 mL
Particle Count <sup>(1)</sup>		
>5 µm	303/mL	128/mL
>10 µm	63/mL	31/mL
>25 µm	9/mL	4/mL
>50 µm	1/mL	1/mL
>100 µm	0/mL	0/mL

<sup>(1)</sup> Approximate particle count limits obtained by converting the original differential values per 100 mL to cumulative counts per 1mL to conform to ISO 4406

## Conclusions

Phosphate ester fluids and lubricants undergo thermal degradation, oxidation and hydrolysis resulting in acidic by-products that can corrode or oxidize metal-equipment components.

Some of the performance data discussed above (especially, Tables 5 and 6) indicate that proper maintenance and control of mechanical systems can assure excellent performance and result in exceptionally long fluid or lubricant life. The system maintenance should involve three broad areas:

1. Design and preparation of equipment and its components
  2. Maintenance programs to preserve the mechanical integrity of the system and prevent contamination of the fluid
  3. Operating procedures that include conditioning and periodic chemical analysis of the fluid.
- Design and preparation of equipment is an important first step. Suppliers of phosphate ester fluids and lubricants have detailed recommendations on materials of construction. These suppliers should be consulted early in the design stage of new equipment or when conversion of older equipment to phosphate esters is being planned.
  - Recommended procedures and control limits for phosphate ester systems should be followed as a means of establish a maintenance program. Use of specialist handbooks describing operating and maintenance procedures for systems using phosphate ester fluids and lubricants is also recommended.
  - **Maintenance of low water and acid contents is the best operational procedure for maintenance and control of mechanical systems to assure excellent performance and long fluid or lubricant life.**
  - We have already identified several limitations with current techniques for the measurement of water, TAN and chlorine levels in PEs as: interference from contaminants; range & sensitivity the outside our scope; high maintenance and calibration requirements; high costs; sensor fouling; prone to EM interference.
  - **We intend to transcend the limitations of state-of-the-art sensor analysis techniques for the measurement of water, TAN and chlorine in PE's by developing innovative sensing technologies based on solid state laser diode technology.** The use of solid state laser diodes for the measurement of water, TAN and chlorine in lubricants is entirely new. We also aim to develop new diagnostic algorithm/software to determine the condition of the PE and recommend and implement corrective actions (water removal and/or acidity removal).
  - Furthermore we aim to develop innovative dehydrating and degassing technology that will operate on-line with minimal or no manual intervention for the PE.
  - These innovative sensors, diagnostic algorithm and dehydrating/degassing technologies will be integrated to deliver a complete monitoring and conditioning prototype system for PEs in power turbines.

## **Deliverable D2; Computational model to predict the operational life of the lubrication & resin**

The aim of this deliverable was a computational model to predict the operational life of the lubricant and resin. At month 6, when the deliverable was due the consortium did not have a turbine supply partner in it and therefore was not able to specify which lubricant, and hence which resin, the model should be based on. This partner was not found by the termination date of the project and therefore this deliverable has not been completed.

## **Deliverable D3; Target specification for PE monitoring & conditioning system**

The partners working together with the RTD performer have developed a detailed performance specification. This performance specification acts as the target document that will be used at the end of the project to review the performance of the prototype Safelube system demonstrator. The specification covers the following aspects:

- Target life for PE
- Definition of moisture analysis range and target limits for the PE
- Definition of target acid number range and limits for the PE
- Verification moisture sensor
- Verification of TAN sensor
- Definition of moisture and acid monitoring frequency
- Definition of moisture “correction” period
- Target life for ion-exchange absorbent resin
- Capital costs.
- Physical size

## **TECHNICAL OBJECTIVES**

The main technical objectives for the Safelube project are follows:

- Develop laser diode water sensor to measure absorption peaks in the 4000-800cm<sup>-1</sup> range
- Develop laser diode TAN sensor to measure absorption peaks in the 4000-800cm<sup>-1</sup> range.
- Development of a diagnostic expert system to assist users in PE condition monitoring
- Development of a regenerative dehydration system based on turbulent flow membrane filtration
- Robust fibre-optic transmission and scada communications architecture

In addition:

- System must be competitive with existing manual solutions
- Small footprint to enable retrofit installation

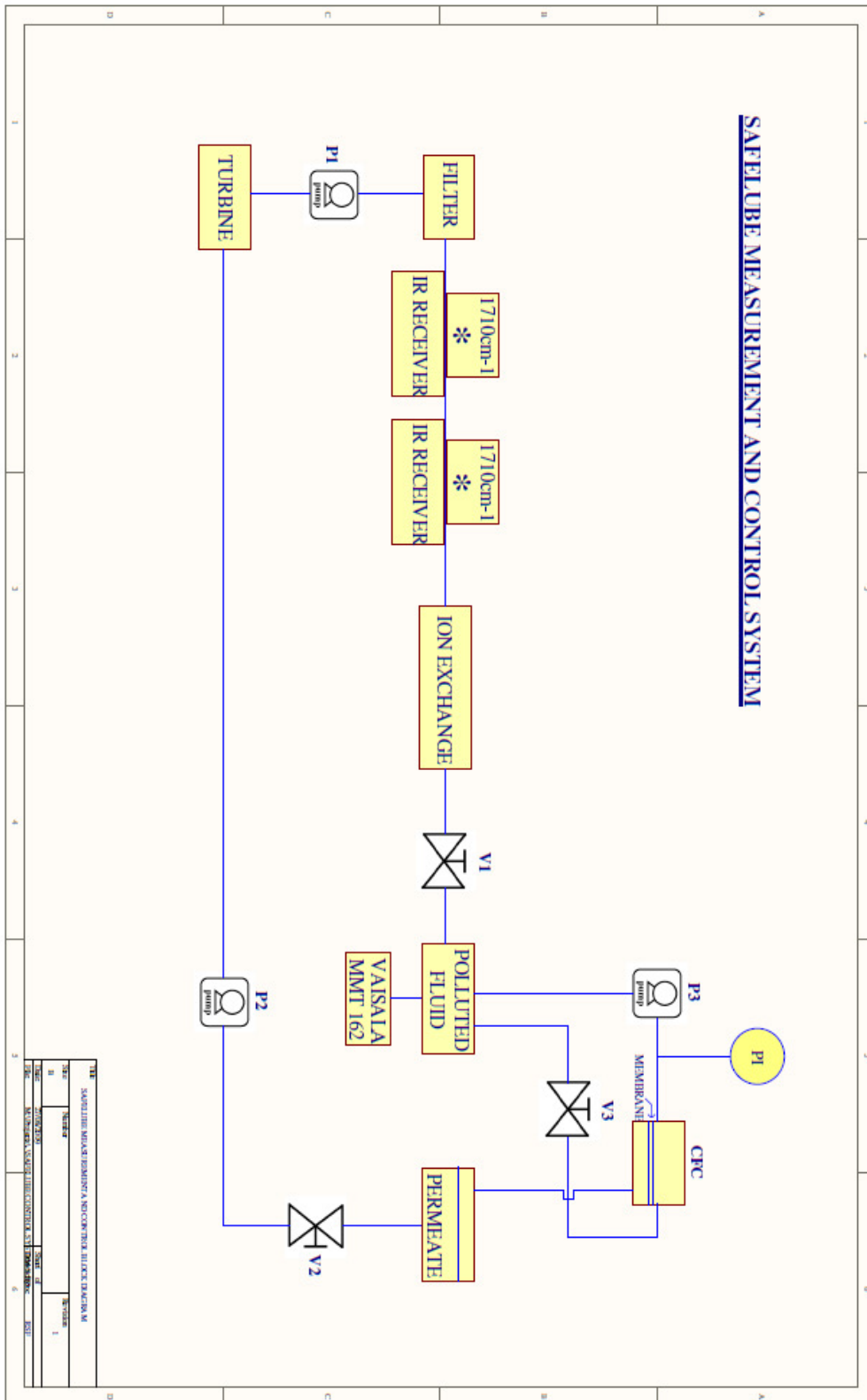
## **TARGET SPECIFICATION**

- Target life for PE to be 30 years to align with the lifespan of the turbine generator
- The target moisture analysis range will be 0 to 2100ppm to control limits of 0 to 2000ppm within the PE.
- The target acid number range will be 0 to 0.20 mgKOH/g and 0 to 0.15mgKOH/g limits for the PE

- The moisture and TAN sensor will be measured with a Alpes SB105 QCL laser.
- The target moisture and acid monitoring laser will operate within the wavelength range of 6200 to 6300nm.
- The target moisture “correction” period will be defined at a later stage when the demonstration turbine has been defined as this is a function of the turbine lubricant design and the space available.
- Target life for ion-exchange absorbent resin one year to align with the routine service intervals of turbine
- The target capital costs is <€50,000.
- The target physical space for the system is <1.5m<sup>3</sup>.

## **FUNCTIONAL CONTROL AND COMMUNICATIONS DIAGRAM**

Figure 4 presents the functional control and communications diagram.



**Figure 4: Functional Control and Communications Diagram**

## **CONCLUSION**

The above target specifications have now been established and represent the current views and requirements expressed by the consortium members. The specifications will be reviewed at regular intervals throughout the project to ensure that the specifications remain relevant.

## WORKPACKAGE 2 – LASER DIODE SENSOR DEVELOPMENT

**Objectives:** To develop solid state single frequency laser diodes as follows: water sensor - specifically for measuring the water content in PE's in the region of 0-1500ppm with a sensitivity response of 5%; TAN sensor - specifically for measuring the TAN in the range 0-0.5 mg/KOH/g with a sensitivity response of 5%.

Deliverables D4, D5, D6, D7 and D8 have been produced to meet these objectives.

### **Deliverable D4; Specification for laser diode design to enable Identification of laser diode properties**

#### ***Measurement of water content***

**Water absorbs strongly at  $3333\text{ cm}^{-1}$  however there is currently not a low cost solid state laser diode available at this wavelength, it is therefore necessary to use a broad band source to interrogate this region of the spectrum** – a technique widely applied in oil quality monitoring<sup>4</sup>. Indeed, water in oil analysis technology using solid state probes is relatively mature technology and can be readily sourced and implemented<sup>5</sup>. Greater challenges are posed by the monitoring of other parameters

#### ***Measurement of total acid number***

It was decided at a technical Working Group meeting that it would be beneficial to refine the target of Total Acid Number (TAN) as there exists in the market place a number of instruments that offer relatively swift on-site (but not on-line) analysis of oil samples for this important parameter<sup>6</sup>. It was highlighted that TAN, although being employed as a standard parameter throughout the industry, does not provide any information about the nature of the different acids within the sample. The TAN value is normally derived from titration with KOH, and does not make any distinction between the different types of acid present in the sample.

### **Different Acid Species in Oil Samples**

There is potentially a large range of different acid species in an oil sample; a number of these are of great importance as they have the potential to impact severely on the performance of the lubricant:

- Formic acid -  $\text{HCO}_2\text{H}$
- Acetic acid -  $\text{CH}_3\text{CO}_2\text{H}$
- Levulinic acid -  $\text{CH}_3\text{COCH}_2\text{CH}_2\text{CO}_2\text{H}$

The above acids are considered “strong” acids – the acidic proton is easily dissociated from the anion. Hence, such species can have a large impact upon lubricant condition.

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<sup>4</sup> For Example, H2Oil system: [www.parker.com](http://www.parker.com)

<sup>5</sup> For example, Vaisala HUMICAP® Moisture and Temperature Transmitter for Oil MMT318: [www.vaisala.com](http://www.vaisala.com)

<sup>6</sup> For example the Fluidscan system: [www.foster-miller.com](http://www.foster-miller.com)

In comparison, other acidic species present in oil samples do not have a drastic impact on lubricant condition as they are considered “weak” acids, such as:

- Naphthenic acid -  $C_{10}H_7CO_2H$
- Stearic acid -  $C_{17}H_{35}CO_2H$
- Oleic acid -  $C_8H_{17}CH=CHC_8H_{16}CO_2H$

Since there may be a very significant contribution to the TAN from the weak acids within the oil, it may be the case that TAN could be misrepresentative in two ways:

False negative: if [strong acid]  $\gg$  [weak acid], but total TAN below warning level, action would not be taken. The high concentration of strong acids within the oil would however, have a significant negative contribution to lubricant performance, creating potential fire hazard.

False positive: if [strong acid]  $\ll$  [weak acid], TAN value would trigger remedial action where none was required, weak acids having little detrimental impact upon lubricant performance and posing little inflated risk, yet may be unnecessarily disposed of on the basis of TAN analysis.

## Definition of the Exact Nature of Acids

**It was therefore decided to target part of the SAFELUBE system tasked with defining the exact nature of the acids found within oil samples. It was thought that this should prove to be possible by examining the FTIR spectra of the oil very closely.**

- It is known that the position of the C=O vibration found within the FTIR spectra of carboxylic acids is dependent up the nature of the rest of the molecule. The wavenumber position of this peak will be reduced as the chain length of the associated molecule increases.
- Previous studies of a number of carboxylic acids in oils have been undertaken by monitoring the acid group FTIR spectra at  $1710\text{ cm}^{-1}$  for the protonated acid and at  $1565\text{ cm}^{-1}$  for the deprotonated acid. Contributions from short chain “strong” acids would modulate the peak position, as would those from long chain molecules.
- It is thought viable that employing the highly tunable QCL light source provided by project partner Alpes it would be viable to scan the FTIR spectra and find the position of the C=O vibration with a good degree of accuracy.
- **It would then be possible to relate the position of this peak to the contributions from short chain “strong” acids, and hence be able to provide a significantly improved data set compared to the traditional TAN value.**

## Specification for Laser Diode Design

The assembly of the laser diode design capable of detecting water and total acid contents in phosphate esters is illustrated in Fig. 5 below.

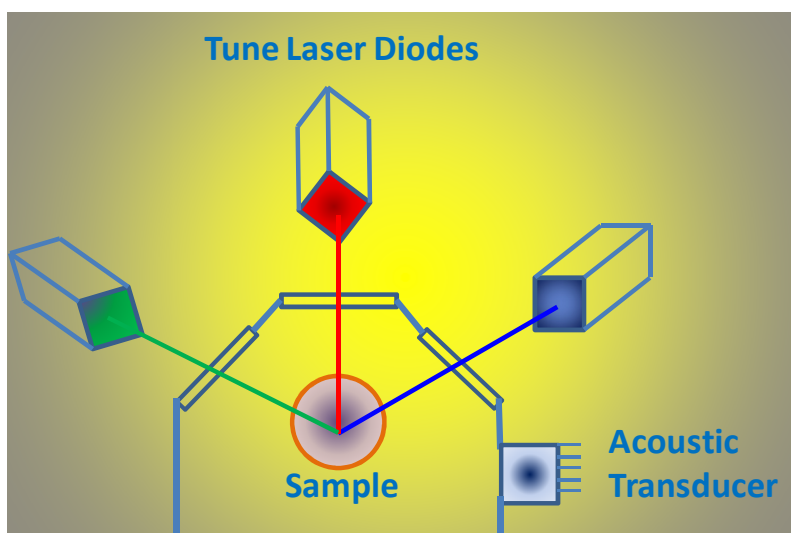


Fig.5. Schematic of Photo Acoustic Spectroscopy cell Using Tuned Laser Diode

### **Solid state Infra-red sources**

Safelube project partner Alpes manufacture a range of solid state IR sources, and one has been highlighted that could be manufactured to overlap with the C=O vibration in carboxylic acids, as shown below in Table 10. An important factor to consider in light source selection is the available power at the desired spectral region – it can be seen that several mW of power could be possible with the QCL source.

**Table 10. Optical output power of Alpes SB105 QCL laser as function of operating parameters**

$\lambda$ [nm]	$\nu$ [ $\text{cm}^{-1}$ ]	P [mW]	Temp. [ $^{\circ}\text{C}$ ]	$U_{\text{LDD}}$ [V]	$I_{\text{pulse}}$ [A]
6246.4	1600.9	1.3	-30	14	4.0
6248.9	1600.3	2.5	-30	16	4.9
6260.7	1597.3	1.0	0	15	4.4
6262.0	1596.9	1.5	0	16	4.9
6264.8	1596.2	2.3	0	18	5.8
6276.4	1593.2	0.4	30	15.5	4.7
6278.0	1592.9	0.6	30	16.5	5.1
6282.2	1591.8	1.1	30	19	6.2

### **Performance criteria**

- The water and TAN contents will be measured with a Alpes SB105 QCL laser (specifications are shown in Table 10 above).
- The water and total acid monitoring laser diode will operate within the wavelength range of 6200 to 6300nm.

- The target water content range will be 0 to 2100ppm to control limits of 0 to 2000ppm within the PE.
- The target total acid number range will be 0 to 0.20 mg KOH/g and 0 to 0.15 mg KOH/g limits for the PE
- The target water “correction” period will be defined at a later stage when the demonstration turbine has been defined as this is a function of the turbine lubricant design and the space available.
- Target life for ion-exchange absorbent resin one year to align with the routine service intervals of turbine

## **Conclusion**

The partners working together with the RTD performer have developed a detailed performance specification. This performance specification acts as the target document that will be used at the end of the project to review the performance of the prototype Safelube system demonstrator. Full specification details are presented in Deliverable 3.

### **Deliverable D5: Prototype laser diode sensors showing proof of concept functionality for water & TAN detection**

The aim of this deliverable was prototype laser diode sensors showing ‘proof of concept’ functionality for water and TAN detection. At month 10, when the deliverable was due the consortium did not have a turbine supply partner in it and therefore was not able to specify which lubricant, and hence what sensors would be appropriate to detect water and TAN. This partner was not found by the termination date of the project and therefore this deliverable has not been completed.

### **Deliverable D6: Sensors for measuring (i) water content in the region of 0-1500ppm with a sensitivity response of 5%; (ii) TAN content in the range 0-0.5mg/KOH/g with a sensitivity response of 5%**

The aim of this deliverable was prototype sensors for measuring: (i) water content in the region of 0-1500ppm with a sensitivity response of 5%; (ii) TAN content in the range 0-0.5mg/KOH/g with a sensitivity response of 5%. At month 16, when the deliverable was due the consortium did not have a turbine supply partner in it and therefore was not able to specify which lubricant, and hence what sensors would be appropriate to detect water and TAN. This partner was not found by the termination date of the project and therefore this deliverable has not been completed.

### **Deliverable D7: Matrix/interference correction for target components.**

The aim of this deliverable was a Matrix/interference correction for target components. At month 16, when the deliverable was due the consortium did not have a turbine supply partner in it and therefore was not able to specify which lubricant, and hence produce a prototype on which a matrix/interference correction for target components could be developed. This partner was not found by the termination date of the project and therefore this deliverable has not been completed.

### **Deliverable D8: Sensor shield**

The aim of this deliverable was a sensor shield. At month 16, when the deliverable was due the consortium did not have a turbine supply partner in it and therefore was not able to

specify which lubricant, and hence produce a prototype sensor on which a sensor shield could be developed. This partner was not found by the termination date of the project and therefore this deliverable has not been completed.

## **WORKPACKAGE 3 – MEMBRANE REGENERATION TECHNOLOGY FOR WATER REMOVAL & ION-EXCHANGE SELECTION FOR TAN REMOVAL**

Objectives: To dehydrate the oil, in-situ, with minimal or no manual intervention using membrane filtration/adsorption technology in an automated system. The moisture removal will be facilitated by chilling the oil in a localised environment prior to membrane filtration to reduce its solubility properties in the oil. To regenerate the membrane filter by electrically heating the filter system to liberate the captured moisture/gases and vent to atmosphere. To remove acids from the oil using in-situ ion-exchange technology.

Deliverables D9, D10, and D11 have been produced to meet these objectives.

### **Deliverable D9: Membrane technology capable of removing water levels to <10ppm.**

#### **Activities:**

During this period a number of different membrane filtration test rigs were constructed and implemented within UMSICHT. These will be used to trial the filtration membrane systems for Safelube. These test rigs are described in detail below:

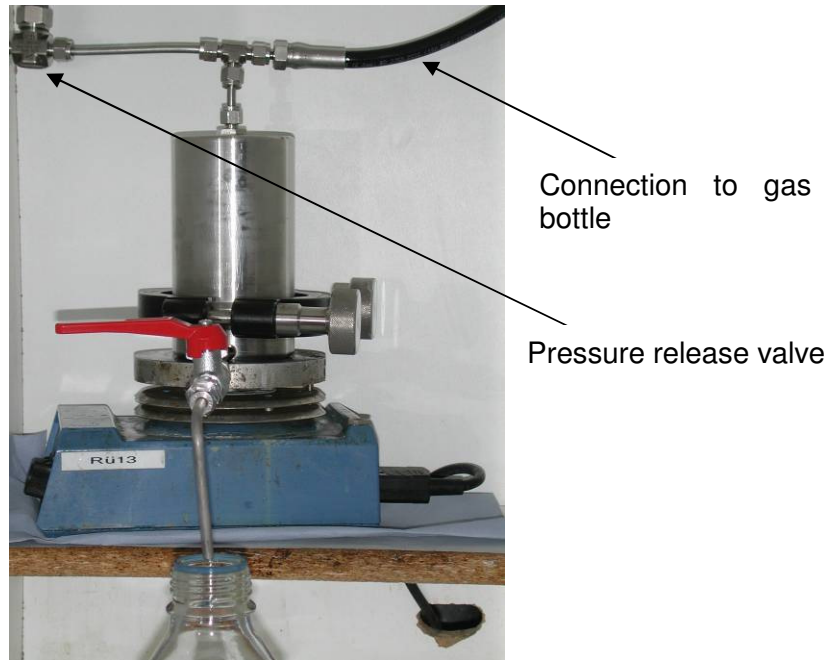
- Discontinuous filtration cell (DFC),
- Cross flow cell (CFC)
- Cross flow cell for ceramic membranes (CM)
- Microfiltration test plant (MTP)
- Tubular rotor filter from MinerWa (TRF)

#### **Discontinuous filtration cell (DFC):**

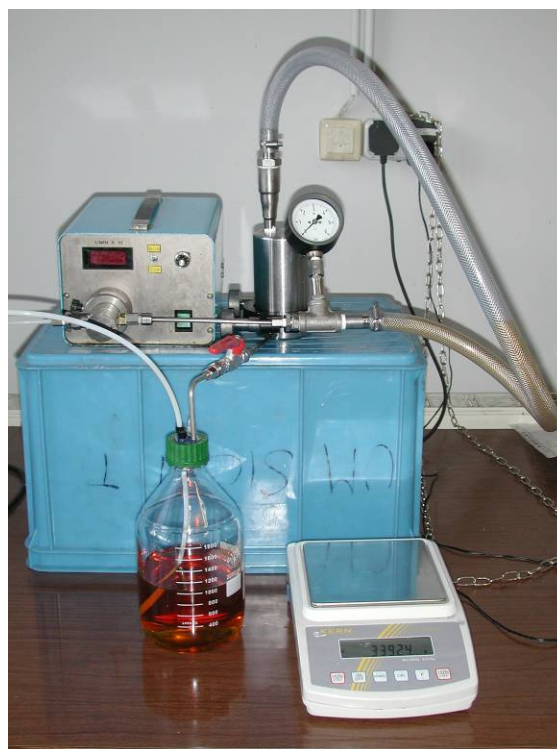
The DFC device is suited for permeability tests. It is made of a straightforward stainless steel arrangement with a filter placed on a perforated plate within a filtration cell that can be pressurised up to 50 bar. The test chamber is filled with phosphate esters and pressurised either with nitrogen from a connected gas bottle or by a pump. This offers the possibility to use the test cell for both batch and continuous processes. Photographs of these possibilities can be seen in Fig. 6 and Fig. 7.

A magnetic stirrer can be mounted in the cell for homogenisation and enhancement of fluid flow over the membrane surface. The filtration is started by opening the permeate valve. The permeate flow is then measured by analysing the weight or the volume of fluid that comes out of the membrane relative to the time that has elapsed. Equal time steps will immediately give an idea of the constancy of the permeate flow.

Further analysis is conducted using a computer. Normally the experiments will be done at ambient temperature, however, if required, to conduct tests at lower temperature it is recommended to put the filtration cell into an ice bath. There it is possible to cool the fluid down to 3°C. Tests at higher temperatures with the batch process can be done by heating the fluid and the test cell before a run separately in an oven. For continuous filtration a heat exchanger could be assembled in the filtration test rig.



**Fig. 6: Photograph of the discontinuous filtration test rig (DFC) in a batch process**



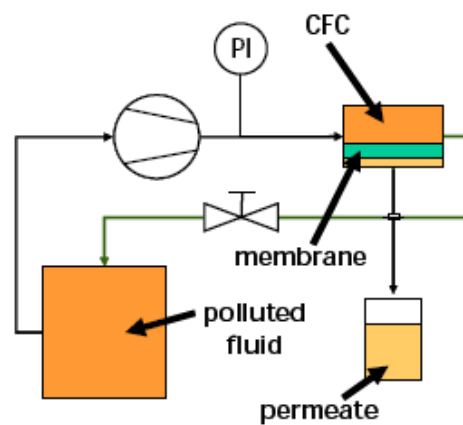
**Fig. 7: Photograph of the discontinuous filtration test rig (DFC) in a continuous process**

The DFC has a screw thread inside. This allows mounting a magnetic stirrer, which is used to prevent particle deposition on the membrane surface. Nevertheless the DFC is prone to blockage of membrane pores. Alternative options to prevent particle deposition on the membrane surface are the use of the cross flow cell (CFC) or the tubular rotor filter (TRF) from MinerWa. The CFC can be used for preliminary tests for the TRF.

### Cross flow cell (CFC):

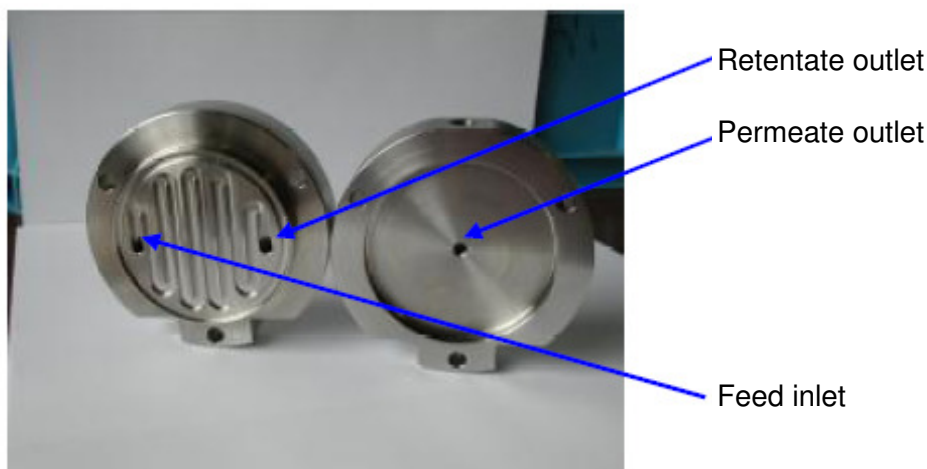
A schematic diagram shows the cross flow filtration principle (Fig. 6). The polluted fluid is sampled from a tank and pumped into the filtration cell. The feed is then directed over the membrane surface. The membrane itself is placed on a perforated plate to provide it with mechanical stability. During the fluid flow, the feed is divided by the membrane into two fractions – the retentate and the permeate. The retentate is directed back into the tank and the permeate is collected separately.

The filtration process can be done at different pressures. The pressure is controlled by the frequency of the pump and the valve that is placed between the retentate outlet and the feed tank. The CFC is able to work at pressures up to 6 bar and maximum temperatures of 45°C. Currently new test cells will be made of stainless steel. This should provide the possibility to increase the maximum operating conditions.



**Figure 8: Schematic of cross flow cell**

Figure 9 below shows the inside of the CFC. The left of the picture shows the loops inside the test cell and the feed inlet as well as the retentate outlet.

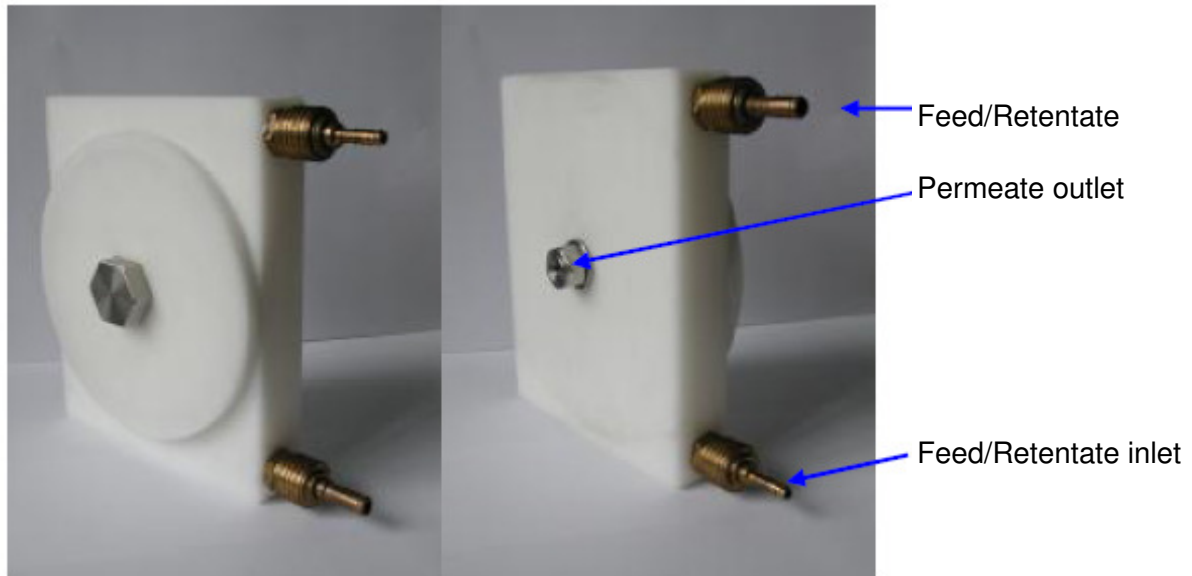


**Figure 9: The upper (left) and lower (right) sides of the cross flow filtration cell**

A similar system to the CFC has been assembled for ceramic membranes; the feed is able to flow over the ceramic membrane on both sides.

#### **Cross flow cell for ceramic membranes (CM):**

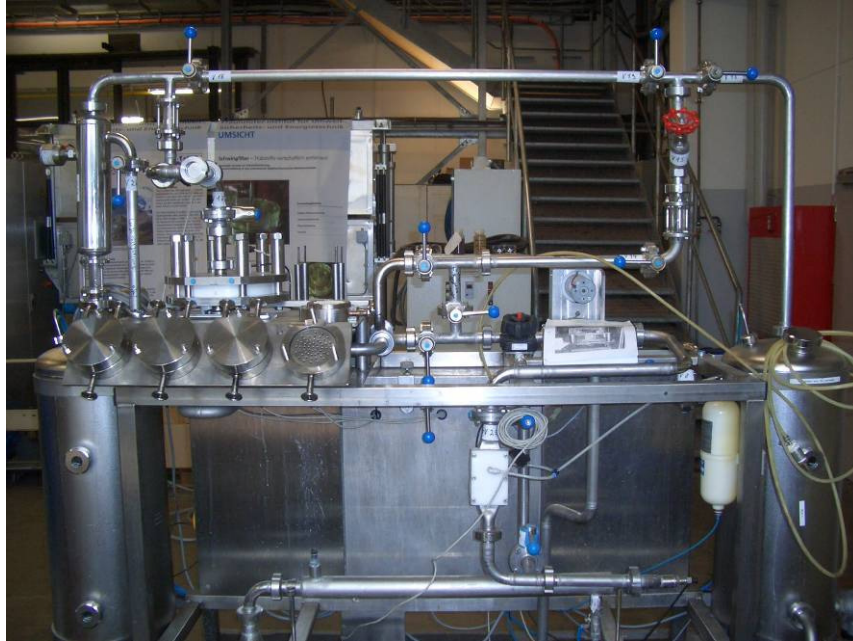
The permeate will be directed into the middle of the membrane where small channels will guide the permeate to the centre of the cell. A hollow screw in the centre of the cell will let the fluid flow out to be collected separately. This test cell is used for preliminary tests with the TRF. Photographs of the test cell are shown in Fig. 10.



**Figure 10: Cross flow cell for ceramic membranes**

#### **Microfiltration test plant (MTP):**

Figure 11 shows a further filtration plant that can be used for microfiltration tests. In general this plant consists of a feed pump, a feed tank, a storage tank for the permeate, and a series of filtration units. A test can be done with one to four of these units, depending on the process parameters. The units can be operated in parallel or in series connection. Three different module arrangements are possible and four different filter media can be tested in parallel.



**Fig. 11: Photograph of the MTP**

#### **Tubular Rotor-Filter (TRF) from MinerWa:**

This system has been supplied by project partner Minerwa, and is intended to form the basis of the filtration approach. The test rig consists of a tank with a volume of up to 100 litres, inside of which anything from 2 to 30 membranes can be mounted. The fluid flows over both sides of the membranes and is then directed into internal channels that collect the permeate. The membranes are mounted on stacks that can be assembled on a rotor, thus allowing movement of the membranes during the filtration process. This working principle is similar to the CFC and will keep the membranes from particle deposition on their surface. Unlike the CFC there will be no retentate. Particles accumulate within the tank of the TRF. A significantly decreased permeate flow indicates the point when the concentrate should be discharged.

The filtration process will start with the filling of the tank. Once some fluid comes out of the valve on the upper side of the tank, a flow sensor will activate the rotor. The upper valve will be closed and the permeate valve will be opened. In ambient conditions it will take some time until the filtration process reaches stable working conditions, because the rotating rotor will increase the temperature inside the tank due to friction forces. Normally, an increased temperature will lower the viscosity of a fluid. This leads to an improved permeate flow because the resistance to enter the membranes will be lower.

Figure 12 is a photograph of the TRF.



**Figure 12: Tubular Rotor-Filter**

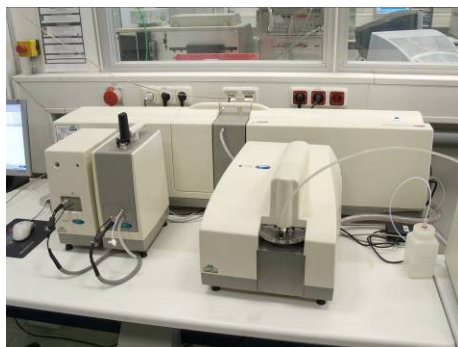
### **Membrane & particle analysis systems**

To assess the effect of pore dimensions it is necessary to analyze the filtered fluid itself. Therefore several particle size measurement devices are available: Mastersizer Hydro 2000, Nano Zetasizer and Accusizer 780.

Furthermore, to assess the effect of membrane pore dimensions it is necessary to observe them with optical methods. Therefore a number of analytical devices from Fraunhofer UMSICHT were used: Light optical microscope, Keyence and  $\mu$ Surf®. With these devices it was also possible to make observations of the filter material itself. This enhances the conditioning of lubricating fluids.

#### **Mastersizer Hydro 2000:**

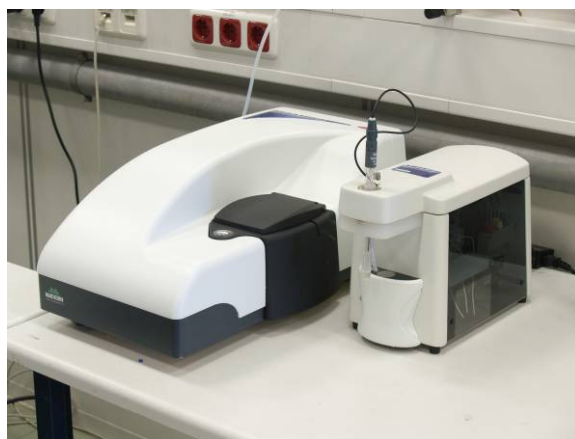
A particle sizer (Mastersizer, Malvern) is applied due to measuring particle size distributions. The working principle of the Mastersizer is based on the interaction of laser light with particles. Due to the fact that the particle size determines the deflection angle of the laser light, the particle size can be measured by detecting the scattering of the light under different angles (static light scattering method). The smaller the particle size, the greater the light deflection and vice versa. By means of complicated mathematic algorithms the particle size distribution can be calculated. Red and blue laser light is applied which allows the adjustment of a measuring range from 0.04 to 2000  $\mu\text{m}$ . Both a measuring cell for aqueous solutions and another for organic solutions with a capacity of 18 ml are provided. An auto sampler for simultaneous measuring of 36 samples is also available. Figure 13 shows a photograph of the Mastersizer Hydro 2000.



**Fig. 13: Mastersizer Hydro 2000**

### **Nano Zetasizer:**

The Nano Zetasizer will measure a particle size distribution on the basis of light intensity that is back scattered from the fluid sample. The fluid sample is filled into a cuvette which is placed in the Nano Zetasizer. There the sample is tempered to a default value. When starting the measurement a laser beam is aimed upon the sample. A sensor is placed at an angle of  $173^\circ$  relating to the incident light. The sensor will detect the intensity of light that is back scattered from the sample. This method is called NIBS (non-invasive-back-scatter). The amount of light that is back scattered is dependent on the particles in the sample. The measured signal will be processed with mathematical algorithms. The Nano Zetasizer has a measuring range from  $0,006 \mu\text{m}$  to  $6 \mu\text{m}$ . Figure 14 shows a photograph of the Nano Zetasizer.



**Fig. 14: Photograph of the Nano Zetasizer**

### **Accusizer 780:**

The Accusizer 780 is a particle counter. The measuring range is from  $0,5 \mu\text{m}$  to  $500 \mu\text{m}$ . Then material from a sample is sampled through a tube by a syringe. The fluid will flow through a channel where it passes a laser beam. Every crossing of a particle through the laser beam will be detected by two sensors.

One sensor is placed in that way that the incident light of the laser beam hits it perpendicularly. This sensor has the function of measuring the extinction of a particle. The sensitivity of this sensor will decrease with decreasing particle size. Therefore a second sensor with a growing sensitivity for small particles is placed in a known angle to the incident light. This sensor will recognize the light that is produced by diffraction when a particle crosses the laser beam.



**Fig. 15: Photograph of the Accusizer 780**

### Light-optical microscope:

A number of light-optical microscopes are available that will allow the investigation of the blockage of membrane pores. This is possible due to a magnification from 500 to 5000 times. A mountable camera will offer the possibility to take shots as shown in Figure 16. Figure 17 shows the surface of an unused nickel sieve. In case of a filtration it would be possible to observe the particle deposition on the membrane. Another feature of the microscopes is that they have the possibility to transmit light. This will offer the possibility to analyse the pore blockage of metallic membranes or sieves.



**Fig. 16: Light-optical microscopes**



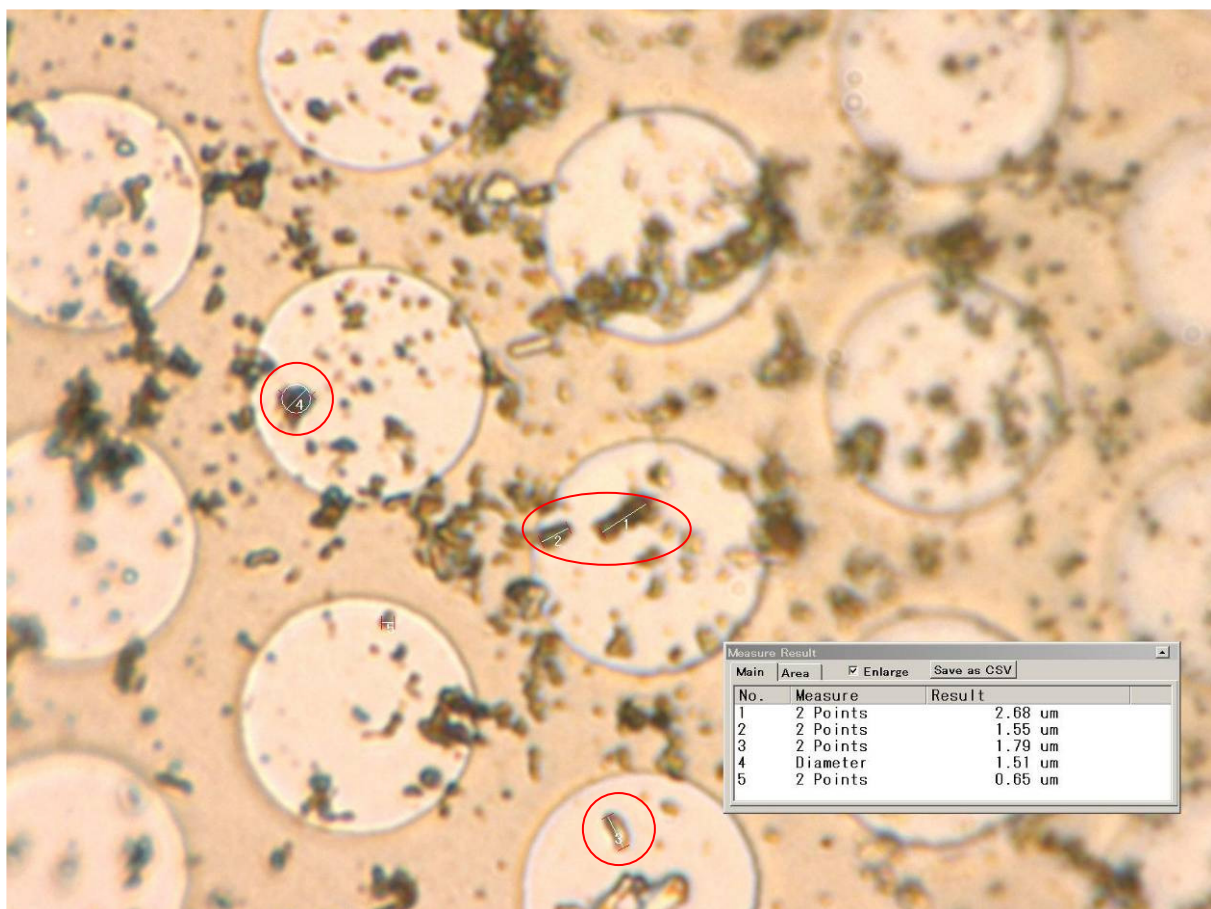
**Fig. 17: Snapshot of the surface of a nickel sieve (open pores are identifiable)**

### Keyence VHX-100

The Keyence microscope (Figure 18) is able to analyse membranes with a magnification up to 5000 times. Furthermore a 3D-analysis is possible by overlaying of pictures that were taken at different heights. Size measurements can be made as well, an example for this can be seen in figure 19. Figure 19 shows the surface of a metal membrane that is contaminated by particles that were filtered from oil. Now it is possible to determine the length of a particle or its diameter.

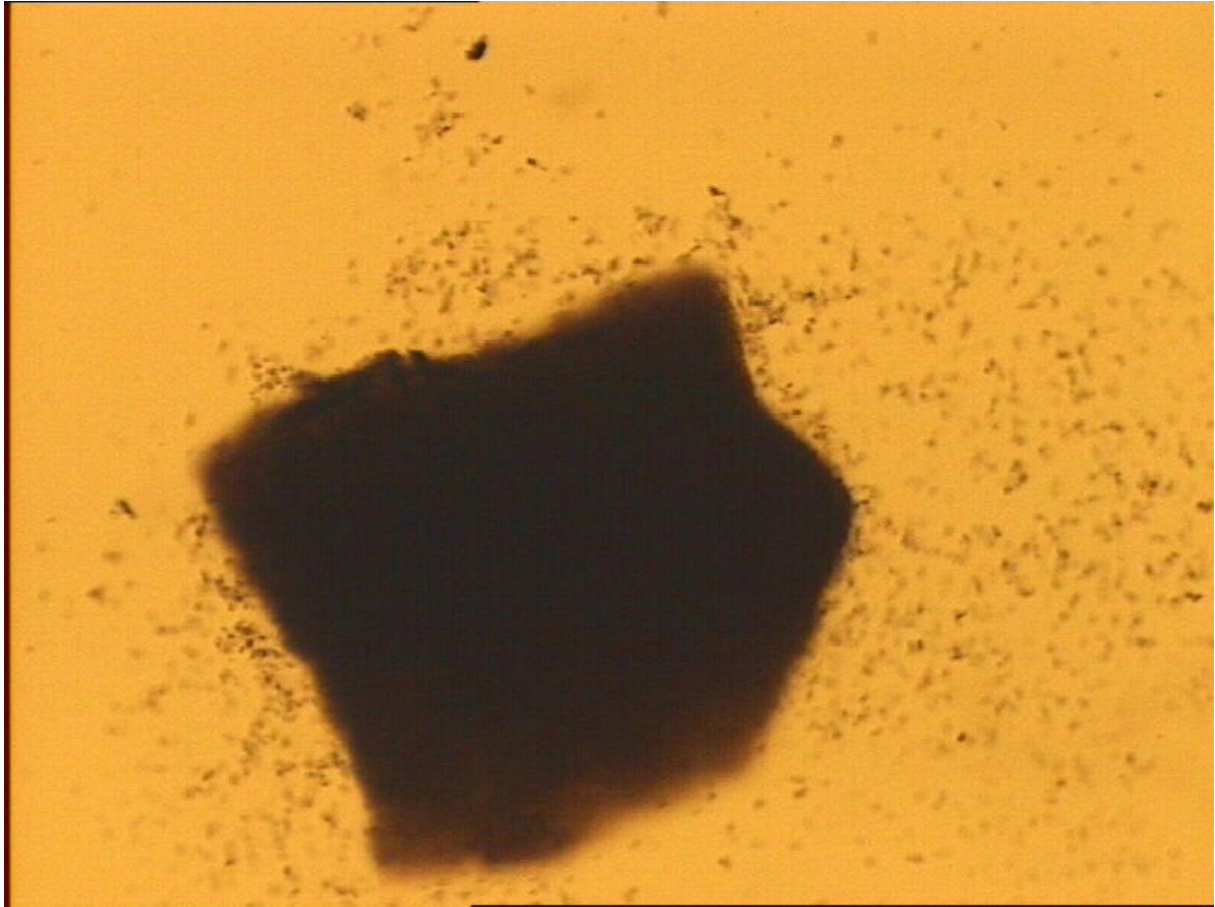


**Fig. 18: Keyence VHX-100 microscope**



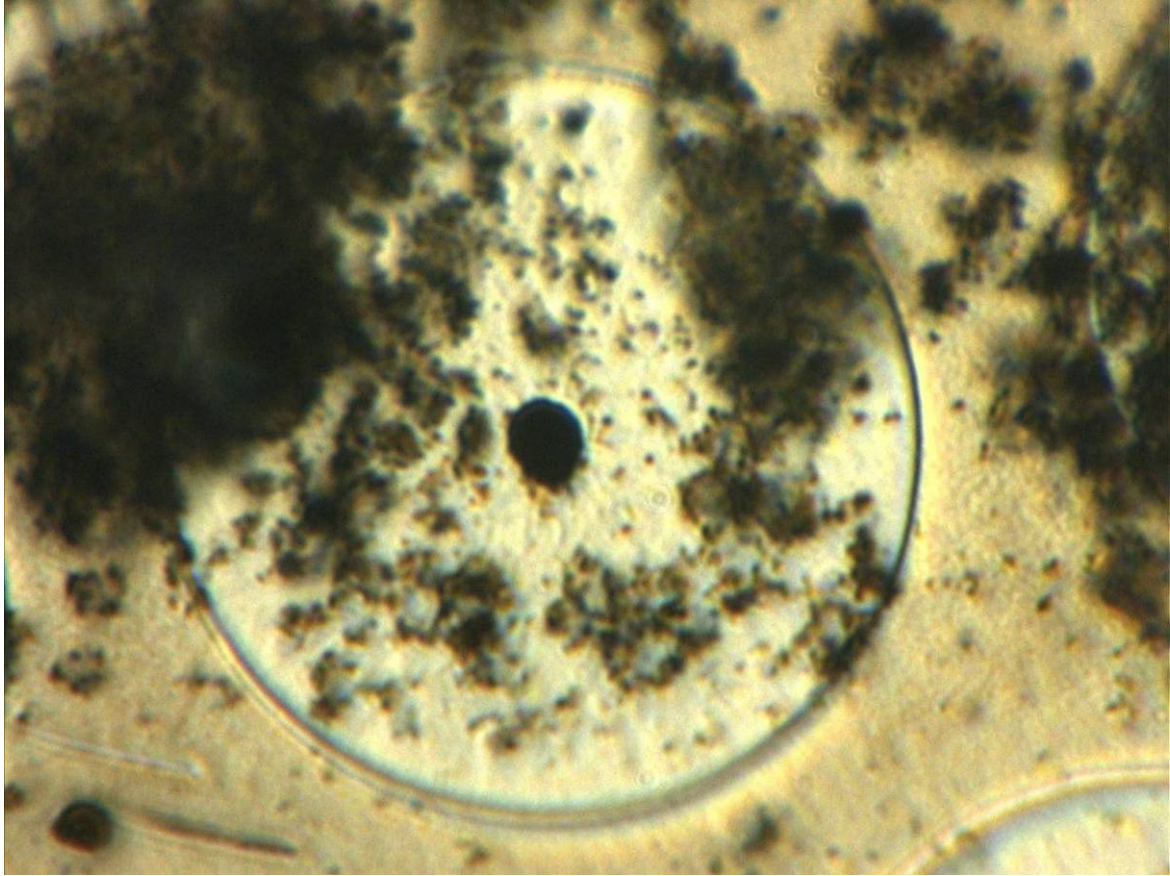
**Fig. 19: Membrane surface contaminated with particles**

Figure 20 shows an example of a particle that was separated from oil. The analysis of this particle helps to determine if it is a wear particle or a particle that was built due to oxidation processes. To determine the parameters of a filtration process for lubricants it is necessary to analyse such particles. First of all this will help to determine the right pore size of the filter. Normally the biggest possible pore size is chosen because this will result in higher permeate fluxes. The second positive finding of the particle analysis is that the operator of the turbine gets some valuable information of the lubricant and which problems maybe arouse. Furthermore the operator has now the chance to change the operating mode to enhance the life of the turbine.

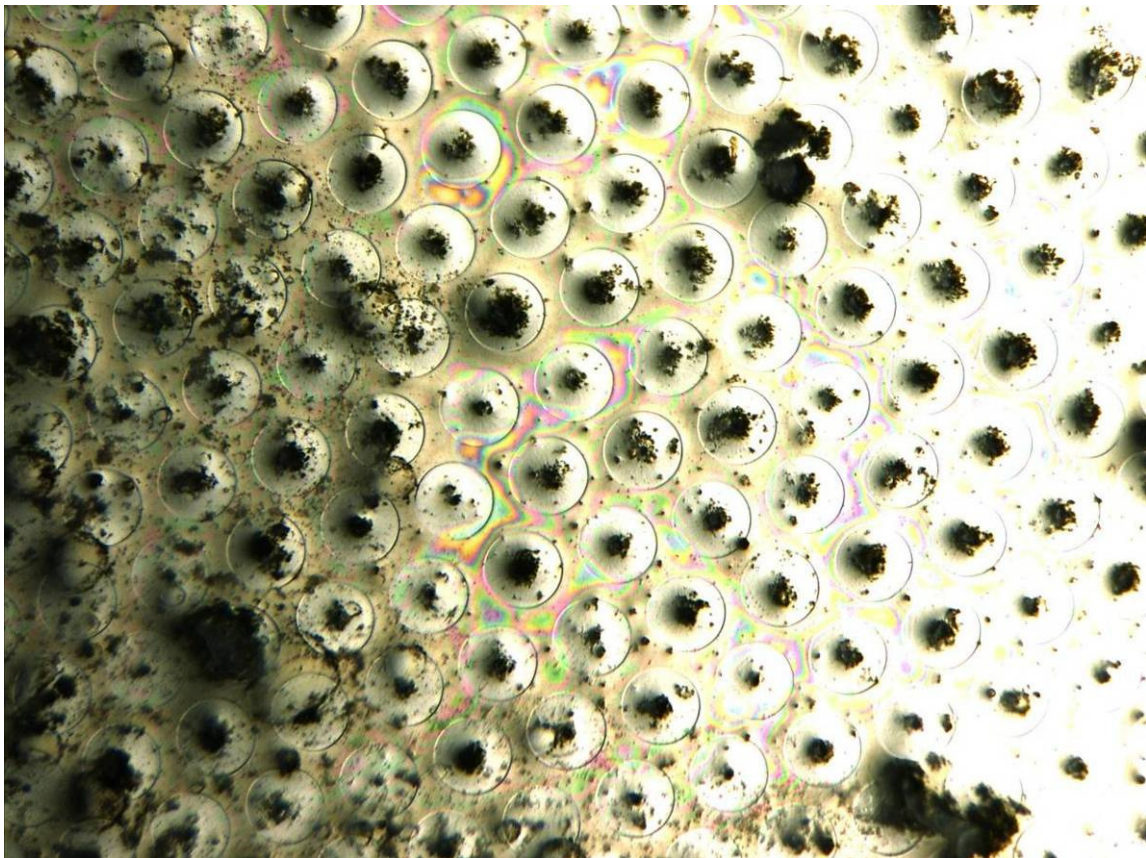


**Fig. 20: Particle in oil**

The next two figures (figure 21 and 22) show a surface of a membrane that was used for an oil filtration for some time. These pictures help to determine the build-up of a filter cake and how fast pore blocking will occur. This delivers valuable information in determination of the lifetime of a filter module when this filter material is used.

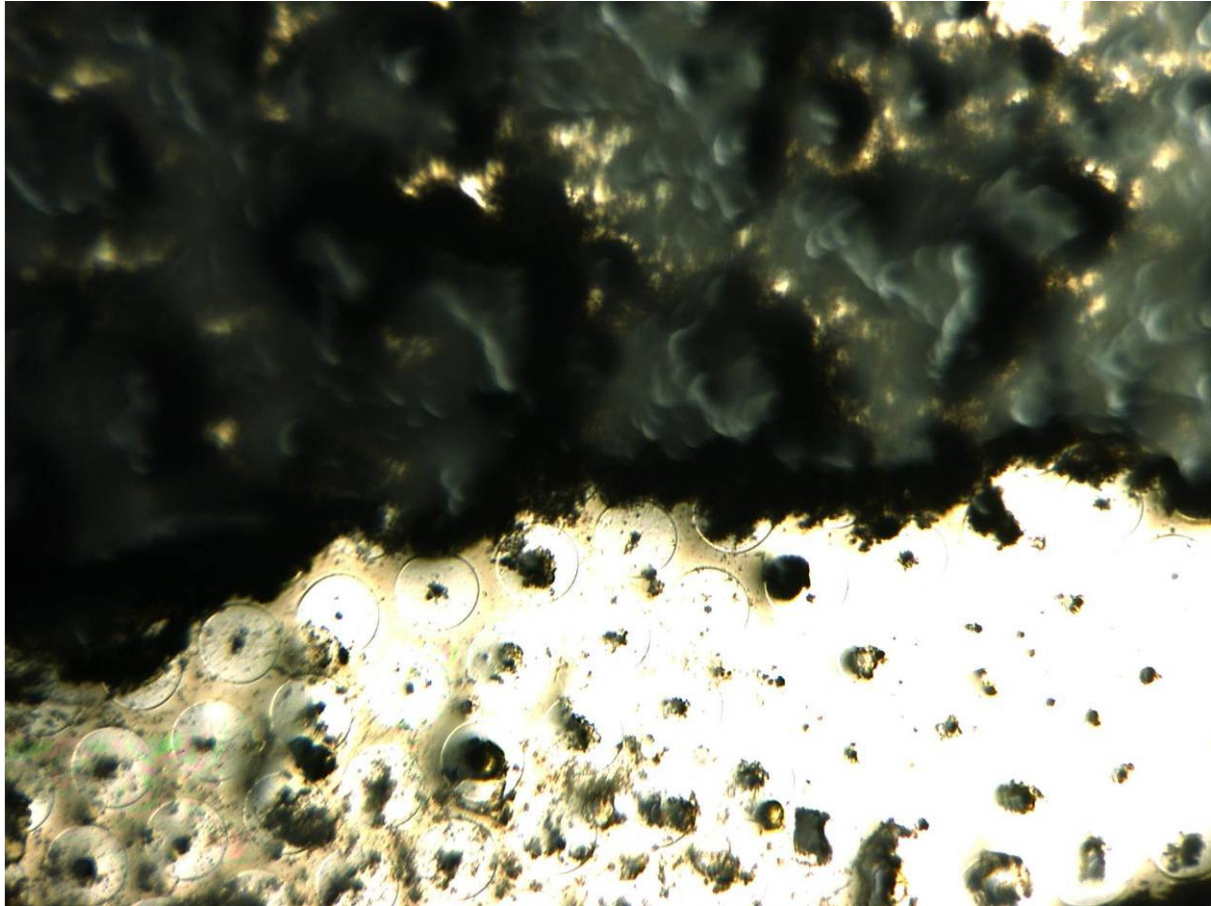


**Fig. 21: Build-up of a filter cake**



**Fig. 22: Build-up of a filter cake and analysis of pore blocking**

Figure 22 shows clearly what happens during the filtration process when using a sieve. The particles will block the pores first. The filtration test was done in dead end mode. This means that the lubricant is pressed through the membrane. This indicates that maybe a different filtration mode should be used for the planned filtration process. In figure 23 the results of a cross flow filtration are shown.



**Fig. 23: Results of a cross flow filtration**

Figure 23 shows the border of two different areas. The lower area was overflowed by oil. The upper area on the other side was not overflowed and thus prone to particle deposition. This leads to the result that a turbulent flow should be created over the membrane surface to enhance the lifetime of the filter. The dependency of the velocity of particles was also reviewed in Sobisch (2007). The advantage of cross flow filtration to prevent pore blockage becomes even clearer when the membrane is analysed by transmitted light (Figure 24). In this picture the lower part shows the area of the membrane that was not overflowed. The pores are blocked, because no light goes through the sieve.



**Fig. 24: Sieve analysed by transmitted light**

### **μSurf®:**

One further device used to analyse the surface of membranes or particles is the μSurf® (figure 21). The μSurf® is a confocal microscope that is able to make a non-contact 3D-measurement of complex surfaces. The physical filtering principle leads to a significant reduction of range in zoom compared to other measurement methods. The ability to measure in the x-, y- and z-axis makes this microscope special. The resolution in the x- and y-axis is 0,3 μm. The analysis is computer controlled in all three directions. The microscope can be used to determine the abrasion of materials in use or the change in material properties. Furthermore the measurement of shapes and surface roughness is possible. The advantage of this microscope for this project lies in the possibility to characterise contaminations and the loading or blocking of a filter.



**Fig. 25: Confocal microscope μSurf®**

**Assessment of membrane pore dimensions:**

Due to the fact that new phosphate esters (Reolube Turbofluid 46XC) were used, no analysis was done regarding particle size and particle deposition on membrane surfaces. The input of a turbine manufacturer or lubricant supplier with dirty or used lubricant was unavailable. Thus the parameters that have been investigated were the flow rate performance, the water removal and the changing of the acid content. Table 11 summarises the results of the accomplished flow rate tests.

**Table 11: Results of flow rate performance tests**

membrane	pore size [μm]	temperature [°C]	pressure bar]	permeate flow [l/h m <sup>2</sup> ]
nickel sieve	1,15	18	0,5	approx. 40
nickel sieve	1,15	18	1	approx. 80
PTFE*	0,1	18	2	approx. 45
PTFE*	0,1	18	2,5	approx. 80
PTFE*,**	0,2	20	2,5	approx. 160
PTFE*,**	0,2	2	2,5	approx. 14

\* A flow rate below 2bar could not be observed

\*\* Concentration of cellulose fibres was approx. 1 wgh.% referred to the total amount of filtered PE's

In the above table it is recognisable that the flow rate of the nickel sieve will increase proportional to the pressure that is applied. Thus, a doubling of the pressure doubles the permeate flow. A flow rate below 2 bar was not observed with PTFE-membranes. In comparison to the nickel sieve the flow rate is not proportional to the applied pressure. An increase of 0,5 bar up to 2,5 bar is nearly doubling the flow rate. It was observed that the pore size of the membrane is proportional to the flow rate.

Temperature appears to influence the flow rate more significantly than other parameters. Cooling down of the fluid from 20°C to 2°C decreased the permeate flow by more than 10 times.

Nickel sieves have regular channels and use PTFE-membranes that are made of a dense fibrous material. In respect of the water removal, no difference could be observed. (Note: all water in the PE's is dissolved). The cellulose powder on the other hand was able to lower the water content to approximately half of the initial value. These tests give some indications regarding the design process of the membrane technology in the project. It should be noted that the combination of a membrane and cellulose powder could give some advantages.

**Assessment of membrane configurations, e.g. hollow fiber, tubular, spiral, etc for water removal performance:**

According to the results presented, a different design of membrane configuration (channels in the metal sieve and dense fibrous material of the PTFE-membranes) was not able to remove water from the phosphate esters. A removal of water could only be detected when cellulose powder as an additional absorber was used.

**Assessment of influence of membrane surface chemistry/polarity/surface charge:**

According to the results presented, hydrophobic PTFE-membranes and non-hydrophobic nickel sieves were compared. A removal of water from the phosphate esters was not observed. The water content in all accomplished tests was for all membranes constant from the beginning of the filtration until the end when no additional absorber was used. It should be noted that the investigated PTFE-membranes have a water entry pressure around 2 bar. Because a flow rate below 2 bar was not observed, it is concluded that the PTFE-membranes are not appropriate membranes for the filtration of phosphate esters. The non-hydrophobic nickel sieve also has no potential to remove dissolved water.

**Introduction of localized chilling prior to membrane filtration to reduce moisture solubility. Assessment of the effect of turbulence and increase of oil flow in membranes by varying pump control to achieve the required level of moisture/gas removal:**

New Reolube Turbofluid 46XC was used for this set of testing. All tests were performed with the DFC and in batch process mode. A PTFE-membrane was placed into the cell with cellulose as an additional absorber. Table 12 will present the results.

**Table 12: Results of different filtrations in respect of water content**

adsorber*	temperature [°C]	stirrer	water content [l/h m <sup>2</sup> ]**	
1 wgh.% cellulose	20	no	1 <sup>st</sup> load 100,9 ± 12,0	2 <sup>nd</sup> load 229,2 ± 7,2
0,8 wgh.% cellulose	20	yes	214,3 ± 21,3	
1 wgh.% cellulose	2	no	264,4 ± 0,8	

\* Filtration without additional adsorber does not changed the water content

\*\* Initial water content was about 410 ppm

\*\*\* All tests were performed with a PTFE-membrane of a pore size of 0,2 µm

A volume of approximately 400 ml is filled into the test cell. After applying pressure, a sample of approximately 100 ml was taken. This is the 1<sup>st</sup> load. 2<sup>nd</sup> load means that a second sample from the same volume was taken. It is obvious that the water content is much higher in the

2<sup>nd</sup> load, but still lower than the initial water content (410 ppm). This could be explained due to the fact that no stirrer was used to cause turbulence and thereby a homogenization of the sample volume together with the cellulose. The 1<sup>st</sup> load had a much longer contact with the cellulose because of the time that is needed to assemble the test cell after filling with lubricant. The 2<sup>nd</sup> load instead could be taken right after the 1<sup>st</sup> load without any additional waiting time. This theory is validated with the second test in table 2. There a stirrer was used to homogenise the fluid and the absorber. It can be seen that the water content isn't that low compared to the 1<sup>st</sup> load of the previous test.

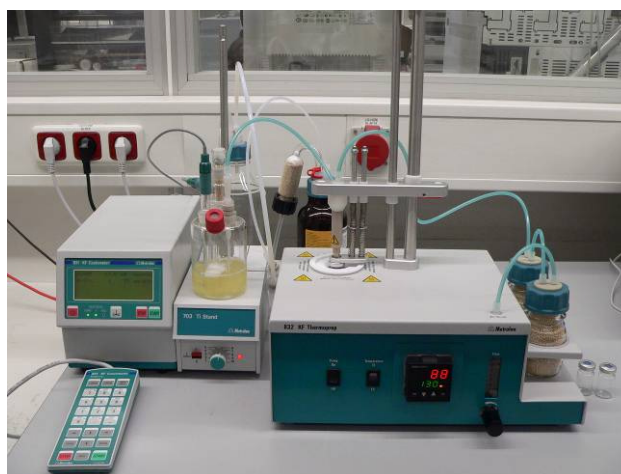
Localized chilling was applied by placing the DFC into an ice bath. This construction was standing for some time to give the phosphate esters enough time to cool down. The contact time between the fluid and the cellulose was much longer compared to the test at 20 °C. By analysing the water content of the fluid after the filtration it was found that the water content could be decreased down to 264 ppm.

Finally several things can be drawn as a conclusion from these tests. Increased turbulence helps to homogenise the fluid sample. To do a filtration without increased turbulence a much higher content of absorber would be necessary. Cooling does not seem to reduce the moisture solubility in phosphate esters. This stands in contrast to known results [CJC 2008]. But the tests at ambient temperature are more promising.

#### **Measurement of dissolved water PE's before and after membrane filtration to quantify level of removal:**

A coulometric Karl-Fischer device was used (figure 26) to analyse the water content of phosphate esters before and after membrane filtration to quantify the level of removal. Two accredited methods exist to measure the water content with a Karl-Fischer device [Scholz 2006]. The first is the direct method, where a sample of fluid is injected directly into the test chamber by a syringe. The injected fluid mass is determined by differentiation measurement of the syringe.

The indirect method is working with an oven. The fluid sample is filled into a vial and sealed with a septum. The vial is placed in the oven and pierced by a needle that directs compressed dry air into the vial. The water in the fluid is extracted and pressed through another whole in the needle into the reaction chamber. In the reaction chamber the water vapour reacts with a special solution. During the reaction an electrical current is needed to neutralise the reaction by-products of the water and the special solution. With the total needed electrical current the water content is calculated.



**Fig. 26: Coulometric Karl-Fischer device**

## **Conclusion**

According to the results presented, none of the membrane filtration systems alone were able to remove water from the phosphate esters. Even hydrophobic PTFE-membranes could not remove water from PE's. The removal of water could only be detected when cellulose powder as an additional absorber was used. Thus it is deduced from these tests that probably simple membrane technology is not suitable to remove water from PE's, because there is no water separation when the water is dissolved in the fluid. Furthermore the dissolved state is the most common appearance in fire retardant lubricant fluids. The use of adsorption technology could be an efficient possibility to reduce the water content.

Increased turbulence helps to homogenise the fluid sample. To do a filtration without increased turbulence a much higher content of absorber (cellulose powder) would be necessary. Turbulence also helps to prevent the membranes, especially metallic membranes, from pore blockage. Back pulsing in combination with metallic membranes would provide the possibility to concentrate particles in the retentate before it has to be removed. This could be efficiently realised with the TRF for a later particle filtration application for PE's.

Cooling the phosphate esters prior to membrane filtering them (no absorbers) reduced the permeate flow rate by a factor of 10 due to the higher viscosity of the fluid. Cooling does not seem to reduce the moisture solubility in phosphate esters. This stands in contrast to known results [CJC 2008].

The tests at ambient temperature are promising. Following a further series of tests with cellulose powder acting as an absorber during filtration water loading was reduced by approximately 50% from 410ppm to between 100 and 240 ppm. Thus it is suggested to treat PE's at the current fluid temperature. Special adsorbers, for example zeolites, could still guarantee to remove water from PE's while the filtration unit could work without additional coolers. This would reduce the overall investment costs and the operating costs due to the lower energy consumption.

Table 13 provides a summary of permeate flow rates found with different membranes when using of Reolube Turbofluid 46XC.

Membrane	Pore Size [µm]	Temperature [°C]	Pressure [bar]	Permeate Flow [l/hm <sup>2</sup> ]
Metal sieve	1.15	18	0.5	approx. 40
Metal sieve	1.15	18	1	approx. 80
PTFE*	0.1	18	2	approx. 45
PTFE*	0.1	18	2.5	approx. 80
PTFE*,**	0.2	20	2.5	approx. 160
PTFE*,**	0.2	2	2.5	approx. 14

\* A flow rate below 2 bar could not be observed

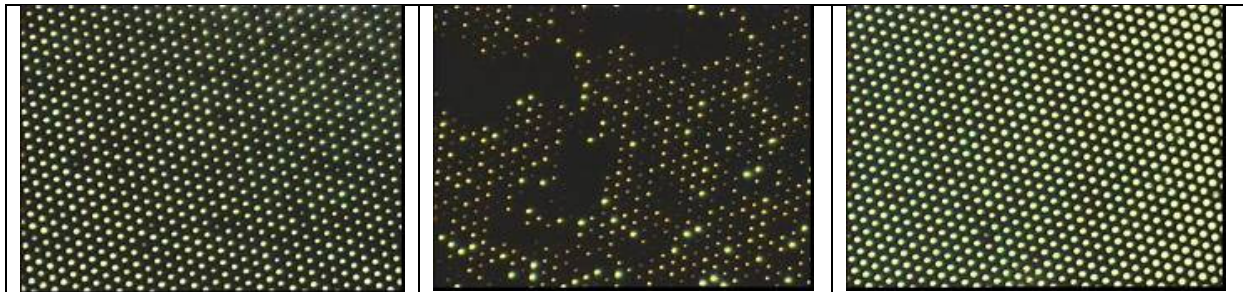
\*\* Concentration of fibrous material was approx. 1 wgh.%

**Table 13: Results of flow rate of Reolube Turbofluid 46XC using various membranes**

### **Deliverable D10: Membrane regeneration design technology.**

The aim of this deliverable was a prototype membrane technology to predict an in-situ automated membrane regeneration system. At month 16, when the deliverable was due the consortium did not have a turbine supply partner in it and therefore was not able to specify which lubricant, and hence which membrane, the prototype should use. This partner was not found by the termination date of the project and therefore this deliverable has not been completed. The following was determined.

The regeneration of filter membranes by back pulsing is analysed by optical microscopy in Figure 27 below. For a new membrane (left) all pores are open. After filtering contaminated oil some pores become blocked, leading to decreasing permeate flow until no further filtration is possible (centre). Fluid was then back-flushed through the membrane from the permeate side to remove the particles from the pores and the original filter quality was restored (right). This shows how effective back pulsing can be to enhance the lifetime of a filter for oil applications.



**Figure 27: New membrane (left), contaminated membrane (centre), regenerated membrane (right)**

### **Deliverable D11: Identification of suitable ion-exchange technology for removal of TAN in PE's**

#### **Ion exchange for TAN removal**

An intensive study was undertaken and some potential ion-exchange resins for the removal on TAN from phosphate esters and maybe turbine oils have been identified.

- Rohm & Haas (<http://www.rohmmaas.com>) have a potential resin called Amberlite that could be applicable for acid removal from turbine lubricants.
- Purolite (<http://www.purolite.com>) also provide an applicable ion exchange resin called A100-dry. This is a weakly basic resin that can be used to remove acids from lubricants.
- Serva Electrophoresis GmbH (<http://www.serva.de/>) does not provide ion exchange resin for the acid removal, but does sell an adsorber for that application. Due to the high costs of this adsorber (approx. 175€ for 100g) it was decided that it is not applicable in the project.

In addition to the task in the project, there were also some additional investigations conducted with respect to acid removal by membrane filtration with and without an adsorber. The hope was to develop one single unit for both the acid and water removal. This would provide the possibility to develop a more cost-efficient conditioning unit. Results of these tests are summarized in Table 14. No significant change of the TAN was observed, even with the use of a fibrous material as an adsorber.

Tests regarding TAN removal with membranes under different conditions	Before	After
PE-01-Metal-1.15µm-01	0.06	0.06
PE-01-PTFE-0.1µm-01	0.06	0.06
PE-01-PTFE-0.2µm-fibrous adsorber-01	0.06	1st sample 0.06    2nd sample 0.07
PE-01-PTFE-0,2µm-fibrous adsorber-02	0.05	0.06
PE-01-PTFE-0,2µm-fibrous adsorber-Cooled (2°C)-03	0.05	0.06

**Table 14: TAN removal test using membranes**

### Identification of strategic location of the ion exchange resins in turbine systems

In respect to statements in the literature<sup>78</sup> ion exchange resins seem to have diminished activity under dry conditions. Therefore it is suggested to place the ion exchange treatment between the particle removal and the water removal unit in the conditioning system. The particle removal unit will prevent the ion exchange resins from contamination. The water removal unit after the ion exchange treatment ensures good activity of the resins due to wet fluid and this assists the prevention of contamination of a possible absorber in the water removal unit.

### Applicable adsorbers for TAN removal

After consultation of the literature<sup>9</sup> and discussions within the consortium an alternative to ion exchange resins was envisaged if the project scope is increased to cover a number of lubricant types other than phosphate esters.

Ion exchange resins require a certain polarity of the surrounding medium to work sufficiently and therefore may not perform as required if mineral oil based formulations form the focus of research instead of PE-types. Additional input from a turbine manufacturer is needed to focus this work. Furthermore, ion exchange resins can add water to the system that also has to be removed. Because of this uncertainty Fraunhofer UMSICHT worked on alternative possibilities to reduce the acid content without increasing the water content at the same time.

Another promising possibility to reduce the acid content in a fluid – phosphate ester or turbine oil – is titanium dioxide. Fraunhofer UMSICHT made several tests with different absorbers that are potentially able to reduce the acid content of a fluid. The tests were performed using acidic oil with a fibrous material, Fuller’s Earth and titanium dioxide. The fibrous material showed no influence on the TAN of the oil. Fuller’s Earth on the other hand was able to reduce the TAN, but the reduction was not as high as hoped for. Furthermore Fuller’s Earth is known as a material that will cause additional problems in the lubrication system, as already discussed. Titanium dioxide showed promise, displaying increased

<sup>7</sup> Phillips, W.D.; The high-temperature degradation of hydraulic oils and fluids; Journal of synthetic lubrication, 23, pp. 39-70, 2006

<sup>8</sup> Duchowski, J.K.; Collins, K.G.; Ion exchange/vacuum dehydration treatment: an improved approach for conditioning and reclamation of phosphate ester hydraulic fluids; Journal of the society of tribologists and lubrication engineers, pp. 29-35, 2000

<sup>9</sup> Troyer, D.; Wurzbach, R.; „Will ion-exchange resins remove acid from mineral oils?“, Machinery Lubrication Magazine, Issue Number 200211, 2002

effectiveness compared to Fuller's earth. Titanium dioxide is widely available, relatively low in price, non-hazardous and chemically very inert. This helps to regulate the acid removal process of the lubricants without unwanted side reactions. In principle, titanium dioxide is nearly insoluble. It also has a melting point higher than 1800°C. There are small variations depending on its crystalline structure but nevertheless it is applicable to work in the high temperature area of turbines.

The most common crystalline structures of titanium dioxide are the Anatas and the Rutil structures. Fraunhofer UMSICHT is currently analysing the advantages of these structures regarding the effectiveness of acid removal. Furthermore Fraunhofer UMSICHT is working to build pellets of titanium dioxide. This will help to handle this material in a very simple manner. This also provides the advantage that no fine or small particles will go into the fluid.



**Fig. 28: TiO<sub>2</sub> in Rutil structure**



**Fig. 29: TiO<sub>2</sub> in Anatas structure**



**Fig. 30: TiO<sub>2</sub> in Pellet structure**

## Obtain up-to 6 samples of ion exchange resins for laboratory trials

The identified samples were obtained, these are shown in Figures 31-35. Specific details for each of the samples are available in Appendix 1 of Deliverable 11.



**Fig. 31: Amberlyst™ A21 from Rohm & Haas**



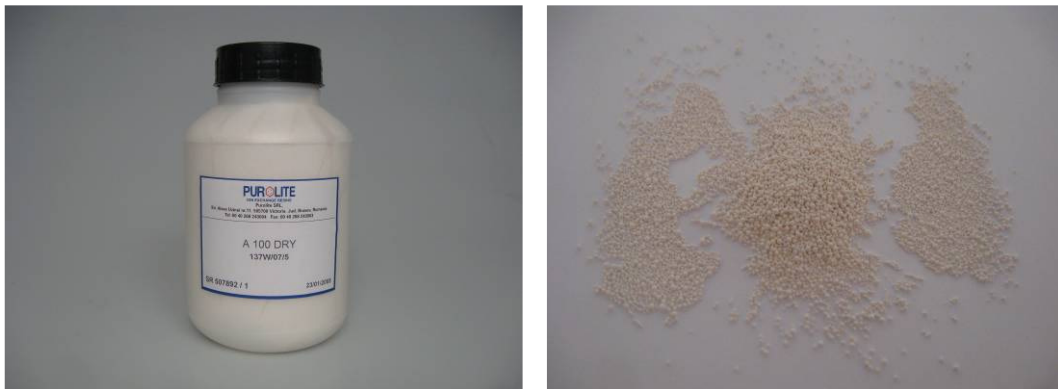
**Fig. 32: Amberlyst™ A24 from Rohm & Haas**



**Fig. 33: CTA 190 from Purolite Deutschland GmbH**



**Fig. 34: A103 S from Purolite Deutschland GmbH**



**Fig. 35: A100 DRY from Purolite Deutschland GmbH**

### Conclusion:

No significant change of the TAN was observed, even with the use of a fibrous material as an absorber in any of the membrane filtration tests. The hope was to develop one single unit for both the acid and water removal, i.e. membrane filtration, but this was not possible so other systems were investigated.

Tests were performed using acidic oil with a fibrous material, Fuller's Earth and titanium dioxide. The fibrous material showed no influence on the TAN of the oil. Fuller's Earth on the other hand was able to reduce the TAN, but the reduction was not as high as hoped for. Titanium dioxide showed promise, displaying increased effectiveness compared to Fuller's earth. Titanium dioxide is widely available, relatively low in price, non-hazardous and chemically very inert. In principle, titanium dioxide is nearly insoluble. It also has a melting point higher than 1800°C and therefore it is applicable to work in the high temperature area of turbines.

Fraunhofer were in the process of developing alternative forms of Titanium Dioxide when the project was terminated.

5 samples of ion exchange resins for laboratory trials had been obtained.

## **WORK PACKAGE 4: WATER AND TAN MANAGEMENT AND COMMUNICATIONS PROTOCOL**

Objectives: To develop an intelligent moisture and TAN management process for PE's in turbine systems. This will encompass an embedded control system comprising a micro-controller, firmware and interface electronics to manage the PE drying and TAN scavenging process. If the water or TAN contents are within acceptable limits (as defined in WP1), then no action will be taken, if they are outside the limits then water/TAN removal steps will be implemented (as defined in WP3). To develop a fibre optic communications and scada technology to report PE condition to a central software database e.g. at the generator's operations centre, where the recorded data will be extrapolated to provide intelligence on the life expectancy of the PE. To alert the data controller using the database, where moisture/TAN levels in the PE are exceeding defined limits – i.e. warning system.

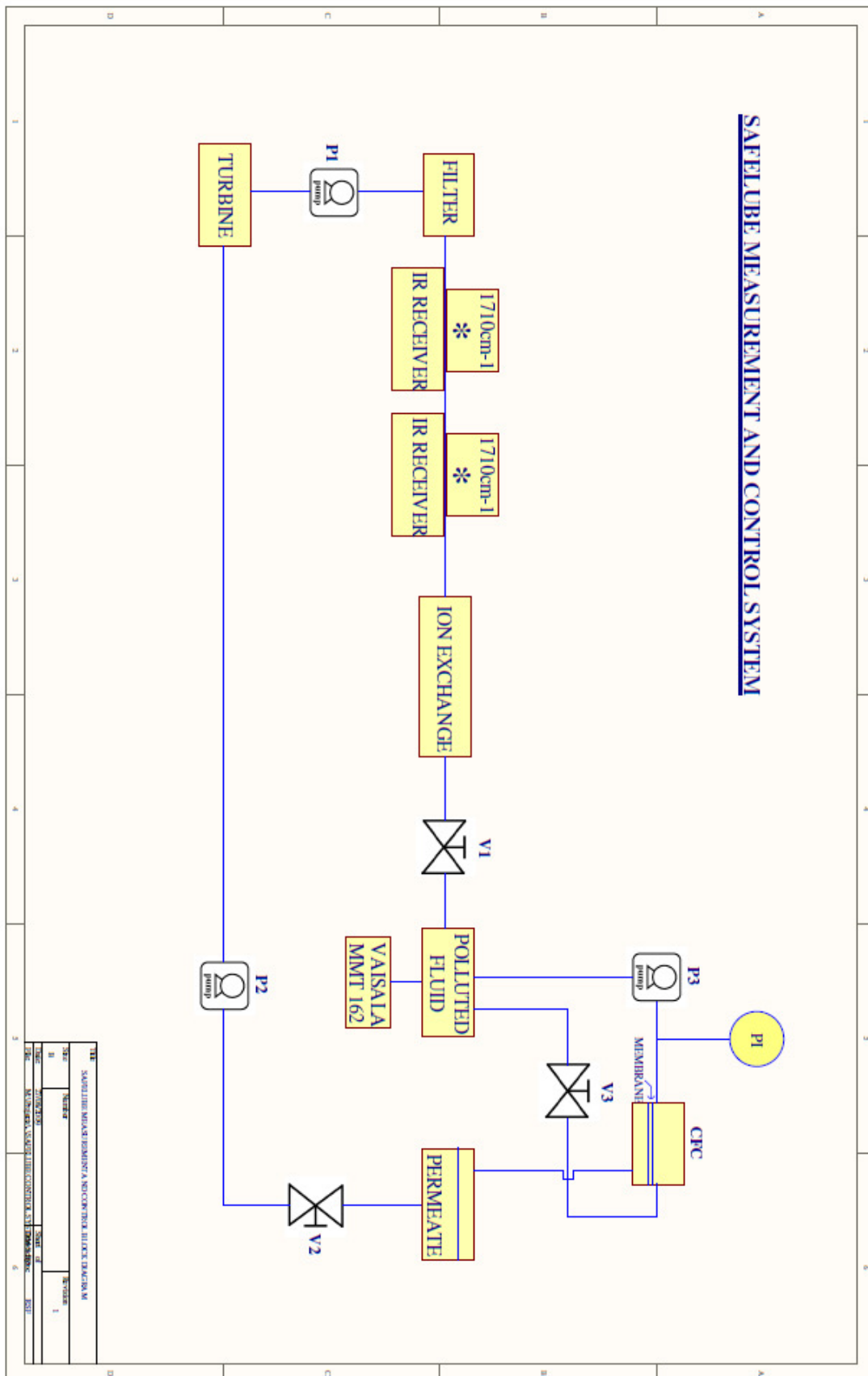
Deliverables D12, D13, D14, and D15 have been produced to meet these objectives.

### **Deliverable D12: Schematic electrical system design for management and communications protocol**

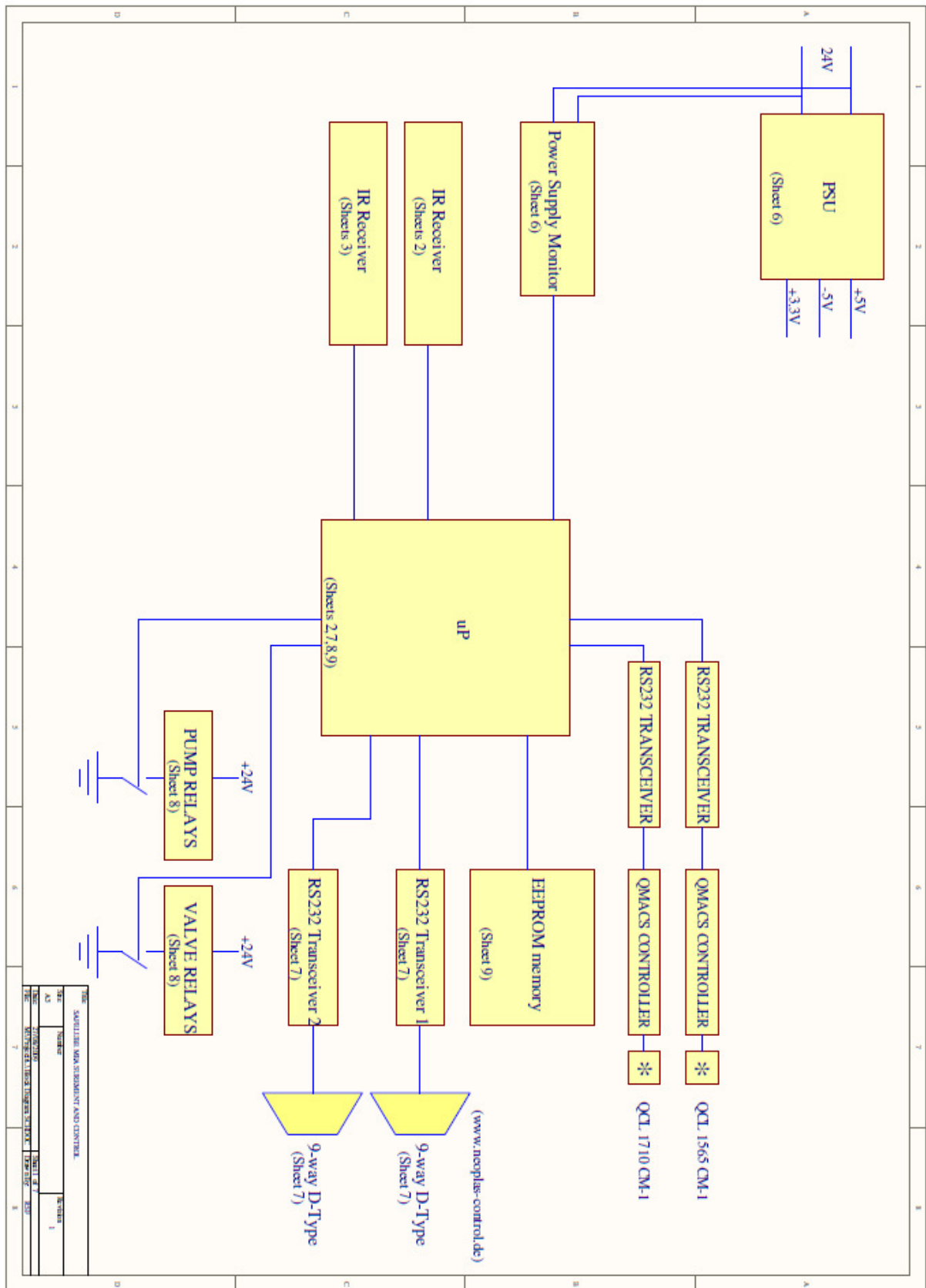
#### **1.0 Initial System Design**

The first step to developing a management and communications protocol was to produce an initial system design in the form of a schematic diagram. The following pages display the results of an initial system design for the SAFELUBE electronic control system.

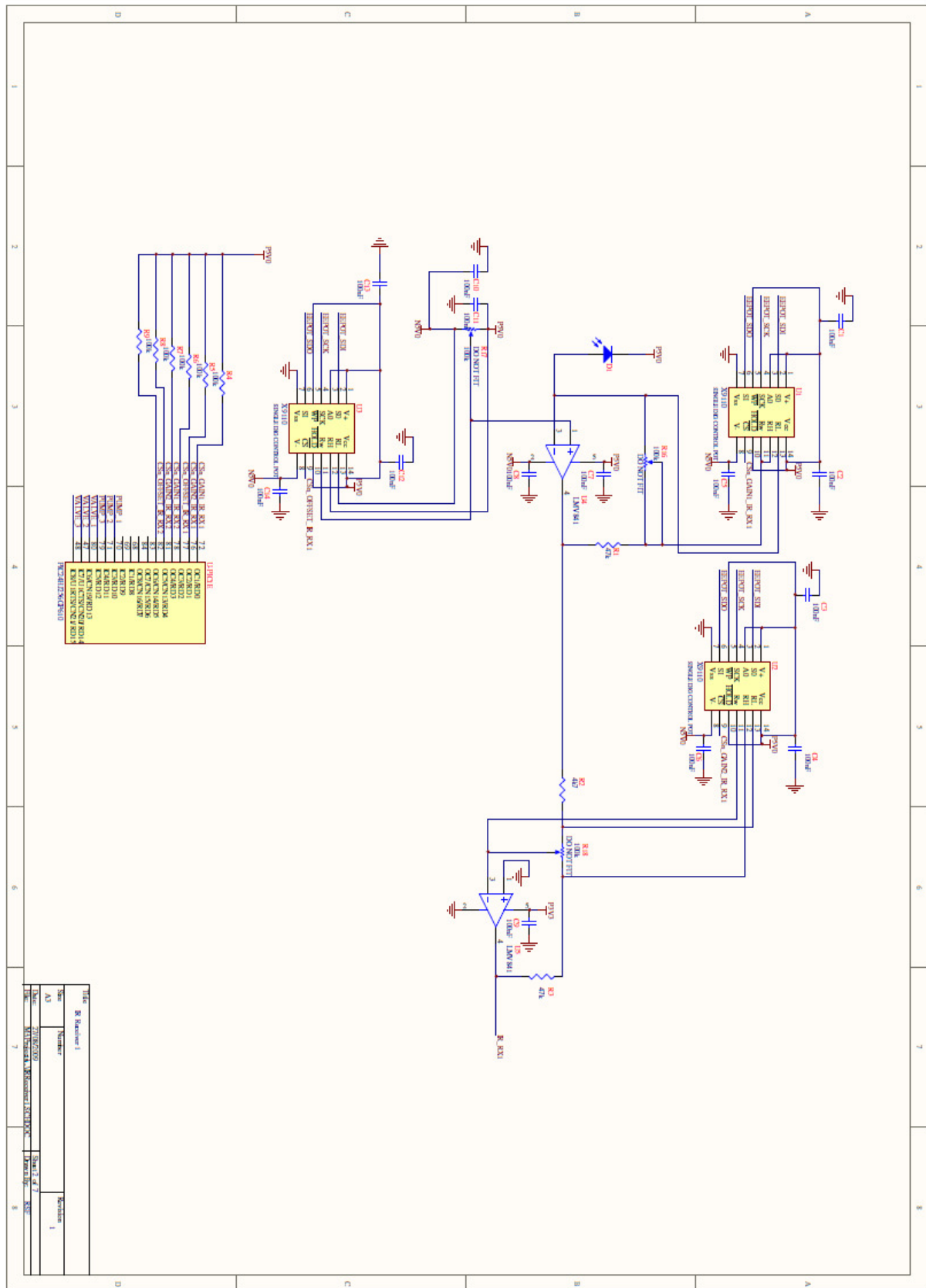
## 2.0 Schematic Overview of System



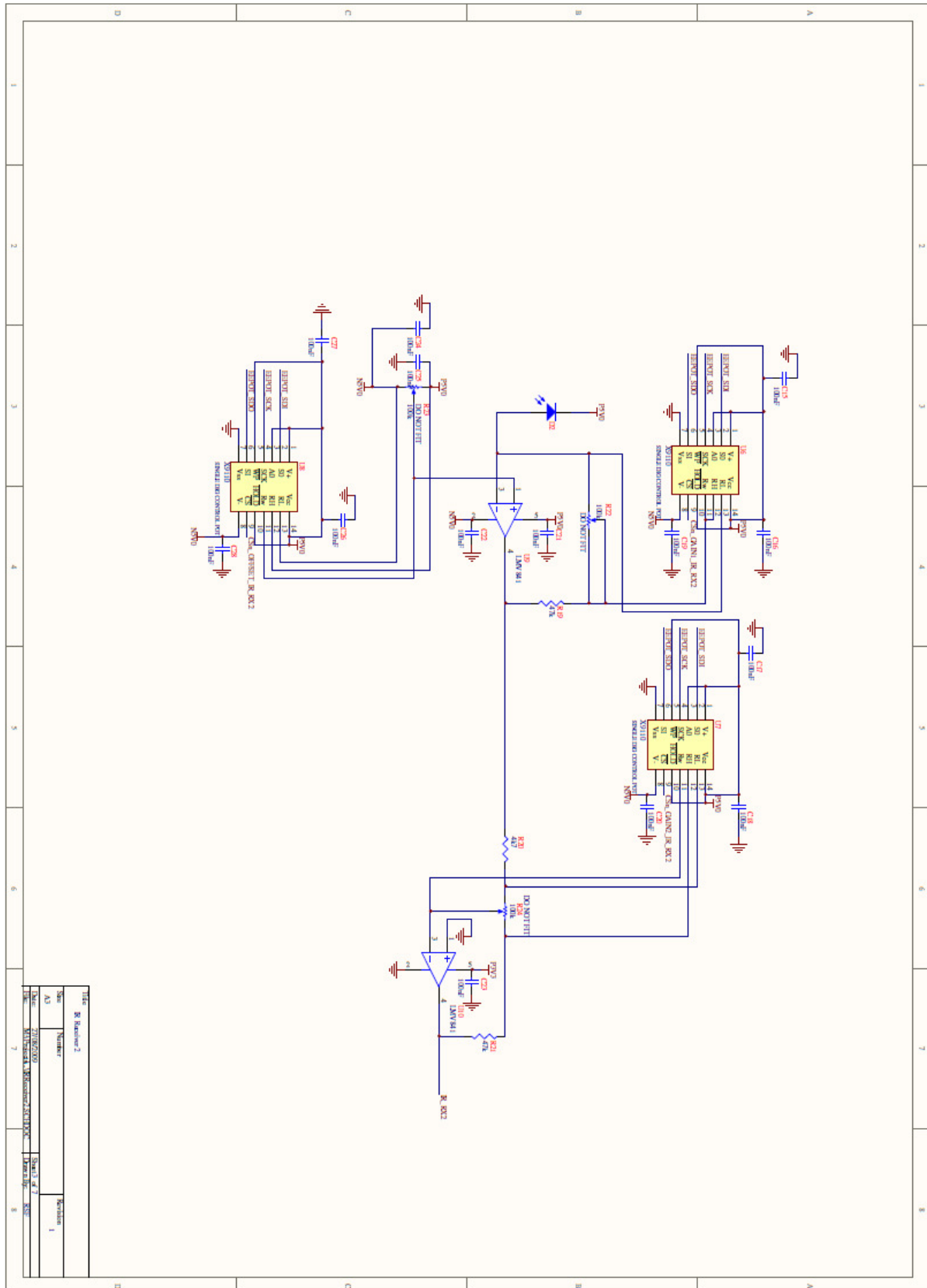
### 3.0 System Block Diagram



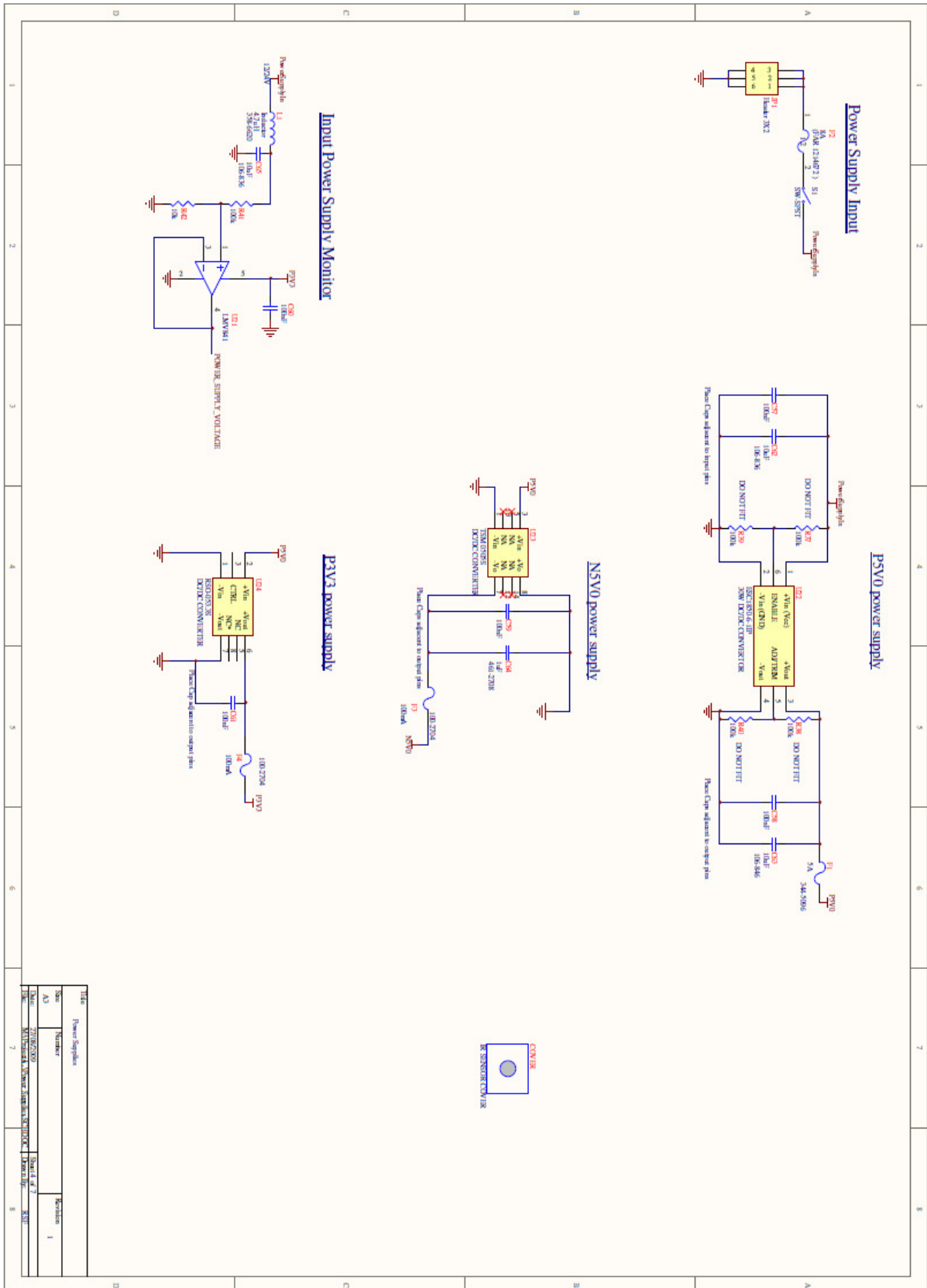
# 4.0 Infra-Red Receiver 1



## 5.0 Infra-Red Receiver 2



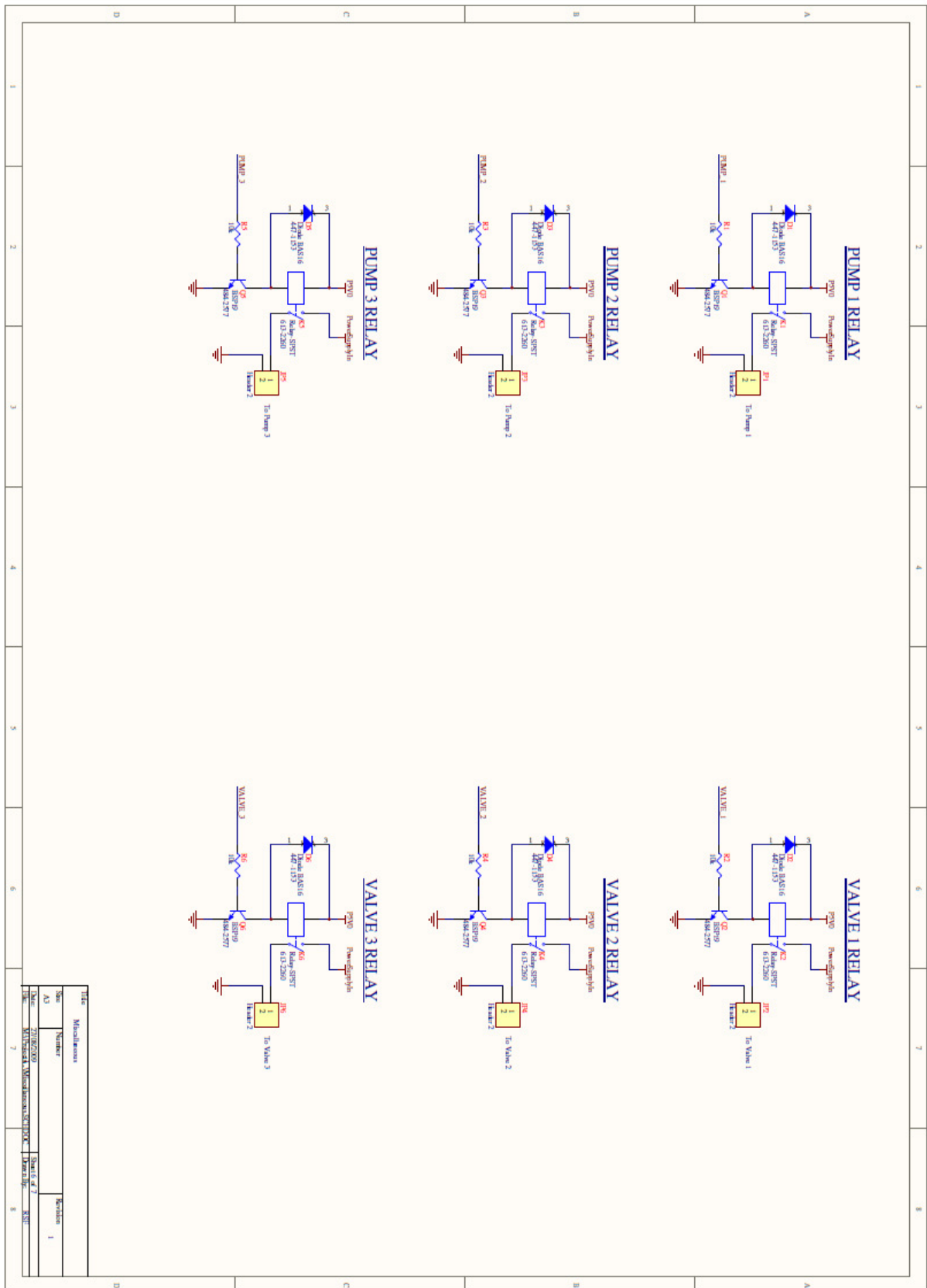
# 6.0 System Power Supplies



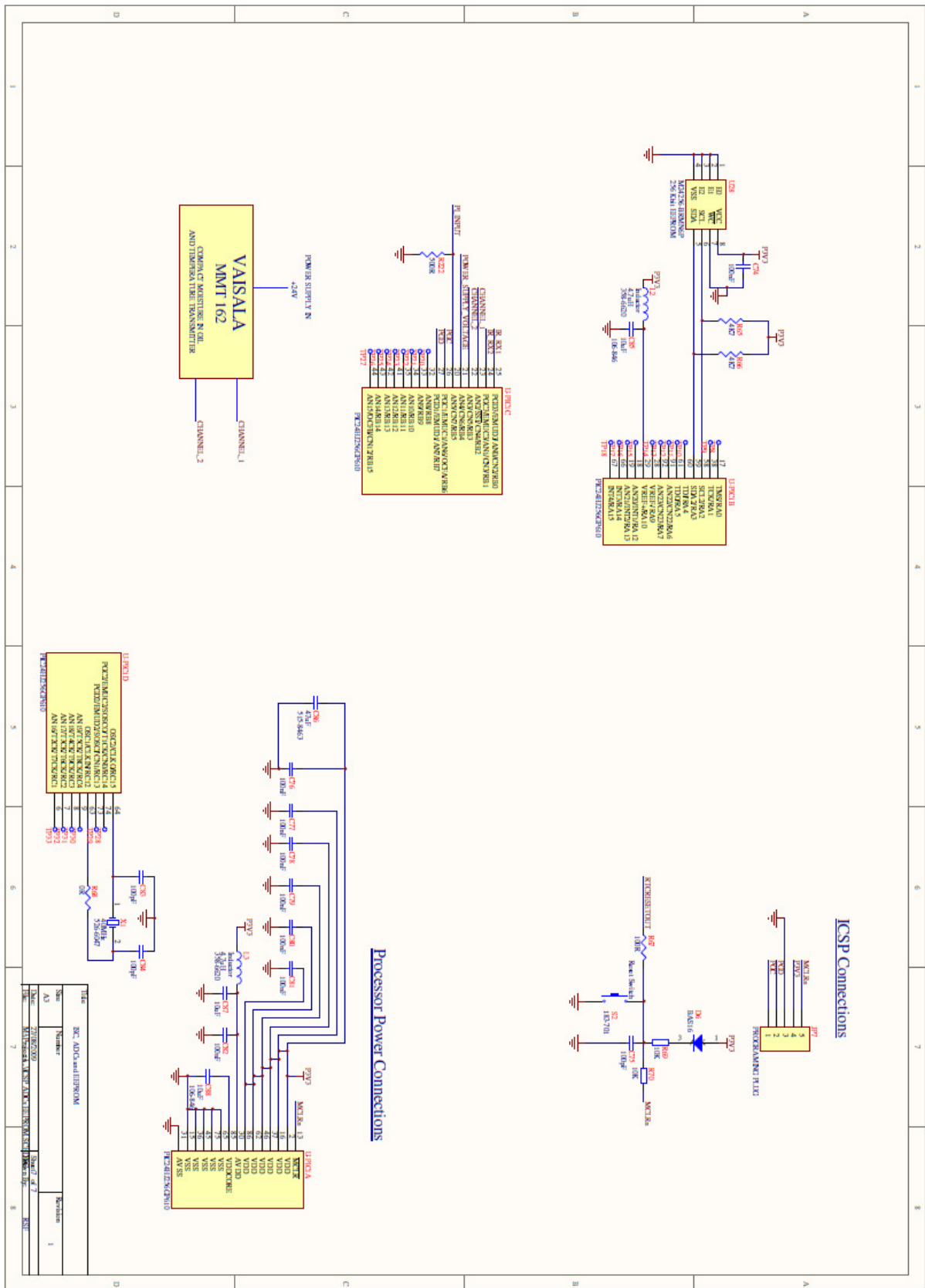
Title		Revision	
Power Supplies	Number	1	
Size	Number	1	
Rev	Number	1	
Date	2/11/2009	Sheet	1 of 7
Name		Author	
M. J. ...		M. J. ...	
Title		Project	
Power Supplies		...	



## 8.0 Pump and Valve Control



# 9.0 ISC, ADC's and EEPROM



## 10.0 Conclusion

This deliverable was to design the schematic electrical system for the management and communications protocol of the cleaning system. This has been done, and the deliverable is complete.

### **Deliverable D13: Master and slave electronics.**

The aim of this deliverable was master and slave electronics. At month 16, when the deliverable was due the consortium did not have a turbine supply partner in it and therefore was not able to make the full prototype specifications for master and slave electronics to be developed. This partner was not found by the termination date of the project and therefore this deliverable has not been completed.

### **Deliverable D14: Central unit control design.**

The aim of this deliverable was a central unit control design. At month 16, when the deliverable was due the consortium did not have a turbine supply partner in it and therefore was not able to make the full prototype specifications for a central unit control system to be developed. This partner was not found by the termination date of the project and therefore this deliverable has not been completed.

### **Deliverable D15: Communications protocol system to enable PE monitoring and management and life forecasting of the lubricant.**

The aim of this deliverable was a Communications protocol system to enable PE monitoring and management and life forecasting of the lubricant. At month 16, when the deliverable was due, the consortium did not have a turbine supply partner in it and therefore was not able to specify the lubricant, and hence what communications protocol the prototype should use. This partner was not found by the termination date of the project and therefore this deliverable has not been completed.

## **WORK PACKAGE 5: PROTOTYPE INTEGRATION AND VALIDATION**

Objectives: To design and construct a fully functional, PE monitoring and conditioning system for simple retrofitting into a selected case study turbine. To integrate the prototype system with the intelligent water and TAN management and communications protocol (delivered in WP 4). To conduct a series of tests to ensure the functionality of the water and TAN management system and communications protocol. To generate “benchmark” validation test data from the case study demonstrator. To review the data, and explore opportunities of optimising performance as necessary.

This work package is associated with deliverables D16 and D17.

As the project terminated in M17 and the deliverables for this work package were due by M22, the associated deliverables have not been completed.

## **WORK PACKAGE 6: INNOVATION RELATED ACTIVITIES**

Objectives: To ensure that all the project results are formulated and compiled into a form that can be protected and all necessary patents are made. To transfer specific knowledge from the RTD performers to the SME participants to enable them to rapidly apply and embed the technology to manufacture and exploit the developed insulation monitoring and conditioning system. To broadcast the benefits of the developed lubrication monitoring and conditioning system and knowledge beyond the consortium to the end-user communities.

Deliverable D18 has been produced to meet these objectives.

Deliverables D19 and 20 were due to be delivered by M24, as the project terminated in M17 these deliverables have not been completed

### **Deliverable D18: Project Presentation.**

#### **The Consortium:**

The project partners, lead by the coordinator Kelman, are spread throughout six EC member states. Kelman Ltd and Houghton PLC are from the UK; Safibra S.R.O. are from the Czech Republic; Macq Electronique are from Belgium; Minerwa Umwelttechnik GmbH are based in Austria and Alpes Lasers in Switzerland. RTD partners Pera Innovation and Fraunhofer-UMSICHT are based in the UK and Germany, respectively.

#### **Project Objectives:**

The SAFELUBE project aims to develop new systems and technologies that will promote the up-take of fire-retardant lubricant technology in turbines and therefore to help significantly reduce turbine fires. This will be achieved by developing systems to enable the more efficient management of fire-retardant lubricants. By increasing the working lifetime these lubricants, the SAFELUBE project will also help to significantly reduce the quantity of acidic and mineral oil from the power generation sector.



**Project Website:**

A SAFELUBE project website has been set-up (<http://Safelube.pera.com>), and is being accessed regularly by the partners. Project progress, meeting minutes, travel arrangements, future meeting dates, partner reports and presentations are among the items on the website. A public-facing domain will soon be established for dissemination purposes.

**Project Presentation:**

A SAFELUBE project presentation has been developed for use by the partners when speaking about the project or indeed promoting it. The slides for this presentation are displayed over the following pages.



## A Novel Method of Continuous On-line Conditioning & Safe Management to Enable the use of Fire Retardant Lubricants in Gas & Steam Power Generation Turbines

EC COOPERATIVE RESEARCH PROJECT  
COOP-CT-2005-017970



## Steam Turbine Fire Hazards

- Steam temperatures rising in search for greater efficiency-currently 550 to 600°C
- Hydraulic system pressures have risen from 40-160 bar.
- Use of mineral oil in hydraulic and lube oil system poses significant fire risk
- Factory Mutual Insurance loss data indicates one significant fire every 40 operating years for steam turbines.
- Allianz Insurance indicate that origin of turbine fires split almost equally between hydraulic and lube oil system
- Cost of losses can run into millions of dollars



# Steam Turbine Fire Hazards

Fire hazard is currently increasing because:

- Operating temperatures are still increasing (7000°C in latest reference unit in Germany)
- Reduced manning
  - fewer operators 'walking the lines'
  - more outsourcing of maintenance
  - fewer experienced personnel
- Wider use of remote operation (small turbines)
- Increasing age of equipment (>30% is more than 30 years old) and trend to life-extension/refurbishment
- Longer times between overhauls



## EC Framework 6

- €15 billion available under Framework 6 to fund the development of Innovative new technology...
- To help European Companies (especially SMEs) survive and prosper in a globalised market, through innovation
- To provide the scientific excellence and technological resources to help European businesses to turn their ideas and aspirations into reality
- To help improve trans-national collaboration throughout Europe
- To improve European Quality of Life



## To Improve Safe Operation

Either:

- Use a fire resistant hydraulic fluid - with no mechanical fire protection - or
- Use mineral oil with mechanical fire protection (sprinklers, pipe-in-pipe hydraulics etc.)

Factory Mutual have shown in full scale tests that sprinklers will not extinguish a turbine oil fire-only control it. Only way to extinguish it is to shut down the turbine and risk bearing damage



## Use of Phosphate Ester Fire- Resistant Hydraulic Fluids

- Now over 50 years use globally
- Specified by most of the turbine builders
- Very effective in avoiding turbine fires – but:
  - Expensive
  - Sensitive to moisture - form acids which further catalyse fluid degradation
  - Need in-situ conditioning to extend life. This means taking regular samples and checking a minimum of acidity, water, particulates and resistivity



# Use of Phosphate Ester Turbine Lubricants

- Even with fire-resistant hydraulic fluids, turbine oil fires still occur.
- Fire-resistant lubricants are being used in some applications e.g. in Russian turbines up to 1000MW
- Turbine builders are, however, reluctant to move to a more expensive fluid.
- VGB (European utility assocn.) supports their use and wants a reference unit in Germany



**PERA**  
THE INNOVATION NETWORK

## Other Benefits of PE Turbine Lubricants

- Improved operator safety
- Compliance with EU Machinery Safety Directive
- Increased security of energy supply
  - no excess capacity at utilities
  - reduced risk of terrorism
- Increased availability of equipment
- Reduced insurance premiums?
- Savings on payment of deductibles/cost of mechanical fire protection
- Favourable life-cycle costs possible



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# Technical Objectives

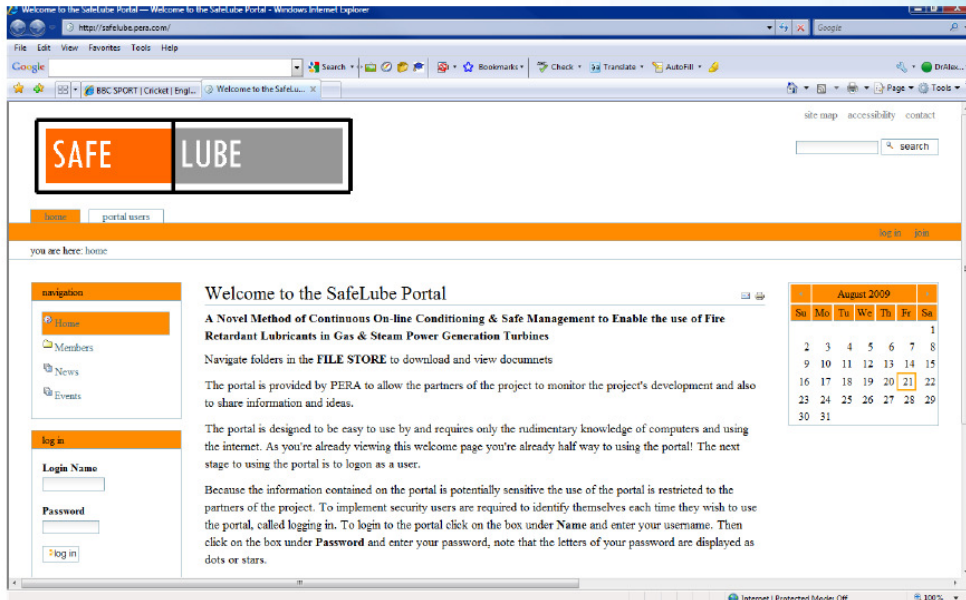
- Develop water sensor
  - **Detect presence of water between 0-1500 ppm**
- Develop laser diode TAN sensor to measure absorption peaks in the 4000-800  $\text{cm}^{-1}$  range.
  - **Detect level of TAN between 0-0.5 mg KOH/g**
- Development of a diagnostic system to assist users in PE condition monitoring
  - **Decision-making accuracy of at least 98% in controlled tests**
- Development of a regenerative dehydration system based on turbulent flow membrane filtration
  - **Capable of reducing water levels below 50 ppm**
- Robust fibre-optic transmission and scada communications architecture
  - **Transmission of 2 data streams with 100% data integrity within high EM field test environment**



## The SAFELUBE Partners



# Project Website



Thankyou for your attention!

<http://safelube.pera.com>



## **WORK PACKAGE 7: PROJECT MANAGEMENT**

**Objectives:** To manage, from a technical perspective, the project resource, timing, delegation, intra partner communications, coordination of cross partner activities within work packages and review/reporting of progress against gateways/milestones.

A Month 12 meeting did not take place due to the non-accession to the SAFELUBE contract of two key project partners providing knowledge of the lubricant industry (Great Lakes) and power generation turbines (Man Turbo). This proved to be a considerable stumbling block for the SAFELUBE consortium. On 10<sup>th</sup> March 2008, the consortium requested a six month suspension of the project in order that potential replacements be put in place.

During the period of suspension, Kelman, the project coordinator, was acquired by GE Energy during the first months of Period 2. Following the take over, Kelman's focus was aimed more specifically at transformer monitoring and so they decided to withdraw from the SAFELUBE project following the suspension on 10<sup>th</sup> March 2008. It is at this point that Pera Innovation Ltd, the lead RTD contractor, also became the project Coordinator.

### **Section 3. Consortium Management**

#### **WORK PACKAGE 8 : CONSORTIUM MANAGEMENT**

To co-ordinate all project activity and act as the administrative interface with the Commission. To manage time, resources and facilities allocation to optimise the application of resource and establish exploitation mechanisms.

There were no consortium meetings during Period 2 of the project.

In P2, there was a sustained effort to recruit replacement partners, however due to the nature and limitations of the turbine technology, this was not ultimately successful and the project was terminated in Month 17 by the European Commission.

#### **Deliverable D21; Draft Dissemination Plan**

A draft DUP plan has been submitted, this document forms Appendix 1 to this report.

#### **Deliverable D22; Final Dissemination report.**

Deliverables 22 was due to be delivered by M24, as the project terminated in M17 this deliverable have not been completed

## Project Progress Against Objectives, Deliverables & Milestones

Deliverable Number	Description	Nature	Dissemination Level	Delivery Date	Date Submitted to the EC
1	Relationship between water and TAN content and condition/state of PE's	R	CO	M6	
2	Computational model to predict the operational life of the lubrication and resin	O	CO	M6	N/A
3	Target specification for PE monitoring and conditioning system.	O	CO	M6	September 2009
4	Specification for laser diode design, to enable detection of water and TAN in infra-red band	R	CO	M6	September 2009
5	Prototype laser diode sensors showing 'proof of concept' functionality for water and TAN detection	P	CO	M10	September 2009
6	Sensors for measuring: (i) water content in the region of 0-1500ppm with a sensitivity response of 5%; (ii) TAN content in the range 0-0.5mg/KOH/g with a sensitivity response of 5%	P	RE	M16	September 2009
7	Matrix/interference correction for target components.	O	RE	M16	September 2009
8	Sensor shield	P	RE	M16	September 2009
9	Membrane technology capable of removing water levels to <10ppm.	P	RE	M10	September 2009
10	Membrane regeneration design technology	P	RE	M16	September 2009
11	Identification of suitable ion-exchange technology for removal of TAN in PE's	O	RE	M16	September 2009
12	Schematic electrical system design for management and communications protocol	O	CO	M10	September 2009
13	Master and slave electronics.	O	RE	M16	September 2009
14	Central unit control design	O	RE	M16	September 2009

15	Communications protocol system to enable PE monitoring and management and life forecasting of the lubricant.	O	RE	M16	September 2009
16	Turbine case study	O	PU	M22	September 2009
17	PE monitoring and conditioning system validation data	R	PU	M22	September 2009
18	Project Presentation	O	PU	M6	September 2009
19	4 case studies	O	PU	M24	September 2009
20	Exploitation strategy document	R	CO	M24	September 2009
21	Draft Dissemination and Use Plan	R	PU	M12	January 2008
22	Final Dissemination and Use Plan	R	PU	M24	September 2009

## Milestones Update Month 1 – Month 17

<b>Milestone No.</b>	<b>Milestone Title</b>	<b>Completion Date</b>	<b>Update</b>
M1	Completion of scientific studies to identify PE properties and capabilities of laser diodes	M6	15
M2	Completion of 'proof of concept' technical development, demonstrating feasibility of implementing scientific knowledge.	M10	18
M3	Completion of technical development of water and TAN sensors and management system.	M16	20
M4	Completion of integration and validation, resulting in a working prototype system	M22	23
M5	Completion of project, including all dissemination and exploitation planned for project duration	M24	24

## Project Progress against Work Programme

		WORK PROGRAMME																								
		Partners	MONTHS																							
			1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24
<b>WP1</b>	<b>Characterization of vital properties of PE's</b>	<b>6, 1, 8, 9</b>																								
	1.1 PE degradation chemistry																									
	1.2 Predictive model for PE's																									
	1.3 Performance specification																									
<b>WP2</b>	<b>Sensor development</b>	<b>2, 1, 6 8, 9</b>																								
	2.1 Identification of laser diode properties																									
	2.2 Laser diode development																									
	2.2 Matrix effects																									
	2.3 Sensor shielding																									
<b>WP3</b>	<b>Water and acid removal</b>	<b>4, 3, 6, 9</b>																								
	3.1 Membrane assessment																									
	3.2 Membrane regeneration																									
	3.3 Ion exchange selection																									
<b>WP4</b>	<b>Water &amp; acid manag. &amp; communications protocol</b>	<b>3, 1, 2, 4, 5, 6, 8, 9</b>																								
	4.1 Initial System design																									
	4.2 Iterative Design																									
	4.3 Slave mode electronics																									
	4.4 Central control unit																									
	4.5 Communication protocol																									
<b>WP5</b>	<b>Prototype integration &amp; validation</b>	<b>7, 1, 2, 3, 4, 5, 6, 8, 9</b>																								
	5.1 Design & build																									
	5.2 Simulation testing																									
	5.3 Performance optimisation																									
<b>WP6</b>	<b>Protection of IPR</b>	<b>1, 2, 3, 4, 5, 6, 7, 8, 9</b>																								
	6.2 Technology transfer																									
	6.3 Dissemination of knowledge																									
	6.4 Social economic aspects																									
	6.5 Promotion of exploitation																									
<b>WP7</b>	<b>Project management</b>	<b>1, 2, 3, 4, 5, 6, 7, 8, 9</b>																								
	7.1 Review and Management of Project Progress																									
	7.2 Review and Assessment of Economic and Social Impact																									
	7.3 Workflow Scheduling and Change Management																									
	7.4 Intra-Consortium Comm'n & Co-ordin'n of Technical Activities																									
	7.5 Provision and Monitoring of Technical Minutes																									
<b>WP8</b>	<b>Consortium Management</b>	<b>1</b>																								
	8.1 Coordination of Knowledge Management and IRA.																									
	8.2 Collation of Cost Statements and Audit Certificates.																									
	8.3 Co-ordination of Legal, Contractual, Ethical and Financial Asp.																									
	8.4 Comm'n between Consortium & EC. Organis'n of Proj. Meetings																									
	8.5 Co-ord'n of Gender Equality, Ethical and Soc. Aspects																									
	<b>Milestones</b>							X			X						X						X		X	

## **SECTION 4. Conclusions**

The key technologies and systems developed in SAFELUBE were subject to the restriction of a limited project time. Key consortium members withdrew from the project and their replacement proved too difficult to fulfil within the project lifetime. Therefore the limited development and results generated are still subject to Intellectual Property Rights (IPR) protection and cannot be published. When the consortium are reformed and in a position to take this technology to its next developmental stage then the benefits and knowledge of the SAFELUBE project can then be further disseminated.

## Appendix 1: D21 Draft Dissemination and Use Plan

### Dissemination and Use Plan

Project number: COOP-CT-2005-017970

Project acronym: SAFELUBE

Project title: *A Novel Method of Continuous On-line Conditioning & Safe Management to Enable the use of Fire Retardant Lubricants in Gas & Steam Power Generation Turbines*

#### Section 1 – Exploitable Knowledge and its Use

##### 1.1. Exploitable Results

Overview Table

<b>Exploitable Knowledge</b>	<b>Exploitable Product(s) or Measure(s)</b>	<b>Sector(s) of Application</b>	<b>Timetable for Commercial Use</b>	<b>Patents or Other IPR protection</b>	<b>Owner and Other Partners Involved</b>
<b>A -</b>	Tunable laser diode sensor for SAFELUBE system	Power generation/turbine manufacture industry	End of project	Expected	Alpes
<b>B</b>	Dehydration & degassing equipment for the SAFELUBE system	Power generation/turbine manufacture industry	End of project	Expected	Minerwa
<b>C</b>	Process control instrumentation for the SAFELUBE system	Power generation/turbine manufacture industry	End of project	Expected	Macq
<b>D</b>	Optical communications for the SAFELUBE system	Power generation/turbine manufacture industry	End of project	Expected	Safibra
<b>E</b>	Integrated SAFELUBE system	Power generation/turbine manufacture industry	End of project	Expected	Kelman

**A: Tunable laser diode sensor for SAFELUBE system**

Solid state laser diode based sensor system for measurement of acids in turbine lubricants.

**B: Dehydration & degassing equipment for the SAFELUBE system**

Dehydration & degassing membranes for application to turbine lubricants.

**C: Process control instrumentation for the SAFELUBE system**

Process control system to enable turbine lubricant monitoring and management.

**D: Optical communications for the SAFELUBE system**

Communications system to enable turbine lubricant monitoring and management.

**E: Integrated SAFELUBE system**

Integrated turbine lubricant monitoring and conditioning system

## **Section 2 – Dissemination of Knowledge**

The technology that had been developed will be available for dissemination through the use of the project's case studies. The pilot manufacturing unit will be used as a demonstration facility, which will be a valuable tool for the participants in building a network of licensees. All participants will play an active role in technology transfer and dissemination, promoting the technology development to customers, and through networks of industrial contacts. In the first instance publications, editorials and demonstrator CD ROMs will be supplied to the Managing/Financial Directors of the 200 largest EC power distribution utilities, all the main rail network operators and a selection of the largest industrial and retail turbine owners. Where possible, links will be made with existing EC funded projects and relevant Thematic Networks. Trade Associations throughout Europe will be used to network the results and help demonstrate the technology to end users in a variety of industry sectors, (for example CIGRE, IEEE). We will also place of advertorials and technical articles in industry journals, such as GDS International Ltd

The exploitation manager will coordinate continued evaluation of the primary and all secondary market potentials (including sales forecasts and monitoring of initial feedback from the target market end users) and in so doing will create a market development plan appropriate to the needs of the consortium. An End User Interest Group of interested parties, including industry consults, power distribution utilities and large enterprise end users from various primary and secondary market application sectors will be gathered to assist in the marketing of the new product. For European and global regions beyond the reach and ability of the primary partners to supply to, licensees will be actively recruited by a variety of measures including trade shows, industry seminars, targeted media campaigns and Internet marketing. These activities will make use of prototype technology demonstrators (both virtual and real) to impart the benefits of the new product and provide validation of the results achieved.

The project has an established internet portal for use by project partners (<http://safelube.pera.com>). This would have supported a public-facing website in order to make appropriate information available to the public should the project had fulfilled all its objectives and completed the timeplan. There will potentially be publishable aspects to some of the reports completed for the project, although these are currently limited in content due to lack of patent protection.

It had been planned that once the concept was proven, and available in a demonstration unit with patent protection the consortium will increase their dissemination activities. This would involve:

- More information in the public documents for the EC and others
- More information on the web site
- Press releases to specific journals
- Attendance and exhibiting at conferences.

If the SAFELUBE technology was taken forward in a new EC project, then sufficiently developed, established and protected, coordinated activities would then need to be

undertaken to bring the product towards the market. These would include Public Relations, Direct Marketing, Third Party Marketing, Back up material, e.g. literature and possibly advertising. Information would be disseminated to specific interested parties e.g. HSE and Environment Agencies. An advertising strategy would be considered, including factors such as the budget, the nature of the media and the timing.

### **Section 3 – Publishable Results**

The key technologies and systems developed in SAFELUBE were subject to the restriction of a limited project time. Key consortium members withdrew from the project and their replacement proved too difficult to fulfil within the project lifetime. Therefore the limited development and results generated are still subject to Intellectual Property Rights (IPR) protection and cannot be published. When the consortium are reformed and in a position to take this technology to its next developmental stage then the benefits and knowledge of the SAFELUBE project can then be further disseminated.