LEAD IN POWER CABLES

Lead Diffusion from High Voltage Cables

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Report title:	Lead Diffusion from High Voltage Cables	Materials & Testing - Oslo-4100-NO
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Applicable contract(s) governing the provision of this Report:
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Objective:

The objective of the work is dismantling of seven (7) high voltage cable samples, extraction of polymer samples from each of the cable polymer sheath in contact with lead sheath for further testing. The aim has been to check for lead diffusion through the polymer sheath.

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Keywords:
Power cables, lead diffusion

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1 EXECUTIVE SUMMARY

Europacable and ENTSO-E have commissioned DNV AS, section for Materials Advisory at Høvik as a 3rd party, to carry out a dissection and analysis of selected submarine and land power cables, that have been in service for some of the samples up to more than 40 years. The dissection was carried out to extract polymer sheath samples in order to check for lead diffusion through the polymer sheath. In total 7 lengths of cables have been received for examination.

The objective of the work was to extract samples of polymer sheath that has been positioned outside the lead sheath, without contaminating the area to be analysed and to check if lead diffusion has occurred to outer polymer sheath surface. This is to understand if there is a risk that lead is leaking out of the cable.

A general illustration of high voltage power cable and where samples from PE sheath was extracted is shown in Figure 1-1. Please note that Figure 1-1 is an illustration, and the surrounding layers of PE sheath are not represented in the sketches. As such it looks like an underground cable.

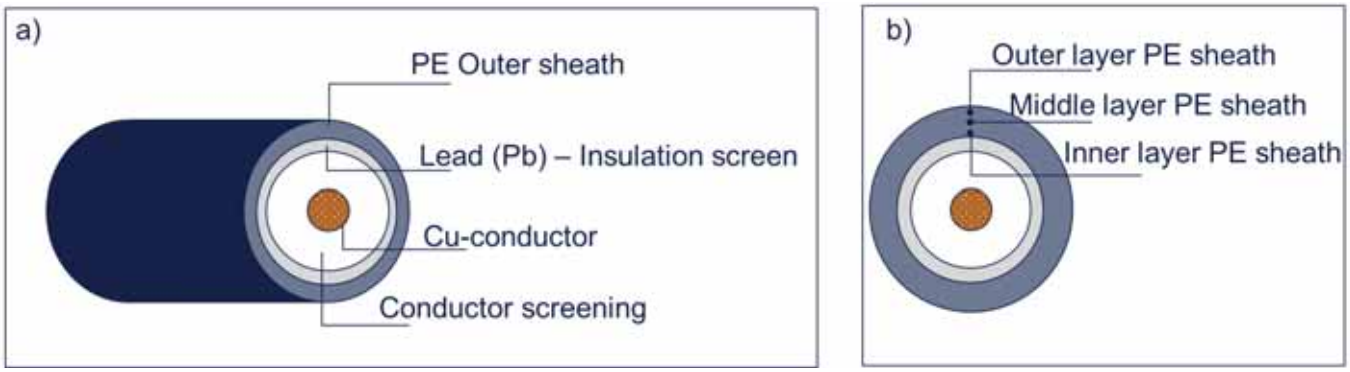

Figure 1-1 Illustration of where test samples have been extracted a) Illustration of high voltage cable b) cross-sectional view - sample extracted from three (3) positions of the PE sheath positioned outside Lead (Pb) layer.

1.1 Conclusions

The results showed similar findings for six (6) of the power cables with Polyethylene (PE) sheath outside the lead sheath.

Based on the results from the analysis the following is concluded for the Polyethylene (PE) samples:

Sample No. 1, 2, 4, 5, 6 and 8:

- It was not found Lead (Pb) diffusion through the polymer sheath to the outside in any of the samples. The samples extracted from the outer layer (shavings + microtome cutting) and middle layer showed low or non-detectable levels of lead.
- The inner layer of polymer sheath in direct contact with lead showed for three of the samples a higher but still within the level of ppms of lead compared with the outside samples, which is contamination from lead sheath to the inner surface of polymer sheath. The risk of contamination to inner polymer sheath surface is expected since it is in direct contact with the lead.

See also Section 5 Discussion for more details.

2 INTRODUCTION

Europacable and ENTSO-E have commissioned DNV AS, section for Materials Advisory at Høvik as a 3rd party, to carry out a dissection and analysis of selected submarine and land power cables that have been in service for up to more than 40 years some of the samples. The dissection was carried out to extract polymer sheath samples in order to check for lead diffusion through the polymer sheath. In total 7 lengths of cables have been received for examination.

2.1 Objective

The objective of the work was to extract samples of polymer sheath that has been positioned outside the lead sheath, without contaminating the area to be analysed and to check if lead diffusion has occurred to outer polymer sheath surface. This is to understand if there is a risk that lead is leaking out of the cable.

2.2 Scope of work

Seven (7) lengths of cable have been dissected. Each length provided is between 1 and 3 meters.

Scope of work carried out:

- Establish a procedure for the work
- Photo documentation of sample at arrival
- Dissection until polymer sheath
- Pre-cleaning of surface before cutting incl. photo documentation of surface
- Longitudinal cut along length of sample
- Visual inspection of lead cover, incl. photo documentation
- Sectioning of samples for further examination of polymer sheath
- Chemical analysis to check for lead diffusion. Two methods were used. Method 1 were carried out at the DNV Laboratories while method 2 were carried out at an external supplier.

    1) To perform qualitative/semi-quantitative chemical microanalysis, a scanning electron microscope (SEM) equipped with an energy dispersive spectroscopy (EDS) system will be used.

    2) ICP-SFMS method according to standard DIN EN ISO 17294-2, external supplier. A microtome will be used to extract 3 samples per cable, one from the internal surface (facing the lead), one from the bulk of the sheath/material and one from the external surface. In addition, 1x shavings were taken out from outer surface. The extractable amount of lead by acid digestion will be used for further analysis by ICP-SFMS.

2.3 Background information received from client

The environmental and human health protections governing the safe use of chemicals are of fundamental concern also for the power cable industry. This concern also includes lead since lead is used in many power cable applications. As part of a lead investigation program, Europacable and ENTSO-E is investigating whether lead used in power cables can diffuse out of the lead sheath into the next sheathing materials in a power cable, with the main objective to determine if lead is at risk of “leaking” from the cable into the environment.

Europacable, together with ENTSO-E, is executing this project to understand the potential implication of the use of lead in power cables.

2.4 Received samples

Samples of used submarine and land power cables were provided by the manufacturers and system operators of power systems. Overview of samples received is shown in Table 2-1

Table 2-1 Received information related to the samples examined. Samples are numbered from 1 to 8. Sample 7 is not included due to sample was not received in time.

#	Name of the link/Project	Company	Underground Submarine	Voltage	AC/DC	Type of cabe	Commission date
1	Skagerrak 2	Statnett	Submarine	250 kV	DC	MI cable	1976
2	Ionian Islands	IPTO	Submarine	150kV	AC	Paper with oil duct	
3	EHV sunstation Acharnes	IPTO	Underground	150kV	AC	XLPE	Around 2000
4	Kontiskan 2	Svenska kraftnät	Submarine	285 kV	DC	MI cable	1989
5	Copenhagen 400kV	Energinet	Underground	400kV	AC	XLPE	1999
6	IFA 2000	RTE	Submarine	270 kV	DC	MI cable	1986
~~7~~	~~Suomi underground cable~~	~~Fingrid~~	~~Underground~~	~~110 kV~~	~~AC~~	~~XLPE~~	~~1992~~
8	Fenno-Skan 1	Fingrid/SVK	Submarine	400 kV	DC	MI cable	1989

Sample 7 was not received in time to be included in the test program.

The polymer sheath information received from client are according to drawing/ received information:

- Sample No. 1: Polyethylene (PE) sheath
- Sample No. 2: Polymer sheath of unknown polymer material outside of the lead sheath.
- Sample No. 3: Polyvinyl chloride (PVC) with lead (Pb) stabilizer
- Sample No. 4: PE sheath
- Sample No. 5: PE sheath
- Sample No. 6: PE or Polypropylene (PP) sheath
- Sample No. 8: PE sheath

Due to material composition sample no. 3 is not included in final results from SF-ICP-MS testing.

2.5 Kick-Off Meeting and Witnessing

A kick-off meeting was held the 25th of March 2022 where the procedure for the work was agreed upon.

Client witnessing via Teams and onsite was arranged for the following activities:

- Dissection of power cables for extraction of samples from the polymer sheath covering the lead (Pb) was carried out at DNV facilities at Høvik, Norway. Witnessing held the 30th and 31st of March 2022 via Teams and onsite visitor.

- Microtome cutting of samples extracted and prepared microtome cutting at Norner AS Porsgrunn, Herøya, Norway. Witnessing via Teams only held the 8th of April 2022.

3 METHOD

High level description of method:

- Dissection of power cables, extraction of samples from the polymer sheath outside the lead sheath
    - Shavings from the outer surface
    - Cuts from the polymer sheath sectioned by microtome into 3 samples, outer surface, mid-thickness sample and inner surface (facing the lead sheath)
- Chemical analysis measurements to measure the content of lead on samples extracted. The different steps are illustrated in images below

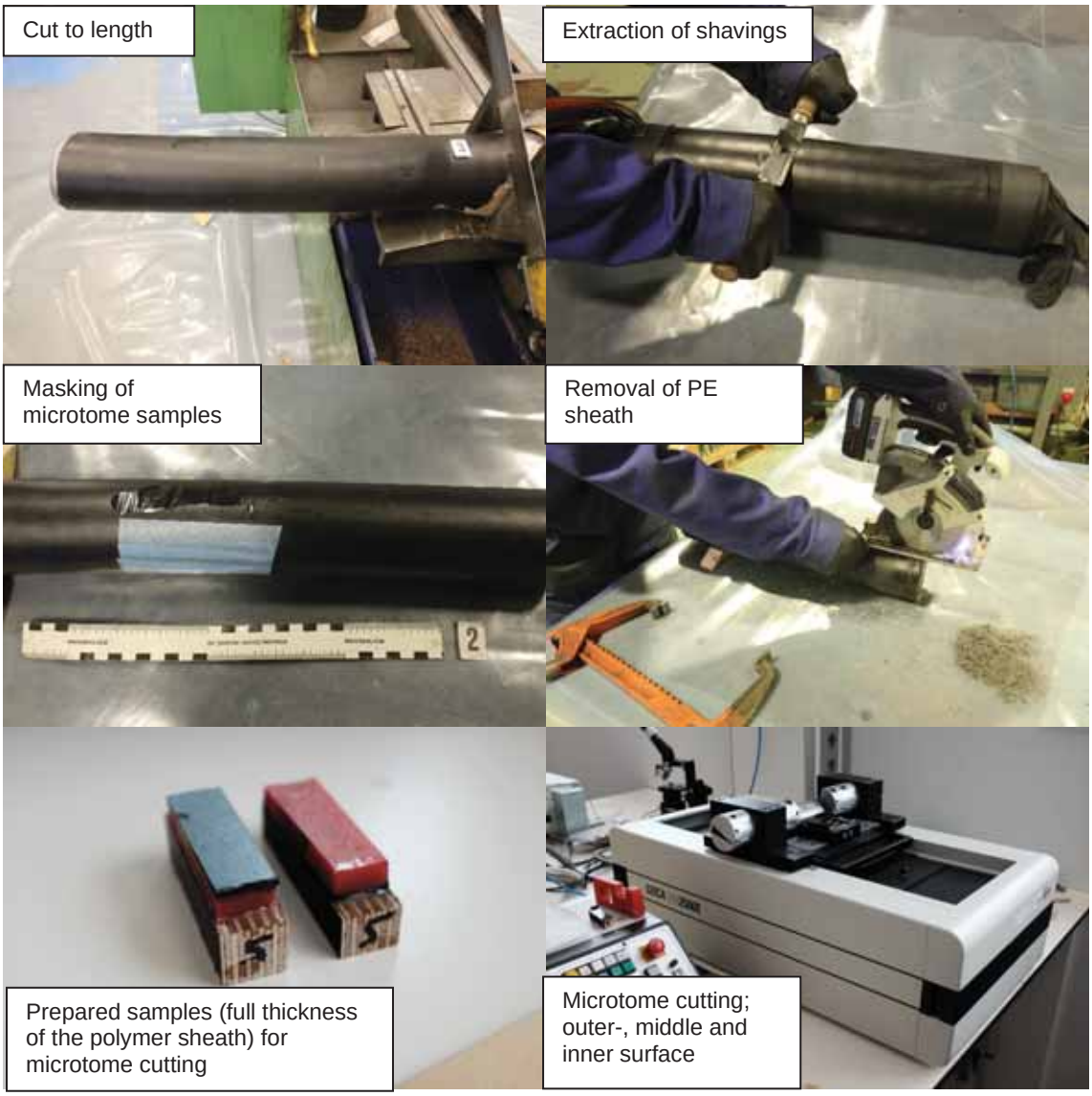

Figure 3-1 Illustration over the main steps during dissection and sample extraction.

3.1 Dissection

The dissection work was carried out according to Procedure in Memo No 1521198-1 (2022-04-25), see Appendix A.

Note, the procedure has been updated with regards to microtome cutting and sample extraction for further testing, after attempting microtome cutting on dummy samples.

The updates of the procedure are comprising of the following.

The samples were first cut to length of 0.5 m.

Extra measures in order to avoid contamination were taken during dissection to avoid cross contamination of lead. Cover foils were placed on the table where samples were dissected, new clean cover foils were applied when arriving to the polymer-sheath. In addition, the cut surfaces were sealed with rubber cover (nitrile rubber gloves).

In order to have PE sheath material with as less handling as possible, samples were shaved off by use of a plane knife. There were taken in addition to the samples extracted by microtome cutting. Shavings were collected in individual bags:

- Including outer surface (Shaving including outer surface of polymer sheath, decided to be tested)
- Below outer surface (Shaving samples not including outer surface of polymer sheath, was decided not to be tested)

All cleaning of the samples was done by using ethanol and clean rags.

3.2 Microtome

The microtome cutting at Norner AS was carried out according to Procedure in Memo No 1521198-1 (2022-04-25), see Appendix A. The procedure was updated after initial procedure had been approved due to improvements identified after initial trials of microtome cutting of dummy samples. The samples needed to be reduced in size and glued to a support in order to carry out microtome cutting. The steps and images from microtome cutting are included in Appendix A.

The thickness of the samples was measured with a digital Vernier calliper prior to microtome cutting. The microtome layers were taken in three positions: outer surface, middle section and inner surface. The depth the layers were taken from (distance from outer surface of the sheath) were noted in addition to the sample extraction weight, see Appendix B.

3.3 SF-ICP-MS testing

The Section Field-Inductively Coupled Plasma-Mass Spectrometry (SF-ICP-MS) testing was carried out by Sintef Norlab AS Porsgrunn at Herøya, Norway. Sintef Norlab Progrunn holds an accreditation for chemical analysis from Norwegian Accreditation enclosed in Appendix D.

The instrument limit of quantification (detection threshold) is 0.2 ppm or 0.2mg/ kg.

The samples were prepared after microwave assisted decomposition in a closed vessel based on Milestone application recommendations. The PE-based sample materials are analysed with SF ICP-MS technique, after an internal method based on EN ISO 17294-2 and EPA 200.8.

3.4 Scanning electron microscope – EDS

To perform qualitative/semi-quantitative chemical microanalysis, a scanning electron microscope (SEM) equipped with an energy dispersive spectroscopy (EDS) system was used. The EDS system functions by measuring the energy of the X-rays that are emitted from the samples due to interaction with the incoming high energy electron beam. The samples were sputtered with gold (Au) to achieve conductivity. Each element produces a characteristic spectrum of emitted X-rays, which can be identified during the analysis.

- (The identification is used to produce a qualitative or semi-quantitative elemental distribution.)
- Due to contaminations from organic compounds etc. carbon (C) may be present to some degree in all spectra. This is not further elaborated in the results table.
- EDS spectra shown have x-ray energy on the x-axis and count rate (partly proportional to content) on the y-axis.

4 RESULTS

The samples received are shown in Figure 7-1 to Figure 7-6.

4.1 Dissection of cables

Observations during dissection are listed in Table 4-1.

Table 4-1 Observations made during dissection

Sample No.	Observations	Figures
1	No significant observations. The shavings were extracted up to a depth of max. 2 mm from PE outer surface.	See Section 7.1
2	Inside of polymer sheath was covered by a substance that appeared sticky and was not removed by cleaning with ethanol. The shavings were extracted up to a depth of max. 1.5 mm from outer surface.	See Section 7.2 and 7.8
3	No significant observations. The shavings were extracted up to a depth of max. 2 mm from PVC outer surface.	See Section 7.3
4	Tape stuck to outer surface of PE-sheath making it difficult to clean. The shavings were extracted up to a depth of max. 2.3 mm from PE outer surface.	See Section 7.4
5	No significant observations, PE-sheath was black and red in colour. The shavings were extracted up to a depth of max. 3.1 mm from PE outer surface.	See Section 7.5
6	Inside of polymer-sheath (white in colour) was covered by substance that appeared sticky and was not removed by cleaning with ethanol. The shavings were extracted up to a depth of max. 2.9 mm from outer surface.	See Section 7.6 and 7.8
8	Lead sheath showed discoloration also visible on inside of PE sheath. The discoloration on inside of PE sheath was not possible to remove by cleaning with ethanol. The shavings were extracted up to a depth of max. 2 mm from PE outer surface.	See Section 7.7 and 7.8

The polymer sheath outside the lead sheath has been assumed to be PE (Polyethylene) except sample No. 3 that is PVC material. Information regarding composition has not been received for sample No. 2.

4.2 SF-ICP-MS testing

The SF-ICP-MS test results for PE-sheath samples are summarized in Table 4-2 below.

Table 4-2 PE-samples; Table of the results from the SF-ICP-MS testing, Lead content [mg/kg]

Sample No.	1	2	4	5	6	8
	Units [mg/kg]					
Shaving	0.3	0,2	0,2	0,2	< 0,2	0,8
Outer surface	0,3	< 0,2	0,5	< 0,2	0,7	0,2
Middle	< 0,2	< 0,2	0,2	0,2	0,3	0,3
Inner Surface	0,5	37	0,7	0,3	28	640

The results show that the level of lead (Pb) detected on samples extracted at outer surface and middle section is low and close to and below in some cases the instrument limit of quantification (detection threshold), 0.2 ppm or 0.2mg/ kg. The highest lead content that was measured on an outer surface was up to 0.8 ppm, however, corresponding sample either extracted by shaving or microtome cutting showed a low value.

The inner surface samples, sample No. 2, 6 and 8, in direct contact with the lead sheath showed significantly higher lead (Pb) content compared to the other values achieved, it is assumed to be contamination of lead (Pb) from being in contact with the lead sheath. Test report is enclosed in Appendix C.

4.3 Scanning electron microscope – EDS

A cross section of PE sheath from Sample 1 was examined by SEM/EDS. No lead (Pb) was detected on the outer- and inner surface. The accuracy by SEM/EDS analysis is significantly lower than SF-ICP-MS testing and typically the lower limit to what could be detected is 0.1-0.3 Wt% of an element.

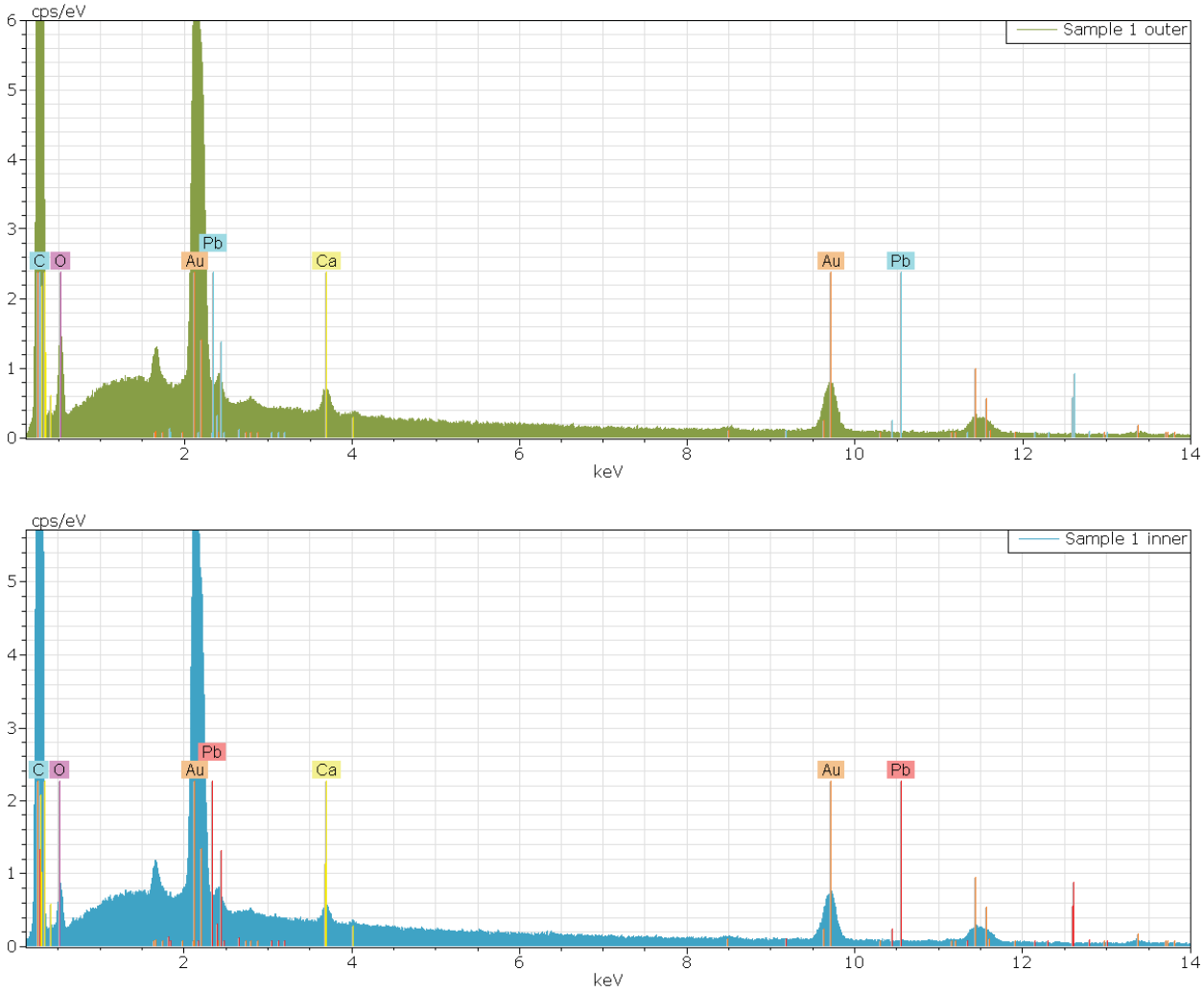

Figure 4-1 SEM/ EDS spectrum on cross section sample No. 1 at outer- and inner surface. Note position of lead (Pb) is added to show where peak of lead should have been, no Pb detected in neither of the positions.

The polymer sheath from sample No. 3 it was detected lead by SEM/ EDS, see spectra below.

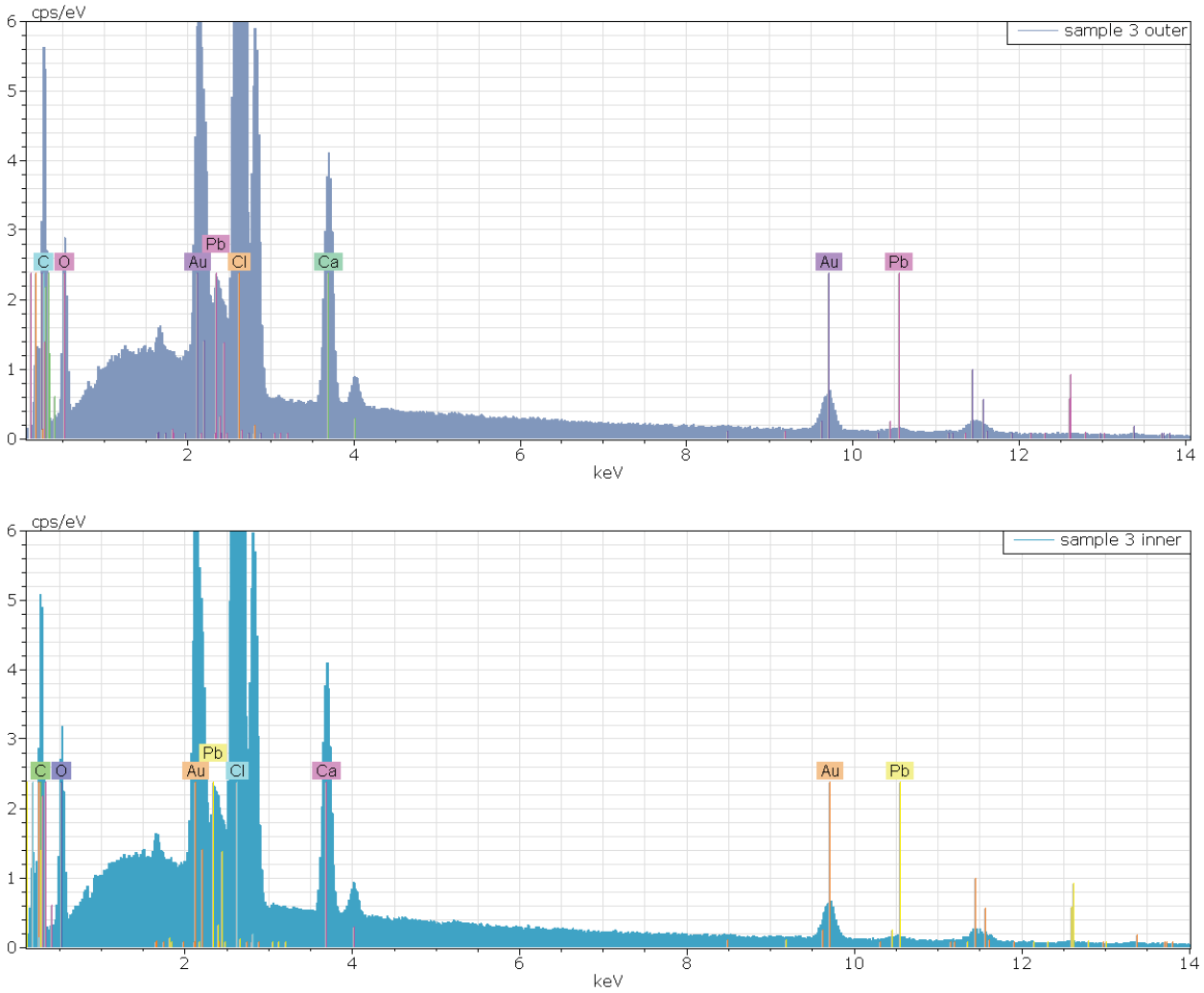

Figure 4-2 SEM/ EDS spectrum on cross section sample No. 3 at outer- and inner surface. A small peak of Pb detected in addition to chlorine (Cl).

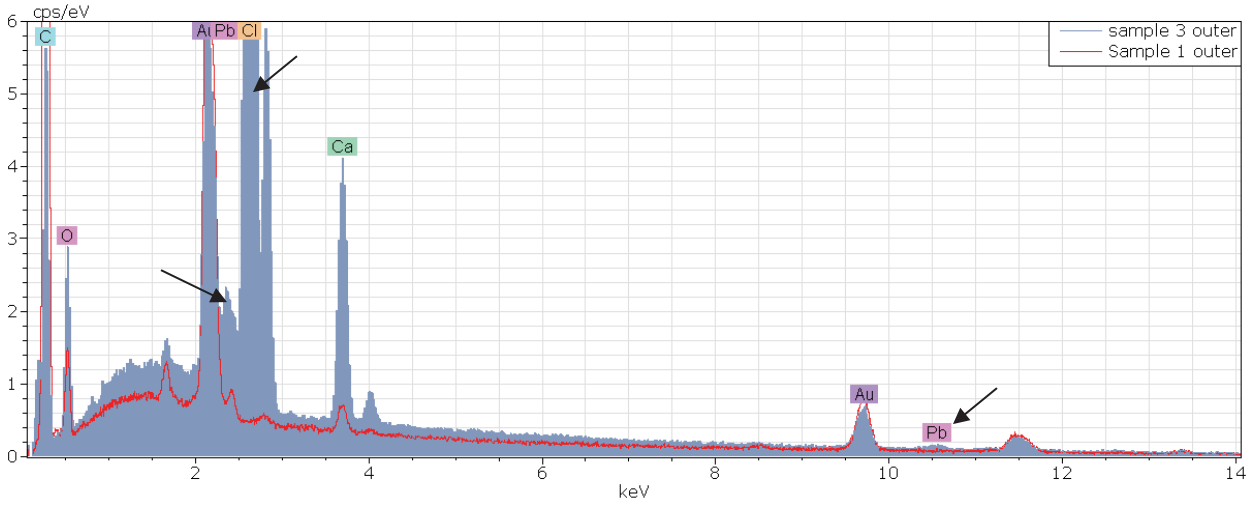

Figure 4-3 EDS comparison of sample No. 1 (red) and 3 (blue) showing that sample 3 has different composition with chlorine (Cl) and some lead (Pb), indicating that sample No. 3 is PVC with lead stabilizer as informed. Arrows show where differences in composition to sample No. 3 is compared to No. 1.

Table 4-3 Semi-quantitative chemical composition analysis by SEM/ EDS

Wt%	Sample 1 inner surface towards lead	Sample 1 outer surface	Sample 3 inner surface towards lead	Sample 3 outer surface
C	86.62	73.44	35.55	35.79
O	4.84	4.94	10.67	9.62
Cl	-	-	37.48	35.50
Ca	0.53	0.53	4.24	4.12
Au	8.01	21.09	11.82	13.03
Pb	0.00	0.00	2.24	1.93

SEM/ EDS was not able to detect any lead (Pb) by SEM/EDS to Sample No. 1. As shown in Figure 4-3 a comparison between sample no. 1 and no. 3 indicates that sample no. 3 is PVC with lead stabilizer as informed, due to chlorine (Cl) and lead (Pb) detected.

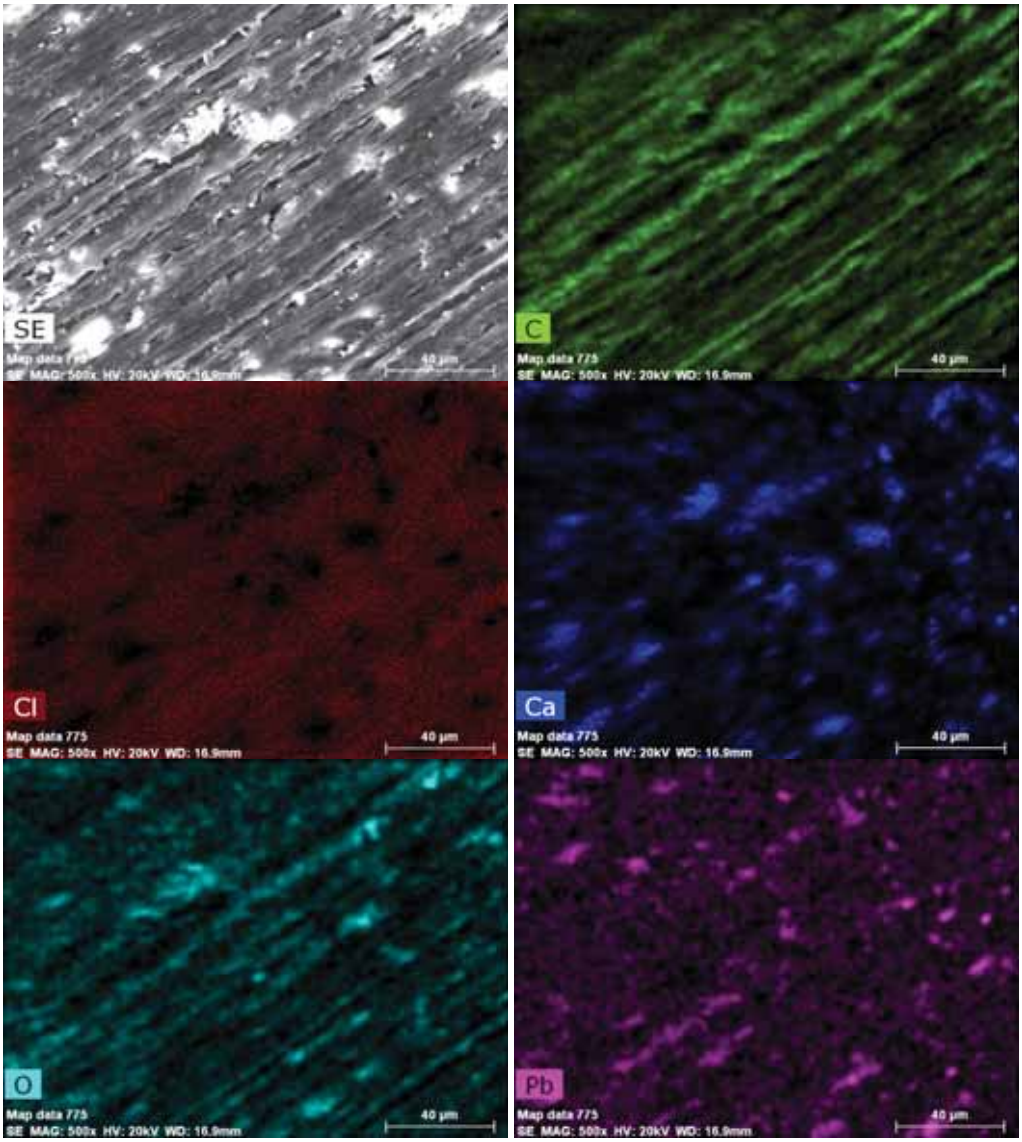

Figure 4-4 SEM/ EDS mapping of sample No. 3. Mapping indicates that the observed lead (Pb) is concentrated as particles (increased intensity shows higher content of an element).

5 DISCUSSION

The lead content measured by SF-ICP-MS was in general low and close to the detection limits of the instrument (0.2 ppm). The lead content measured for the samples extracted from the mid-thickness was low, which shows that no lead diffusion has occurred. The highest lead content that was measured on an outer surface was up to 0.8 ppm, however, corresponding sample from outer surface either extracted by shaving or microtome cutting showed a low value. This is indicative of variation in content due to sample handling and/ or extraction.

From the samples extracted from the inner surface in direct contact with the lead sheath, three of the samples (sample no. 2, 6 and 8) showed a higher lead content compared with the other samples. The reasons for the significantly higher lead content at inner surface for sample No. 2, 6 and 8 are considered to be:

- Sample No. 2 and 6 had a sticky inner surface that was not possible to remove with clean rags and ethanol. The sticky/ gluey surface could easily collect any residues from handling of the samples during dissection and sample extraction, which later was not removed by cleaning with ethanol (see Figure 7-40).
- On sample No. 8 there was an evident discoloration observed on lead- and PE-sheath which was not possible to remove by ethanol cleaning (see Figure 7-40).

For the other samples (sample No. 1, 4 and 5) the lead content was measured to be low also at the inner surface.

Sample No. 3 showed a distinctly higher lead content compared to the sheaths of all the other cable length samples received. High lead content was measured on all samples extracted from Sample No. 3. The sample was informed to be PVC material with lead stabilizer, SEM/ EDS analysis showed that chlorine (Cl) and lead (Pb) was present in sample No. 3 indicative of PVC material. It was informed that PVC lead (Pb) stabilizers has been a commonly used additive for more than 20 years ago. It was not observed any degradation of the polymer in the form of embrittlement. Sample 3 is an underground cable. Due to the high Pb content in sample No. 3 this sample has not been possible to use for evaluation of lead (Pb) diffusion.

6 CONCLUSION

The results showed similar findings for six (6) of the power cables with Polyethylene (PE) sheath outside the lead sheath.

Based on the results from the analysis the following is concluded for the Polyethylene (PE) samples:

Sample No. 1, 2, 4, 5, 6 and 8:

- It was not found Lead (Pb) diffusion through the polymer sheath to the outside in any of the samples. The samples extracted from the outer layer (shavings + microtome cutting) and middle layer showed low or non-detectable levels of lead.
- The inner layer of polymer sheath in direct contact with lead showed for three of the samples a higher but still within the level of ppms of lead compared with the outside samples, which is contamination from lead sheath to the inner surface of polymer sheath. The risk of contamination to inner polymer sheath surface is expected since it is in direct contact with the lead.

7 FIGURES

Received samples pr. 25-03-2022

Figure 7-1 Sample 1 at arrival

Figure 7-2 Sample no. 2 and 3 at arrival

Figure 7-3 Sample 4 at arrival

Figure 7-4 Cable lengths of sample no. 5 (Red PE-sheath)

Figure 7-5 Sample No. 6 at arrival

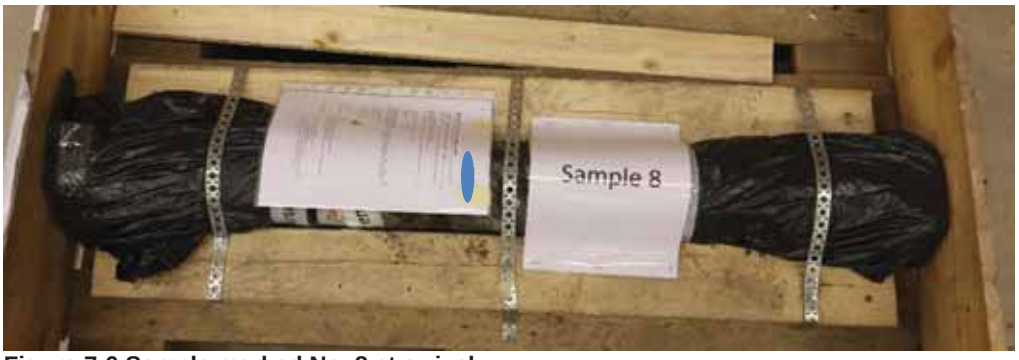

Figure 7-6 Sample marked No. 8 at arrival

7.1 SAMPLE 1

Figure 7-7: Sample 1 as received and cut to length.

Figure 7-8: Sample 1 PE sheath overview.

Figure 7-9: Sample 1 PE sheath close-up

Figure 7-10: Sample 1 PE sheath after extraction of shaving samples.

Figure 7-11: Sample 1 after removal of PE sheath – lead sheath revealed.

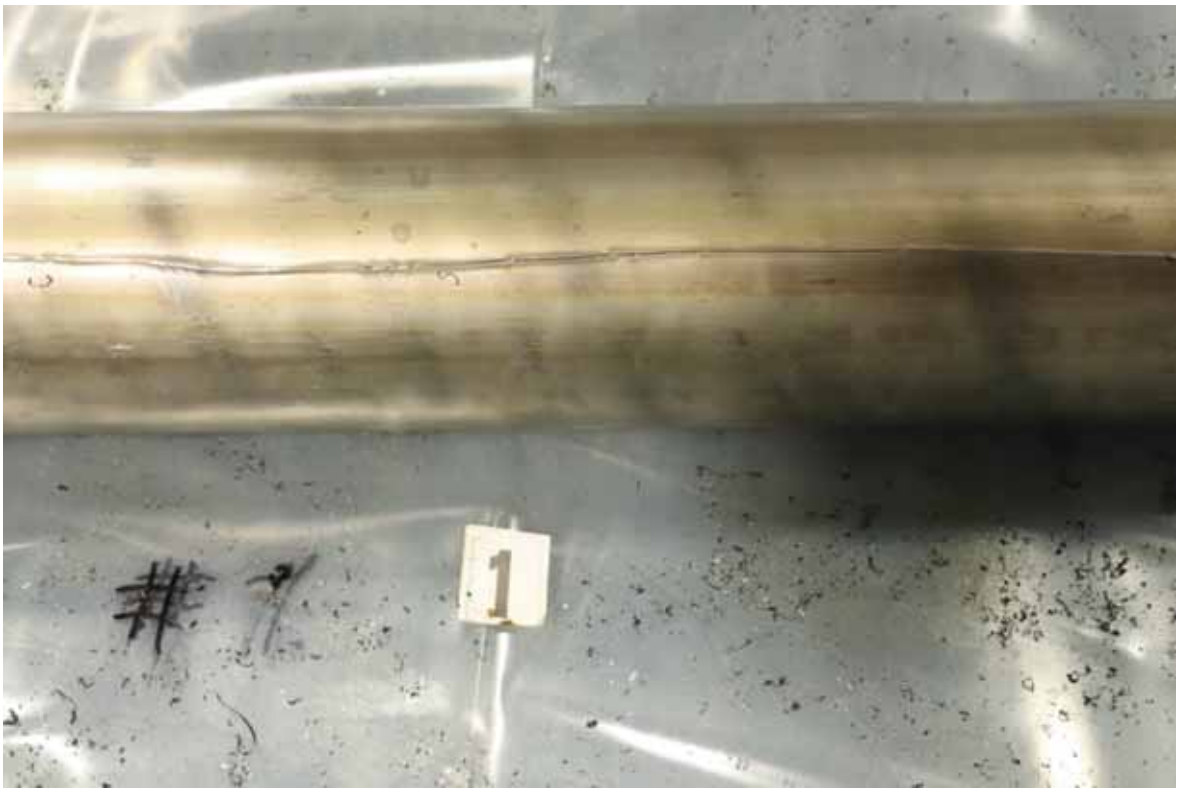

Figure 7-12: Sample 1 close-up of lead sheath.

7.2 SAMPLE 2

Figure 7-13 Sample 2 as received prior to cutting to length.

Figure 7-14: Sample 2 overview of polymer sheath and with protection on the cut surfaces (nitrile gloves).

Figure 7-15: Sample 2 close-up of the outer surface of the polymer sheath.

Figure 7-16: Sample 2 polymer sheath after extraction of shaving sample.

Figure 7-17: Sample 2 after removal of polymer sheath – Pb sheath revealed.

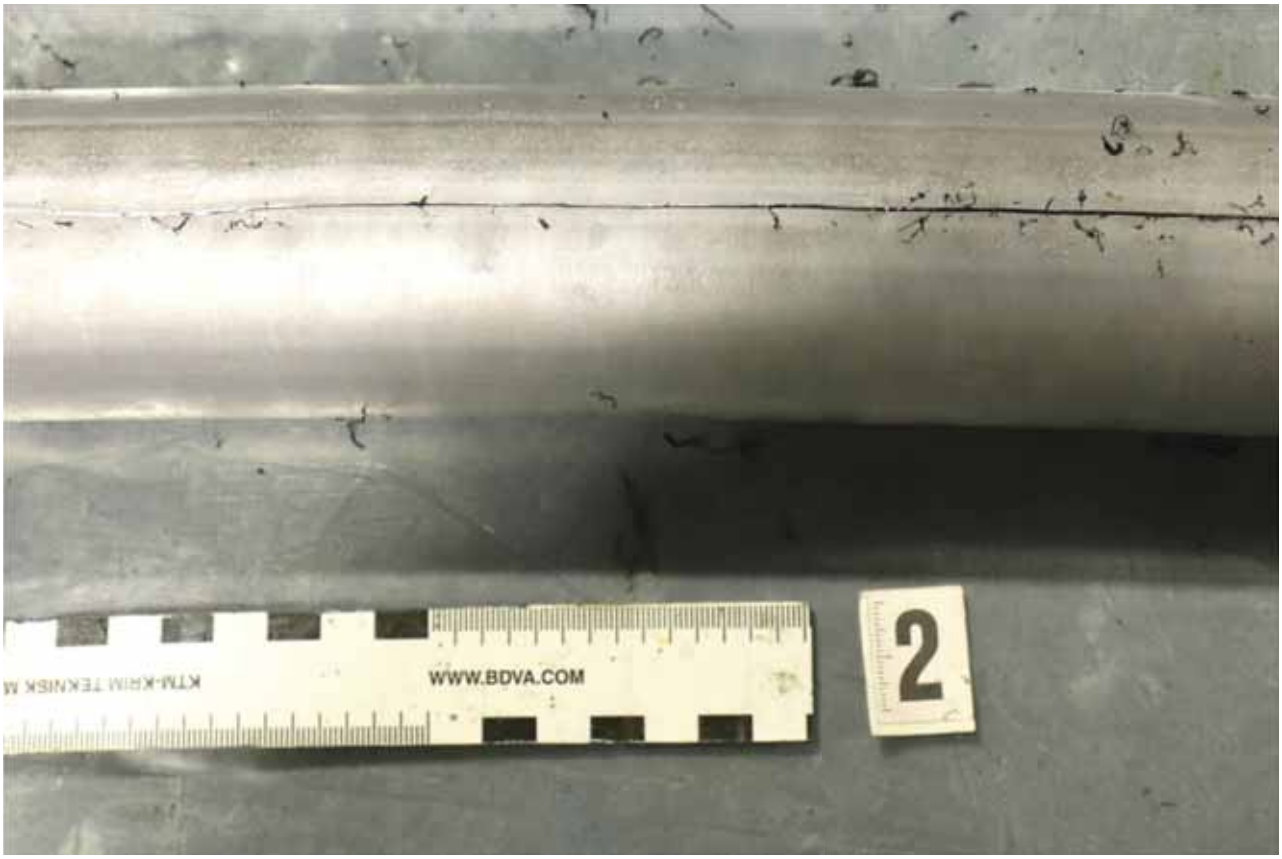

Figure 7-18: Sample 2 close-up of Pb sheath.

7.3 SAMPLE 3

Figure 7-19: Sample 3 as received and cut to length, and with protection on the cut surfaces (nitrile gloves).

Figure 7-20: Sample 3 close-up of the outer surface of the PVC sheath.

Figure 7-21: Sample 3 PVC sheath after extraction of shaving sample.

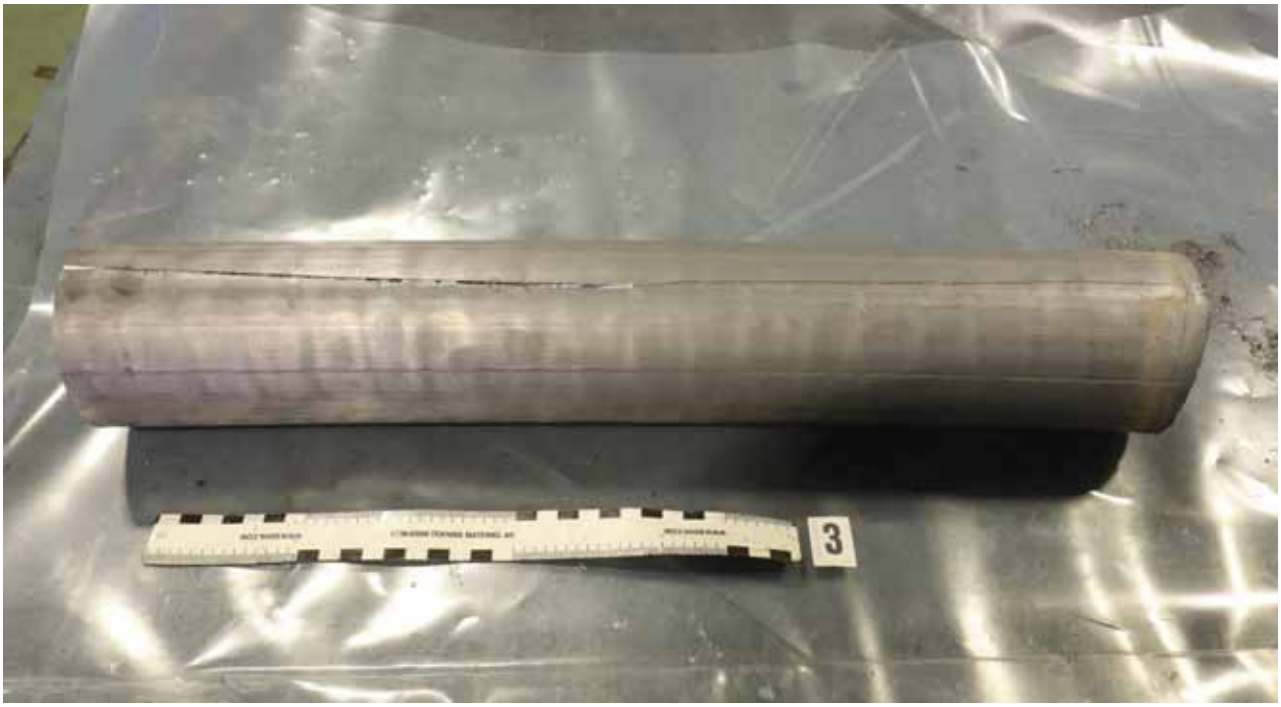

Figure 7-22: Sample 3 after removal of PVC sheath – Pb sheath revealed.

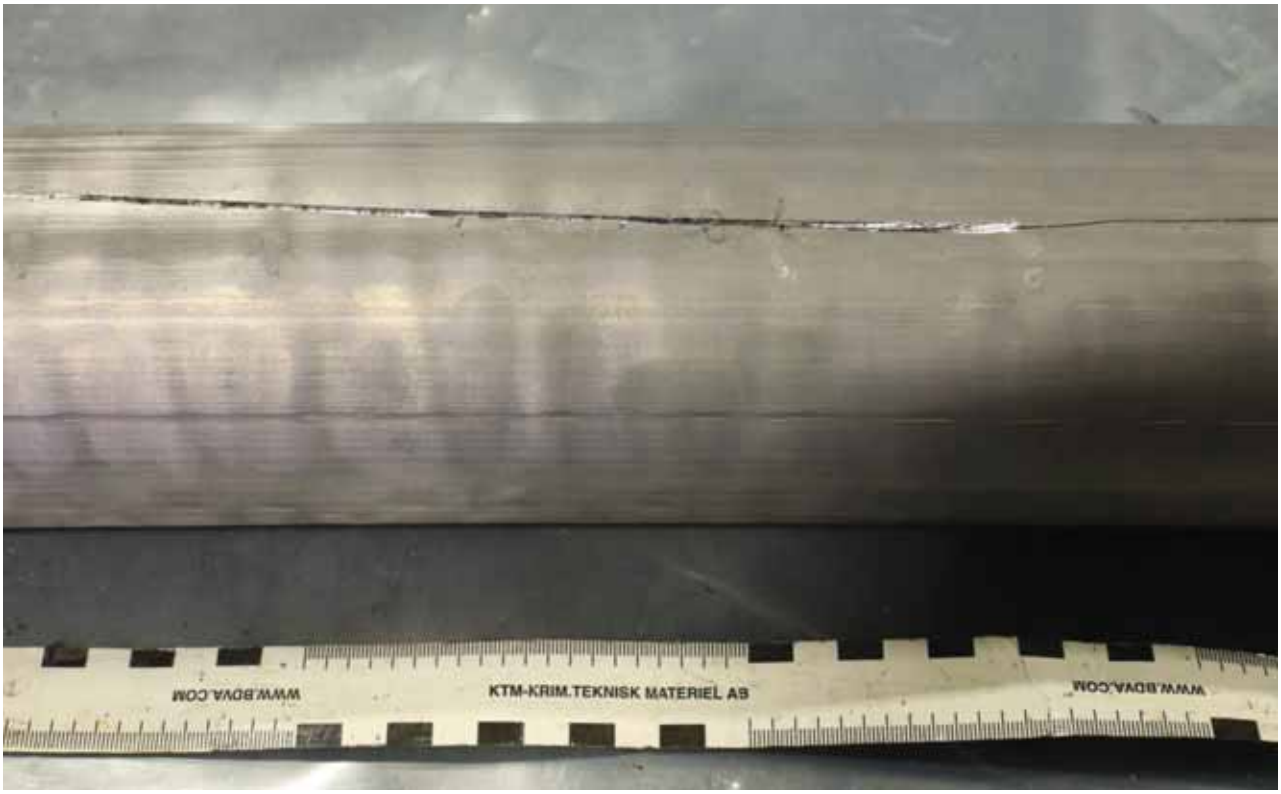

Figure 7-23: Sample 3 close-up of Pb sheath.

7.4 SAMPLE 4

Figure 7-24: Sample 4 as received and cut to length.

Figure 7-25: Sample 4 PE sheath overview.

Figure 7-26: Sample 4 close-up of the outer surface of the PE sheath.

Figure 7-27: Sample 4 after removal of PE sheath – lead sheath revealed.

7.5 SAMPLE 5

Figure 7-28: Sample 5 as received and cut to length.

Figure 7-29: Sample 5 close-up of the outer surface of the PE sheath.

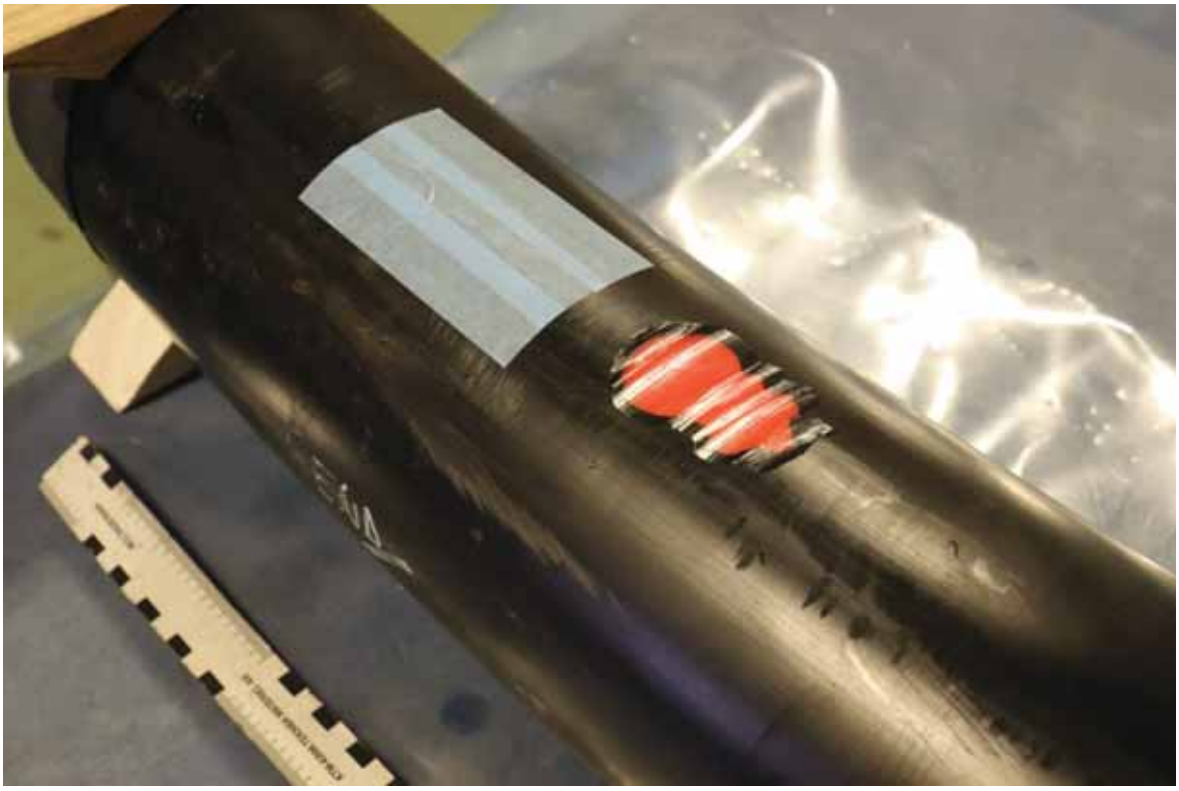

Figure 7-30: Sample 5 PE sheath after extraction of shaving sample.

Figure 7-31: Sample 5 after longitudinal cut in PE sheath. A layer of paper was present between the PE and the lead layer.

Figure 7-32: Sample 5 after removal of PE sheath – lead sheath revealed.

7.6 SAMPLE 6

Figure 7-33 Sample No. 6 as received prior to cutting to length

Figure 7-34: Sample 6 overview of PE/PP sheath, and with protection on the cut surfaces (nitrile gloves).

Figure 7-35: Sample 6 after removal of PE/PP sheath – lead sheath revealed.

7.7 SAMPLE 8

Figure 7-36 Sample 8 as received prior to cutting to length

Figure 7-37: Sample 8 overview of PE/PP sheath, and with protection on the cut surfaces (nitrile gloves).

Figure 7-38: Sample 8 after removal of PE sheath – lead sheath revealed. Discoloration of lead sheath.

7.8 Extracted polymer samples for ICP testing

Figure 7-39 Samples extracted from polymer sheath masked and section to size of approx. 50 x 50 mm.

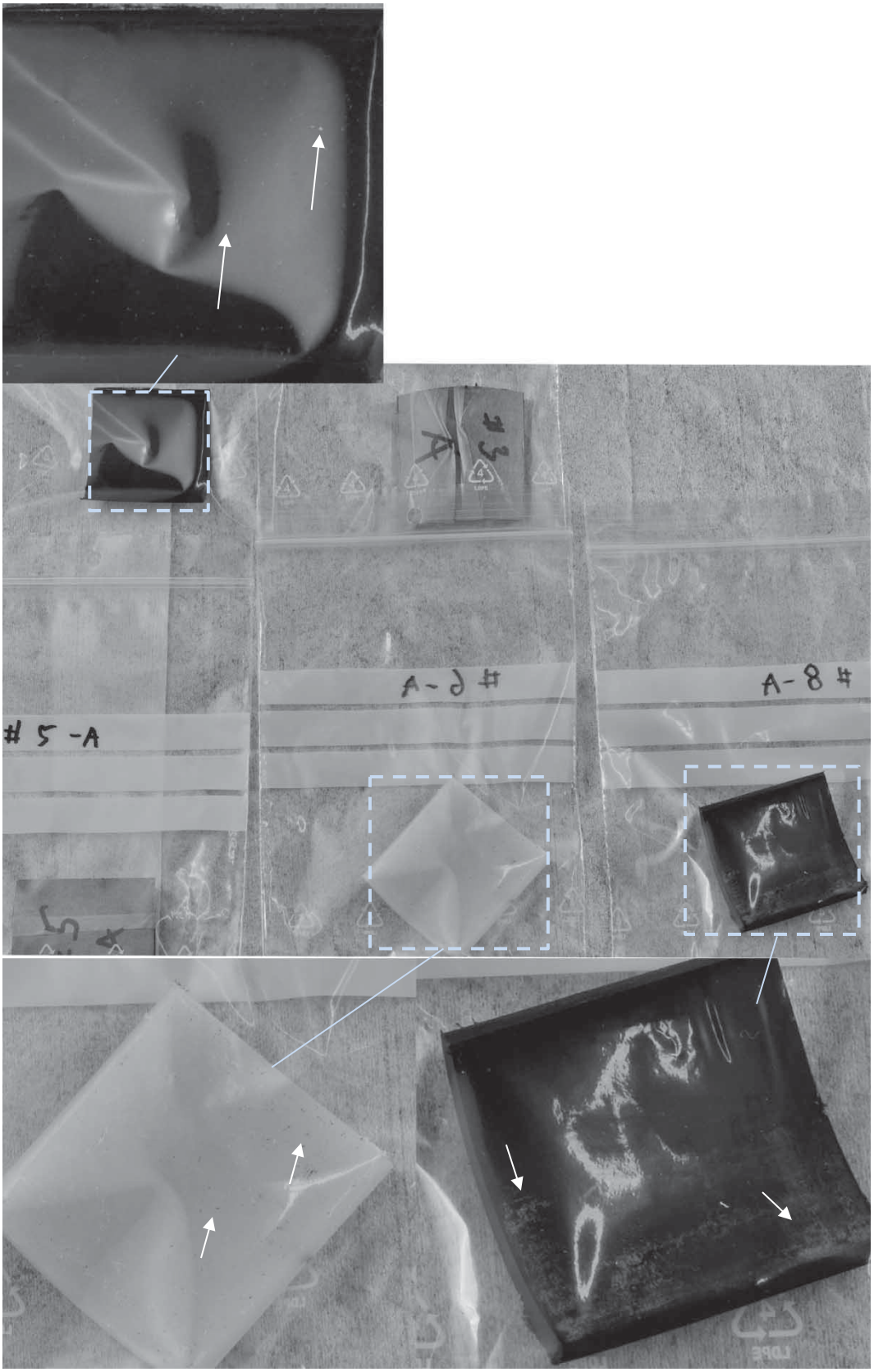

Figure 7-40 Images illustrating observations on the inner surface of sample No. 2, 6 and 8. Sample 2 and 6 was not possible to clean on polymer sheath inner surface (facing the lead) with ethanol due to a sticky residue. Sample No. 8 had staining on inner surface facing the lead not removed by cleaning. Arrows indicate particles/ residue on inner surface and staining.

APPENDIX A
Test procedure

Memo to:
Europacable

Memo No: **1521198-1**
Date: 2022-04-25
Prep. By: Jan Henrik Olsen
Verified By: Anne Kirsti Noren Egtvedt

Update since MEMO issued 2022-03-29

New revision of the Memo to include final procedure for microtome cutting after trial cut. The changes to sample preparation and sample size are included and description of microtome cutting.

Procedure for dissection and laboratory examination of PE sheath

Europacable has commissioned DNV AS, section for Materials Advisory at Høvik, to carry out a dissection of selected power cables to extract PE sheath in order to check for lead diffusion in the polymer sheath. In total 8 lengths of cables will be received for examination.

The objective of the work is to extract samples of polymer sheath outside the lead layer, without contaminating the area to be investigated and to check if lead diffusion has occurred to outer PE sheath surface. A diffusion curve will be made from 3x cuts at inner surface, mid-thickness and outer surface of PE-sheath.

The following scope of work is covered in the procedure:

- Photo documentation of sample at arrival
- Dissection until PE sheath (no photo documentation of layer-by-layer dissection)
- Pre-cleaning of surface before cutting incl. photo documentation of surface
- Longitudinal cut along length of sample, incl. photo documentation of cut
- Visual inspection of lead cover, incl. photo documentation
- Sectioning of samples for further examination of PE sheath
- Chemical analysis for lead penetration. Two methods will be used:
    1) SEM/ EDS
    2) ICP-SFMS method according to standard DIN EN ISO 17294-2. Note, analysis is not covered by this procedure only sampling and handling is covered by procedure.

This document is a procedure for extracting and examination of PE sheath samples for lead diffusion. Experience has shown that the risk for lead contamination during testing is high. Great care is therefore taken during all process step to as far as possible avoid contamination.

Witnessing points during layer-by-layer dissection and of microtome cutting is planned. Witnessing via Teams will be made possible

1 HSE GUIDELINES

1.1 PPE

PPE according to internal operating procedure, OP-E-NQ-TM-13-HMS-02-NO, shall be used. With special note/consideration to section 4.1.4. Handling of lead. **All handling of sample lengths and samples sectioned out from PE sheath to be handled with nitrile rubber gloves, to be disposed after use.**

For visitors, necessary PPE will be:

- Safety shoes
- Safety glasses
- Lab coat or boiler suit (optional)
- Hearing protection (supplied if needed)

SJA will be performed prior to work on the samples will start.

Care shall be taken when cutting the armouring layers, including securing surrounding workspace. Samples to be dissected down to lead layer, all surfaces that exposes lead to be wrapped in plastic.

1.2 Handling

Samples will be stored in separate room prior to dissection. Floor where samples are stored will be covered by plastic. Individual samples will be moved to workshop for dissection.

During handling and storage special care to PE sheath to avoid cross contamination samples to be wrapped in plastic prior to further sectioning.

1.3 Cutting

When dissecting the cable suitable fixation shall be used to secure the sample and to avoid injury. Special caution is to be taken when cutting through outer sheath of cable with armouring layer.

An evaluation of gas generation due to cutting of non-metallic sheath shall be evaluated and means shall be taken if this is found necessary.

1.4 Secured working area

The work location, weld shop with an overhead crane, shall be marked as restricted area to ensure that personnel not familiar with the project do not enter. Other work to be carried out in the area during dissection shall be agreed with PM.

Personnel in the work location should be kept to a minimum during the dissection. Entrance to the work location shall be agreed with PM and minimized to avoid disruptions and delays.

2 CABLE SAMPLE LAYER-BY LAYER DISSECTION

Eight (8) cable lengths will be dissected sequentially.

Prior to dissection the samples will be cut to length, 0.5 m, using a band saw. After cutting to length cut surface to be documented by photo and then wrapped in plastic.

The cables will be dissected layer-by-layer until lead layer, see Section 2.3.

2.1 As-received condition – External examination

Photographic documentation shall be made of cable length as received. Cross-sectional image to be taken, preferable after cutting to length.

2.2 Marking

The individual samples will be stored in pallets marked with number 1 to 8 according to received list from client. An extraction of the list is shown in Table 2-1.

Table 2-1 Received information related to the samples to be examined. Samples are numbered from 1 to 8.

#	Name of the link/Project	Company	Underground Submarine
1	Skagerrak 2	Statnett	Submarine
2	Ionian Islands	IPTO	Submarine
3	EHV sunstation Acharnes	IPTO	Underground
4	Kontiskan 2	Svenska kraftnät	Submarine
5	Copenhagen 400kV	Energinet	Underground
6	IFA 2000	RTE	Submarine
7	Suomi underground cable	Fingrid	Underground
8	Fenno-Skan 1	Fingrid/SVK	Submarine

The PE sheath marking and handling of sections for further testing is covered in Section 3 of this procedure. It will be extracted shavings from outer surface to PE sheath from all 8 samples in addition to 8 samples for microtome cutting that will cover through thickness. The samples will be marked by sample number 1 to 8 + designated letter for sample type (shaving, outer surface, mid-thickness and surface towards lead).

2.3 Layer-by-layer dissection procedure

Visitors will not be able to enter into the workshop during dissection and will be given access for inspection of PE-sheath, cut through PE sheath and inspection of lead layer.

Witnessing will be made possible via Teams for activities marked in green. The layer-by-layer dissection is completed when the PE sheath is removed. All other handling and sample preparation is covered in Section 3.

Tools needed:

- Electric circle saw
- Knife
- Masking tape
- Pen marker
- Plane knife
- Plastic for wrapping samples and remaining
- Tape
- Zip lock plastic bags for shavings and sections for further testing.

2.3.1 Sample No. 1

Table 2-2 Dissection layer-by-layer Sample No. 1. Action marked in Green are witnessing points.

Activity	Action	Observation/ Reporting
Marking	• Mark pallet with sample number 1 • Mark plastic bag for PE-sheath, No. 1	
Cut-to-length	• 1x Cross-sectional cut – sample length 1m. • Wrap exposed surfaces in plastic and seal by tape.	• Overview photo of received sample • Cross section photo
Dissection	• Strap around circumference both ends • Longitudinal cut through Corrosion protection layer • Remove outer armouring layer • Remove inner armouring layer • Inspect PE-sheath • Clean PE sheath outer surface with ethanol and rag. Dispose rag after cleaning. Surface to PE sheath at sample ends not to be cleaned.	• Document by photo PE-sheath • Photo of longitudinal cut PE sheath • Photo lead layer

	• Inspect PE-sheath after cleaning • Shave outer sheath surface samples store in marked zip lock bags • Apply masking tape to sample area at PE sheath outer surface • Longitudinal cut of PE sheath • Wrap PE sheath in clean plastic bag. Seal closed with tape. • Inspect lead layer • Wrap remaining sample length in plastic. • Store parts in pallet.	

2.3.2 Sample No. 2

Table 2-3 Dissection layer-by-layer Sample No. 2. Action marked in Green are witnessing points.

Activity	Action	Observation/ Reporting
Marking	• Mark pallet with sample number 2 • Mark plastic bag for Polymer-sheath, No. 2	
Cut-to-length	• 1x Cross-sectional cut – sample length 1m. • Wrap exposed surfaces in plastic and seal by tape.	• Overview photo of received sample • Cross section photo
Dissection	• Strap around circumference both ends • Longitudinal cut through Corrosion protection layer/ outer cover • Remove outer armouring layer • Remove inner armouring layer • Remove layer on top of Polymer-sheath • Inspect Polymer-sheath • Clean Polymer sheath outer surface with ethanol and rag. Dispose rag after cleaning. Surface to Polymer sheath at sample ends not to be cleaned. • Inspect Polymer-sheath after cleaning	• Document by photo Polymer-sheath • Photo of longitudinal cut Polymer sheath • Photo lead layer

	• Shave outer sheath surface samples store in marked zip lock bag • Apply masking tape to sample area at Polymer sheath outer surface • Longitudinal cut of Polymer sheath • Wrap Polymer sheath in clean plastic bag. Seal closed with tape. • Inspect lead layer • Wrap remaining sample length in plastic. • Store parts in pallet. • Move pallet to storage area.	

2.3.3 Sample No. 3

Table 2-4 Dissection layer-by-layer Sample No. 3. Action marked in Green are witnessing points.

Activity	Action	Observation/ Reporting
Marking	• Mark pallet with sample number 2 • Mark plastic bag for Polymer-sheath, No. 2	
Cut-to-length	• 1x Cross-sectional cut – sample length 1m. • Wrap exposed surfaces in plastic and seal by tape.	• Overview photo of received sample • Cross section photo
Dissection	• Longitudinal cut through Corrosion protection layer/ outer cover • Inspect Polymer-sheath • Clean Polymer sheath outer surface with ethanol and rag. Dispose rag after cleaning. Surface to polymer sheath at sample ends not to be cleaned. • Inspect Polymer sheath • Shave outer sheath surface samples store in marked zip lock bag • Apply masking tape to sample area at Polymer sheath outer surface • Longitudinal cut of Polymer sheath	• Document by photo Polymer-sheath • Photo of longitudinal cut Polymer sheath • Photo lead layer

	• Wrap Polymer sheath in clean plastic bag. Seal close with tape. • Inspect lead layer • Wrap remaining sample length in plastic. • Store parts in pallet.	

2.3.4 Sample No. 4

Table 2-5 Dissection layer-by-layer Sample No. 4. Action marked in Green are witnessing points.

Activity	Action	Observation/ Reporting
Marking	• Mark pallet with sample number 2 • Mark plastic bag for PE-sheath, No. 2	
Cut-to-length	• 1x Cross-sectional cut – sample length 1m. • Wrap exposed surfaces in plastic and seal by tape.	• Overview photo of received sample • Cross section photo
Dissection	• Longitudinal cut through Corrosion protection layer/ outer cover • Remove protection layers and armouring layers (7x layers) • Inspect PE-sheath (mantle) • Clean PE sheath outer surface with ethanol and rag. Dispose rag after cleaning. Surface to PE sheath at sample ends not to be cleaned. • Inspect PE sheath • Shave outer sheath surface samples store in marked zip lock bag • Apply masking tape to sample area at PE sheath outer surface • Longitudinal cut of PE sheath • Wrap PE sheath in clean plastic bag. Seal close with tape. • Inspect lead layer	• Document by photo PE-sheath • Photo of longitudinal cut PE sheath • Photo lead layer

	• Wrap remaining sample length in plastic. • Store parts in pallet.	

2.3.5 Sample No. 5

Table 2-6 Dissection layer-by-layer Sample No. 5. Action marked in Green are witnessing points.

Activity	Action	Observation/ Reporting
Marking	• Mark pallet with sample number 2 • Mark plastic bag for PE-sheath, No. 2	
Cut-to-length	• 1x Cross-sectional cut – sample length 1m. • Wrap exposed surfaces in plastic and seal by tape.	• Overview photo of received sample • Cross section photo
Dissection	• Longitudinal cut through Corrosion protection layer/ outer cover (black in color) • Inspect PE-sheath (red in color) • Clean PE sheath outer surface with ethanol and rag. Dispose rag after cleaning. Surface to PE sheath at sample ends not to be cleaned. • Inspect PE sheath • Shave outer sheath surface samples store in marked zip lock bag. • Apply masking tape to sample area at PE sheath outer surface • Longitudinal cut of PE sheath • Wrap PE sheath in clean plastic bag. Seal close with tape. • Remove half conductive band • Inspect lead layer • Wrap remaining sample length in plastic. • Store parts in pallet.	• Document by photo PE-sheath • Photo of longitudinal cut PE sheath • Photo lead layer

2.3.6 Sample No. 6

Table 2-7 Dissection layer-by-layer Sample No. 6. Action marked in Green are witnessing points.

Activity	Action	Observation/ Reporting
Marking	• Mark pallet with sample number 2 • Mark plastic bag for PE-sheath, No. 2	
Cut-to-length	• 1x Cross-sectional cut – sample length 1m. • Wrap exposed surfaces in plastic and seal by tape.	• Overview photo of received sample • Cross section photo
Dissection	• Longitudinal cut through Corrosion protection layer/ outer cover (PP Yarns) • Remove armouring layer • Remove tapings • Remove Plastic sheath (black) • Inspect PE/PP-sheath (W-T compound) • Clean PE sheath outer surface with ethanol and rag. Dispose rag after cleaning. Surface to PE sheath at sample ends not to be cleaned. • Inspect PE/ PP sheath • Shave outer sheath surface samples store in marked zip lock bag. • Apply masking tape to sample area at PE sheath outer surface • Longitudinal cut of PE sheath • Wrap PE sheath in clean plastic bag. Seal close with tape. • Inspect lead layer • Wrap remaining sample length in plastic. • Store parts in pallet.	• Document by photo PE-sheath • Photo of longitudinal cut PE sheath • Photo lead layer

2.3.7 Sample No. 7

Table 2-8 Dissection layer-by-layer Sample No. 7. Action marked in Green are witnessing points.

Activity	Action	Observation/ Reporting
Marking	• Mark pallet with sample number 2 • Mark plastic bag for PE-sheath, No. 2	
Cut-to-length	• 1x Cross-sectional cut – sample length 1m. • Wrap exposed surfaces in plastic and seal by tape.	• Overview photo of received sample • Cross section photo
Dissection	• Inspect PE-sheath • Clean PE sheath outer surface with ethanol and rag. Dispose rag after cleaning. Surface to PE sheath at sample ends not to be cleaned. • Inspect PE sheath • Shave outer sheath surface samples store in marked zip lock bag. • Apply masking tape to sample area at PE sheath outer surface • Longitudinal cut of PE sheath • Wrap PE sheath in clean plastic bag. Seal close with tape. • Inspect lead layer • Wrap remaining sample length in plastic. • Store parts in pallet.	• Document by photo PE-sheath • Photo of longitudinal cut PE sheath • Photo lead layer

2.3.8 Sample No. 8

Table 2-9 Dissection layer-by-layer Sample No. 8. Action marked in Green are witnessing points.

Activity	Action	Observation/ Reporting
Marking	• Mark pallet with sample number 2 • Mark plastic bag for PE-sheath, No. 2	
Cut-to-length	• 1x Cross-sectional cut – sample length 1m. • Wrap exposed surfaces in plastic and seal by tape.	• Overview photo of received sample • Cross section photo

Dissection	• Longitudinal cut through Corrosion protection layer/ outer cover (PP Yarns) • Remove outer armouring layer • Remove bedding • Remove inner armouring layer • Remove Bedding • Remove steel tapes • Inspect PE-sheath • Clean PE sheath with ethanol • Shave outer sheath surface samples store in marked zip lock bag. • Longitudinal cut of PE sheath • Wrap PE sheath in clean plastic bag. Seal close with tape. • Inspect lead layer • Wrap remaining sample length in plastic. • Store parts in pallet.	• Document by photo PE-sheath • Photo of longitudinal cut PE sheath • Photo lead layer

2.4 Sectioning of PE-sheath into sizes for further testing

Larger sample sections for chemical analysis will be sectioned out in sizes as shown in Table 2-10. The samples will be extracted from PE-sheath by band saw and then placed in clean plastic zip-lock bags, bags marked with sample number. Outer sheath surface is marked by masking tape and pen marker.

Table 2-10 Number of sections and size to be taken out from each sample length for further analysis. Thickness is given by sample thickness (approx. 3-6mm).

Chemical analysis method	Number of samples	Sample size [mm] (Length x Width)	Comment
SEM	1	Approx. 30x10	Sample size depending on PE-sheath thickness
ICP-SFMS	2*	15x45 (3x) 50x50 (spare sample)	

*One (1x) sample as spare

All cuts will be made from outside towards inner side. **Cut surfaces will be treated as contaminated after this cutting and further cutting/ grinding is needed before chemical analysis.** Further handling of samples is described in Section 3.

3 CHEMICAL ANALYSIS FOR LEAD PENETRATION

The longitudinal section of PE-sheath will be further sectioned to take out samples for SEM/ EDS and ICP-SFMS method of testing. General handling of samples will be covered in own section 3.1.

3.1 Sample extraction and handling for further testing

It will be taken out 2x samples for ICP testing and shavings from outer sheath surface, in addition 1x sample for SEM examination. Handling steps of the samples are illustrated in figure below:

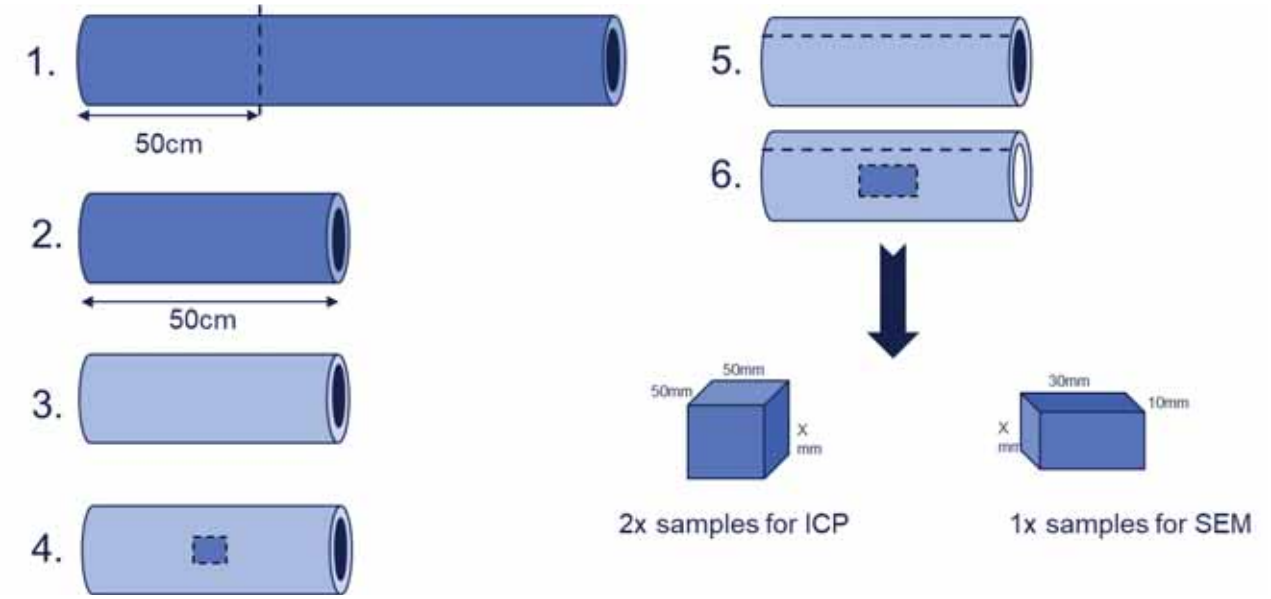

Figure 3-1 Steps of handling until sample extraction (thickness varies from approx. 3 to 6 mm)

1. Cut to length
2. Remove outer layers until PE-sheath position outside lead layer.
3. Inspect and clean PE-sheath
4. Shave PE-sheath samples – Store in Zip-lock plastic bag and mark with sample no. Outer surface of samples for ICP testing will be masked with tape.
5. Longitudinal cut through PE-sheath – Remove from Lead layer
6. Split into two half shell and clean inner surface towards lead with rag and ethanol. Cut out samples with band saw for further testing.
7. Place samples for ICP testing on plastic covered tray for further grinding.

3.2 SEM/ EDS Analysis

Sample for SEM extracted from PE sheath is shown in Figure 3-2.

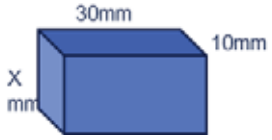

Figure 3-2 Illustration SEM sample cut from PE-sheath

The cross-sectional sample surface to be examined will be ground using silica paper at grit 320 and 1000, using a rotating turn table with water applied. The sample will be oriented with outer surface facing towards rotating direction and at <90°. The last grinding step will be with outer surface facing the rotating direction, to avoid lead cross contamination from inside surface to outside surface. A new silica paper to be used at final stage for each sample number, as illustrated in Figure 3-3.

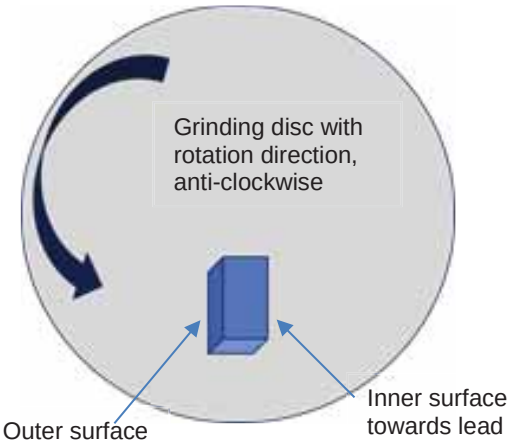

Figure 3-3 Illustration of grinding step(s) in order to "mirror" finish and remove eventual residues from band saw cutting.

The samples will then be rinsed with water and ethanol.

Prior to SEM examination the samples will be sputtered with gold in order to get good conductivity and avoid charging when analyzing for lead (Pb).

SEM/ EDS analysis will be presented with EDS spectra.

This analysis is aimed to be qualitative to identify presence of lead or not. One sample will be examined to check for lead over thickness.

3.3 Microtome cutting and ICP-SFMS method

The analysis will be carried out by sub-supplier and a work description will be issued to the sub-supplier. ICP-SFMS method will be carried out according to standard DIN EN ISO 17294-2.

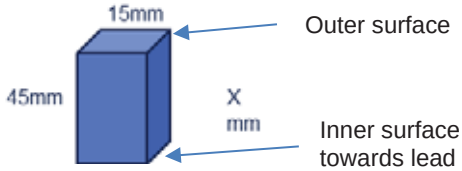

Figure 3-4 Sample first sectioned to 50x50x thickness, then cut down to size to width of approximately 15 mm for further microtome cutting.

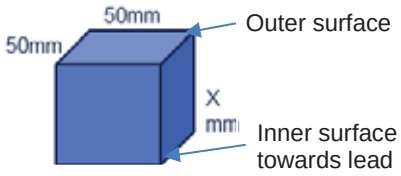

Figure 3-5 Illustration of spare samples.

All cut surfaces will be grinded to “mirror finish” according to same procedure as for SEM sample, as shown in Figure 3-3. The outer surfaces will be masked with masking tape, sample will be packed in plastic zip lock bag and marked with sample number and inner surface towards lead. The samples will be clued to a support piece before microtome cutting. The samples to be taken from outer surface and mid-tickness will be extracted from one sample (A in image below) and the inner surface sample (facing the lead) will be extracted from another sample (B in image below). Image showing sample 5 ready for microtome cutting.

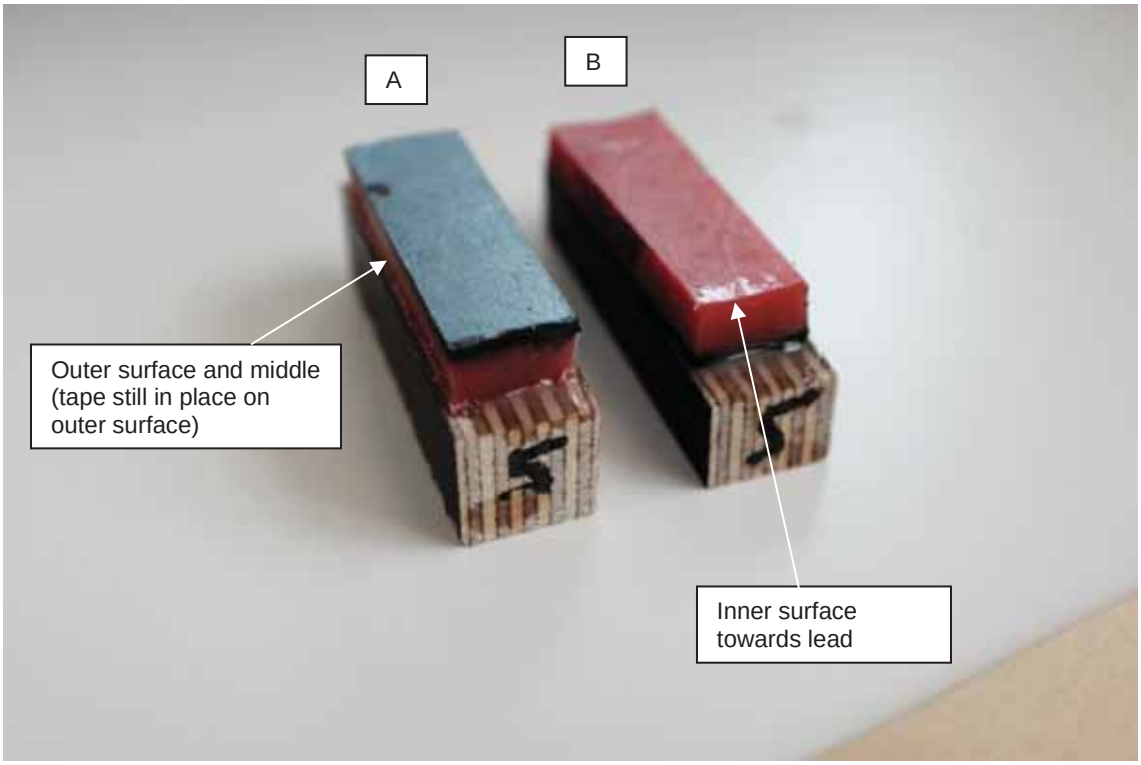

Figure 3-6 Example image of sample No. 5 prepared for microtome cutting

Microtome cutting will be done in minimum 3 steps as illustrated in Figure 3-4.

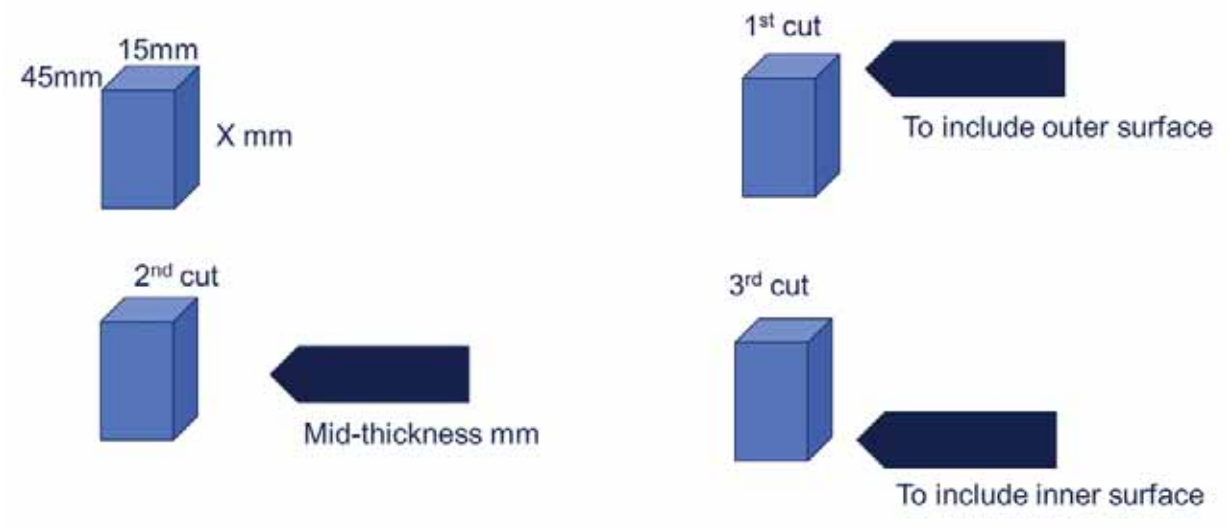

Figure 3-7 Illustration of microtome cutting (sample thickness varies approx. 3 – 6 mm)

Note! The inner surface has been in contact with lead and cross-contamination from sample handling and cutting should be avoided between the individual cuts.

1) Install New cutting blade pr. sample
2) Remove masking tape
3) Zero stage position
4) First cut to be made at outer surface sample to be bagged in a marked paper bag note down depth where layers are taken from – change cut position on knife blade
5) Second cut at mid-thickness sample to be bagged in a marked paper bag note down depth where layers are taken from - change cut position on knife blade
6) Change sample for Inner surface towards lead
7) Third cut at inner surface sample to be bagged in a marked paper bag note down depth where layers are taken from – change cut position on knife blade
8) Repeat step 1 to 6 for each sample

Images illustrating the microtome cutting is shown in Figure 3-8 to Figure 3-11.

The samples from microtome cutting to be marked as follow:

- Sample No. – Microtome cut position (O, M or I)

O = Outer

M = Middle

I = Inner

Figure 3-8 Stage position (encircled) to be zeroed before cutting, layer depth sampled to be noted on paper bag.

Figure 3-9 Example image of marked paper bags for sample no. 2

Figure 3-10 Overview image of microtome cutting machine

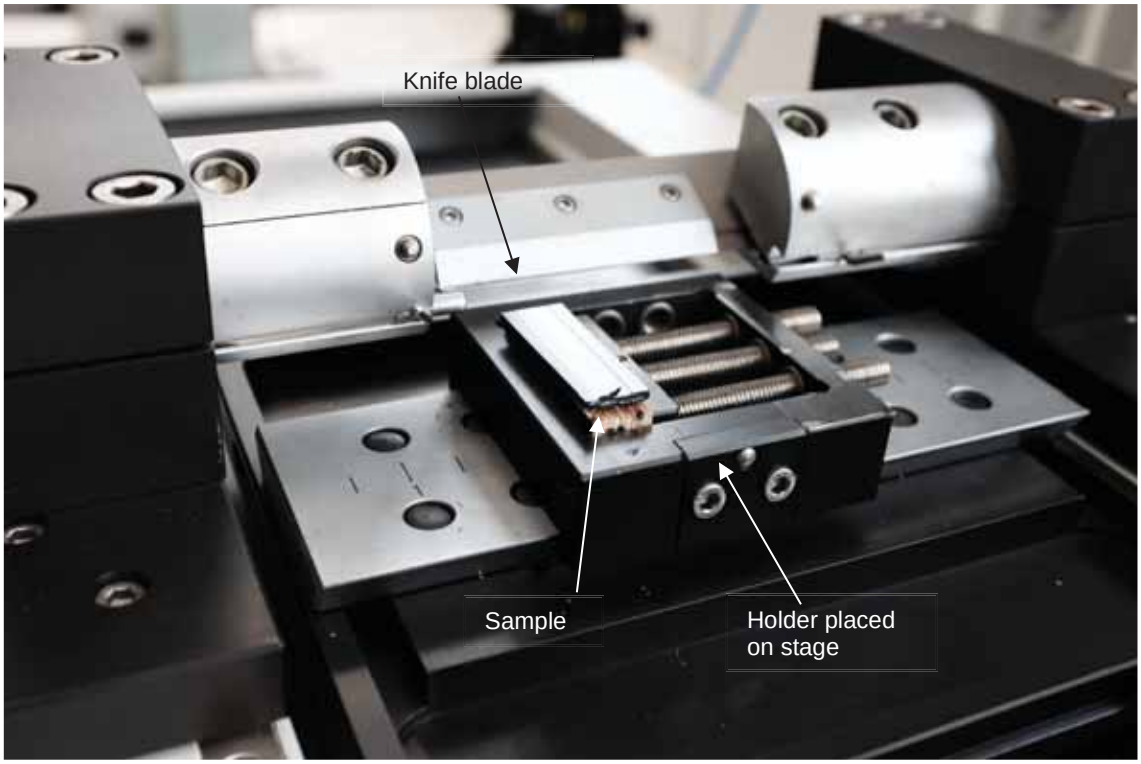

Figure 3-11 Stage with holder and cutting blade to microtome cutting machine. Blade will be changed for each sample No. Position on blade changed for the different positions, inner sample was cut last.

Test matrix for ICP testing example is shown in Table 3-1.

Table 3-1 ICP Test matrix example

Sample No.	Test samples ICP	Sample Marking
1	Shavings outer surface	1-S
	Outer surface cut	1-O
	Mid thickness cut	1-M
	Inner surface cut	1-I
2	Shavings outer surface	2-S
	Outer surface cut	2-O
	Mid thickness cut	2-M
	Inner surface cut	2-I

The bags and marking on bags of samples for testing are shown in Figure 3-12.

Figure 3-12 Samples for ICP testing. Paper bags are samples from microtome cutting and samples in plastic bag is shavings taken during dissection.

4 REPORTING

All the results will be presented in a Technical Report including photo documentation. The report shall content at least the following items:

a. Why are these analyses performed?

b. What is the background and context?

c. How are performed analyses (identification of samples, method and apparatus used, illustrations)?

d. Who did perform analyses?

e. Equipment calibration certificates,

f. Analysis of measurements,

g. Potential source of errors, incl. detection limit

h. Relation between age, type of cable and results,

i. Conclusion

APPENDIX B

Microtome cutting weight of samples extracted and depth of layers included for the different samples

Sample No.	Test samples ICP	Sample Marking	Thickness [mm]	Depth microtome layers [mm]	Weight [mg]
1	Outer surface cut	1-O	3.1	0-0.8	350
	Mid thickness cut	1-M		0.96-1.44	286
	Inner surface cut	1-I		0-1.44	245
2	Outer surface cut	2-O	3.9	0-0.8	300
	Mid thickness cut	2-M		0.96-1.41	280
	Inner surface cut	2-I		0-1.27	430
3	Outer surface cut	3-O	5.4-5.9	0-0.82	390
	Mid thickness cut	3-M		1.00-1.39	300
	Inner surface cut	3-I		0-1.14	450
4	Outer surface cut	4-O	3.5-3.7	0-0.81	300
	Mid thickness cut	4-M		1.05-1.50	250
	Inner surface cut	4-I		0-1.31	400
5	Outer surface cut	5-O	7.2-7.4	0-0.80	400
	Mid thickness cut	5-M		1.40-2.00	325
	Inner surface cut	5-I		0-1.10	380
6	Outer surface cut	6-O	3.7-3.8	0-0.75	250
	Mid thickness cut	6-M		1.40-2.00	325
	Inner surface cut	6-I		0-1.10	380
8	Outer surface cut	8-O	3.8-3.9	0-0.95	350
	Mid thickness cut	8-M		1.50-2.00	325
	Inner surface cut	8-I		0-1.10	250

For outer surface cut and mid thickness cut the depth is measured from outer surface. Inner surface cut depth is measured from inner surface facing the lead.

APPENDIX C
Sintef Norlab Test Report

Appendix C - Comments Sintef Norlab test report in English - DNV

The samples were prepared after microwave assisted decomposition in a closed vessel based on Milestone application recommendations. The PE-based sample materials are analysed with SF ICP-MS technique, after an internal method based on EN ISO 17294-2 and EPA 200.8.
The samples were totally decomposed. The instrumental readings were unambiguously in low, medium, and high resolution for each sample and a significant difference between Pb-isotopes were observed. There are high differences in Pb-concentrations between the samples, ranging from below LOQ to %-level. The limit of quantification, LOQ is set to 0.2 mg Pb/kg in polymer material samples. A study of the isotopes 206Pb, 207Pb and 208Pb, showed that no significant interference is observed. Hence, it is concluded that the method is suited for the purpose. A full validation of the method requires relevant reference material and Pb-analyte control over a wide lead concentration range. Reference material with known Pb content is not been available so the accuracy and uncertainty of the measurements are not covered by laboratory certification.

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DNV GL AS
v/ Jan Henrik Olsen / Anne Kirsti Noren Egtvedt

RAPPORT

Analyse av Pb i polymermateriale fra kabler

Vårt ordrenummer: 115181 | **Rev.nr.** 0

Ordre mottatt dato: 19.04.2022 | **Dato rapportert:** 22.04.2022

Kundens bestillingsnr./ref: Jan Henrik Olsen

SINTEF Norlab signatur: Robert Gibala

PRØVEINFORMASJON

Kundens prøvemerking	Vår prøve ID	Parameter/Problemstilling
1-S	115181-001	Bestemmelse av Bly (Pb)
1-O	115181-002	Bestemmelse av Bly (Pb)
1-M	115181-003	Bestemmelse av Bly (Pb)
1-I	115181-004	Bestemmelse av Bly (Pb)
2-S	115181-005	Bestemmelse av Bly (Pb)
2-O	115181-006	Bestemmelse av Bly (Pb)
2-M	115181-007	Bestemmelse av Bly (Pb)
2-I	115181-008	Bestemmelse av Bly (Pb)
3-S	115181-009	Bestemmelse av Bly (Pb)
3-O	115181-010	Bestemmelse av Bly (Pb)
3-M	115181-011	Bestemmelse av Bly (Pb)
3-I	115181-012	Bestemmelse av Bly (Pb)
4-S	115181-013	Bestemmelse av Bly (Pb)
4-O	115181-014	Bestemmelse av Bly (Pb)
4-M	115181-015	Bestemmelse av Bly (Pb)
4-I	115181-016	Bestemmelse av Bly (Pb)
5-S	115181-017	Bestemmelse av Bly (Pb)
5-O	115181-018	Bestemmelse av Bly (Pb)
5-M	115181-019	Bestemmelse av Bly (Pb)
5-I	115181-020	Bestemmelse av Bly (Pb)
6-S	115181-021	Bestemmelse av Bly (Pb)
6-O	115181-022	Bestemmelse av Bly (Pb)
6-M	115181-023	Bestemmelse av Bly (Pb)
6-I	115181-024	Bestemmelse av Bly (Pb)
8-S	115181-025	Bestemmelse av Bly (Pb)
8-O	115181-026	Bestemmelse av Bly (Pb)
8-M	115181-027	Bestemmelse av Bly (Pb)
8-I	115181-028	Bestemmelse av Bly (Pb)

Prøvematerialet forelå klart for analytisk arbeid i laboratoriet 19-20.04.2022. Prøvematerialet består av to typer.

1. «Shavings», mottatt fra oppdragsgiver DNV direkte i zip-poser.
2. Mikrotomert materiale levert av DNV til Norner as som er lagt i små papirposer for å begrense statisk elektrisitet.

METODIKK

Uttak fra hver prøve ble veid inn og totalt dekomponert til en klar løsning vha. av en syreblanding bestående av HNO_3 og H_2O_2 i lukket system ved hjelp av et program i en mikrobølgeovn, Milestone Ethos. Prøvene ble fortynnet til ett gitt volum, og ytterligere fortynnet før avlesning på en SF-ICP-MS. Det er benyttet en intern metode basert på beskrivelse av måleprinsippet i EN ISO 17294-2 og EPA 200.8.

RESULTATER

Analyseresultater er gitt i tabell 1

Tabell 1: Resultater fra bestemmelse av Pb

Vår prøve ID	Kundens prøve ID	Benevning	Pb
115181-001	1-S	mg/Kg	0,3
115181-002	1-O	mg/Kg	0,3
115181-003	1-M	mg/Kg	<0,2
115181-004	1-I	mg/Kg	0,5
115181-005	2-S	mg/Kg	0,2
115181-006	2-O	mg/Kg	<0,2
115181-007	2-M	mg/Kg	<0,2
115181-008	2-I	mg/Kg	37
115181-009	3-S	mg/Kg	18000
115181-010	3-O	mg/Kg	22000
115181-011	3-M	mg/Kg	28000
115181-012	3-I	mg/Kg	27000
115181-013	4-S	mg/Kg	0,2
115181-014	4-O	mg/Kg	0,5
115181-015	4-M	mg/Kg	0,2
115181-016	4-I	mg/Kg	0,7
115181-017	5-S	mg/Kg	0,2
115181-018	5-O	mg/Kg	<0,2
115181-019	5-M	mg/Kg	0,2
115181-020	5-I	mg/Kg	0,3
115181-021	6-S	mg/Kg	<0,2
115181-022	6-O	mg/Kg	0,7
115181-023	6-M	mg/Kg	0,3
115181-024	6-I	mg/Kg	28
115181-025	8-S	mg/Kg	0,8
115181-026	8-O	mg/Kg	0,2
115181-027	8-M	mg/Kg	0,3
115181-028	8-I	mg/Kg	640

KOMMENTARER

Prøvene ble analysert for Pb etter kundens ønske. Prøvene ble løst i en syreblanding bestående av HNO_3 og H_2O_2 mikrobølgeassistert dekomponering. Alle prøvene ble total dekomponert.

Analyttene ble bestemt vha. SF-ICP-MS, mot intern og ekstern standardisering.
Massene Pb206, Pb207 og Pb208 ble benyttet. Siden bestemmelsen av Pb i miljøsammenheng, gjerne krever at Pb bestemmes med isoptopene Pb206, Pb207 og Pb208 (sum, snitt), så kan det være mulighet for interferenser fra komplekse matrikser som kan påvirke resultatene negativt. Det ble ikke observert noe av dette for disse prøvene. Mulighetene for interferenser er imidlertid ikke studert inngående, siden dette kan være en omfattende jobb. Men det har blitt lagt ned en innsats med å fjerne så mange interferenser som mulig, innenfor oppdragets rammer. Dette involverer blant annet å bestemme analyttene med elementenes forskjellige isotoper der det er mulig (noe som er nødvendig.), samt å utnytte instrumentets oppløsningsevne for å skille analyttene ifra eventuelle interferenser der det er nødvendig og hensiktsmessig.
Metoden som ble benyttet, er ikke validert, eller akkreditert – så nøyaktighet og presisjon er ikke kjent.

APPENDIX D
Accreditation of sub-supplier

NORWEGIAN ACCREDITATION

Accreditation scope for

TEST 032

SINTEF Norlab AS

NO-8607 Mo i Rana

Telephone: +47 404 84 100
E-mail: info@sintefnorlab.no
Internet: http://www.sintefnorlab.no

The laboratory meets the requirements in
NS-EN ISO/IEC 17025:2017

Accreditation was first granted: **07/03/1995**

Accreditation requires regular follow-up, and is valid to: **30/08/2022**

P19 Sensorics

Object	Parameter	Reference standard	Internal method identity	Comment
Indoor air and outdoor air	Determination of odour concentration	EN 13725	D01436	Dynamic olfactometry

P31 Flexible accreditation

Object	Parameter	Reference standard	Internal method identity	Comment
Parameter, object, reference standard	Organic analysis	Internal method	D01666	An updated list of methods included in the flexible scope of accreditation, is available with the organization.

Field sampling

P3002 Air sampling

Object	Parameter	Reference standard	Internal method identity	Comment
Indoor air and outdoor air	Determination of odour cocentration	Internal method	D01435	Method based on NS-EN 13725:2003 and VDI 3880 (Norwegian environment agency, TA-3019)

The administrative / geographical unit:

SINTEF Norlab AS, Porsgrunn
Herøya Forskningspark, Hydroveien 67
3905 Porsgrunn

Permanent facility

P12 Chemical analysis

Object	Parameter	Reference standard	Internal method identity	Comment
Absorption solutions	As, Cd, Cr, Co, Cu, Mn, Mo, Ni, Pb, Sn, Sb, TI, V and Zn	Internal method	D01514	ICP-MS Internal methode based on NS-EN 14385
Produced water / saline water	Ethane acid, propanoic acid, methane acid, butyric acid, pentanoic acid, hexanoic acid	Internal method	D01950	Ion chromatography
Produced water/Saline water	Cd, Co, Cr, Cu, Ni, Pb, Zn, V, As, Fe, Ba and Mn	Internal method	D01943	ICP-MS Internal method based on EPA Method 200.8
Produced water/Saline water	Fe, Ba and Mn	Internal method	D01923	ICP-OES Internal method based on EPA Method 200.
Produced water/Saline water	Hg	Internal method	D01936	CV-AFS Internal method based on NS-EN ISO 17852:200
Nitric acid extracts from solid materials and nitric acid solutions	Cd, Cu, Ni, Pb, Zn, As, Cr	Internal method	D02191	ICP-MS Based on EPA method 200.7
Nitric acid extracts from solid materials and nitric acid solutions	Hg	Internal method	D02527	CV-AFS Method based on NS 4770:1994 and NS-EN ISO 17852:2008
Probe Wash Solution	As, Cd, Cr, Co, Cu, Mn, Mo, Ni, Pb, Sb, Sn, Ti, V and Zn	Internal method	D01514	ICP-MS Internal methodebased on NS-EN 14385
Probe Wash Solution	Hg	NS-EN 13211	D01513	CV-AFS
Dust on quartz fiber filter	Hg	NS-EN 13211	D01513	CV-AFS
Stationary source emissions	As, Cd, Cr, Co, Cu, Mn, Mo, Ni, Pb, Sn, Sb, TI, V and Zn	Internal method	D01514	ICP-MS Internal methode based on NS-EN 14385
Dust on quartsfiberfilter, sonic wash solution, absorption solution	Cu, Mn, Ni, Pb, Zn	Internal method	D01647	ICP-OES Method based on NS-EN 14385

P31 Flexible accreditation

Object	Parameter	Reference standard	Internal method identity	Comment
Changing parameters, object, reference standard and not permanent changes	Inorganic analysis	Internal method	D01666	An updated list of methods included in the flexible scope of accreditation, is available with the organization.


DNV

About DNV

DNV is the independent expert in risk management and assurance, operating in more than 100 countries. Through its broad experience and deep expertise DNV advances safety and sustainable performance, sets industry benchmarks, and inspires and invents solutions.

Whether assessing a new ship design, optimizing the performance of a wind farm, analyzing sensor data from a gas pipeline or certifying a food company's supply chain, DNV enables its customers and their stakeholders to make critical decisions with confidence.

Driven by its purpose, to safeguard life, property, and the environment, DNV helps tackle the challenges and global transformations facing its customers and the world today and is a trusted voice for many of the world's most successful and forward-thinking companies.