RESEARCH REPORT

VTT-R-00094-22

Joint sealants and their ageing in nuclear power plants

Authors: Konsta Sipilä

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Summary

Joint sealants are polymer-based sealing solutions that are applied in between structures, such as floor and wall elements, thus joining them together and forming a gas-tight structure. Such sealants are widely applied in constructions, including nuclear power plants (NPPs). However, their ageing in the nuclear power plant environment is a less studied phenomenon. A wide range of polymers, such as acrylic, silicone, polybutene, latex, butyl, polysulphide and polyurethane, can be used as a base polymer in joint sealants. Each type of joint sealant will have slightly different properties and targets of application. The ageing stressors that the joint sealants experience in NPPs are heat, moisture and radiation. It seems that oxidation induced by thermal ageing and loss of plasticizers are the most apparent ageing mechanisms for joint sealants in an NPP environment. Swelling and leaching become relevant if there is exposure to excess moisture or water. Various methods were listed for condition monitoring of joint sealants, and it seems that the most applicable ones include hardness measurement, differential scanning calorimetry (DSC) and Fourier transform infrared spectroscopy (FTIR). Joint sealant samples from Olkiluoto 1 and 2 units were obtained with additional information on their tradename and service environment. The proposed analysis methods are planned to be applied to these materials as part of future work.

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Written by	Reviewed by
Konsta Sipilä Research Scientist	Timo Saario Principal Scientist

VTT's contact address
P.O.Box 1000, FI-02044 VTT

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Approval

Date:	
Signature:	
Name:	Mikko Vepsäläinen
Title:	Research Team Leader

Preface

This work was completed as part of the Finnish Research Programme on Nuclear Power Plant Safety 2019 – 2022 (SAFIR2022) within the SAMPO (Safety criteria and improved ageing management research for polymer components exposed to thermal-radiative environments) project's work package 2 "Improvements in ageing management of polymer components".

Espoo 31.1.2022~~28.1.2022~~
Author

Contents

1. Introduction

Sealing solutions are an integral part of each building. The components (e.g. concrete blocks, wall and floor elements, glass structures etc.) of which buildings are constructed are often bonded or sealed to each other by joint sealants. Joint sealants are defined here as polymer-based materials that are applied in a groove between two structures and join tightly these components to each other preventing moisture or gases from penetrating through the sealed area. They are commonly used in various types of structures due to their ease of use, wide range of properties and applicability. An example of applying a joint sealant between two concrete elements is shown in Figure 1. Such joint sealants are widely applied in nuclear power plants but there is very little information available on their ageing and how to monitor their condition in the nuclear power plant environment.

Figure 1. Application of a joint sealant between two concrete blocks. Picture was taken from [1].

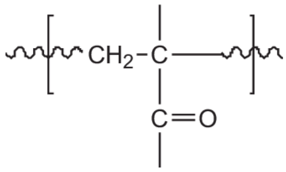
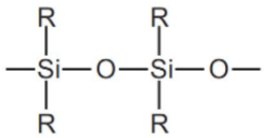
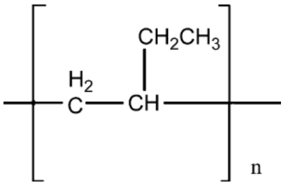
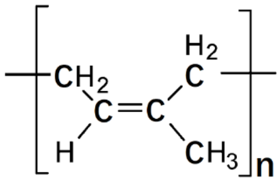
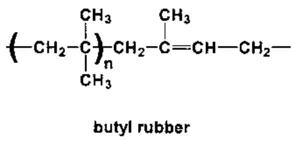
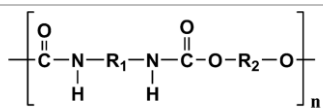
This report focuses on listing different types of joint sealants that are generally used in constructions. Typical ageing stressors and mechanisms are gathered from the open literature for these materials. Finally, samples obtained from Olkiluoto NPP are listed and suitable tests to be performed on these materials are discussed.

2. Different types of joint sealants used in buildings

In the classification of different types of joint sealants, several approaches can be used. Joint sealants can be classified based on the material type, elasticity or form. In this context classification based on the material will be used.

Before 1950 traditional caulks, based on either oleoresins (e.g. linseed oil), bitumen or tar were used in joints. By the 1970's the industrially manufactured polymer-based materials had replaced the traditional caulks due to their improved material properties. Currently, a broad range of different polymer-based materials is applied in joint sealants. These materials and their common properties and applications are summarized in Table 1.

There are some differences between the different polymers. They may have limitations regarding the environment, e.g. regarding temperature, or they may display different type of material properties, e.g. elasticity.

Sealant material	Application	General properties	Molecular structure	Reference
Acrylic	Wide range of building materials	Retains pliability, cannot be applied at low temperatures, good water resistance		[2, 3]
Silicone	Household use (kitchen and bathroom) and construction use (wood, stone, metal bricks)	Flexible, retains pliability, sticks to painted surface but cannot be overpainted		[3, 4]
Polybutene	Wide range of temperatures (e.g. road construction products)	Low cost, excellent durability and water resistance, good package stability and adhesion, will withstand cyclic joint movement, can stain adjacent surfaces and relatively poor recovery from extension		[5, 6, 7]
Latex	Bathroom joints, cracks in plaster, tile, glass and plastic.	Easy to use, water resistant, not very flexible		[8, 9]
Butyl	Wide range of building and dissimilar materials	Good water resistance, very durable, poor adhesion to painted surfaces		[3, 10]
Polysulphide	Basements, glazing frames, ceiling joints, floors, roofs, external walls, cladding, retaining walls, water retaining structures, joints in bridges, roads and aerodomes	Good water and chemical resistance	-S-S- (polymer containing a chain of sulfide atoms)	[11]
Polyurethane	Wide range of building materials, wider and irregular cracks	Retains pliability, good water resistance, overpainting recommended on foam (susceptible to UV-light)		[3, 12]

3. Ageing of joint sealants

3.1 Environmental stressors

The ageing mechanisms of these joint sealants have been previously studied by Burström [13]. Weather-related changes, such as exposure to rain, ultraviolet radiation from the sun and temperature changes during the seasons, are more extreme in outdoor than indoor constructions. These effects are thus secondary when stressors present indoors are considered.

Stressors for indoor applications are somewhat similar to those faced in outdoor applications, but obviously, the environment is more stable. In a nuclear facility stressors present include temperature, radiation and moisture. The temperature during normal use inside containment can vary between different plants. One estimate is that the temperature inside containment would not increase above 50°C at any point [14]. Data obtained from TVO indicated that the joint sealants in Olkiluoto units would be exposed to slightly above room temperature, but still below 30°C. No heavy cycling of temperature is expected to be present during normal operation. The absence of temperature cycles would have an effect on the thermal expansion of structural elements and thus result in absence of the cyclic mechanical loading of the sealants.

Similarly to temperature, radiation dose rates inside containments can be evaluated plant specifically. Maximum dose rates during a normal operation inside containments are locally less than 1 Gy/h. Joint sealant materials are not typically located in the vicinity of these radiation hot spots, which is indicated also by the dose rate data obtained from TVO regarding the joint sealant samples, where the maximum dose rate was estimated to be 10^{-5} Gy/h. This would mean an about 0,4 Gy total annual dose and after several decades of operation, the radiation dose levels remain relatively low, less than 20 Gy. This would indicate that the irradiation would not be a significant stressor for these specific sealants, since typically tens or hundreds of kGys are required in order to initiate radiation damage to polymers [14].

The effect of water on joint sealants can be thought to be due to moisture and humidity. Moisture can be defined as water or liquid containing traces of water being in contact with a surface while humidity is water vapor. Not much data is available on the moisture levels at the plants, one estimate is that the relative humidity during normal operation is less than 90% [14].

It seems that oxidation and temperature would be the most significant stressors for joint sealants used inside nuclear power plants.

3.2 Ageing mechanisms

3.2.1 Oxidation

The oxidation of a polymer is a series of molecular level reactions that end up scission or cross-link the copolymer chains, yielding in the degradation of macroscopic material properties. This is frequently referred to as auto-oxidation reaction [15] and is illustrated in Figure 2. In a containment environment, heat and radiation are present which interact with the polymer material generating molecules with an unpaired electron, i.e. radicals ($R^{\cdot}$). Oxygen reacts with the radicals forming peroxy radicals ($ROO^{\cdot}$). These peroxy radicals can abstract hydrogen species from the surrounding molecules (RH) forming hydroperoxide (ROOH), which is further decomposed to non-radical species or additional radicals.

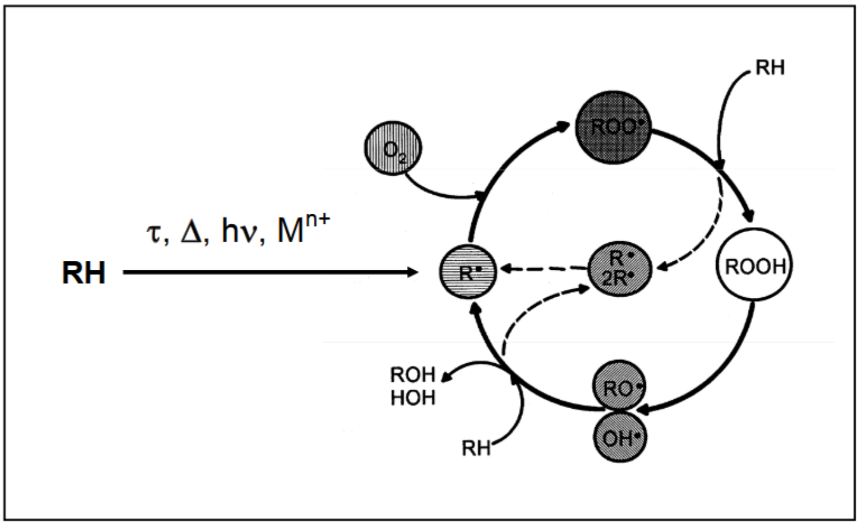


Figure 2. Schematic illustration of the auto-oxidation process. [15]

The oxidation of the polymer may cause material changes visible on the component scale. Decrease in strength and elongation are commonly observed as a result of oxidation as well as colour and weight changes.

3.2.2 Evaporation of plasticisers

Plasticizers are added to polymers during their manufacture to improve processability and optimize their material properties by affecting the interactions between the polymer chains and improving their mobility. A broad range of properties can be affected by plasticizers as can be seen from Figure 3. Adjusting these properties is essential when manufacturing otherwise rigid polymers, such as PVC. A large part of the plasticizer consumption is by the PVC industry. Plasticizers can be divided into phthalates and non-phthalates. Di-isodecyl- and di-n-buthyl phthalates, adiapates, phosphates and polyesters are applied on other than PVC materials.

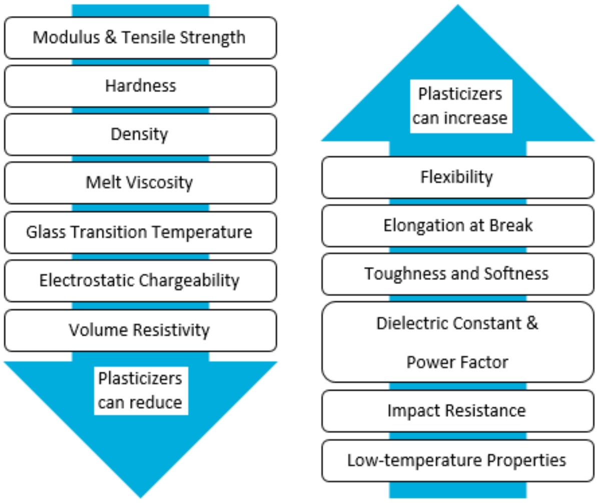


Figure 3. The effect of plasticizers on material properties. Picture was taken from [16].

The evaporation of plasticizers can be thought to comprise of two phases. First, the plasticizer migrates to the surfaces and is followed by evaporation. The evaporation may be thus diffusion- or evaporation-controlled, the latter at higher temperatures and former at room temperatures. Several factors affect this phenomenon, including the polymer−plasticiser interactions, plasticiser concentration and gradient within the material, temperature, plasticiser features (size, shape, polarity, vapour pressure), flow rate of the gas above the surface and the volume of the atmosphere around the material. [17]

3.2.3 Swelling and leaching

Water has a very complicated effect on ageing and it is often combined with other factors, such as temperature and radiation. Generally, it is considered that water has an accelerating effect. This is thought to be mostly due to increased mobility of the reaction products and an increased rate of dissociation. [13]

Some of the polymers may contain parts that are decomposed by water, i.e. hydrolysable groups. When these hydrolysable groups are part of the polymer main chain, the reaction with water may cause chain scission and a decrease in strength.

Swelling and leaching are ageing phenomena related to water ingress. Hygroscopic materials tend to absorb moisture from the air. This absorption of water causes swelling in the polymer. Water may reversibly increase the plasticity of some polymer types (e.g. urethane rubber and ester-type). These polymers regain their properties after drying.

Some of the polymer ingredients may be water-soluble and thus susceptible to leaching. The leaching of antioxidants and light stabilizers has a negative effect on polymer ageing resistance. On the other hand, some of the radicals or catalysts for oxidation may be removed by leaching, thus being beneficial from the ageing point of view.

3.3 Testing methods

3.3.1 Tensile test

Tensile testing is a well-established destructive methodology where a sample is pulled until rupture with a constant strain rate and the elongation and force is measured during the procedure. From the resulting data stress-strain behaviour of the material can be analysed which is convenient in ageing studies as the mechanical properties, such as elongation, are heavily influenced by ageing. However, the tensile test requires sample removal and from a sealant joint, sample removal for a tensile specimen is in practice impossible due to the narrow dimensions of such joints. Using tensile tests in condition monitoring of joint sealants would require surveillance samples.

3.3.2 Hardness

Hardness is also a well-established measurement where a force is applied on a surface and the depth of indentation is measured. It can be considered to be non-destructive if the indentation is not too severe. Hardness has been shown to correlate well with elongation at break values [18], making it a quite convenient method for the non-destructive evaluation of polymers. The measurement is sensitive to several factors such as temperature, clamping of the measurement device around the sealant, applied force and intender tip.

3.3.3 Density

Ageing increases the amount of oxidation products in the polymer which tends to increase the density of the material. The evaporation of volatile species from the polymer causes changes in weight, dimensions and density. The method requires sample removal but the amount is relatively small.

3.3.4 Thermogravimetric analysis

In thermogravimetric analysis (TGA) the sample is heated in a pre-defined atmosphere and the weight of the remaining ash is measured. TGA requires a small amount of sampling. The method is applied to the analysis of volatile compounds of coatings (ASTM D2369-20). Thus, it would be a potential method to be used in the evaluation of joint sealants as well.

3.3.5 Fourier transform infrared spectroscopy

In Fourier transform infrared spectroscopy (FTIR) infrared wavelength radiation is directed to the sample and the absorption of the radiation is measured. The oxidation products have characteristic wavelengths at which they absorb infrared radiation enabling thus identification of these products. The method requires sample removal.

4. Materials obtained from TVO outage

During the Olkiluoto 1 and 2 2021 outages, several joint sealant materials were obtained from various structures. A summary of the obtained materials is presented in Table 1~~Table 2~~. Generally, two types of sealant materials were in use, tradenames Thiostät and Sika. Most of the samples were from the original installations, thus having been in use for four decades. The environmental parameters have not been too severe. The ageing has occurred in the air atmosphere and at temperatures below 30°C. Some of the samples have been exposed to low dose rate radiation. However, the total absorbed doses for the samples are low despite the long exposure time. It is estimated that the maximum annual dose that the samples have obtained is up to 0.4 Gy. Thus, it can be stated that the irradiation-induced ageing of the components has been negligible compared to thermal ageing.

A bit more detailed data on the sample condition is attached in Annex 1. Some of the sealants were applied on a polyethylene ribbon, which is a typical solution in some structures. Some of the sealants were reported to contain fractures or have sticky surfaces, both being signs of ageing. The formation of cracks in such old and aged sealant may well be due to the migration of plasticizers leaving the sealant brittle and susceptible to cracking. The formation of sticky surfaces would indicate degradation of the polymer by chain scission of the crosslinking bonds.

Table 1~~2~~. Summary of the joint sealant materials obtained from Olkiluoto 1 and 2 outages in 2021.

Sample number	Sealant info	Age	Temperature / °C	Dose rate	Sample dimensions / mm
1.1	Thiotät	Original	25,8	-	170
1.2	Sika?	-	25,8	-	140
1.3	Thiotät	Original	28,4	-	-
1.4	Thiotät	Original	27,2	-	-
1.5	Thiotät+Sika?	-	22,3	-	-
1.6	Sika?	-	22,3	-	-
1.7	Sika?	-	25,7	-	-
1.8	Sika?	-	25,7	-	170
1.9	Thiotät	Original	25,6	-	170
1.10	Thiotät	Original	26,5	-	170
1.11	Thiotät	Original	26,5	-	150
1.12	Thiotät	Original	26,2	-	130
1.13	Thiotät	Original	26,5	-	-
1.14	Sika?	-	26,5	-	100
1.15	Sika? + Thiotät	-	26,5	-	300
2,1	Thiotät	Original	24,5	-	240
2,2	Sika?	-	24,5	-	220
2,3	Thiotät	Original	27,8	-	93
2,4	Sika?	-	27,8	-	130
2,5	Thiotät	Original	28,1	-	100
2,6	Sika?	-	28,1	-	130
2,7	Thiotät+ sika?	-	26,4	-	110
2,8	Thiotät	Original	26,4	-	100
2,9	Thiotät	Original	27,5	-	110
2,10	Thiotät	Original	27,5	-	100
2,11	Thiotät	Original	27,9	-	-
2,12	Thiotät	Original	27,9	-	100
2,13	Thiotät	Original	27,8	-	130
2,14	Thiotät	Original	27,8	-	130
2,16	Sika?	-	27,1	-	-
2,17	Thiotät	Original	-	0.0250 mSv/h	100
2,18	Thiotät	Original	-	0.0250 mSv/h	120
2,19	Thiotät	Original	27,1	0.0200 mSv/h	100
2,20	Thiotät	Original	27,1	0.0200 mSv/h	100
2,21	Thiotät	Original	28,1	0.0020 mSv/h	110
2,22	Thiotät	Original	28,1	0.0020 mSv/h	115

2,23	Thiotät	Original	29,5	0.0500 mSv/h	-

5. Future work

The next steps for the joint sealant study would include:

- Evaluating the applicability of the presented testing methods for joint sealants obtained from Olkiluoto units
- Evaluating the condition of the most interesting samples obtained from Olkiluoto
- Evaluating the performance of the newly installed joint sealant material (tradename Sikaflex Construction +)

It is already recognized that applying tensile testing for the samples obtained from Olkiluoto might be too challenging due to the sealant geometry. Thus, the focus while applying the methods should be on hardness, TGA and FTIR. However, it should be considered how the lack of reference data affects the interpretation of the results with these methods too.

The materials chosen for the further condition analysis should be decided together with Olkiluoto material experts. Based on the data received the ageing conditions have been rather similar (less than 10 degrees temperature differences and low dose rates). For the newly installed material, thermal ageing treatment will be considered to evaluate the lifetime of the material in its designed use.

6. Summary

Joint sealants are polymer-based materials used to bond and seal construction elements to each other. Such sealants are applied also in nuclear power plants and their ageing in these environments are a less studied topic. The most common polymer-based joint sealant materials were introduced and their properties were summarized in the report. Ageing mechanisms that these joint sealants may experience during their use were discussed. Experimental methods that could be used to evaluate the condition of joint sealants were presented. Joint sealant materials from Olkiluoto 1 and 2 units were obtained from the outage of 2021. Finally, experimental work to be performed with the samples obtained from Olkiluoto and the newly installed joint sealant were discussed.

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Annex 1 – Details on the samples obtained from Olkiluoto 1 and 2

Sample information from Olkiluoto 1.

Perustiedot					
Näyte	pvm.	Huone	Positio	Näytemassa	Huomautus / havainnot
1.1	4.5.2021	1.B01.11	Seinä, oikea	"Thiotät"	- Massan väri: tumman harmaa - sauman leveys noin 50 mm, - näytteen pituus noin 170 mm
1.2	4.5.2021	1.B01.11	Lattia, oikea	"Sika?"	- Massan väri: vaalean harmaa - sauman leveys noin 30 mm, - näytteen pituus noin 140 mm
1.3	4.5.2021	1.B91.15	Lattia, vasen	"Thiotät"	- Massan väri: tumman harmaa - sauman leveys noin 30 mm, - näytteen pituus noin xxx mm - Saumassa 2 kerrosta samaa massa päällekäin, välissä tummaa ainetta - Sauma pinnasta halkeillut
1.4	4.5.2021	1.B01.15	Seinä, oikea	"Thiotät"	- Massan väri: tumman harmaa - sauman leveys noin 50 mm, I3 - näytteen pituus noin xxx mm
1.5	5.5.2021	1.B07.42	Seinä, vasen	"Thiotät"+Sika?	-Näytekohdassa 2 eri massaa, ylempänä THIOTÄT (tummanharmaa) alla Sika (Vaaleanharmaa) - Sauman leveys 30mm - Saumassa ei pohjanauhaa, alta näkyy mineraalivilla -Vaaleanharmaata massaa vain pinnassa n. 5mm vahvuinen kerros
1.6	5.5.2021	1.B07.42	Lattia, vasen	Sika?	- Väri vaaleanharmaa - Leveys 30mm - Massa alareunastaan irti seinästä
1.7	5.5.2021	1.B06.50	Lattia, oikea	Sika?	- Väri vaaleanharmaa - Leveys 30mm - Massa yläreunastaan irti seinästä

1.8	5.5.2021	1.B06.50	Lattia, oikea	Sika?	- Vaaleampi kuin 2 tavattua muuta - Sauma halkeillut keskeltä - Ominaisuuksiltaan erittäin kova - Sauman leveys 50mm - Näytteen pituus 170mm
1.9	5.5.2021	1.B05.31	Lattia, vasen	THIOTÄT	- Näytteenpituus 170mm - Sauman leveys 30mm - Saumassa paljon reikiä ja halkeilua - Valkoinen pohjanauha PE
1.10	5.5.2021	1.B03.31	Seinä, vasen	THIOTÄT	- Sauma maalattu - Näytteen pituus 170mm - Sauman leveys 30mm
1.11	5.5.2021	1.B03.31	Lattia, oikea	THIOTÄT	- Saumanauha näkyvissä nurkassa - Sauman leveys 30mm - Näytteen pituus 150m
1.12	5.5.2021	1.B02.29	Seinä, vasen	THIOTÄT	- Sauma maalattu - Sauman leveys 30mm - Pituus 130mm - Saumanauha pohjalla, sauma ohut
1.13	5.5.2021	1.B01.42	Seinä, vasen	THIOTÄT	- Saumassa pientä halkeilua
1.14	5.5.2021	1.B01.42	Lattia, vasen	Sika?	-Päällä vaaleanharmaa - Alla tummanharmaa - Sauman leveys 30mm - Näytteenpituus 100mm
1.15	5.5.2021	1.B91.29	Lattia, vasen	Sika + THIOTÄT	- Näytteessä kahta eri massaa päällekkäin - Vanha massa elastisempaa -Sauman leveys 30mm - Pituus 300mm

Sample information from Olkiluoto 2.

Perustiedot					
Näyte	pvm.	Huone	Positio	Näytemassa	Huomautus / havainnot
2,1	27.5.2021	2.B07.42	Seinä vasen	Thiotät	-Pystysauma seinässä - Halkeilua - Sauman pituus 240mm
2,2	27.5.2021	2.B07.42	Lattia vasen	Sika?	- Ei halkeilua - Sauman pituus 220mm
2,3	27.5.2021	2.B06.49	Seinä vasen	Thiotät	- Tumma elastinen massa - Pituus 93mm - PE pohjanauha - Ei halkeilua
2,4	27.5.2021	2.B06.49	Lattia vasen	Sika?	- Vaalea ei niin elastinen sauma - Pituus 130mm - Ei halkeilua - PE pohjanauha
2,5	27.5.2021	2.B05.31	Seinä vasen	Thiotät	- Tumma elastinen massa - Pituus 100mm - Ei halkeilua
2,6	27.5.2021	2.B05.31	Seinä vasen	Sika?	- Vaalea leveä - Sauman pituus 130mm - 2 Massaa päällekkäin - PE pohjanauha, ei halkeilua
2,7	27.5.2021	2.B03.47	Lattia oikea	Thiotät ja sika	- Sauman pituus 110mm - Sauman pinta ruskean keltainen, maalattu?

					-Ei halkeilua, 2 eri massaa päällekkäin
2,8	27.5.2021	2.B03.47		Thiotät	- Sauman pituus 100mm - Ei halkeilua - Tumma elastinen sauma
2,9	27.5.2021	2.B02.29	Seinä vasen	Thiotät	- Halekeilua paljon - Tumma elastinen massa - Sauman pituus 110mm - Saman huoneen toisen kulman saumassa ei halk. (2.10)
2,10	27.5.2021	2.B02.29	Seinä oikea	Thiotät	- Sauman pituus 100mm - Tumma elastinen massa - Ei halkeilua
2,11	27.5.2021	2.B01.11	Seinä vasen	Thiotät	- Elastinen tumma massa - Ei halkeilua
2,12	27.5.2021	2.B01.11	Lattia vasen	Thiotät	- Massan päällä epoksi - Sauman pituus 100mm
2,13	27.5.2021	2.B91.29	Lattia oikea	Thiotät	- Sauman pituus 130mm - Vesi maannut kulmassa jossa sauma on - Elastinen sauma
2,14	27.5.2021	2.B91.29	Lattia oikea	Thiotät	- Sauman pituus 130mm -Elastinen tumma massa
2,16	27.5.2021	2.B07.14	Seinä vasen	Sika?	- Vaakasauma seinässä - Vaalea usittu massa

					- Avarrettu seinä -Ilma virtaa saumasta sauman avaamisen jälkeen - PE pohjanauhaa ei koko matkalta
2,17	4.6.2021	2.B06.09	Seinä vasen	Thiotät	- Elastinen ja tahmea - Pituus 100mm - Yleissäteily huonetilassa tehoajolla 0.0250 mSv/h
2,18	4.6.2021	2.B06.09	Lattia vasen	Thiotät	- Elastinen ja tahmea - Pituus 120mm - Yleissäteily huonetilassa tehoajolla 0.0250 mSv/h
2,19	4.6.2021	2.B06.14	Seinä vasen	Thiotät	- Elastinen ja tahmea - Pituus 100mm - Yleissäteily huonetilassa tehoajolla 0.0200 mSv/h
2,20	4.6.2021	2.B06.14	Seinän japalkin välinen	Thiotät	- Elastinen ja tahmea - Pituus 100mm - Yleissäteily huonetilassa tehoajolla 0.0200 mSv/h
2,21	4.6.2021	2.B02.08	Katon ja seinän välinen vaakasauma	Thiotät	- Elastinen ja tahmea - Pituus 110mm - Yleissäteily huonetilassa tehoajolla 0.0020 mSv/h
2,22	4.6.2021	2.B02.08	Lattian ja seinän	Thiotät	- Elastinen - Pituus 115mm

			välinen vaakasauma		- Yleissäteily huonetilassa tehoajolla 0.0020 mSv/h
2,23	4.6.2021	2.B03.11	Seinä vasen	Thiotät	-Elastinen - Pystysauma Säteilevän putken vieressä. - Yleissäteily huonetilassa tehoajolla 0.0500 mSv/h