5. Exemption 1(a) of Annex IV: Pb and Cd in Ion-selective Electrodes

The exact wording of the current exemption 1(a) of Annex IV is as follows:

"Lead and cadmium in ion selective electrodes including glass of pH electrodes"

The exemption expires on 21 July 2021 for EEE of category 8 other than in-vitro diagnostic medical devices (IVD) and for EEE of category 9 others than industrial monitoring and control instruments (IMCIs). For IVDs, the exemption expiry date was scheduled for 21 July 2023, and for IMCIs for 21 July 2024.

Declaration

In the sections preceding section 5.4 "Critical review" the phrasings and wordings of applicants' and stakeholders' explanations and arguments have been adopted from the documents they provided as far as required and reasonable in the context of the evaluation at hand. Formulations were only altered or completed in cases where it was necessary to maintain the readability and comprehensibility of the text. These sections are based exclusively on information provided by applicants and stakeholders, unless otherwise stated.

Acronyms and definitions

Ba	Barium
BaO	Barium oxide
Cd	Cadmium
CdS	Cadmium sulphide
IMCI	Industrial monitoring and control instruments
ISE	Ion-selective electrode
ISFET	Ion Sensitive Field Effect Transistor
IVD	In-vitro diagnostic medical devices
Pb	Lead
PbO	Lead oxide
pH	'potential of hydrogen', 'power of hydrogen', a scale used to specify the acidity or basicity of an aqueous solution
RoHS 1	Directive 2002/95/EC
RoHS 2, RoHS	Directive 2011/65/EU

TCE	thermal coefficient of expansion

5.1. Background

JBCE (2020a) and JBCE (2020b) (amendment of original renewal application) requested the renewal of exemption 1(a) on 17 January 2020 and 6 July 2020 respectively (amendment) for the maximum validity period of seven years with the following modified wording:

Lead in pH glass electrodes and ion selective electrodes equipped with a pH glass electrode with complex shape as following:

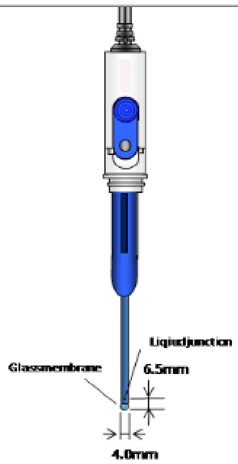
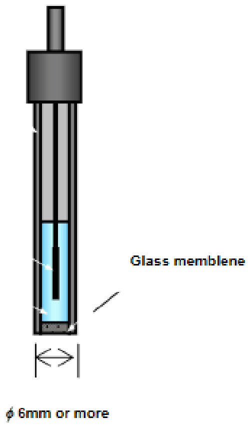
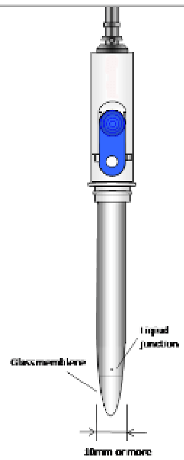
I) Micro type pH glass electrode
Composite electrode that has a spherical or tube-shaped pH responsive glass membrane with a diameter of 4.0 mm or less and a reference electrode with a liquid junction at a position vertically within 6.5 mm from the tip;

II) Flat type pH glass electrode
pH glass electrode with a flat pH response membrane at the tip of a glass tube with a diameter of 6.0 mm or more;

III) Needle type pH glass electrode
Composite electrode that has a conical pH response membrane with a tip angle of 40 ° or less and with a diameter of 10 mm or more.

JBCE (2020a) request the above renewed wording for EEE of cat. 9 monitoring and control instruments including industrial monitoring and control instruments (IMCI). JBCE (2020b) added the illustration in Figure 5-1 to demonstrate the types of glass pH glass electrodes addressed in their proposed wording:

Figure 5-1: Types of pH glass electrodes addressed in the proposed new wording of exemption 1(a) of Annex IV

<table>
<tr><th>1. Micro Type pH Glass Electrode</th><th>2. Flat Type pH Glass Electrode</th><th>3. Needle Type pH Glass Electrode</th></tr>
<tr><td>
</td><td>
</td><td>
</td></tr>
</table>

Source: JBCE (2020b)

No contributions were received during the stakeholder consultation.

5.1.1. History of the Exemption

Exemption 1(a) of Annex IV was not part of RoHS Directive 2002/95/EC (2003) (RoHS 1). It was first evaluated by Goodman (2006) and subsequently listed on Annex IV of RoHS Directive 2011/65/EU (2011) (RoHS 2) when this was officially published in 2011. Applications for renewal were submitted in time, and exemption 1(a) was reviewed for the first time to adapt it to scientific and technical progress.

5.1.2. Summary of renewal request

JBCE (2020a) state that *“The current exemption is for both lead and cadmium in ion selective electrodes and pH glass electrodes, however JBCE requests renewal only for lead as it has no knowledge of electrodes that contain cadmium [Goodman (2006) stated that cadmium concentration in aqueous solutions was measured with an electrode sulphide membrane at the time, the consultants].*

pH meters are used by a wide variety of purposes including use in laboratories, process control, quality control, workplace safety, environment (pollution) analysis. Over the last 14 years, pH electrode manufacturers have carried out research into lead-free glass pH electrodes and this work has been successful for many designs. However, where complex or unusual shapes of electrodes are required, lead-free glass causes cracks during the manufacturing process that result in premature failures. Therefore, this exemption is needed for these designs. pH electrodes are also used as components inside electrodes for analysis of substances, such as ammonia and this exemption is also needed for these electrodes. Lead-free pH glass electrodes are available on the market. However, lead in glass of pH glass electrode is required to create intermediate layer for the connection between stem tube and pH-responsive glass or pH glass membrane. Some complicated shapes explained in this document cannot be formed without lead and currently there is no alternative technology that allow glass to be substituted.”

5.2. Technical description of the requested exemption

5.2.1. Amount of lead used under the exemption

JBCE (2020a) state that they do “not have access to all EU data. pH glass electrodes and ion selective electrodes are made by many manufacturers and are used in a wide range of final products and markets, it is therefore impossible to provide a precise figure of the amount of lead included in glass of all pH electrodes supplied in the EU. However due to the developments of lead-free alternatives for some designs of electrodes it would be reasonable to expect that the annual quantity of lead used is reduced from the previous exemption request even if the exact amount cannot be calculated.

The amount entering the EU market annually from manufacturers of JBCE members has been calculated to be approximately 14 g.”

5.2.2. Applications in the scope of the requested exemption

According to JBCE (2020a), pH glass electrodes are used in pH meters that are employed for a wide variety of purposes including use in laboratories, process control, quality control, workplace safety, environment (pollution) analysis. JBCE (2020a) explain that pH electrodes are also used as components inside electrodes for analysis of substances, such as ammonia, and this exemption is also needed for these electrodes (ion selective electrodes). Both applications are described in more detail in the following.

pH meters

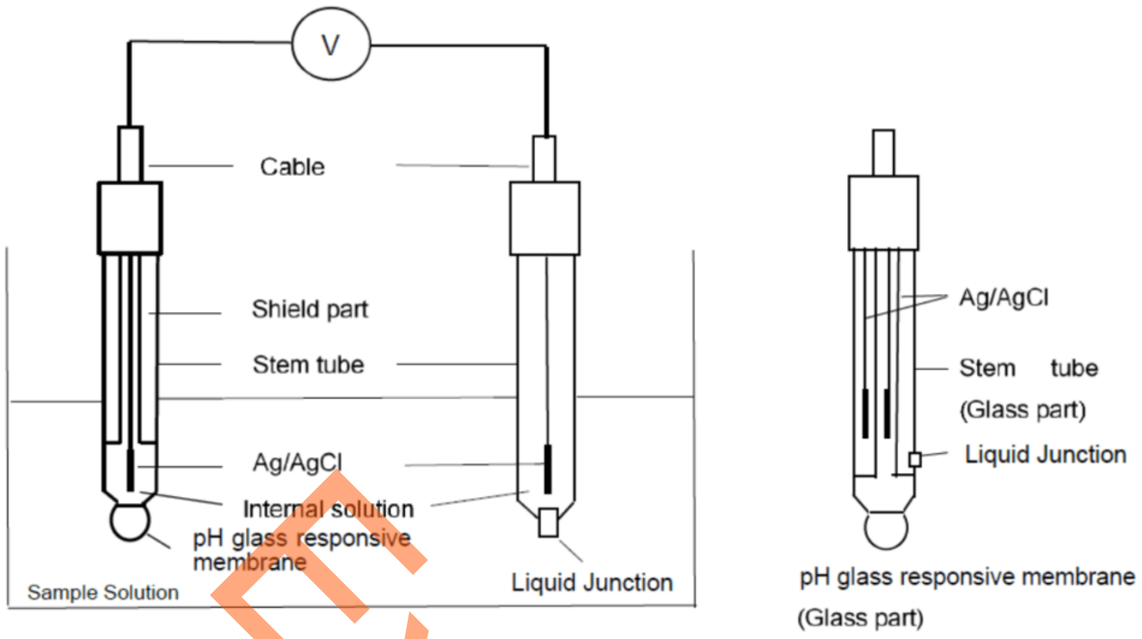
According to JBCE (2020a), the "pH meter is one of the most widely used instruments with a wide variety of purposes. The applications listed below are categorized and are not exhaustive:

- *Use in laboratories of universities, companies, research institutes, educational institutions and quality control in manufacturing industries;*
- *Process control in the industrial facilities: use and control for production and manufacturing lines;*
- *Quality control; control of pH in food, drinking water and sewerage;*
- *Use for workplace control and safety for safety check before work; and*
- *Use for environment (pollution) analysis."*

JBCE (2020a) explain the principle of pH measurement using a pH meter is that "the silanol groups formed in the hydrated layer on the pH-responsive glass surface respond to hydrogen ions, and the potential generated across pH-responsive glass affected by the hydrogen ion concentration is measured with a potentiometer".

Diagrams of pH measurement with pH glass electrode and a reference electrode, as well as a structure of pH glass combination electrode (pH electrode combined with a reference electrode) are shown in Figure 5-2.

Figure 5-2: Configuration example of a pH electrode

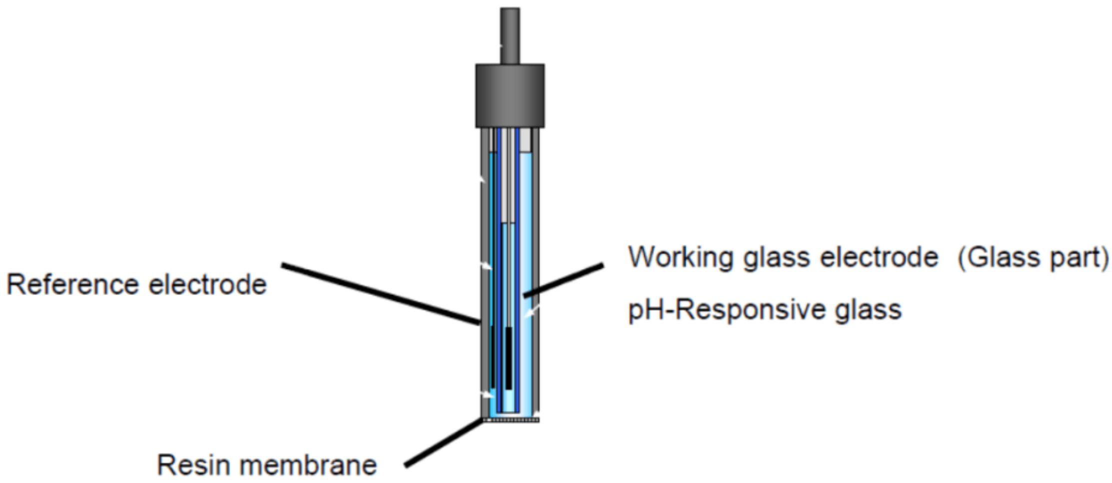


Source: JBCE (2020a)

Ion selective electrode

According to JBCE (2020a), "diaphragm type ion selective electrode (ammonia electrode) contain a pH glass electrode and a reference electrode. In this case, ammonia gas migrates through the membrane, and is changed to ammonium ions in an alkaline internal solution. Ammonium ions are measured by the change in pH and converted into ammonia concentration". The structure of an ion selective electrode equipped with a pH glass electrode is shown in Figure 5-3.

Figure 5-3: Configuration example of ion-selective electrode equipped with a pH glass electrode



Source: JBCE (2020a)

Function of lead in the described applications

JBCE (2020a) explain that in pH glass electrodes, there is pH responsive glass and a stem tube glass, which are joint as shown in Figure 5-2. JBCE (2020a) explain that "the pH responsive glass is a glass that selectively responds to hydrogen ions and has a special composition containing about 30 mol % of an alkali metal such as lithium. This glass generally has a linear thermal expansion coefficient of 100 × 10^{-7} / degree or more due to its high alkalinity. The glass is joined by heat with a stem glass tube which is a different kind of glass with electrically insulating character. Typical thermal expansion coefficient of the stem glass tube is 94 × 10^{-7} / degree."

JBCE (2020a) continue to explain that "at the joint of the two different glasses with different thermal expansion coefficients, it is easy for cracks to occur shortly after manufacturing due to temperature change or static fatigue. To prevent the cracks, after the stem tube glass and pH-responsive glass are joined by heat, air pressure is manipulated to expand and contract the pH responsive glass several times so that an intermediate layer is formed between the two different types of glasses. When there is enough intermediate layer formed, no cracks occur, and the electrode is able to be manufactured without failure."

However, JBCE (2020a) explain further that "it is difficult to form an intermediate layer in the case of electrodes that have complicated shape, for example flat responsive glass type, microelectrode type, or needle tip type. The complicated shapes of these electrodes allow unique functionality which would not be able to be achieved without this shape, which is discussed further below. Since the shape of responsive glass of these electrodes are complicated, it is necessary to join the responsive glass membrane and stem glass tube in a very short time, otherwise the shape of the glasses is deformed due to the heat. Due to the limited time to form an intermediate layer between the response glass membrane and the stem glass tube, cracks can easily occur."

JBCE (2020a) state that when lead containing glass is used as stem glass tube, "no cracks occur, even if there is a limited processing time to allow for an intermediate layer to be formed. The reason is that lead is an element which has a low chemical potential, and so rapidly diffuses into different types of glass at the time of bonding to form an intermediate layer even in a short time. This is the reason why lead is needed for electrodes with complicated shapes."

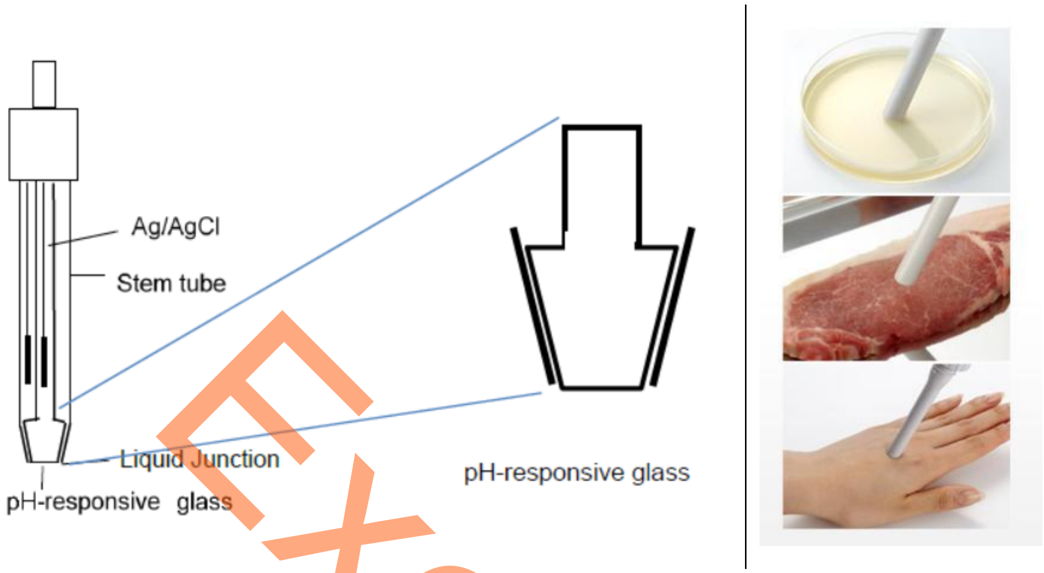
Specific electrode types requiring the exemption

Flat type electrode

According to JBCE (2020a), the "flat type electrode is used for measuring the pH of a thin film such as paper, and is measured by penetrating a small amount of water into paper. Measurement is performed by bringing a film into contact with the surface of a flat object. Ammonia electrode has a flat pH responsive glass electrode inside. The flat membrane is joined to stem glass tube. The place where cracks are likely to occur is the joint between the responsive glass and the stem tube. The reason of the cracks is that the upper part of the joint portion of the stem tube is on the thread during manufacture and heat cannot be applied to the stem tube with a burner. The responsive glass and the stem tube have *different thermal coefficient of expansion (TCE) values and so cracks are likely to occur, unless lead glass is used as the stem glass tube."*

JBCE (2020a) provided the images in Figure 5-4 for illustration.

Figure 5-4: Flat type example configuration (left) and application examples (right)



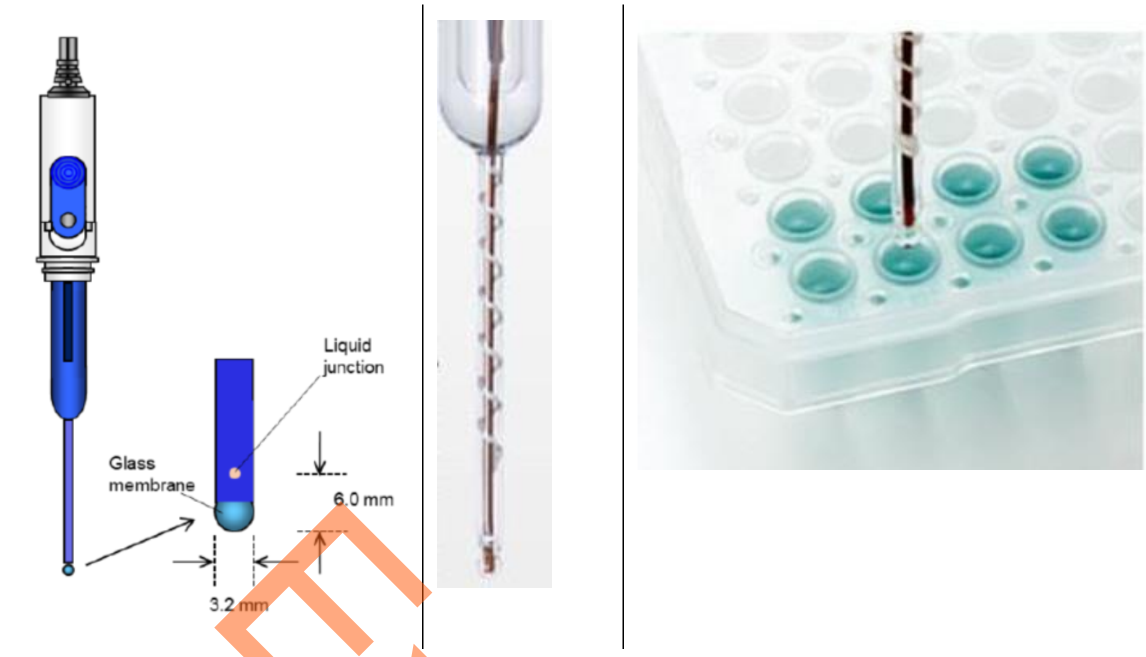
Source: JBCE (2020a)

Microelectrode type

JBCE (2020a) stated that "since the pH responsive glass membrane is small, it is necessary to make the glass composition rich in lithium oxide to reduce the responsive glass resistance to 300 MΩ or less, which is necessary for the electrode to function correctly as well as being a measurement method standard of the Japan Measurement Act. A large amount of lithium oxide inevitably increases the linear thermal expansion coefficient. Since the linear thermal expansion coefficient difference with the stem tube glass is large, cracks are likely to occur. Lead is what eases the stress between the two glass types."

JBCE (2020a) provided the images in Figure 5-5 for illustration.

Figure 5-5: Microelectrode type example configuration (left), close up of the pH responsive glass electrode (middle), and application example (right)



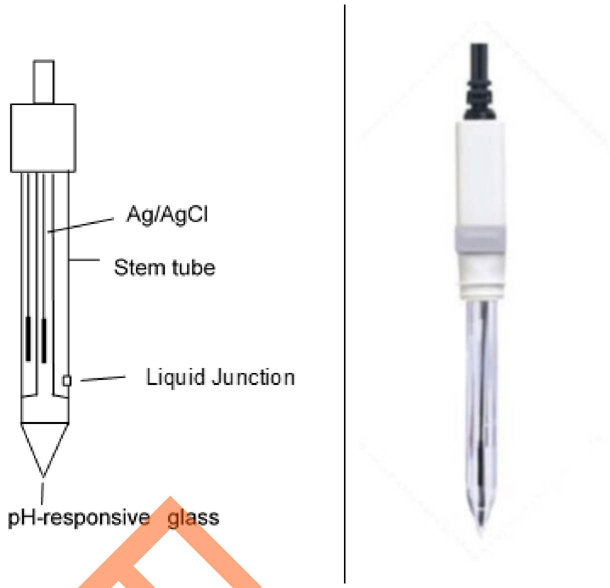
Source: JBCE (2020a)

Needle type

JBCE (2020a) stated: "In case of needle type, the stem tube is rotated with a lathe, the responsive glass is melted with a burner and placed on the stem tube. The shape is processed into the needle shape while gradually stretching the molten glass and therefore is unable to be heated further to join the glasses and form an intermediate layer without the use of lead. The place where the crack is likely to occur is the joint between the responsive glass and the stem tube, unless lead glass is used. This electrode has a sharp tip, to allow pH measurement by inserting it into a soft solid such as cheese or yogurt."

JBCE (2020a) provided the images in Figure 5-6 for illustration.

Figure 5-6: Needle type example configuration



Source: JBCE (2020a)

5.3. Justification for the requested exemption

5.3.1. Substitution of lead in pH glass electrodes

Lead-free glass

According to JBCE (2020a), "over the last 14 years, pH electrode manufacturers have carried out research into lead-free glass pH electrodes and this work has been successful for many designs. Electrodes that are simple in shape, such as general purpose electrodes are possible to process already with lead-free glass. However, where complex or unusual shapes of electrode are required, lead-free glass causes cracks during the manufacturing process that result in premature failure. Therefore, this exemption is needed for these designs."

JBCE (2020a) state that "lead-free pH glass electrodes are available on the market. However, lead in glass of pH glass electrodes is required in order to create an intermediate layer for the connection between stem tube and pH-responsive glass or pH glass membrane. Some complicated shapes explained in [section 5.2.2] cannot be formed without lead and currently there is no alternative technology that allow glass to be substituted."

Adding a more in-depth explanation, JBCE (2020a) state that "the thermal expansion coefficient of lead glass is close to that of pH responsive glass", stating that generally, the difference of the coefficient should be within 10 % for joining different kinds of glass firmly. JBCE (2020a) provided a table that shows the thermal properties of typical lead glass and lead-free glass (Table 5-1).

Table 5-1: Comparison of thermal properties of lead glass and lead-free glass

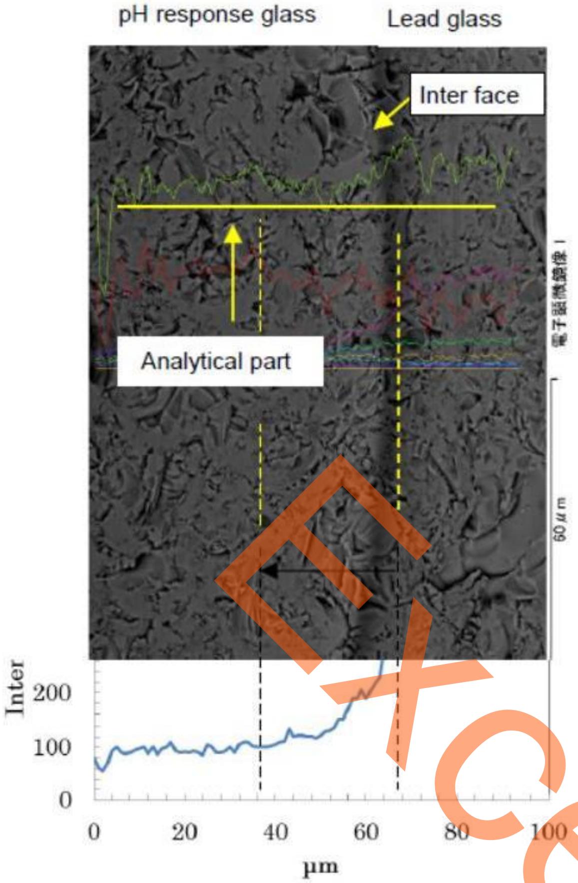
Glass Type	Stem GLASS		
Characteristic	L-29(NEG) *1 Lead Glass	PS-94(NEG) *2 Lead free glass	Schott AR GLASS *3 Lead free glass
Thermal expansion coefficient at 30-380℃ ($\times 10^{-7}$/K)	94	94.5	91.0
Density $\times 10^3$kg/m3	3.05	2.57	2.50
Strain point ℃	395	440	-
Annealing temperature ℃	435	480	530
Softening point ℃	625	665	720
Working point ℃	965	980	1040
WT% of PbO	29	None	None

Source: JBCE (2020a), citing third sources

JBCE (2020a) further explain that "since the thermal expansion coefficient of our pH responsive glass is 95 to 110 × 10^{-7} / K, bonding with lead glass is possible without any problem. Lead free glasses seem to have no problem because of their similar thermal expansion coefficients, however, they have different thermal characteristics other than expansion coefficient. For example, the softening point is higher at 665 °C and 720 °C, whereas lead glass is 625 °C, which differs by 40 °C or more. This difference means that the rate of shrinkage is different in the cooling process, so that higher strain stress is generated and cracks between different glasses are more easily occurred. This is the reason why lead-free glass is difficult to use for stem glass."

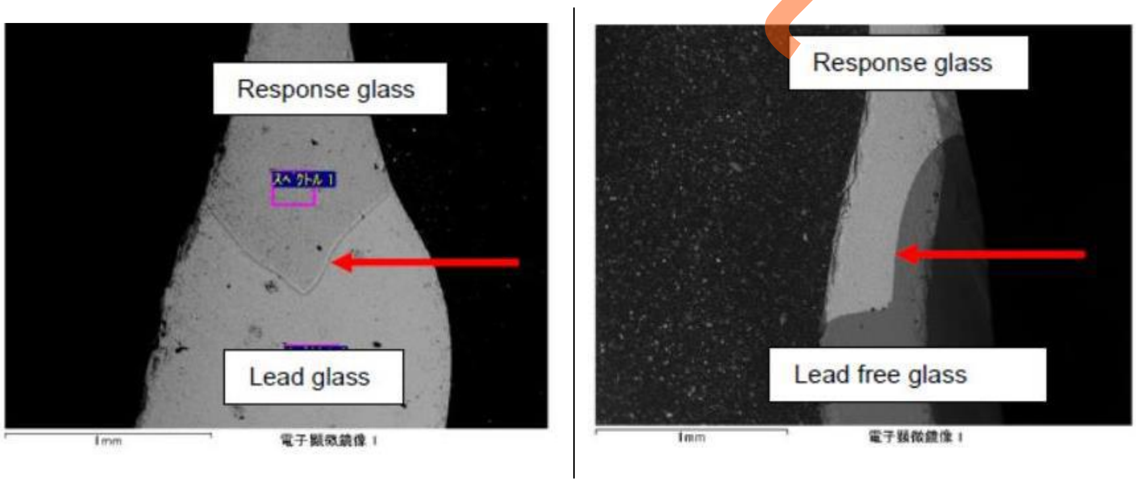
According to JBCE (2020a), another reason for the use of lead is that "lead glass is easily bonded to pH responsive glass firmly. This is because lead glass diffuses toward pH responsive glass and forms an intermediate layer between the two glasses". Figure 5-7 shows the result of line chemical analysis of lead in the bonding interface determined by EDX (energy dispersive X-ray analysis), which shows a distribution of lead in the glass. Figure 5-8 (left hand image) is a SEM (electron microscope) photograph of the joint of lead glass and pH responsive glass. As shown in the image, lead diffuses and makes a bonding interface layer which is seen on the image as a double line. In contrast, in using lead free glass, the interface is a sharp boundary as shown on the image as one clear line Figure 5-8 (right hand image). According to JBCE (2020a), "this means that the compatibility between the two glasses is small and does not have enough intermediate layer. In this case the bonding is performed only at the interface, and the bonding strength is weak and cracks can more easily occur."

Figure 5-7: Analysis of lead distribution in the joint between lead glass and pH response glass



Source: JBCE (2020a), citing an unnamed JBCE member company

Figure 5-8: SEM photographs of the joint between lead glass and pH response glass showing the diffuse interface layer (left) and SEM photograph of the joint between lead free glass and pH response glass (right)



Source: JBCE (2020a), citing an unnamed JBCE member company

JBCE (2020a) describe that "since there is no diffusion of elements such as lead when lead-free glasses are used, the joint interface becomes clear and cracks are likely to occur. The pH glass electrode measures the pH of an aqueous solution of acid or alkali. Ions contained in these measurement samples can enter the glass bonding interface and cause cracks after several weeks to several months (cracks that generate cracks over time are referred to as static fatigue cracks)."

JBCE (2020a) further state that "analyzing these phenomena by fluorescence analysis and optimizing the processing conditions can be improved to a large extent the ability to withstand crack formation. Conventional shape pH glass electrodes are able to be manufactured by blowing glass which has lead below the regulated value. However, pH electrodes which have a special shape (as described in section 5.2.2) has been devised to increase the alkali content of the response glass to lower the electrical resistance, and but this has the disadvantage of having a larger linear thermal expansion coefficient than lead glass. If a lead-glass support stem tube is used, the difference in expansion coefficient does not become a problem, but if a lead-free glass is used, the difference in TCE causes cracks. Because there is no lead diffusion (intermediate layer), static fatigue cracks are likely to occur at the joint bond between the tube glass and the responsive glass."

JBCE (2020a) argue that "one difficulty is that lead-free glass is much less flexible at the highest temperature that can be used to bond the stem glass tube to the pH sensitive glass. If the temperature is raised to further soften the glass, the stem glass becomes too soft and distorts so it is impossible to make complex shapes. Trials with the complex pH electrode types shown in section 5.2.2 resulted within a few hours of making the bonds, cracks will form within the bonds due to the stresses within the glass as it cools.

As a consequence of a higher softening temperature the more complex geometries of Flat type electrodes, Needle type electrodes and Microelectrode type lead free glasses are unable to be used as the higher temperature causes the deformation of the electrode during manufacturing."

Barium glass

When asked whether substances other than lead have been investigated to fulfil the same function in the stem glass of glass electrodes, JBCE (2021a) stated to not be aware of any "*examples of research for adding other elements in a stem tube that can achieve the same functionality as lead. The glass of stem tubes used for pH electrodes is commercially available product for a wide range of applications, not dedicated to pH electrode application. The development of stem tubes dedicated to pH electrode is not feasible for a large-scale production because the demand of pH electrode is far smaller than demand for other applications. Therefore, a glass responsive membrane that can be adapted to commercially available stem glass is being developed.*"

When specifically asked about whether barium glass might be a substitute for lead glass (this was stated by another company, cf. Table 5-6 in section 5.4 "Critical Review"), JBCE (2021a) stated: *"Barium is one of the general additives when making the glass, so we believe the main purpose of adding barium is not to achieve the same functionality as lead"*. JBCE (2021a) further clarified that in the glass-forming process, lead (specifically: lead oxide) is a glass-forming intermediate, while barium (specifically: barium oxide) is a glass modifier. JBCE (2021a) explain the difference as follows: *"Since glass modifiers have weak bonds, metallic cations (such as Ba) can diffuse inside a glass during the high temperature fabrication process but will not contribute in bridging the glass network. On the other hand, "glass forming intermediates" such as lead oxide (PbO) are essentials for the formation process of glass. Lead cations (Pb) act as bridges in the glass network for the diffusion of different kinds of others elements by heat during the formation of the glass. PbO is therefore essential to bond glasses with different thermal characteristic, such as a relatively large variability in thermal expansion coefficient."* Therefore, according to JBCE (2021a), as barium is classified as a "modifier", not as a "glass-forming intermediate", barium does not have the same function than lead in the glass.

JBCE (2021a) provided a table with a classification of glass-forming inorganic substances, taken from the Glass Engineering Handbook (Table 5-1).

Table 5-2: Classification of glass-forming inorganic substances

Glass-network formers	Glass-forming intermediates	Glass modifiers
SiO2	TiO2	Li2O
B2O3	TeO2	Na2O
P2O5	Al2O3	K2O
GeO2	Bi2O3	MgO
BeF2	V2O5	**BaO**
	Sb2O3	CaO
	PbO	SrO
	CuO	LiCl
	ZrF4	NaCl
	AlF3	BaF2
	InF3	LaF3
	ZnCl2	
	ZnBr2	

Source: JBCE (2021a), stating the Glass Engineering Handbook as source

With respect to the glass-forming intermediates other than lead oxide, JBCE (2021a) provided the following comparison of required properties (**Table 5-3**). In this comparison, every compound has a negative impact in its application in pH electrodes.

Table 5-3: Comparison of glass-forming intermediates in necessary properties

	pH sensitivity	Alkaline error	Water proof	workability	Other disadvantages
Al2O3 Bi2O3 Sb2O3 AlF3 InF3 ZnCl2 ZnBr2	Low sensitivity	Cause of error	No negative impact	No negative impact	N/A
V2O5	No negative impact	No negative impact	Less water proof[1]	No negative impact	N/A
CuO	No negative impact	No negative impact	No negative impact	Less workability	N/A
TiO2 TeO2 ZrF4	No negative impact	No negative impact	No negative impact	No negative impact	Cause of less formability
PbO	No negative impact	No negative impact	No negative impact	No negative impact	N/A

Source: JBCE (2021a)

5.3.2. Elimination of lead

JBCE (2020a) describe ISFET electrodes and the use of fluorescent dyes as alternative technologies to lead-containing glass electrodes. The listed methods are stated to be shown as examples and not exhaustive.

Ion Sensitive Field Effect Transistor (ISFET) electrodes

JBCE (2020a) explain that instead of pH responsive glass membranes, metal oxide semiconductors can be used to measure the "potential generated depending on hydrogen ion concentration in a sample. It can be used in almost the same measurement range as the conventional electrode with responsive glass membrane. Since the surface is stronger than a glass membrane, and because there is no need to use glass for stem tube, the strength and the design of the liquid contact part is advantageous compared to conventional glass electrodes." JBCE (2020a), however, also point out problems with ISFET:

a. The ISFET resin body and semiconductor are susceptible to damage
b. The shape of ISFET electrodes cannot be completely flat
c. ISFET have a limited measurement range
d. Measurements are affected by light
e. Lower battery life of portable ISFET instruments

These four issues are described in more detail the following sections.

a. The ISFET resin body and semiconductor are susceptible to damage

JBCE (2020a) states that one disadvantage of ISFET is that its plastic body is damaged by many organic solvents or other similar substances: ISFET has a resin body, which can be damaged by organic solvent, chlorine and other chemicals that can permanently damage the ISFET chip. When asked to specify which components of an ISFET electrode are damaged by which substances, JBCE (2021a) specified the following three factors:

- *"A pH-sensor based on an ISFET chip is very sensitive to electrical static discharges (ESD) such as every Field-Effect transistor-based technology. Unlike most integrated circuits, which are operating in a closed, well-protected, environment, ISFET are often exposed to very high intensity ESD events. For example, very low conductivity waters or hands of someone manipulating the pH sensor can generate ESD events of dozens of kV that may deteriorate permanently an ISFET chip. Despite ESD-protection integrated in the ISFET pH sensor, some applications are not suitable for ISFET due to the high occurrence of ESD-events. On the other hands, glass-made pH electrodes are totally insensitive to ESD." JBCE (2021a)*
- *"The sensing layer of an ISFET is a metal oxide (for example, Al_2O_3 or Ta_2O_5) deposited during the CMOS process. Despite being chemically inert in most aqueous solutions, metal oxide materials will be dissolved in highly concentrated alkali solutions (such as 1M NaOH or KOH), with a etching rate increasing exponentially with the temperature." JBCE (2021a)*
- *"The body of the pH sensor, the substrate of an ISFET chip or the hydrophobic coating used to protect the ISFET connection can be damaged by long exposure in organic solvents." JBCE (2021a)*

When asked to explain the reasons why the body of ISFET electrodes is made from resin, and whether other materials, such as ceramics or glass, might be used alternatively, JBCE (2021a) responded that *"In the encapsulation process of the ISFET sensor, the ISFET chip and several additional components (such as a glass thermistor or a SMD capacitor) are mounted directly inside the plastic body, which has been designed on purpose to fit each component. This would be probably extremely difficult to fabricate a ceramic-based body that can accommodate such a complex geometry with the tolerance on size and the reproducibility that can be achieved with the current polymer body. We also believe that glass might not be suitable because some steps of the encapsulation process require some mechanical constraints on the sensor that can ultimately break a glass material."*

The consultants identified ISFET electrode products that use materials such as PEEK (Polyether ether ketone), FFKM (perfluorinated rubber) and ceramics[19]. When asked whether such products would have similar issues with damage from organic solvents or other substances, JBCE (2021a) responded: "*We agree that polymer materials you proposed can increase the chemical resistance for the body of the ISFET pH sensor, although some fluorinated materials may not bonded with adhesives. On the other hand, we believe that changing the material of the sensor body will not be effective to overcome*

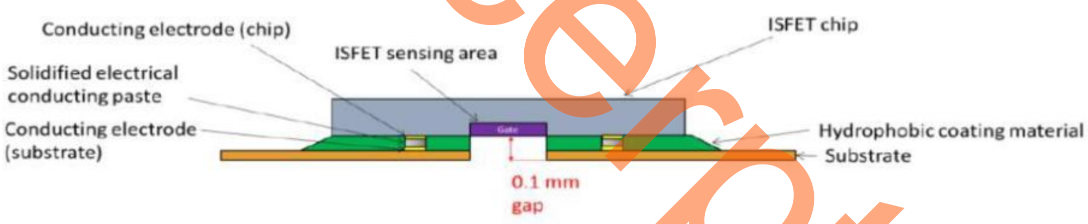
[19] Digital non-glass sensor Memosens CPS47D: https://www.de.endress.com/en/field-instruments-overview/liquid-analysis-product-overview/pH-digital-sensor-cps47d (last accessed: 2nd August 2021)

essential issues of the ISFET which are ESD events and low lifetime in strongly alkaline solutions. In addition, changing the material of the body will not remove the requirement of using a hydrophobic coating material in order to connect the ISFET chip with the substrate. A coating material will not have increased chemical resistance by changing the material body. ISFET pH sensor made with a PEEK or a FFKM body will certainly share most of limitations in the ISFET technology we stated earlier […]."

b. The shape of ISFET electrodes cannot be completely flat

Another disadvantage of ISFET described by JBCE (2020a) is that the shape cannot be completely flat, therefore ISFET cannot be applied in use cases where a completely flat electrode is required. The consultants identified suppliers ISFET pH electrodes that are designed to take pH measurements from the surface of solid samples via their flat-tipped sensor[20] *and have the semiconductor-based sensor located less than 100 µm from the flat surface of the tip*[21]*. When asked about these product examples, JBCE (2021a) stated that the sensing area of the ISFET electrode must be in contact with the measured samples (typically water) but the conducting electrodes (the terminal to take out the electrical signal from a field effect transistor) must be kept strictly isolated from liquids to protect the sensor against malfunctions. This protection is typically performed by depositing a thin layer of a hydrophobic coating material, which leads to the sensing area of the ISFET chip being around 0.1 mm depth the level of the substrate. JBCE (2021a) provided the illustrating schematic produced in Figure 5-9.*

Figure 5-9: Schematic of an ISFET



Source: JBCE (2021a)

JBCE (2021a) stated that "While the gap of 0.1 mm might be considered small compared to previous generation of ISFET products, this cannot prevent the formation of air bubble on the top of the sensing area during measurement. Air bubbles can be ultimately removed by stirring the solution. However, since there are some applications in which it is impracticable to stir during measurement, an ISFET pH electrode is not always an alternative to glass pH responsive electrode due to the 0.1 mm gap existing on the top surface of ISFET pH electrode."

When asked whether ISFET electrodes can generally be designed to be used in applications which require the three complex shapes relevant for this exemption request

[20] Horiba Flat ISFET pH Electrode - 0040-10D: https://www.agriculturesolutions.com/horiba-flat-isfet-ph-electrode-0040-10d (last accessed: 2nd August 2021)

[21] 0040-10D LAQUA Electrode pH ISFET https://www.alphaomega-electronics.com/en/electrodes/4599-0040-10d-laqua-electrode-ph-isfet-ion-sensitive-field-effect-transistor.html (last accessed: 2nd August 2021)

(flat, microelectrode, or needle type), JBCE (2021a) stated that *"It might be technically possible. However, the microelectrode design cannot accommodate the ISFET chip that is bigger than the 3 mm diameter of the pH sensing area. We address that the flat electrode cannot be done perfectly flat using ISFET for reasons explained on the previous point."*

c. Limited measurement range

JBCE (2020a) stated that while pH electrodes fitted with a glass membrane feature a measurement range of pH -2 to 16, ISFET has a more limited measurement range of pH 0 to 14.

d. Lower battery life of portable ISFET instruments

Another disadvantage mentioned by JBCE (2020a) is that "when using a common measuring equipment for conventional glass electrode, ISFET requires another signal conversion circuit, which increases the size of instruments and electric power consumption. Lifetime of batteries of portable instruments is shorter than the instruments with glass pH electrodes."

e. Measurements are affected by light

Lastly, according to JBCE (2020a), "the measured value is affected by light during measurement because of its semiconductor property, and so ISFET is not suitable for outdoor measurement or measurements in bright areas."

Optical measurements using fluorescent dye

JBCE (2020a) describe that "in this method, pH is measured by measuring the amount of emitted fluorescence according to the hydrogen ion concentration in sample solution. Since it is not necessary to use reference electrode, there is no concern that potassium chloride (KCl) solution derived from reference electrode will flow out to sample solution, which is particularly useful for measurement in a closed system.

However, this method also has some problems. One is that the range in which pH can be measured is narrow, and in some cases only pH 4 to 10 can be measured. Its measurement resolution is also low, making it difficult to see the difference in the order of 0.01 or 0.001. In addition, since it is affected by the influence of sample temperature and concentration of ions contained therein, it can be used only to know trends in limited situations. As described above, fluorescent method cannot be an alternative method of conventional glass electrode."

Comparison of glass membrane and potential alternatives

JBCE (2020a) provide a table comparing glass membrane and its potential alternatives regarding several aspects in **Table 5-4**.

Table 5-4: Comparison of glass membrane and potential alternatives

	Glass membrane	ISFET	Fluorescence
Response location	pH Response glass	Semiconductor (metal oxide compounds)	Fluorescent dye
Necessary equipment	Voltmeter (high impedance)	Signal coverter circuit	Fluorescence measurement
Measurement range	-2 to 16	0 to 14	4 to 10
pH resolution	0.001	0.001	0.1
Body material	Glass or Plastic	Plastic	Plastic
Influencing factors	-	Outer light	Outer light Ion concentration Temperature

Source: JBCE (2020a)

5.3.3. Roadmap towards substitution or elimination lead

JBCE (2020a) point out that for the three types of pH electrodes, micro size, needle type and flat type, that use lead glass in the scope of this exemption request, efforts are underway to make these without lead or reduced lead content.

With respect to the stages necessary for substitution of lead in the glass of these three electrode types and the timeframe needed for completion, JBCE (2020a) stated that glass of pH electrodes and ion selective electrodes require the intermediate layer in the glass and that there is currently no known way of creating an intermediate layer in the complex geometries described in section 5.2.2 without lead. Therefore, JBCE (2020a) cannot provide a specific time frame for searching for the composition of a lead-free glass substitute. JBCE (2020a) state, citing a third party report[22], that new product development time for many Category 8 and 9 products is over 4 years and can be 7 years or longer. This is considered an expected timeframe by JBCE (2020a) as instrument manufacturers will need to undertake engineering changes and evaluate the functionality of the alternative solution. The change would also mandate the update of global approvals, one of which is the submission of change of products as required by the Measurement Act of Japan. JBCE (2020a) cannot specify the period of the schedule but due to the complex nature of the product expect this to be over 7 years.

[22] ERA Technology (2006) , Review of Directive 2002/95/EC (RoHS) Categories 8 and 9 – Final Report, 2006, p.29 https://ec.europa.eu/environment/pdf/waste/weee/era_study_final_report.pdf (last accessed: 3rd August 2021)

5.3.4. Environmental arguments and socioeconomic impacts

JBCE (2020a) state that if this exemption is not renewed, "reliable pH electrodes could no longer be sold in the EU which will prevent many EU industries from operating and pollution could not be prevented (e.g. as water quality could not be monitored). EU industry would be at a very significant competitive disadvantage and there would likely be significant loss of EU jobs."

5.4. Critical review

5.4.1. REACH compliance – Relation to the REACH Regulation

Art. 5(1)(a) of the RoHS Directive specifies that exemptions from the substance restrictions, for specific materials and components in specific applications, may only be included in Annex III or Annex IV "*provided that such inclusion does not weaken the environmental and health protection afforded by*" the REACH Regulation. The article details further criteria which need to be fulfilled to justify an exemption, however the reference to the REACH Regulation is interpreted by the consultants as a threshold criteria: an exemption could not be granted should it weaken the protection afforded by REACH. The first stage of the evaluation thus includes a review of possible incoherence of the requested exemption with the REACH Regulation.

Lead

Lead is a substance of very high concern but so far, aside from a few specific compounds, has not been adopted to REACH Annex XIV. The fact that lead is a candidate substance therefore at the time being does not weaken the *environmental and health protection afforded by"* the REACH Regulation if the requested exemption would be granted/renewed.

REACH Annex XIV (2021)[23] *lists a few substances which include lead compounds, the placing on the market and use of which would require an authorisation in the European Economic Area:*

- *Lead chromate (entry 10);*
- *Lead sulfochromate yellow (entry 11);*
- *Lead chromate molybdate sulphate red (entry 12);*

The applications in the scope of the exemption at hand do not use any of the above lead compounds.

REACH Annex XVII (2021) also contains entries restricting the use of lead compounds:

- *Entry 16 and entry 17 restrict the use of lead carbonates and lead sulphates in paints;*

[23] ECHA, https://echa.europa.eu/authorisation-list?p_p_id=disslists_WAR_disslistsportlet&p_p_lifecycle=1&p_p_state=normal&p_p_mode=view&_disslists_WAR_disslistsportlet_javax.portlet.action=searchDissLists

- *Entry 19 refers to arsenic compounds but includes a few lead compounds such as lead arsenide and restricts their use as anti-fouling agent, for treatment of industrial water or for the preservation of wood;*

The above applications are not applicable to the use of lead in oxygen sensors.

- *Entry 28*[24] *addresses substances which are classified as carcinogenic. In this context, it stipulates that various lead compounds, e.g. lead chromate, shall not be placed on the market, or used, as substances, constituents of other substances, or in mixtures for supply to the general public;*
- *Entry 30*[25] *addresses substances which are classified as reproductive toxicants. Like for entry 28, entry 30 stipulates for some lead compounds that they shall not be placed on the market, or used, as substances, constituents of other substances, or in mixtures for supply to the general public;*

The above restrictions are not applicable to the use of lead in glass electrodes. Further, the substances are part of an article and thus are not placed on the market or used as substances, constituents of other substances or mixtures supplied to the general public.

- *Entry 63*[26] *restricts the use of lead and its compounds in jewellery, e.g. wristwatches, and in articles or accessible parts thereof that may, during normal or reasonably foreseeable conditions of use, be placed in the mouth by children. This entry lists many lead compounds, including lead sulphide (PbS) and lead selenide (PbSe).*
- *Entry 72*[27] *stipulates that lead and various lead compounds listed in entries 28, 29 and 30 shall not be used in textiles, clothing and foot wear.*

Lead oxides are constituents of leaded glass. In the scope of the exemption at hand, lead oxides are, however, not used in wristwatches or any other jewellery in the scope of entry 63, nor are conditions foreseeable where the oxygen sensors or the related equipment may be placed in the mouth by children. The same applies to entry 72, where it is not expected that leaded glass electrodes might be used in textiles, clothing or shoes in the scope of entry 72.

No other entries, relevant for the use of lead in the requested exemption could be identified in Annexes XIV and Annex XVII. Based on the status of these Annexes, the requested exemption would not weaken the environmental and health protection afforded by the REACH Regulation. An exemption could therefore be granted if the respective criteria of Art. 5(1)(a) apply.

[24] ECHA, https://echa.europa.eu/substances-restricted-under-reach?p_p_id=disslists_WAR_disslistsportlet&p_p_lifecycle=1&p_p_state=normal&p_p_mode=view&_disslists_WAR_disslistsportlet_javax.portlet.action=searchDissLists

[25] ECHA, https://echa.europa.eu/substances-restricted-under-reach?p_p_id=disslists_WAR_disslistsportlet&p_p_lifecycle=1&p_p_state=normal&p_p_mode=view&_disslists_WAR_disslistsportlet_javax.portlet.action=searchDissLists

[26] ECHA, https://echa.europa.eu/substances-restricted-under-reach/-/dislist/details/0b0236e1807e30a6

[27] ECHA, https://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:02006R1907-20210825&from=EN:#page=546

Cadmium

With regards to **Annex XIV** of the REACH Regulation, cadmium in general or in compounds is not mentioned in the list of substances that require an **authorisation** for use.

With regards to **Annex XVII** of the REACH Regulation, cadmium is mentioned in a few of the listed restrictions.

Paragraph 1 of entry 23[28] *of Annex XVII refers to cadmium and several of its compounds including cadmium sulphide. Under this entry, several restrictions are mentioned for cadmium and the compounds, among others:*

- *A list of various polymers in which cadmium may not be used unless required in colour for safety reasons.*
- *Shall not be used for cadmium plating*[29] *metallic articles or components of articles used in equipment and machinery in certain branches and applications, e.g. cooling and freezing, food production, etc.*
- *Shall not be used in brazing fillers unless used for safety reasons*
- *Shall not be used or placed on the market if the concentration is equal to or greater than 0.01 % by weight of the metal in metal beads and other metal components for jewellery making, or metal parts of jewellery and imitation jewellery articles and hair accessories, e.g. in wristwatches.*

In the scope of the exemption at hand, cadmium is neither used in polymers nor in platings or as brazing filler, and its use under the exemption is not related to jewellery. The above stipulations are therefore not applicable.

Due to their carcinogenicity, *entry 28*[30] *of Annex XVII does not allow the placing on the market, or use of various substances as such, as constituents of other substances, or in mixtures. Various compounds are mentioned in this respect, including among others, cadmium sulphide.*

As cadmium sulphide in ISE is a constituent of an article as opposed to substance or mixture, the exemption would not weaken the protection afforded by entry 28.

Entry 63 restricts the use of lead and its compounds as part of jewellery articles and provide a list of affected lead compounds, including

- *Cadmium sulphide (CdS), solid soln. with zinc sulphide, copper and lead-doped*
- *Cadmium sulphide (CdS), copper and lead-doped*

[28] C.f. ECHA, https://echa.europa.eu/substances-restricted-under-reach/-/dislist/details/0b0236e1807e2518

[29] 'Cadmium plating' means any deposit or coating of metallic cadmium on a metallic surface

[30] ECHA, https://echa.europa.eu/de/substances-restricted-under-reach?p_p_id=disslists_WAR_disslistsportlet&p_p_lifecycle=1&p_p_state=normal&p_p_mode=view&_disslists_WAR_disslistsportlet_javax.portlet.action=searchDissLists

Cadmium sulphide is not used as part of jewellery in this exemption.

Entry 72[31] *lists substances which are classified as carcinogenic, mutagenic or toxic for reproduction. It refers among others to cadmium and its compounds as listed under entries 28, 29 and 30 (germ cell mutagenic substances) and restricts their use in clothing and textiles. The entries list several cadmium compounds, among others cadmium sulphide.*

Like entry 28, this entry does not address cadmium as it is applied in exemption 1.

To conclude, none of the entries currently listed under REACH would apply to the case at hand. The use of cadmium in ISE cannot be considered to weaken the protection afforded by REACH. The exemption can therefore be renewed if the relevant stipulations of Art. 5(1)(a) apply.

5.4.2. Scientific and technical practicability of substitution or elimination of lead

Scope clarification

JBCE (2020a) requested the renewal of exemption 1(a) with a slightly modified wording that does no longer mention cadmium, as *JBCE has no knowledge of electrodes that contain cadmium*. JBCE (2020b) amended the renewal request specifying the exact applications for which the exemption is needed in the following updated wording. When asked, JBCE (2020c) agreed to a slight rewording that specifically highlights the stem glass as the part of the pH glass electrodes that requires the addition of lead, as other parts of the pH glass electrodes (e.g. the pH-responsive glass) do not require lead (changes in the requested formulation are underlined):

"Lead in the stem glass of pH glass electrodes and ion selective electrodes equipped with a pH glass electrode with complex shape as following:

I) Micro type pH glass electrode
Composite electrode that has a spherical or tube-shaped pH responsive glass membrane with a diameter of 4.0 mm or less and a reference electrode with a liquid junction at a position vertically within 6.5 mm from the tip;

II) Flat type pH glass electrode
pH glass electrode with a flat pH response membrane at the tip of a glass tube with a diameter of 6.0 mm or more;

III) Needle type pH glass electrode
Composite electrode that has a conical pH response membrane with a tip angle of 40 ° or less and with a diameter of 10 mm or more."

The consultants note that no application was received for a renewal of the exemption for cadmium to be used in electrodes in scope of this exemption. Cadmium-selective electrodes traditionally use a cadmium sulphide membrane, as reported by Goodman (2006). The consultants requested three relevant companies to provide information on whether

[31] ECHA, https://www.echa.europa.eu/web/guest/substances-restricted-under-reach?p_p_id=disslists_WAR_disslistsportlet&p_p_lifecycle=1&p_p_state=normal&p_p_mode=view&_disslists_WAR_disslistsportlet_javax.portlet.action=searchDissLists

cadmium may still be needed in this application, from which two responded. (Metrohm 2021c) stated that to their knowledge, cadmium would only be used in cadmium-selective electrodes, adding that Metrohm does not offer such electrodes, and that none of their electrodes contain cadmium. Another company, that did not agree to be named, stated that their ion selective electrodes for cadmium offered on the EU market do in fact contain cadmium sulphide (CdS). Another company stated that the cadmium-selective electrodes in their portfolio are free of cadmium. The latter two responses were received after the draft report for this review had been submitted. Therefore, further in-depth investigation was not feasible.

Substitution of lead glass with lead-free glass

Lead-free glass

The core technical reason argued by JBCE (2020a) why lead-free glass cannot be used in the three types of glass electrodes for which the exemption is requested is that *the intermediate layer required for the bonding of the stem glass and the pH responsive glass cannot form sufficiently when using lead-free stem glass*. Two characteristics of lead are highlighted by JBCE (2020a) that make the use of lead indispensable:

- *Lead is an element which has a low chemical potential, and so rapidly diffuses into different types of glass at the time of bonding to form an intermediate layer even in a short time.*
- *The thermal expansion coefficient of lead glass is close to that of pH responsive glass, and its softening point (625°) leads to a shrinkage rate during the cooling process that prevents higher strain stress, thereby avoiding cracks.*

When asked, JBCE (2021b) specified that the required softening point on the stem tube for bonding pH responsive glass membrane is between 625 and 700 degrees Celsius. Therefore, the physical requirements for a lead-free glass as a potential substitute for lead glass are:

- Thermal expansion coefficient of 95 to 110 × 10−7 K^{-1} ±10 %
- Softening point between 625°C and 700°C

JBCE (2021b) also confirmed that there are no other required physical properties for the glass, and that "*joining is possible as long as the physical characteristics (thermal characteristics) are within the above required range*".

During the evaluation of the exemption request, the consultants identified a few lead-free glass types, including SG036 and Schott 8366, the properties of which are compared with the requirements stated by JBCE in Table 5-5.

Table 5-5: Comparison of required physical properties with lead-free glass types

	TCE	Softening point
Requirements stated by JBCE	$95\text{-}110\times10^{-7}$ K^{-1} ± 10 %	625-700 °C
SG036 lead-free glass	$105x10^{-7}$ K^{-1}	675 °C
Schott 8366 lead-free glass	$92x10^{-7}$ K^{-1}	675 °C

When asked about SG036, JBCE (2021b) explained that the TCE in the specification sheet of SG036 is defined differently from the definition used by JBCE: "*Thermal Expansion you referred as 105 is calculated based on the amount of change between Room Temp (20 deg C) and Set Point (about 485 deg C). TCE definition we are adopting is change between 100 to 300 deg C. Our required TCE range is 95-100 between 100-300 deg C.*" The specification sheet of SG036 also states the TCE between 0°C and 300°C to be $92.5x10^{-7}K^{-1}$, which is also below the required range stated by JBCE.

Concerning the glass type Schott 8366, JBCE (2021b) stated: "*is also one of the candidates, because the softening point seems to be within (close to) our required range." JBCE further explained that since the market demand for pH electrodes is extremely small and unsteady compared to other applications, the procurement of Schott 8366 for pH electrodes is problematic with respect to timing. According to JBCE's knowledge, the next lot of 8366 is planned to be manufactured around Summer 2022. Further, JBCE state that "Even after getting 8366, it will take 5-7 years for us in order to evaluate the lot-to-lot variation, and to optimize our production process.*" It should be noted that the TCE is also slightly below the required range stated by JBCE.

The consultants can follow the argument that both lead-free glass types are not within the required TCE range specified by JBCE and may therefore not be considered "drop-in" replacements for currently used lead glass. Both glasses, however, may be considered as potential candidates to substitute lead glass in the future, if production processes can be optimized by JBCE.

In correspondence with Metrohm, another supplier of pH glass electrodes and ion selective electrodes (cf. Table 5-6), they stated to have *transitioned from lead to barium glass for most types of electrodes*. When asked whether using barium instead of lead glass would also be feasible for JBCE, the applicant stated that *barium is one of the general additives when making glass and that the main purpose of adding barium is not to achieve the same functionality as lead*. Further investigating barium as a potential substitute for lead, the consultants identified scientific literature discussing the substitution of lead glass with barium-containing glass in technical applications as opposed to decorative and other applications of such glasses. (Lityushkin et al. 2000) positively evaluated the substitution potential of high-lead glass with barium-containing glass in the production of light sources, stating: "*Glasses known as barium crystal with BaO mass content up to 20 % and total content of alkaline oxides up to 18 % have recently gained wide acceptance. The TCLE* [thermal coefficient of linear expansion; the consultants] *of such glasses is (99 - 109) x* 10^{-7} *K […]. They have low softening point and viscosity* 10^{10} *Pa sec (510- 520°C), rather high temperature* T_k*-100 (290-300°C), and are easily melted, molded, and heat-treated.*" While the TCE seems to be well aligned with TCE of the pH responsive glass of 95 - 110 × $10^{-7} K^{-1}$, as stated by JBCE (2020a), the softening point deviates from that of the pH responsive glass (625°C) by more than 100 °C.

This difference in the softening point is larger than the difference pointed out by JBCE (2020a) when comparing lead glass to commercially available lead-free glass. *JBCE (2021b) confirmed that the softening point of the described glass was lower than the required range (625 to 700 °C), and added that it is likely not on the market, as the only commercially available stem glass tubes were in the range of 91 to 94 x 10^{-7} K^{-1}.*

Adding to the above, the substitution potential of the described glass with high barium in the lighting industry does not necessarily indicate the feasibility of substitution in pH electrodes, as these are entirely different applications with different requirements.

Market-available lead-free pH glass electrodes

The consultants investigated whether other manufacturers and suppliers of pH meters or glass electrodes offer lead-free products corresponding to the three complex electrode shapes relevant for this exemption renewal request. Eight suppliers that operate within the EU were contacted. Answers were received from five companies and are summarized in **Table 5-6**. Only the names of companies are explicitly stated that engaged in more in-depth discussions leading for relevant conclusions for the exemption evaluation, other companies are not named in the table.

Table 5-6: Summary of company inquiries for lead-free pH glass electrodes

Company	Relevant products	Response	Conclusions
Hanna Instruments	pH electrodes, ISE	All pH glass electrodes are manufactured with lead-free glass	Offer lead-free flat type, micro type and needle type electrodes
Metrohm	pH electrodes, ISE	Transitioning from lead glass to barium glass for many electrode types, incl. flat-type electrodes.	Offer lead-free flat type, micro type and needle type electrodes
Company 3	pH sensors for water	All products are lead-free, but only “standard products” - cannot make statement on the three complex shapes.	No conclusions for the specific electrode types relevant for this exemption request.
Company 4	pH meters incl. with flat electrode	Initially confirmed to manufacture all electrodes without added lead.	Did not respond to request to confirm that the electrode types relevant for this exemption request

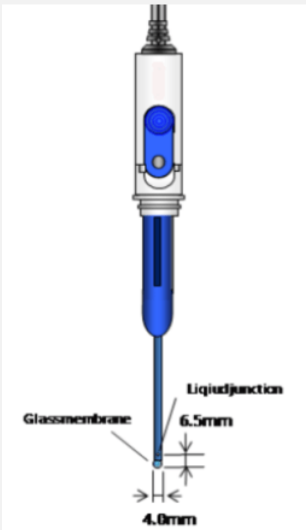
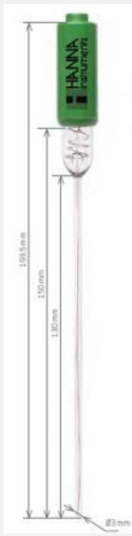
			are indeed manufactured without added lead.
Company 5	Glass products	Insufficient insights into electrode manufacturing	n/a

(Hanna Instruments 2021a) stated that the glass used in their pH electrode production is lead-free and provided information on the properties of the used glass type SG036 (see TCE and softening point data cited in Table 5-5). The company offers flat type, micro type and needle type electrodes in their product catalogue and confirmed that all electrodes are manufactured using the lead-free glass.

(Metrohm 2021a) stated to have transitioned from lead glass to barium glass for most of their pH electrodes, stating that many glass electrodes were already manufactured using barium glass. In further correspondence, (Metrohm 2021a) confirmed that their flat, micro and needle type electrodes are manufactured without the addition of lead.

A comparison of electrode types in the scope of the requested renewed exemption and those offered by Hanna Instruments and Metrohm is provided in the following tables.

Table 5-7: Comparison of Micro Type pH Glass Electrodes

JBCE specified electrode	Example electrode from Metrohm	Example electrode from Hanna Instruments
		
"Composite electrode that has a spherical or tube-shaped pH responsive glass membrane with a diameter of 4.0 mm or less and a reference electrode with a liquid junction at a position	Combined electrode that has a spherical pH responsive glass membrane with a diameter of 3 mm and a reference electrode with a liquid junction at a position	Combined electrode that has a spherical pH responsive glass membrane with a diameter of 3 mm and a reference electrode. *Source: https://hannainst.de/707-*

vertically within 6.5 mm from the tip."	vertically within 7 mm from the tip. *Source: https://www.metrohm.com/de-de/products-overview/60224100*	*hi1093b-kombinierte-mikro-ph-elektrode.html*

Table 5-8: Comparison of Flat Type pH Glass Electrodes

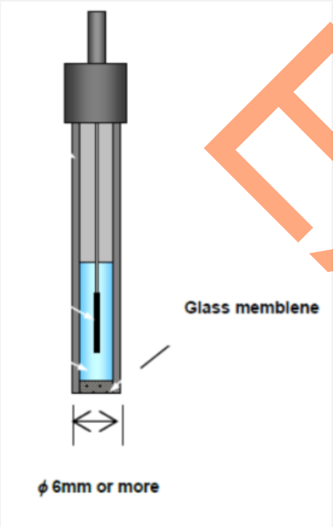
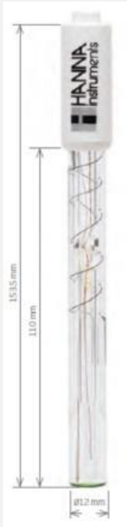
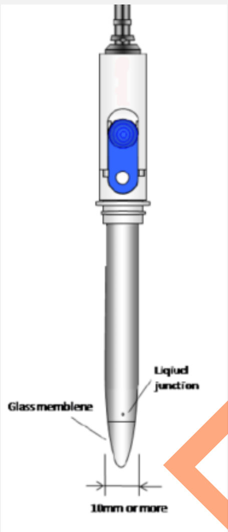
JBCE specified electrode	Example electrode from Metrohm	Example electrode from Hanna Instruments
		
"pH glass electrode with a flat pH response membrane at the tip of a glass tube with a diameter of 6.0 mm or more."	pH glass electrode with a flat pH responsive membrane at the tip of a glass tube with a diameter of 12 mm. *Source: https://www.metrohm.com/de-de/products-overview/60256100*	pH glass electrode with a flat pH responsive membrane at the tip of a glass tube with a diameter of 12 mm. *Source: https://hannainst.de/731-hi14143-ph-elektrode-mit-flacher-spitze-quick-din-anschluss.html*

Table 5-9: Comparison of Needle Type pH Glass Electrodes

JBCE specified electrode	Example electrode from Metrohm	Example electrode from Hanna Instruments
		
"*Composite electrode that has a conical pH response membrane with a tip angle of 40 ° or less and with a diameter of 10 mm or more.*"	Combined electrode that has a conical pH response membrane with a diameter of 6 mm. *Source: https://www.metrohm.com/de-de/products-overview/60226100*	Combined electrode that has a conical pH response membrane with a diameter of 10 mm (at the tip). *Source: https://www.hannainst.com/fc2100-digital-ph-temperature-electrode-for-dairy.html*

When asked to point out technical differences between the JBCE electrodes (using lead) and the electrodes from Metrohm and Hanna Instruments (using no lead), JBCE (2021c) stated: "*there does not seem be significant difference between HANNA instruments and our required pH electrode performance in comparison*" when specifications are compared, and further: *"[…] mechanically, the electrodes offered by Metrohm and Hanna Instruments closely match our specification. However, we believe that it will take 5-7 years for us in order to evaluate the lot-to-lot variation, and to optimize our production process in order to bond our pH responsive glass membrane with necessary property to the lead-free stem glass."*

Only with respect to the micro type pH electrode, JBCE found the measurement range to be different, in that the JBCE reference electrode has a wider measurement range (the specific Hanna Instruments electrode has a measurement range of pH 0-12). JBCE (2021c) provided a table comparing basic specifications of the leaded electrodes described by JBCE with the lead-free products by Metrohm and Hanna Instruments, reproduced in Figure 5-10.

Figure 5-10: Specification comparison of the lead vs. lead free electrodes

	Flat type			Needle type			Micro type		
	Leaded	HANNA	METROHM	Leaded	HANNA	METROHM	Leaded	HANNA	METROHM
model number		HI14143	6.0256.100		FC2100	6.0226.100		HI1093B	6.0224.100
Measurement range	0-12pH	0-12pH	0-13pH	0-12pH	0-12pH	1-11pH	0-14pH	0-13pH	1-11pH
Temperature range	0-50°C	0-50°C	0-60°C	0-60°C	0-60°C	0-60°C	0-60°C	0-50°C	0-60°C
Size	Φ12 x 150mm	Φ12x153.5mm	Φ12x125mm	Φ12x150mm	Φ10x163.5mm	Φ6x98mm	Φ3x151.5mm	Φ3x150mm	Φ3x113mm

Source: JBCE (2021c)

Due to the above information, the consultants consider the basic specifications of the leaded and lead-free electrodes to be very similar. With respect to the measurement range of the micro type electrode, *(Hanna Instruments 2021b) stated that the measurement range of the micro type electrode HI1093B is actually pH 0-14 and therefore equal to the lead electrode*.

With regards to differences that may justify the exemption, *JBCE (2021b) added that they found differences in the measurement performance caused by the difference in the pH responsive glass membrane, stating: "We strongly believe that "the reliability of substitutes is not ensured", because pH responsive glass membrane we use (which has better performance) is not suitable to bonding to the stem glass which HANNA Instruments uses."*

When asked whether non-confidential data can be shared to substantiate these claims, JBCE (2021c) provided a further statement in combination with the data table reproduced in **Table 5-10** and the diagram reproduced in Figure 5-12.

The consultants note that the tested Hanna Instruments electrode (type HI10832) is a lead-free micro electrode, having a spherical tip with a diameter of 3 mm, shown in Figure 5-11 (rotated by 90°).

Figure 5-11: Hanna Instruments electrode HI10832

Source: Hanna Instruments[32]

[32] HI10832 in the Hanna Instruments online shop: https://hannainst.de/2338-hi10832-halo-ph-elektrode-fuer-laboranwendungen.html [last accessed: 16 September 2021]

Table 5-10: Performance comparison of the lead vs. lead-free electrode

Performance parameters		Leaded	HANNA-HI10832 (Lead free)
Sensitivity (%)	pH4.01-pH6.86	99.4	97.3
	pH6.86-pH9.18	99.4	99.2
	pH4.01-pH9.18	99.4	98.2
Repeatability (mV)	pH6.86	1.3	5.3
	pH4.01	1.3	2.2
	pH9.18	1.1	5.3
Responsiveness (mV)	pH6.86	0.7	-0.6
	pH4.01	0.3	-0.1
	pH9.18	0.3	0.4
Alkaline error (mV)	0.1 M NaOH	12.8	12.9
Tap water response time(s)	Tap water	12.2	199.0
Resistance of glass ($\times 10^8 \Omega$)	3.33 M KCl	1.19	5.62

Source: JBCE (2021c)

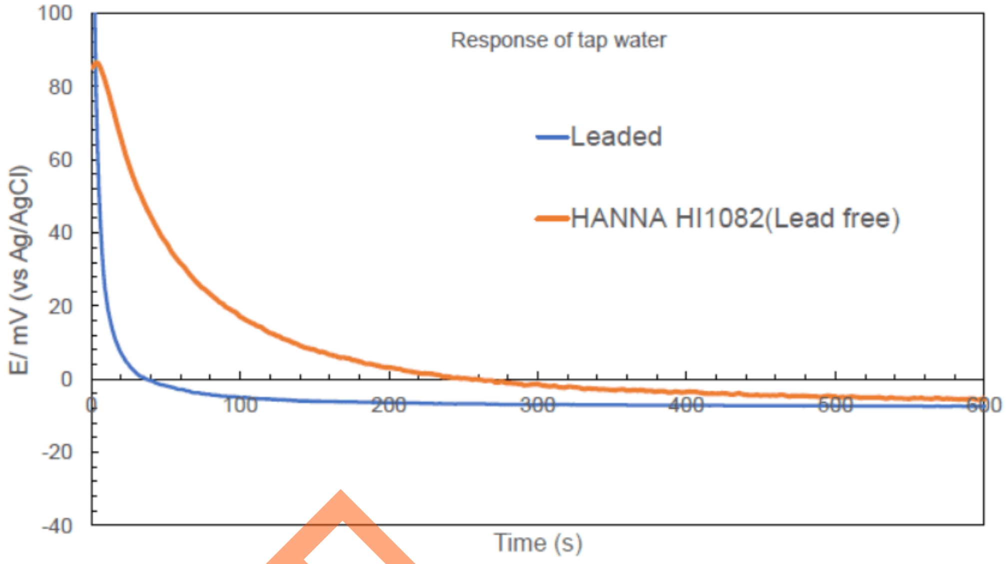
Upon request, JBCE (2021d) stated that *the buffer solutions were compliant with the Japanese Industrial Standard JISZ8802 at 25°C*. JBCE (2021d) also explained that test results in the category repeatability are to be interpreted as follows:

“Regarding the relationship between mV and pH, it follows Nernst equations as described in BN EN60746-1.

- *In the case of 5.3mV is converted to 0.089 pH (at 25 deg C).*
- *In the case of 1.3mV is converted to 0.021 pH (at 25 deg C).*

We strongly believe that the sensitivity is very essential to detect the proper condition in the aquatic ecosystem. For some of fish species, small change of pH value will cause increasing mean percentage mortality.”

Figure 5-12: Response data comparison of the lead vs. lead-free electrode

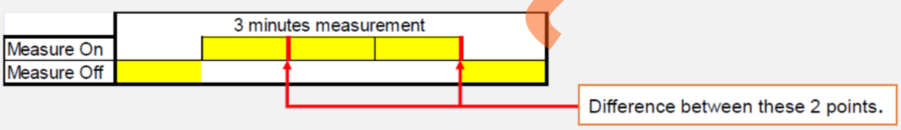


Measured by BS EN 60746 "Expression of performance of electrochemical analyzers", (2003)
10 min measurement of tap water, after the standards solution(pH4) measurement.

Source: JBCE (2021c)

JBCE (2021c) provided measurement conditions for each of the performance parameters reported on in the above figures. The parameters in which the lead-free electrode is shown to perform worse are repeatability and tap water response time. These are defined by JBCE (2021c) as reproduced in Table 5-11.

Table 5-11: Measurement condition for repeatability and tap water response time

Aspect	Definitions provided by JBCE (2021c)
Repeatability (mV)	*"Measure the buffer solutions pH7, pH4, pH9 for 3 minutes each. Difference between reading value at one minute, and at 3 minutes."* 
Response of tap water (T90)	*"Measured by BS EN 60746 "Expression of performance of electrochemical analyzers (2003).* ▪ *10 min measurement of tap water, after the standards solution (pH4) measurement. Then calculate the 90 % of the electric potential at 10 min later after starting the tap water measurement.* ▪ *Then calculate the elapsed time to reach the above 90 % of the electric potential from the starting time of the tap water measurement."*

When asked whether the reduced repeatability and tap water response of the lead-free electrode was directly caused by the electrode's characteristic of being lead-free, JBCE (2021d) stated: *"Adding lead does not contribute to the response time, but adding alkaline metal elements or alkaline earth metal elements do contribute. So, it is necessary to add a large amount of alkaline oxide as glass modifiers. In general, the coefficient of thermal expansion of glass is determined by the property and amount of each oxide component. As alkaline metal elements or alkaline earth metal elements have a large coefficient of thermal expansion, the coefficient of thermal expansion of the response glass membrane increases when it is optimized to perform with higher response time. Therefore, the better performance response glass membrane makes it difficult to bond with lead-free glass."*

When requested to comment on the data provided in the above figures, *confirmed the longer response time of the sensor HI1082, which was stated to be due to the internal design of the sensor, including diaphragm type, electrolyte, and other factors. This was said to be also the case for the sensors HI14143 und FC2100. While Hanna Instruments stated that the response time is not directly causally related to the stem glass being lead-free, as this is not the ion-sensitive component of the pH electrode, they did not disagree with the above explanation provided by JBCE. (Hanna Instruments 2021b) could not immediately provide their own measurement data for the parameter "repeatability", citing the short time available for responding. (Metrohm 2021b) suspected that not the glass type of the electrode, but the composition of the membrane (diaphragm) was the decisive factor to determine the "repeatability" and "response time".*

In the consultants' view, the test result data on one electrode from one supplier does not allow drawing conclusions on the entire range of available lead-free electrodes from several manufactures. When asked whether test data on other lead-free electrodes was available, JBCE (2021d) stated: *"To our best knowledge, Methrom has not been identified as a supplier of lead-free pH electrode. So, we have never evaluated ones from Methrom. We are planning to evaluate, once we get it. However, as long as we confirmed, there is no stock in Japan, so it will take around 2 to 3 months to get."* (Hanna Instruments 2021b) stated that it cannot be assumed that other lead-free electrodes also have a higher response time and lower repeatability.

Elimination of lead

Other technologies that may be used instead of glass electrodes are ISFET electrodes and optical sensing using fluorescent dyes. JBCE provided a range of reasons arguing that currently, neither technology can substitute pH glass electrodes in all applications.

ISFET

The applicant pointed out a range of disadvantages of ISFET compared to glass electrodes that may hinder them to substitute glass electrodes in all applications. Among those, those that appeared most relevant to the consultants were researched further:

a. *The ISFET resin body and semiconductor are susceptible to damage:* This argument appears plausible. However, it should be noted that glass membranes are also susceptible to physical damages to the glass. ISFET electrodes are often advertised by manufacturers and suppliers to be useful in cases in which the more fragile glass electrodes cannot be used. However, glass may be more resistant to the types of damage (i.e. ESD, organic solvents, etching by alkali solutions) described by JBCE.

b. *The shape of ISFET electrodes cannot be completely flat*: The consultants identified market-available ISFET electrodes that are advertised to be of flat shape, however, JBCE correctly pointed out that the sensing element of the electrode is commonly offset from the surface by 0.1 mm. *JBCE argued that air bubbles tend to form in the gap, which disturb the measurement and which cannot always be dispersed by shaking or stirring the measured solution.* This argument appears plausible to the consultants.

c. *ISFET have a limited measurement range*: It was confirmed in a non-exhaustive market research that ISFET electrodes are advertised to measure the range of max. pH 0 to 14 and do not cover the extreme ends of the spectrum (pH -2 up to +16) so that this argument is technically correct and can be followed.

With respect to shapes other than flat type, JBCE (2021a) stated that "*it might be technically possible. However, the microelectrode design cannot accommodate the ISFET chip that is bigger than the 3 mm diameter of the pH sensing area*". When asked whether ISFET packages could be manufactured as small as with a 3 mm diameter, JBCE (2021b) stated: "*To our best knowledge, it is achievable to manufacture less than 3mm ISFET chips (sensing area). However, it needs to be larger size than 3mm when making it the ISFET package*. *To make smaller ISFET package result in sacrificing the durability, because the smaller package size, the less reliability on ESD [electrostatic discharge; the consultants] resistance.*"

As to the needle type, JBCE (2021b) responded "*Needle type can be made if the dent is acceptable." When asked to elaborate in this statement, JBCE (2021c) stated that although needle type electrodes with ISFET exist in the market, not all types of measurement allow the operator to check whether air bubbles exist on the tip of the sensor. JBCE also state to believe that the dent on the tip of the electrode will sometimes cause electrode failure, because debris and particles may easily get stuck there, and brushing them into the sensor may damage it when cleaning the electrode.*

In the consultants' view, the arguments presented by JBCE are plausible. Electrodes fitted with ISFET may be suitable for some applications, but due to the wider measurement range and higher geometric flexibility, they may not substitute glass electrodes in all applications.

Fluorescence

Regarding pH measurement via fluorescent optical sensors, *JBCE (2020a) argued that due to the narrow measurement range of pH 4-10 as opposed to -2 to 16 using glass electrodes, it cannot substitute glass electrodes in all use cases*. The argument of limited measurement range could be confirmed in a non-exhaustive survey of publicly available information. This is also confirmed by other publications such as (Gotor et al. 2017), stating that the working range of fluorescent pH indicators is commonly 2 pH units, which only allows for the determination of pH values in specific pH windows. (Gotor et al. 2017) further explain that researchers have conceived various strategies to broaden the pH range of optical indicators, but that "*among the large amount of optical pH sensors reported, only very few cover a range of ≥10 pH units and all of these systems rely on the combination of dyes*".

In the consultants' view, therefore, the argument provided by JBCE appears plausible, and it seems measurements using fluorescent optical sensors can indeed not substitute glass electrodes in all applications.

5.4.3. Environmental arguments and socioeconomic impacts

The reasons for the requested renewal of this exemption are solely technical and no arguments have been presented that favour pH glass electrodes using leaded glass over its alternatives in environmental or socio-economic terms. JBCE (2020a) stated that *reliable pH electrodes for specific applications could no longer be sold in the EU if the exemption expired and that the EU industry would be disadvantaged and jobs may be lost.* The consultants can follow this line of argument.

With respect to the amount of lead entering the EU market through this exemption, JBCE only provided an estimate for the products of its member companies (14 grams per annum) and did not provide any data on the entire EU market. Goodman (2006) did also not report amounts of lead placed on the market through pH glass electrodes per se, but reported 200 grams of lead from ion-selective electrodes per annum. It is not immediately clear whether this number also includes pH glass electrodes, which can be considered a type of ion selective electrode that is selective for hydrogen.

Due to the fact that lead has been substituted in many pH glass electrodes including in ion-selective electrodes since then, the consultants assume that the order of magnitude of lead entering the EU market through products in scope of this exemption request is likely in the range of a few dozen or hundred grams up to several kilograms. However, this is to be considered a best guess in the absence of factual data.

As glass electrodes can reasonably be assumed to be used by professionals rather than private consumers, it can be reasoned that it is more likely that an appropriate collection and recycling pathway is taken at their end of life. Therefore, the uncontrolled release of glass bound in the glass matrix becomes less likely.

5.4.4. Conclusions

Article 5(1)(a) provides that an exemption can be justified if at least one of the following criteria is fulfilled:

- their elimination or substitution via design changes or materials and components which do not require any of the materials or substances listed in Annex II **is scientifically or technically impracticable**;
- the **reliability** of substitutes is not ensured;
- the total negative **environmental, health and consumer safety impacts** caused by substitution are likely to outweigh the total environmental, health and consumer safety benefits thereof.

In their initial exemption renewal request, JBCE described the progress over the past 14 years and stated that the transition to lead-free glass has been successful for pH electrodes that are simple in shape. The applicant consequently narrowed the scope of the exemption request to three specific and well-defined types of electrodes: those with a flat shape, micro electrodes, and needle type electrodes. Extensive descriptions and evidence on material level was presented to demonstrate why lead-free glass cannot substitute lead-containing glass in the manufacturing of those complex-shaped electrodes. The applicant further narrowed the scope of the exemption request by stating that cadmium is no longer needed in the wording of the exemption, as JBCE are unaware of any electrode that uses cadmium.

Initially, JBCE primarily argued with the lack of manufacturability of three specific types of pH glass electrodes when using lead-free glass. However, during the evaluation, the consultants identified other suppliers that stated to manufacture the same three specific types of pH glass electrodes without the use of lead. Two of them, Hanna Instruments and Metrohm, engaged in extensive communication and provided documents to support their statements. JBCE confirmed that the products from those two manufactures closely match the specifications of the three specific types of glass electrodes for which the renewal of this exemption was requested.

The main argument by JBCE after this point was that “the reliability of alternatives is not ensured”, which JBCE demonstrated with measurement data from a lead-free electrode from Hanna Instruments compared with a lead electrode from an unnamed manufacturer, in which the lead electrode performs better in the tests on “repeatability” and “tap water response time”. *Hanna Instruments confirmed the longer response time of their lead-free electrode.*

In the consultants’ view, JBCE could have performed measurements with other lead-free electrodes, such as the flat and needle type electrodes from Hanna Instruments, in order to evaluate whether these could deliver the required performance. This does not seem to have taken place.

Regarding the elimination of lead through the use of alternative technologies, the consultants can follow the arguments presented by the applicant. ISFET and optical measurement with fluorescent dyes both have limitations compared to glass electrodes. Limitations include the limited measurement range of both methods, limited shape, and susceptibility to damage in the case of ISFET.

In conclusion, the technical barrier described by JBCE – the lack of manufacturability of glass electrodes with complex shapes using lead-free glass – has been disproven in the consultants’ view. Other manufacturers, such as Hanna Instruments and Metrohm, in fact offer such electrodes using lead-free glass. Nevertheless, the secondary argument presented by JBCE – the reduced responsiveness and repeatability – was not disproven, even when requesting the manufacturers of lead-free electrodes to comment. It should be noted, however, that JBCE based this argument on testing a single lead-free electrode by one supplier. Now that JBCE is aware of the fact that more suppliers of lead-free electrodes exist, more comparative testing can be carried out.

The consultants conclude that the available information does not allow the conclusion that lead-free glass electrodes can substitute leaded glass electrodes in all applications and find it reasonable to renew the exemption with the new wording. However, it should be noted that the real-life relevance of the performance differences of lead-free glass electrodes compared to leaded glass electrodes (i.e. repeatability, tap water response time and the ability to measure extreme ends of the pH spectrum) in the various applications of such electrodes could not be unambiguously clarified. In a possible next review, it should be clarified whether these aspects indeed prevent the substitution of leaded glass electrodes in practice and in which specific applications.

The consultants further conclude that a renewal of the exemption for a time frame below the maximum validity period would be sufficient to allow JBCE and other stakeholders to comprehensively test available lead-free electrodes against leaded electrodes and gather information on applications where repeatability, tap water response time and extreme ends of the pH spectrum are of relevance.

Scope and timing of the renewal request

As explained under section 5.4.2 “Scope clarification”, no application was received for a renewal of the exemption for cadmium to be used in ion selective electrodes. After the submission of the draft report, the consultants received information that cadmium is in fact contained in some ion selective electrodes that detect cadmium ions. In this late stage of the review process, it was not feasible to further investigate whether cadmium-free ISE are available for all types of measurements that are conducted using cadmium-containing ISE.

The applicant requested the renewal of exemption 1(a) only for the use of lead in EEE of cat. 9 IMCI. As a result, the renewed exemption would no longer cover the use of cadmium in these appliances, which might cause shortages of ISE containing cadmium and of the respective IMCI for specific measurements.

For cat. 9 IMCI, the expiration date is on 21 July 2024, i.e. the current review started three years before the exemption would have expired. The fact that no request to renew the exemption for cadmium was submitted can be interpreted in two ways: Either, cadmium is no longer needed, or companies that still require the exemption for cadmium were not aware that the review of this exemption is taking place three years ahead of its expiry. For this reason, the consultants consider it reasonable to suggest two options for the wording of the renewed exemption.

For cat. 8 IVD, exemption 1(a) expires on 21 July 2023, and as the request for renewal does not pertain to category 8 devices, the exemption in its current wording could continue to apply to category 8 IVD until 21 July 2023 with the possibility to request further renewal in due time.

5.5. Recommendation

The available information suggests that substitution and elimination of lead in pH glass electrodes and ion selective electrodes equipped with a pH glass electrode is not yet feasible in all cases as the reliability of substitutes is not ensured. In the consultants’ view, Art. 5(1)(a) would therefore allow granting an exemption.

In reference to the section "Scope and timing of the renewal request" above, the consultants suggest two options for the wording of the exemption.

- Option A builds on the stakeholders' and their associations' responsibility to follow with due diligence the developments in the context of regulations which are applicable to their products. It therefore only takes account of the exemption request situation, i.e. that no renewal was submitted for cadmium in ISE applied in cat. 9 IMCI. This approach results in the below exemption wording and timing:

Exemption wording option A

	Exemption	Scope and dates of applicability
1(a)	*Lead and cadmium in ion selective electrodes including glass of pH electrodes*	*Expires on* - *21 July 2021 for cat. 8 other than in vitro diagnostic medical devices* - *21 July 2023 for cat. 8 in vitro diagnostic medical devices*
1(a)-I	*Lead in the stem glass of pH glass electrodes and ion selective electrodes equipped with a pH glass electrode with complex shape as following:* - *Micro type pH glass electrode* *Composite electrode that has a spherical or tube-shaped pH responsive glass membrane with a diameter of 4.0 mm or less and a reference electrode with a liquid junction at a position vertically within 6.5 mm from the tip;* - *Flat type pH glass electrode* *pH glass electrode with a flat pH response membrane at the tip of a glass tube with a diameter of 6.0 mm or more;* - *Needle type pH glass electrode* *Composite electrode that has a conical pH response membrane with a tip angle of 40 ° or less and with a diameter of 10 mm or more."*	*Expires on 21 July 2025 for cat. 9 monitoring and control instruments including industrial monitoring and control instruments.*

- Option B, besides the renewal request from JBCE, takes into account that the renewal request was submitted around three years earlier than the minimum 18 months prior to exemption expiry so that the evaluation of the exemption with relevance to cat. 9 IMCI also took place several years prior to the exemption expiry date. Some stakeholders and their associations despite due diligence may not have been aware of the consequences that might arise from this situation. Considering that this is the first larger review of Annex IV exemptions, this may be justifiable. If the COM wishes to follow these considerations, the consultants recommend renewing the exemption for cadmium in ISE in cat. 9 IMCI as exemption 1(a)(I) for the originally foreseen validity period until July 2024:

Exemption wordings option B

	Exemption	Scope and dates of applicability
1(a)	*Lead and cadmium in ion selective electrodes including glass of pH electrodes*	*Expires on 21 July 2023 for cat. 8 in vitro diagnostic medical devices*
1(a)-I	*Cadmium in ion selective electrodes including glass of pH electrodes*	*Expires on 21 July 2024 for cat. 9 industrial monitoring and control instruments*
1(a)-II	*Lead in the stem glass of pH glass electrodes and ion selective electrodes equipped with a pH glass electrode with complex shape as following:* - *Micro type pH glass electrode* *Composite electrode that has a spherical or tube-shaped pH responsive glass membrane with a diameter of 4.0 mm or less and a reference electrode with a liquid junction at a position vertically within 6.5 mm from the tip;* - *Flat type pH glass electrode* *pH glass electrode with a flat pH response membrane at the tip of a glass tube with a diameter of 6.0 mm or more;* - *Needle type pH glass electrode* *Composite electrode that has a conical pH response membrane with a tip angle of 40 ° or less and with a diameter of 10 mm or more."*	*Expires on 21 July 2025 for cat. 9 monitoring and control instruments including industrial monitoring and control instruments.*

The consultants recommend renewing the exemption for lead in ISE in cat. 9 in the scope of exemption 1(a)(I) in option A or 1(a)(II) in Option B for a period below the maximum possible as suppliers may be able to demonstrate substitutability within a shorter time than the maximum validity period requested by the applicant.

For the next review of this exemption, the consultants expect that the applicant will be able to provide comparative measurement data on the performance of all or most available lead-free pH glass electrodes with the geometric specifications of the electrodes in the scope of the renewed exemption, as such and as part of ion selective electrodes. It should be demonstrated whether any insufficient performance compared to specific lead electrodes (naming manufacturer, product identifier and the electrode geometries) still exist that hinder substitution. It is also expected that the applicant can provide a list of applications in which glass electrodes are needed for measurements of the extreme ends of the pH spectrum (below zero and above 14). Further, in case the "repeatability" and "tap water response time" of lead-free electrodes still do not match the performance of lead electrodes, and this is brought forward as an argument for another renewal of this exemption, the applicant is expected to provide technical insight into the relevance of these parameters in specific real-life applications where the substitution with lead-free electrodes is prevented due to their subpar performance in these two test criteria.

5.6. References

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