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Wiesbaden, 31.Mar 2008

Prolongation of exemption 5 "lead in glass of cathode ray tubes in electroniccomponents and fluorescent tubes"

Dear Sirs,

Attached please find our comments to the stakeholder consultation on adaptation to scientific and technical progress under Directive 2002/95/EC.

With our comments we do want to support the prolongation of exemptions 5. A termination of above mentioned exemptions would result in the end of life of our products since at the moment there are no feasible substitutes available.

Attached please also find the report of Dr. Paul Goodman from ERA Technology LTD. (UK) who supported us with his independent opinion on this matter.

The attached documents were prepared in collaboration with Fraunhofer IPA Stuttgart.

In case of further question please contact us.

Best regards

i.A. Carsten Jaehnisch

General questionnaire for exemption 5

For which substance(s) or compound(s) should the requested exemption be valid?	Lead bound in glass for Channel Photomultiplier (CPM).
What is the application in which the substance/compound is used for and what is its specific technical function?	CPM have a photocathode screen which is sensitive to light. When a photon strikes this thin coating, an electron is emitted and travels towards an anode at the other end of the CPM. However due to its novel shape, the electron will strike the wall of the CPM tube where it causes the emission of many more secondary electrons. The tube consists of lead based glass. The CPM is suited for a variety of life science applications, medical equipments and high-end physics. This high performance device offers substantial advantages for analytical applications like emission-spectroscopy, flouroscopy, atomic-absorption as well as bio- and chemoluminescene.
What is the specific (technical) function of the substance/compound in this application?	CPM – Channel Photomultiplier uses a specially developed lead oxide glass. This glass has the special characteristics of forming a defined semiconductive surface while being reduced in hydrogen. The semiconductive surface is needed as a functional layer for producing secondary electrons in the photomultiplier tube.
Please justify why this application falls under the scope of the RoHS Directive (e.g. is it a finished product? is it a fixed installation? What category of the WEEE Directive does it belong to?).	i.) Why does a CPM fall under the scope of the RoHs Directive: - It does use electric current for its primary function. - It is built into an application which falls under the scope of the RoHS ii.) What category of the WEEE does it belong to: - Category 8: Control and measurement device - Category 9: Medical Equipment

What is the amount (in absolute number and in percentage by weight) of the substance/ compound in: **i) the homogeneous material** **ii) the application** **iii) total EU annually for RoHS relevant applications?**	i) The glass contains 41% Pb (weight%) ii) Product type 9mm: 0.861g Product type 13mm: 1.025g Product type 19mm: 2.214g iii) The total amount put on the EU market is 45.1 kg
Please check and justify why the application you request an exemption for does not overlap with already existing exemptions respectively does not overlap with exemption requests covered by previous consultations.	Not applicable
Please provide an unambiguous wording for the (requested) exemption.	Lead in glass of Channel Photomultiplier.

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Specific questions for exemption 5

"Lead in glass of cathode ray tubes, electronic components and fluorescent tubes"

PerkinElmer request the prolongation of the existing exemption 5. Especially the part "Lead in glass of electronic components"

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1. **Please specify in detail the "electronic components" in the wording above where lead is used in glass.**
   CPM – Channel Photomultiplier uses a specially developed lead oxide glass. This glass has the special characteristics of forming a defined semi-conductive surface while being reduced in hydrogen. The semi-conductive surface is needed as a functional layer for producing secondary electrons in the photomultiplier tube (see figure 1).

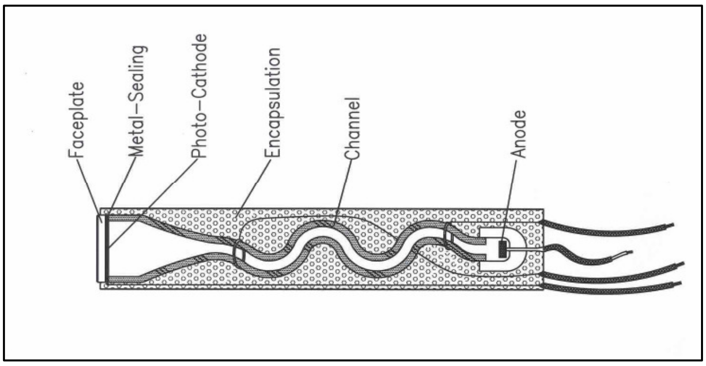

Figure 1: Construction of Channel Photomultiplier

More information about the function of a CPM can you find under:
http://www.microscopy.fsu.edu/primer/java/digitalimaging/photomultiplier/channel/index.html

2. **Please state the amount of lead used per application, the lead content in the homogeneous material, the annual production volume as well as the number of applications put on the EU market annually in applications falling under the scope of RoHS for**
   a. **cathode ray tubes - not applicable**
   b. **electronic components**
   c. **fluorescent tubes - not applicable**

bi) amount of lead per application:
The glass contains 41% Pb (weight%)

bii) lead content in the homogeneous material:
Product type 9mm: 0.861g
Product type 13mm: 1.025g
Product type 19mm: 2.214g

biii) annual production volume:
The total amount put on the EU market is 45.1 kg

3. **Please provide detailed information about the specific function and related performance criteria of lead in glass for**
   a. **cathode ray tubes - not applicable**
   b. **electronic components**
   c. **fluorescent tubes - not applicable**

Channel Photomultipliers (CPM) operate according to the same general principles as Photomultiplier tubes (PMT), but are built from different materials and lack the PMT´s internal parts. Photons enter both devices through an entrance window and hit the photocathode. Inside the photocathode the photons are converted into electrons, which are emitted into the vacuum tube. These electrons are accelerated by an electrical field through a multi-curved path through the vacuum chamber toward an anode (see figure 2).

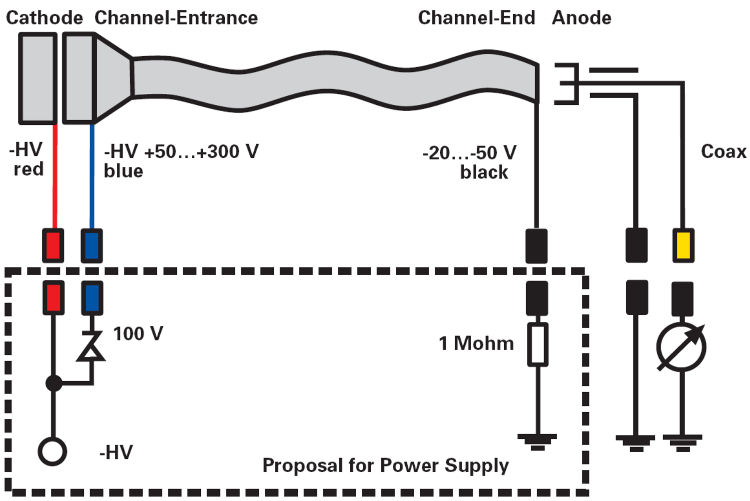

Figure 2: Physical specification

In a PMT, electrons hit specially coated and separately charged metal dynodes on their way through the glass vacuum tube, and these dynodes emit secondary electrons. The electrons travel to the next dynode, which emit still more electrons, and the multiplication is repeated up to approximately ten times to produce a gain on the order of one million. Finally the electrons are collected and read out at the anode.

A CPM works in much the same way, but without the PMT's multi-element dynode structure. Instead, the device contains a single hollow curved tube (in the follow named as channel) internally coated with a semiconductor. On the way from the photocathode to the anode the electrons hit the curved channel walls several times and at each collision with the channel wall several secondary electrons were created. Like an avalanche more and more electrons were generated at each collision. Every electron emitted from the photocathode can multiply up to a gain of 100 millions or more.

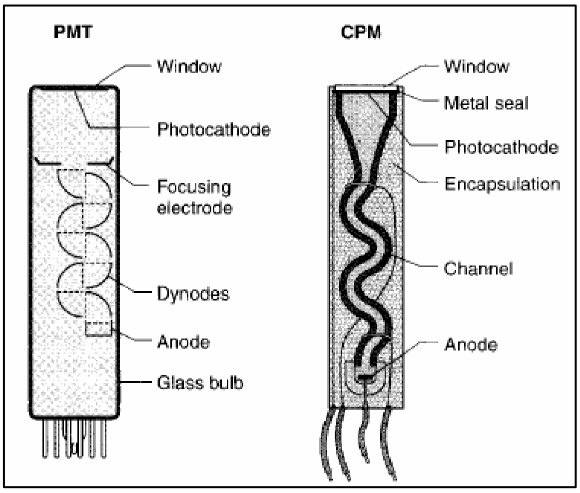

Figure 3: Construction of the classical photomultiplier tube (left) is compared to that of the Channel Photomultiplier (right). The CPM is small, simple and rugged (far right).

The CPM, however, operates with far less "background noise" – the current generated by the device itself in absence of photocathode illumination. In the PMT, emissions from the photocathode *and* the dynodes produce significant dark current. In the CPM, the vacuum channel is virtually silent, and dark counts originate from the photocathode alone. Also, in the CPM, there is no leakage current of the kind that results from voltage applied to dynodes. Hence, a CPM's background level is generally one or two orders of magnitude lower than in a PMT (see figure 4).

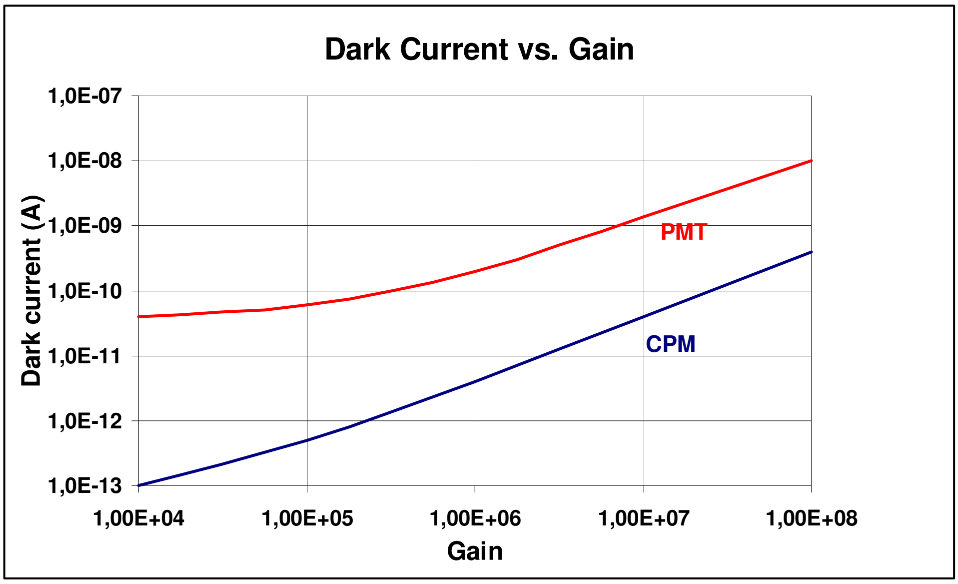

Figure 4: Background level of a CPM compared with a PMT

This result in a high dynamic range and significantly better detection limits. The device can operate with a low and stable background in *both* photon-counting mode and DC-analog mode. The CPM is more reliable than the PMT, because it is 50 times more immune to external magnetic fields.
Unlike the PMT, the CPM can apply an incorporated electronic shutter, which prevents excessive illumination and enables time-resolved measurements. This is especially important for applications like blood analyses when samples are activated with short-time high light flash lamps or lasers and might overexpose the detector. PMTs require mechanical shutters, which are far slower and wear out.

4. What technical characteristics do substitutes need to fulfil as a minimum requirement?

Substitutes have to fulfil follow technical characteristics:
- high gain
- low dark noise and counts
- detectior sensitivity
- dynamic range
- time resolution
- large sensitive area
- immunity to magnetic fields
- single photon detection capability
- UV sensitive
- IR sensitive

The following table shows a comparison between CPM and other technologies, with the result that the CPM is in certain areas outstanding and in the sum of properties absolutely unique.

CPM compared with other technologies:

	Performance Characteristics for Low light level applications (Luminescence)					
Alternative Products	high Gain	low dark noise (DC background)	low dark counts	Detection sensitivity	Dynamic range	Time resolution
CPM	++	++	++	++	++	+
PMT glass tube	++	+	+	+	++	+
PMT metal can	+	+	+	+	+	++
APD	-	-	-	-	-	++
SPCM (APD based)	++	na	o	+	+	++
Hybrid Photon Detector	o	o	++	++	o	++
CMOS Single Photon APD	++	--	--	o	-	++

Table1a: Performance characteristics for low light level applications

Alternative Products	Performance Characteristics for Low light level applications (Luminescence)				
	large sensitive area	immunity to magnetic fields	Single Photon detection capability	UV sensitive	IR sensitive
CPM	+	++	++	++	o
PMT glass tube	++	-	++	++	o
PMT metal can	++	++	++	++	o
APD	-	++	-	-	o
SPCM (APD based)	--	++	++	-	o
Hybrid Photon Detector	+	++	++	+	o
CMOS Single Photon APD	+	++	++	--	o

Table 1b: Performance characteristics for low light level applications

5. **Please provide evidence that manufacturers have put effort in research on alternatives for lead. What are the alternatives to lead and which ones are (likely to be) used as substitutes? Are there any results about strengths and weaknesses expressed in results relating to (technical) performance criteria?**

PerkinElmer put effort in research. But an alternative material for the PbO-glass has to fulfil all these characteristics:

- resistivity
- high emission of secondary electrons
- low dark noise
- ultrahigh vacuum compatible
- functional surface
- high voltage stability over a long period
- stability against electronic bombardment

At the moment several materials are under review (see figure 5a and 5b) but none of the below listed materials are considered to be suitable. As it is obvious every other material is at least in one category worse than PbO-glass.

Alternative Products	Performance Characteristics					
	high secondary emission	Appropriate resistivity	Control resistivity	high voltage stability	Stability against electron bombardment	Vacuum outgasing due to electron tube requirements
Lead oxide glass	++	++	++	++	++	++
Volume conductive ceramics (ZnO - TiO)*)	o	+	o	+	+	--
Volume conductive glass **)						
a-Si coating	+	?	?	?	?	?
CrO coating	+	?	-	?	?	?
SnO coating	+	?	-	?	?	?
Sb alcalides	++	-	--	-	+	+

*) Murata Ceratron taken from market
**) CdO-BiO not RoHS --> not regarded
**) AsO-TeO-TlO toxic --> not regarded
**) V based glass not usable since ionic conductivity

Table 2a: Performance characteristics

	Performance Characteristics					
Alternative Products	high temperature UHV bakable	smooth emissive layer and resistivity inside channel	compatible to photo cathode material	long life stability	no dark noise background	high gain
Lead oxide glass	++	++	++	++	++	++
Volume conductive ceramics (ZnO - TiO)*)	o	o	-	?	++	++
Volume conductive glass **)						
a-Si coating	-	--	?	?	?	?
CrO coating	++	--	?	?	?	?
SnO coating	+	--	?	?	?	?
Sb alcalides	--	--	++	?	-	++

*) Murata Ceratron taken from market
**) CdO-BiO not RoHS --> not regarded
**) AsO-TeO-TIO toxic --> not regarded
**) V based glass not usable since ionic conductivity

Table 2b: Performance characteristics

Table 2a and 2b shows that at time no other material as lead oxide glass is available which fulfil all items under point 5.

6. Are manufacturers still investigating alternatives?

Today there are no alternative materials available which fulfil all requirements (see item 5). The assessment of our evidence is confirmed by independent experts.

a) Report prepared for **PerkinElmer** (see appendix A)

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b) Review of Directive 2002/95/EC (RohS) Categories 8 and 9 – Final Report p.55 (CPM use the same physical effects like MCP)

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PerkinElmer continues to search for substitutes of the environment and human hazard substance lead.

7. Assuming the current exemption will be given an *expiry date*, what date do you think is technologically feasible for industry?

Alternative material or design that would provide the required performance can not be envisaged from current research. Therefore an expiry date can not be predicted.

Appendix A

ERA Project No. 043122289
ERA Report No. 2008- 0164

Review of Directive 2002/95/EC, RoHS exemptions Nos 5 and 7c- ERA's opinion on their validity for two specific applications

Please refer to: 043122289 PE Report final.pdf

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27 March 2008

Report: Review of Directive 2002/95/EC, RoHS exemptions Nos 5 and 7c- ERA's opinion on their validity for two specific applications

ERA Project No. 043122289
ERA Report No. 2008- 0164

1. Background

The European Commission is currently reviewing D2002/95/EC (RoHS) and this includes the review of all existing exemptions listed in the Annex. Perkin Elmer relies on exemptions 5 and 7c and is submitting evidence to demonstrate that these exemptions will be needed in the foreseeable future. Perkin Elmer has asked ERA to provide its independent opinion on the technical issues relating to these exemptions and in particular to the reasons for use of lead in Channel Photomultipliers "CPM" (exemption 5) and lead in pyroelectric detectors (exemption 7c).

The main applications for CPM and pyroelectric detectors are as electronic components in electrical equipment within WEEE categories 8 and 9 although they may also be used in products within other WEEE categories. Categories 8 and 9 are currently excluded from the scope of RoHS but ERA concluded in its review for the European Commission that it would be possible to include these categories in the scope of RoHS. ERA also determined that Category 8 and 9 products would require several of the existing exemptions in the RoHS Annex. ERA did not investigate the technical issues relating to these in detail as part of its review as the exemptions listed in the Annex of the RoHS Directive were going to be reviewed by the European Commission under a separate contract. At least some of the existing exemptions will be important for category 8 and 9 equipment manufacturers if these products are included in the scope of RoHS.

2. CPM

Channel photomultipliers (CPM) are a fairly new type of device that is very sensitive light detector. These are so sensitive that they are able to detect single photons and are used mainly as components in analysis instruments and in medical applications. CPMs have been developed for use in specific applications that require very high sensitivity and accuracy and which is superior to other technologies such as photomultiplier tubes (PMT) and various types of Avalanche Photo-diodes (APD).

CPM have a photocathode screen which is sensitive to light. When a photon strikes this thin coating, an electron is emitted and travels towards an anode at the other end of the CPM. However due to its novel shape, the electron will strike the wall of the CPM tube where it causes the emission of many more electrons. This occurs many times along the tube resulting in very large amplification of the original weak signal. The electron amplification effect is also utilised in microchannel plate (MCP) detectors which are also made of lead glass with a hydrogen reduced semiconducting surface layer.

2.1 Alternative detectors

Alternative types of detectors cannot duplicate the combination of performance characteristics of CPM. The important characteristics include in particular:

- High Gain – CMP is one of the best
- Low dark noise – This is the most important advantage of CPM which has the lowest noise output with no light input. This is due to its novel design. Dark noise can be as low as 1 count per second (cps) with CPM whereas PMT dark noise can be ~100 cps.
- Low dark counts – CPM is one of the best detectors although hybrid photon detectors also give low dark counts but hybrid photon detectors are less accurate as the dark noise characteristics are inferior
- Detection sensitivity – CPM are one of the most sensitive types. CPM amplification can be as high as 10^8 times whereas PMTs are typically ~10^6 – 10^7 times and APDs are much less sensitive (~10^3 times). Hybrid photon detectors can be very sensistive but amplification is less than CPM.
- Dynamic range – CPM and glass tube PMT have the widest dynamic range (light intensity range)
- UV sensitivity – only CPM and PMT have high UV sensitivity

CPM has other advantages over other types of detector, for example, due to its design; CPM requires far fewer additional electrical components than PMT. CPM, photomultiplier tubes and APD are all commercially available products whereas hybrid photon detectors are available only as relatively new research tools.

2.2 Substitute materials for lead in glass

Lead based glass is used for CPM because the glass surface can be chemically reduced by hydrogen gas to form a semiconducting surface layer. When an electron strikes this layer, multiple electrons are emitted and when each of these strikes the opposite semiconducting wall, more electrons are produced resulting in very large multiplication of the original electron. Gains of up to 10^8 times are possible. The CPM tube has a characteristic shape, has a high vacuum and the only feasible production technique is currently with a glass tube. Research on ceramic CPM has been carried out but not commercialised however these also use an internal lead-glass coating.

There are many glass formulations used by the electronics industry but the glass required for this application must have a specific range of characteristics. It must be capable of having its surface reduced to a semiconductor layer by the action of heat and a reducing gas such as hydrogen. The semiconductor must also have a high secondary electron emission capability. To date only lead based glass has been found to show all of the essential properties and this is due to the chemistry and physical properties of lead which are different to all other materials that can be used in glass formulations. The essential requirements are:

1. Capable of producing a readily formable glass
2. Does not cause crystallisation of the glass at fairly high concentrations
3. Is not susceptible to degradation by atmospheric moisture
4. Is chemically reduced to a thin surface semiconductor layer by a reducing gas
5. The semiconductor must be stable inside the CPM and have high secondary electron emission capability.

These requirements are considered separately although all five are essential

1. The glass is fabricated into a precise and fairly complex shape and has various types of optical windows and internal metal electrodes attached. Fabrication requires a glass which is capable of having glass/metal seals (to maintain the vacuum with electrical connections), capable of attachment to various types of optical window and has a melting point that allows accurate fabrication of the required shape. Several types of glass could be used to fabricate complex shapes including soda- and lead- glasses but the thermal coefficient of expansion (TCE) of some lead-free glasses is incompatible with some of the types of windows used such as magnesium fluoride.

2. “Glass” is an amorphous material which softens at a high temperature to allow fabrication and does not crystallise on cooling but maintains its amorphous form. Glass formulation is complex as the combination of ingredients must be balanced to ensure that the amorphous form is stable and will not crystallise. Some additive elements can be used in glasses only at low concentrations because they have a tendency to cause crystallisation. The semiconductor forming ingredient must be capable of being added at a fairly high concentration to allow the formation of a semiconducting layer at the internal surface. Many of the elements that might be investigated as potential alternatives to lead when added to glass at high concentrations cause crystallisation which destroys the glassy structure and so are unsuitable.

3. Some novel lead-free glasses have been developed such as phosphate based materials but these are susceptible to hydrolysis by water which would cause the structure to degrade.

4. One of the most important characteristic for CPM is the ability to be reduced to a semiconductor by a reducing gas at a temperature below the softening temperature of the glass. The two main reducing gases are hydrogen and carbon monoxide. Carbon monoxide is a much more potent reducing agent than hydrogen and will readily reduce many types of oxide to their metals and so it is not used to produce semiconductor coatings for CPMs. Hydrogen reduction is more easily controlled and so is preferable for the formation of semiconductor coatings. The reduction process requires the removal of a small proportion of the oxygen atoms from the surface layers of the glass by hydrogen, which forms water vapour:

$$nPbO + H_2 \rightarrow n\text{-}1PbO + Pb + H_2O$$

n = a fairly large number.

5. Few semiconducors have been found to be both stable inside CPM tubes and have high secondary electron emission. Only a few types of semiconductor films could be produced inside CPM tubes although several others can also be applied as coatings, for example by chemical vapour deposition techniques. However research has shown that no semiconductors, including those deposited internally as coatings, other than partially reduced lead oxide give the required combination of electron emission performance and stability inside the CPM (more details on internal coatings in section 2.3)

2.3 Semiconducting Coatings

Some research into the deposition of semiconducting coatings has been carried out but none of the materials evaluated has been found to be suitable. Several including silicon, chromium oxide and tin oxide semiconducting coatings were found to be unsuitable as the emission of secondary electrons is far weaker than from lead-based glass. Antimony compounds with alkali metals are used as photocathodes in PMTs and these were found in CPMs to be reasonable electron emitters but had poor stability and rapidly deteriorated during use.

2.4 CPM conclusions

The most common elements used in glasses as major constituents are:- Na, K, Ca, Ba, Al, Si, B and Pb. Of these elements, hydrogen is capable of reducing only lead oxide to give a semiconductor. Hydrogen will readily reduce certain metal oxides but none tested to date have the required combination of glass-forming and semiconductor characteristics (copper oxide, for example cannot be added to glass at high enough concentrations as it would cause crystallisation and it also tends to be completely reduced hydrogen to copper metal). Nobel metals such as silver are unsuitable, as they do not form stable oxides. Hydrogen can reduce oxides of more reactive metals such as zinc but only at temperatures well above the glass softening point and so these are unsuitable. Research with many other materials, some applied as coatings has not identified an alternative that has suitable characteristics. Therefore the choice of metals that can be added to glass is limited and of those that could form suitably active semiconductors, only lead has been found to date to provide the required physical properties.

3. Pyroelectric detectors

Pyroelectric detectors use materials that generate a voltage when their temperature changes. This property is very similar to piezoelectric materials which generate a voltage when a force is applied. Pyroelectric materials are also piezoelectric and one of the most effective piezoelectric materials, lead zirconate titanate (PZT) is also widely used as a pyroelectric material. PZT is a useful pyroelectric material because its composition can be varied to tailor its physical and electrical properties. There are many other materials known that have pyroelectric properties but these have different characteristics. Some of these alternatives are used commercially as heat sensors but these cannot be used as intruder or movement detectors because of their characteristics.

3.1 Intruder and movement detection

Intruder detection using pyroelectric detectors relies on the very weak infrared emission from the human body which is usually at a higher temperature than the surroundings. The temperature difference can be fairly small and so the infrared energy to be detected is at very low levels and so very sensitive detectors are required. Various techniques such as the use of special lenses are used to optimise sensor sensitivity although these are not relevant to this submission.

3.2 Types of pyroelectric sensor material

The pyroelectric material that is used in intruder sensors must have a range of specific characteristics:

- Very high sensitivity – requires a high dielectric constant and pyroelectric coefficient

- Curie temperature of about 200°C – The reasons for this are fairly complex. Pyroelectric (and piezoelectric) materials are manufactured by application of a strong magnetic field across the material which is held at a temperature above the Curie temperature. This aligns the dipoles within the material giving it the pyroelectric property. The material is then cooled to below the

Curie temperature to retain the dipole structure. Sensitivity is greatest at temperatures close to the Curie temperature but the rate of randomisation of dipoles increases with temperature (this degrades the performance) and so pyroelectric materials are usually used at temperatures of at least 100°C below the Curie temperature. Short periods close to the Curie temperature, for example during a soldering process are not detrimental but longer periods at commonly used adhesive cure temperatures would cause degradation of low Curie temperature materials.

- Stability in use – PZT is a multicrystalline material that is resistant to sudden temperature changes and is not damaged. It is also chemically inert and so is unaffected by moisture and environmental pollutants.

The properties of more common pyroelectric materials are used in heat sensors are compared in table 1.

Table 1. Comparison of more commonly used pyroelectric materials

Material	**Curie temperature °C**	**Dielectric constant**	**Pyroelectric coefficient ($10^{-8}.cm^{-2}.K^{-1}$)**	**Stability/comments**
Ideal material	~200	High	High	Inert
PZT	200	250	5.0	Very stable
LiTaO3	618 - 620	54	2.3	Stable but less sensitive / used as heat sensor
$Sr.BaNb_2O_6$	115	380	6.5	Stable but Curie temperature too low
Triglycine sulphate	49.5	38	3.5	Water soluble so affected by moisture. Curie temperature very low.
Polyvinylidene fluoride	120	11	0.24	Inert but low sensitivity and low Curie temperature

Of the materials listed in table 1, lithium tantalate ($LiTaO_3$) is used commercially in heat sensors but research by Perkin Elmer has found that it is not suitable for intruder detection because it generates excessive noise when exposed to fast changes in temperature. This noise consists of spontaneous and random voltage spikes that would cause false alarms. This effect is less important when $LiTaO_3$ is used as a heat detector which monitor temperature because the output can be averaged and so noise is cancelled out. The noise is due to the very high electrical resistance of $LiTaO_3$. PZT has a lower electrical resistance and as a result much less noise.

Strontium barium niobate is a newer pyroelectric material, not widely used commercially because its Curie temperature is too low for components to be soldered and also degrades at adhesive cure temperatures. Research into other novel pyroelectric materials is being carried out. For example "$NaBiNbTiO_3$" is a new pyroelectric material but this has too low a Curie temperature (112°C) for commercial use as a movement sensor. Bismuth vanadate is also new and has a Curie temperature of ~ 500°C which is high for movement sensors and so has relatively low sensitivity at room temperature, unlike PZT.

3.3 Pyroelectric movement sensor conclusions

Many materials exhibit pyroelectric properties but none have yet been developed that have the necessary combination of characteristics that are able to provide the required performance and reliability of PZT in intruder and movement detection applications. This is because PZT is one of the most stable and sensitive materials which functions without random noise outputs that would cause false alarms. Therefore there is currently no commercially available lead-free substitute material.

Report prepared by:

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