

Clarification Questionnaire Exemption No. 32

Exemption for „Lead oxide in seal frit used for making window assemblies for Argon and Krypton laser tubes“

Abbreviations and Definitions

CET	Coefficient of Thermal Expansion
EEE	Electrical and Electronic Equipment
Lumentum	Lumentum Operations LLC
Pb	Lead
PbO	Lead Oxide
RoHS	Directive 2011/65/EU on the Restriction of Hazardous Substances in Electrical and Electronic Equipment

Background

The Oeko-Institut has been appointed by the European Commission, within a framework contract¹, for the evaluation of applications for exemption from Directive 2011/65/EU (RoHS), to be listed in Annexes III and IV of the Directive.

Your organisation (Lumentum) has submitted a request for the renewal of the above-mentioned exemption, which has been subject to an initial evaluation. A summary of the main argumentation for justifying the request is provided below as a first basis to be used in the stakeholder consultation planned as part of this assessment.

Please read the summary of the argumentation provided to ensure that your line of argumentation has been understood correctly and provide answers to the questions that follow that address aspects requiring additional information and/or clarification.

1. Summary of argumentation of applicant on the justification of the exemption

1.1. Background

Lumentum applies for the renewal of Ex. 32 of Annex III of the RoHS Directive:

“Lead oxide in seal frit used for making window assemblies for Argon and Krypton laser tubes”

Lumentum applies for the exemption in its current formulation and requests it to be renewed for 5 years for RoHS annex I categories 6, 8 and 9. In its application, it addresses only argon lasers and it is not clear if the exemption is still needed for krypton lasers or not.

¹ The contract is implemented through Framework Contract No. ENV.B.3/FRA/2019/0017, led by Ramboll Deutschland GmbH.

1.1.1. Volume of lead to be placed on the EU market through the exemption

The content of lead in the frit used for making window assemblies in argon lasers is >50% of the homogeneous material (%weight). The average annual Lumentum usage of PbO in the sealing glass frits of Argon lasers is about 200 g. However, only a fraction of that enters the EU. Based on Lumentum's direct shipments, only 17g of PbO enters the EU market annually in argon lasers. This amount has not changed since Lumentum's previous application for the renewal of RoHS exemption 32 in 2015.

1.2. Technical description

Argon laser products are used as coherent light sources in a broad range of critical applications, a majority of which are in research, bioinstrumentation and semiconductor manufacturing. This includes flow cytometers, DNA sequencers, and hematology equipment. Instruments are used internationally by both government and private sector agencies for health care, drug discovery, and research applications. In semiconductor manufacturing, argon lasers are used in inspection equipment.

1.3. Applicant's justification for the requested exemption

The lead-oxide glass provides a critical thermo-mechanically stable and vacuum-tight seal between the optics and laser tube in applications of relevance to this exemption. Lead-oxide as a raw material lowers the melting temperature of the solder glass/glass frit. This glass frit joins glass of a mirror to the laser metal tube without thermally damaging complex coating layers of the mirror. The softening point of the lead-oxide based material occurs at a narrow temperature range around 420°C and does not thermally damage the nearby fragile components being joined.

The processing temperatures are restricted by the potential of damage to the components, primarily the optics. Because the optics utilize complex multilayer coatings (>30 layers) using higher temperatures or longer processing times is not advised by the supplier of the optics. The coating fabrication process only allows for stabilization of the key optical properties to 500°C. Processing at temperatures above 500°C will cause failure of the coatings.

Additionally, the material has a coefficient of thermal (CET) expansion closely matched to the components for stress-free sealing. The seal frit is required to have a thermal expansion coefficient at 7.0 to 8.0 10⁻⁶/K to match to the components for stress-free sealing.

1.3.1. Availability of alternatives (Substitution or Elimination, roadmap to substitution, reliability of substitutes)

LUMENTUM has not identified lead-free glass that meets the softening temperature and CET requirements specified above.

Commercially available alternatives to the leaded glass sealing material (frits) are bismuth based. Bismuth based glasses have significantly higher (540°C) melting temperatures than the Pb based frits. In trial builds the lead-free frit did not produce a consistent good flow of the frit material (see illustration photo's in application). To further substantiate this, Lumentum provides a comparison of the coefficients of thermal expansion and sealing temperature of leaded and bismuth based lead-free sealing glass and sealed components in the argon lasers in Table 1 of the application.

Thus, the bismuth-oxide based material (frit) is not considered a viable alternative at this time.

1.3.2. Environmental and health arguments (also LCA aspects)

Environmental arguments were not raised as the main justification for this exemption.

2. Clarification Questions

1. Lumentum refers in the application to CET as Coefficient of thermal expansion. In other exemption assessment CTE is referred to as the abbreviation for this term. Can you confirm that CTE can be used in this case as well?

CTE is the correct abbreviation for a coefficient of thermal expansion.

CET was a typo.

2. Lumentum request the exemption for both laser types, whereas the application only provides detail on argon lasers.
 - a. On what basis does Lumentum assume that the exemption is still needed for krypton lasers?

In section #2 of our application we indicated that Lumentum does not manufacture krypton laser tubes. We don't have any visibility if exemption #32 is still needed for krypton lasers. However, in previous RoHS exemptions reviews Coherent applied for RoHS exemption #32 extension for their krypton and argon lasers.

- b. Can Lumentum name suppliers of krypton lasers to allow their invitation to the stakeholder consultation?

We recommend to reach out to Coherent, as Coherent previously applied for RoHS exemption #32 extension and still advertises krypton and argon lasers on their website.

Besides Coherent, also Lexel-Cambridge (lexellaser.com) and LASOS (lasos.com) manufacture krypton lasers.

3. Is Lumentum aware of additional manufacturers of argon lasers or is Lumentum the single supplier of such lasers to the EU?

Besides Lumentum, also Coherent, Lexel-Cambridge and LASOS manufacture argon ion laser tubes. All of the above manufacturers are likely selling and shipping either directly or indirectly around the globe, including to the EU.

4. Lumentum states that the bismuth-oxide based material (frit) is not considered a viable alternative at this time, since Lumentum's optics are not designed to be subjected to temperatures beyond 500°C. However, Lumentum also refers to performance of the lead-free frit during trial builds. Please explain how the trial build was performed in this respect also explaining at what temperature/s it was performed.

The trial builds with bismuth-oxide based frits were performed at different temperatures starting at 480°C. A good melting, per visual inspection, was only obtained starting from 540°C.

5. Table 1 of the application provides a comparison between softening temperature and CET of lead based and lead-free frit. As a lead-free option, it appears that only bismuth-based

frit is specified. Please provide details of these two parameters for other frit materials to further show the lack of suitable alternatives.

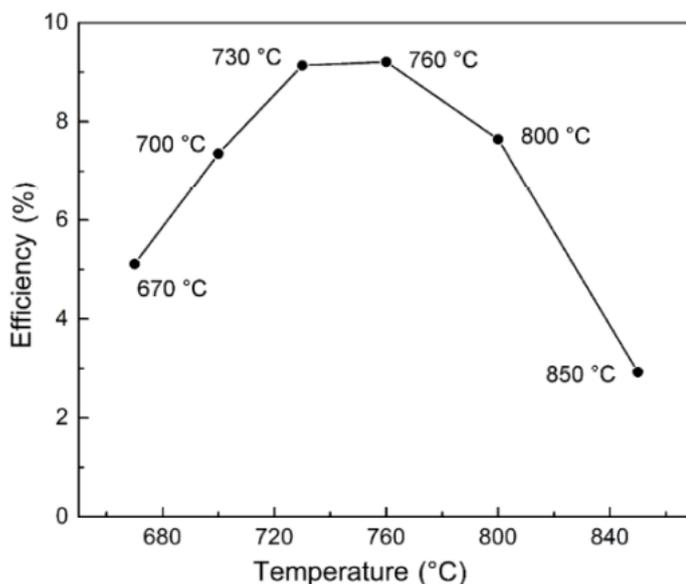
Bismuth-based frits are typically used as a replacement for leaded frits due to their lower melting temperatures compared to other type of glass material.

Bismuth-free frits are used by the industry for other type of applications but their sintering or firing temperatures are already above 700°C, and their actual melting point is even above 900°C.

Below are examples from two research papers.

One investigated several frits based on SnO-B₂O₃ glass [1] which have peak sintering temperature of > 730°C.

The diagram below shows firing temperatures for a frit made with 45 SnO – 53 B₂O₃ glass.



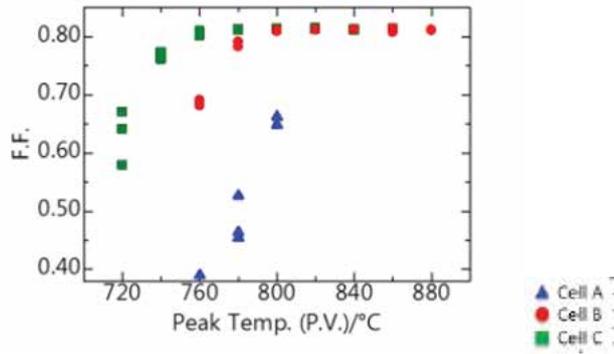
[1] Fig.3: Influence of the peak firing temperature on the efficiency of the cells fabricated with SP02.

Other study, investigated several frits based on TeO₂-WO₃-ZnO-B₂O₃ glass [2] with peak sintering temperatures above 720°C.

COMPOSITIONS OF THE FRITS (MOL%)

Sample Name	TeO ₂	WO ₃	ZnO	B ₂ O ₃	In ₂ O ₃	ZrO ₂	Y ₂ O ₃	Sum
Frit A	40.3	24.5	22.2	8.7	4.3	0	0	100.0
Frit B	40.3	24.5	22.2	8.7	0	4.3	0	100.0
Frit C	40.3	24.5	22.2	8.7	0	0	4.3	100.0

[2] Table 1: Composition of the frits



[2] Fig.1 (a): I-V characteristics of Al-BSF cells fired at various peak temperatures

[1] J. Jiang, C. Li, Y. He, J. Wei and L. Li , “Pb-free silver pastes with SnO-B2O3 glass frits for crystalline silicon solar cells”, 2017 18th International Conference on Electronic Packaging Technology, IEEE 978-1-5386-2972-7/17, 2017 pp. 44-49

[2] M.Kurahashi, N. Shindo, K. Nishimura, K. Shirasawa and H. Takato, “Investigation of the Reaction Mechanisms of Lead-free and Bismuth-free Tellurite glass in Front Silver Paste for c-Si Solar Cells”, IEEE 978-1-5386-8529-7/18, 2018, pp. 1033-1036

6. There are currently other exemptions which cover the use of lead in materials such as glass frit. For example, exemption 7(c)-I of annex III refers among others to lead in glass and exemption 25 of Annex III and exemption 4 of annex IV refer specifically to seal frit and glass frit. Please explain whether the use of lead-based frit in argon lasers is in your interpretation also covered by other exemptions specified in annexes III and/or IV of the RoHS Directive.

Gas laser tubes are optical components that depend on electric current in order to function. If exemption #7c-I covers glass used for this type of components, Lumentum would welcome to consolidate exemption #32 within exemption #7c-I.

Exemption 4 of annex IV for “lead in glass frit binder for assembly of gas lasers” seems to be suitable to cover our application however, it would limit it only for medical devices, monitoring and control instruments. It would not cover the use of argon laser tubes in other tool applications.

In case parts of your contribution are confidential, please provide your contribution in two versions (public /confidential). Please also note, however, that requested exemptions cannot be granted based on confidential information!

Finally, please do not forget to provide your contact details (Name, Organisation, e-mail and phone number) so that Oeko-Institut can contact you in case there are questions concerning your contribution.