campine

EU COM Study to support the review of the list of restricted substances and to assess a new exemption request under RoHS 2 (Pack 15)

1st Stakeholder Consultation
Questionnaire for diantimony trioxide (CAS 1309-64-4; EC 215-175-0)

Responses submitted by Campine
13 June 2018

Contents

Campine statement

Campine welcomes the opportunity to participate in the Stakeholder Consultation.
Campine is producer of Antimony Trioxide (hereafter ATO) since 1924, and lead registrant for ATO. We are market leader in Europe. We actively contributing to the safe use of ATO in all the markets and to the recycling of antimony . Campine is also supplier of halogen and non halogen Flame retardant (here after FR)-master batches.
Therefore we are well positioned to give a good view on the uses, alternatives and recycling potential.

With regard to the specific use of ATO in flame retardant applications, it is a primary moral duty to first consider the benefit of an efficient flame retardant to avoid any loss of lives. The use of ATO, combined with brominated and chlorinated additives or plastics, has **saved millions of lives in the last 50 years**, showing its effectiveness and allowing FR plastics to be used “safe” in the most critical applications like electrical and electronic equipment (EEE). Many alternative synergists were investigated and presented over the last decades. None of them are equally performing at similar addition levels or are ‘totally safe’.

The evaluation of FR-additives used in plastics, including ATO, **is ongoing as well in Reach as in Rohs.** We recommend to evaluate all fire retardants side by side to generate a holistic view before taking a decision in Rohs.

The potential health risk emanating from the use of ATO is limited to the inhalation to the fine particles. We, as supplier are actively promoting the use of dust free ATO products (dispersions, wetted powder, ATO incorporated in plastic granules, doseable bags, ..), this way preventing downstream EEE facility operators to be exposed to ATO. In EEE market almost all ATO is supplied in **safe granulate dust-free form**.

The European union **recognized antimony as a critical raw material** (CRM) as published by the European commission in the CRM list. As such, it is recognized that antimony has unique properties that can be used to manufacture high performing products and contribute to the competitiveness of the European industry. Since the market is reasonably small, every ban of an application will threaten the whole antimony market.

Because of its inorganic nature ATO can be recycled over and over again. Direct reuse as recycled plastic is demonstrated and done. The fact that it **could** have been combined with PBDE’s has lead to solutions as landfill and waste to energy. Sorting techniques will enable to extract the reusable plastic fractions. The work started to summarize best practices in a BREF document for the waste recycling industry will help to introduce these techniques.

The WEEE –recycling sector is actively working on processes to create a circular economy for WEEE waste, containing flame retardants. **The feasibility of re-using halogenated FR’s with ATO has already been proven** in the projects of Fraunhofer and NLPR. Driving factor is the potential re-use of the ingredients. **A negative Rohs evaluation could stop these initiatives and leave more than 50 million tons of recyclable material unused .**

As Antimony metal is for a considerable part a co-mined product , linked to Cu & Pb mining, the shrinkage of the ATO market would disrupt the equilibrium in the metal market . This will end up in massive dumping of all co- mined byproducts . We recommend a full economic study of the impact of an ATO ban on the non ferrous industry. We expect European non ferrous industry will be obliged to buy with less impurities and thus more expensive ores, **reducing their competitive capabilities** on the international market.

In summary :

1. **ATO has saved million of lives, confirming the product as a reliable synergist in flame retardancy.**
2. **We deliver safe dust free products in order to mitigate risks to the well conditioned ATO production environment only.**
3. **REACH evaluation of ATO nor of its alternatives is finalized. It is premature to take decisions on RoHS- directives.**
4. **ATO can be recycled over and over again. FR Plastic recycling will be stimulated by recent development in circular economy.**

We request that the European Commission does not proceed to any final proposal or decision-making in the Rohs context related to ATO before all above mentioned concerns have been clarified.

Wim De Vos

CEO Campine

Hilde Goovaerts

Operation Excellence Manager

Hans Vercammen

Business unit manager Specialty Chemicals

Questions

1. Applications in which diantimony trioxide is in use

a. Please provide information concerning products and applications in which the substance is in use.

i. In your answer please specify if the applications specified are relevant to EEE products and applications or not.

ii. Please elaborate if substitution of the substance is already underway in some of these applications, for example in relation to the properties for which diantimony trioxide is used (for example synergist for halogenated flame-retardants) and/or in relation to specific applications in which it is used (for example in specific plastic materials, etc.) and where relevant elaborate which chemical or technological alternatives may be relevant for this purpose.

We did summarize in table 1 the European antimony trioxide consumption in all applications, with the EEE relevance. Specific figures for the EEE industry are not available as far as we know.

Table 1. Products and applications using ATO and their relevance to EEE –see text i2a

b. Please specify if you are aware, if aside from actual use of the substance, it may be reintroduced in to the material cycle through the use of secondary materials.

i. Please detail in this case what secondary materials may contain diantimony trioxide impurities and at what concentrations as well as in the production of what components/products such materials are used.

It is perfectly possible to re-melt and reuse plastics containing ATO, depending on the polymer type. When properly separated (as mandatory in the waste of electrical and electronic equipment (WEEE) Directive (EC, 2012)), the recycler will sort out the antimony containing product. After determination of the antimony content, he adapts the mix with virgin material (virgin plastics and ATO or other FR's) to produce flame retardant master batches, ready for use for e.g. injection molding of new EEE.

Recycled antimony (either out of lead, copper, printed circuit boards or others) will be introduced in the supply chain at the level of ATO production with final use in the traditional applications.

The recovery of antimony out of plastics is not yet implemented on a broader scale because the operation is not economically viable yet and rather complex (due to pop's in FR plastics). But the implementation is only a matter of time as circular economy will put more pressure and opportunities on the development of solutions to remove pop's).

New sorting techniques for plastic waste are in full development e.g. at the Vrije Universiteit Amsterdam[1]

ii. If possible please provide detail as to the changing trends of diantimony trioxide concentrations in such secondary materials as well as the changing trend of use of the respective secondary material in EEE manufacture.

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[1] Study of Ballesteros-Gómez, A.M., A. Covaci, J. de Boer (2016). Screening of additives in plastics with high resolution time-of-flight mass spectrometry and different ionization sources: direct probe injection (DIP)-APCI, LC-APCI and LC-ion booster ESI. *Anal. Bioanal. Chem.* 408, 2945-2954

The concentration of ATO used in flame retardant EEE components depends on the required flame retardancy performance, which is measured with a standardized test (ISO 4589 and ASTM D2863). This performance will be dependent on the 'natural' flame retardancy of the polymer in which the flame retardant issued , and the final application the polymer is aimed to be used in.

The addition of ATO is today already limited to the minimum . Because of cost producers of flame retardant polymers always minimize the flame retardant to the absolute minimum that is technically needed. Overloading of additives reduces the mechanical strength of plastics , which also motivates compounders to aim for minimal addition levels .

Since ATO keeps all its characteristics (like all metals do), there is no need for differentiation in Sb content in recycled products.

c. Please specify in which applications diantimony trioxide is used as a material constituent, as an additive or as an intermediate and what concentration of diantimony trioxide remains in the final product in each of these cases (on the homogenous material level). If diantimony trioxide is used as a synergist flame retardant, please specify the brominated flame retardant with which it is used and at what concentrations they are applied.

In the specific case of ATO, it is an additive which functions as a synergist with halogens, to confer high flame retardancy (and hence, fire safety) performance to plastic components used in EEE. Halogens provide flame retardancy via the so-called "Radical Trap Mechanism". This is a process where reactive radicals are fed to the decomposition gas, which in turn efficiently cuts the supply of the oxygen fuel, to cease the combustion chain reaction. The halogens released by the plastic they are contained in, quench the chemical reaction occurring in the flame by isolating the various factors, preventing the material from reaching its ignition temperature.

Whereas halogenated flame retardants are moderately effective on their own, they become significantly more effective when combined with ATO. ATO effectively enables a reduced use of halogens while increasing the flame retardancy of the plastics. The use of a combined halogen + ATO flame retardant solution provides flame retardancy via an additional "Gas phase (heat absorption and dilution) Mechanism" of flame retardancy. The halogens with the ATO act as heat-absorbing substances which i) trigger endothermic reactions and cool down the polymer, and ii) dilute the decomposition gas and cuts the supply of oxygen fuel to the combustion region, thereby ceasing the combustion chain reaction.

Because of the two mechanisms they facilitate, the combined use of halogenated flame retardants and ATO is known to provide the most performant flame retardant solution[2].

The amount of flame retardant needed will depend on two main factors:

i) the relative 'natural' resistance to burning of each plastic
ii) the minimum flame retardancy performance that is required for the specific use or application of the polymer.

The flame retardancy performance can be defined by the Limiting Oxygen Index (LOI), which measures the minimum percentage of oxygen concentration it takes to support the candle-like or flaming combustion of plastics in an air-like gas mixture (ISO 4589 and ASTM D2863). Other methods include: Lateral Ignition and Flame spread Test (LIFT), upward flame spread tests (UL-94V and NASA 6001 test 1), cone calorimeter, Steiner Tunnel test (ASME E84)

PP (polypropylene), PE (polyethylene), or PC (polycarbonate) to be used in applications exposed to heat and have an elevated risk of fire, will require much more flame retardant than PVC, which appears to have

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[2] Source: Kitano, M. 2016. Introduction to Flame Retardancy: Protect your life and property from fire. Published by: Osamu Odajima. Japan. Pages 33-46

an intrinsic high resistance to ignition if they would be in the same application and thus compared to the same FR requirements.

The flame retardancy of plastics is preferably determined by the UL 94 V method (Underwriters Laboratories Inc. Standard of Safety, "Test for Flammability of Plastic Materials for Parts in Devices and Appliances", p.14 to p. 18, Northbrook 1998). The UL94 identifies a combustion or flame retardancy level after exposing the material to an open flame. The existing levels are (from lowest (least flame-retardant) to highest (most flame-retardant) are:

- HB: slow burning on a horizontal specimen; burning rate < 76 mm/min for thickness < 3 mm or burning stops before 100 mm
- V-2: burning stops within 30 seconds on a vertical specimen; drips of flaming particles are allowed.
- V-1: burning stops within 30 seconds on a vertical specimen; drips of particles allowed as long as they are not inflamed.
- V-0: burning stops within 10 seconds on a vertical specimen; drips of particles allowed as long as they are not inflamed.
- 5VB: burning stops within 60 seconds on a vertical specimen; no drips allowed; plaque specimens may develop a hole.
- 5VA: burning stops within 60 seconds on a vertical specimen; no drips allowed; plaque specimens may not develop a hole.

In practical terms, a UL94 V0 level is typically the highest flame-retardant level for the most common EEEs in scope of the RoHS Directive[3].

The polymer of choice for a given EEE application and its ‘natural’ fire-resistance vs the LOI or UL level necessary for that application will determine the type and amount of flame retardant needed. It is generally recognized that only the combination of halogen + ATO can achieve UL94 V0 flame retardancy.

In general terms, the amount of ATO added as flame retardant synergist will typically be in a 1:3 or 1:4 ratio to the bromine or chlorine content. Overall, ATO will be present in levels between 2 and 8% in flame retardant plastics.

Table 2 provides an overview of the typical properties of the various polymers which can be used in EEE, and whether ATO can be expected to be present in these : see text i2a

Table 2. Plastics commonly used in electric and electronic equipment[4] - see text i2a

ATO can be used in combination with PVC or DBE Decabromodiphenyl-ethane, FR 1025 (brominated polyacrylate). In printed circuit boards with tetra bromo bisphenol A.

2. Quantities and ranges in which diantimony trioxide is in use

a. Please detail in what applications your company/sector applies diantimony trioxide and give detail as to the annual amounts of use. If an exact volume cannot be specified, please provide a range of use (for example – 50-100 tonnes per annum).

Table 1 gives the overview of the sector.

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[3] Underwriters Laboratories. Available from: https://standardscatalog.ul.com/standards/en/standard_94_6.

[4] British Plastics Federation. Available from: http://www.bpf.co.uk/innovation/Plastics_in_Electrical_and_Electronic_Applications.aspx and http://www.bpf.co.uk/plastipedia/polymers/polymer-thermoplastics.aspx.

Figure 1[5] shows the world consumption of ATO. In figure 2[6] price evolution of antimony metal shows the sudden increase of price in 2011. This drastic change forced a lot of customers to change to another, cheaper flame retardant system when it was technical feasible to meet the needed FR characteristics and mechanical strength. Unless these change overs, the total consumption of antimony trioxide increased. Location of this consumption did change (e.g. from the Nordic to Turkey).

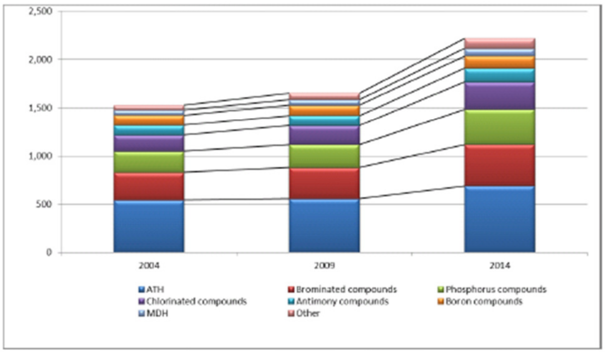

Figure 1. World consumption flame retardants by compound 2004 – 2014 in (000 ton)

Roskill

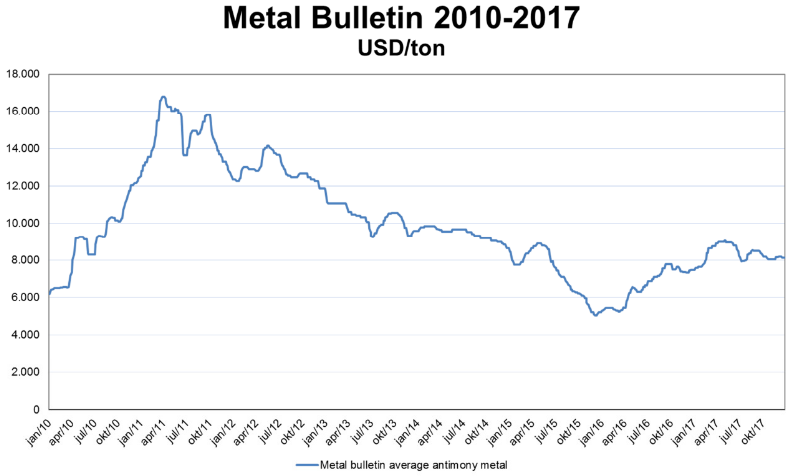

Figure 2. Antimony Metal MMTA standard Grade II in USD/ton

Metal Bulletin

Conclusion : Substitution of brominated FR with ATO is implemented in 2011 – 2012 in applications with low to moderate performance requirements. At present, the global flame retardants market witnesses vivid opportunities due to the increase in demand for flame retardant chemicals in a wide number of end use industries. Moreover, rise in use of flame retardants in the electronic industry due to its property of decreasing the flammability of combustible semiconductors and substances is another key factor that augments the growth of the market. ATO demand is consequently increasing.

b. Please provide information as to the ranges of quantities in which you estimate that the substance is applied in general and in the EEE sector.

The EEE market stands for about 50 % of the total turnover of our ATO business, being some 5000 tonnes per annum for Campine, 10000 ton in Europe. Loss of the EEE market would mean an immediate shutdown of our ATO activities because the large negative impact on our viability.

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[5] Roskill Magmin 2014

[6] https://www.metalbulletin.com/non-ferrous/minor-metals/antimony.html

c. If substitution has begun or is expected to begin shortly, please estimate how the trend of use is expected to change over the coming years.

We do not have any rumor nor signal that there would be already an alternative or substitute for ATO as a synergist, or a FR with equal performances as the ATO-FR solution. Campine is not in the position to answer this question.

3. Potential emissions in the waste stream

a. Please provide information on how EEE applications containing diantimony trioxide are managed in the waste phase (with which waste is such EEE collected and what treatment routes are applied)?

Articles 5, 6 and 8 of the EU WEEE Directive foresee that EEE waste (WEEE) is collected and treated separately and adequately from other wastes in permitted facilities.
Annex VII of the WEEE Directive provides measures to guarantee the selective treatment for materials and components of waste electrical and electronic equipment referred to in Article 8(2) of the Directive, and foresees that plastics containing brominated flame retardants and external electric cables are to be removed from any separately collected WEEE.
In line with the implementation of the WEEE Directive at national levels, it can be reasonably expected in the future that ATO containing WEEE will not be mixed with municipal or other wastes. This sorting is a first step to enable a separate treatment of ATO containing WEEE. Even older sorting techniques based on density will allow to separate ATO due the specific weight in combination with relatively low filling percentages.

Once sorted, the antimony-containing plastics can be directly reused in extrusion[7] if the waste material does not contain any POP's. The loss of characteristics of the recycled plastic depends on the basic material. The ATO itself will still fulfill his function without any needed addition : it can be recycled over and over again. Of course this type of recycling is not yet evident due to other characteristics : color, unknown ingredients, … This is in full development since 'circularity' is in and Europe stresses the recycling of plastics.

If ATO is in a polymer-mix with PBDE or other banned substances, different methods could be used :

1. Reuse of the polymer by dissolving and recomposition. E.g. Vinyloop[8] for PVC, Creasolv[9] for the other applications. The antimony trioxide is recuperated together with other additives, it can be recycled in the traditional pyrometallurgical way.

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[7] CLOSED LOOP RECYCLING OF TV HOUSING PLASTICS, Vanegas Pena, Paul ; Peeters, Jef ; Devoldere, Tom ; Cattrysse, Dirk ; Duflou, Joost;2014-11 and further investigations of this group.

[8] https://plasticker.de/news/docs/VinyLoop_A4_ENG.pdf

[9] http://publica.fraunhofer.de/dokumente/N-452396.html

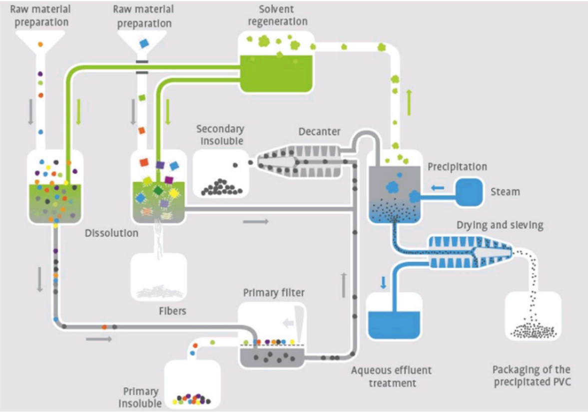
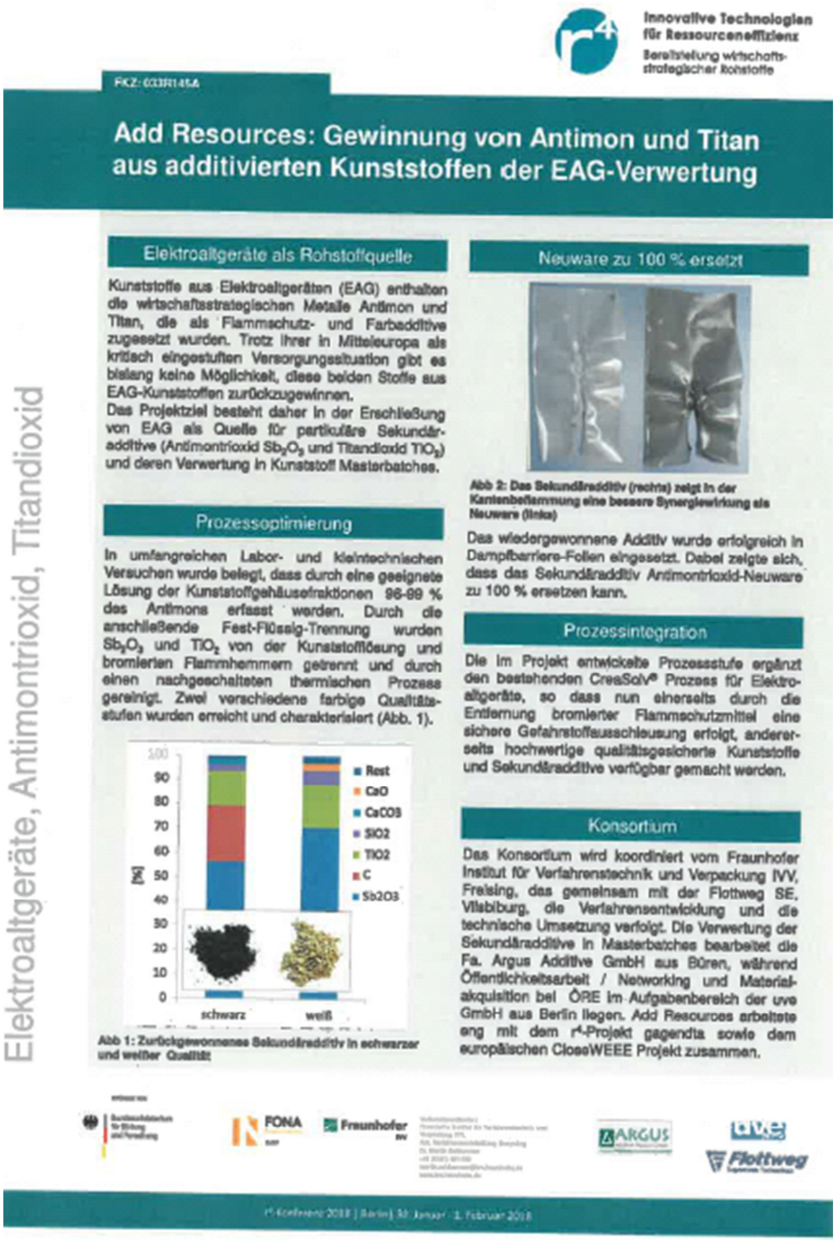


Figure 3 Vinyloop process, recuperation of ATO in the decanter.



Innovative Technologien für Ressourceneffizienz

Bereitstellung wirtschaftsstrategischer Rohstoffe

FKZ: 033R145A

Add Resources: Gewinnung von Antimon und Titan aus additivierten Kunststoffen der EAG-Verwertung

Elektroaltgeräte, Antimontrioxid, Titandioxid

Elektroaltgeräte als Rohstoffquelle

Kunststoffe aus Elektroaltgeräten (EAG) enthalten die wirtschaftsstrategischen Metalle Antimon und Titan, die als Flammschutz- und Farbadditive zugesetzt wurden. Trotz ihrer in Mitteleuropa als kritisch eingestuften Versorgungssituation gibt es bislang keine Möglichkeit, diese beiden Stoffe aus EAG-Kunststoffen zurückzugewinnen.

Das Projektziel besteht daher in der Erschließung von EAG als Quelle für partikuläre Sekundäradditive (Antimontrioxid Sb_2O_3 und Titandioxid TiO_2) und deren Verwertung in Kunststoff Masterbatches.

Prozessoptimierung

In umfangreichen Labor- und kleintechnischen Versuchen wurde belegt, dass durch eine geeignete Lösung der Kunststoffgehäusefraktionen 96-99 % des Antimons erfasst werden. Durch die anschließende Fest-Flüssig-Trennung wurden Sb_2O_3 und TiO_2 von der Kunststofflösung und bromierten Flammhemmern getrennt und durch einen nachgeschalteten thermischen Prozess gereinigt. Zwei verschiedene farbige Qualitätsstufen wurden erreicht und charakterisiert (Abb. 1).

Abb 1: Zurückgewonnenes Sekundäradditiv in schwarzer und weißer Qualität

Neuware zu 100 % ersetzt

Abb 2: Das Sekundäradditiv (rechts) zeigt in der Kantenbeflammung eine bessere Synergiewirkung als Neuware (links)

Das wiedergewonnene Additiv wurde erfolgreich in Dampfbarriere-Folien eingesetzt. Dabei zeigte sich, dass das Sekundäradditiv Antimontrioxid-Neuware zu 100 % ersetzen kann.

Prozessintegration

Die im Projekt entwickelte Prozessstufe ergänzt den bestehenden CreaSolv® Prozess für Elektroaltgeräte, so dass nun einerseits durch die Entfernung bromierter Flammschutzmittel eine sichere Gefahrstoffausschleusung erfolgt, andererseits hochwertige qualitätsgesicherte Kunststoffe und Sekundäradditive verfügbar gemacht werden.

Konsortium

Das Konsortium wird koordiniert vom Fraunhofer Institut für Verfahrenstechnik und Verpackung IVV, Freising, das gemeinsam mit der Flottweg SE, Vilsbiburg, die Verfahrensentwicklung und die technische Umsetzung verfolgt. Die Verwertung der Sekundäradditive in Masterbatches bearbeitet die Fa. Argus Additive GmbH aus Büren, während Öffentlichkeitsarbeit / Networking und Materialakquisition bei ÖRE im Aufgabenbereich der uve GmbH aus Berlin liegen. Add Resources arbeitete eng mit dem r⁴-Projekt gagendta sowie dem europäischen CloseWEEE Projekt zusammen.

r⁴-Konferenz 2018 | Berlin | 30. Januar - 1. Februar 2018

Figure 4. Add Resources project, recovering trace metals from waste electrical and electronic equipment.[10]

2. Energy recuperation from plastic mass in industrial of municipal waste incinerator. Depending on the temperature Sb can be recuperated in the residues :
   Bottom ash (very low and controlled levels of Sb 80 ppm) or fly ash [11](1200 ppm ATO). Bottom ash is re-used as raw material for industrial building (roads/ dykes) after stabilization of the

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[10] https://www.ivv.fraunhofer.de/en/forschung/verfahrensentwicklung-polymer-recycling/add-ressources.html

different components. Fly ash is stored in approved storage area's (old salt mines or other mines).
The composition of the fly ash, and thus the recuperation of the valuable elements, is very complex : main components are Al, Ca, K, Na, Mg, Zn, chloride. In Switzerland recuperation of valuable elements is stimulated, resulting in new processes as Flurec[12]. With comparable techniques it is be possible to recuperate antimony.

Currently incineration methods are in use, due to the fact that good sorting techniques (to make the difference in Pop's and tolerated bromium compounds) are in pilot phase. The plastic waste treatment has to follow the routes chosen in function of the potential contaminated waste stream, not in function of ATO as ATO is combined with Br.

Further development of recycling techniques is ongoing as explained. European stimulation programs, economical benefit and general commitment of the industry towards sustainability is driving this development.

Conclusion : there are upcoming best practices for reuse of ATO. Depending on prices of virgin material, cost for landfill and financial stimulation of the governments, implementation can go faster. The uncertainty about the future prevents investments decisions. A ban to reuse ATO in EEE will result in at least 50 million ton of plastic waste to incinerate, with landfill of the fly ashes.

b. How are waste wire and cables containing diantimony trioxide managed in the waste phase and how is copper extracted from such waste to enable recycling?

The sorting and recycling of wires and cables is stimulated by the need to recover the Cu containing them.
There are different methods for recycling of wire and cable. In most cases, electric wires and printed circuit boards (PCB) are taken out before crushing/shredding operations. The pcb's are taken out before shredding, because there are typically established recycling routes for these, aimed to recover the precious and minor/rare metals they contain.
The cables can be stripped or they can be shredded in whole. In both methods, the plastic mantle can be recuperated (e.g. by density sorting).

CABLO[13], a subsidiary of the Aurubis Group, is specialized in the separation of metals and plastics, in particular in the recycling of cable production waste and cable scrap, contributing to the largest Cu recycling enterprise worldwide. In addition to recovering the brass, Al, Cu, Fe and Pb, CABLO also extracts the almost metal-free plastic components and makes them available for reuse. These products with excellent contour accuracy and surface finish are produced using an innovative injection moulding technique which has been specially developed to recycle plastics (PVC, PE).

The tonnage of recycled cables is increasing due to direct reuse of the PVC and recycling via the Vinyloop process. In 2016, 127 kTon of PVC cables were recycled (containing ATO) (Figure 5).

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[11] Sb and Br in EfW residues AVR Waste incineration – control of bottom ash and flue dust by Tauw
[12] https://www.bsh.ch/en/waste-to-energy-plants/flurec-valuable-metal-recovery-process
[13] http://www.cablo.de/service-products/recycling/?glossary=jpfnqvhk

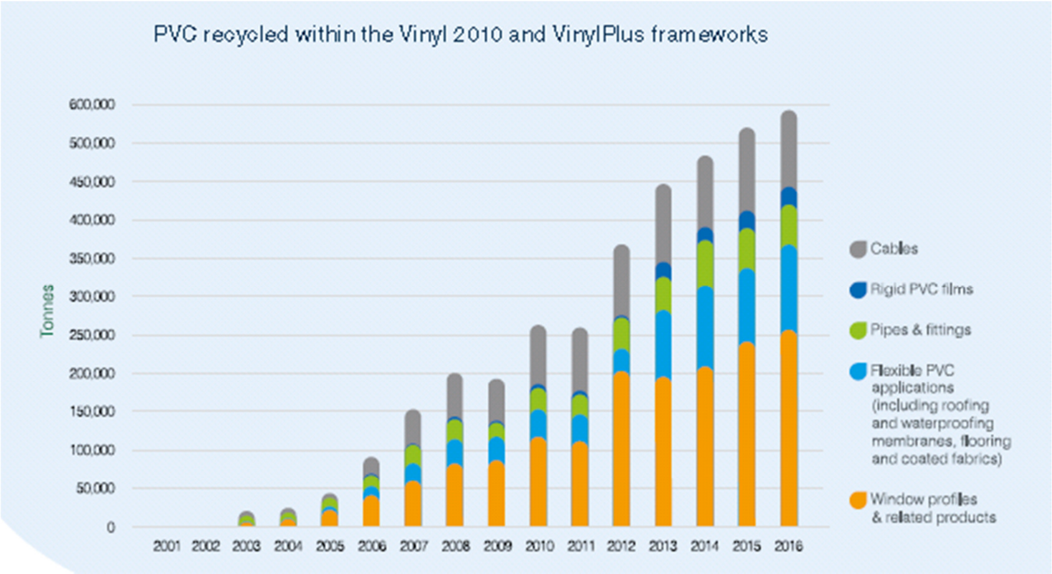

Figure 5: PVC recycled as part of the Vinyl 2010 and Vinyl Plus frameworks[14]

The Vinyloop© process developed by Solvay is a mechanical recycling process using an organic solvent to separate the PVC compound from other types of plastic waste or from the other materials in a PVC composite.

A special solvent is used to dissolve the PVC waste selectively in a closed loop pressurized batch process. The impurities are separated in a filter from the PVC solution; all the constituents of the PVC waste such as PVC, plasticizer, filler, stabilizer and pigments are present in the filtrated liquid. The secondary material is washed to eliminate the entirely dissolved PVC compound and afterwards stripped with steam to recover all the solvent. The liquid is recovered in a precipitation tank, where steam is injected to completely evaporate the solvent.

All the components of the original PVC formulation are recovered in an aqueous suspension. The suspension is split by a decanter into cake and process water. The humid PVC compound is dried with hot air and is then ready to be reused. The solvent is regenerated by a multi-step condensation and separation process, and is entirely recycled.

A first VinyLoop© plant, located in Ferrara (Italy), was built in 2001 to treat post-consumer PVC cable waste. In 2011, about 7500 t of waste PVC, mainly cables, were processed in the VinyLoop Ferrara plant. Besides cables, trials are going on to demonstrate the feasibility of recycling other types of waste PVC.

Conclusions : Good techniques for recuperation of Cu and PVC/ATO are in place. We expect them to grow further as current trends are.

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[14] Source: VinylPlus. 2017. Progress Report. Available from : https://vinylplus.eu/documents/44/59/New-Progress-Report-2017.

c. How are waste glass and ceramics containing diantimony trioxide dealt with in the waste phase?

The glass will not contain ATO, as during the production of the glass, it will be chemically transformed with the other raw materials added to the batch into mixed silicium- mineral -oxides . There is no risk of exposure to ATO during waste glass treatment. The glass is molten and the mixed silicium-minerals remain in the melt . New glass is produced from this melt .
When the glass (eventually containing antimony) is remolten, the antimony will stay in the matrix.

As regards ceramics, the process is comparable to glass . Antimony is built into the ceramic minerals at very high temperatures (800-1000°C)as a mixed- mineral oxide. When ceramics , at end of life cycle , are recollected they are grinded and re-used in cement . During this process the temperature is never raised above 800 or 1000°C : the mixed mineral oxide cannot be broken down below these temperatures

d. Please detail potentials for emissions in the relevant treatment processes.

As to the workplace exposure, there are various national OELs for Sb, set at 0.5 mg/m^3. This ensures that workplace exposure will be either very low (because of the same reasons mentioned for the description of the environmental emissions above), or controlled below the OEL.

Dust free ATO products are available in the market and are actively promoted by Campine, The ATO powder is incorporated into masterbatches, dispersions, powder in closed doseable bags or in wetted conditions, this way preventing downstream EEE facility operators to be exposed to ATO.
The European ATO producers have already transformed more than 50% of their market share into these safe formats. For the EEE market , almost all ATO is supplied in safe granulate format. (ATO concentrates at 80/20 level in ABS,PA,PP,PBT ,PE , SAN) .

RISK = HAZARD X EXPOSURE
During the production of ATO the exposure is known, **all measures are taken to eliminate the risk** (exhaust, automatization, personal protection equipment, training, strict procedures) and people are monitored for exposure.
Along the supply chain, people will not be exposed since they don't come in contact with ATO, because for each market segment there are specific DUST FREE formats of ATO available to allow working with ZERO – exposure .
If there would be any dust forming during the waste-phase in the shredding e.g. countermeasures are taken. But even without good extraction,… the dust particles will be a combination of ATO with plastics, bigger than 10 µ and thus not inhalable .

A study of EBRC[15] demonstrated that the operator handling final products typically could be exposed to 0,002 mg Sb/m3 (not differentiating for the respirable fraction). There is no reason why emissions should exceed this exposure.

The various techniques through which ATO containing EEE waste is treated and their associated potential emission pattern are :

- Sorting techniques (NIR, XRF): waste in its original form/unchanged, solid; no emission
- Sorting based on density: done in liquid, water; liquid is filtered after sorting
- Remelting of plastic or extrusion of plastic: demonstrated processes with low imissions in line with current production since the processing temperatures are moderate and ATO stays in the plastic matrix. (described in the EBRC report)

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[15] 'Occupational, dermal and inhalation exposure to diantimony trioxide during the production of flame retardant non PVC plastics' Study of EBRC Consulting Gmbh 2006

- Creasolv/Vinyloop: liquid process conditions without significant emission
- Shredding: installations are closed, and combined with dust extraction and filter; controlled emissions only.
- Melting: All established smelter work according to best available technology, as described in Bref[16]. For Cu smelters, the limit value for total dust emissions is typically 5 mg/Nm3. In most cases, because Sb does not pose any major environmental concern, there is no specific limit value for Sb. However, the following rule is applied to control emissions to air: the sum of Hg, Th, Pb (max. 0,5 mg/m3), Co, Ni (max. 0,2 mg/m3), Se, Te, Sb, Cr, CN, F, Cu, Mn, V and Sn must not be higher than 2 mg/m3. But in actual operation the total dust emissions are measured continuously and can be even below 1 mg/m3.
- Thermal cracking/pyrolysis: processes are equipped with filter installations; controlled emissions only. Waste to energy or waste incineration plants: equipped with dust filter; controlled emissions only.
- As regards material that is landfilled, strict composition and leaching limitations are imposed before a material can be landfilled. (salt mines)

3. Substitution

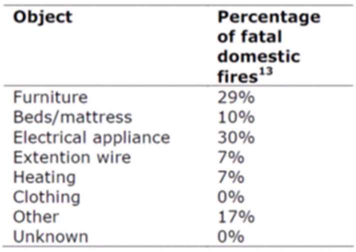
a. For which applications is substitution underway?

For which applications is substitution scientifically or technically not practicable or reliable and why?
Our response reflects 2 undesirable aspects of substitution : The functionality of the Flame retardant and the desirability of replacing one chemical by another of equal or unknown concern.

First of all we want to stress that flame retardants are necessary in EEE.
Elimination of flame retardants or substitution by less performing alternatives is to be considered as " a crime ".
The fire in the Grenfell towers in London ,exactly one year ago, is illustrating this .
Even when release of FR-product would be a topic, it is our moral duty to safe lives.
With relation to the use of ATO in FR- applications , there is NO indication that in the short exposure time during a fire, the inhalation of ATO would harm a victim.
Fire fighters are equipped with the necessary Protective clothing and inhalation protection .
The use of " effective" flame retardants creates " escape time ":

ATO in combination with halogenated flame retardants is particularly effective in extinguishing the flames and delaying the flashover – point !

As Electrical & Electronic equipment is a primary root cause for the initiation of fires, it is of great importance to use the most effective flame retardant products in these equipments.
Root causes of a fire in the Netherlands (2008) ;



Object	Percentage of fatal domestic fires[13]
Furniture	29%
Beds/mattress	10%
Electrical appliance	30%
Extention wire	7%
Heating	7%
Clothing	0%
Other	17%
Unknown	0%

Flame retardants give you the potential to escape of fire :

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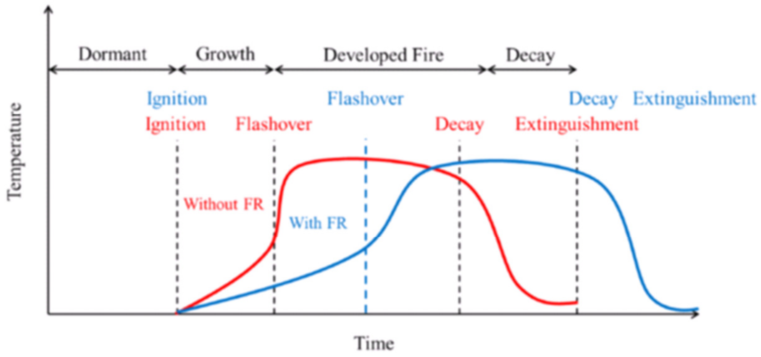
[16] http://eippcb.jrc.ec.europa.eu/reference/nfm.html



Substitution of ATO as synergist for halogenated flame retardants is studied in depth & presented by Campine at the AMI FR conference in dec 2016. We add the paper in the addenda.

<u>Why are other groups of Flame retardants not fit for E&E – applications, compared to halogenated FR's in combination with ATO ?</u>

Mode of action

In electronic & electrical equipment a short circuit is the most frequent cause of fire .
The flame is caused by a strong electrical current in contact with plastic " in " the equipment.
The only way to extinguish the flame is by using a **primary** flame retardant technique.
Halogenated additives with ATO as synergist are radical stabilizers .
These radical stabilizers extinguish the flame, as they stabilize the radicals from the plastic so that the flame disappears.
The UL 94 test method, standard for E&E, is focused on reaching this direct extinguishing.
The flame retardant must fulfill UL 94 V0 : the FR-additive must kill the flame!

Alternative flame retardant techniques like phosphates function as **secondary** flame retardants : they do NOT extinguish the flame.
Phosphates come into action when the plastic is already partially burned.
The phosphates interact with the burned plastic to form together a char layer or an intumescent layer . This is a **secondary** action that helps spreading of the flames .

Mechanical requirement for E&E plastics

Phosphates, aluminium or magnesium hydrides need high loading levels (around 30-60%) for their functionality. The E&E equipment however needs a firm/rigid & strong casing to avoid damage to the incorporated conducting or electronic parts (resistors, capacitors, cables etc.)
At high filling levels engineering plastics like ABS, PBT, PA, PP, etc. loose their physical strength . The plastics become brittle , loose tensile strength & impact strength and become very vulnerable for breakage/ damage.

Brominated flame retardants with ATO as synergist, are functional at a very low level of loading. Therefore they meet the specifications of the EEE component which combine heat and fire resistance, as well as dimensional stability, impact resistance , hardness, wear, friction, abrasion and chemical resistance.

<u>conclusion :</u>
None of the Claimed " alternatives " is reaching the same LOI or UL 94 V0- levels as ATO in the tested PVC, PA or PE - formulations .

Toxicological review

In this paragraph we make the overview of toxicity and environmental aspects when considering ATO compared to some other types of flame retardants, without looking to the flame retardancy performance. We took one of the existing, and objective scores, just to demonstrate that there are several concerns to all of the potential substitution substances. The confirmation of these findings has to be done in detail, based on finalized Reach dossiers.

GreenScreen, a US based NGO evaluated substances on their toxicological performance, summarized in a GreensScreen Benchmark score[17] (GreenScreen class). In the details, the GreenScreen information is combined with REACH data (where Reach data got preference). The assessment of the recycling potential, the carbon footprint, sociological effects and the criticality is based on existing information, e.g. critical raw materials list (CRM)[18].

We compared ATO with other currently used potential alternatives based on the characteristics:

Global classification is a summary of the health and environmental effects.

- Type I human effects : carcinogenicity, genotoxicity, reprotoxicity, developmental toxicity and endocrine toxicity.
- Type II : STOTE, Neurotox, skin sensitizer or irritating to skin, respirator, irritating to eyes
- Environmental : ecotox, chronic aquatic, persistant (the NGO estimated this for metals as very high)

		GREENSCREEN+ REACH + Literature																			own judgement									
		I Human effects					II Human effects									Envi														
Flame retardant	**Class Greenscreen**	**Carcinogen**	**Genotox**	**Reprotox**	**Developmental tox**	**Endocrine**	**Acute mammalian**	**STOTE**	**STOTE repeat**	**Neurotox**	**Neurotox repeat**	**Skin Sens**	**Respirator**	**Skin Irrit**	**Eye Irrit**	**Ecotox**	**Chroniq Aquatic**	**Persistance**	**Bioaccumulation**	**Reactivity**	**Recycle potential**	**CO2**	**Positive Side effects - socio by substitution**	**Negative Side effects - socio by substitution**	**positive CRM by substitution**	**negative CRM by substitution**				
ATO (2014)	1	M	L	M	L	-	L	L	H	L	L	L	-	M	M	H	M	vH	vL	L	3	2	0	4	0	4		vH	very high	4
Triphenyl phosphate	2	M	L	L	L	H	L	L	M	M	L	L	-	L	M	vH	vH	L	L	L	1	2	0	4	0	0		H	high	3
Melamine cyanurate	1	M	M	M	M	-	L	-	H	-	L	M	-	L	M	L	L	vH	vL	L	0	2	0	0	0	0		M	moderate	2
Melamine polyphosphate	2	M	M	L	L	M	M	M	H	M	M	L	-	L	H	L	H	H	vL	L	1	2	0	4	0	0		L	low	1
Red phosphorus	2	L	M	L	L	L	L	vH	M	-	M	H	vH	H	H	L	vH	vH	vL	M	3	1	0	4	0	0		vL	very low	0
Aluminium hydroxide	1	L	L	L	M	M	L	L	M	-	M	L	M	L	L	H	H	vH	vL	L	3	1	0	0	0	0		⬜	own judgement	
Zinc Borate	1	L	M	H	H	M	L		M			L	H	L	M	vH	vH	vH	L	L	3	1	0	0	0	0		🟫	Literature + REACH + Greenscreer	
Ammonium Polyphosphate	3	L	L	L	L	L	L	L	L		L	L		M	L	L	L	vL	vL	L	3	2	0	4	0	0		🟩	Greenscreen	

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[17] https://www.greenscreenchemicals.org/ Benchmark 2014

[18] https://ec.europa.eu/transparency/regdoc/rep/1/2017/EN/COM-2017-490-F1-EN-MAIN-PART-1.PDF

- **ATO**

Overall GreenScreen classification is 1, based on the potential Human effects of type 1 and 2. Recent research has shown ATO does not resort a genotoxic effect, thus lowering the risk defined in the description. ATO is recognized STOTE Repeat.
The report mentions ‘Persistance’ as very high and a reason for classification as score 1. Since this is a heavy metal, it is a logical conclusion that once ATO is released in nature it is indefinitely persistant. Antimony is a naturally abundant substance. Since bioaccumulation is low one can conclude that the risk is very low and a score 1 is maybe exaggerated.
We rate recycling potential, given the fast evolving trend into circular economy as high, strengthened by the fact that Sb doesn’t loose any functionality. Making it a valuable alternative for organic based alternatives.
CO2 production has been rated as average because since the recycle potential for reuse of ATO should be rated lower than de novo production.

Although ATO is used in plastics applications it is used in very low concentrations and compared to other non ferrous metals its spread will be almost non detectable. This might be the reason why authorities never include antimony as an element of concern in their monitoring plans. For EEE applications the issue becomes even more irrelevant because of the EU legislation about separation of WEEE waste and recycling.

Regarding the environmental fate of ATO it is important to note the detoxification reactions that normally unfold in nature. Nature is humid and after it is admitted as a particle into the air it will settle to the soil and run off with rain water into rivers and ponds. Slowly ATO will start to dissolve into Sb-ions, because ATO is slightly soluble. From the ATO point of view the toxicity ends. It is important though to consider the endpoint of the potential toxicity of the degradation products. Like all metals Sb has a positive charge and will adhere to natural clay and organic particles that are generally abundant in nature. Those particles will clog in river meanders or still waters and slowly precipitate to the river soil. Due to biochemical processes and bioturbation the oxidized material will be buried and become anoxic, yet anaerobic. Because of the intrinsic nature of most sludges the anaerobic areas produce free sulfur ions that immediately combine with free metals to form almost insoluble sulfide precipitates. Note that the Ksp for antimony sulfide is as low as 10-93. Taking into account its low solution rate one can easily state that antimony became biologically inactive, the final detoxification step

- **Triphenyl phosphate**

The substance has been put on CoRAP listing for suspected **endocrine disrupter** activity and very acute toxic for the aquatic life and chronic toxic for the aquatic life. The GreenScreen evaluation did not take that into account yet.
Recycle potential is low because it is an organic compound and maybe some elements will be able to be recycled, but not the carbon bounds.
CO2 production is average because of the one time use, meaning that de novo production will lead to extra energy for every new compound.
Social side effects when choosing to substitute ATO for this substance would lead to extra use of phosphate. Phosphate mining **creates radio active wastes** in several mines, which is a burden for society, this can be considered as a severe risk.
Phosphate is also, as well as ATO a CRM. The use of Phosphate for fire retartants is only a fraction compared to its use in agriculture. Banning or using would not have any effect on the status of the CRM.

- **Melamine cyanurate**

Is, following the Greenscreen methodology a class 1 substance based on its persistence and its High effect in **STOTE**. Further studies are going on within REACH. The substance is labeled as being **dangerous for human organs**.
None of the atoms in the molecule are marked as CRM. The use of this substance as a flame retardant will never be high enough to push an atom into a CRM.
Recycling potential is very low because no component of the molecule is able to be recycled once the plastic went into the waste phase.
Melamine cyanurate substitution has no positive side effects. The negative social side effect of using it, is that it is not phased out because it has proven in the past to contaminate (intentionally) the food chain, with toxicological consequences for children and adults.
CO2 production is grosso modo comparable to triphenlyphosphate as it will not be recycled and thus has to be synthesized “de novo”.

- **Melamine phosphate**

Is considered a class 2 because of the moderate Human effects I and II. In the mean time REACH concluded that there is serious risk for **eye irritation**, indicating Greenscreen should be updated. It is also **damaging organs on the long term and harmful for the aquatic life** for the long term. Since phosphorus is part of the cristal, the same conclusions apply as triphenyl phosphate.

- **Red phosphorus**

Classified as class 2 because of the moderate human effects I and II. There are reasons to put this component in class 1 because of more recent evidence in the REACH context showing that it is **fatal when swallowed or inhaled, causes severe skin burns and eye damage and is very toxic to the aquatic life also on the long term.**
Phosphor is in principle recyclable with an efficiency compared to ATO. It is estimated that CO2 production for recycling and reworking is small.
The other evaluation points are coherent with triphenyl phosphate and show ample reasons to be an adequate substitute for ATO.

- **Aluminium hydroxide**

Greenscreen considered this substance as Class 2 because of the moderate Human I and II effects. REACH did not detect any risks either. It is not classified according to the REACH legislation.
The IARC (International Agency for Research on Cancer) however, has classified the **production of aluminum as carcinogenic.** Therefor a class 1 has to be taken into account.
The other risks are comparable to triphenyl phosphate leading to a bad substitute for ATO.

- **Zinc borate**

Is a class 1 because of its **severe human effects**. The substance has the same profile as Aluminium hydroxide thus being a bad substitute for ATO.

- **Ammonium polyphosphates**

Classified as class 3 and has no observed risks of relevance. According to the REACH classification ammonium polyphosphates can cause serious **eye irritation and are harmful if swallowed**. This information is more recent than the Greenscreen summary. Has the same profile as other phosphates. The major drawback of the use of APP is the production of **radio active wastes**.

Conclusion:

potential substitutes all have ecological and social drawbacks making them not suitable for being an adequate alternative for ATO. Reach evaluation has to be finalized before promoting one or the other solution.

i. Please specify in this respect which alternatives are available on the substance level (substitution) and which are available on the technological level (elimination). For example, which alternatives can be applied instead of diantimony trioxide used in PVC cables or in plastic components and which alternative isolating materials can be applied instead of PVC in order to eliminate the need for diantimony trioxide in such applications?

Possible substitution is studied in the research work for preparation of the presentation " the pallet of synergists ", included as annex 1. The study is published by Campine, tests are executed at independent institutes. (VKC and WRF Gent).
None of the claimed alternatives reaches the same levels of fire extinguishing compared to ATO .
They have interesting complementary properties , as reduced smoke . But they do not act as radical stabilizers to reinforce the function of Bromine or Chlorine.

These findings, are supported/confirmed by the findings of the European Union :
The European union recognized antimony as a critical raw material.
Critical Raw Materials are those raw materials which:
(a) have a high economic importance for key sectors of the European economy,
(b) have a high-supply risk, and
(c) do not have viable substitutes due to their unique and reliable properties for existing and future applications.
As such, it is recognized that antimony has unique properties that can be used to manufacture high performing products and contribute to the competitiveness of the European industry.

In any case, it is not wise to compare existing solutions with potential alternatives, which are by far less investigated than the existing solutions.
It is premature to conclude that ATO is more dangerous for human health ,compared to alternatives that are NOT yet studied in full detail.
Unsolicited restrictions generate market uncertainties that put pressure on companies who aim to remove the listed substances from their portfolios, inventories and supply chain as quickly as possible. Although such reactive behavior may seem honorable and wise, in practice, decisions taken under such pressure may precipitate 'regrettable substitution'. Regrettable substitution is not only the substitution of a substance or a technology by an alternative which may actually pose similar or worse risks, but also the substitution by alternatives which are unsustainable from an energy consumption, sourcing, or resource efficiency standpoint, and which shift or transfer the risk elsewhere.

What constraints exist to the implementation of the named substitutes in a specific application area (provide details on costs, reliability, availability, roadmap for substitution, etc.). For example, for what range of the diantimony trioxide applications can specific substitutes be used for?

Technical specifications of the EEE components are the main (justified) constraint to implement substitutes for fame retardancy in EEE.

The absence of a truly holistic AoA is the second main constraint to (successfully, and not regrettably) implement substitutes for flame retardancy in EEE.

4. Socio economic impact of a possible restriction

Please provide information as to the socio-economic impacts of a scenario in which diantimony trioxide were to be restricted under RoHS. Please specify your answers in relation to specific applications in which the substance is used and/or in relation to the phase-in of specific alternatives in related application areas. Please refer in your answer to possible costs and benefits of various sectors, users, the environment, etc. where possible; please support statements with quantified estimations.

ANNEX 3

5. Further information and comments

The information compiled on diantimony trioxide for the stakeholder consultation has been prepared as a summary of the publicly available information reviewed so far. If relevant, please provide further information in this regard, that you believe to have additional relevance for this review, as well as references where relevant to support your statements.

It is important to note that the presence of ATO in flame retardant plastics not only is not an obstacle to recycling, but even facilitates it because ATO actually can be used to sort out brominated flame-retardant plastics from other (flame retardant) plastics, using density and weight. There is an increased potential to improve the recyclability and subsequent resource-efficiency and sustaining circular economy when the flame retardant plastics contain ATO. Since these studies are running and confidential, no evidence can be included in this document, but can be asked if needed.

Campine recycles yearly more than 650 ton of Sb, partly for lead alloys, partly for ATO production.

As regards environmental issues, Sb is indeed mentioned in various legislations, despite the fact that recent risk assessments have demonstrated that Sb is not toxic for the environment.

Campine has produced ATO since 1924 . Campine has a 14001 certification and has respected at any point in time the valid regulations.

As regards human health, only animal data exists that show lung toxicity, under extremely high exposure conditions. Levels of exposure below the OEL of 0.5 mg/m^3 have led to the absence of lung toxicity in workers producing and using Sb substances.
Health of the people is extremely important for Campine.
Best practices for avoiding dust, best Pbm's combined with 3 monthly biomonitoring with feedback to the operator are in place. Campine monitors the Operator health closely: IDEWE (external service, monitoring health at Campine) has published it's study of operator health to NTP (US) and ECHA (EU) recently. Investigations to correlate workplace exposure, seniority in the ato production and respiratory functions demonstrated that there is no effect on the health of the people. (study in annex 2)

Campine has no evidence of increased carcinogenity of its employees compared to a reference group that has not been exposed to ATO –dust .

About Campine

Campine is involved in the recycling of lead, antimony and tin. We produce ATO and flame retardant masterbatches.

For further information: www.campine.be

ANNEX

Annex 1.
presentation " the pallet of synergists ", included as annex 1. The study is published by Campine, tests are executed at independent institutes. (VKC and WRF Gent). Rien Repriels, Campine

Annex 2.
Investigations to correlate workplace exposure, seniority in production of ATO and respiratory functions, demonstrating there is no effect on the health of the people. IDEWE 2017 Lieve Vandersmissen

Annex 3.
Confidential information Campine