



Adaptation to Scientific and Technical Progress under Directive 2000/53/EC (ELV Directive)

Review of exemption 8 (i)

Final Report

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1 Review of exemption 8 (i)

Abbreviations, terms and definitions

Liquidus	The liquidus temperature is the temperature above which an alloy is completely liquid so that no crystal phases coexist with the molten phase.
Solidus	The solidus temperature is the temperature below which the alloy is completely solid so that no liquid phases coexist with the molten phase.
	In the temperature range between the solidus and the liquidus, solid, crystal and molten phases of the alloy coexist.
OEM	original equipment manufacturer, used as a synonym for vehicle manufacturer

1.2 Description of requested exemption

The technical background of this exemption was described in detail in (Öko-Institut 2008).

1.3 Overview on past reviews of this exemption

This review is the third review of exemption 8 (i) since 2007.

In 2007, Antaya had applied for repealing exemption 8 "Solder in electronic circuit boards and other electric applications" for glazing applications. The company claimed to have a lead-free solder based on indium to substitute the lead-containing solders for soldering on glass (Öko-Institut 2008). The vehicle manufacturers (OEMs) and their suppliers, the glass manufacturers, opposed this claim. It could not be fully clarified whether the Antaya solder would be a viable substitute to a degree that would have justified revoking the exemption. It was hence recommended to continue the exemption, but to review it in January 2009. (Öko-Institut 2008)

In August 2008, the Commission changed the exemption in Annex II into exemption 8(b) and added an expiry date: "Solder in electrical applications on glass for vehicles type approved before 31 December 2010 and spare parts for these vehicles". The Commission set a review date for the exemption in 2009.

In 2008 and 2009, the stakeholders worked in an open joint working group that agreed on a joint lead-free solder test program for soldering on glass. The objective was to find out whether Antaya's lead-free solder could actually substitute the lead solders in all glazing applications. Antaya's proposed lead-free solder was tested according to this program. In the tests, "[...] the lead-free indium-based alloy did not show performances inferior to the lead alloy." (Öko-Institut 2009)

At the time when the joint test program was designed, it was, however, not yet known that the melting point of the proposed lead-free solders (109 °C solidus, 127 °C liquidus¹ (Öko-Institut 2009) was much lower compared to the lead solders that had been used so far in this application (160 °C and 227 °C). The lower melting points thus could not be taken into account in the joint test program. It was hence recommended to continue the exemption until end of 2012. The OEMs and their suppliers should have time to qualify the Antaya solder for their applications and to apply for the continuation of the exemption in case the low melting point would turn out to cause reliability problems.

In February 2010, the Commission amended Annex II and the exemption was changed into exemption 8 (i) "Lead in solders in electrical glazing applications on glass except for soldering in laminated glazing, in vehicles type approved before 1 January 2013 and spare parts for these vehicles". The exemption must, however, be reviewed again before 1 January 2013, which is the reason for this new review of exemption 8 (i).

1.4 Stakeholder constellation and main arguments in this review

The OEMs request the continuation of exemption 8 (i), Antaya pleads to maintain the current expiry date 31 December 2012. St. Gobain, a third stakeholder, submitted documents claiming that St. Gobain will have an alternative lead-free solution available in the foreseeable future, the time frame depending on the testing and validation of the substitute in by the OEMs.

The main discussion line between the OEMs on the one hand and Antaya on the other hand is the maximum temperatures that may occur in solder joints used to fix connectors of heated backlights to the backlight. The OEMs submitted simulation test results showing that the temperatures in connectors may be well above 100 °C, which the Antaya lead-free alloy with 65 % indium with only 109 °C solidus cannot withstand. Additionally, the OEMs put forward that the Antaya lead-free alloy is principally inappropriate for reliable long term applications due to insufficient mechanical properties.

¹ The liquidus temperature is the temperature above which the alloy is completely liquid. The solidus temperature is the temperature below which the alloy is completely solid. In the temperature range from 109 °C to 127 °C, solid and the molten phases of the alloy coexist.

Antaya counters the OEMs' claims with own tests, which shall prove that the maximum temperatures will only arrive at around 80 °C. Antaya additionally provided information that the alloy has been used without failures in several car models, which would prove that the mechanical properties of the lead-free alloy are not a principle obstacle against its use in automotive glazings.

1.5 Justification by ACEA et al. for the continuation of the exemption after 2012

ACEA submitted documentation to prove that the temperatures of the connectors in vehicles may well achieve more than 100 °C. ACEA wants to show that the Antaya lead-free solder is not an appropriate substitute for the lead-containing solders that have been used so far due to its lower melting point and its material properties.

1.5.1 High temperature tests

(ACEA 3) reports about solar radiation tests, most of them conducted in laboratory climate chambers under defined company specific test conditions. According to (ACEA 7), the "[...] tests were conducted by members of the joint association industry expert group applying OEM's and suppliers appropriate test specifications." The tests were thus not implemented by independent organizations.

Solar radiation performances

(ACEA 3) reports about maximum recorded solar radiation values for Europe of around 1.050 W/m^2 and may reach 1.100 W/m^2 or slightly higher depending on solar activity.² The quoted source³, however, is a complex database, from which these values cannot be retrieved without further expertise.

(ACEA 3) presents Figure 1 showing that 1.150 W/m² were recorded in Italy in summer of 2010.

² http://re.jrc.ec.europa.eu/pvgis/apps/radmonth.php?lang=de&map=europe; http://re.jrc.ec.europa.eu/pvgis/apps4/pvest.php; sources referenced in (ACEA 3)

³ http://re.jrc.ec.europa.eu/pvgis/apps/radmonth.php?lang=de&map=europe; <u>http://re.jrc.ec.europa.eu/pvgis/apps4/pvest.php</u>; sources referenced in (ACEA 3)



Figure 1: Solar radiation performance recorded in Italy in July and August 2010 (ACEA 3)

According to (ACEA 7), the measurements were done at the NSG-Pilkington manufacturing plant in San Salvo, Italy, at 42.3°N, 14.43°E. The pyranometer was installed on a horizontal plane, the sensor pointing vertically, i.e. not directly at the sun. The pyranometer used was type CM6B manufactured by Kipp & Zonen. (ACEA 7)

(ACEA 3) references the SoDa Project⁴ as an independent source for solar radiation performances.

⁴ SoDa Project, http://www.soda-is.com/doc/enviroinfo2002.pdf



Figure 2: Solar radiation performances in Tenerife, Spain (SoDa project 2005 in ACEA 3)

According to (ACEA 3), Figure 2 shows the radiation values for a common summer day on Tenerife. (ACEA 3) says that there exist even hotter days with more radiation, but does not provide evidence. (ACEA 3) explains that the chance is 68% that the actual value is comprised between lower and upper bounds. The real measured radiation amounts to 1.017 W/m^2 . With the same input and without secondary radiation contributors it could be even 1.117 W/m^2 or 987 W/m². There is a 32 % chance that the real value is not between these extremes. (ACEA 3)

Results of high temperature tests of various cars

(ACEA 3) presents results from different high temperature tests on various cars. Concerning the test conditions, (ACEA 3) explains that the recorded solar radiation load in the test varied between 850 and 1,050 W/m².

Thermocouples were attached to connectors on backlights in vehicles. The vehicles were positioned in solar test chambers, exposed to varying levels of solar intensity and the temperatures at the connectors recorded without and with powered heat grid (defrost).

	°C	°C	°C	°C	°C	Light	Glass	Angle /	
	Defrost 0 min	Defrost 10 min	Defrost 15 min	Defrost 20 min	Defrost 50 min	Transmission	Thickness in mm	Inclination	Remarks
Japan 4, Sedan, Lab	104	116.5	117.5			81 %; 0 % at black carbon	3 mm	25.8	normal glass; further details
Test Japan 4, Hatch, Lab	93	100	100.3			81 %; 0 % at black carbon	3 mm	38.3	see test report normal glass; further details
Japan 1, Lab Test	98					77%	3,1 mm	10	1000 W/m ² ; 2h load ; 35 °C ambient temp.; further details see test report
EU 7, Sports Car, Lab Test	100			118		71%	4 mm	15	1050 W/m ² ; further details see test report
EU 5, Wagon, Lab Test	126.3				132	20%	3,5 mm	20	dark tinted glass; 1000 W/m²; further details see test report
EU 6, Test Car		102				72%	3,85 mm	24	normal glass; 1049 W/m²; further details see test report
EU 9, Lab Test	95		118			70 % minimum	3 mm		1000 W/m ² ; 42 °C; further details see test report
EU 6, Lab Test	98 - 102					30%	3,85 mm	13	max. value 1050 W/m²; defrost not tested; further details see test report
Japan 2, Sports Coupé Car, Lab Test	104 - 109					78%	4 mm	15.4	1000 W/m ² ; 42 °C ambient air; different points measured front & rear; heater off; further details see test report
EU 2, Roadster, Outdoor Test	80		91			72%	3.15	65.5	around 950 W/m²; outdoor; normal green glass; further details see test report

Table 1: Maximum temperatures recorded in the tested vehicles (ACEA 13)

According to (ACEA 7), the exposition to the ambient temperature was generally done until near equilibrium or longer, i.e. more than one hour. In real life, a parked car has time to equilibrate as the intensity and angle of the sun increases slowly. The ambient temperature used in lab tests varied according to the specifications of the OEM that performed the tests, usually between 35 °C and 50 °C. The lamps were positioned at the ceiling of the climate

chambers. This corresponds to outdoor weathering in locations where the sun at noon reaches a completely vertical position, like in Arizona, USA. As in the very south of Europe like Cyprus or south of Spain the angle is only a few degrees different, the results are comparable. Table 1 shows a summary of the test results including some information about the test settings.

Beyond the information in the above Table 1 and two test descriptions for the vehicles EU5 and EU7 (see next sections), ACEA et al. submitted all other information about the test conditions and settings, the tested vehicles and the detailed test results as confidential information in the "test reports" mentioned in the "Remark" column of the above table. As a general remark, (ACEA 7) stated that "Test conditions mainly were oriented to German OEM specification. Tests were carried out on backlights made on standard production equipment using standard inks." No detailed test documentation is publicly available.

(ACEA 3) reports the maximum temperature recorded in the test with 126.3 °C without defrost in the EU5 car, and 132 °C with powered heat grid in this same car. This test car was equipped with dark tinted glass and exposed to 1,000 W/m² of solar performance. The other cars reached a maximum of 104 °C without and 117.5 °C with defrost, both measured in the Japan 4 Sedan with normal glass.

Details on the solar radiation laboratory test on vehicle "EU5"

(ACEA 3) provided details for the test on a wagon with a dark tinted backlight (EU5) in Table 1 on page 6.

- Light transmission of the backlight: 20 %
- Infrared radiation applied: ~ 1.000 W/m²
- Ambient temperature around 50 °C

Thermocouples of type J (Fe-Const) were used at two different locations (ACEA 3):

- 1 couple at the bus bar
- 1 couple at the soldering joint

(ACEA 3) indicates the test times as follows:

- 60 Minutes without heated backlight
- 50 Minutes with heated backlight

Figure 3 displays temperatures recorded during the test.



Figure 3: Temperatures without (60 minutes) and with heated backlight (50 min) (ACEA 3)

With defrost off, 126 °C and with defrost on, 132 °C have been measured. (ACEA 3) does not specify whether these temperatures were recorded at the bus bars or at the connectors (ACEA 3) maintains that this experiment confirms that dark tinted glass results in higher temperature load at bus bar and solder joint.

Details on the solar radiation laboratory test on vehicle "EU7"

(ACEA 3) provides an example of the test conditions and results for vehicle "EU7" in Table 1.



Figure: "EU 7" test vehicle (ACEA 3)

In (ACEA 3), an OEM conducted solar load simulation tests on the test vehicle displayed in Figure. The tester used its own vehicle climatic chamber to determine temperature loads that vehicles could experience in extremely hot territories. The "EU 7" sports car was equipped with a 4 mm green and toughened backlight, installed in an angle of 15 ° to the horizontal,

corresponding to 75 ° angle to the vertical. The test conditions shall simulate thermal soak of a closed vehicle parked with the rear facing midday sun at ambient temperatures of 40 °C and 50 °C exposed to solar radiation of 1050 W/m². Table 2 shows the temperatures recorded at the inner surface of the windshields measured at the locations indicated in Figure on page 8.

Table 2: Temperatures recorded on the inner surface of the installed windshields (ACEA 3)

test	location	result
Mesurement of temperature of inner surface of a windshield installed in a car. Heater not operated. Ambient temperature : 42°C Incident solar energy : 1000W/m ² Glass type:light green : 4mm	Laboratory / climate chamber	FR WDW : 109°C RR WDW UPPER : 104°C MID : 104°C LOWER : 104°C

Additionally, the temperatures were measured by thermocouples embedded in the solder joints during soldering of the metal electrical connectors to the glass (see "inner thermocouple" in Figure 4 on page 10). Table 3 shows the recorded maximum temperatures inside the connectors and on the busbars.

Table 3: Maximum temperatures	recorded inside connectors	and on bus bars of the	"EU7" test vehicle (ACEA 3)
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Solar Load intensity	Vehicle Code EU 7	Glass type 4 mm green	Glass type 4 mm green
		Connector	Busbar
1050 W/m ²	Power Off	97,2	100,1
	Power On	107,6	118,1

The testers conclude in (ACEA 3) that, based on the test results, the Antaya indium solder with a solidus of 109 °C cannot work in territories with extreme climates experienced in Europe and North America, Australia, and North Africa.

Inner versus outer connector temperature

(ACEA 3) explains that the temperatures in the tests in the section 1.5.1 were measured using thermocouples attached to the outside of the contacts. In addition, the temperature inside the solder joint was determined via some measurements in a laboratory of a supplier. The effect of positioning connectors underneath a soldered T-piece (between solder and silver print) and on top of the soldered T-piece was investigated.

An IR lamp was positioned over the glass and the distance from the lamp to the glass was varied. The outside surface of the glass was facing the lamp to simulate real conditions (glass protecting T-piece from direct radiation). Figure 4 shows the test setting.



Figure 4: Test setting for the temperature assessment inside connectors (ACEA 7)

Figure 5 displays the temperatures recorded on the outside and inside of the connector.



Figure 5: Inner and outer temperatures at connectors (ACEA 3)

(ACEA 3) states that in all three tests the thermocouple underneath the connector was always slightly hotter than the thermocouple on top of the connector. The difference between the two temperatures varied depending on how close the lamp was to the glass. So when the lamp was closer the difference was greater. This result would equate to a greater difference at higher solar intensities.

(ACEA 3) admits that the test is not conclusive enough to justify an adjustment of the temperature data, but it indicates that temperatures would be higher if thermocouples are positioned underneath the connector. Typically the difference is between $2 - 4^{\circ}$ C in these tests. At higher intensities the difference would be greater, according to (ACEA 3).

1.5.2 Dark glass computation study

With dark tinted glass, ACEA et al. claim that the temperatures at the connectors will be even higher. The highest temperatures (126 °C and higher) were actually measured in the EU 5 vehicle (Table 1 on page 6), however, in a laboratory test.

(ACEA 3) provides Figure 6 showing a computational simulation in which the glass temperature is linearly correlated to energy or light transmission.



Figure 6 Linear correlation of glass temperature and light transmission of the used backlight glass (ACEA 3)

As a consequence, (ACEA 3) claims the temperatures in dark glass to be about 15-20 °C hotter than standard glass for the same glass thickness, and the darkest glass should even be about 25 °C hotter than standard glass. According to (ACEA 3), the thickness of the glass

as well should cause significant differences. For the same raw color, 5 mm glass should be around 3 °C hotter than 4 mm glass, and 4 mm glass again 3 °C hotter compared to 3 mm glass.

(ACEA 3) concedes some limitations of the simulation. It is assumed that the temperature inside the car has not changed (always 80°C) when changing glass type from standard tint to "privacy" tint, since it is difficult to simulate in a car the real inner conditions for given heat flux, external temperature and exposure time. But (ACEA 3) concludes that the glass color influence is significant. Indeed, if the temperature inside the car is lowered by 10°C (i.e. 70°C) thanks to the use of a whole rear set of privacy glazings on the exposed car (a single "privacy" glass on a car will not lead to such a temperature reduction), the computed temperature result on the glass is lowered by only around 5 °C. (ACEA 3) therefore still speaks about a 15 to 20 °C temperature increase on glass for privacy glazing vs. standard tint.

1.5.3 High temperature requirements for paint repairs

(ACEA 8) puts forward that materials used in vehicles must stand high temperatures for paint repair. Fiat, for example, states in (ACEA 8) that in production they use irradiation lamps reaching temperatures of 140 °C for paint repairs, during which, however, the windows are protected with thermal shields. Other OEMs put forward similar claims in (ACEA 8) asserting that the lead-free Antaya alloy cannot withstand these temperatures.

1.5.4 Mechanical instability of indium solders

Intermetallic phases and micro-cracks in indium solders

(ACEA 9) puts forward that new evidence since the review in 2009 proves all indium solders that have been tested to develop harmful intermetallic layers when exposed to elevated temperatures for a period of time. It is not possible to stipulate time and temperature limits because the intermetallic growth is affected by both time and temperature. The Antaya 65 % indium lead-free solder was one of the tested alloys. The intermetallics are present immediately after soldering, which Figure 7 shall illustrate. Intermetallic layers are present in both images. The intermetallics are evident in the original condition as a thin layer. (ACEA 9) does not reveal whether "indium solder 1" or "indium solder 2" actually is the Antaya lead-free indium solder.



Figure 7: Intermetallics in indium solders directly after soldering (ACEA 5)

(ACEA 9) interprets that the intermetallics grow over time. The intermetallics are evident in the original condition as a thin layer and as a much bigger layer with cracks after the conditioning. The growth of the intermetallic layer with indium solders is also seen after thermal cycle tests and humidity tests with a maximum temperature of 80°C. Figure 8 shows the growth of the intermetallic layers compared to the status in Figure 7 and the formation of cracks after environmental conditioning (1,000 hours at 100 °C according to an OEM test specification). (ACEA 5, ACEA 9)



Figure 8: Growth of intermetallics in indium solder after 1,000 h at 100 °C (ACEA 5, ACEA 9)

The intermetallic phases are brittle, have a different CTE (coefficient of thermal expansion, thermal mismatch). They may thus cause cracks in the solder joint. The intermetallic phases as well affect the cohesion of the solder joint to the copper and silver substrate. (ACEA 7)

(ACEA 5) explains that high temperatures accelerate the growth of the intermetallics. As a consequence, the soldered connection is unstable. (ACEA 7) presents a lead solder

reference showing a joint soldered with a lead-containing solder after 63 thermocycles between 120 °C and -40 °C and 30 A electric current.



Figure 9: Pb70Sn27Ag3 solder joint after 63 thermocycles between -40 °C and 120 °C, 30A (BMW in ACEA 7)

(ACEA 5) says that these findings are in line with a supplier's analysis of indium solder on glass conducted at the NSG Research and Development facilities in Japan with guidance from the European Technical Centre in UK. Figure 10 shows intermetallic growth formation after high humidity treatment, which, according to (ACEA 9), is an OEM test specification.



Figure 10: Formation of microcracks in indium based solder after high humidity treatment (ACEA 9)

According to (ACEA 5), micro-cracks can be observed. The customer rejected the indium solders because of the cracks in the solder from the intermetallic layer that grows during the conditioning test. For confidentiality reasons, (ACEA 9) did neither reveal the details of the high humidity treatment nor the composition of the tested indium solders. A test reference with lead-containing solders is missing as well.

Incapability of the lead-free alloy to pass all tests of all OEMs

(ACEA 9) reports that a number of indium containing solders (including Antaya's 65 % indium solder) have been tested against the standard durability tests required of the various global OEM's. Based on the pass/fail criteria in the standard customer tests, the indium containing solders failed some specific tests consistently. Unless OEMs are willing to revise their pass/fail criteria for these specific tests either for a specific vehicle or for all future vehicles, it is clear that Indium solders in general cannot meet global customer requirements and consequently ACEA et al. recommend lead-containing solders.

The table below shows some of more than 100 customer specific validation tests that have to be passed before vehicle launch. Table 4 summarizes the assessment of lead-free solders with 65% indium against those tests.

Test	Result – Pb solder	Result – Indium solder
1000 hours, 100°C.	Pass	Fail – low pull strength, cracks in solder due to intermetallic growth
1500 hours, 90°C	Pass	Fail – low pull strength
Salt spray – 168 hours, 336 hours, 672 hours	Pass	Fail – low pull strength
500 hours, -30°C	Pass	Pass
336 hours, 50°C, 95%RH	Pass	Pass
720 hours, 80°C 98%RH	Pass	Fail – low pull strength, cracks in solder due to intermetallic growth
24 hours, -40°C	Pass	Pass

Table 4: Selection of customer specific validation tests of lead-containing versus lead-freesolders (ACEA 9) (ACEA 9) claims the above summary table with OEM specific tests to be clear evidence that indium solders do not meet all OEMs existing requirements. (ACEA 9) further on points out that the joint test program in 2009 and the new proposed tests since then are not considered as validation tests by the industry. All individual OEMs have their own specific tests and any new solders have to pass all those tests.

With this evidence, (ACEA 9) concludes that at temperatures of 80 °C already, whereas temperatures around 120°C are possible, the intermetallic layers will grow and will result in cracks in the soldered joint. This makes the joint unacceptably weak and is rejected by OEMs.

ACEA et al. remarks on the 2009 joint test program

As described in section 1.3 on page 1, several OEMs and glass makers had discussed and set up a joint test program in 2009, which can be found in Annex III.

(ACEA 9) states that in the 2009 joint test program, many indium solder joints that were soldered by Antaya on the Toyota Aygo glass samples provided by a supplier detached at low pull forces while the Pb-solder joints soldered on the same glass samples performed all well. The indium solders thus, according to (ACEA 9), has performed inferior to the lead-solder reference. Figure 11 shall illustrate this claim.



Figure 11: Test results Aygo after environmental conditioning according to the 2009 Joint Test Program (ACEA 9) Left side: lead-soldered connectors; right side: lead-free soldered connectors

(ACEA 9) explains that the rate of intermetallic growth occurring with all Indium solders tested is affected by time and temperature. The test carried out in 2009 was at 90°C for 500 hours. When that test programme was being designed the OEMs wanted to test at higher temperatures but Antaya refused (ref: Minutes of meeting at CLEPA 17 July 2008). Had the solder been tested at higher temperatures then based on new evidence it could be anticipated that the intermetallic layer would have grown more and failure at lower pull loads would have been more noticeable. Equally it could be anticipated that storage at lower temperatures for longer time would also create a failure. The formation of the intermetallic layer with the components tested will occur – it is a question of time and temperature and variations in those will affect the time to failure. For example, there is another customer specific test that requires conditioning at 90°C for 1500 hours. Indium solders fail this test.

Corrosion of indium solders

(ACEA 5) says that indium solders corrode more in humidity tests and salt spray exposure. According to (ACEA 7), several, but not all vehicle manufacturers have applied salt spray tests for interior components for years. As corrosion has never been a major issue with leadcontaining solders, not all OEMs performed salt spray tests. As corrosion is a critical point with indium solders, the salt spray test becomes important now. Vehicles have to fulfill their functions also in coastal regions. The resistance to deterioration of the components after salt spray exposure has to be considered in the design of the product. (ACEA 7) Figure 12 demonstrates the different corrosion behavior of lead based solder and of indium based solder. (ACEA 5)

Test	Lead Solder	Indium Solder
Humidity Exposure (Optical images)		Cherry Contraction
Salt Spray (SEM Cross section)		

Figure 12: Lead (left) and indium based solder after salt spray exposure (ACEA 5)

(ACEA 7) puts forward that grain boundary corrosion leads to mechanical failures of the junction. Corrosion reduces the area of the current carrying junction, which will cause higher resistance against electrical currents. This creates a hot spot, where the temperature will increase.

Further on, (ACEA 7) explains that the bond strength becomes weaker, if the solder corrodes. Heating circuits and connectors on automotive glass products regularly experience moisture and high temperature, as removing moisture by temperature is their purpose. (ACEA 7) says that indium solders deteriorate much more than standard Pb solders in humidity tests. As evidence, (ACEA 7) presents the results in Table 5, which shows the mechanical bond strength of solder joints at 80 °C temperature and 98 % of relative humidity.

Exposure period	Pb solder	45%indium solder	55%indium solder	65% indium solder
	Pull Str	ength Values – star	ndard "T-piece	' design
Original condition	352.1N	354.2N	360.0N	362.3N
2 weeks	374.9N	175.4N	210.0N	219.6N
4 weeks	373.3N	177.6N	202.1N	186.2N
8 weeks	353.2N	146.8N	142.2N	132.3N
12 weeks	339.3N	75.2N	30.1N	89.4N

Table 5: Bond strength of solder joints after conditioning at 80 °C and 98 % relative humidity(ACEA 7)

According to (ACEA 9), the tests were conducted at the NSG European Technical Centre in UK. The 65 % indium solder was not the Antaya lead-free alloy, but another alloy composed of 65 % indium, 2.5 % silver and 32.5 % tin. This composition differs from the Antaya alloy, which is composed of 65 % indium, 30 % tin, 4.5 % silver and 0.5 % copper.

(ACEA 9) explains that the above test is a customer specific test. The customer's pass criterion is a pull strength of more than 78.4N for all connectors tested. The results in Table 5 are average values. The table below shows the spread of results with 65 % indium solder for clarification.

Test period	Average strength	Pull	Minimum	Maximum
4 weeks	186.2N		116N	247N
8 weeks	132.3N		77N	172N
12 weeks	89.4N		ON	197N

Table 6: Average, minimum and maximum pull strength results for 65 % indium solder test results (ACEA 9)

(ACEA 9) states that the standard customer test is 4 week duration, but for new materials (i. e. lead-free solder) this customer had requested an extended test period of 12 weeks. According to (ACEA 9), this specific customer meanwhile does no longer require the 12 week test for validation, but accepts a four weeks maximum test duration. (ACEA 9) insists, however, that there is still evidence of some low pull strength values with some indium solders after the four weeks so that further evaluation would be required.

Additionally, OEMs specify in their acceptance criteria that there should be no change in appearance. Indium solders change significantly even after the 720 hours (four weeks) humidity exposure.

Pb containing	65%Indium containing		
solder	solder		

Figure 13: Optical appearance of lead (left) and indium based solder after 720 h humidity exposure (ACEA 9)

(ACEA 7) says that the exposure to salt spray also reduces the performance of indium solders as shown in Table 7 below. In a customer specific test, the solder joints were exposed to a 5 % salt spray mist at 35 °C in the NSG European Technical Centre in UK. (ACEA 9) indicates the pass criterion with a pull strength of more than 120 N in all cases, which the indium containing solders do not meet, while the lead solders easily pass the test. The 65 % indium solder is composed of 65 % indium, 32.5 % tin and 2.5 % silver (65In32.5Sn2.5Ag). It is not the lead-free Antaya alloy (65 % indium, 30 % tin, 4.5 % silver and 0.5 % copper). (ACEA 9) states that results for the Antaya alloy are available, but cannot be published due to confidentiality issues. According to (ACEA 9), other indium solders with compositions similar to the tested ones also fail this test.

Salt Spray		Average Pull Strength		Minimum Pull Strength		Maximum Pull	
Exposure Tim	ie					Strength	
		Pb	65% Indium	Pb	65% Indium	Pb	65% Indium
		Solder	Solder	Solder	Solder	Solder	Solder
168 hours		209.5N	144.8N	184.4N	123.6N	231.5N	156.0N
336 hours		251.5N	119.3N	176.6N	68.7N	307.1N	166.8N
672 hours		209.5N	26.3N	191.3N	ON	219.7N	65.7N

Fable 7: Bond strength	of solder joints after salt s	prav conditioning tests (ACEA 9)

(ACEA 7) concludes that the performance of indium solders deteriorates significantly when exposed to salt spray conditions so that with time the bond strength is below customer requirements. The standard lead containing solder does not show the same level of deterioration and maintains a good level of performance. For a product with expected long term reliability this loss of performance in the humidity and the salt spray tests is a significant concern.

1.5.5 Environmental, resource- and health-related arguments

Indium resource availability

(ACEA 5) puts forward that the EU Commission has identified indium as a critical raw material. The (Ad-hoc Working Group 2010) actually put indium on the EU's list of critical raw materials in 2010. The (Commission 2011a) interprets the study "Critical Metals in Strategic Energy Technologies" stating that five metals commonly used in these technologies – neodymium, dysprosium, indium, tellurium and gallium – show a high risk of shortage. Europe depends on imports for many of these, for which there is rapidly increasing global demand and limited supply, often concentrated in a few countries with associated political risks. Furthermore, they are not easily recyclable or substitutable.

(ACEA 5) states that, because the availability of indium cannot be guaranteed, industry cannot rely on it to replace lead in solders. Indium use is essential e.g. for the production of certain solar cell types (thin film), for the production of ITO (indium tin oxide layers) in LCD displays and specific semiconductors. Even in white LED's used for energy efficient illumination Indium is needed. With 500 to 600 t/a annual worldwide production (ACEA 5), indium is very rare and in about 6 years it is expected that demand will exceed current production capacity. (ACEA 5)

"In solders for soldering on automotive glass, indium is not essential. Replacing lead in the specific automotive application by indium would influence the Indium availability for these applications and sharpen the critical indium availability. The limited indium availability and the

scarcity are reflected in volatile high prices. The price for one kilogram of indium currently is around 800 USD, whereas one kg of lead is around 2.4 USD." (ACEA 5)

Higher lead-emissions and energy consumption for indium

(ACEA 1) purports that the use of indium increases the lead emissions to the environment and the energy consumption. To substantiate this claim, (ACEA 2) submitted a "Selected Life Cycle Inventory and Impact Assessment Results for Indium and Lead". The scope of the study is limited to mining and refining of indium and lead. Table 8 shows a summary of the results.

	GWP 100 (kgCO₂e/kg)	Primary Energy Demand (MJ/kg)	Lead Emission to air (gPb/kg)	Lead Emission to fresh water (gPb/kg)
Indium	599.96	8290	24.54	1.015
Lead – Primary (Germany)	1.80	26	0.04	0.012
Lead – Primary (N. America)	2.32	30	0.13	0.013
Lead – Primary & Secondary (Sweden)	0.43	28	0.01	0.023
Lead – Primary (ELCD Mix)	1.77	27	0.05	0.009
Lead – Primary & Secondary (Germany)	1.53	20	0.02	0.005
Lead – Primary & Secondary (N. America)	1.74	22	0.05	0.005
Lead – Primary & Secondary (ELCD Mix)	1.52	21	0.02	0.004

Fable 8: Selected impact categor	and inventory emissions	for indium and lead (ACEA 2)
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The following figures illustrate the results from the above table.



Figure 14: Lead emissions into air from mining and refining of 1 kg of indium and lead (ACEA 2)



Figure 15: Lead emissions into water from mining and refining of 1 kg of indium and lead (ACEA 2)



Figure 16: Global warming potential from mining and refining of 1 kg of indium and lead (ACEA 2)

(ACEA 1) claims this study to be the scientific evidence that the use of indium instead of lead in automotive glazings increases energy consumption and lead emissions.

(ACEA 1) quotes the ELV Directive to substantiate its environmental arguments. According to its recital 1, the ELV Directive targets, "[...] first, to minimize the impact of end-of life vehicles on the environment, thus contributing to the protection, preservation and improvement of the quality of the environment and energy conservation [...]". Article 1 stipulates that the ELV Directive aims at "[...] the improvement in the environmental performance of all of the economic operators involved in the life cycle of vehicles [...]".

(ACEA 1) concludes that, due to the results shown above, the intentions and objectives of the ELV Directive exclude the use of indium as a lead substitute in exemption 8 (i).

Recycling aspects

(ACEA 5) brings forward that the ELV Directive and the elements of the European waste legislation ensure that end-of-life vehicles (ELVs) as well as waste from the repair and maintenance of vehicles enter well defined utilization and recycling paths. Vehicles have to be registered and deregistered. This is a significant difference to other waste sources, where

smaller appliances and parts are not always entering the foreseen utilization routes. (ACEA 5)

(ACEA 5) further on explains that the current trend in the recycling of vehicle glazing is for the glass to remain in the vehicle when it is sent for shredding. In the shredding process, the glass is broken into small fragments. Any solder will remain adhered to the "T" piece and connector wiring together with some small glass fragments. The predominately metal pieces pass on through the separation process. Ferrous materials are separated in a magnetic field. Aluminum, using Eddy Current technology and non-ferrous materials, including copper, are separated in the dense media separation section of the process. The solder content is passed on with the copper based waste stream for recycling. (ACEA 5)

A specific recycling of indium based solders is not possible and the indium will end as tramp element in other recycled metals or in residues of further metals refining processes. So indium will be lost by dissipation, whereas the conventional solder enters the established recycling routes and is recovered. (ACEA 5)

The glass is included in a mixed aggregate stream consisting of stone and brick etc. which is used as a secondary aggregate material for road making, pipe laying and building construction work. (ACEA 5)

Missing risk assessment for indium

(ACEA 5) recommends the EU Commission to consider risk assessment aspects regarding ecotoxicity and adverse effects in humans of indium, before making any decision for substitution. Some toxicological investigations have shown that Indium compounds can create a lot of adverse effects regarding health and environment. Expanding usage of indium should not be recommended until comprehensive data about indium and indium compounds are published. Assuming that indium is used in EU in a quantity above 100 t/a, the risk assessment should be available until 2013 for final registration under REACH. (ACEA 5)

Occupational health aspects

According to (ACEA 5), during the processing of solder joints containing lead on vehicle screens, state of the art devices prevent workers' exposition to solder dust. This is also in line with EU occupational health regulations. There is no evidence of any challenge known. (ACEA 5)

Summary of ACEA's environmental arguments

From the perspective of the total environmental impact and the critical resource availability, (ACEA 5) sees the use of indium in this application very questionable. The higher lead emissions into the environment, the inevitable loss of indium in this application during vehicle utilization, the high energy consumption and the overall environmental load being higher than for lead use disqualify the use of indium in automotive glazings as substitute for lead.

1.5.6 Time for the implementation of lead-free solutions

Like in the previous review (cf. Öko-Institut 2009), (ACEA 6) claims that 54 months of transition time would be needed, as illustrated in Figure 17. With some new potential substitutes claimed to be found recently, the implementation process is currently more or less at 54 month baseline point of the roadmap (see stars in Figure 17). According to (ACEA 6), this gives evidence that there are around 3 to 5 years required for implementing the substitute in new type approved vehicles.



Figure 17: Time for implementation of lead-free glazings (ACEA 6)

(ACEA 6) describes the transition in more details.

- Determination of applications requiring further development

 Preparation of samples for all applications
 3 months
 The samples must originate from "normal" production.
 - b. Soldering 1 month
 Roughly: 20 applications x 200 connectors = 4000 connectors must be soldered. Since no adapted tooling for the series designs is available, all connectors must be processed by hand on a manual series production line.
 - c. Tests and evaluation

- 2 months
- 2. A lab-development has to be started for all applications where no drop-in is feasible
 - a. Optimistically, the stakeholders assume for all such applications (only) two development cycles á lab-analysis of the failure-reasons, definition of process or product actions
 1–2 months
 - Execution of optimization 2 months
 If new tooling is required (e.g. new printing screens), time of delivery has to be taken into account

C.	Preparation of samples incl. soldering	0.5 months

d. Tests and evaluation 2 months

Total: 6 months per cycle, ergo a good 12 months of development. The Parallelization of the developments for different applications are limited by man-power. Hence, if the number of applications is big (>3), delays are unavoidable.

- Presentation of prototypes for all applications at all OEMs, as all OEMs will want to apply their validation program
 - a. Preparation of prototypes on series or pilot lines 3–6 months depending on OEM demands.

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D.	Validation program OEMs	3 months
c.	Field tests with prototypes	15 months
	Can be done in parallel to industrialization	

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After this validation, introduction of the new technology into running development projects can start. The minimum lead-time until SOP (start of production) is 1 year, if no big engineering of production lines is required. Otherwise (e.g. new printing room), 1.5 - 2 years are realistic.

4. Industrialization (including required invest)

a.	Invest preparation	(dossier)	and decision	3 months
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- b. Engineering (compilation of list of requirements until PO) 3–6 months
- c. Time of delivery 3–6 months
- d. Waiting for shut-down 0–6 months Larger modifications of the shop floor, e.g. an additional printing room or a modification of an automated soldering line, are only possible during summer or Christmas shut-down
- e. Start-up and ramp-up 3 months

Total: 1 to 1,5 years

The extent of the required actions is crucial, e.g. if no space on the shop floor is available to install a new printing machine + curing station (which unfortunately is quite often the case), it is necessary to change the complete line design. Then 1.5 year is quite challenging. If "only" a flux application apparatus has to be added to a line with amply shop floor space (and amply cycle time!!!), the engineering can be done in 6 months.

5. Testing in second prototype phase of OEM

In the 2nd prototype phase cars are produced with toolings that are either series or close to series. The cars are used for thorough testing of the complete system. As for example chemicals used in the interior may interfere with the solder contacts, it is

required that also the material used for the solder contacts is the same that will be used for series, thus the glass for these prototypes must have been produced under conditions close to series.

- a. Car buildup (prototypes!) 1 month
- b. Laboratory testing (climate, shaker, ...) 2 months
- c. Summer- and winter testing 2 months each, not including the time for waiting for correct weather conditions in the relevant countries.

Total: around 9 months.

- Laboratory-testing of glass soldered in series-process 3 months As for the second prototype phase no parts from series-process may be available, additional laboratory testing is necessary with series parts, which requires time including production of test specimen
- 7. Testing in third prototype phase for new model with glass produced and soldered in series process

As for the second prototype phase no parts from series-process may be available, there is only the third prototype phase to test the parts coming from series tooling and series process.

a. Car buildup (prototypes!) 1 month
b. Laboratory testing (climate, shaker, ...) 2 months
c. Summer and winter testing 2 months each, not including the time for waiting for correct weather conditions in the

Total: around 9 months

relevant countries.

8. Field-/laboratory testing of prototype parts that passed the first "drop-in-test" of the glass-suppliers

In order to get first results and hints on what to focus on in the further development, first prototypes of glass with lead-free solder connectors are tested in laboratory as well as in current series-cars under heavy driving conditions and special climates. The main purpose of this test and the corresponding laboratory-test is to get a comparison between laboratory and real-life conditions: does the laboratory test really reflect real-life conditions?

According to (ACEA 6), these tests do not influence the total time needed for the development, they are done in parallel.

(ACEA 6) arrives at a total time of 54 months confining that in all cases of mentioned periods it was assumed that all work can be perfectly parallelized for all applications, products, plants, lines, customers, etc. Since this technology concerns all customers and all plants and service centers, the limiting resource is man-power. The required know-how according to the stakeholders is very specific and cannot be studied at universities. All engineers are trained by the glass industry and there are only about 15–20 experts in all companies in total all over the world. Such experts hence are difficult to find, according to the stakeholders.

1.6 Antaya's arguments for repealing the exemption

Antaya wants exemption 8 (i) to expire end of 2012 and tries to prove that its lead-free 65 % indium solder is a full substitute for the lead solders that have been used in glazings so far. The main arguments are

- the lead-free alloy has been used successfully in several vehicles from several manufacturers
- the low melting point of the lead-free alloy is not a concern as the temperatures at the connectors using the alloy remain well below 100 °C.

1.6.1 Maximum temperature rise at powered heat grid connectors of backlights

Antaya opposes the OEMs' request to extend the exemption beyond 2012. Antaya presented several tests to prove that the temperatures at the connectors of backlights remain well below 100 °C, even if the backlight heating is switch on additionally.

(Antaya 2) describes a test of a BMW backlight conducted by Trialon⁵. The test according to Antaya proves that the temperature rise in a backlight and the connectors is lower if the heat grid is switched on at higher temperatures.

⁵ Trialon Corporation, <u>http://www.trialon.com/;</u> last accessed 19 October 2011

The tested BMW windshield was placed in a climate chamber with thermocouples placed on the center, right and left bus bar, left top and left bottom ground, right top and right bottom power. Figure 18 shows the test setting and the identification labels on the tested backlight.



Figure 18: Test arrangement and labels of the tested backlight (Antaya 2)

The test was started at 20 °C powering the windshield for 30 minutes with 14 V, switching off for five minutes and then back on for another 30 minutes. The same procedure was repeated at temperatures between 30 °C and 80 °C in steps of 10 °C respectively. Figure 19 shows the maximum temperature rises after powering the connectors at different ambient temperatures.
Ambient	0	Left Bus	Left Top	Left	Right Bus	Right Top	Right	
Temperature C°	Center	Bar Ground		Ground Bar		Power	Power	
20	24.83	17.63	10.67	9.27	8.37	11.68	8.48	
30	10.27	8.00	3.94	2.98	1.10	3.92	1.19	
40	9.46	6.79	2.53	2.16	0.95	3.18	0.99	
50	13.08	5.19	5.41	2.24	1.81	4.54	2.20	
60	12.67	8.14	5.85	3.05	2.59	5.10	2.82	
70	8.28	5.05	3.85	1.30	0.46	2.19	1.20	
80	11.46	6.45	4.56	2.77	2.59	4.08	3.17	



Figure 19: Maximum temperature rise in °C (Antaya 2)

(Antaya 2) concludes from the above test results:

- The center grid is always hotter than the bus bars or the connectors.
- The temperature rise is significantly greater when the ambient temperature is in the lowest range.
- The highest absolute temperature on the connectors was less than 85 °C

For the understanding of the result it is important to know that the connectors are to be found under left top and left bottom ground, and under right top and right bottom power. The maximum temperature thus must be calculated adding the maximum temperature rise of 4.56 °C at 80 °C ambient temperature to the 80 °C ambient temperature resulting in 84.56 °C. (Antaya 9)

1.6.2 High temperature test in Seville

Test setting

(Antaya 7) describes a test conducted in Seville, Spain, from 6 to 30 August 2010. EDAG⁶ has done the test report summary by order of Antaya. Antaya has designed and conducted the test.

EDAG, according to (Antaya 9), is a large independent development organization for automotive companies. Antaya asked EDAG for their opinion, how realistic the Antaya measurements are: (e.g. the possibility that the temperature on glass under realistic conditions will not reach a temperature above 100°C). "For EDAG, Antaya's test setup is understandable (referring to the available test documentation)" (Antaya 7) and "Seville is a suitable place for the temperature measurements."

Four test vehicles with a hatchback were parked in Seville, Spain, facing south with their backlite. They are directly exposed to solar radiation with no obstruction between sun and vehicle. The tested vehicles are:

- VW Eos
- Skoda Octavia
- Audi TT
- Peugeot 407

In each car 5 temperature sensors are installed at following places:

- 1. Passenger side heat grid terminal
- 2. Center of glass
- 3. Driver side heat grid terminal
- 4. Ambient temperature inside vehicle (backseat)
- 5. Ambient temperature outside vehicle (feed through hatch)

⁶ EDAG Group, <u>http://www.edag.de/index_html?set_language=en&cl=en;</u> EDAG contact for the Seville project: Markus Renkert, <u>Markus.renkert@edag.de</u>, +49 (0) 661 / 6000-489



Figure 20: Sensor positions

Figure 20 shows the location of the outside ambient temperature sensor (5) in the location above the trunk lid. (Antaya 12) explains the illustration shall show that the sensor should be outside the vehicle. The ambient temperature sensors (5) were put under the cars so that they would be completely shaded from sunlight and to accurately record the ambient temperature.

The temperature of the sensors and time was recorded and saved every 5 minutes. The total test duration for each car was 2 weeks. Every day the test administrator downloaded the data from the measuring equipment. Before downloading the data, the administrator turned on the heated defrost in the vehicle for two complete cycles. The time when the first cycle starts has to be noted. This should as close to 2 pm as possible.

Test results

Table 9 shows the maximum temperatures recorded during the test period with powered heat grids.

Table 9: Car models and maximum temperatures in test vehicles with powered heat grid (prepared from Antaya 7and Antaya 12 data)

		Car Model							
Se	nsor Position	VW EOS	Skoda Octavia	Audi TT Coupé	Peugeot 407				
1	Passenger Heat Grid Terminal	88.0	85.5	89.0	89.1				
2	Center of Glass	92.1	92.3	90.3	96.6				
3	Driver Heat Grid Terminal	87.1	85.0	93.3	88.4				
4	Ambient Inside Vehicle	51.6	56.8	56.1	58.8				
5	Ambient Outside Vehicle	47.3	41.1	42.5	43.3				
	Rise Passenger Heat Grid Terminal	48.6	48.6	50.4	47.1				
	Rise Center of Glass	52.1	55.6	53.6	54.9				
	Rise Driver Heat Grid Terminal	48.4	47.5	53.7	46.9				
	Angle of Glass to Vertical	65.6°	55°	70°	65.6°				
	Glass Thickness (mm)	3.15	-	3.2	3.85				
	Light Transmission Backlight (%)	73,5 ±1,5%	Green Glass 70 % (estimated)	Green Glass SGN, 70 % (estimated)	71% ±1,5%				

According to (ACEA 12), the information was not readily available from the glass manufacturers for the "estimated" values. (ACEA 12) highlights that in all cases the terminals were on the opposite side of the black ceramic with 0% light transmission.

The (Antaya 7) data show that the highest temperature with powered heat grid was 93.3 °C measured at the driver heat grid terminal of the Audi TT Coupé. The hottest overall temperature of 96.6 °C occurred at the center of the Peugeot 407 glass.

The hottest day during the test period in Seville was 43.3 °C on 10 August 2010. The vehicle with the highest temperatures on the heat grid terminals on this day was the Peugeot 407. Table 10 shows the temperatures before and after the heat grid was turned on. (Antaya 7)

Sensor Position	Heat Grid Off	Heat Grid On
Passenger Heat Grid Terminal	77.3 °C	87.4 °C
Center of Glass	74.0 °C	93.6 °C
Driver Heat Grid Terminal	72.5 °C	86.1 °C

Table 10: Highest temperatures in the Peugeot 407 on the hottest day (prepared from Antaya 7 data)

EDAG states in (Antaya 7) that the highest actual measured terminal temperature without powered heat grid was 77.3 °C, which coincides with the maximum temperature measured in the Peugeot 407 on the hottest day on 10 August 2010, where 43.3 °C ambient temperature were recorded. The maximum temperatures with powered heat grid on that day are lower than the maximum temperatures with heat grid switched on. (Antaya 9) explains that on this day the test administrator missed the peak temperature for the day. The heat grid was cycled on that day at the time when the ambient temperature was 41.2 °C.

The hottest temperature recorded was 96.6 °C with powered heat grid at the center of the Peugeot 204 glass (see Table 9). This temperature was, however, not measured on the hottest day, but on the next day, 11 August 2010. (Antaya 9) explains that on that day, the maximum ambient temperature during the period that the heat grid was being cycled was higher (42 °C) which resulted in this higher temperature at the center of the glass.

Connector temperature extrapolation for the hottest day recorded in Europe

The highest temperature in the test period in August 2010 was 43.3 °C, while the highest temperature ever recorded in Europe was 48.8 °C, measured in Seville, Spain, according to (Antaya 7). The maximum recorded temperature in Europe thus is 5.5 °C higher than the maximum ambient temperature during the test. EDAG presents a worst case scenario calculation in (Antaya 7) adding the + 5.5 °C to the highest temperature measured during the test at a connector on the hottest day, which are the 77.3 °C recorded in the Peugeot 407 on 10 August. This 77.3 °C are at the same time the highest connector temperature in the test period. EDAG states that the highest temperature to be expected at a connector would be 82.8 °C. EDAG assumes, however, that physical effects (such as "cooling" of the glass towards the air outside the vehicle would lead to a connector temperature below these 82.8 °C at the unpowered connectors.

EDAG does the same worst case calculation for the powered connector. The highest terminal temperature under power was 87.4 °C (see Table 9). Adding the + 5.5 °C to this

temperature would result in a maximum temperature of 92.9 °C at the powered connector. (Antaya 7)

This worst case scenario does not take into account that the highest temperature at the powered connector was not measured on the hottest day, but one day later at 41.2 °C ambient temperature. The temperature difference to the hottest day recorded in Europe thus is 48.8 °C - 41.2 °C = + 7.6 °C. The worst case temperature thus should be 95 °C at the powered connector.

Beyond the ambient temperature measurements, Antaya did not provide information about the weather conditions during the test besides the statement that it was hot and sunny (Antaya 9). No data are available about the solar radiation performance during the test.

Figure 21 shows average climate data for Seville.

Month	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Year
Average high °C	15.9	17.9	21.2	22.7	26.4	31.0	35,3	35.0	31.6	25.6	20.1	16.6	24.9
(°F)	(60.6)	(64.2)	(70.2)	(72.9)	(79.5)	(87.8)	(95.5)	(95)	(88.9)	(78.1)	(68.2)	(61.9)	(76.8)
Daily mean °C (°F)	10.6	12.2	14.7	16.4	19.7	23.9	27.4	27.2	24.5	19.6	14.8	11.8	18.6
bally mean c(1)	(51.1)	(54)	(58.5)	(61.5)	(67.5)	(75)	(81.3)	(81)	(76.1)	(67.3)	(58.6)	(53.2)	(65.5)
Average low °C	5.2	6.7	8.2	10.1	13.1	16.7	19.4	19.5	17.5	13.5	9.3	6.9	12.7
(°F)	(41.4)	(44.1)	(46.8)	(50.2)	(55.6)	(62.1)	(66.9)	(67.1)	(63.5)	(56.3)	(48.7)	(44.4)	(54.9)
Precipitation mm	65	54	38	57	34	13	2	6	23	62	84	95	533
(inches)	(2.56)	(2.13)	(1.5)	(2.24)	(1.34)	(0.51)	(0.08)	(0.24)	(0.91)	(2.44)	(3.31)	(3.74)	(20.98)
Avg. precipitation days (≥ 1 mm)	6	6	5	7	4	2	0	0	2	6	6	8	52
Sunshine hours	179	183	224	234	287	312	351	328	250	218	186	154	2,898

Figure 21: Climate data for Seville, Spain (World Meteorological Organization (UN), Agencia Estatal de Meteorología, in "Antaya 9")

August in average is the second hottest month in Seville with the second highest number of sunshine hours, the lowest precipitation and zero precipitation days.

1.6.3 High temperature test in Death Valley

Test setting

(Antaya 8) describes another temperature test conducted at Devil's Golf Course in Death Valley, California, USA, on 1 September 2009. Antaya conducted the test.

(Antaya 9) explains that Devil's Golf Course is a location in the middle of Death Valley where car manufacturers frequently test their cars, for example Mercedes-Benz⁷. According to (Antaya 9), July and August are equally hot, and on 1 September 2009, the ambient temperature was 47.7 °C.

Two vehicles were tested, a Ford Crown Victoria and a GM Chevy Impala, both equipped with standard glass. The glass is probably a 70% light transmission glass (clear green glass) to be found on 90 % of vehicles. (Antaya 9) The backlights were removed from these vehicles and replaced by new OEM backlights with Antaya lead-free #65 alloy on the terminals of the Ford, and Antaya #10 lead-free alloy on the Chevy terminals.

Vehicle	Alloy	Liquidus	Solidus
		Temperature in °C	Temperature in °C
Ford Crown Vic	**30Sn 65In 0.5Cu 4.5	127	109
	Ag		
Chevy Impala	26.5Sn, 1 Ni/Fe, 1Cu,	131.5	123.63
	1Sb, 1Bi, 65In, 4.5Ag		

Table 11: Lead-free alloys used in the test cars (Antaya 9)

A 0.5 kg weight was fixed to each terminal, and the power and ground terminals were connected with the vehicles.



Figure 22: Connector with 0.5 kg weight, thermocouple, and indicator light wire (Antaya 8 and 9)

⁷ **Article** "Mercedes-Benz SLK Gets Final Endurance Tests on New MAGIC SKY CONTROL Roof", http://www.emercedesbenz.com/autos/mercedes-benz/slk-class/mercedes-benz-slk-gets-final-endurance-testson-new-magic-sky-control-roof/; last accessed 23 October 2011

The vehicles were parked in direct sunlight with the backlight facing the direction of the sun. Seven thermocouples were fixed at the cars, as indicated in Table 12.

METER #		METER #
1	CAR INTERIOR	11
2	OUTSIDE AIR	22
3	OUTSIDE GLASS	33
4	INSIDE GLASS	44
5	DRIVER'SIDE CLIP	55
6	PASSENGER SIDE CLIP	66
7	OUTSIDE TEMP (CAR THERM.)	

Table 12: Thermocouples ("meters") fixed in the cars (1-7: Ford; 11-66: GM Chevy)

The vehicle doors were closed and the temperature monitored for at least thirty minutes prior to the start of the test. The heat grid was turned on in both cars during 2 to 4 pm, which according to (Antaya 8) is the warmest period of the day, and the heat grid was cycled for one hour while recording temperatures at all thermocouple locations every five minutes.

Table 13: Defroster cycle (Antaya 8)

Ford D	Defroster C	Chevy	Chevy Defroster Cycle			
1	3:15	3:31	1	3:15	3:36	
2	3:31	3:47	2	3:36	3:46	
3	3:47	4:02	3	3:46	3:57	
4	4:02	4:15	4	3:57	4:08	
			5	4:08	4:15	

After completion of the test, the terminals were inspected for damages or separation from the glass.

Test results

Table 14 shows the temperatures recorded during the test.

		FORD					#65			CHEVY				#10
	1	2	3	4	5	6	7		11	22	33	44	55	66
Time	°F/°C	1	°F/°C	°F/°C	°F/°C	°F/°C	°F/°C	°F/°C						
2:50	135/57.2	129/53.8	163/72.7	167/75	173/78.3	166/74.4	116/46.6	1	132/55.5	124/51.1	163/72.7	171/77.2	169/76.1	170/76.6
2:55	135/57.2	121/49.4	161/71.6	166/74.4	173/78.3	163/72.7			132/55.5	119/48.3	160/71.1	169/76.1	168/75.5	168/75.5
3:00	136/57.7	124/51.1	159/70.5	165/73.8	173/78.3	161/71.6			132/55.5	122/50	158/70	167/75	168/75.5	166/74.4
3:05	136/57.7	120/48.8	159/70.5	164/73.3	173/78.3	159/70.5	() (132/55.5	121/49.4	156/68.8	165/73.8	167/75	162/72.2
3:10	136/57.7	130/54.4	160/71.1	165/73.8	174/78.8	162/72.2	0 0		132/55.5	12451.1	159/70.5	167/75	169/76.1	166/74.4
3:15	136/57.7	123/50.5	160/71.1	165/73.8	174/78.8	165/73.8			132/55.5	123/50.5	161/71.6	169/76.1	169/76.1	168/75.5
3:20	137/58.3	121/49.4	171/77.2	186/85.5	192/88.8	188/86.6			132/55.5	121/49.4	161/71.6	190/87.7	191/88.3	192/88.8
3:25	136/57.7	118/47.7	165/73.8	182/83.3	189/87.2	181/82.7			132/55.5	120/48.8	151/61.1	186/85.5	189/87.2	190/87.7
3:30	136/57.7	120/48.8	170/76.6	187/86.1	194/90	186/85.5			132/55.5	120/48.8	154/67.7	190/87.7	195/90.5	195/90.5
3:35	137/58.3	126/52.2	176/80	191/88.3	196/91.1	192/88.8			133/56.1	122/50	157/69.4	195/90.5	195/90.5	202/94.4
3:40	138/58.8	123/50.5	175/79.4	191/88.3	197/91.6	191/88.3		-	133/56.1	122/50	157/69.4	194/90	195/90.5	199/92.7
3:45	137/58.3	122/50	176/80	191/88.3	195/90.5	192/88.8	3		134/56.6	127/52.7	156/68.8	195/90.5	195/90.5	200/93.3
3:50	137/58.3	117/47.2	169/76.1	184/84.4	188/86.6	186/85.5	116/46.6		134/56.6	119/48.3	150/65.5	189/87.2	191/88.3	194/90
3:55	137/58.3	122/50	170/76.6	185/85	190/87.7	186/85.5			134/56.6	122/50	152/66.6	191/88.3	194/90	191/88.3
4:00	138/58.8	120/48.8	176/80	189/87.2	194/90	192/88.8			134/56.6	121/49.4	155/68.3	191/88.3	191/88.3	195/90.5
4:05	138/58.8	116/46.6	174/78.8	187/86.1	191/88.3	190/87.7	117/47.2		135/57.2	123/50.5	153/67.2	193/89.4	190/87.7	197/91.6
4:10	139/59.4	123/50.5	176/80	189/87.2	194/90	192/88.8	8	2	135/57.2	121/49.4	156/68.8	194/90	193/89.4	195/90.5
4:15	139/59.4	119/48.3	171/77.2	184/84.4	189/87.2	187/86.1	118/47.7	1	136/57.7	118/47.7	154/67.7	190/87.7	190/87.7	187/86.1

Table 14: Temperatures recorded during test (Antaya 8)

The first reading with defroster turned on was at 3:20 pm. The highest temperatures recorded at the connectors are 91.6 °C in the Ford and 94.4 °C in the Chevy, in both cases with powered connectors. Before powering the connectors, the highest connector temperatures remained below 80 °C in both car models.

According to (Antaya 8), no damages or separations were detected on any of the terminals after the test.

1.6.4 Application of Antaya lead-free connectors in vehicles worldwide

Antaya claims that its parts are used in many vehicles on the road, and have qualified for several upcoming car models. According to Antaya, this proves that the use of lead in exemption 8 (i) is avoidable.

Table 19 in Annex I shows information from Antaya about several vehicles from various manufacturers, which, according to Antaya, use the Antaya lead-free 65 % indium solder on glazings. Table 20 in Annex I lists vehicle manufacturers and vehicle models for which Antaya claims that its lead-free solder will be used in upcoming programs. (Antaya 12) states that "upcoming programs" refers to vehicles where Antaya's lead-free solder has passed the manufacturers' tests prior to start of production and hence has been identified and approved for use.

1.6.5 Antaya B6 alloy

(Antaya 1) says to have developed the new lead-free alloy "B6" to address high temperature concerns.

	Solidus	Liquidus
Alloy 65	109 °C	127 °C
Alloy B6	129.24 °C	140.64 °C

Table 15: Melting points of Antaya lead-free solders (Antaya 9, Antaya 11)

(Antaya 6) provides a test showing that the alloy B6-01 could hold 30 connectors loaded with 500 g of weight and stored at 125 °C over 500 hours without failures in the soldered contacts. Annex IV lists a series of tests, which this alloy has passed, among those several of the higher temperature tests ACEA suggests in Annex II for the higher temperature requirements.

No evidence is available that this alloy has been successfully qualified and/or applied in automotive glazings. (Antaya 9) states that one glass company asked Antaya for an alloy with higher temperature ranges, but Antaya was not able to button down the exact specs and as there was no programme that called for higher temperatures. According to (Antaya 9), the routine is that the glass companies give Antaya the standards and details of the programme and Antaya supplies the most appropriate part and alloy. ACEA and the OEMs' suppliers thus had no access to this alloy so far.

The B6 alloy may thus be an appropriate lead-free solder for automotive glazings. Unlike for the #65 alloy, there is no evidence that the B6 solder has passed OEM and glass maker qualification programs. Until that time it remains unclear whether and how far the B6 alloy is a general lead-free substitute.

1.6.6 Time required for implementation of lead-free glazings

(Antaya 1) claims that lead-free programmes can be launched in approximately 90 days from request for quotation. The average transition time is four months, and three months, according to Antaya, is a reasonable timeframe offering adequate time for pre-production testing, soldering equipment optimization for lead free, and other customary administration. The total time required from request for quotation would then be around six to seven months starting from request for quotation.

1.7 St. Gobain lead-free solder developments

(St. Gobain 3) announces to have a lead-free substitute developed and industrialized by the end of 2012. The lead-free solder is based on bismuth with a melting point of 139 °C solidus and 141 °C liquidus. Table 16 presents St. Gobain's schedule.

Table 16: Schedule towards the industrialization of the St. Gobain lead-free solder solution (St. Gobain 2 and 4)

Flexible connector,	Lab development of product and process	finalized		
small bridge for	Availability of prototype parts for car manfuacturer testing	since July 2011		
antenna	Industrialization	September 2011 - December 2012		
	Lab development of product and process	targeted for February 2012 (rigid)		
Multingle for antenna	Lab development of product and process	and June 2012 (multipole)		
wullipole for antenna,	Availability of prototype parts for car manfuacturer testing	from April 2012 on		
rigid heating connector	Inductrialization	March (rigid) and July (multipole)		
	Industrialization	2012 to end of 2013		

(St. Gobain 2) believes the draft test requirements in Annex II set up by ACEA et al. to be the relevant tests. Figure 23 shows test results obtained with the "flexible bridge" connector. (St. Gobain 2) states that all eight tests defined in Annex II have been passed successfully.









lead ref.





Figure 23: (St. Gobain 2) test results

Concerning the question whether and how long the exemption should be extended, (St. Gobain 1) thinks two timescales to be of importance:

- Component development and industrialization on the glass manufacturers' side
- Component validation on the complete vehicle on the car manufacturers' side.

(St. Gobain 1) can start production of components end of 2012, and can already supply prototypes of lead-free flexible connectors now. Other connector geometries can follow in mid-2012. (St. Gobain 3) has supplied backlights with lead-free soldered standard flexible bridge connectors to several major OEMs within Europe. Due to confidentiality agreements (St. Gobain 3) cannot provide any further details.

(St. Gobain 4) says that it will have three suppliers in mid 2012, and will develop more suppliers for the further ramp-up in 2014 to 2018. For 2013, (St. Gobain 4) can supply around 2 million heating field connectors, 1 million of lead-free antenna connectors, and around 0.5 million units of various other connectors. With these 3.5 million units, (St. Gobain 4) can satisfy the supply needs of its customers. Additionally, (St. Gobain 4) is ready to start licensing discussions with competing glass suppliers to enable lead-free supplies from these glass manufacturers.

(St. Gobain 2) believes that there is no reason to continue the use of lead-containing solders once the St. Gobain lead-free solution is industrialized and the car manufacturers have validated it.

1.8 Critical review

Article 4 (2) (b) (ii) allows an exemption from the substance restrictions if the use of these substances is unavoidable. The use of a restricted substance is unavoidable

- if scientifically and technically viable alternatives are not available.
- if the negative environmental or health impacts caused by substitution are likely to outweigh the environmental, health and safety benefits of the substitution.

1.8.1 Use of Antaya indium solders in vehicles

Generally, the use of a restricted substance is avoidable in a specific application if there are manufacturers who have found ways to avoid the use of the restricted substance in the same or a comparable application. It was therefore first checked whether any OEMs apply the indium-based lead-free solder successfully in their products.

Uses of the Antaya lead-free alloy as indicated in Table 19 of Annex I

(Antaya 13) submitted information that its lead-free solder has been or will be used in several car manufacturers' vehicles. Table 19 and Table 20 in Annex I show the details. To consolidate Antaya's claim, the OEMs were asked to either confirm the information in Table 19 (uses) and Table 20 (intended uses), or to otherwise identify incorrect information.

• Volkswagen

(Volkswagen 1) confirmed that its car models "Jetta" and "New Beetle" have been using the Antaya lead-free solders on the backlights. According to (ACEA 9), around 250.000 cars were equipped with lead free connectors. (ACEA 12) claims that this use was unauthorized. According to (ACEA 9), Volkswagen will switch back to lead-soldered connectors completely before 19 December 2011, as the lead-free soldered connectors do not comply with the new draft test requirements (see Annex II).

• SAAB

(ACEA 9) says not to have any information for SAAB, which is also listed as using the Antaya lead-free alloy in Table 19 of Annex I.

• Ford

Ford confirmed the use of the Antaya lead-free solder in (ACEA 9) stating, however, that it is not in use since longer time. It could already be proved in (Öko-Institut 2009) that the Ford "T" Bird used the alloy in a heated wiper circuit along the bottom edge of the windshield. Around 70,000 units of this vehicle were built from 2002 to 2005. The fact that the design using lead-free solders was only used for around 3 years does not go back to failures, but has other reasons, which are not known to the reviewers.

General Motors/Opel

The first lead free application on annealed glass was in one series of vehicles launched in the early 1990s. (Öko-Institut 2009) already could confirm that the GM "U" Van (Chevrolet Venture, Pontiac Montana and Oldsmobile Silhouette) used the Antaya lead-free alloy in an integrated circuit replacing the antenna in the windshield. The design was in use from around 1999 to 2001 or 2002.

In order to comply with the European ELV Directive, (GM 1) has started using a lead free alloy with 65 % of indium on tempered glasses in selected new model vehicles starting in 2008. (GM 2) states that Antaya is a supplier of this lead-free alloy. (GM3) shows an analysis of this solder proving that it contains less than 0.1 % of lead and thus is lead-free according to the requirements of the European RoHS Directive.

(ACEA 9) confirms that both the Chevrolet "Volt" and the Opel "Ampera", the technical equivalent for the European market, use Antaya indium-based lead-free solder with 65 % indium as listed in Annex I.

Intended uses of the Antaya lead-free alloy as indicated in Table 20 of Annex I

• Nissan

In (ACEA 12), Nissan confirms the intended use of the lead-free alloy for two NISSAN vehicles as indicated in Table 20 of Annex I. (ACEA 12) explains that none of the models is for the European market. One model is a van type and the other one a station wagon type. Both vehicles have backlites with quite low inclination (near to vertical). (ACEA 12) contests that there is a decision of NISSAN for Antaya materials. The test phase for one vehicle has started and will be started for the other one soon. There are no test results available yet.

• AGC North America/GM

According to (ACEA 12), AGC North America has not yet validated the Antaya leadfree solder, but just started some preliminary tests with the Antaya solder in the perspective of the GM programs.

• Daimler

In (ACEA 12), Daimler contests the correctness of information in Table 20 of Annex I about upcoming uses of the Antaya lead-free alloy in Daimler cars. Daimler fails, however, to specify whether this just refers to a part of the information such as the start date of the program or to the fact that the lead-free alloy shall be used in Daimler cars.

Field failures of the Antaya lead-free alloy

The OEMs that used or have been using the Antaya lead-free alloy were asked whether any field failures occurred, which demonstrably go back to the use of this alloy.

General Motors

(GM 1) is not aware of field use failures of the indium alloys in its products using the Antaya lead free alloy, neither in the vehicles produced in the 1990s nor in the ones produced since 2008. (ACEA 12) confirms that currently there is no particular indication for failures or warranty issues for the lead free soldered components in GM and Opel cars.

• Volkswagen

(ACEA 12) alleges that an actual VW quality check (20.12.2011) reports mid of November one first malfunction for a Jetta backlite (heater without function) after 7 months. (ACEA 12) does not provide any evidence that this failure goes back to the use of the lead-free solder.

General remarks

(ACEA 12) claims that in North America and Europe vehicle use profiles and customers are different. Even (USCAR/SAE 40) and the VDA/ACEA draft specifications (see Annex II) are different. (ACEA 12) asserts that in Europe the specifications do more consider long life functional aspects of backlites. ACEA et al. do not substantiate this claim with facts.

(ACEA 12) assumes that the application in vans and SUV's are not the critical ones in terms of screen inclination and glass color. (ACEA 12) also puts forward that compared to the total vehicle production there is only a low volume of vehicles equipped with Antaya lead free solder. (ACEA 12) concludes that field failure counts possibly would not cut through threshold limits and be indicated.

(ACEA 9) states that based on vehicle quantities built worldwide between 2000 and today with estimated 400 Mio vehicles, less than 700 000 vehicles in this period and thus less than 0.2 % were equipped with lead-free soldered contacts. These figures may be correct, but do not prove that the lead-free solder is principally inappropriate, as such a situation will always occur with changes from one material to another one. Additionally, it must be taken into account that until a few years ago the OEMs relied on exemptions and that there were thus no lead-free connects in vehicles. The overall number of vehicles produced since lead-free solder connects have been discussed is thus less than the estimated 400 million, the share of vehicles with lead-free connects higher.

Conclusions

There is evidence that GM/Opel, Ford and Volkswagen have been using the Antaya leadfree alloy. The earliest uses date back to the nineties of the last century (Ford, GM) and to the beginning of the 2000s (GM). Heated applications are of particular concern. The earliest use in the Ford starting 2002 was a heated wiper, which, according to PPG in (Öko-Institut 2009), has a current load of around 5 Ampere and thus is less critical than heated backlights with 20 - 30 Amperes of electrical load. GM, however, has been using the alloy in heated backlights since around 2008. GM/Opel and Ford state that no field failures have been reported so far in any of the lead-free soldered applications. Even though ACEA et al. assert that Volkswagen had not authorized the use of the Antaya alloy, around 250,000 Volkswagen cars were equipped with lead-free soldered connectors. ACEA et al. suspect field failures, but do not provide evidence that failures actually occurred, and that such failures were related to the use of the lead-free alloy.

The reviewers hence conclude that the lead-free alloy has been in use in several applications in vehicles, partially for years. The uses comprise heated applications with lower electrical currents used around 10 years ago and heated backlights with higher electrical currents since around 2008. No cases of field failures have been reported so far, which demonstrably go back to the use of the lead-free alloy.

1.8.2 Test failures and mechanical instability of the lead-free alloy

Test failures

(ACEA 9) negates that the uses and intended uses of applications of the lead-free alloy in Annex I can be seen as evidence that the lead-free alloy actually is a viable substitute for the lead solders. (ACEA 9) says that the products supplied by Pilkington in North America were tested according to GM's test specification only. As the lead-free soldered product was supplied to GM as a vehicle specific development, the fact that Antaya's lead-free solder is being used on those vehicles according to (ACEA 9) is not an indication that it is capable of passing all OEMs' requirements.

ACEA et al. assert that user profiles are different in Europe and Northern America, and that in Europe, the specifications do more consider long life functional aspects of backlites. (USCAR/SAE 40) tests and specifications and the new draft test requirements (Annex II) are different. Each OEM has its own tests, which the solder has to pass. The tests are thus different within the European manufacturers as well, and there is no evidence that all tests of all OEMs for all automotive glazing applications are stricter than those of US manufacturers. On the contrary, the Opel Ampera using the lead-free alloy, even though a brand of the US company GM, is or will be sold in Europe. ACEA et al. do not back their European longevity claim and the shorter life of backlights in the USA, and there is no evidence how different user profiles would affect the life time of lead-free solder joints in automotive glazings.

ACEA says that a lead-free alloy must pass all tests of all OEMs' in order to be accepted as substitute and thus justify the repealment of the exemption. ACEA et al. vice versa do, however, not provide evidence that the indium lead-free alloy does not pass any OEMs' requirements for any application ranging from antennas to backlights and other applications. ACEA admits, however, that the Antaya lead-free solder may be appropriate for niche applications.

ACEA argues that OEMs are responsible for the functioning and the safety of their products. They hence must carefully decide what kind of materials and technologies they use. Tests are important decision tools in this context. OEMs therefore should have the right to test materials and technologies according to their established test programs and to accept materials only if they pass the OEM-specific test for a specific application. The consultants share this point of view.

(ACEA 9) also says, however, that there are more than 100 customer specific validation tests solders have to pass before being launched in vehicles. Following the OEMs' request that each material must pass all tests of all OEMs would thus put the OEMs into a position to block each substitution, as there may always be an OEM who applies a test, which a potential substitute cannot pass. Additionally, given the multitude of different tests, it must be assumed that lead-solders do not pass all tests of all OEMS either without adaptations and redesigns.

Although the consultants support ACEA's argument that substitutes must pass the OEMspecific tests, they do not agree with ACEA's static view on testing. On the one hand, the consultants understand that OEM-specific tests reflect each OEMs' knowhow and experience gathered over the years, and that changing tests thus may be delicate. On the other hand, tests should reflect the real conditions. ACEA et al. say that temperatures at connectors depend on geometries and other factors. A proper redesign and constructive changes of glazing applications therefore can reduce temperature and mechanical loads. The temperature field and lab experiments show that the temperatures are different depending on the position of the connectors on the glass, and on the car's geometries. Alternative connector designs, geometries and constructions may as well enable reducing mechanical and thermal loads. An optimum design may thus expands the usability of the lead-free alloy. If the thermal and mechanical requirements are lowered by design optimization, the tests could be re-specified as well reflecting the lower field requirements. ACEA et al. applied this approach when they increased the test requirements for solders used in automotive glazings (cf. Annex II) after they had measured high temperatures of more than 120 °C at connectors in their lab tests.

ACEA's argument that each substitute must pass all tests of all OEMs therefore is too static and not in line with the requirements of Art. 4 (2) (b) (ii) requiring the substitution of banned substances wherever their use is avoidable.

Mechanical instability

In section 1.5.4 on page 12, ACEA et al. claim that the lead-free alloy is mechanically unstable and thus not appropriate. They put forward that intermetallics occur, which grow over time and thus result in the failure of the solder joint.

The presence of an intermetallic phase at the interfaces of the solder joint with the surfaces of the joined articles (silver print, connector) is a pre-condition for the forming of a solder

joint, regardless whether lead-free or lead solders are used. It is also clear that intermetallic phases grow and coarsen over time in both cases and in the end result in the failure of a lead-free as well as a lead solder joint. Every solder joint thus has a limited life time. The presence of intermetallic phases and their growth thus is not an evidence that the alloy will fail during the life time of a vehicle. The crucial question is whether the intermetallics grow and coarsen so fast that the solder joint fails within the intended life time of the vehicle. This, however, has never been proven for field uses of the alloy listed in Table 19 of Annex I, even though the first vehicles using the alloy were shipped in the early nineties of the last century.

(ACEA 5) argues that high temperatures accelerate the growth of the intermetallics. The lead-free alloy thus may be more prone to premature failures in applications, where the solder joint continuously or permanently operates at higher temperatures like for example in heated backlights. Higher temperatures actually increase the growth and coarsening of intermetallic phases. The evidence presented (Figure 7, Figure 8 and Figure 9 on page 13 ff) is, however, inconsistent. The environmental conditioning conducted with the indium solders is different from the conditioning of the lead reference. A proper comparison would require the same conditioning applied on the test sample and on the test reference. Additionally, ACEA et al. do not reveal, whether the tested indium solders actually are the Antaya lead-free alloy stating that confidentiality agreements do not allow the publication of the tests with the Antaya lead-free solder. The transparency of the review process requires, however, clear and transparent evidence. The exact composition of a solder alloy may have strong influence on the intermetallics and their growth in a solder joint.

There is thus no transparent and consistent evidence available proving that the Antaya leadfree alloy principally is not a viable substitute due to its adverse mechanical properties.

Additionally, the alloy has already been used in the early 1990s and in the early 2000s without proven failures. Neither intermetallic growth nor corrosion as presented in the corrosion tests in Figure 12 on page 17 demonstrably have caused failures. The application in the early 2000s of the lead-free alloy was in a heated wiper, and no failures have been proven so far. The lead-free alloy could thus withstand the mechanical stress and corrosion at least to a degree that no failures have been observed, even in a heated application, which increases the growth rate of intermetallics.

The first heated backlight applications, which might operate at higher temperatures, started around 2008. No problems have been reported since then. As 2008 is a short period of time, a certain risk is of failure is remaining. GM/Opel and Nissan obviously classify the remaining risk as acceptable, as they have been using or intend to use the lead-free alloy on heated backlights.

The information provided by ACEA et al. shows that the Antaya lead-free alloy is probably not be a generally viable substitute. Vice versa, however, it cannot be concluded that the lead-free alloy is principally inappropriate for all applications and that the general exemption 8 (i) therefore must be continued.

1.8.3 ACEA et al. high temperature tests

The melting point of the Antaya lead-free solder with 65 % indium is 109 °C solidus and 127 °C liquidus. The liquidus temperature is the temperature above which the alloy is completely liquid. The solidus temperature is the temperature below which the alloy is completely solid. In the range from 109 °C to 127 °C, crystals and the molten phase of the alloy coexist.

ACEA et al. want to prove that the melting point of the Antaya lead-free indium solder is too low. They claim that solar irradiation may rise the temperatures at backlight connectors to as much as 126 C and even to 132 °C if additionally the defroster is switched on (Table 1 on page 6).

Intransparency of evidence

(ACEA 7) says that "Specific car test reports and tested alloys may contain competitive information and are sent separately as confidential information". "The tests were conducted by members of the joint association industry expert group applying OEM's and suppliers appropriate test specifications." No independent test results are thus available, and details of the temperature tests such as pictures and descriptions of the thermocouple placements, the exact test settings, etc., are not available. Antaya, for example, submitted such information about its temperature tests, and ACEA et al. excessively commented and criticized the test settings and other details. This shows the importance of detailed and transparent information.

Thermal laboratory testing versus field testing

ACEA et al. substantially base their high temperature arguments on laboratory simulations. The simulations are based on DIN 75220 "Ageing of automotive components in solar simulation units". Mr. Trubiroha, whom (ACEA 8) acknowledges as a known expert for weathering of polymers and polymer photo ageing, explains in (Antaya 18) that this standard specifies methods to determine the (photochemical) ageing behavior of polymeric automotive components in their original position and type of mounting. A field (bank) of metal halide lamps at the ceiling (and walls) of the chamber simulates the spectral irradiance of the global solar radiation on a horizontal reference plane in the climatic chamber. The irradiance of solar radiation on the horizontal reference plane is set to 1,000 W/m² ± 5 %. Within the test volume - limited by this plane - the irradiance levels on horizontal surfaces of a car shall be within 1000 W/m² ± 10 %. The air temperature in the test chamber is set to 42 °C ± 3 °C.

(Antaya 18) criticizes the simulation. The radiation is only diffuse radiation, while sunlight consists of 90 % of parallel radiation. In the thermal simulation, there is hence no beam of parallel radiation. (Antaya 18) further on puts forward that according to DIN 75220, the global solar radiation is not simulated by xenon arc lamps, which would be the best radiation sources for this purpose. Because the thermal load to the climatic chamber would be too high, the radiation sources are metal halide lamps. According to (Antaya 18), these types of lamps have clear deficiencies in simulating the whole spectrum of global solar radiation.

(Antaya 18) says that the backlights in the thermal simulation will be exposed to a more or less poorly simulated global solar radiation with irradiance levels of up to 1.100 W/m^2 . Irradiance by terrestrial radiation emitted and reflected by the car, the walls and the ceiling will be at least 550 W/m². The additional influence of the hot filters of the radiation sources can hardly be estimated. That means that the total irradiance in the climatic chamber is at least 1,600 W/m².

(Antaya 18) concludes that the heat load to the glazing is at least 15 % to 20 % higher than outdoors. The radiation sources are very different and hard to compare, as there is only diffuse radiation in the simulation experiment. As a consequence, the glazings of a car are irradiated at the same time. The varying arrangement of the lamps demonstrated in Figure 24 will also influence the results of the studies on the heat load of the interior. DIN 75220 climatic chambers, according to (Antaya 18), are optimized and only suitable for photo degradation studies of exterior components of a car and are unsuitable to simulate the heat load of a car and its glazing in an open air climate.



Figure 24: Climatic chambers according to DIN 75220⁸

ACEA et al. were asked to comment on Mr. Trubirohas arguments raised in (Antaya 18). (ACEA 8) insists that the total incident power to an exposed surface in the climatic chamber must be comparable to the power load in the real world and doubts that the irradiation performance in the climatic chambers is higher than in the real world. (ACEA 8) also insists that the spectrum of the used lamps matches the solar spectrum quite well asserting that it does not matter whether the light is collimated or diffusely reflected. Only the power integral is of importance. According to (ACEA 8) the calibration of the climatic chamber and the real world can be approved by experiments: for example dash board temperature, vehicle cabin temperature, glazing temperature etc. Thus, the climatic chamber test has a high degree of reliance. Based on that, (ACEA 8) is sure that 120 °C for dark glazing can be reached in the

⁸ Sources of pictures from top left to bottom right: <u>http://www.go2ross.eu/Deutsch/fahrzeug-validie.html</u>; http://www.imtech.de/fileadmin/redaktion/Nachhaltige_Energietechnik/Leistungen/Umsi/Pruefkammern/Fahrze ugpruefkammern/Downloads/D-06-035_Bewitterungskammer.pdf; http://www.iabg.de/automotive/erprohung_gualifikation/gesamtfahrzeug/sonpensimulation_de.php; http://atlas-

http://www.iabg.de/automotive/erprobung_qualifikation/gesamtfahrzeug/sonnensimulation_de.php; http://atlas-mts.com/en/products/solar_simulation/solarconstant_system/index.shtml

field. Nevertheless, (ACEA 8) admits that there is a spectral mismatch and that the climatic chamber test conditions are idealized, as only radiation and temperature can be adjusted.

The DIN 75220 standard is not designed for the thermal simulation of vehicles. The sense of a standard is to make sure that test procedures are appropriate to measure and to test what shall be tested, and to enable test results, which are reproducible, comparable and, above all, reflect real conditions, even if these conditions may be extreme ones. The application of a standard hence only makes sense if it is applied to what it was specified for. In the reviewers' opinion, a thermal simulation of a vehicle in a climate chamber would only make sense using a standard for the thermal simulation of vehicles, which then would have to include the walls, the geometries and other aspects of the chamber.

In this case, where no standard test is available, it would have been even more important to substantiate the high temperature claims with results from field tests. (ACEA 8) admits that field tests are still necessary. ACEA et al. claim that high solar loads can heat up connectors on normal glass to temperatures of 100 °C. If the dark glass computation study (see next section) is correct, it would have been easy to prove in a field test that the 15 °C and more temperature increase with dark glass results in connector temperatures, which are clearly too high for the alloy's solidus of 109 °C, even if the available solar load outdoors would remain below the maximum of around 1,000 W/m². Given the fact that the review date for exemption 8 (i) has been known since February 2010, there was sufficient time to set up and conduct some field trials with transparent and well documented experimental conditions and test settings in hot areas with high solar irradiation performance.

On 26 January 2012, ACEA et al. declared the document (Volkswagen 2) as nonconfidential, which before had been submitted as confidential information. For time constraints, this document could not be integrated into the report properly. (Volkswagen 2) measures temperatures of around 90 °C at non-powered backlight connectors in a field experiment in Arizona.

Overall, the field experiment submitted by ACEA et al. shows that temperatures of around 90 °C can be achieved without defrosts switched on. For further measurements of ACEA et al. resulting in temperatures of far beyond 100 °C, a field test confirmation is not available.

The field results show that at least for some geometries and designs, the Antaya lead-free alloy is not an optimum alloy, as it offers little security margin for higher temperatures. An application in areas where high temperatures may occur or cannot be avoided thus requires careful design, consideration about the placement of the connectors, or the use of the alloy may even be impossible.

Antaya as well provided several temperature measurements showing maximum temperatures of around 80 °C. The burden of proof for their high temperature claim was on ACEA et al. As temperatures far beyond 90 °C with switched-off defrosters have not been confirmed in vehicles with reliable tests, the Antaya measurements were not further critically reviewed against the ACEA measurements and arguments.

Computational study for thermal loads on dark glasses

ACEA et al. provided a computational study claiming that temperatures at backlight connectors will be around 15-20 °C hotter than standard glass for the same glass thickness. The darkest glass should even be about 25 °C hotter than standard glass.

The computation study results are, however, questionable. In (ACEA 3) it is stated that "[...] it is really difficult to simulate the real temperature reached on glass in a vehicle parked under intensive sun light in very hot areas (desert areas), the physical phenomenon being numerous and complex to model, as well as the whole geometry of the vehicle [...]." For this reason, according to (ACEA 3), "[...] a very simple model was used for the following computations [...]". The question arises whether and how far such a simple model is appropriate to reliably simulate the complex real situation.

Mr. Trubiroha in (Antaya 18) and (ACEA 8) discuss the assumptions and results of the computational study. (Antaya 18) claims that the adopted model is too simple and provides a gedankenexperiment that shall prove that temperatures of 120 °C and more are unrealistic. (ACEA 8) criticizes this, stating that "[...] each author made correct computations, and each one could be criticized for some of its choices. From our side, from a simulation point of view, we confirm a glass temperature in the black painted area of the glass well above 100°C is realistic. 120°C is possible and an actual record at 126°C is not improbable." (ACEA 8) continues that "the real interest or concern is here to measure temperatures in the peripheral black painted area of the glass, that is where the soldered terminal effectively is for most of the rear screens [...], for which area energy transmission is nearly null (hence absorption is maximized at 95-96%, whatever the color of the glass); but the actual results in the black painted area will be indeed somewhat influenced by the properties of the glass in the (more or less) transparent area and by the temperatures hence reached in that "transparent" area, up to 20°C higher in case of dark tinted glass, further to heat transfer by conduction through the glass [...]".

The above paragraph touches upon another critical point. The connectors of a backlight are soldered behind the black print area (see Figure 4 on page 10), which, according to (ACEA 8), has zero percent of light transmission and thus is like the darkest glass available already. The question is whether and how far the use of dark glass with only few percentages of light

transmission for the rest of the backlight would affect the temperature in the black print area. A clear answer to this question would require a field test.

Summing up, the computational study shows uncertainties and remains contentious. Actually, it is stated even in (ACEA 3) that "[...] it make sense for OEMs to redo some new measurement campaigns this summer in severe operating conditions, using very dark "privacy" glass (growing demand is very probable) for backlite, instead of standard tinted." These field tests are, however, not available. ACEA et al. thus cannot substantiate their high temperature claim with clear and reliable evidence. It must be stated additionally that the Opel Ampera using the Antaya lead-free alloy will actually be used with dark glass.

Paint repair at high temperatures

(ACEA 8) reminds that paint repair procedures may not be disregarded. Annex II of (ACEA 8) lists examples that paint repairs are conducted at temperatures of up to 140 °C. ACEA et al. therefore assert that the lead-free alloy cannot be used as its melting point is too low to withstand the paint repair procedures.

A paint repair temperature of 140 °C does not automatically result in a temperature of 140 °C at the backlight connectors, the more as, according to (ACEA 8), the backlights are covered with heat shields during the paint procedure. As the lead-free alloy demonstrably has been used in vehicles already, paint repair must be technically viable with the lead-free alloy. In case some OEMs actually generate higher temperatures at connectors during such repair processes, they can be expected to adapt their processes in order to achieve compliance with the ELV-Directive.

Temperature increase with switched on defoggers

ACEA et al. argue that the temperatures at backlight connectors may rise even higher if the defogger is accidentally switched on while the car parks in full sunlight. In Table 1 on page 6, ACEA et al. therefore present additional temperature measurements with defoggers switched on.

Antaya recorded temperatures of cars parked in strong sunlight in the range of 50 °C to 60 °C inside the vehicles (Table 9 on page 34 and Table 14 on page 39). (Volkswagen 2) measured around 80 °C inside the car. It is not plausible to imagine that a passenger enters such a hot car, keeps all doors and windows closed, switches on the ignition without starting the air condition and the engine, starts the defogger on purpose or accidentally and remains sitting in the car for several minutes. Defoggers could, however, be switched off automatically if the temperature inside or outside the car exceeds a certain level.

There are cars with such a feature. The manual of the Peugeot 407 with Diesel engine, for example, states on page 46: "Demisting is switched off automatically to prevent an excessive consumption of current and in relation to the exterior temperature." Mercedes Benz of Indianapolis9 for example advertises the "2012 Mercedes-Benz SLK350" with a "Rear window defroster w/auto-shutoff based on time & driving speed & exterior temp?. Daimler says in (ACEA 9) that this feature extends the heating time at low temperatures near 0°C to ensure a complete de-icing of the rear window. Peugeot explains in (ACEA 8) that this feature is used to minimize the defogger operation time and thus the electricity consumption.

(ACEA 7) opposes the idea of an automatic defogger switch-off at higher temperatures to prevent the excess heating of backlights and connectors at hot weather conditions. Peugeot says that it is usual with Diesel vehicles to include the power load of the heater operation to achieve higher exhaust temperatures for cleaning the particle filter of the catalytic converter. This action could be asked even with high temperatures outside. The regeneration of the converter is necessary and may not be suppressed and can occur at very high temperatures outside. Like above, the scenario that a passenger enters the hot car and starts such an action at closed windows and switched-off air condition is, in the contractor's view, not plausible.

According to (ACEA 7) and (ACEA 8), fogging can occur at higher temperatures under special weather conditions, e.g. at high humidity. They therefore insist that customers should have the possibility to switch on the defrost function in any case.

If, as ACEA et al. claim, fogging actually occurs in certain conditions such as in hot, humid climate, it is impossible that it happens when the backlight is exposed to strong sunlight and therefore heated above the temperature of the air inside and outside the vehicle. In case it is just hot and humid without direct sunlight on the backlight, the temperatures at the connectors must be closer to the inside air temperature, in each case, however, far from temperatures that might result in the damage of the lead-free alloy. In case fogging occurs in such a situation, it would not be a problem to switch on the defogger. Technically, it should be viable to allow the switch-on of a defogger, which had been switched off automatically before. Otherwise, the automatic switch-off for example in the Peugeot would not allow customers to switch on the defogger after the automatic operation control had switched it off and fogging might occur in adverse circumstances.

⁹ Mercedes Benz of Indianapolis, <u>http://www.mercedesofindy.com/vehicle/2012-Mercedes Benz-SLK350-Indianapolis-367192.html</u>, last accessed 8 November 2011

For the reasons explained above, the reviewers conclude that the intended or accidental operation of defoggers at high temperatures endangering the solder joints can be solved with an automatic switch-off depending on the temperature inside the vehicle. In parked cars, the defoggers normally do not operate, as the ignition is switched off, and the battery would be stressed. In the case that a passenger enters such a hot car and switches on the defogger accidentally, it should be possible to switch it off automatically. If the passengers actually intends using the defogger nevertheless, it is technically possible to allow this switch-on nevertheless without increasing the temperature of the connectors to a dangerous level. The high temperature measurements with operating defoggers thus, if relevant at all, should be possible to be solved technically.

Homologous temperature

(ACEA 3) says that, following a publication of Fraunhofer IZM, solder alloys for long term reliable solder connections should not be used at a homologous temperatures of more than 0.9 at maximum, better at 0.8 to 0.85. The homologous temperature h is the ratio of the operating temperature T_o and the solidus T_s of a solder alloy in Kelvin.

$$h = \frac{T_o}{T_s}$$

Equation 1: Homologous temperature

With temperatures of around 90 °C, which (Volkswagen 2) measures at unpowered connectors in field experiments, the homologous temperature ratio of 0.9 would be clearly exceeded for the Antaya lead-free alloy. The same applies to the around 80 °C maximum, which Antaya measures in its field tests.

Fraunhofer IZM's recommendation is a thumb rule, not a natural law. Operating solder joints at high homologous temperatures affects the life time of solder joints and their mechanical strength. The crucial aspect in the end is, however, whether the solder joint works over the expected life time. The Antaya lead-free alloy has, however, been used without known failures over years in vehicles.

Moreover, the 90 °C temperatures measured at connectors are not permanent operating temperatures, but temperatures which may occur. Additionally, in the reviewers' opinion it is important to state that not all applications necessarily must result in such high temperatures. Design and geometry changes may also enable reducing the thermal and mechanical loads.

The high homologous temperatures, which the Antaya lead-free alloy can be exposed to, thus is not a final argument against the use of the alloy, but shows that the use of the alloy requires constructive and design measures.

1.8.4 Use of Indium in the Light of EU Policies

ACEA puts forward that the Commission considers indium as a critical substance from the supply point of view (section "Indium resource availability" on page 20). The report of the (Ad-hoc Working Group 2010) actually lists indium as a critical raw material for the European Union. The (Commission 2011b) indicates a high risk of supply for indium, as illustrated in Figure 25 below.

	Market	Factors	Political	Political Factors				
Metal	Likelihood of rapid demand growth	Limitations to expanding production capacity	Concentration of supply	Political risk	Overall risk			
Dysprosium	High	High	High	High				
Neodymium	High	Medium	High	High				
Tellurium	High	High	Low	Medium	High			
Gallium	High	Medium	Medium	Medium				
Indium	Medium	High	Medium	Medium				
Niobium	High	Low	High	Medium				
Vanadium	High	Low	Medium	High				
Tin	Low	Medium	Medium	High	Medium			
Selenium	Medium	Medium	Medium	Low				
Silver	Low	Medium	Low	High				
Molybdenum	Medium	Low	Medium	Medium	Low			
Hafnium	Low	Medium	Medium	Low				
Nickel	Medium	Low	Low	Medium				
Cadmium	Low	Low	Low	Medium				

Figure 25: Bottleneck analysis for metals used in strategic energy technologies (Commission 2011b)

The (Commission 2011b) assesses that the availability of indium, among other metals, could be a potential bottleneck to the deployment of low-carbon energy technologies in the EU and thus might endanger important strategic energy technologies.

The (Adhoc-Working Group 2010) therefore recommends substituting the use of such critical metals by other, less critical ones, wherever technically possible. Substituting lead by indium in exemption 8 (i) clearly contravenes this recommendation, as according to (USGS 2011), the abundance of lead is much higher than that of indium. The annual mining quantities of 3,860,000 tonnes of lead versus 546 tonnes of indium in 2009, according to (USGS 2011), demonstrate this. The (Commission 2011c) considers the critical metals as part of the Resource-efficient Europe flagship initiative in the Europe 2020 Strategy. The substitution of lead by indium therefore contradicts EU resource policies and, based on the Commission's assessments, increasing the demand of indium might have adverse impacts on sustainable development in the EU.

According to Antaya in (Öko-Institut 2008), the use of indium in automotive glazings would require around 15 tonnes of indium per year. This figure is probably too low, as Antaya states that 90 t of lead would be replaced. Based on the considerations in Table 17 on page 61, the replacement of 90 t of lead in this application would probably demand more than 15 tonnes of indium, but less than 90 tonnes per year, not taking into account any recycling, which might reduce the net demand. The additional demand should thus be between 3 % to 16 % related to the annual mining capacities.

The question arises whether and how far the consultants could recommend the continuation of exemption 8 (i) based on the scarcity of indium and the conflict with EU policies. The use of lead in glazing applications instead of lead-free, indium-based solders would be unavoidable in line with Art. 4 (2) (b) (ii) and the review practices applied so far, if indium currently or in the foreseeable future would not be available physically and thus make it impossible to buy indium-containing lead-free solders on the market regardless of the price a manufacturer would be ready to pay. The substitution would then be technically impossible, and the use of lead become unavoidable. This would justify an extension of exemption 8 (i). The consultants do, however, not have clear evidence that this situation will actually occur.

Unavailability, beyond the absolute physical unavailability, is expressed via the price. Scarce metals are more expensive than those with abundant supplies. As long as the "scarcity" of indium is expressed by higher prices, an exemption would thus not be in line with Art. 4 (2) (b) (ii), as economic arguments do not justify an exemption.

Summing up, in the reviewers' opinion the substitution of lead by indium in automotive glazings contributes to increase the demand of indium and thus contravenes important EU resource policies like the resource-efficient Europe flagship initiative. The weighting and prioritization of conflicting policies – the EU resource policy versus the legal ban of lead in

the ELV Directive - is, however, beyond the reviewers' mandate. The reviewers, according to their mandate, must prioritize the legal ban of lead in the ELV Directive in this exemption review.

1.8.5 Environmental impacts of lead versus indium use

(ACEA 5) says that substitutes introduced because of material restrictions should have proven environmental benefits. (ACEA 1) additionally puts forward that indium causes higher lead emissions and increases the energy consumption compared to the use of lead in automotive glazings (see section "Higher lead-emissions and energy consumption for indium" on page 21). According to ACEA et al., the overall environmental impacts from the use of indium in automotive glazings will cause higher environmental impacts than the use of lead. ACEA et al. therefore plead for the continuation of exemption 8 (i) for environmental reasons.

In the exemption reviews so far an exemption based on environmental arguments in line with Art. 4 (2) (b) (ii) was only possible, if applicants could prove that the adverse environmental impacts of the substitution are at least likely to outweigh the positive environmental substitution effects. The strongest argument of ACEA et al. is the study presented in (ACEA 2) according to which the use of indium results in higher lead emissions. If this argument is correct, the continuation of exemption 8 (i) for environmental reasons would be justified. A crucial question hence is whether the facts and evidences ACEA et al. provide actually substantiate their environmental arguments.

Lead emissions and energy consumption in mining and refining of lead and indium

(ACEA 2) submitted the study titled "Selected Life Cycle Inventory and Impact Assessment Results for Indium and Lead". According to (ACEA 1), this study is the scientific evidence that the use of indium as substitute of lead

- results in higher lead emissions into the environment
- results in higher energy consumption
- causes higher environmental impacts than the use of lead in automotive glazings

It must be taken into account that the results of life cycle assessments (LCA) may highly depend on the scope, the system borders, the allocation rules, and on the assumptions made. The transparency of life cycle assessment studies is hence of paramount importance. Such an LCA study should at least suffice the requirements of ISO 14040/44, which are the generally acknowledged standards for life cycle assessments (LCA). (ACEA 15) claims that "The study is prepared in accordance with ISO14040/44. The report as provided so far does not cover all formal ISO requirements. Certainly a comparative study following ISO needs an

ISO conform report and in case of being a comparative study intended for publication an external review, which was not possible to provide in the short time frame."

(ACEA 15) lists several of the requirements of an LCA according to ISO 14040/44. In the reviewers' opinion, the study presented in (ACEA 2) is not compliant with ISO 14040/44. If this study shall prove that the use of indium as a substitute for lead in automotive glazings increases the overall environmental impacts, several crucial points need to be addressed:

- The study assesses "[...] the production of 1 kg of material (indium or lead) from cradle-to-gate, that is from raw material extraction, through to a finished, high purity metal product ready for transport from the production site." A clearer goal and scope definition is missing. The system boundaries are not made clear.
- The study only takes into account the mining and refining stage. The later processing
 of lead and indium or the glazings in vehicles and the end-of-life phase are not taken
 into account. (ACEA 2) thus does not provide any base for the statement that the use
 of indium results in overall higher environmental impacts than the use of lead in
 automotive glazings.
- The study compares energy consumption and lead emissions for 1 kg of lead and indium respectively. The substitution of the lead solder for lead-free solder will, however, be volume equivalent, not mass equivalent. As the density of the lead-free solder is lower, the substitution of 1,000 g of lead solder would require roughly between 700 and 900 g of lead-free solder as shown in Table 17.

	Lead-solder	Indium-solder		
Density of solders (g/cm ³)	8.5 to 10.5 (ACEA 16)	7.54 (Antaya 14)		
Content in solder (%)	40 to 70 lead (ACEA 16)	65 indium (Antaya 14)		
Volume equivalent solder	1,000	720 to 880		
(g)				
Metal content	400 to 700 (100 %)	470 to 570 (81 % to 118 %)		

Table ²	17:	Volume-	equivalen	t substitution	of solders
rabic		volume	cquivalen	i SubStitution	01 3010013

In consequence, the consumption of indium is different from the consumption of lead. In tendency it will probably be lower assuming that the indium-solder will replace lead-solder with around 60 % lead content. The lead content of the solder used in the joint test program (Annex III), for example, was 62 % (Öko-Institut 2009) resulting in the use of a higher amount of lead than indium. The emissions of (ACEA 2) would have to be corrected accordingly and the emissions of lead would hence increase, while those of indium would decrease.

- The inventory analysis is provided as confidential information. The basic underlying data on which the results are based thus are not transparent.
- A critical third party review is missing.
- Assumptions and uncertainties are neither revealed nor appraised with respect to their potential effects on the final results. A sensitivity analysis is missing.
- The study results are based on a market-based allocation using the prices of the metals. This method is common. Prices of metals are volatile, and the prices of different metals may develop differently. The study results thus, among other factors, depend on the metal prices. A sensitivity analysis of the price influence is not available.
- The higher emissions of lead related to indium use are only plausible for the mining and refining of indium from lead-containing ores, such as the zinc ores assessed in (ACEA 2). The assessment is based on the ores and processes of a specific mine. Neither is it clear how much of the indium on the market is mined from such ores, nor how far the assessed indium mining and refining route is representative for indium mining. The lead emissions may be different for other ores. In case indium is mined from ores, which do not contain lead, there would be no lead emissions related to the use of indium. No alternative indium-containing ores, mines and refining processes were assessed.
- It is not clear which ore types were taken into account for the assessment of lead emissions from lead mining and refining. It is further on not clear whether and how far the assessed lead ores as well as the mining and refining processes are representative for the mining and refining of lead. The lead emissions may be different for other mines and processes.
- It is not clear whether lead emissions (toxicity) and energy consumption are the only relevant environmental burdens and impacts. (ACEA 2) does not specify why and how these two environmental impacts were chosen only.

(ACEA 2) thus does not provide clear and transparent evidence that the use of indium results in overall higher lead emissions.

The same in principle applies to the energy consumption for indium compared to lead as far as the concrete numbers are concerned. Comparing the prices of indium and lead, it is, however, plausible to assume that indium is much scarcer than lead and that the energy consumption for indium in mining and refining is higher compared to that of lead. On the other hand, however, the energy consumption for the soldering processes would be lower due to the lower melting points of the indium alloy. The production of the solder alloys from the refined metals as well might cause lower energy consumption for the same reason. A life cycle based assessment of energy consumption including solder manufacturing, the soldering process and the end-of-life phase would hence be important.

Assuming the overall energy consumption would be higher for the use of indium, there is no method based on natural science to decide, whether for example potential hazardous effects from lead use or from higher energy consumption related to indium are more negative for the environment. This would require a weighting of different environmental impacts. Such a weighting, however, is a societal and political task and thus beyond the contractors' mandate, unless the Commission would authorize the use of a specific weighting method to be used in such cases. Principle considerations and a similar case can be found in (Öko-Institut 2006), page 12 ff, and in the monthly report 3 in Annex I of that report.

Recycling of lead from end-of-life vehicles

ACEA et al. put forward that, due to the ELV Directive and other legislation, end-of-life vehicles (ELVs) as well as waste from the repair and maintenance of vehicles are recycled (see section "Recycling aspects" on page 23). They argue that the metals in the solder on backlights and other glazings are recycled from the copper fraction. Contrary to that, according to ACEA et al., a specific recycling of indium based solders is not possible and indium will be lost.

The recycling of end-of-life vehicles (ELVs) is politically intended and environmentally beneficial. The fact that an ELV is recycled, however, is not a justification for an exemption. If the use of a restricted substance is avoidable, an exemption would not be in line with Art. 4 (2) (b) (ii), regardless of the recycling of ELVs. Recycling thus in this exemption review process can only be relevant if it contributes to reduce the environmental impact from the use of lead to a degree that it is likely to be more environment friendly than the use of indium.

(ACEA 2) claims that around 50 % of lead on the market are secondary lead. It is, however, not clear how much lead is actually recycled from ELVs in general, and from the solders used in automotive glazings in particular. The fact that ELVs enter recycling processes does not mean that all materials in the ELV are recycled. (ACEA 2010), a study report about "Costs and environmental benefits of the end of life-vehicles (ELV) Directive (2000/53/EC)

'heavy metals ban" does not contain information on how many percentages of lead in an ELV are actually recycled, and (ACEA 9) cannot provide such data either. The overall recycling success in the end is the result of the individual performance of each step in the end-of-life process chain. Crucial for the recycling success is how much of the glazing solders actually end up in the copper fraction, from which lead can be recycled, while it is unlikely to be recycled from other fractions. (ACEA 8) cannot give exact figures which route the connectors go. The actual recycling rate of lead from ELVs thus is not known. Additionally, the (Commission 2009) reports that several member states do not achieve the reuse and recycling targets stipulated in the ELV Directive, and that ELVs are directly exported to Africa and Asia, where their fate at end of life is not known.

The recycling of indium is critical. According to (Deubzer 2007), depending on the installed production technologies, there are copper smelters claiming that they can recycle indium from copper fractions generated during the shredding and mechanical separation of waste electrical and electronic equipment. (UNEP 2009) confirms that indium recycling capacities are available, even though still limited. Like for lead, it is thus not clear how many percentages of indium in automotive glazing solders of an ELV will actually be recycled. To improve the recyclability, an appropriate pre-treatment – possibly the manual removal of the indium-soldered connectors prior to shredding – may enable higher recycling rates for indium. The higher economic value of indium compared to lead may be an incentive for removing the connectors. Due to the high concentration of indium, it could probably be recycled in a smelter, which has the technical capabilities.

Summing up, even though the recycling of ELVs is environmentally beneficial, it is not clear how many percentages of lead or indium in ELVs are or actually can be recycled.

1.8.6 Missing risk assessment for indium

(ACEA 5) mentions toxicological investigations showing that indium compounds can create a lot of adverse effects regarding health and environment. ACEA did not provide any more concrete or substantiated data, but asks the Commission to wait with a decision on the use of indium until a risk assessment is available for indium within the REACH registration.

So far, elementary indium is neither banned nor restricted in Annexes XIV or XVII of the REACH Ordinance. Indium phosphide has been classified as carcinogenic substance in the Classification and Labeling of hazardous substances, resulting in a possible ban of indium phosphide in REACH Annex XVII. Placing indium phosphide on the market for consumer uses in substances and in mixtures will no longer be allowed. Such a decision on

classification of a substances as carcinogens can be, in some cases, the starting point of a number of legislative actions, including an in depth risk assessment. As far as this development raises concerns about the toxicity of indium, this issue needs further examination.

An exemption would be justified in line with Art. 4 (2) (b) (ii) and the review practices developed and applied in the past exemption reviews if the toxicity of indium is likely to at least outweigh the positive effects of lead substitution in automotive glazings. Lead and its compounds are already restricted in RoHS Annex XVII. There are currently no data showing that indium as it is used in the lead-free solder might be equally or even more toxic than lead. An exemption based on the toxicity of indium would hence not be justified.

Recommending an exemption based on a missing risk assessment and the prospect of future in-depth investigations about the toxicity of substitutes in the course of REACH registration, authorization and restriction processes so far has been beyond the consultants mandate. Any such decision therefore is under the authority of the Commission only.

1.8.7 Time required for implementation of lead-free glazings

As ACEA et al claim that the remaining time until the expiry of exemption 8 (i) is not sufficient to implement lead-free glazings, the question becomes relevant how long the implementation of such solutions would take.

Like in the previous reviews, the stakeholders' point of views on the time needed for the implementation of lead-free automotive glazings deviate considerably ranging from a few weeks (Antaya 19) on the one hand and up to five years (ACEA 6) on the other hand. Given the huge differences, it must be assumed that each stakeholders' reasoning is guided by its own business and other interests so that an in-depth investigation would be required to arrive at a more comprehensive and impartial result. The consultants' mandate does not cover such an independent in-depth study, and the consultants therefore check the plausibility and consistency of the stakeholders' data and draw conclusions from this scrutiny.

In its statements, Antaya does not take into account the time for validation and qualification of lead-free solutions at OEMs and glass makers. The OEMs claim that they need one year minimum for in-field testing in a pre-series of vehicles. In the (Joint Working Group 2008), Antaya stated that such in-field testing is not required, as field experience is available from the uses of the Antaya alloy in several vehicles. The consultants' approach is that, as OEMs are responsible for the safety and reliability of their vehicles, they should be given the

opportunity to follow their customary qualification procedures. Further on, as in (Öko-Institut 2009), the Antaya schedule is based on the assumption that all lead-free solutions are dropin replacements of lead-based solutions, meaning that no or only minor adjustments would be required at glass makers and OEMs. Even though this may be correct for some applications, the changeover from one material – lead solders – to another one – lead-free solders – in many cases may require more comprehensive adaptation and redesign works at glass makers. The low melting point of the alloy, for example, may require a careful design or redesign to ensure a security margin for higher temperatures. Temperatures of around 90 °C as measured by Volkswagen and around 80 °C as indicated by Antaya in field tests result in a homologous temperature of 0.9 and higher for the lead-free alloy (cf. section "Homologous temperature" on page 57). This may affect the long-term reliability and therefore needs design and constructive measures to limiting for example the mechanical load on connectors, and to avoid or at least minimize the time where the lead-free alloy is exposed to such high temperatures. Additionally, the lead-free alloy does not pass all OEMs' usual tests, which requires more intensive adaptation work at least at and for those OEMs.

St. Gobain, as a stakeholder offering a lead-free solution, indicates the time for industrializing lead-free glazings with around 15 to 18 months (cf. Table 16 on page 41). During the last review of exemption 8 (i), Antaya had provided a contact to PPG, the glassmaker who had worked with Antaya's lead-free solders for the use in GM and Ford cars. PPG stated in (Öko-Institut 2009) that "[...] this is no way an endorsement of Antaya's indium based solder as it is up to each manufacturer to test and evaluate materials they use. Different silvers, paint compositions and processing parameters can all affect the reliability of the final product." "[...] the automotive application of this new technology will require validation and performance testing before indium based lead free solder could be certified for use in production. The lead time for this type of effort typically requires a 2 year minimum to complete exposure testing and an additional 1 year lead time to establish supply."

The information available to the consultants from this and the previous review thus does not support Antaya's claim that lead-free soldering for automotive glazings can be implemented within a few weeks, even though it cannot be excluded that it might be possible in certain cases. Typically, the transition time required in the glass plants is in the range of around three years.

In the previous review in 2009, ACEA et al. had submitted the same roadmap like the one presented in Figure 17 on page 25 as a worst case scenario justifying a transition time of 54 months (4.5 years). (ACEA 6) submitted this figure again for the current review claiming that "This gives evidence that there are around 3 to 5 years required for implementing the substitute in new type approved vehicles." Taking into account that ACEA et al. consider the
scenario as a worst case scenario, three years of total transition time should be sufficient to reliably implement lead-free glazings. This would be in line with the result of the last revision, where a three year period was recommended in (Öko-Institut 2009). Additionally, contrary to the situation in 2009, with the Antaya B6 alloy and the St. Gobain lead-free alloy, two more lead-free solder candidates are available, which reduces the probability that the worst case scenario actually applies. It should be easier to find the most viable solution for each application within three years. Further on, it must be stated that the exemption would only expire for new type approved vehicles. The number of annual lead-free implementation programs is thus limited. Besides soldering, there are other solutions thinkable as well such as inductive couplings, which would not require solders at all.

Finally, it must be stated that there is no progress at the OEMs concerning the implementation of lead-free solutions since 2007/2008, when the exemption was first reviewed. Instead, given the fact that this is the third time ACEA asks for the continuation of the exemption, it is justified to demand ACEA et al. to give highest priority the implementation of lead-free solutions instead of further delaying the progress towards lead-free soldering solutions in automotive glazings. The fact that GM/Opel have started the implementation of lead-free solutions already is a further argument not to grant a longer transition time.

The consultants conclude that technically, a total of three years should be sufficient to implement lead-free solutions in automotive glazings. Taking into account the overall situation, a longer implementation time would not be in line with the requirements of Art. 4 (2) (b) (ii).

1.8.8 Summary

ACEA et al. claim that the Antaya indium-based lead-free solder principally is not a technically viable solution as the alloy is not an optimum substitute, and that use of indium increases the overall environmental impacts compared to the use of lead in automotive glazings. ACEA et al. therefore request the continuation of exemption 8 (i) for another three years after its expiry end of 2012.

ACEA et al. did not provide transparent evidence that the use of the indium-based lead-free alloy in automotive glazings would cause an overall higher environmental impact compared to the use of lead-based alloys. The technical arguments submitted show that the Antaya lead-free alloy may not be appropriate for all glazing applications in automotive glazings, and that its use may require redesign and constructive changes. ACEA et al. did, however, did not prove that the lead-free alloy is principally not a viable solution. The documented uses of the lead-free alloy in the past and in an increasing number of recent applications underpin the conclusion that the Antaya lead-free alloy can be used, even though possibly not for each and every application of each OEM and not without design adaptations.

ACEA et al. rejects this view claiming that a lead-free solution must be an optimum substitute meaning that it is appropriate for all kinds of automotive glazing applications of all OEMS or otherwise is no solution. This claim is not in line with the legal requirements. Article 4 (2) (b) (ii) of the ELV Directive restricts the use of lead to those applications, where its use is unavoidable. In the review practices developed in the last years in agreement with the Commission, OEMs are obliged to implement lead-free solutions and to adapt the design, constructions and geometries and to take all measures enabling the implementation of lead-free glazing solutions wherever the use of lead is avoidable.

Table 18 summarizes ACEA's arguments and Antaya's position taken from the submitted stakeholder documents, and the consultants' positions. St. Gobain is not listed as its lead-free solution has only become available around mid/end of last year and therefore is not yet in the center of the stakeholders' discussions.

Table 18: Overview on arguments

ACEA's arguments	Antaya's position	Consultants' position					
	Testing and mechanical properties						
Antaya lead-free alloy does not pass all tests of all OEMs	 Alloy passed the tests of the Joint Industry Working Group in 2009 Several glass makers have validated the alloy 	 OEM-specific tests are important for OEMs to decide about appropriate materials and technologies No evidence that it does not pass some OEMs' tests More than 100 OEM-specific tests; if each substitute has to pass all tests of all OEMs, OEMs will be in a position to block all future substitutions, as there will always be at least one OEM whose test a substitute cannot pass Design changes can reduce thermal and mechanical burdens on solder joints, and test requirements can thus be adapted as well, because tests should reflect real life requirements 					
Antayalead-freealloymechanically unstable and not along-termreliablesubstitutebecause of-growth of intermetallics-corrosion		 Intermetallics occur and grow in lead-solders as well Test results for intermetallics in indium and lead reference not comparable Tests intransparent as tested solders not sufficiently specified Alloy applied successfully in vehicles in the 1990ies already and later on 					
Use in GM vehicles only proves that alloy can pass GM specifications; as each OEM has own test specifications, this is not a proof that alloy can be used in other OEM's vehicles	Alloy is appropriate for all applications, and several glass makers have validated the lead-free alloy	- There is no evidence that the lead-free alloy is only applicable in GM's vehicles, but not in other OEMs' vehicles					

ACEA's arguments	Antaya's position	Consultants' position				
	High temp	erature arguments				
Very high temperatures of up to 130 °C can occur at connectors, which is too high for the Antaya lead-free alloy	 Thermal simulations do not reflect real life situation Maximum temperatures of around 80 °C not exceeded in own field tests 	 High temperatures beyond around 90 °C without powered defrosts assessed in thermal lab tests or computer simulations only; verification in field tests required, but missing Temperature measurement scenarios with switched-on defroster not plausible; in case they would be accepted, lead-free alloy can still be used in non-heated applications Alloy has been used in heated and non-heated applications in vehicles without proven failures 				
90 °C more than 90% of homologous temperature of Antaya lead-free alloy; long- term reliable solder joints should not be operated beyond 90 % of homologous temperature		 90 °C not permanent operating temperature 90 % homologous temperature limit recommended as thumb rule, not a natural law Constructive and design chances can reduce thermal and mechanical loads on solder joints Alloy has been used in vehicles without proven failures 				
ACEA's arguments	Antaya's position	Consultants' position				
	Environmental a	nd availability arguments				
Use of indium in automotive glazings results in higher energy consumption and higher lead emissions	Submitted study intransparent and not representative	 Submitted study lacks transparency Essential aspects missing, e.g. assumptions made, sensitivity analysis Covers only mining and refining, not entire life cycle Representativeness of results for indium and for lead not clear 				

EU has classified indium as critical metal; indium therefore not an appropriate substitute for	Indium is abundantly available	 Use of indium in automotive glazings raises questions in the light of EU resource policies, as indium is classified as a critical metal Prioritization of EU policies (ban of lead versus avoiding use of
lead in automotive glazings		critical metals) beyond the consultants' mandate
 Indium is toxic Risk assessment of indium in REACH is not available 	Indium is not toxic, different from lead	 ACEA et al. do not provide any data about the toxicity of indium Risk assessment in REACH so far has not been a pre-condition substitutes of substances restricted in the ELV Directive have to fulfill Lead and its compounds already restricted in REACH Annex XVII Currently no data available showing that indium as used in the lead-free solder is equally or more toxic than lead In the REACH context there seem to be concerns about the toxicity of indium; this issue needs further examination Recommending the extension of the exemption would require evidence that the use of indium in automotive glazings is likely to at least outweigh the positive effects of lead substitution. Such evidence is currently not available. The consultants can only base their recommendation on information and evidence currently available, not on information that might become available in the near or more distant future. Any recommendation to extend the exemption until more indepth data becomes available within the course of REACH registration
		authorization or restriction processes would be beyond the
 Antaya lead-free alloy patented and the expiry of the exemption would create 	 Lead-free alloy only patented in the US Sufficient supplies are 	 Considerations of patents and monopoly situations beyond the consultants' mandate, as long as physical non-availability does not block the technical practicability of substitution

a monopoly situation - Supplies may be insufficient and availability of substitutes not be secured	secured	 Higher prices of substitutes due to patents or monopolies are economic arguments, which the consultants according to their mandate cannot take into account Since mid/end of 2011, St. Gobain offers lead-free solutions as well, no monopoly situation Non-availability of lead-free connector supplies not proven 					
ACEA's arguments	Antaya's position	Consultants' position					
	Time required for implementation of lead-free soldering						
 Three to five years required Exemption must therefore be extended for another three years until end of 2015 	Lead-free soldering can be implemented within a few weeks or months	 With respect to Antaya's arguments: Implementation of lead-free soldering typically requires more than a few weeks or months at glass makers, the more as Antaya lead-free solder is not a drop-in substitute for all applications and hence requires redesign, constructive and other adaptations Around three years required in total according to OEMs and glass makers, including those who have worked with Antaya's lead-free solder, to arrive at an industrial scale production and including at least a one year field testing period of lead-free solutions in vehicles 					
		 With respect to ACEA's arguments ACEA claims three to five years based on a worst case scenario Three available lead-free substitutes make implementation of lead-free soldering easier, worst case scenario hence not likely Alternative solutions not depending on soldering may open further 					

-	ways to avoid the use of lead Exemption only expires for new type approved vehicles, which limits total amount of adaptation and implementation work at glass makers and OEMs; worst case scenario hence even less likely ACEA requests extension of exemption for third time without visible progress in implementation of lead-free soldering in last four years; OEMs should therefore prioritize lead-free soldering in automotive glazings and implement it within three years The fact that at least one OEM has started implementation of lead-
	free glazings does not allow a longer transition period

1.9 Recommendation

Taking into account the information submitted, and based on the review practices developed in agreement with the Commission during the last years of exemption reviews, the further extension of the exemption 8 (i) is not in line with the requirement of Art. 4 (2) (b) (ii) to only use banned substances where their use is unavoidable. The contractors hence recommend repealing the general exemption 8 (i).

However, even though Art. 4 (2) (b) (ii) in the consultants' point of view requires revoking the exemption, the consultants would like to make clear the consequences of repealment at the end of 2012. Most OEMs have not implemented any lead-free glazing solutions. The implementation of lead-free solutions should be possible within a maximum of three years. The detailed arguments are listed in in section 1.8.7. Repealing the exemption end of 2012 would thus mean that most OEMs are not able to put compliant vehicles on the European market, as the 11 months remaining would not be sufficient. Technically, the implementation of lead-free solutions would thus require extending exemption 8 (i) for a maximum of two years to arrive at a total time to expiry of three years.

In case the Commission grants the OEMs additional time, the consultants suggest the following wording for exemption 8 (i):

Lead in solders in electrical glazing applications on glass, except for soldering in laminated glazing, in vehicles type approved before 1 January 2015, and lead in spare parts for these vehicles.

Since the first review in 2007/2008, there is no visible progress at most OEMs towards the implementation of lead-free glazing solutions. The consultants recommend not to set a review date again in order to spur progress towards the substitution of lead in automotive glazings. With the Antaya 65 % lead-free alloy, the Antaya B6 lead-free alloy and St. Gobain's lead-free solution, three substitutes are available meanwhile. It should therefore be possible to properly implement lead-free soldering solutions within almost three years.

Beyond technical and environmental arguments, the availability of indium has been discussed in this review process. The Commission has classified indium as a metal with high supply risk and therefore as a critical metal. The use of indium in automotive glazings contravenes the recommendation of the (Adhoc Working Group 2010) to substitute indium where its use is not technically required, as indium is indispensable for the deployment of strategic energy technologies such as low carbon energy technologies, and the use of indium

in automotive glazings might hamper sustainable development, as well as the use of toxic lead in automotive glazings. The substitution requirements for lead in the ELV Directive in this case are in conflict with important Commission resource policies such as the Resource-Efficient Europe flagship initiative. A prioritization of conflicting policy goals is, however, beyond the reviewers' competence. The reviewers, according to their mandate, must give priority to the legal ban of lead in the ELV Directive and therefore cannot take into account the EU resource policies in their recommendation.

In case the Commission would like to give priority to its resource policies over the ban of lead stipulated in the ELV Directive, the contractors nevertheless recommend the same timing for exemption 8 (i) like in the case of an extension after 2012:

Lead in solders in electrical glazing applications on glass, except for soldering in laminated glazing, in vehicles type approved before 1 January 2015, and lead in spare parts for these vehicles.

As mentioned before, a total of around three years should be enough to implement other lead-free solutions. The consultants recommend not setting a review date for the reasons explained above.

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Volkswagen 2	Stakeholder document "Volkswagen 2.pdf" sent to Otmar Deubzer via e-mail by Reinhard Hoock, BMW, as confidential document originally, declared as non-confidential on 26 January 2012 by Rakus Hagen, Volkswagen

Annex I

Uses and intended uses of the Antaya 65 % indium lead-free alloy in vehicles according to (Antaya 13)

Application	Antaya Part Number	Part Description	Installation Angle	Thickness (mm)	Light Transmission	OEM	Glass Company	Parts Shipped to Date	First shipped
Laminated Windshield	2252-IN31	Wire Assembly with solder clad terminal			70%	GM Minivan	PGW	550,000	12. Mai 00
Laminated Windshield	2355-CUP	Wire Assembly with solder clad terminal			70%	GM Minivan	PGW	142 375	13 Jul 00
Taminata d Windshiald	2240 1/205	Wine A second la suith a state state description			709/	Faul Course	DOW	141.250	20 1-1 00
Lammated windshield	2249-101203	whe Assembly with solder clad terminal			/076	r ora Coupe	row	141,200	20. Jul 00
-			0.08						
Tempered Backlite	65-3417-XD15LF	Heat Grid Solder Clad Terminal	90-	3.90 ± 0.1	20%	GM H2T Truck	Vitro	320,926	07. Apr 08
-						C 1/4 07.77			
Tempered Backlite	65-3564-166BL	Heat Grid Braided Buss Bar	45.12°	4.0 ± 0.1	20%	Cadillac SUV	Vitro	356,092	01. Mai 09
Tempered Quarter Window	65-3561-ATCWL	Wire Assembly with solder clad terminals		4.0 ± 0.1	20%	Cadillac SUV	Vitro	178,046	01. Mai 09
Tempered Quarter Window	65-3563-ATCW	Wire Assembly with solder clad terminals		4.0 ± 0.1	20%	Cadillac SUV	Vitro	178,024	01. Mai 09
							Vitro		
Tempered Backlite	65-3564-166BL	Heat Grid Braided Buss Bar with wire and terminal	39.3°	3.5 ± 0.1	20%	Saab SUV	Vitro	5,000	10. Jul 10
Tempered Quarter Window	65-3561-ATCWL	Wire Assembly with solder clad terminals			20%	Saab SUV	Vitro	2,499	10. Jul 10
Tempered Quarter Window	65-3563-ATCW	Wire Assembly with solder clad terminals			20%	Saab SUV	Vitro	2,499	10. Jul 10
							Vitro		
Tempered Backlite	65-3567-CCGM	Heat Grid Braided Buss Bar with wire assembly	30°	40 ± 01	70%	Cadillac Sedan	Vitro	27 252	08 Sep 09
Tempered Backlite	65 3560 PBGM	Heat Grid Braided Buss Bar with wire assembly	309	4.0 ± 0.1	70%	Cadillac Sedan	Vitro	27,232	08 Sep 09
Tempered Backlite	65-3525-MSS1P	Solder Clad Button Span Terminal	300	4.0 ± 0.1 4.0 ± 0.1	70%	Cadillac Sedan	Vitro	54 474	08. Sep 09
Tempered Dackine	05-5525-1415511	Solder clad Button Shap Tenninar	39	4.0 ± 0.1	7070	Caunac Sedan	Vitro	24,474	00. Sep 05
Tempered Backlite	65-3559-ROBH	Heat Grid	39°	4.0 ± 0.1	70%	Cadillac Sedan	Vitro	7,100	24. Feb 09
Tempered Backlite	65-3560-LOBH	Heat Grid Solder Clad Terminal	39°	4.0 ± 0.1	70%	Cadillac Sedan	Vitro	10,040	24. Feb 09
Tempered Backlite	65-3525-MSS1P	Heat Grid Solder Clad Terminal	39°	4.0 ± 0.1	70%	Cadillac Sedan	Vitro	20,080	24. Feb 09
Tempered Backlite	65-3523-EFPB	Heat Grid Braided Buss Bar with wire assembly	40°	3.2	20% and 70%	Chevrolet Volt	Pilkington	36,720	16. Apr 09
Tempered Backlite	65-3513-WJC13	Heat Grid Solder Clad Terminal	40°	3.2	20% and 70%	Chevrolet Volt	Pilkington	36,720	16. Apr 09
Laminated Windshield	65-3525-MSS1P	Solder Clad Button Snap Terminal	40°	3.2	20% and 70%	Chevrolet Volt	Pilkington	18,360	16. Apr 09
Towners d Pastilita	65 2522 EEDD	Uset Grid Preided Pues Per with wire accombing	40°	2.2	2004 and 7004	Onal Amnara	Dillington	5 000	16 Apr 00
Tempered Backlite	65-3513-WIC13	Heat Grid Solder Clad Terminal	40°	3.2	20% and 70%	Opel Ampera	Pilkington	4 998	16 Apr 09
Laminated Windshield	65-3525-MSS1P	Solder Clad Button Snap Terminal	40°	3.2	20% and 70%	Opel Ampera	Pilkington	2,499	16. Apr 09
Tempered Backlite	65-3555-GLMR	Heat Grid Solder Clad Terminal with wire and connector	28.75°	3.15 ± 0.1	35% and 76%	VW Jetta	Vitro	701,113	08. Sep 09
Tempered Backlite	65-3556-5463	Antenna Wire assembly with solder clad terminals	28.75°	3.15 ± 0.1	35% and 76%	VW Jetta	Vitro	350,557	08. Sep 09
Tempered Backlite	65-3557-5462	Antenna Wire assembly with solder clad terminals	28.75°	3.15 ± 0.1	35% and 76%	VW Jetta	Vitro	350,557	08. Sep 09
Tempered Backlite	65-3581-J90	Antenna Wire assembly with solder clad terminals	28.75°	3.15 ± 0.1	35% and 76%	VW Jetta	Vitro	20,000	08. Sep 09
-	(C. 0.000 000 000		24.000				Vitro		10.02.10
Tempered Backlite	65-3555-GLMR	Heat Grid Solder Clad Terminal with wire and connector	21.89*	3.15 ± 0.1	76%	VW Bettle	Vitro	30,000	12. Nov 10
Tempered Backlite	65 3761 2	Antenna Wire assembly with solder clad terminals	21.89 21.80°	3.13 ± 0.1 3.15 ± 0.1	76%	VW Bettle	Vitro	15,000	12. Nov 10
Tempereu Dacknie	05-5701-2	rancinia wite asseniory with solder clau terminials	21.07	5.15 ± 0.1	1070	, w Dettie	VILO	15,000	12.100 10
Tempered Backlite	65-3512-WJA	Heat Grid Solder Clad Terminal		3.5 ± 0.1	70%	Chevrolet Sonic	Vitro	15.333	24. Aug 10
							-	Parts	
								3,624,751	

Table 19: Vehicle and connector types with Antaya lead-free solder shipped worldwide according to (Antaya 13)

Upcoming Programs								Start of Production	
Tempered Backlite	65-3794-PM7	Heat Grid Solder Clad Terminal with wire and connector		3.20 ± 0.2	20%	GM Colorado/CanyonTruck	Vitro	March 1, 2012	
Tempered Backlite	65-3512-WJA	Heat Grid Buss Bar with terminal				GM AVEO	Vitro	March 1, 2012	
Laminated Windshield	65-39/6-HWALF	Heated Wiper Area				Damler	PGW	April 1, 2012	
Tempered backlight	65-3512-WJA	Heat Grid Buss Bar with terminal				Holden	Fuyao	February 1, 2012	
Tempered Backlite	65-3513-WJC-13	Heat Grid Buss Bar with terminal				Nissan X11M	Vitro	October 1, 2012	
Tempered Backlite	65-4004-JO2C	Heat Grid Buss Bar with terminal				Nissan J02C	Vitro	February 1, 2013	
Tempered Backlite	65-3815-GM351-R	Heat Grid Buss Bar with terminal				GM Car	PGW	T 1 2012	
Tempered Backlite	65-3816-GM351-L	Heat Grid Buss Bar with terminal				GM Car	PGW	January 1, 2012	
Tempered Backlite	65-3895-GM352	Heat Grid Buss Bar with terminal				GM Car	Pilkington	Tanuary 1, 2013	
Tempered Backlite	65-3525-MSS1P	Solder Clad Button Snap Terminal				GM Car	Pilkington	January 1, 2015	
Tempered Pacifite	65 3805 P1CP	Heat Grid Praided Puss Par with wire assembly				GM Car	Guardian		
Tempered Backlite	65 2806 P1PP	Heat Grid Braided Buss Bar with wire assembly				GM Car	Guardian	March 1 2012	
Tempered Backlite	65 2525 M881P	Solder Clad Putton Span Terminal				GM Car	Guardian	Water 1, 2012	
Tempered Backine	05-5525-1415511	Solder Clau Button Shap Tenninar				divi cai	Guardian		
Tempered Backlite	65-3954-K2PS	Heat Grid Buss Bar with wire assembly				GM Truck	Pilkington		
Tempered Backlite	65-3955-K2GS	Heat Grid Buss Bar with wire assembly				GM Truck	Pilkington	April 1, 2013	
Tempered Backlite	65-3945-K2PB	Heat Grid Buss Bar with wire assembly				GM Truck	Dura		
Tempered Backlite	65-3946-K2GB	Heat Grid Buss Bar with wire assembly				GM Truck	Dura		
Tempered Backlite	65-3913-K2BT	Heat Grid Solder Clad Terminal				GM Truck	Dura	March 1, 2013	
Tempered Backlite	65-3914-K2CT	Heat Grid Solder Clad Terminal				GM Truck	Dura		
Tempered Backlite	65-3938-K2WJ	Wire Assembly with Solder Clad Terminals				GM Truck	Dura		
Town out 4 Doubling	65 2054 VODC	Hart Crid Dree Derrich min anorthe				CM Trush	Differente en		
Tempered Backite	65 2055 V200	Heat Ond Buss Bar with wire assembly				GM Truck	Pilkington	March 1, 2013	
I empered Backlite	03-3933-K2G8	Heat Orid Buss Bar with wire assembly				GM Truck	Piikington		
Tempered Backlite	65-3945-K2PB	Heat Grid Buss Bar with wire assembly				GM Truck	Vitro		
Tempered Backlite	65-3946-K2GB	Heat Grid Buss Bar with wire assembly Heat Grid Buss Bar with wire assembly				GM Truck	Vitro	March 1, 2013	
Tempered Backlite	65-3948-Y1BC-L	Heat Grid Buss Bar with terminal				Chevrolet Coupe	AGC	Terre 1, 2012	
Tempered Backlite	65-3949-Y1BC-S	Heat Grid Buss Bar with terminal				Chevrolet Coupe	AGC	June 1, 2013	
Town of Doubles	(5.2052 A 100	Hast Cald Dave Descriptions	21.20	2.5		C- 49 C- 4	11. m		
Tempered Backlite	65-3952-AIPB	Heat Grid Buss Bar with terminal	21.2*	3.0		Cadillac Sedan	Vitro	July 1, 2012	
Tempered Backlite	65-3525-MSS1P	Solder Clad Button Snan Terminal	21.2*	3.5		Cadillac Sedan	Vitro		
Tempered Backate	05-5525-1415511	Solder eilde Buttorr Shap Teinmin	21.2	5.5		Caunae Sedan	vide		
Tempered Backlite	65-3952-A1PB	Heat Grid Buss Bar with terminal				Cadillac Sedan	AGC		
Tempered Backlite	65-3953-A1GB	Heat Grid Buss Bar with terminal				Cadillac Sedan	AGC	June 1, 2013	
Tempered Backlite	65-3525-MSS1P	Solder Clad Button Snap Terminal				Cadillac Sedan	AGC		
T (D (C)	(5.005) E10EC	T COMP D Stars 11				at 1 : 03 (0)			
1 empered Backlite	02-3926-E18PG	Heat Ond Buss Bar with wire assembly				Shanghai GM Sedan	Yaopi	0	
1 empered Backlite	02-3957-E18PB	Heat Ond Buss Bar with wire assembly				Shanghai GM Sedan	Таорі	September 1, 2012	
1 empered Backlite	03-3525-M881P	Solder Clad Button Snap Terminal				Shanghai GM Sedan	1 aopi		
Tempered Backlite	65-3950-GAM	Wire Assembly with Solder Clad Terminal				Shanghai GM SUV	Vaoni	September 1, 2012	
a competent Daciante	00.000000000000	macrossenory with bolder older reminist				onangina on oo ,	1.0001	September 1, 2012	

	Table 20: Vehicle and connector types of	f upcoming program	nes using Antaya lead-free	 solder according to (Antaya 13)
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According to (Antaya 12), "upcoming programs" refers to vehicle programs that have already been identified for production and are in various stages of release for production. Each OEM has specific requirements for testing and builds that occur before the start of production. Included in the above table are all vehicles where Antaya has been sourced as the supplier for the program and lead free solder has been identified and approved for use. (Antaya 12) confirms that all applications listed in

Table 19 and Table 20 above use the Antaya alloy with 65 % of indium.

Annex II

Revised draft test specification for assessing potential substitutes according to (ACEA 3)

Section I component specific tests

1. Temperature cycling test

According to DIN EN ISO 16750-4-H section 5.3.1.2

Before test: Temperature of climate chamber -40°C to 105°C Humidity not controlled (dry) Min. 60 cycles Electrical current loading with 14V (+/- 0.2) starting at end of low temperature phase Temperature-time following current loading eg. acc. VW 80101

After test: Pull test (perpendicular) 10N for antenna, theft warning system 50N for heater connectors (14V)



Figure 19 – Temperature profile

Table 21 - Temperatures and durations of a temperature cycle

Time	Temperatures
(min)	(°C)
0	20
60	T _{ve}
150	Tue
210	20
300	Tat
420	Tai
480	20

 T_{uB} : - 40°C

T_{oB} : +105°C

Heat soak test according DIN EN ISO 16750-4-K, section 5.1.2.2

Before test:

Temperature of climate chamber	105°C
Test time at 105°C inside chamber:	96 hours

Electrical current loading with 14V (+/- 0.2) throughout the test for heater connectors, only

Mechanical load p	to soldering joints during heat storage: osition of screen horizontally, inside down fechanical load: vertical down, directed as acceleration of gravity
Pull forces:	2N for antenna connectors, theft warning system 6N for heater connectors (14V)
After test:	Electrical testing (see below, section III) Pull test (perpendicular) 10N for antenna, theft warning system 50N for heater connectors (14V) Electrical testing (tbd)

3. High temperature storage test

Considers recent investigated max. data for thick dark tinted glass and requirements for adhesives testing (Polyurethane for glass bonding).

Duration:	24 h
Temperature:	120°C
After test:	Electrical testing (see below, section III)
	Pull test (perpendicular) 10N for antenna, theft warning system
	50N for heater connectors (14V)

4. Long term test with electrical load

(purpose of test: Dissolution of silver print and solder)

Temperature of climate chamber: 105°C Test time at 105°C inside chamber: 500 hours Electrical current loading with 14V (± 0.2) throughout the test

After test:

Pull test (perpendicular) 10N for antenna, theft warning system 50N for heater connectors (14V)

5. Heat shock test following DIN EN ISO 16750-4-H

Splash water test following section 5	.4.2
Temperature of climate chamber:	105°C, 1 hour
Temperature of splash water:	23°C ± 5°C or lower (from refrigerator)
Number of cycles:	10, checks after 5, 10
Dry samples after every cycle.	
Can be done on lab samples (difficul	t with backlights).

After test: Pull test (perpendicular) 10N for antenna, theft warning system 50N for heater connectors (14V)

6. High Humidity test: constant climate

Storage at:	80°C reflects bus bar temperature under load, >96 %RH (condensing),
Duration:	500 hours
electrical load:	14 V 15 min. every 24h, first cycle after 10 hours
After test:	Pull test (perpendicular) 10N for antenna, theft warning system 50N for heater connectors (14V)

If Silver separates from the glass, pull-tests and electrical tests cannot be performed. In this case, the solder contact is assessed as good.

7. Resistance to screen washer fluids:

Test fluid: Immersion in or continuous wetting with glass washing liquid consisting of:

69,5 Vol% water 20 Vol% ethanol 10 Vol% Isopropoanol 0.09% weight sodium lauryl sulphate 0.5 Vol% ethylene glycol

Duration of immersion or wetting: 24 hours at 23C pre-conditioning: 24 hours at 23C

After test: Pull test (perpendicular) 10N for antenna, theft warning system 50N for heater connectors (14V)

8. Salt spray test according to DIN EN ISO 9227

Salt spray test (neutral): duration 96 hours After test: Pull test (perpendicular) 10N for antenna, theft warning system 50N for heater connectors (14V)

Remark: revision of pull off forces before after / ageing in discussion / harmonization.

Section II OEM Company specific vehicle tests

Target: test behavior of screen and connectors in cars

1. Lab tests

Company specific hydropulse test equipment applying dynamic mechanical load to car body at low and high temperatures

- On the road tests Screens are tested in regular development car test program
 - a. Summer tests program
 - b. Winter tests program

Annex III

2009 Joint Test Program (ACEA, CLEPA, Antaya et al.)

2009 Joint Test Program

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2 Background and Approach

This test program is designed to determine whether Antaya's proposed lead-free indiumbased solders are an industrially feasible substitute for lead-containing solders used on glass in vehicles. This requires a test program compliant to typical European requirements and procedures which can be applied to all proposed lead-free solutions.

The OEMs are responsible for the safety and reliability of their products. They hence decide about the reliability and safety tests to be conducted before they accept Antaya's proposed lead-free indium-based solders in their vehicles.

Each test consists of three parts:

- 1. ageing procedure
- 2. the specific parameters describing how the ageing procedure is conducted
- 3. testing and acceptance criteria

Although test standards are available, there is no standard test program. In principle, each OEM applies its ageing procedure with its own parameters as well as its specific testing and acceptance criteria. The OEM-specific test programs are based on years of in-field experiences and testing, and these experiences are specific for each OEM.

Nevertheless, there is a group of ageing procedures, which all or almost all OEMs apply, although with different parameters and testing/acceptance criteria. Other tests are only applied by a few OEMs.

It was therefore necessary to select

- the ageing procedures to be conducted
- the parameters describing how these procedures shall be conducted
- the testing and acceptance criteria for each test

The frame conditions for the selection were the following

- The ban of lead in solders for soldering on glass would affect the use of solders in all applications on glass in all vehicles under the scope of the ELV Directive. The test program must therefore be broad enough to allow assessing the industrial feasibility of Antaya's lead-free solders as a general substitute for lead-containing solders in all these applications.
- 2. The test program must be as slim and simple as possible. It cannot take into account all tests of all OEMs, as such a test program would neither be accomplishable within a reasonable time frame nor could it be paid for.
- 3. The test program must ensure a fair assessment of the Antaya solders. It must follow the usual testing practice unless sound technical arguments justify a deviation from these usual testing practices.

The selection of the ageing procedures therefore is conducted following these principles:

 The ageing procedures, which all or most OEMs conduct, must be assumed to be of central importance to assess the reliability of solder applications on glass. These ageing procedures must hence become part of the test program (chapter 3 on page 95 ff).

The OEMs discussed the parameters for these ageing procedures, and an agreement could already be achieved. The parameters are a compromise between the parameters of different OEMs.

- 2. After the ageing procedures, the samples must be tested. It was necessary to agree on the testing and acceptance criteria (chapter 5 on page 99 ff).
- 3. Testing is material and application specific. A test shall allow conclusions whether a specific material can suffice the requirements in a specific application over a specified period of time. If one material is substituted by another one with different material properties, these differences in material properties may require an adaptation of the test programs. Some tests therefore may become part of the test program depending on the material parameters of the lead-free solders compared to the lead-containing ones (chapter 6 on page 101)

4. Several tests are only conducted by a single or few OEMs. These tests did not become part of the test program, as a test program as summary of all tests of all OEMs for all applications would neither be manageable nor could it be paid for. The OEMs agreed to drop these tests to facilitate a viable test program (chapter 7 on page 105).

3 Laboratory test program

3.1 Temperature cycle test ISO 16750-4:2003

- -40C to +90 C
- 30 cycles
- Temperature change rate: 1K/min
- Dwelling time: 90 min

Background and objective of this ageing procedure:

The difference of the coefficients of thermal expansion between glass and metal leads to mechanical stress during thermal cycling. When the car is operated at very low temperatures (e.g. -40°C – Scandinavia, Russia) the metal connector applies a huge tensile stress to the glass. This stress is reduced by the solder alloy, a ductile material like Pb. This test ensures that the stress relaxation mechanism of the solder alloy is sufficient. About 20-30 cycles are required to simulate the ageing (e.g. workhardening) of the alloy.

Test and acceptance criteria

Specified in section 5 on page 97.

3.2 High temperature storage test according to UNECE Global

- 90C
- 500 h

Background and objective of this ageing procedure:

This procedure describes typical accelerated ageing. The micro-structure of the solder alloy after the soldering process is not necessarily in an equilibrium state, because the metallic micro-structure is frozen at (or slightly below) the melting point of the alloy. During the life-time of the car, this micro-structure relaxes towards its equilibrium state, which might for example be harder than the state right after soldering. This ageing procedure simulates the microstructural changes in the solder joint over the life-time of the car on an accelerated time scale to enable the testing of an aged sample. Typically, this procedure is upstream to tests, which test the short-time functionality of the product (e.g. pull-off test, temperature cycling, etc.)

Test and acceptance criteria:

Specified in section 5 on page 97.

3.3 Climatic temperature with humidity tests according to DIN EN ISO 6270-2 under load

- 14 cycles of 24 h total
 - 8 hours, 40 C at 100% humidity (condensation)
 - 16 h at room temperature (18-28C +-3C) with hum. less than 100%
 - Change time included in 8 h and 16 h, temp. change within 1 h
 - Continuous change of 14 V electrical load: 1 h under load, 1 h without load

Background and objective of this ageing procedure:

Water is a more aggressive chemical than usually thought. If the water layer grows to a thickness of about 50 molecules, the corrosion reactions start to prevail. The thermal cycling of this test leads to a thick layer of water on all structures. The material absorbs the water and can cause a swelling. Besides the mechanical degeneration, the dominant corrosion mechanism is electrolysis, which is amplified by the electrical load. Electro-corrosion and (with the load) electro-migration are the results, which might cause functional cut-offs or even mechanical break-down of the joint. Condensation is a typical phenomenon in the cabin of a car. This procedure ensures that the product functionalities persist under condensation during the life-time of the car.

Test and acceptance criteria:

Specified in section 5 on page 97.

3.4 Constant climatic humidity tests

ECE-TRANS-WP.29-GRSG-2007-28e, now replaced by ECE/TRANS/180/Add.6/ and ECE/TRANS/180/Add.6/Appendix 1

- 50°C at 100% (-5%) rel. humidity WITHOUT condensation
- duration 336h

Background and objective of this ageing procedure:

Even more aggressive than water is water vapour. It can easily creep into the interfaces of the solder joint (without being limited by wetting mechanisms) and thus penetrate the structure of the joint. Some alloys are vulnerable to this type of exposure. The alloy may become brittle or the adhesion at the interfaces might fail. The result is a cohesive or adhesive failure of the joint. This procedure ensures that the product functionalities persist during the life-time of the car.

Test and acceptance criteria:

Specified in section 5 on page 97

3.5 In-field testing and vibration test

3.5.1 In-field testing

The OEMs (vehicle manufacturers), according to their general qualification procedures for new materials and parts, would not accept the lead-free indium solders to be generally used in their vehicles without a previous in-field testing phase.

In-field testing means that vehicle manufacturers, after the solders have successfully passed the lab test program, would apply the indium-containing solders in a smaller number of their vehicles (around 500 to 1,000 units). The manufacturers would closely monitor the respective vehicles. After at least one year, if no failures occur,

they would then give green light for the use of the solder at least in the car models using the glass for which the solders had been qualified during lab testing. The final approval will be given after a total of 3 years assuming no problems have come up until then.

They agreed to participate in the testing program and to start in-field testing as soon as it had been proved that the indium-containing solders can successfully pass the obligatory laboratory test program.

3.5.2 Vibration test according to ISO 16750 (Shaker test)

Most OEMs apply such a test. It is not specific for the solder joint or for glasses. It is either a component test or applied to the entire vehicle.

- > 8 h at room temperature
- 10 to 1000 Hertz

Background and objectives of this ageing procedure:

Not specified

Test and acceptance criteria:

Not specified

4 OEM-specific thermal shock test as part of the general test program?

One OEM insists on the thermal shock test as part of the general test program. The other OEMs conducting this test agreed to only integrate this test into the general test program, if deviating material parameters and material properties of the indium-based lead-free solders justify the inclusion of this test (see chapter 6 on page 101 ff).

As the OEMs decide on the test program, and in order to achieve a broad acceptance of the test program, this thermal shock test shall be integrated into the testing program. The fact that only one OEM insists on this test regardless of any material property differences as a justification for inclusion (see chapter 6 on page 101 ff) could be taken into account properly and fair in the selection of samples for testing. The samples on which this test should be performed could be selected specifically for this OEM. It remains, however, to be discussed depending on the outcomes of the general test program whether and how far a possible failure of the indium-based lead-free solders can prove that they are no appropriate substitutes for the lead-containing solders.

For details of the thermal shock test, see chapter 6.1 on page 102.

5 Acceptance criteria for laboratory tests

The following test and acceptance criteria apply to the above ageing procedures. The criteria are based on the CLEPA proposal taking into account OEMs' comments. The objective is not to show whether lead-containing or lead-free solder joints perform better. The tests are accepted as passed if the results match with the OEMs' requirements.

5.1 Testing Order

- 1. Inspection for micro cracks
 - 1.1. All soldered joints are visually inspected to detect micro cracks with the method described below.
 - 1.2. The test has to be performed prior to all other tests.
 - 1.3. If a micro crack is detected, the test is not passed.
- 2. Infra-red inspection
 - 2.1. All soldered joint are inspected by IR-camera during operation. If a hot spot is detected indicating a defect, the test is not passed.
- 3. Resistance measurement
 - 3.1. The resistance of at least 2 joints per glass panel is measured and logged with the method described below.
 - 3.2. The resistance values attributable to the specific connectors in order to allow a comparison to the measurement after the environmental tests.
- 4. Pull-off test
 - 4.1. The pull-off test ensures good adhesion of all interfaces (glass to enamel to silver print to solder to connector).
 - 4.2. The test has to be performed as described below. A maximum pull force of 150 N is expected for an OK outcome. If a lower value is defined in the drawing of the glazing,

this value is valid instead. For acceptance, the specified pull force must be achieved without breakage.

- 4.3. The first and the last glass panel soldered of every model are tested at all soldering positions, reducing the net output by two samples.
- 4.4. If a single pull-off test fails of the first panel, the process parameters might be adjusted and the spare panel is used instead.
- 4.5. If a single pull-off test fails of the last panel, the test has failed for this model. No further testing is performed and it is not investigated if the capability of the processes or the feasibility of the product is responsible.

5.2 Testing and Acceptance Criteria

- 1. Inspection for micro cracks
 - 1.1. The glass side oppositely to the silver printed side is inspected for micro cracks.
 - 1.2. This glass surface is cleaned with a detergent leaving no impurities.
 - 1.3. A bright ring lamp or a bright spot light is used to illuminate the soldered area.
 - 1.4. The area below the soldered connector is searched for a micro crack. A micro crack is visible due to its light scattering properties as a white/silver spot.
 - 1.5. An experience operator is required for this test, because these defects open up only to glass experts. If adverted to it, also non-experts with a good vision are able to see the defect.

Remarks:

- Applying a black covering of the solder joint reduces reflections and increases the exactness of the measurement; this method is applied in in case of doubt.
- The inspection for micro cracks is a test to show the capability of the soldering process
- 2. Infra-red inspection
 - 2.1. A voltage of 12V is applied to the connectors at their contact positions. The voltage has to be adapted if the connector is not at its designated position in the case that one panel is used to solder many connectors.
 - 2.2. Antennas or other structures with one pole only, are contacted on the connector and on the Ag-print directly. The voltage has to be adapted to the resistance of the antenna structure in order to reach typical temperatures of the printed wires.
 - 2.3. The glass is heated for 3 minutes.
 - 2.4. An IR-camera is used to document the temperature distribution of the soldering area from both sides of the glass panel during the heating operation.
 - 2.5. The soldering area is searched for hot spots which indicate a defect.
- 3. Resistance measurement
 - 3.1. The contact resistance is measured and logged with a 4-wire resistance meter with a measurement accuracy of 1mOhm.
 - 3.2. The first resistance probe is in contact with the connector at its designated contact position. Contact to the solder must be avoided.

- 3.3. The other resistance probe is in contact to the silver print as close as possible to the soldering area. Contact to the solder must be avoided. For every model this position is defined prior to the test.
- 3.4. The temperature of the glass has to be determined and logged with a contact thermometer and the resistance values have to be corrected to room-temperature values.
- 4. Pull-off test
 - 4.1. The pull-off test has to be performed 15min ±2min after the soldering of the connector or after ageing of the sample in case of accelerated ageing tests.
 - 4.2. The direction of pulling is vertical to the glass surface. If a different direction is defined in the drawing of the glazing, this direction is used instead.
 - 4.3. The glass must be fixed, but the clamping itself must not apply any force to the glass.
 - 4.4. A constant pull-off velocity of (100±10) mm/min has to be used. Otherwise the values are meaningless.

6 Tests to be Applied under Certain Conditions

A testing program like this here on the application of solders on glass shall allow drawing conclusions whether a material with its specific properties can suffice the specific requirements in a specific application. Tests thus are material and application specific. If a material is substituted by another one in a specific application, the test program may have to be adapted if the material properties of the substitute material deviates in important, application-relevant parameters.

The following ageing procedures are not conducted by all OEMs. They may nevertheless become part of the test program, if the differences in relevant material properties of the currently used tin-lead solders and Antaya's lead-free indium-based solders give reason to include them.

As the composition of Antaya's lead-free indium-based solders is not available, these tests must be decided to be integrated into the test program when the exact material composition – and thus the material properties – of the lead-free solders are known.

6.1 Thermal shock (ISO 16750/4), IEC 60068/2/14 under load

6.1.1 Setting

- 24h at -40C(+/-2)
- 24h at +90C(+/-2)
- Under load (applying electrical voltage according to drawing or: alternating load /unload 16V 5 minutes during change of chambers)
- Storage under recirculation air
- Temperature change rate: as fast as possible (within less than 1 minute)
- Cycles: 10 cycles

Background and objectives of this ageing procedure:

The ageing procedure shall test for solder breakage (cracks in solder joint) as a consequence of thermal mismatch arising from different coefficients of thermal expansion (CTE). The real life background is, e. g., that customers might wash their cars including the backlight and the wind screen with hot water in a cold winter.

Additionally, higher melting points of the lead-free solders might bring in increased tensions into the glass, e. g. a backlight, during the cooling-down process. The increased tensions might result in the damage of the glass during car operation, or chip fractures in case of mechanical burdens and accidents. Both effects would affect the reliability and safety of vehicles.

The number of 10 cycles is a compromise of different OEM's specifications ranging from 5 to 500 cycles.

Testing and acceptance criteria:

Pull force of 150N must be achieved without breakage (OEM1)

Conformity of the part concerning the electrical characteristic of power; no pulling test on the connector in the standard specification but OEM believes this kind of acceptance criteria to be necessary, as the lead-free solders are a new material (OEM2)
6.1.2 Material parameters for integration into general test program

Given the background of this procedure as described above, the coefficient of thermal expansion (CTE) of the soldering material is a crucial parameter. If the difference of the CTE of the lead-free, indium-based solders between the lead-free solder material and the glass to be soldered is bigger than between the lead-containing solders and the glass to be soldered, the thermal shock test must be included into the test program.

A more ductile solder possibly can compensate higher tensile stress. If the leadfree solders are less ductile than the lead-containing solders, it would be another reason to integrate the thermal shock test into the test program.

If the new solder material means a higher soldering temperature there could be a risk that there is more tension in the tempered glass in the area of the solder joint. This would in this case mean a risk for breakage of the glass or different shattering behavior of the glass in a crash situation. The thermal shock test can reveal such weaknesses resulting from these interactions between the solder joint, the soldering process and the glass.

A higher melting point of the lead-free solders thus would also motivate the inclusion of the thermal shock test into the test program.

Summing up, differences in the following parameters between the lead-containing and the lead-free solders would require the integration of the thermal shock test into the test program:

- Differences in the CTE
- Differences in ductility
- Higher melting points of the lead-free solders

The final decision will remain to the OEMs depending on the above parameters.

6.2 Salt spray test

6.2.1 Setting

Glass solder is used on tempered and laminated glass. The evaluation should be on both under the same conditions. The test does not refer to PVB.

Test method: JIS C 60068-2-11, IEC 68-2-11

Conditions:

- Concentration 5%
- Temperature 35C
- Hours 16, 24, 48, 96, 168, 336, 672

Background and objectives:

Simulation of the effect, which snow melting chemicals might have on open backdoor or open back window. Glass solder is used on tempered and laminated glass, so the evaluation should be on both under the same condition.

Testing and acceptance criteria:

Pull force of 150N must be achieved without breakage

6.2.2 Material parameters for integration into general test program

Salt water is more corrosive than water without salt. If the corrosiveness of the lead-free solders is higher compared to the lead-containing solders, the inclusion of the salt spray test into the test program might become necessary.

The final decision will be with the OEMs depending on the corrosiveness of the lead-free solders.

7 OEM-specific Laboratory Tests (Not part of the test program)

These tests are not part of the test program. They are OEM specific. To keep the test program manageable, the OEMs agreed to forego these tests.

7.1 Salt spray tests

7.1.1 Salt spray test version I (OEM2)

• 96 h of salt spray test made directly on the serigraphy and connector

Background and objectives of this ageing procedure:

Test and acceptance criteria:

- after this test, the pull test must be passed
- electrical characteristic change less than 5 %

7.1.2 Salt spray test version II (OEM4, OEM5; other OEMs have agreed to drop this test)

The salt spray test is part of a life test on parts with heating network and connector (OEM4). The test is only applied on electrical heated windows were the heating wires are printed on the surface which is in contact with the environmental climate (OEM5).

Salt spray test is made for 200h. The part is under power for the first 48h. This salt spray test is part of a all test, as follows:

- 140 heating cycles at -10 °C -2/0 °C,
- 200 heating cycles at 23 °C ± 5 °C,
- exposure to salt spray in accordance with Test Method D171058.
 - Expose the parts inside the passenger compartment for 200 hours and the parts outside the passenger compartment for 400 hours.

- Unless otherwise specified on the functional drawing, the heating network shall be supplied with 13 volts ± 0.1 volt for the first 48 hours. The connectors shall be installed.
- Glazing part shall be installed at 30° from vertical position. No drop of water shall run on the glass part.
- In the case of networks, a part of which is located outside of the passenger compartment, the test can be conducted protecting the part inside the passenger compartment for 200 hours with a box, then not protecting it for 200 hours.
- 140 heating cycles at 70 °c ± 2 °C,
- 200 heating cycles at 23 °C ± 5 °C,
- resistance to voltage surge tests in accordance with 5.2.5.
 Glazing is exposed to power of
 - > 18V ± 0,2 V for 60 min
 - > $24V \pm 0.2 V$ for 60 s.
- 140 heating cycles at -10 °C to 20°C
- 200 heating cycles at 23 °C ± 5 °C.

Unless otherwise specified on the functional drawing, a heating cycle consists of 8 minutes operation at 13 V \square 0.1 V across the terminals followed by 2 minutes stop

Background and objectives of this ageing procedure:

Testing and acceptance criteria:

At the outcome of these tests, the consumed electrical power, shall not have varied by more than 5 %.

Connectors shall be still present on glass, and resist to a folding test:

- Folding at 180°
- Soldering to be solicited

7.2 Chemical tests

For these tests it will be difficult to integrate them into the test program. Although it is acknowledged that these tests are important for OEMs, the objective of the tests is to exclude visual changes. Such changes have a mere esthetical background. A visual change would not indicate a change in the solder joint to a degree that might endanger its proper technical function. As the subject at stake is the substitution of a material – lead – due to its toxic properties, pure esthetical reasons would hardly justify recommending the continuation of exemption 8b. Additionally, not all OEMs do these chemical tests, which further on

weakens the importance for the exemption review process. Finally, OEMs could take secondary esthetical measures in case visual changes actually would occur on lead-free indium solder joints.

7.2.1 Water-Bath storage (OEM3)

The test specimens are to be stored in surface-active water (rainwater), such that the test parts are entirely submerged in the water.

Storage period: 28 days

Water temperature: 23 \pm 5 °C

Background and objectives of this test:

Not specified

Test and acceptance criteria:

Compare with non-exposed specimen, no visual differences allowed.

7.2.2 Storage in glass-cleaning fluid (OEM3)

Glass cleaner test fluid:

- o 20 vol.% ethanol (denatured); highest grade
- o 10 vol.% I-propanol; for synthesis in demineralised water
- o 0.09 wt. % sodium lauryl sulphate; highest grade
- o 0.5 wt. % ethylene glycol; for synthesis in demineralised water

The specimen are to be stored in the glass cleaner test fluid and then reverseconditioned.

Storage period:	24 h
Storage temperature:	$23\pm2~^{\circ}C$
Drying period:	24 h
Drying temperature	$23 \pm 2 \ ^{\circ}C$

Background and objectives of this ageing procedure:

Test and acceptance criteria:

Following reverse-conditioning, a comparison is to be performed with a nonexposed limiting sample (visual inspection), no visual differences allowed.

7.2.3 Storage in conservation media (OEM3)

Conservation test fluid:

- 15 wt. % paraffin; dropping point 55...60 °C, DAB 8; highest grade in depreservation agent test fluid
- 5 wt.% calcium stearate (soap)
 - highest grade in depreservation agent test fluid

The mixture is heated over a water bath at 50 °C for 30 min to dissolve the paraffin and then cooled down again under running cold water.

The mixture is to be stirred before use.

Deconservation test fluid:

- 25 vol.% isoctane, for synthesis
- 10 vol.% decalin (cis/trans); for synthesis
- 25 vol.% n-heptane; highest grade
- o 10 vol.% 1,3,5, trimethylbenzene; for synthesis
- 20 vol.% cyclohexane; highest grade
- 10 vol.% xylene (mixture); highest grade

Ageing procedure

- $\circ~$ Immersion of the specimen in conservation test fluid for 10 min. at 23 \pm 2 °C;
- Then allowed to dry for 15 min. at 23 ± 2 °C;
- $\circ~$ 2h storage at 50 \pm 5 °C in heating cabinet (air recirculation with fresh air supply).
- \circ 15 min acclimatisation at 23 ± 2 °C.
- \circ 30 min storage in deconservation test fluid at 23 ± 2 °C
- \circ Rinsing with water jet (50 ± 5 °C)
- \circ 1h (minimum) acclimatisation at 23 ± 2 °C

Background and objectives of this ageing procedure:

Test and acceptance criteria:

After end of test, a comparison is to be performed with a non-exposed limiting sample (visual inspection), no visual differences allowed.

7.2.4 Alkaline Test (OEM1)

o 2 hours soak in 0.1N Na-OH (JISK8576, ISO6353-2)

Background and objectives of ageing procedure Simulation of conditions at car wash

Test and acceptance criteria

Pull force of 150N must be achieved without breakage

Annex IV

Antaya B6 alloy results for the ACEA draft tests listed in Annex II as submitted by Antaya

ACEA Draft Test Specification (Annex II)	
Temperature cycling test	Pass
According to DIN EN ISO 16750-4-H section 5.3.1.2	Trialon report
Before test: Temperature of climate chamber -40°C to 105°C	27833 A
Humidity not controlled (dry)	
Min. 60 cycles	
Electrical current loading with 14V (+/- 0.2) starting at end of low temperature phase	
Temperature-time following current loading eg. acc. VW 80101	
After test: Pull test (perpendicular) 10N for antenna, theft warning system	
50N for heater connectors (14V)	
Heat soak test according DIN EN ISO 16750-4-K,	Pass
section 5.1.2.2	Exova Report
Before test:	9058-2 and
Temperature of climate chamber 105°C	9058-3
Test time at 105°C inside chamber: 96 hours	
Electrical current loading with 14V (+/- 0.2) throughout the test for heater connectors,	
only	
Mechanical load to soldering joints during heat storage:	
position of screen horizontally, inside down	
Mechanical load: vertical down, directed as acceleration of gravity	
Pull forces: 2N for antenna connectors, theft warning	
system	
6N for heater connectors (14V)	
After test: Electrical testing (see below, section III)	
Pull test (perpendicular) 10N for antenna, theft warning system	
50N for heater connectors (14V)	
Electrical testing (tbd)	

High temperature storage test		Pass
Considers recent investigated max. data for thick dark tinted glass and requirements		Trialon test report
for adhesives testing (Polyurethane for glass bonding).		27833 A
Duration: 24 h		
Temperature: 120°C		
After test: Electrical testing (see below, section III)		
Pull test (perpendicular) 10N for antenna, theft warning system		
50N for heater connectors (14V)		
Long term test with electrical load		Pass
(purpose of test: Dissolution of silver print and solder)		Trialon report
Temperature of climate chamber: 105°C		27833 A
Test time at 105°C inside chamber: 500 hours		
Electrical current loading with 14V (\pm 0.2) throughout the test		
After test:		
Pull test (perpendicular) 10N for antenna, theft warning system		
50N for heater connectors (14V)		
Heat shock test following DIN EN ISO 16750-4-H		Pass
Splash water test following section 5.4.2		Exova report
Temperature of climate chamber: 105°C, 1 hour		8819-2
Temperature of splash water: $23^{\circ}C \pm 5^{\circ}C$ or lower (from refrigerator)		
Number of cycles: 10, checks after 5, 10		
Dry samples after every cycle.		
Can be done on lab samples (difficult with backlights).		
After test: Pull test (perpendicular) 10N for antenna,		
theft warning system		
50N for heater connectors (14V)		
High Humidity test: constant climate	Trialon report 27523	Passed surrogate
Storage at: 80°C reflects bus bar temperature under	50 degrees C	
load, >96 %RH (condensing),	100% RH	
reflects the experience to discern "good" and "bad"		

solders	336 Hours	
Duration: 500 hours		
electrical load: 14 V 15 min. every 24h, first cycle after		
10 hours		
After test: Pull test (perpendicular) 10N for antenna, theft warning system		
50N for heater connectors (14V)		
If Silver separates from the glass, pull-tests and electrical tests cannot be performed. In this case, the solder contact is assessed as good.		
Resistance to screen washer fluids:		Passed
Test fluid: Immersion in or continuous wetting with		Trialon report
glass washing liquid consisting of:		28152 A
69,5 Vol% water		
20 Vol% ethanol		
10 Vol% Isopropanol		
0.09% weight sodium lauryl sulphate		
0.5 Vol% ethylene glycol		
Duration of immersion or wetting: 24 hours at 23C		
pre-conditioning: 24 hours at 23C		
After test: Pull test (perpendicular) 10N for antenna,		
theft warning system		
50N for heater connectors (14V)		
Salt spray test according to DIN EN ISO 9227		Passed
Salt spray test (neutral): duration 96 hours		Trialon report
After test: Pull test (perpendicular) 10N for antenna,		28152 A
theft warning system		
50N for heater connectors (14V)		

The Trialon test reports are available in a separate folder named "B6 Alloy Tests".