



GLASS BEADS (AND GLASS PARTICLES) – FOR USE IN AND WITH PAVEMENT MARKING MATERIALS

1 SCOPE

- a) This specification applies to glass beads and glass particles:
 - i. Added as a component in the production of pavement marking materials, and
 - ii. Combined with pavement marking materials when applied to surfaces, or
 - iii. Both.
- b) This document is prepared in a manner compliant with the requirements of AS/NZS ISO/IEC 17065.
- c) APAS certification to this specification shall only be claimed by APAS participating manufacturers (and suppliers), listed in APAS document AP-D152, that demonstrate conformance to the requirements of APAS document AP-D192 and those documented in this specification, when assessed by the APAS.
- d) APAS® is a trademark registered with IP Australia, owned by CSIRO, the Scheme Owner, and protected under applicable laws. Use of the trademark or the Certification Scheme is prohibited unless prior approval in writing is obtained from CSIRO via the APAS Secretariat.

2 BACKGROUND

- a) To obtain a broad overview of the Australian Paint Approval Scheme (APAS), refer to APAS document AP-D001.
- b) To obtain an overview of restricted ingredients in APAS certified products, refer to APAS document AP-D123.
- c) To obtain the current list of APAS participating manufacturers (and suppliers) and resellers, refer to APAS document AP-D152.
- d) To obtain an overview of how to participate in the APAS, refer to APAS document AP-D177.
- e) APAS approval to this specification may be gained by compliance with the requirements detailed in this specification and those in APAS document AP-D192.

3 DEFINITIONS AND ACRONYMS

3.1 Definitions

The definition of terms used in this document and in the Certification Scheme can be found in APAS Document AP-D001. In addition, the following definitions shall apply:

- a) **Agency for Conformity Assessment:** An organisation or testing authority recognised by APAS that is either part of the Clients Recognised Manufacturing Unit (RMU) and perform all of the required tests, or a specialist laboratory contracted either by the APAS Secretariat or by the Client, to carry out specific tests that are beyond the capability of the Client RMU.
- b) **APAS Signatory:** A person(s) authorised by APAS to exercise local control over the production, testing and application for certification. The role of the Signatory is to ensure compliance with APAS rules.
- c) **Campaign Run:** A continuous production run of one type of glass bead or glass particle.
- d) **Certification Scheme:** The Certification system related to specified products (Paint, Surface Coating Material and Non-Paint Products) to which the same specified requirements, specific rules and procedures apply. APAS is the applicable Certification Scheme.
- e) **Certification Requirement:** The specified requirement(s), including product requirement(s), that is fulfilled by the Client as a condition of establishing or maintaining certification.
- f) **Client:** The organisation responsible to the Certification Body (APAS) for ensuring that certification requirements, including product requirements, are fulfilled. The Client nominates a person(s) directly responsible (APAS Signatory) within its organisation, and to communicate directly with the Certification Scheme (APAS).
- g) **Glass:** Inorganic amorphous solid shaped out of a melt of silica and one or more basic oxides.
- h) **Glass Beads:** Transparent, clear, colourless, smooth and spherical glass balls used to provide visibility at night, in conjunction with pavement marking materials, by retroreflecting a vehicle headlight beam back towards the driver.
- i) **Glass Particles:** Small pieces or fragments of glass, typically irregularly shaped, can be of virgin or recycled origin, used primarily for the provision of skid and slip resistance in place of traditional aggregate; can also be referred to as crushed glass, silica, silicon dioxide, fused quartz, sodium carbonate, pot ash or similar.
- j) **Heavy Metals:** Elements as nominated in clause 8, Table 1. For the purposes of this specification, **heavy metals** are arsenic, antimony, cadmium, hexavalent chromium (Cr6+), lead and mercury.
- k) **Lot:** A sub-sample of a Campaign Run, where the glass beads or glass particles are made from known soda lime glass cullet feed stock at essentially the same time, by essentially the same processes and essentially under the same system of control, is defined as:
 - i. 20 metric tonnes (maximum) for glass beads manufactured by Direct Melt process (furnacing virgin raw materials).
 - ii. 10 metric tonnes (maximum) for glass beads manufactured by Non-Direct processes (which use existing materials).
 - iii. Defined Lot size for glass particles is as per clause 3.1 k) i. and ii. where applicable.
- l) **Product Requirement:** The specified requirement(s) that relates directly to a product, specified in standards or in other normative documents (APAS Specifications) identified by the Certification Scheme (APAS).
- m) **Retroreflectivity:** A measure of the light visible to the driver compared to the light entering the pavement marking (refer to **NOTE**¹ for further information).
- n) **Scheme Owner:** The organisation responsible for developing and maintaining the certification scheme. CSIRO is the APAS Scheme Owner.
- o) **Secretariat:** The organisation that provides administrative support and other resources necessary

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to keep the Certification Scheme functioning. The Secretariat is vested in CSIRO.

NOTE¹: The popular definition of the night time visibility of a pavement marking is defined as its retroreflectivity – a measurement of the efficiency of the pavement marking to return light in the general direction from which it is generated. It is simply a measure of the light visible to the driver compared to the light entering the pavement marking. The majority of retro-reflected light is usually a function of the surface applied glass beads, which are a component part of the pavement marking.

3.2 Acronyms

The following acronyms appear in this document:

- ACE** Agency for Conformity Assessment
- APAS** Australian Paint Approval Scheme
- AS** Australian Standard
- AS/NZS** Australian Standard / New Zealand Standard
- CSIRO** Commonwealth Scientific and Industrial Research Organisation
- g** Grams
- HR** High Retroreflectivity
- IAF** International Accreditation Forum
- ICPMS** Inductively Coupled Plasma Mass Spectrometry
- IEC** International Electrotechnical Commission
- ILAC** International Laboratory Accreditation Cooperation
- ISO** International Organisation for Standardization
- JAS-ANZ** Joint Accreditation Society – Australia & New Zealand
- mg** Milligrams
- mm** Millimetres
- PDS** Product Data Sheet
- RAPMG** Road Authority Pavement Marking Group
- RI** Refractive Index
- RIAA** Roadmarking Industry Association of Australia
- SDS** Safety Data Sheet
- TDS** Technical Data Sheet
- NATA** National Association of Testing Authorities
- WHS** Workplace Health and Safety
- XRF** X-ray Fluorescence
- µm** Micrometres

4 DESCRIPTION AND GUIDE FOR USERS

4.1 General Requirements²

- a) Pavement marking materials are used by road authorities to:
 - i. Improve road safety and other surfaces used to carry all types of traffic including cars, trucks, pedestrians, cyclists, buses and aircraft.
 - ii. Provide delineation, acting as a guide to drivers to maintain their respective lane position, deterring accidents and providing direction.
- b) Pavement marking materials require the addition of glass beads to provide retroreflectivity performance under all conditions, compared to an un-beaded marking. Glass beads are typically available in both standard and High Retroreflectivity forms.

- c) Glass beads are utilised during both the manufacturing process, such as in Cold Applied Plastics and Thermoplastics, as well as being added, when required, at the same time as surface coating materials to a road surface.
- d) Glass beads are normally applied directly onto the pavement marking material during application and/or immediately after application of a pavement marking material by one of two methods:
 - i. Hand-strewn
 - ii. Automatic drop-on mechanism
- e) Glass particles are typically included in the manufacturing process as a substitute for an aggregate material to aid in the materials slip and skid resistance and are not involved in retroreflectivity performance.
- f) The retroreflectivity performance for a given type of glass bead can also vary depending on the source of the glass used for manufacture, method of manufacture, and quality controls applied by the manufacturer.
- g) The chemical composition of glass beads and glass particles can vary greatly depending on the source of the glass used for manufacture. Glass beads are typically comprised of, but not limited to, soda lime cullet. Glass particles can be comprised soda lime cullet but can also be comprised of recycled glass materials.
- h) For the purposes of this specification, the following types of glass beads and glass particles are recognised:
 - i. **Type A:** Also known as premix beads, typically range in size from 200-425µm, predominantly added into pavement marking materials during the manufacturing process. **This glass bead type is not being considered for accreditation due to low market demand.**
 - ii. **Type B:** Also known as drop-on beads, typically range in size from 300-850µm, predominantly used in surface application to a wet product. This type typically contains a moisture-proof coating² that is added to the beads to assist it avoiding the absorption of air humidity, thus assisting bead flow and anti-caking properties.
 - iii. **Type C:** Also known as inter-mix beads, typically range in size from 425-1000µm, predominantly used in both pavement marking materials during manufacture (Cold Applied Plastics and Thermoplastics) and in surface application to a wet product. This type **do not** typically contain a moisture-proof coating.
 - iv. **Type D:** Also known as larger, wet-weather beads, typically range in size from 850-1700µm, predominantly used in both pavement marking materials during manufacture and in surface application to a wet product. This type **do not** typically contain a moisture-proof coating.
 - v. **Type E:** Typically, a similar sizing to Type D beads (but can be produced in any size) but with a higher RI and can have less compression strength. An example of such a bead is a Type III, barium titanate glass bead with a refractive index greater >1.9 that complies with US FAA Specification TTB 1325D used in airport runway markings (refer to APAS specification AP-

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S0041/6 Pavement Marking Material – Airport Runway Markings for further information).

- vi. **Specialty Grade:** This grouping encompasses “beads” of novel technologies; innovative pathways are being made to increase night-time visibility of roads including, but not limited to, blends, hybrid glass beads and ceramic beads with prismatic elements, all with different gradation profiles.
- vii. **Glass Particles**³: Small pieces or fragments of glass, as defined in clause 3.1 i), of any size similar to those stated above for glass beads.

NOTE²: Additional coatings can also be added to glass beads depending on the end results required. Adhesion coatings are added in order to enhance the adhesion to and embedment of the glass beads to the pavement marking material, whereas floatation coatings have oleophobic properties, allowing beads to float and not penetrate too far into pavement marking materials when dropped on to pavement marking materials when wet.

NOTE³: Previously, only glass beads were noted for inclusion in this specification. In consultation with industry members of RIAA and RAMPG, the inclusion of glass particles in this specification was deemed necessary to aid in the regulation of their use, specifically with regard to the long term heavy metal leaching potential.

4.2 Sub-Classes

- a) This specification incorporates the following sub-classes:
 - i. **0042/1:** Type B (standard grade) or Type B-HR (high retroreflectivity grade)
 - ii. **0042/2:** Type C (standard grade) or Type C-HR (high retroreflectivity grade)
 - iii. **0042/3:** Type D (standard grade) or Type D-HR (high retroreflectivity grade)
 - iv. **0042/4:** Type E
 - v. **0042/5:** Specialty Grade⁴
 - vi. **0042/6:** Glass Particles

NOTE⁴: Technical innovation: Where a manufacturer of glass beads (or other type bead/material) believes a new technical innovation will allow their product to pass the performance requirements specified in clause 8, Table 1, but not the compositional limitations of sub-classes 0042/1, 0042/2 and 0042/3, then a request may be made to APAS for application to sub-class 0042/5. This variation request shall be treated Commercial-in-Confidence by APAS, who shall decide on the validity of the variation request. In making this decision, APAS officers may consult with industry experts.

4.3 Technical Basis of this Specification

- a) This specification is based on AS/NZS 2009 with the addition of:
 - i. Heavy metals content restrictions.
 - ii. Retroreflectivity testing and performance requirements for Type B-HR, Type C-HR, D-HR, Type E and Specialty grade glass beads.

- iii. Inclusion of glass particles.

5 REFERENCED DOCUMENTS

- a) The following standards are referenced in this document:
 - i. **AS 1199** – Sampling procedures for inspection by attributes
 - ii. **AS/NZS 1580** – Paints and related materials: Methods of test.
 - iii. **AS/NZS 2009** – Glass beads for pavement marking materials
 - iv. **AS 4049.3** – Paints and related materials - Pavement marking materials – Part 3: Waterborne paint - For use with surface applied glass beads
 - v. **AS/ANZ ISO 9001** – Quality management systems - Requirements
 - vi. **AS ISO/IEC 17011** – Conformity assessment – Requirements for accreditation bodies accrediting conformity assessment bodies
 - vii. **AS ISO/IEC 17025** – General requirements for the competence of testing and calibration laboratories
 - viii. **AS/NZS ISO/IEC 17065** – Conformity assessment: Requirements for bodies certifying products, processes and services

These documents may be purchased through the Reference Standards Australia website:
<https://www.standards.org.au/>

- b) The following documents were utilised in the creation of this document:
 - i. AASHTO M 247 – Standard Specification for Glass Beads Used in Pavement Markings
 - ii. AASHTO T 346 – Standard Method of Test for Glass Beads Used in Pavement Markings
 - iii. AASHTO TP 106 – Standard Method of Test for Determination of Heavy Metal Content of Glass Beads Using X-Ray Fluorescence (XRF)
 - iv. EN 1423 – Road marking materials – Drop on materials – Glass beads, antiskid aggregates and the mixture of the two
 - v. EN 1424 – Road marking materials – Premix Glass beads
 - vi. TT-B-1325D Federal Specification Beads (Glass Spheres) Retro-reflective
- c) The following APAS documents are referenced in this document:
 - i. AP-D001 Rules Governing How APAS® Operates
 - ii. AP-D114 Rules Governing APAS® Recognition as a Testing Authority
 - iii. AP-D123 Restrictions on Ingredients in Product Formulations
 - iv. AP-D152 APAS® Participating Manufacturers and Resellers
 - v. AP-D177 Rules Governing How Product Manufacturers participate in APAS®
 - vi. AP-D192 Rules Governing APAS® Product Certification Scheme



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- vii. AP-D200 Application for Glass Bead Certification
- viii. AP-S0041/6 Pavement Marking Material – Airport Runway Markings

All APAS documents and specifications are available for download from the APAS website:

<https://vs.csiro.au/apas/documents/>

<https://vs.csiro.au/apas/specifications/>

6 COMPOSITIONAL REQUIREMENTS

6.1 General

- a) Glass beads in sub-classes 0042/1, 0042/2 and 0042/3 shall be made from glass conforming to clause 4, Materials of Manufacture of AS/NZS 2009.
- b) Glass beads or glass particles in sub-class 0042/4, 0042/5 and 0042/6 have no specific composition requirements.

6.2 Heavy Metal Content

- a) The heavy metals content in the glass beads and glass particles shall not be greater than that specified in clause 8, Table 1.
- b) As the preferred test method for heavy metals is ICPMS (refer to Appendix A, clause A.3), previous measures for Cadmium Oxide and Chromium 6+ have been changed to Cadmium and Total Chromium respectively, due to limitations of the ICPMS test method.
- c) The use of the XRF method is allowable provided that the XRF method is validated against ICPMS or, a certified glass matrix reference standard is used for measurement validation, refer to clause A.4.

7 PRODUCT CERTIFICATION REQUIREMENTS

7.1 General Requirements

- a) The product and its application for product certification shall comply with the relevant requirements of APAS document AP-D192 and this specification during the life of the approval.
- b) APAS document AP-D200 shall be supplied by the Client in lieu of APAS document AP-D139 (refer to clause 8.3, 8.6, 8.8 and 20 of APAS document AP-D192).
- c) Glass bead and glass particle products applying for initial product certification, having demonstrated conformance to all the relevant requirements of APAS document AP-D192 and this specification, as tested by an organisation holding AS ISO/IEC 17025 accreditation with all glass bead and glass particle test methods defined in clause B.3.7 under their Scope of Accreditation, shall be granted a once-off CLASS II (interim) certification for a period of 12 months.
- d) CLASS I (full) certification of glass beads and glass particles shall be granted for a 24-month period after the initial product certification, having demonstrated conformance to all the relevant requirements of APAS document AP-D192 and this specification, as tested

by an organisation holding AS ISO/IEC 17025 accreditation with all glass bead and glass particle test methods defined in clause B.3.7 under their Scope of Accreditation.

- e) Glass beads and glass particles shall conform to the compositional requirements (clause 6.1 above) and applicable performance requirements (clause 8, Table 1 below) of this specification throughout the certification period.
- f) The use of recycled materials in all aspects of manufacture is ever increasing. If a manufacturer chooses to use recycled materials in the production of products seeking accreditation against this specification, that is their choice as long as the compositional requirements (where applicable) and performance requirements of the specification are being met.

7.2 Technical Requirements

- a) The product shall comply with **all** the relevant requirements of clause 8, Table 1 below.
- b) The use of an appropriate particle size analyser for measuring particle size distribution (clause 5.4 of AS/NZS 2009) and shape (clause 5.5 of AS/NZS 2009) of particles is an allowable variance to the sampling methods of Appendix A, Method 1 or Method 2 of AS/NZS 2009. Details of this piece of equipment must be submitted to, and assessed by, APAS for approval prior to product application. Evidence of regular calibration records must be available for inspection at any time requested.
- c) Typically, the manufacturer and/or supplier applying for certification to this standard will undertake the full testing required (as per clause 8, Table 1) in their own AS ISO/IEC 17025 accredited testing facilities with all glass bead / glass particle testing under their Scope of Accreditation.
- d) If the Client applying for product certification does not have the capacity to perform their own testing, an alternative ACE holding AS ISO/IEC 17025 accreditation with all glass bead / glass particle testing under their Scope of Accreditation must be engaged for testing (refer to APAS document AP-D114 8.1 b) i. for further information.) Three (3) x 500g representative samples of the product must be sampled in accordance with Appendix A, Method 1 or Method 2 of AS/NZS 2009, and the sampling method stated. A single 500g sample is then to be supplied to the alternative ACE and two 500g retained samples are to be kept by the Client for a minimum period of twenty-four (24) months.
- e) For all Clients unable to undertake the testing themselves, they must get testing completed through an alternative ACE. At present, the only independent testing body available is the CSIRO Materials Durability Laboratory. Other suitably qualified ACEs will be duly considered and added to this document as their testing facilities become available.

The contact details for the CSIRO Materials Durability Laboratory are as follows:



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Money Arora
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- f) Subject to compliance with all the requirements of this specification, the level of Approval appropriate to the application shall be given.

7.3 Health and Safety Requirements

- a) The manufacturer's Safety Data Sheet (SDS) must be studied closely prior to using the product and complied with during use of the product.
- b) Products intended for sale in Australia and in other countries shall comply with all the requirements of all local WHS and environmental requirements.
- c) The product shall comply with all requirements of clause 6.3 and 6.4 of APAS document AP-D192.

7.4 Test Failure Procedure

- a) Where the glass bead / glass particle manufacturer and/or supplier encounters test failures, the quality assurance system (conforming to ISO 9001) shall describe the process for handling non-conforming results. This shall include re-testing, isolation, rework or disposal, as appropriate.
- b) Records of non-conformities shall be kept.



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8 TABLE 1: PERFORMANCE PROPERTIES

TEST	AS/NZS 2009 CLAUSE / APPENDIX	REQUIREMENTS
Foreign Matter	Clause 5.2	<p>0042/1: ≤ 50 - state results. 0042/2: ≤ 50 - state results. 0042/3: ≤ 25 - state results. 0042/4: Sample dependent* – state results. 0042/5: Sample dependent* – state results. 0042/6: Sample dependent* – state results.</p> <p>*NOTE: If the particle size span of the product is between 425-1000µm, requirement is ≤ 50; if the size is 1000µm+, requirement is ≤ 25.</p>
Optical Quality	Clause 5.3 / Appendix K	<p>Applicable to ALL sub-classes (except 0042/6) – state all results:</p> <ul style="list-style-type: none"> • Sample shall be clear; opacity ≤ 2%. • Sample with >25% of the visible area affected by gas inclusions is considered defective; ≤ 2% by count can show this defect. • Sample shall be smooth; crazing ≤ 2%.
Size	Clause 5.4 / Appendix C	<p>0042/1, 0042/2 & 0042/3: Shall comply with the size requirements of this clause pertaining to the product type (i.e., B/B-HR, C/C-HR, D/D-HR) – state results. 0042/4, 0042/5 & 0042/6: Sample dependent* – state results.</p> <p>*NOTE: If the particle size span of the product is between 425-1000µm, size requirement relates to product type B/B-HR or C/C-HR (state which one); if the size is 1000µm+, size requirement relates to product type D/D-HR.</p>
Shape	Clause 5.5 / Appendix D	<p>Applicable to ALL sub-classes (except 0042/6): ≥ 70% by mass – state results.</p>
Colour	Clause 5.6 / Appendix F	<p>Applicable to ALL sub-classes (except 0042/6): Lighter than reference sample – state results.</p>
Water Resistance	Clause 5.7 / Appendix G	<p>Applicable to ALL sub-classes: ≤ 4.5 mL – state results.</p>
Flow Properties	Clause 5.8 / Appendix H	<p>Applicable to ALL sub-classes (except 0042/6):</p> <p>Products <u>without</u> Moisture-Proof Coatings: Product that has been stored undercover in original unopened packaging for up to 12 months shall flow freely and show no signs of caking – state results.</p> <p>Products <u>with</u> Moisture-Proof Coatings: Product tested in accordance with Appendix H shall flow freely and show no caking – state results.</p>
Adhesion Coating	Clause 5.9 / Appendix L	<p>Applicable to samples with Adhesion Coating only: Confirm presence or absence of coating – state results.</p>



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8 TABLE 1: PERFORMANCE PROPERTIES (Cont.,)

TEST	AS/NZS 2009 CLAUSE / APPENDIX	REQUIREMENTS
Refractive Index	Clause 5.10 / Appendix I	Applicable to ALL sub-classes (except 0042/6): 0042/1, 0042/2 & 0042/3: ≥ 1.5 – state results. 0042/4: ≥ 1.9 – state results. 0042/5: State results.
Heavy Metals	Clause 5.11 / Appendix N & AP-S0042 Appendix A	Applicable to ALL sub-classes – state results: Antimony (Sb); Arsenic (As); Lead (Pb): ≤ 50 ppm each element – state results. Cadmium (Cd); Mercury (Hg); Chromium (Cr⁶⁺): ≤ 10 ppm each element – state results. NOTE*: If Total Chromium is greater than 10ppm, specific testing for hexavalent Chromium Cr ⁶⁺ should be undertaken via colorimetric method or other test method agreed to by APAS.
Retroreflectivity	Appendix M & AP-S0042 Appendix C	Applicable to all HR type products within ALL sub-classes (except 0042/6): 0042/1 & 0042/2: ≥ 450 mcd/lux/m ² – state results. 0042/3 & 0042/4: ≥ 600 mcd/lux/m ² – state results. 0042/5: State results.



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APPENDIX A

Sampling and Testing of Heavy Metals

A.1 SCOPE:

A.1.1 This method details the accepted method for the analysis of glass beads / glass particles for the purposes of conformance to this specification.

A.1.2 Sampling is a key aspect to ensure results are representative of deliveries and the method of sampling is defined in clause A.2.

A.2 SAMPLING:

A.2.1 Sampling shall be carried out in accordance with Appendix A, Method 1 or Method 2 of AS/NZS 2009. The sampling method shall be stated.

A.2.2 Samples shall be taken at the time of manufacture or upon delivery of glass bead / glass particle product, but prior to the glass bead / glass particle product being added into or applied onto pavement marking materials. Samples shall be taken for all APAS certified grades of glass beads / glass particles and Specialty Grades.

A.2.3 Frequency of sampling shall be not less than once per Lot (glass beads and glass particles). Samples will be generated as representative composites of the Campaign Run.

A.3 TESTING VIA ICPMS:

A.3.1 General:

- Testing to the method detailed shall be carried out by a laboratory accredited to AS ISO/IEC 17025 with this method included in their Scope of Accreditation.
- AS ISO/IEC 17025 accreditation shall be provided by an organisation accredited by an ILAC Mutual Recognition Arrangement signatory and having a Scope of Accreditation covering AS ISO/IEC 17025 requirements. In Australia, NATA provides AS ISO/IEC 17025 accreditation. A list of international ILAC accreditation bodies can be found on the ISO website.

A.3.2 Principle^{4,5,6}:

- A representative sample of glass beads or glass particles is milled to a fine particle size (approximately < 5µm), followed by complete dissolution in an acid mixture.
- Analysis for lead, antimony, arsenic, mercury, cadmium and total chromium is then carried out using ICPMS or equivalent analytical techniques capable of measuring parts per million levels of the elements in question.
- Method equivalence for non-ICPMS testing shall be demonstrated by means described in clause A.4.

NOTE⁴: These are analytical test procedures and must be conducted following recognised analytical principles.

NOTE⁵: Dangerous chemical agents are used in this process and must be handled and used in accordance with the SDS and the required laboratory standards.

NOTE⁶: The analysis is to be carried out using recognised reference materials traceable to international standards. Repeatability needs to be demonstrated as part of the analysis process. See clause A.4.

A.3.3 Reagents:

The following reagents are required:

- Analytical grade concentrated nitric acid
- Analytical grade 40% hydrofluoric acid
- Analytical grade concentrated hydrochloric acid
- De-ionised water, 18 M ohm-cm or better

A.3.4 Apparatus:

The following equipment is required:

- Sample milling equipment, zirconium mill
- Microwave digester
- Teflon microwave vessels
- 10 mL polypropylene tubes with lids
- 5 mL polypropylene tubes with lids
- Tube racks
- Fume cupboard
- Adjustable pipettes: 5 mL, 1 mL, 0.5 mL



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APPENDIX A (Cont.)

Sampling and Testing of Heavy Metals (Cont.)

- i. Balance, readable and accurate to 0.0001 g
- j. ICPMS or equivalent instrumentation

A.3.5 Procedure:

i. **Sample Digestion**⁷

- a) The zirconium mill must be **flushed** of any previous samples; 10 to 20 g of clean dry sand is milled and discarded and then the process repeated.
- b) 10 to 20 g of glass beads or glass particles are split from the composite sample obtained as in clause A.2, are placed into the mill and ground to approximately <math><5\mu\text{m}</math>. This material is discarded, and the process repeated. On the second repeat with the sample, the ground material is kept and transferred from the mill to a clean, labelled container and sealed for subsequent analysis.
- c) Weigh out approximately 200 mg of sample into a microwave vessel recording the weight to 4 decimal places. Prepare a duplicate and record sample mass as **m1** and **m2** for the first and duplicate masses of the sample.
- d) In a fume cupboard, add 2 mL nitric acid, 1 mL hydrochloric acid and 1 mL hydrofluoric acid to each vessel containing the previously weighed sample.
- e) Place the lids on the vessels, then place the vessels into the microwave digester.
- f) Digest the samples until no glass beads or glass particles remain. Once the digestion is completed, carefully remove the vessels from the microwave and allow to cool.
- g) Transfer total contents of microwave vessel to a labelled 10 mL tube, rinse vessel thoroughly with de-ionised water and add to the tube. Make tube volume up to the 10 mL mark with de-ionised water.

NOTE⁷: Clean vessels between each digestion with 2 mL nitric acid and 1 mL hydrochloric acid, digested for an appropriate period.

ii. **Analysis**

- a) Carry out an analysis of the digestion sample for lead, antimony, arsenic, mercury, cadmium and chromium using ICPMS or equivalent analytical techniques capable of measuring parts per million levels of the elements in question. Samples may require dilution in order to produce results of an appropriate order of magnitude.

iii. **Reporting**

- a) Results shall be reported as the average of the duplicates and as ppm of each metal per gram of glass beads or glass particles.
- b) The report shall include the details of the person who carried out the sampling and the sampling details.

A.4 **METHOD VALIDATION:**

A.4.1 For ICPMS methodology, method validation shall be demonstrated using certified reference materials.

A.4.2 For non-ICPMS methodology, where the testing authority is AS ISO/IEC 17025 accredited to conduct a non-ICPMS method, the testing authority shall establish and document a process that demonstrates method validity against ICPMS. The method shall be assessed by APAS for approval prior to the non-ICPMS methodology being implemented.



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APPENDIX B

Manufacture and Testing Controls

B.1 SCOPE:

- B.1.1 This Appendix details the controls imposed on glass bead or glass particle manufacture and testing necessary to impart confidence in APAS certification of such products.
- B.1.2 The process is necessary as APAS does not undertake assessments of capability of glass bead or glass particle manufacturers in a similar manner to surface coating material manufacturers.

B.2 PRINCIPLE:

- B.2.1 Confidence in glass bead or glass particle quality and conformance to this specification can be demonstrated by conforming to the requirements of clause B.3.

B.3 REQUIREMENTS FOR THE MANUFACTURE AND TESTING OF GLASS BEADS OR GLASS PARTICLES:

- B.3.1 A local glass bead or glass particle manufacturer is defined as having its manufacturing plant located in Australia. The manufacturer may sell its glass beads or glass particles direct to end-user customers or use a local distributor.
- B.3.2 An overseas glass bead or glass particle manufacturer is defined as having its manufacturing plant located outside of Australia. The manufacturer may sell its glass beads or glass particles direct to end-user customers, to pavement marking suppliers or use a local distributor.
- B.3.3 Manufacturers, pavement marking suppliers and local distributors shall operate under a quality management system, externally assessed as conforming to ISO 9001, by an entity accredited to do so by a member of the IAF. JAS-ANZ is the IAF member for Australia and New Zealand.
- B.3.4 Manufacturers shall operate their own or use an external test laboratory (ACE) to verify product conformity. Tests results shall be provided by a laboratory that fulfils AS ISO/IEC 17025 and its accredited for the relevant Scope of Testing by an accreditation body that fulfils the requirements of AS ISO/IEC 17011 and is a signatory of the ILAC MRA.
- B.3.5 Manufacturers shall demonstrate confidence in their technical controls by conducting random sampling and testing of their production output as follows:
- i. Sampling, which shall be conducted at least once per Lot, shall be in accordance with Appendix A, Method 1 or Method 2 of AS/NZS 2009 or, any alternative method which maintains the intent of, and when tested, is shown to be equivalent to AS/NZS 2009. If an alternative method is used, it is the responsibility of the manufacturer to demonstrate that any alternative method is equivalent to, or exceeds, the requirements of AS/NZS 2009. Any alternative sampling method shall be submitted to and assessed by APAS for approval prior to the sampling method being implemented. The sampling method shall be specified.
 - ii. Testing, which shall be conducted at least once per Lot, shall be in accordance with clause B.3.6. Testing shall be carried out by an authority agreed to by APAS.
- B.3.6 Pavement marking suppliers and local distributors, who shall source glass beads or glass particles only from manufacturers who conform to B.3.4, shall either have the glass bead or glass particle manufacturer undertake sampling and testing of glass beads or glass particles or conduct its own sampling and testing. Sampling shall occur in accordance with B.3.5. Testing shall occur in accordance with B.3.7.
- B.3.7 Mandatory tests⁸ (AS/NZS 2009 reference is noted in brackets) to be conducted for every Lot of glass beads or glass particles sampled are:
- Foreign Matter (clause 5.2)
 - Optical Quality (clause 5.3) – glass beads only
 - Size (clause 5.4)
 - Shape (clause 5.5) – glass beads only
 - Colour (clause 5.6) – glass beads only
 - Flow Properties (clause 5.8) – glass beads only
 - Adhesion Coating (clause 5.9) – glass beads only, where ever an adhesion coating is claimed to be present
 - Retroreflectivity (Appendix M and AP-S0042 Appendix C) – glass beads only, for all HR grades of glass beads



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APPENDIX B (Cont.)

Manufacture and Testing Controls (Cont.)

NOTE⁸: Testing of glass beads or glass particles for Water Resistance (clause 5.7), Refractive index (clause 5.10 – glass beads only) and Heavy metals (clause 5.11 and AP-S0042 clause 6.2 and A.4) is no longer required for every Lot of glass beads or glass particles. These three tests are still required to be performed but will now be at the discretion of the manufacturers testing regime, for example, tests carried out 3-monthly.

- B.3.8 Records of all test results shall be kept for every Lot of glass beads or glass particles tested. Numerical values shall be reported where the conclusion of a test method produces a numerical result.
- B.3.9 Records of test results shall be made available to APAS officers for certification/re-certification applications to this specification or upon request to do so by the APAS.
- B.3.10 Traceable retained samples of test samples, of sufficient quantity to re-do all tests specified in clause B.3.7 (i.e., 2 x 500 g samples) shall be kept for a minimum period of 24 months.



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APPENDIX C

Modified Retroreflectivity Test⁹

C.1 SCOPE:

C.1.1 This Appendix details the method used to assess the retroreflectivity of Type B-HR and Type C-HR glass beads (assessment of Type D-HR and Type E / Specialty Grade beads is referenced in AS/NZS 2009, Appendix M). It is based on a modification of the AS/NZS 2009 Appendix M method as developed by the Department for Infrastructure and Transport – South Australia.

C.2 MODIFICATIONS TO THE PROCEDURE:

C.2.1 In addition to the requirements of clause 8, Table 1 of this specification, **Type B-HR** beads (sub-class 0042/1) shall also be tested to Appendix M of AS/NZS 2009 with the following modifications:

- i. M4 APPARATUS (a) The *draw down paint applicator* shall be capable of casting a 100 mm wide application of 200 - 250 µm dry film thickness of paint.
- ii. M4 APPARATUS (h) *Reference paint material* - GTL-104-14B shall be replaced with an APAS certified white water-borne pavement marking paint conforming to the testing and performance requirements listed under reference 6.1 Laboratory testing of AS 4049.3 with the following modifications: reference 6.1.4 Application properties is excluded; reference 6.1.8 Specular gloss shall not exceed 5 gloss units and reference 6.1.9 Luminance factor shall be 88.5% ±2.5%.
- iii. M5 PROCEDURE (b) Weigh 24 ± 0.5 g of Type B-HR beads. Record the mass of beads.
- iv. M5 PROCEDURE (e) Set the blade of the draw down applicator to apply the paint so that a uniform dry film thickness of 200 - 250 µm is achieved.
- v. M5 PROCEDURE (f) The required dry film thickness shall be 200 - 250 µm over a minimum length of 600 mm on the test panel.

C.2.2 In addition to the requirements of clause 8, Table 1 of this specification, **Type C-HR** beads (sub-class 0042/2) shall also be tested to Appendix M of AS/NZS 2009 with the following modifications:

- i. M4 APPARATUS (a) The *draw down paint applicator* shall be capable of casting a 100 mm wide application of 200 - 250 µm dry film thickness of paint.
- ii. M4 APPARATUS (h) *Reference paint material* - GTL- 104-14B shall be replaced with an APAS certified white water-borne pavement marking paint conforming to the testing and performance requirements listed under reference 6.1 Laboratory testing of AS 4049.3 with the following modifications: reference 6.1.4 Application properties is excluded; reference 6.1.8 Specular gloss shall not exceed 5 gloss units and reference 6.1.9 Luminance factor shall be 88.5% ±2.5%.
- iii. M5 Procedure (b) Weigh 27 ± 0.5 g of Type C- HR beads. Record the mass of beads.
- iv. M5 PROCEDURE (e) Set the blade of the draw down applicator to apply the paint so that a uniform dry film thickness of 200 - 250 µm is achieved.
- v. M5 PROCEDURE (f) The required dry film thickness shall be 200 - 250 µm over a minimum length of 600 mm on the test panels.

C.2.3 In addition to the requirements of clause 8, Table 1 of this specification, **Type D-HR, E and Specialty Grade** beads (sub-class 0042/3, 0042/4 and 0042/5) shall be tested as per Appendix M of AS/NZS 2009 with the following modification:

- i. M4 APPARATUS (h) Reference paint material - GTL- 104-14B shall be replaced with an APAS certified white water-borne pavement marking paint conforming to the testing and performance requirements listed under reference 6.1 Laboratory testing of AS 4049.3 with the following modifications: reference 6.1.4 Application properties is excluded; reference 6.1.8 Specular gloss shall not exceed 5 gloss units and reference 6.1.9 Luminance factor shall be 88.5% ± 2.5%.

NOTE⁹: At the time of writing this specification, no data was available to document a procedure allowing for retroreflectivity testing of HR grade glass beads using an APAS certified white thermoplastic or cold applied plastic material in lieu of an APAS certified white water-borne pavement marking paint.



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APPENDIX D

Document History

Status: Current
 Version: 8
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Document Version No.:	Date Published:	Summary of Changes:
8	13-02-2023	<ul style="list-style-type: none"> Expansion of specification scope to include glass particles in all areas of this document (where applicable). Inclusion of definitions for glass beads and glass particles in clause 3.1 and RIAA and RAPMG in clause 3.2. Update of Appendices with numbering system and moved to the back of the document. Minor formatting changes.
7	06-04-2022	<ul style="list-style-type: none"> Updated clause 7.2 e) regarding glass bead testing and NATA status Updated clause 9.3.7 to remove the requirement for every Lot of glass beads to be tested for Water Resistance (clause 5.7), Refractive index (clause 5.10) and Heavy metals (clause 5.11 and AP-S0042 clauses 6.2 and 8.4) . These three tests are still required to be performed but will be at the discretion of the manufacturers testing regime, for example, tests carried out 3-monthly.
6	02-08-2021	<ul style="list-style-type: none"> Full Technical review of document Change of name from <i>Glass beads for use in pavement marking materials</i> to <i>Glass Beads – For use in and with Pavement Marking Materials</i> Addition of Appendix D Document History and removal of the Editorial Note previously used in specification versions Inclusion of clause 5 b) Reference material Document brought in line with requirements of AS/NZS ISO/IEC 17065 Updated document to the current format Updated internal and external document references Addition of "People + Product = Protection" to Footer
5	05-08-2019	<ul style="list-style-type: none"> Full re-write of the specification
4	19-10-2018	<ul style="list-style-type: none"> Re-write of the specification
3	10-06-2015	<ul style="list-style-type: none"> Changed the certification interval from 12 to 24 months in clause 7.1b) and clarifies some manufacturer controls in Appendix B.
2	23-08-2013	<ul style="list-style-type: none"> General update and format revision Sub-classes added Controls over manufacture added
1	24-05-2007	<ul style="list-style-type: none"> Clarified accreditation requirements for testing in clause A2.1 and A2.5.2