Statement of measurement

ISO 9001 Quality Management certified by BSI under certificate number FS27613

Colloidal gold nanoparticles – nominal diameter 30 nm

Quality Control Material LGCQC5050

Assessed value

	Value	Uncertainty[2]
Number particle concentration[1] NP/g	1.47×10^{11}	2.8×10^{10}

Notes:
1. Number particle concentration of colloidal gold nanoparticles. Value obtained at LGC using single particle inductively coupled plasma mass spectrometry (sp-ICPMS). The value is traceable to the SI.
2. The uncertainty quoted is the half-width of the expanded uncertainty, calculated using a coverage factor of two, which gives a level of confidence of approximately 95 %. This uncertainty includes a contribution for both homogeneity and long term stability.

Indicative values

	Value	Uncertainty[3]
Particle modal diameter[1] nm	32.7	2.0
Gold mass fraction[2] mg/kg	45.1	1.5

Notes:
1. Value obtained at LGC using particle tracking analysis (PTA).
2. Value obtained at PTB, Germany (using ICP-MS).
3. The uncertainty quoted is the half-width of the expanded uncertainty, calculated using a coverage factor of two, which gives a level of confidence of approximately 95 %. This uncertainty does not include a contribution for homogeneity or long term stability.

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Signed: _____
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Material Preparation

The starting material was a commercially prepared solution consisting of colloidal spherical gold nanoparticles (citrate stabilised) with average diameter of approximately 30 nm, suspended in water. After careful mixing, the solution was dispensed into amber glass ampoules in 5.2 mL portions and sealed under argon.

The filled units were irradiated for sterilisation purposes using Co^{60} gamma irradiation at a minimum dose of 35 kGy. Approximately 500 units were prepared and stored at (5 ± 4) °C.

Homogeneity Assessment

The homogeneity of the material has been assessed using fifteen ampoules analysed in duplicate using Particle Tracking Analysis (PTA). A sub-sample of 50 mg was taken from the ampoule for each measurement. The results confirmed that the material was fit for purpose and a contribution for homogeneity uncertainty was included in the combined uncertainty calculation.

Stability

In a short-term accelerated stability study, units were stored at temperatures ranging from 5 °C to 60 °C for up to 14 days. The results showed no significant change at any temperature over the study period.

In a long-term stability study, samples stored at (5 ± 4) °C were analysed over a period of 8 months using PTA. The results showed a marginally statistically significant change during the study period, and a contribution has been included in the combined uncertainty for long term stability uncertainty.

The stability of LGCQC5050 will be monitored on a regular basis as part of LGC's reference materials stability testing programme, and customers will be informed of any significant changes in the material.

Characterisation

Particle number concentration

Analysis was carried out at LGC, UK, on 15 vials. Before opening, vials were allowed to reach room temperature and gently inverted several times to ensure homogeneity of the suspension. Samples were diluted gravimetrically in 1 mM trisodium citrate buffer to prepare the solutions for analysis.

sp-ICPMS measurements were performed using an Agilent 8900 ICP-MS/MS in single particle mode. The samples were introduced into the plasma via a micromist nebuliser, and using a Scott type double pass spray chamber cooled to 2 °C. Determination of the nanoparticle (NP) transport efficiency was performed using the dynamic mass flow method reported elsewhere.[1]

Particle modal diameter

Analysis was carried out by LGC, UK, on five vials using PTA. Before opening, vials were allowed to reach room temperature and gently inverted several times to ensure homogeneity of the suspension. Samples were diluted in 1 mM trisodium citrate buffer to prepare the solutions for analysis.

PTA measurements were performed using an NS500 instrument from Malvern Panalytical, equipped with: violet diode laser 405 nm CW max power <60 mW, EMCCD camera and NTA3.2 software. The samples were analysed using automated focus and camera levels, temperature was maintained at 25 °C (accuracy ± 0.1 °C) and the detection threshold set by the operator.

Gold mass fraction

Analysis was carried out at PTB, Germany, on 5 vials. Samples were allowed to reach room temperature and stirred well using a vortex mixer before opening.

Analysis was carried out by standard addition. Samples were diluted with citrate solution and then thallium was added as an internal standard. Digestion was carried out using a heating block on a hotplate, and then a microwave oven with nitric acid, hydrochloric acid and hydrogen peroxide. The residue was dissolved in hydrochloric acid for measurement.

HR-ICP-MS was used for quantification of the gold concentration, using NIST SRM 3121 gold solution as the primary calibrant.

Intended Use

This QC material is intended, primarily, to evaluate and qualify methodology and/or instrument performance related to the number-based characterisation of nanoscale particles, including particle concentration and diameter.

The indicative values are not suitable for establishing method bias and metrological traceability.

Storage and Shelf life

This material should be stored before use at (5 ± 4) °C in the ampoule as supplied. Before use, the ampoule should be allowed to reach room temperature (20 ± 5) °C and then opened carefully to avoid injury.

The material stability is not affected by short periods of ambient handling during transport or use, but freezing must be avoided.

The material should be handled with care, following normal health and safety precautions.

Providing it is stored in the original packaging under the recommended storage conditions, this statement will remain valid for a period of 12 months from the date of shipment.

Instructions for use

The material is supplied with a temperature strip on the packaging. Please contact your distributor if the ampoule has reached 0 °C, or below, during transit.

Before opening, invert the ampoule carefully several times to ensure thorough mixing of the contents. This is important as settling is likely to occur if the ampoule is left undisturbed for a few days as shown in the photograph.

Any dilutions of the material should be made in 1 mM trisodium citrate buffer, and the recommended minimum sample size for use is 50 mg based on the homogeneity study.

It is recommended that the contents of an ampoule are used the same day as opened. Clean laboratory sealing film can be applied to cover a previously opened ampoule for short term storage (i.e. not more than 7 days). Stability after longer term storage cannot be guaranteed, but may be possible if additional precautions are followed, such as aliquoting under sterile conditions and storage in tightly closed containers.

Metrological traceability

The assessment value (nanoparticle number concentration) is traceable to the SI through gravimetric determination of the sample mass flow, gravimetric preparation of nanoparticle sample dilutions and gravimetric determination of the nanoparticle transport efficiency. Weighing operations are traceable to the SI through the use of calibrated weights traceable to the National Primary Standard of mass via the UK National Physical Laboratory (NPL).

Reference

1 Susana Cuello-Nuñez, Isabel Abad-Alvaro, Dorota Bartczak, M. Estela del Castillo Busto, David Alexander Ramsay, Francesco Pellegrino and Heidi Goenaga-Infante. The accurate determination of inorganic nanoparticles using spICP-MS with the dynamic mass flow approach. J. Anal. At. Spectrom. 2020, DOI: 10.1039/c9ja00415g.

Acknowledgement

LGC would like to thank PTB, Germany for the provision of the gold mass fraction value.

Certificate revision

In June 2019 and March 2020, amendments were made to the wording in the reference section. In November 2021, a correction was made to the wording in the characterisation section

Unit Number	Date of Shipment

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