
**Fine ceramics (advanced ceramics,
advanced technical ceramics) —
Measurement of iso-electric point of
ceramic powder**

*Céramiques techniques — Mesure du point iso-électrique de poudres
céramiques*

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Measurement of iso-electric point of ceramic powder

1 Scope

This document specifies the test method to determine the iso-electric point of fine ceramic powders, which is measured in the state of suspension.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4316, *Surface active agents — Determination of pH of aqueous solutions — Potentiometric method*

ISO 13099-1, *Colloidal systems — Methods for zeta-potential determination — Part 1: Electroacoustic and electrokinetic phenomena*

ISO 13099-2, *Colloidal systems — Methods for zeta-potential determination — Part 2: Optical methods*

ISO 13099-3, *Colloidal systems — Methods for zeta-potential determination — Part 3: Acoustic methods*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 13099-1, ISO 13099-2 and ISO 13099-3 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1

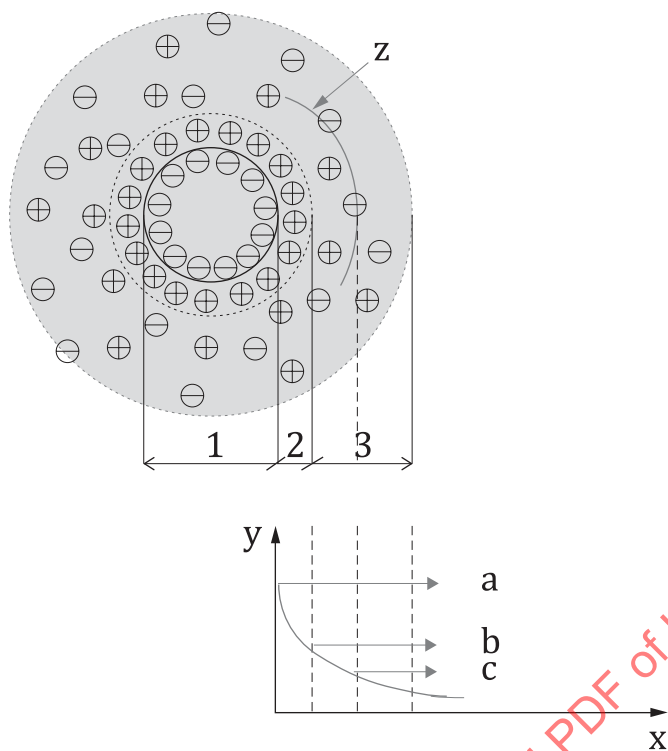
zeta potential

ζ-potential

difference between the electric potential at the slipping plane and that of the bulk liquid, where the electric potential difference is proportional to the electrophoretic mobility of the charged particle to the electrode when the electric field is applied to the dispersed particles in the solution

Note 1 to entry: See [Figure 1](#).

Note 2 to entry: Zeta potential is expressed in volts. Electrophoretic mobility (μ) is electrophoretic velocity divided by electric-field strength. Electrophoretic mobility is positive if the particles move towards a lower potential (negative electrode) and negative in the opposite case. Electrophoretic mobility is expressed in square metres per volt second ($\text{m}^2/\text{V}\cdot\text{s}$).



Key

- a surface potential
- b stern potential
- c zeta potential
- x distance from particle surface
- y potential
- z stern layer
- 1 colloid particle
- 2 stern layer
- 3 ion diffusion layer including zeta potential

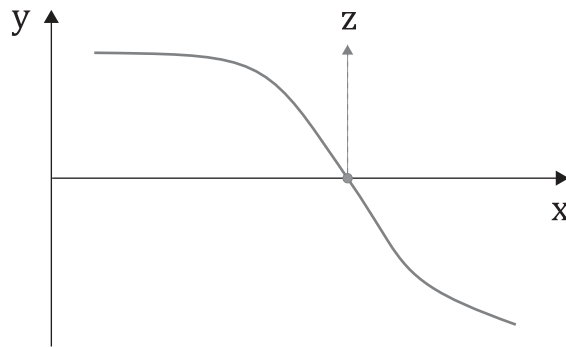
Figure 1 — Schematic of zeta potential for the dispersed colloid particle

3.2

iso-electric point

condition of a liquid medium, usually the value of pH, that corresponds to zero zeta potential of dispersed particles

Note 1 to entry: See [Figure 2](#).

**Key**

x	pH
y	zeta potential
z	iso-electric point

Figure 2 — Representative example of iso-electric point

4 Principle

Zeta potential is the potential in the interfacial double layer at the location of the slipping plane versus a point in the bulk fluid away from the interface. According to general colloid chemistry principles, an electrostatically stabilized dispersion system typically loses stability when the magnitude of the zeta potential decreases. As a result, there will be some region surrounding the condition of zero potential (i.e. the iso-electric point) for which the system is not particularly stable. Within this unstable region, the particles may agglomerate, thereby increasing the particle size. Determining the pH conditions where the zeta potential becomes zero (the iso-electric point) is, therefore, important for zeta potential analysers. Zeta potential may be determined by the electrophoretic light-scattering (ELS) method, the streaming potential method and the electroacoustic method, according to ISO 13099-1, ISO 13099-2 and ISO 13099-3, respectively.

5 Apparatus and calibration

The usual laboratory apparatus and, in particular, the following.

5.1 Zeta potential analyser and calibration

The zeta potential analyser may vary according to the measurement principle, such as electroacoustic and electrokinetic phenomena, the optical method or the acoustic method, and shall conform to ISO 13099-1, ISO 13099-2 and ISO 13099-3, respectively. The calibration of the analyser shall be performed using the standard sample with a predefined zeta potential.

NOTE The standard sample provided by the zeta potential analyser supplier could be applied.

5.2 pH meter

The pH meter should be within the measuring range of pH 2 to pH 10. The calibration of the pH meter shall conform to the standard procedure detailed in ISO 4316.

5.3 Sample dispersion vessel

A vessel of polypropylene or glass in the dimension range of 100 cm³ to 1 000 cm³. It is used for dispersing the powder sample in the electrolyte solution.

Materials that do not elute in the range of pH 2 to pH 10 should be used. Special care is needed for soda lime glass, which may elute in a strong base condition.

5.4 Sonicator

The sample should be dispersed by a tip sonicator, preferably, or a bath-type sonicator. A homogenizer, mortar, or vacuum degassing machine can be applied when needed.

5.5 Balance

A balance with a measuring capacity of 200 g or above with 10-mg precision.

5.6 Temperature-control device

A thermometer with a measuring range of 0 °C to 50 °C with a precision of 0,5 °C. A built-in temperature sensor is also applicable.

5.7 Magnetic stirrer

A stirrer or magnetic stirrer coated with polytetrafluoroethylene resin.

6 Operational procedures

6.1 Dispersion solution

This shall be ion-exchanged distilled water with a conductivity of less than 1×10^{-4} S/m (25 °C) in an electrolyte solution.

6.2 pH-adjusting acid or base and electrolyte solution

The pH-adjusting acids or bases and the type of electrolyte solution shall be selected as follows.

- The pH-adjusting acid and base shall be hydrochloric acid (HCl) or nitric acid (HNO₃) and sodium hydroxide (NaOH) or ammonium hydroxide (NH₄OH), respectively. The electrolyte solution shall be a salt, sodium chloride (NaCl) or ammonium nitrate (NH₄NO₃) solution with a concentration of 0,1 mol/l for acid and base and 1 mmol/l to 10 mmol/l for the electrolyte solution.
- Any combination of inorganic acid and base with an oxidation number 1, except those in a), may be selected, and their electrolyte (1 mmol/l to 10 mmol/l) may be selected.

6.3 Sample preparation and dispersion

6.3.1 Preparation

The dispersion solution of ion-exchanged distilled water shall be selected as in [6.1](#) and an electrolyte solution of 10^{-3} – 10^{-2} mol/l prepared. The powder sample shall be dispersed in the electrolyte solution to produce a ceramic slurry of suspension.

6.3.2 Dispersion

The ceramic slurry shall be well dispersed by using a tip sonicator or ultrasonic bath. The ultrasonic irradiation shall be carried out as follows. If the ultrasonic bath is applied, place the sample above the spot of oscillation for no less than 3 min below 300 W power. If using an ultrasonic tip sonicator, place the sonicator tip in the middle of the beaker bottom and top liquid interface of the ceramic slurry, sonicate for 1 min to 5 min and stabilize for 5 min to prevent any temperature increase.

The recommended concentration of the ceramic slurry is a mass fraction of 1 % to 10 % (depending on particle size).

If the suspension (or colloid) is well dispersed, it may be measured without the assistance of sonication.

6.3.3 Stabilization

The suspension shall be stabilized after sonication, for a recommended time of 30 min, and the supernatant taken for sampling.

Special care is needed in the case of an inhomogeneous mixture sample, because the selection of the supernatant part might not represent the composition of the sample powder.

An additional milling or dispersion method may be applied when the coarse particles are very settled.

The recommended sample concentration of ceramic slurry is 1 wt% to 2 wt% for the streaming potential method, 1 vol% for the electroacoustic method and 0,001 wt% to 0,05 wt% for the ELS. It shall be adjusted depending on the size of particles, where the smaller the size, the higher the concentration needed.

7 Measurement

Zeta potential measurement and pH adjustment shall be performed as follows.

- a) Switch on the analyser and wait until the device is stabilized.
- b) The initial calibration of the device shall be done in accordance with [5.1](#).
- c) Calibrate the pH meter in accordance with [5.2](#), including built-in parts in the zeta potential analyser.
- d) Adjust the concentration of the sample as appropriate and place the sample slurry in the measuring cell.

The zeta potential may vary depending on the concentration of the sample slurry, so the concentration should be recorded.

- e) Measure the pH and temperature of the sample solution after adjusting the concentration.
- f) Measure the zeta potential in accordance with the selected method in [Clause 4](#).
- g) Adjust the pH of the sample slurry from d) using the pH-adjusting acid and base selected in [6.2](#), where pH change is recommended to be within 1, and stabilize the pH-adjusted sample slurry for at least 10 min.
- h) Repeat procedures e) to g) until the pH of the sample slurry is adjusted until it reaches either pH 2 by acid or pH 10 by base.

Special care is needed when the zeta potential is measured near the iso-electric point (which has a zeta potential value smaller than ± 10 mV), because it may have low reproducibility. The pH should be adjusted so that an absolute value of zeta potential of more than 10 mV is obtained.

When the pH adjustment is overshoot, then it is recommended that the pH adjustment not be reversed, because this might result in an additional change in the concentration of electrolyte as well as the sample concentration.

- i) Turn off the power of the zeta potential analyser.

8 Determination of the iso-electric point

Measure the iso-electric point as follows.

- a) Plot the pH versus zeta potential and connect the data points with a smooth curve.