

Dissolution rate (ASTM F1926) and solubility product (ISO 13779-6) of calcium phosphates

Calcium phosphate ceramics are widely used for the repair of bone defects and as coatings for orthopaedic implants. In order to match the growth rate of new bone, the resorption properties of these materials must be carefully controlled. In particular, the resorption rate may be affected by foreign crystalline or amorphous phases or chemical contamination. *In vitro* testing of the dissolution kinetics as well as the (equilibrium) solubility of calcium phosphates provides an indication of the stability of the material in the human body. Therefore, dissolution testing is required in ASTM F1926 and the determination of the solubility product is recommended in ISO 13779-6.

At RMS Foundation, a dissolution test according to ASTM F1926 was recently established and validated according to the requirements of our ISO 17025 accreditation. In this test, a sample is immersed in a buffer solution (MES or TRIS) at 37 °C for 24 hours and the Ca concentration is continuously measured by means of a Ca ion-selective electrode (see figure). In addition, the pH of the solution is measured at the beginning and at the end of the experiment

in order to exclude a pH drift, i.e. to ensure a sufficient buffer strength. The test can be performed on bone substitute products of any shape as well as on coated specimens or powder scraped from a coating. Since a direct correlation of the dissolution rate *in vitro* with the resorption rate *in vivo* is not possible, carrying out parallel tests on a historical control or a certified reference material is recommended.

The solubility product (K_{sp}) is a material property describing a chemical equilibrium that exists between a solid compound and a solution of that compound. We have now defined and validated a test method for the determination of the K_{sp} of hydroxyapatite raw material powders according to ISO 13779-6. This method consists of immersing a sample in dilute phosphoric acid for 60 days at 37 °C and subsequently measuring the pH as well as the Ca and P concentration by inductively coupled plasma mass spectrometry (ICP-MS). K_{sp} is then calculated using an equation provided in ISO 13779-6 (annexe A.8). For comparison, the K_{sp} of a pure fully crystallized reference material (e.g. NIST calcium hydroxyapatite, SRM 2910) can be determined in parallel. Depending on the purity of the sample, it may be possible to use a considerably shorter test duration.

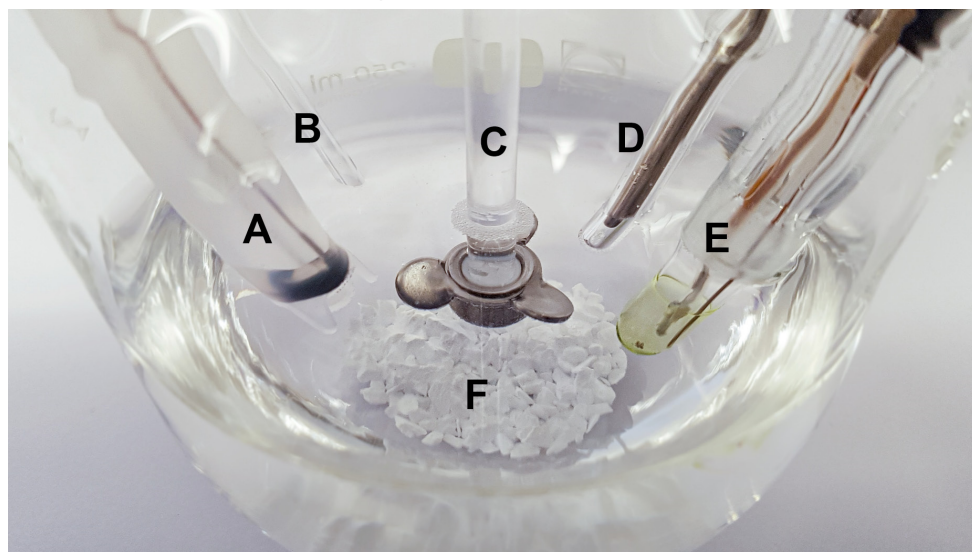


Figure: Dissolution vessel containing buffer solution (ASTM F1926), A) Ca ion-selective electrode, B) N₂ purge gas tube, C) overhead stirrer (in case of powder, granule or preform samples), D) temperature sensor, E) pH electrode, F) sample material.

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Sample requirements:

Dissolution testing (ASTM F1926):
150 mg powder/granules, fabricated forms, powder scraped from coatings, or coated test coupons

Solubility product (ISO 13779-6):
400 mg powder
(6 replicates)

Equipment:

Polymer membrane Ca ion-selective electrode (Metrohm Titrand), pH electrodes, Agilent 7700x ICP-MS

Further dissolution tests offered:

- Dissolution and pH change of calcium phosphates according to ISO 13175-3
- Ceramic degradation products (biological evaluation) according to ISO 10993-14

Discuss your questions with us! We are happy to advise you.

Contact for dissolution and solubility testing:

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More information as well as our service catalogue can be found on our website.

RMS Foundation has been certified according to ISO 9001 and is an ISO/IEC 17025 (type C) accredited laboratory.

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