

PRIMUS – Primary Multi-Ion Standards for Ion Chromatography

With PRIMUS, Fluka GmbH introduces certified primary multi-ion standards to the market. These «ready-to-use» standards allow the validation of analytical methods in a very simple way. PRIMUS standards contain 10 mg/kg of each Br^- , Cl^- , F^- , NO_3^- , PO_4^{3-} , SO_4^{2-} (anion solution) or Ca^{2+} , Li^+ , K^+ , Mg^{2+} , Na^+ (cation solution).

Two metrological institutes – EMPA (Switzerland) and BAM (Germany) – together characterized primary materials and issue a joint certificate. The solutions are produced at EMPA using the facilities of their metrological laboratories. This is why PRIMUS standards are characterised by their traceability to SI as well as a unique low degree of uncertainty of $\leq 0.2\%$.

Increasing demand on CRMs

From a review of recent years certain terms stand out with respect to chemical analysis. These are: «certification», «validation» and «compliance». The demands placed on both the quality and the documentation of the analytical work have continually increased over the last years. A very central part within these requirements is the validation of analytical methods whereby the so-called «performance qualification» can always only be as accurate as the standards with which they are carried out. This is why certified standards are playing an increasingly important role in analytical procedures.

Directives, for example those issued by the European Union, require that a CRM (CRM = Certified Reference Material) must be used for checking the correctness of analyses, whenever available [1]. The American FDA (Food and Drug Administration) stipulates the use of reference standards in analyses in its «Inspection Guide». The ISO (International Organization for Standardization) has also issued more than six publications on the subject of reference materials [2].

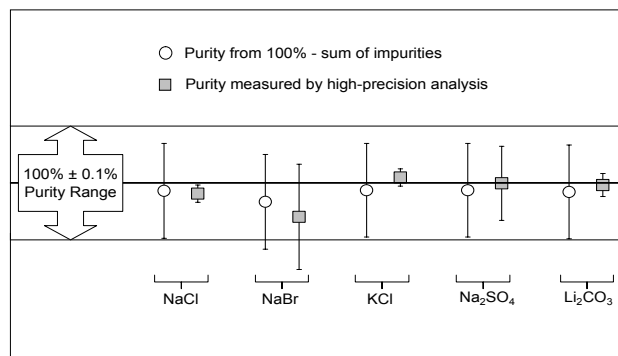
PRIMUS Multi-ion standards

«PRIMUS» stands for **Primary Multi-Ion Standards** and is a joint project of EMPA, Fluka GmbH and Metrohm whereby BAM (Federal Institute for Materials Research and Testing) acts as a partner institute for certification. The standards are prepared gravimetrically based on the kilogram as one of the seven basic SI-units. In gravimetric production the quality of the original substances used is of crucial importance. This is why a dual approach has been selected for the certification of the primary materials for PRIMUS: as many as possible of the trace impurities in the primary materials are quantified or their non-existence is demonstrated (below detection limit). This involves the analysis of up to 85 parameters per substance including cations, anions and non-metals. Residual water is eliminated by drying temperatures up to 500 °C and the successful completion of the drying process is checked by TGA (TGA = thermogravimetric analysis).



Bottled PRIMUS solutions with Al-coated bag and certificate

The purity calculated in this way is then confirmed by an independent high-precision measurement of the contents by coulometry or titrimetry. This additionally substantiates the accuracy of the certified content and the correct stoichiometry of the compound.



Weighing out the primary materials as well as the 50 liter batch is carried out on high-precision balances from Mettler-Toledo in an air-conditioned weighing room (OIML class E2). The primary materials are dissolved in ultrapure water or diluted high-purity HCl in specially manufactured PVDF containers and then mixed for 12 hours in an overhead shaker.

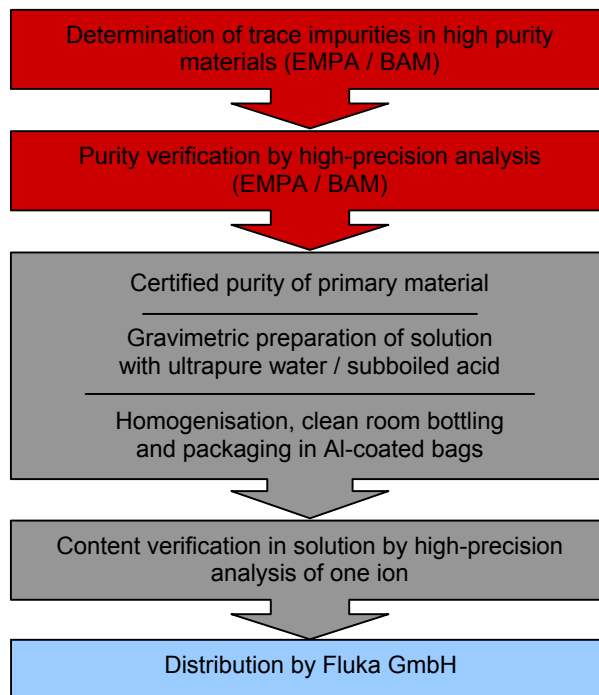


OIML Class E2 weighing room at EMPA Switzerland

The solutions are then filled off into pre-cleaned 50 mL HDPE containers under clean-room conditions. The high-precision analysis of one analyte in the bottled solutions provides a final check of the manufacturing procedure and corresponds to a type of double safeguard. In order to prevent evaporation losses the containers are sealed in Al-coated bags. Long-term tests have shown that solutions packed in this way have a stable shelf life of at least three years.



Schematic manufacture and certification procedure of PRIMUS standards:



[1] Commission decision of 12 August 2002 implementing Council Directive 96/23/EC concerning the performance of analytical methods and the interpretation of results. Official Journal of the European Communities, L 221/8

[2] ISO Guides: **30** (1992) Terms and definitions used in connection with reference materials; **31** (2000): Reference materials – contents of certificates and labels; **32** (1997): Calibration in analytical chemistry and use of certified reference materials; **33** (2000): Uses of certified reference materials; **34** (2000): General requirements for the competence of reference material producers; **35** (1989): Certification of reference materials – General statistical principles

Metrohm highly recommends the use of PRIMUS Standards for the internal calibration and validation of their instruments. Each new Metrohm IC apparatus contains a PRIMUS pack with multianion and multication

see more about CRM's on our webpage: www.sigma-aldrich.com/certified_standards

Ordering Information:

| Cat. No. | Brand | Product | Ions | Produced from | Pack Size |
|----------|-------|---|--|---|-----------|
| 89316 | Fluka | Certified Multication Standard Solution | Lithium, Sodium, Potassium, Magnesium, Calcium | Li ₂ CO ₃ , NaCl, KCl, MgO, CaCO ₃ | 50 mL |
| 89886 | Fluka | Certified Multianion Standard Solution | Fluoride, Chloride, Bromide, Nitrate, Phosphate, Sulfate | NaF, NaCl, NaBr, NaNO ₃ , Na ₂ HPO ₄ , Na ₂ SO ₄ | 50 mL |

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