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Determination of Assigned Values for Cadmium, Cobalt and Manganese in Drinking Water Proficiency Testing

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Abstract

In proficiency tests the selection of an appropriate assigned value is one of the most critical points. Determination of the assigned value for proficiency test can be done in 2 ways: based on reference value and consensus value. The use of such a consensus value as an assigned value is a commonplace in proficiency testing, because there is seldom at present a cost effective alternative. However, the consensus value has a weakness in that it could be significantly different from the true value due largely inexperienced laboratories. Therefore in this work, the reference values were used as the assigned values for all analytes of interest. The reference values in spiked drinking water samples were produced by the Metrology in Chemistry Laboratory of Research Center for Chemistry - Indonesian Institute of Sciences (RCChem-LIPI) with traceable to SI units through standard solutions from the National Institute Standard and Technology, USA. The reference values of cobalt and manganese were obtained by measurement using two from three comparable methods of Graphite Furnace-Atomic Absorption Spectrometry (GF-AAS), Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) and Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) while that for cadmium was obtained by a primary method of ICP-Isotope Dilution Mass Spectrometry (ICP-IDMS).The obtained reference values were used to evaluate the laboratory performance result in a proficiency testing scheme of cadmium, cobalt and manganese in drinking water which was provided in 2014. All fifty nine laboratories reported cadmium and cobalt at low concentration levels which were about more than 82 % showed satisfactory performance results. Similarly, the results of manganese were also comparable at high level concentrations.

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1. Introduction

Proficiency tests (PT) are now widely used to evaluate the analytical competence of field laboratories, are and also used as part of accreditation processes1. However, other benefits can be obtained from PT. These include the comparison of data for given measurement by different methods, the validation of new methods, and the provision of information for the laboratories customers and the accreditation bodies2. In a proficiency test the result of the laboratories should be assessed by comparing them with the true value. Unfortunately, the true value is generally unknown. Therefore a value must be assigned, which is assumed to be as close as possible to the true value3. Usually, the selection of an assigned value is based on:

(1) Value derived by specific test material formulation
(2) Value determined by measurement of the test material and comparison to a reference material or standard, traceable to a national or international standard
(3) Determined by expert laboratories that have proved experience in measuring the test item
(4) Value obtained by the test result provided by the participant laboratories

The assigned value produced by (1), (2) and (3) is called “reference value”, while an assigned value provided by (4), based on the participants results, is defined as “consensus value”4.

Metrologically traceable references values add an essential benefit to interlaboratory comparisons: unlike consensus values, they can be used to establish national and international comparibility. Furthermore, the participating laboratories obtain a reliable and unbiased benchmark to check their results for accuracy5. Since 2000, Research Center for Chemistry - Indonesian Institute of Sciences (RCChem-LIPI) has been conducting proficiency testing study for a range of metal ion concentrations in drinking water. All the data evaluations were made base on consensus values. If a large number of laboratories have bias results in one direction (e.g. losses during the analysis of volatile substances) this method will also result in a bias assigned value. It can be mean that laboratories with accuracy result will be punished3.

The assigned value is the value to which participants results are compared, and must be the best available estimate of the true concentration of analyte. It is important to clearly define the measurand such that the assigned value only relates to that measurement. Traceability of the assigned value is achievable provided there are direct links to stated references, together with sound estimates of the uncertainty of the links. RCChem-LIPI as a Designated Institute (DI) for metrology in chemistry in Indonesia is responsible for dissemination of the national standards with assigned values to set up at least the first link of the traceability chain5, establish and maintain traceability to SI, where this is technically feasible, but not necessarily at the highest metrological level6. The aim of the research is to give confidence on the trueness or accuracy of the PT results by the using of assigned value and its uncertainty for PT sample for all analytes of interest.

To address the measurement capability, all participant laboratories was supplied with one bottles of mix-standard solution together with the proficiency test item. Both standard and the test item are candidate reference materials that can be used as an alternative to fulfill some of the technical requirements such as to evaluate some validation parameters and as quality control materials. This can give additional use of proficiency testing that can help laboratories to reduce the financial impact of the quality assurance process7.

2. Material and Methods

2.1. Preparation of proficiency test items

The proficiency test items are commercially available drinking water which spiked with the desired analytes. A 10-L class A volumetric flask (Duran, Germany) was filled roughly below its mark with the drinking water, and then added with 300 mL of concentric nitric acid (Merck, Germany). A pipette (Eppendorf, Germany) was used to transfer 0.1, 0.4 and 1.6 mL of 1000 mgL^{-1} cadmium, cobalt and manganese standard solution (Merck, Germany), respectively, to the flask in order to get the desired concentration of the analytes of interest. The flask was made up to the mark using the drinking water. It was drain into a 50 L HDPE carboy (Nalgene, USA). The flask was filled up again to its mark for another four times, but solely using the drinking water. Finally, the total volume of the test sample got was 40 L. After homogenisation process, the test sample was bottled into labelled and individual test