

Table of learning points and possible improvements arising from evaluated ILCs

Number and description of the ILC	Description of the observation	Suggestions
IMEP 22 Sulfur in Petrol	The PT shows that there are some doubts that EDXRF is suitable for measuring Sulfur in Petrol at a level of 20 mg/kg.	During the assessment of this EDXRF method this has to be taken into account.
APLAC T057 Metals in Seawater Shrimp	Arsenic showed a bimodal distribution. MdIC explained that this is a result often obtained for Arsenic. Some digestion methods will result in different oxidation states for Arsenic which interfere the analyses leading wrong (smaller) results.	In the assessment of Arsenic in solid materials the assessor should focus on the digestion method. It has to be verified that the digestion leads to one oxidation state (As ⁵⁺).
IfEP HRC 2008 Rockwell Hardness	Bad testing results for Rockwell Hardness Scale C at low levels (10 – 25), especially repeatability.	The test procedure on the low hardness-levels, especial the repeatability should be verified. When assessing Rockwell Hardness the assessors should focus on low level HRC, when applicable or limit the accreditation to higher HRC levels.
IMEP 23 PAH in surface water (WFD)	Some of the laboratories found too low concentrations of PAH compounds of high molecular weight compared with the reference values given.	This was due to the handling of the internal standard. Equilibration times lower than 24 h. In addition the kind and number of the internal standards used did not reflect the properties of the requested PAH compounds. By assessing the analysis of PAH in natural waters and soils special attention should be taken on the handling and composition of the internal standard used.
IMEP 27 Metals in mineral feed	Total and extractable Lead was underestimated in mineral feed by a lot of laboratories	It is assumed that this is due to not applying the recovery rate or using the wrong reference material to estimate the recovery rates of the extraction step (e.g. RM of organic matrix for mineral feed) According to Commission Regulation (EC) No 333/2007 has to be included in the calculation of the test result.

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IMEP 24 Heavy metals in toys (EN 71-3:1994)	Taking into account the dispersion of the results and the differences between the results of the reference laboratories the stated uncertainties from the laboratories seem too small. In the test report the application of the correction factor was not always stated, as required.	To be addressed in the audit: In the audit of the method should be focused on the estimation of the measurement uncertainty. Possibly the reported uncertainty is estimated from the measurement only without taking in account the sample preparation step In the test report it must be made clear if the correction factor according to EN 71-3 was applied or not when reporting the results.
IfEP 1013 Metallography	Test results are critical for personal competence. Calibration of automatic picture analysis equipment is critical in relation to results.	To be addressed in the audit: Competence of personnel evaluating in metallographic images
SYKE Calorific value	- The results of the emission factor (EF) results showed the need of improvements. PT showed that the common procedure for calculation of EF-values within the different laboratories in the EU countries is urgently needed. - Analytical moisture and calorific value should be measured at a same time (within a few hours), if the gross calorific value is analyzed from air dried sample. Analytical moisture has a great effect for calculation the gross calorific value as a dry weight basis.	To be addressed in the audit: - calculation of emission factors - contemporary measurement of moisture content and calorific value
All PTs asking for measurement uncertainty	- Based on the reported measuring uncertainties it is evident that harmonization in the estimating of uncertainties should be continued. Some of the laboratories didn't report the uncertainty statement. EA labs should make effort to evaluate and report uncertainties.	To be addressed in the audit: Estimation and reporting of measurement uncertainty

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IfEP 1102 Rockwell Hardness	Considerable number of results is lower than the certified reference value. Reason: 1. The depth measurement system is critical for wear and un-cleaned surfaces. This leads to lower hardness values. 2. The scale of MPA NRW for the certified reference material was shifted to PTB – Scale in 2009. Laboratories not adopting the “new” scale are expected to report lower values.	To be addressed in the audit: Laboratories and assessors shall check the scale used and look for specific problems of the depth measurement system (conditions of the specimen surface)
IMEP 31 Total arsenic, cadmium, copper, lead and mercury, as well as extractable cadmium and lead in mineral feed	<ul style="list-style-type: none"> - the applied technique seemed to have an influence on the results of all measurands. ICP-MS and HGAAS had the better results - Inappropriate choice of reference material - The analytical methods were not adjusted to the inorganic test material 	To be addressed in the audit: <ul style="list-style-type: none"> - verification of the analytical methods used - reference material should be inorganic as the sample analysed -adjustment on inorganic matrix
IMEP 33 Total cadmium and lead in baby food at a very low mass fraction level	<ul style="list-style-type: none"> - the results were characterized by a high number of "less than" values. This means that many labs have difficulties measuring at these low mass fraction levels, because mass fractions are close to many laboratories' limit of detection - overestimated values especially for lead very likely due to potential contamination, expected at such level of concentration - visible method influence in the case of lead (methods based on atomic absorption were not sensitive enough to attain such low limits of detection) - experience of staff also seems to have an impact on the quality of the results for Pb - participants tended to underestimate the uncertainty 	To be addressed in the audit: <ul style="list-style-type: none"> - verification of the analytical methods used - estimation of measurement uncertainty - competence of personnel

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IMEP 26	Bimodal distribution, number of labs were right and number of them get only 50%. MU was not given.	<ul style="list-style-type: none"> ○ Grinding the test samples <ul style="list-style-type: none"> ○ The extraction technique used (either Soxhlet or ultrasonic), ○ The use of short GC column (10-15 m) ○ The use of GC-MS (as opposed to GC-ECD or HPLC-UV-FLD), ○ Carrying out this type of analysis regularly, ○ Taking part in interlaboratory comparisons for this type of analysis <p>The most influencing parameters on an unsatisfactory value (clustered around the low modal distribution are;</p> <ul style="list-style-type: none"> ○ The use of long GC columns (30-50 m) ○ The use of static extraction <p>Furthermore, the majority of the participants using isotopically labelled internal standards, got a satisfactory z-score.</p>
IMEP 33 Total cadmium and lead in baby food at a very low mass fraction level	<p>- the results were characterized by a high number of "less than" values. This means that many labs have difficulties measuring at these low mass fraction levels, because mass fractions are close to many laboratories' limit of detection</p> <p>- overestimated values especially for lead very likely due to potential contamination, expected at such level of concentration</p> <p>- visible method influence in the case of lead (methods based on atomic absorption were not sensitive enough to attain such low limits of detection)</p> <p>- experience of staff also seems to have an impact on the quality of the results for Pb</p> <p>- participants tended to underestimate the uncertainty</p>	<p>To be addressed in the audit:</p> <ul style="list-style-type: none"> - verification of the analytical methods used - estimation of measurement uncertainty - competence of personnel

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IMEP 34 Trace metals in toys II according to EN 71-3:1994	1/3 of the laboratories failed in assessing compliance to the two directives 88/378/EEC and 2009/48/EC and about half of the participants used the wrong migration limit from the new directive. It is not always the responsibility for the laboratory to report the statement of compliance, but they are supposed to be familiar with the legislation.	<p>The poor outcome of results from zeta-scoring compared to the generally good z-scores, reflects problem with estimation of measurement uncertainty.</p> <p>Even if not all laboratories are responsible for assessing compliance with the regulation, they are supposed to be familiar with the legislation.</p>