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Evaluation of performance of handheld XRF analyzer and possibilities for use in quantitative assessment

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Introduction

IKEA offers home furnishing solutions for every room in the home. The products must be safe for customers and shall have the correct quality. This is secured by product specifications which are legally binding contracts with suppliers.

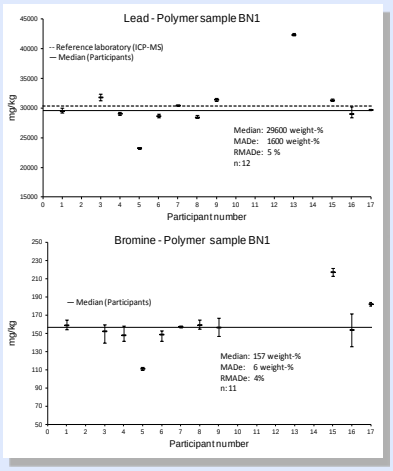
The requirements can be based on safety, health, quality and environmental concerns and secures that IKEA products comply with legal requirements and IKEA's voluntary requirements.

The chemical requirements specify limit values to be met, the test methods to be used and the frequency of testing. These verifying tests are performed at approved laboratories and often with time consuming and destructive methods. In addition to these mandatory tests, IKEAs purchase organization located at sites around the globe is performing chemical testing using handheld portable X-ray fluorescence (XRF) analyzers.

Objectives

•Perform initial proficiency test round of the XRF-methods within the IKEA group.

•Evaluate the measurement uncertainty and capability of handheld XRF as analytical method in laboratory.



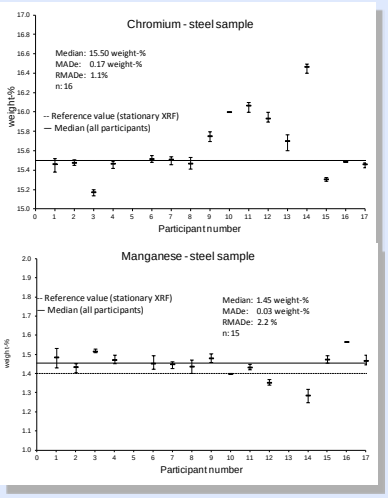
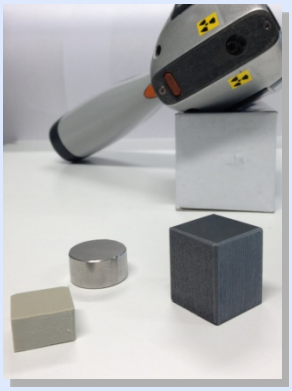


Fig. 1 Data from proficiency test. *Left:* Pb and Br in plastic sample. *Right:* Cr and Mn in steel sample. Outliers, outside the range of ±50% of the median were excluded. Robust statistics, median and MADe (median absolute deviation) were used.



Instrument and proficiency test materials. **The X-ray fluorescence (XRF) instrument** has both a radiation source and a detector behind the small measurement window. When atoms in the sample are hit by radiation - electrons are ejected to higher energy levels. When these vacant positions are filled by electrons from higher levels characteristic fluorescent radiation is emitted. This emission is measured by the instrument.

Background

XRF within purchase organization
With portable XRF-instruments located at different sites near the producers, it is possible to screen many samples and if necessary act immediately. Relevant material should always be tested when the formulation and or supplier is new to IKEA. Spot checks are performed regularly on prioritized materials. The results of XRF-testing are regarded as preliminary. If suspected incompliance is observed, further testing is made.

Examples of prioritized materials are red, yellow, green and dark coloured plastics, galvanized steel, brass and expanded polystyrene. Other materials, that can be tested, are leather, textiles, paint and foams.

When evaluating results, any significant level of for example lead or cadmium in polymeric materials will lead to action. When chlorine or bromine is observed, the assessment is more difficult. The result can indicate banned halogenated flame retardants. But other substances with these elements are allowed. High concentration of chlorine is a clear indication of PVC plastic which is banned in most product types.

XRF in the laboratory
IKEA Test Lab is primarily testing for product and material development, but can also be consulted for investigations and spot testing. In addition to the materials and issues handled by the purchase organization, testing for securing quality of stainless steels is important.

Methods

Portable XRF-proficiency test
Two polymeric materials and one steel test material not intended for IKEA products were selected, cut into pieces and tested for homogeneity. Samples were distributed to 17 participants, including three analytical test laboratories, one external. Six different instrument models from three manufacturers were included.

Estimation of measurement uncertainty
Uncertainty estimation were made for a number of metal elements based on within laboratory reproducibility and bias observed during long term measurements on certified reference materials (CRM).

Results and Discussion

Proficiency test
All participants submitted results. Larger variation in performance is observed for the plastic samples. In general the results is satisfying, a majority of the participants report the relevant elements. Lead, present in very high concentration in one plastic, was reported by all except two participants, three values were outliers. The agreement with the ICP-MS analysis was good. Bromine was not reported by several participants although present in relevant concentration (Fig. 1). In the second plastic, for example antimony, was reported by almost all participants and a relative reproducibility of 7% (relative MADe) was observed.

When the XRF instrument is used as a screening tool to identify suspected samples for further test a large uncertainty in the reported values can be accepted. But, false negative results, *i.e.* when a element present, is not reported, cannot be accepted. It is important to find out, to what extent the observed false negative results are dependent on instrument type, handling of instrument and condition of instrument.

For the steel sample, most participants reported all expected compounds. The agreement with stationary XRF-instrument and the median of participant results are almost perfect for chromium (Fig. 1). For the other metals in concentrations 0.2-2 weight-% the relative difference to the stationary XRF is never more than 12%.

Measurement Uncertainty
The estimated uncertainties of analysis of chromium and manganese in steel (Table 1) is in the same range but higher than the reproducibility in the proficiency test, calculated as MADe ×2, manganese in the lower concentration range ±0.1 weight-% and chromium in the higher concentration range ±0.4 weight-%. Based on these comparisons the estimated uncertainties can be considered reasonable.

These uncertainties are two times or more the uncertainties observed in reports for similar analysis with stationary XRF. But, acceptable for some quantitative analysis performed at IKEA Test Lab and the method is useful for assessment of for example compliance with IKEA stainless steel requirements.

Table 1. Uncertainty of measurement, U (k=2) for analysis of manganese and chromium in metal samples based on data from CRM-measurements.

Concentration range	U, ± weight-%
1-10 weight-%	0.2
10-20 weight-%	0.5

The portable XRF-instrument can, as shown in photo, be connected to a test stand.

Conclusions

•The portable XRF is a useful tool for indicative test on various materials.

•Proficiency test activities will improve the quality of handheld XRF-testing.

•It is important to regularly verify instrument capability to detect target elements in relevant sample matrices.

•The portable XRF provide quantitative results with acceptable measurement uncertainty for metal samples quality assessment.

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