BAM – 6.8

08.05.2013

Information on the test material EDS-TM002 and the BAM software package „*EDX Spectrometer Test*“ for determination of the spectrometer performance

1. Introduction

Energy dispersive spectrometers (EDS) are normally advanced, well-engineered instruments of high operation reliability. Malfunctions are rather seldom but not to be ruled out completely. The causes for this could be e. g. a poor contact between the electronic components (particularly at older instruments) or a leakage in the detector window. Therefore, it is recommended to check periodically the EDS performance. For a testing laboratory accredited in compliance with ISO/IEC 17075 *„General requirements for the competence of testing and calibration laboratories“* such a periodical performance check is even mandatory (section 5.9).

Generally, the performance check consists of a re-calibration of the energy scale (in fact of the channel width of the multichannel analyzer) by means of the positions of the Kα-line of Cu or Mn and another line in the low energy range (or even of the zero-peak), and the quantification of an appropriate reference material. However, this approach may generate the following disadvantages:

- Line broadenings, which may indicate malfunctions of the electronics, are not distinguished.
- The result of the performance check depends on the selection of the reference material, whose X-ray spectrum is possibly insensitive to malfunctions.
- An icing of the detector becomes to be perceptible only if also soft X-ray lines (below 1 keV) are used for quantification of the reference material.
- Updates of the quantification software of the EDS manufacturers could hide a modification of the spectrometer parameters.

BAM offers the test material EDS-TM002, whose spectrum „reacts“ sensitively to malfunctions of the EDS. All the measurements necessary for the performance check of an EDS at an scanning electron microscope in compliance with ISO 15632:2012 „*Microbeam Analysis – Selected instrumental performance parameters for the specification and checking of energy dispersive X-ray spectrometers for use in electron probe microanalysis*“ can be carried out with this one specimen. It consists of a thick layer (of about 6 µm) containing the elements C, Al, Mn, Cu and Zr deposited on a silicon substrate (see Fig. 1).

The evaluation of the measurement is possible in principle with the software package available from spectrometer manufacturers. However, the evaluation of the EDS performance can be carried out in an easier and quicker way by means of the software package “EDS Spectrometer Test” offered (optionally) also by BAM together with the EDS-TM002 test material. The software requires that the detector is provided with a thin film window. For detectors equipped with a beryllium window the software is not applicable.

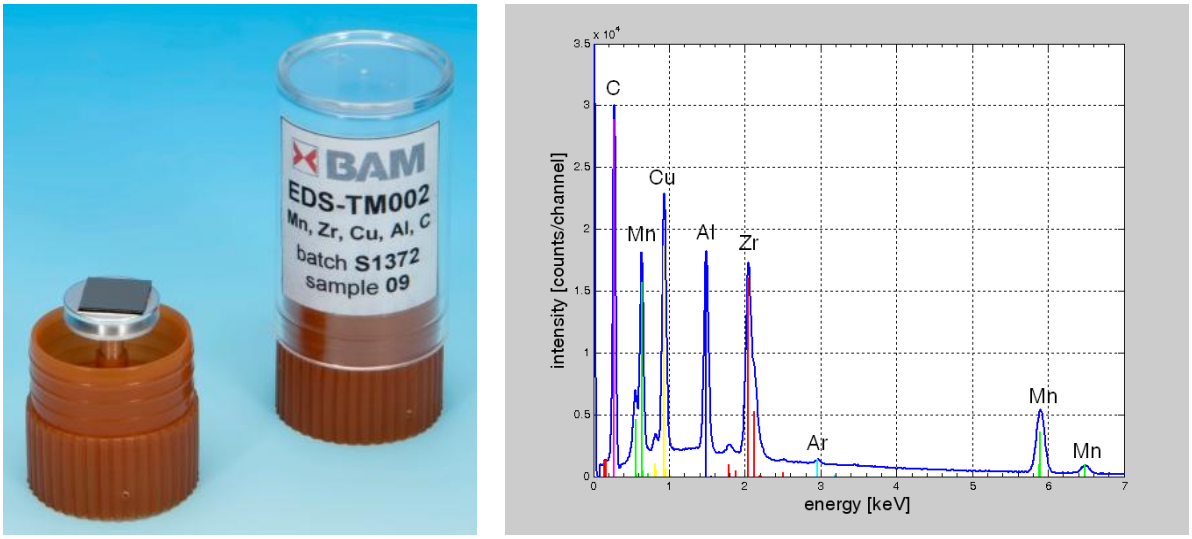

Fig.1 EDS-TM002 (left) and 10 kV spectrum (right)

2. Installation

On the CD accompanying (optionally) the EDS-TM002 test material the self-extractable programs can be found

1. *Performance_V34_pkg.exe*,

and

2. *MCRInstaller.exe*,

together with Info files (in German and English)

3. *Installation&Nutzung.pdf,*
4. *Installation&Operation.pdf,*
5. *Readme.txt*,

as well as

6. a few example spectra.

The installation takes place in two steps:

1. When *MCRInstaller.exe* is started, the *MATLAB Component Runtime* will be installed on *C:\Programme\MATLAB\MATLAB Component Runtime\v78*. The installation requires administrator rights on the corresponding PC. After installation one should make sure that the *PATH environment variable* was also completed accordingly (see indications in *Readme.txt*). When no longer needed, the *MATLAB Component Runtime* can be deinstalled like any other *Windows* application.
2. The file *Performance_pkg.exe* shall be copied into an user directory and then extracted by clicking it in *Explorer*. The new file *Performance_V34.exe* will be created. For this action no administrator rights are necessary.

For the case that a new program version shall be installed only the step #2 is necessary to be executed.

3. Running and setup of the program

By double-click on *Performance.exe* the program *„EDX Spectrometer Test“* gets started. During the extraction of the archive file a DOS window appears on the monitor. In dependence on the performance of the computer this can take a few seconds. Then the graphical user interface (as in Fig. 2) opens up. This extraction procedure will be executed every time when the computer was re-started.

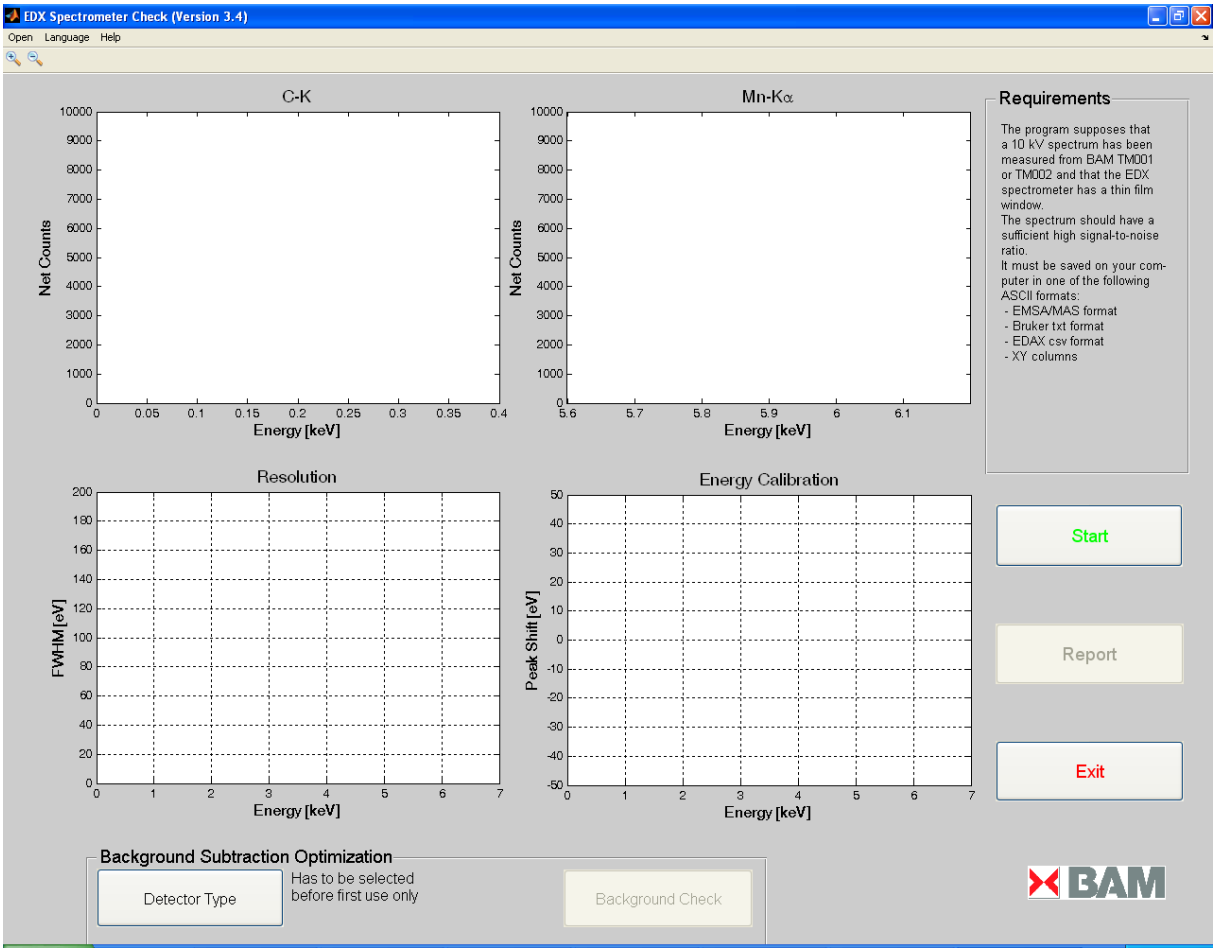

Fig. 2 Graphical user interface after starting the program

Because the program carries out a physical background subtraction the specimen elemental composition and the spectrometer efficiency must be known. The elemental composition of the EDS-TM002 is saved in the program. For specifying the spectrometer efficiency before first use it's necessary to select the detector type (Si(Li) with Ni front contact for *EDAX „Sapphire“* and *Oxford „Pentafet“*, Si(Li) with Au-contact for *Thermo-Noran „Pioneer“*, SDD for *Bruker „XFlash“* etc.). When clicking the button „detector type“ a window opens up for the corresponding selection (see Fig. 3).

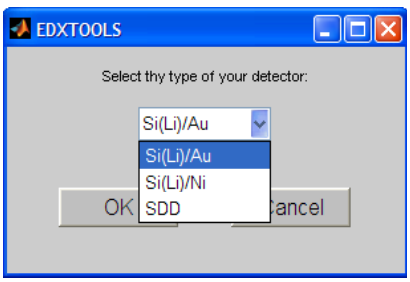

Fig. 3 Selection of the detector type. The window opens up by click on the button „detector type”.

4. Operating with the program

The first step of the performance check shall be the calibration of the energy scale. The EDS-TM002 must be mounted onto the SEM and the surface of the specimen shall be positioned to the working distance specific to the individual SEM/EDS system. The calibration of the energy scale must be carried out with the corresponding software available from the spectrometer manufacturer. For a calibration with Mn-Kα a SEM high-voltage of 20 kV is recommended.

The second step consists of the acquisition of the spectrum of the EDS-TM002 at 10 kV with the longest shaping time and its saving in a text format. The default is the *EMSA/MAS* format (*ISO 22029*), for which spectrometers from *Bruker/Röntec*, *Oxford*, *SAMx* and *Thermo-Noran* are provided with an export filter. Other file formats such as *.txt of *Bruker* or *.csv for *EDAX* spectrometers are also accepted. The spectrum shall be acquired until the height of the C-K Peaks reaches about 10.000 counts.

The third step implies the evaluation of the spectrum. This is automatically carried out by the software, after loading the spectrum (Fig. 4)

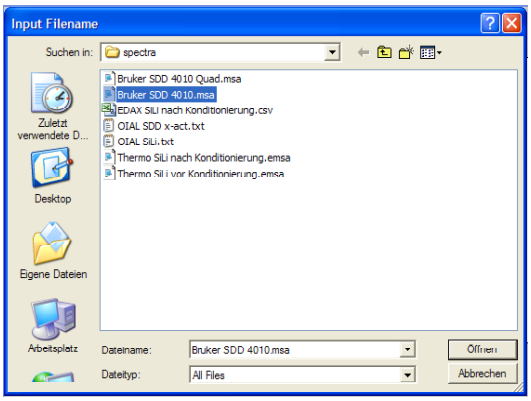

Fig. 4 Loading the spectrum by click on one of the fields under „Open“

The BAM software package for checking the EDS performance shows in the top two diagrams the full-width-of-half-maximum (FWHM) of C-K and Mn-Kα (see Fig. 5). They should be compared with the manufacturer specifications. Moreover, the Mn-Lα/Mn-Kα intensity ratio is provided as an indicator for a possible detector icing. For the case that the values of the Mn-Lα/Mn-Kα intensity ratio decrease steadily one should contact the service. Following values of the Mn-Lα/Mn-Kα intensity ratio corresponding to uncontaminated detectors in the common geometry of 0° tilt angle and 35° EDS port may be considered as typical:

Detector type	Mn-Lα/Mn-Kα
SDD, chip manufacturer #1	1.1±0.1
SDD, chip manufacturer #2	1.4±0.1
Si(Li) with Au front contact	0.8±0.1
Si(Li) with Ni front contact	1.3±0.1

The bottom left diagram shows the energy resolution of the EDS in dependence on the X-ray energy. The blue, continuous line was calculated from the FWHM of the Mn-Kα line. The points represent FWHM of the X-ray lines in the spectrum. An acceptable agreement should be expected. For the case of EDS-TM002 spectra acquired with various shaping times, the dependence of the spectrometer energy resolution on the selected shaping times can be derived from the bottom left diagram.

The bottom right diagram shows the state of the energy calibration. When all the points are located within the ±10 eV interval marked in green, this means that the calibration carried out in the first step was successfully.

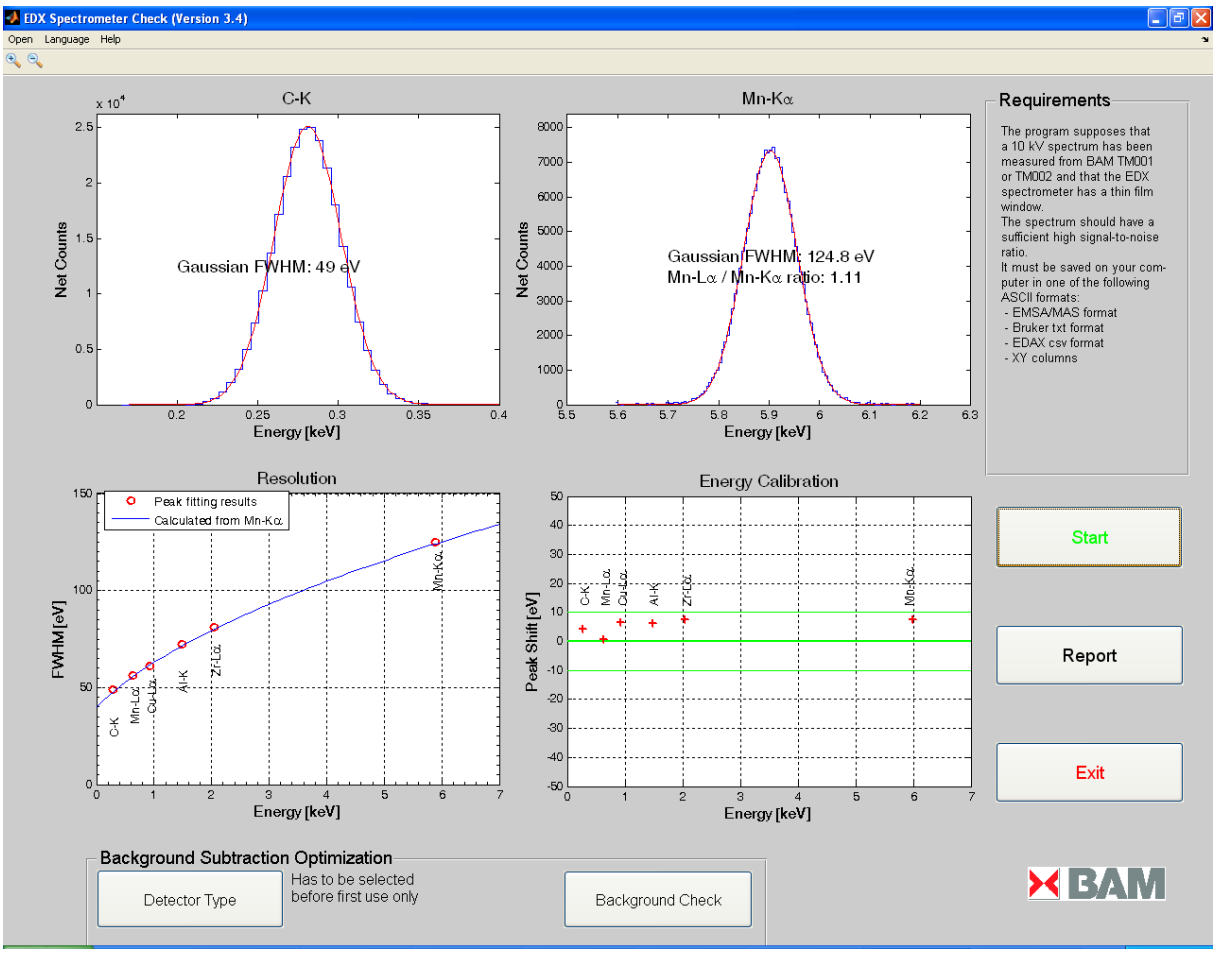

Fig. 5 Result of the EDS performance check

The possibility to check for the background subtraction carried out automatically by the program is offered by means of a click on the field “Background subtraction” (Fig. 6).

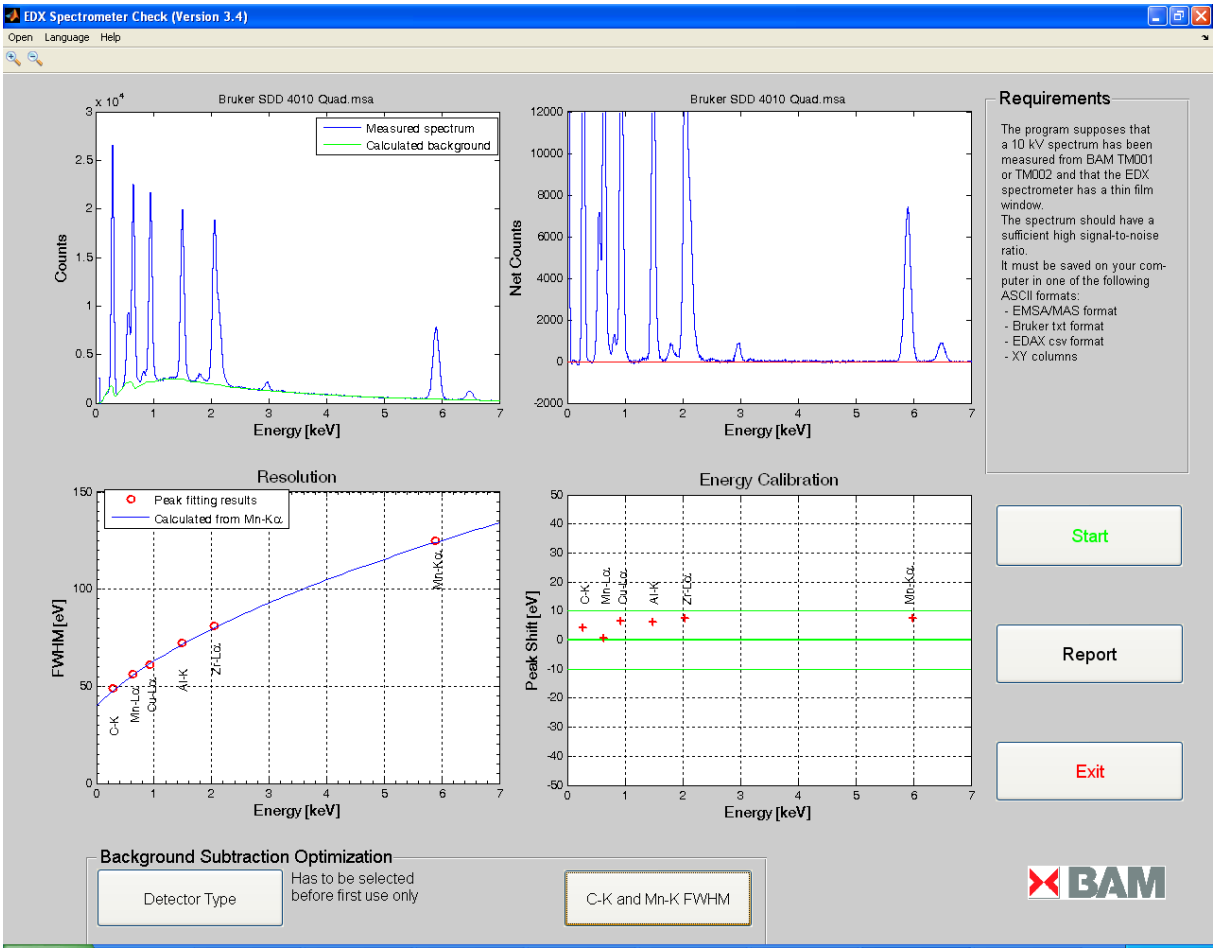

Fig. 6 Check for the background subtraction

The top left diagram shows the measured spectrum (blue) together with the calculated background (green). On the right hand it is represented the spectrum after background subtraction. Negative deviations under the red zero-line shall lie within the noise level.

The fourth and last step is the recording and archiving the results of the performance test carried out. By click on the button „Report“ a window opens up in which file name and path of the result protocol are requested. The accepted file formats are *.txt and *.rtf.

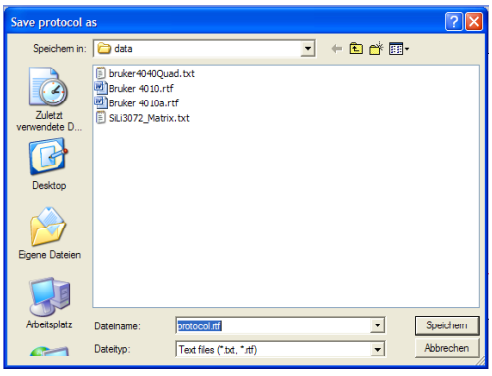

Fig. 7 Saving the results of the performance check as *protocol.rtf*

When saving the data as a text file (*protocol.rtf* in the example of Fig. 7) the result is also saved as a graphic, *protocol.png*. Moreover, all the four diagrams are copied into the computer clipboard. When *protocol.rtf* is opened with *Microsoft Office* the rtf-file is usually converted into a doc-file, in which the graphic from the Clipboard can be inserted. Thus, one gets text and graphic into only one document (see example on the next page).

By a click on the button „Exit“ the program „EDX Spectrometer Test“ will be closed.

Protocol example „*protocol.doc*“:

```
Protocol performance check, Date: 27-Jan-2013
**************************************************

Filename: Bruker SDD 4010 Quad.msa

FWHM Mn-Ka : 124.77 eV
Position Mn-Ka : 5.902 keV
Shift Mn-Ka:  7.47 eV
Intensity Mn-Ka : 1.950281e+005 counts

FWHM C-K   : 48.97 eV
Position C-K : 0.281 keV
Shift C-K  :  4.12 eV
Intensity C-K : 2.617881e+005 counts

Mn-La/Mn-Ka:  1.11
```