

Installation and operation of 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 17025 „General requirements for the competence of testing and calibration laboratories“, such a periodical performance check is even mandatory (cf. section 5.9).

Usually, 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 $K\alpha$ -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 hide malfunctions of the electronics, are not identified.
- 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 (SEM) 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 measured 10 kV EDS spectrum 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. Windowless SDD detectors have been recently tested successfully with the software version 3.6. For detectors equipped with a beryllium window the software is not applicable.

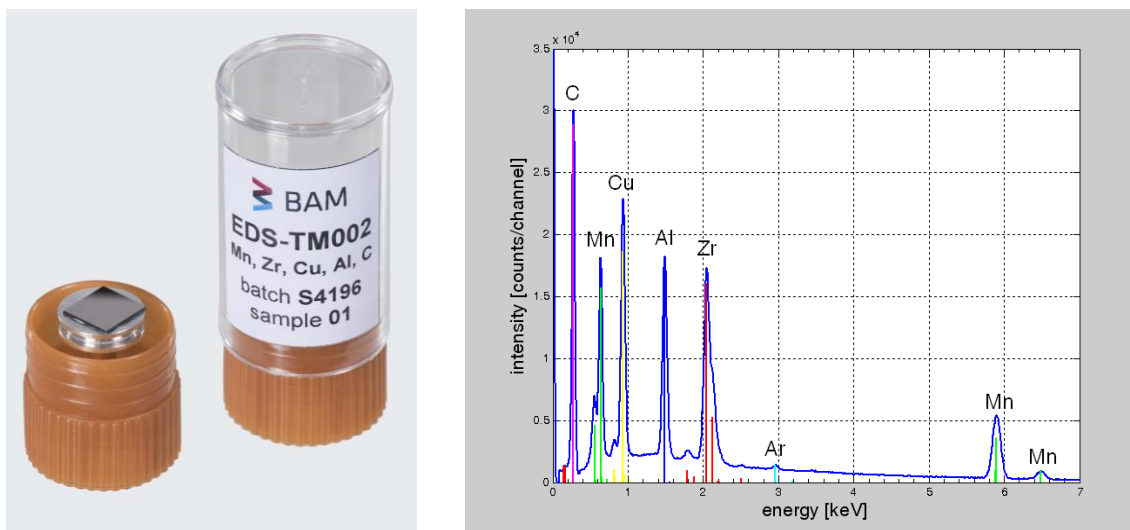


Fig. 1 EDS-TM002 with its 10 kV EDS spectrum excited at an SEM

2. Installation

On the CD accompanying (optionally) the EDS-TM002 test material the setup program can be found:

1. 

together with info files (in German and English)

2. Installation&Nutzung.pdf,
3. Installation&Operation.pdf,

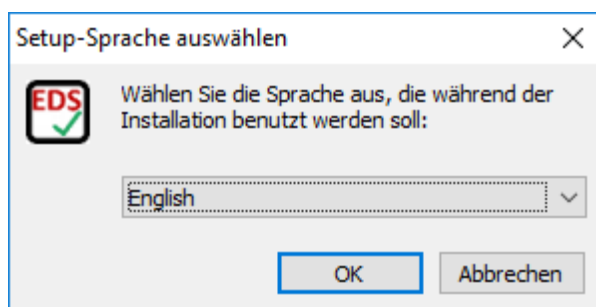
as well as

4. a few 10 kV EDS spectra of EDS-TM002 taken with representative EDS systems.

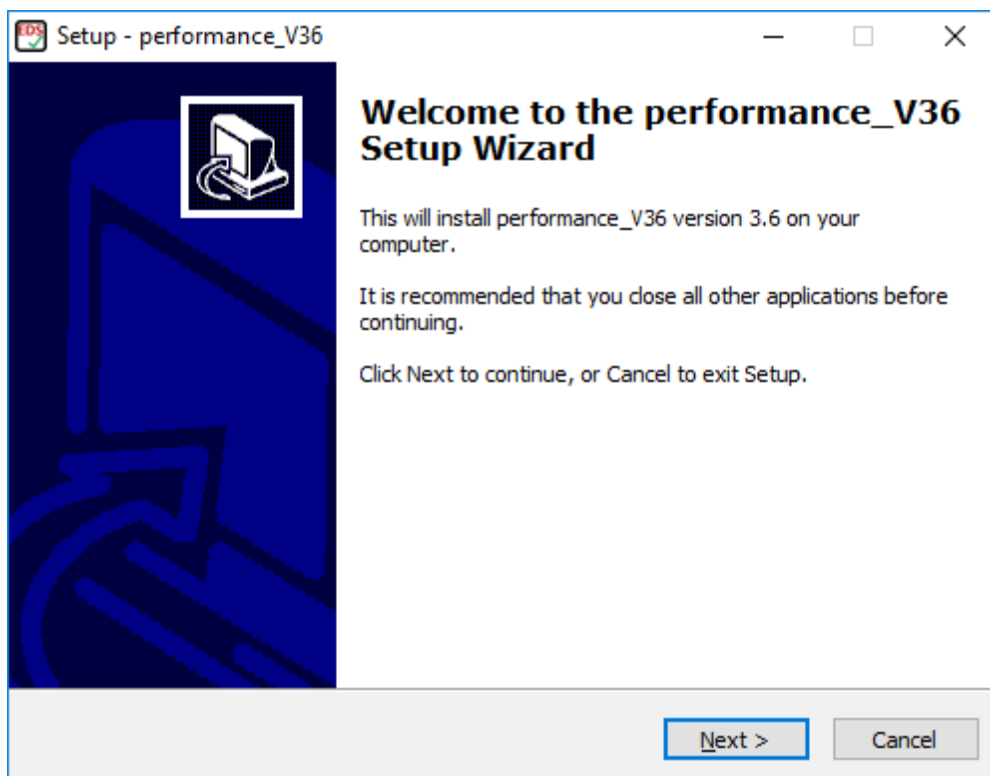
The installation takes place in a few uncomplicated steps as described in detail below. The installation of the setup recognizes and runs automatically on both 32 and 64 bits Windows systems without making necessary any intervention/decision of the operator.

Note that for the installation it is necessary to have administrator rights.

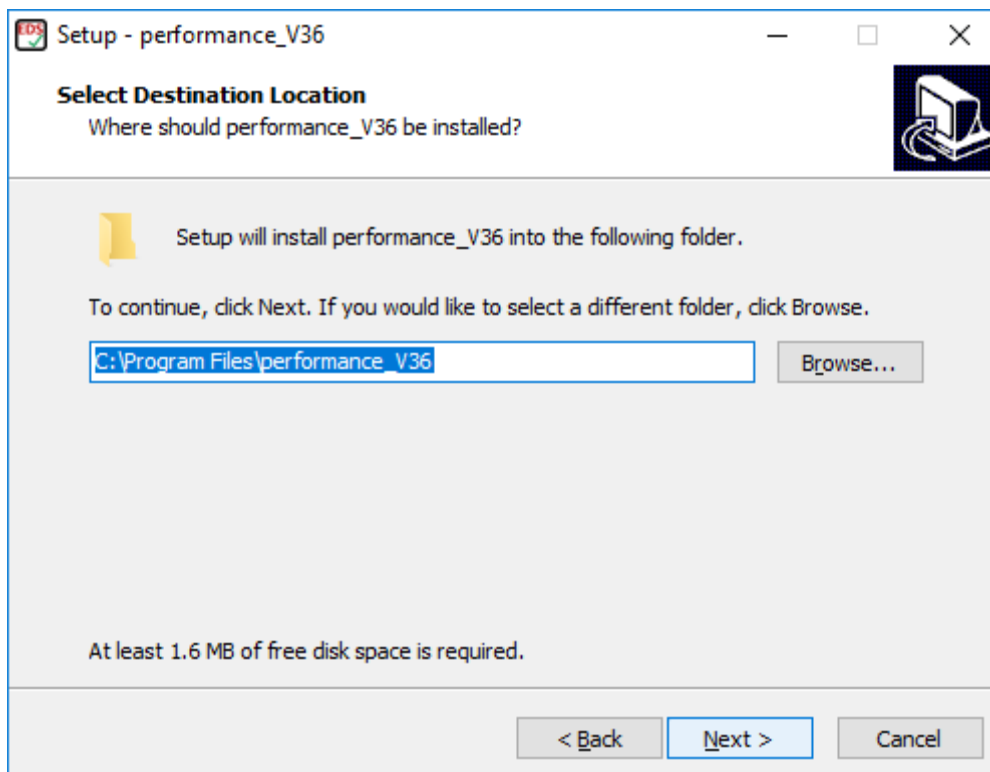
1. When setup.exe is started, the following window will appear where the user must select the language for installation steps:



2. A welcome window will follow:

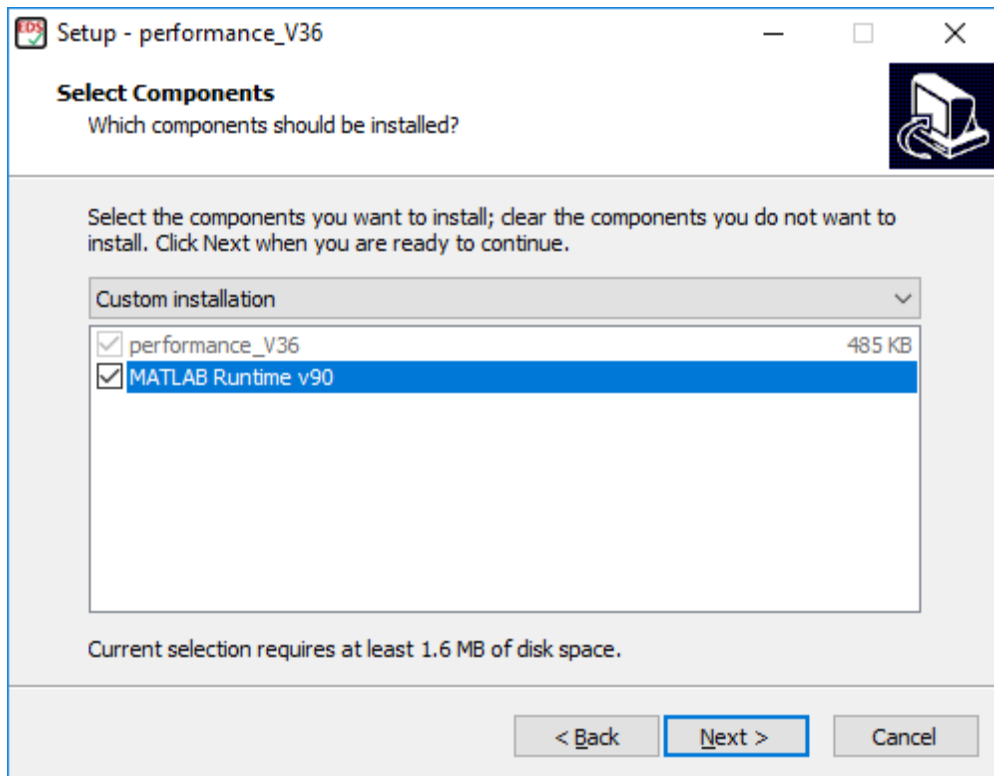


3. After clicking Next button, following window will appear asking for the folder where Performance_V36 shall be installed:



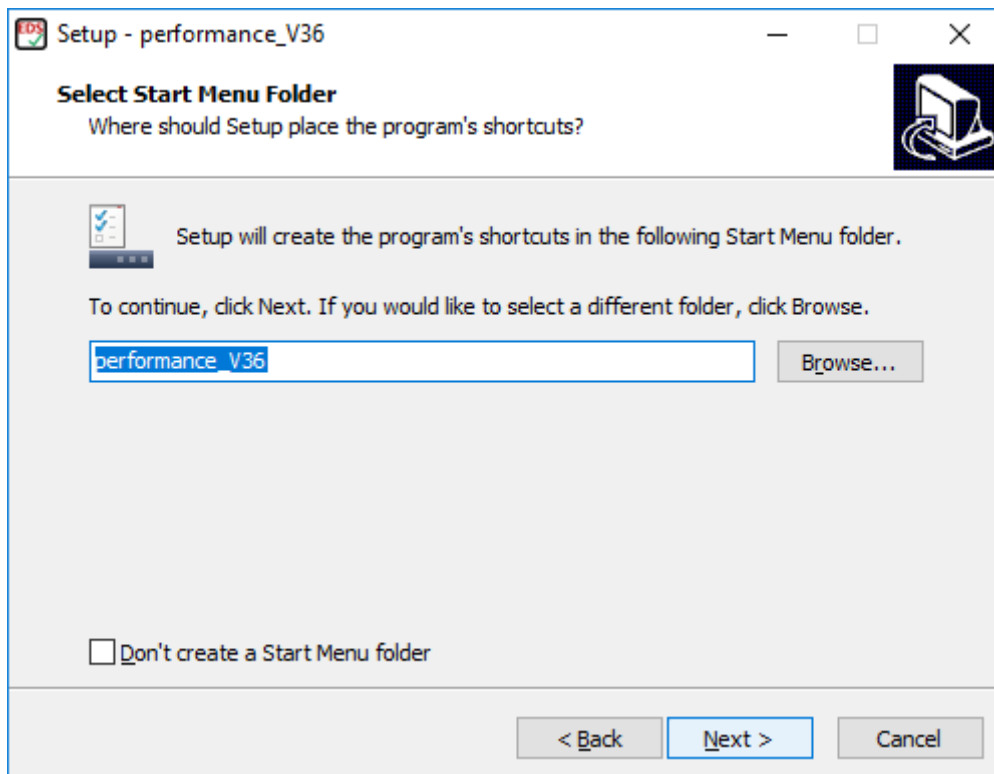
It is recommended to stay with the proposed path.

4. Next step is to select if MATLAB Runtime component shall be also installed additionally to performance_V36:



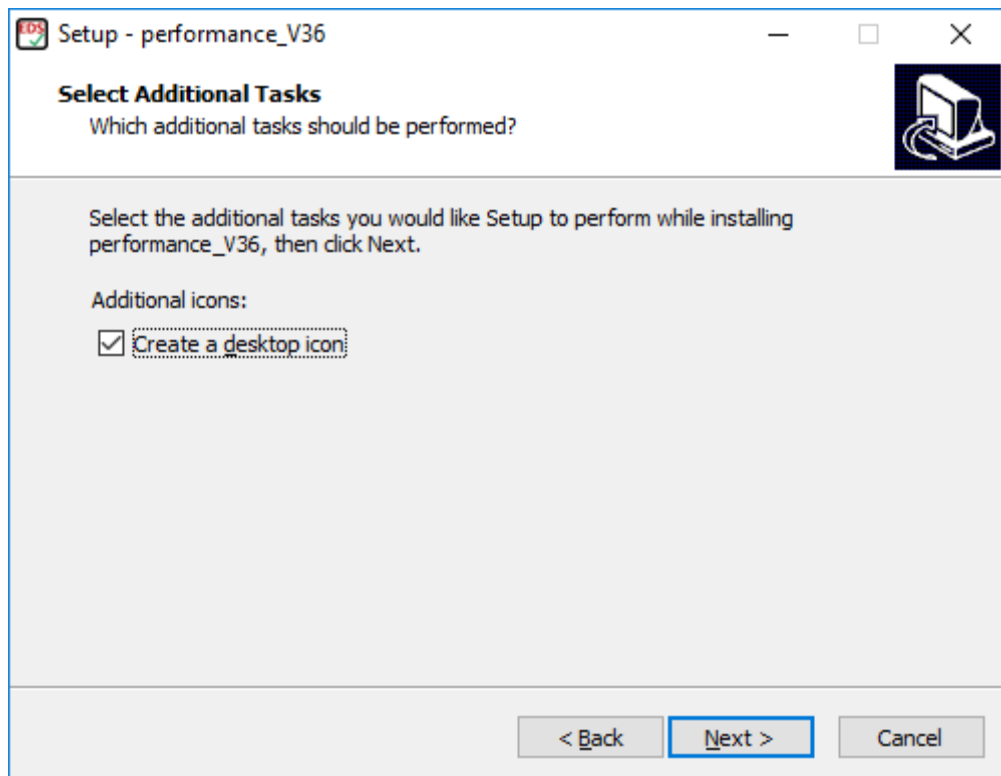
For those not having MATLAB software package installed on their PC it is necessary to select the installation of MATLAB Runtime v90.

5. Next window asks for the Start Menu folder:

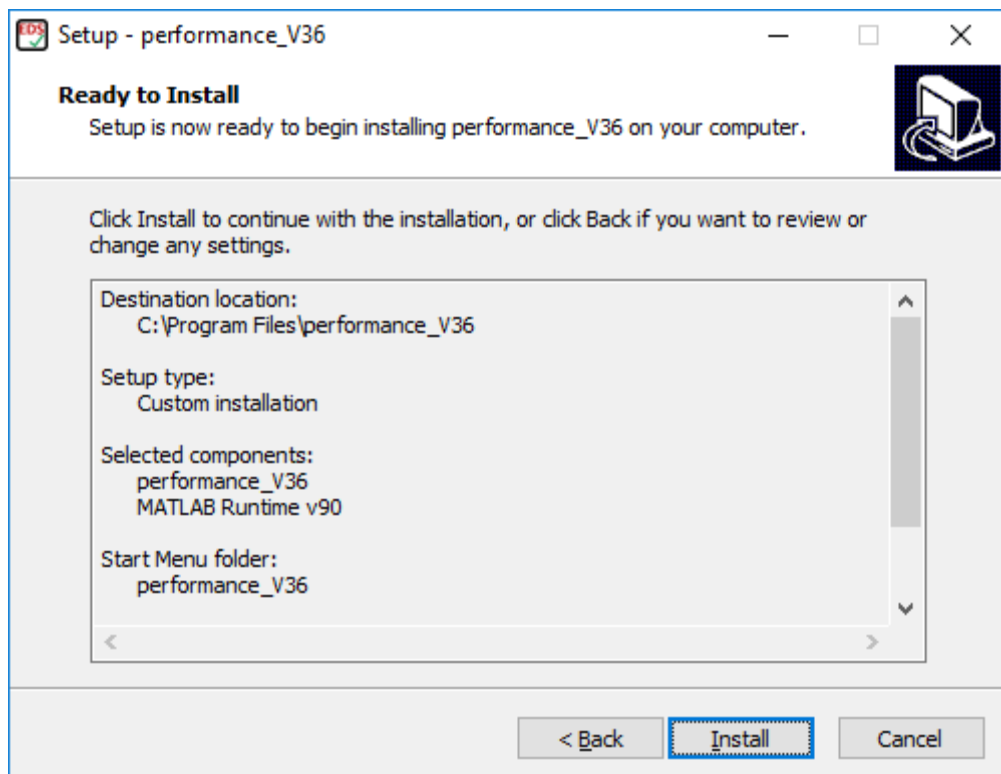


It is recommended to stay with the proposed folder name: performance_V36.

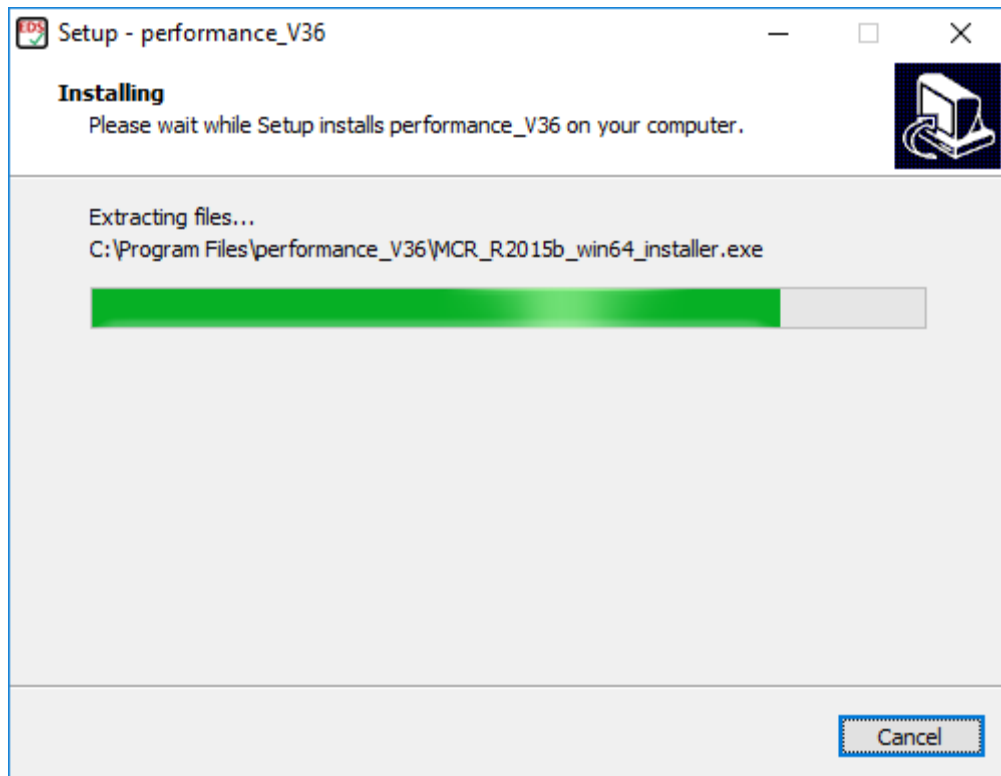
6. The possibility to create a desktop icon is offered by the next window:



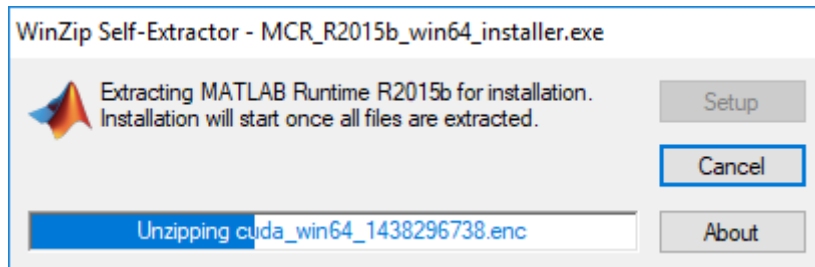
7. After clicking Next, the Setup is now ready to start installing the Performance application on your PC:



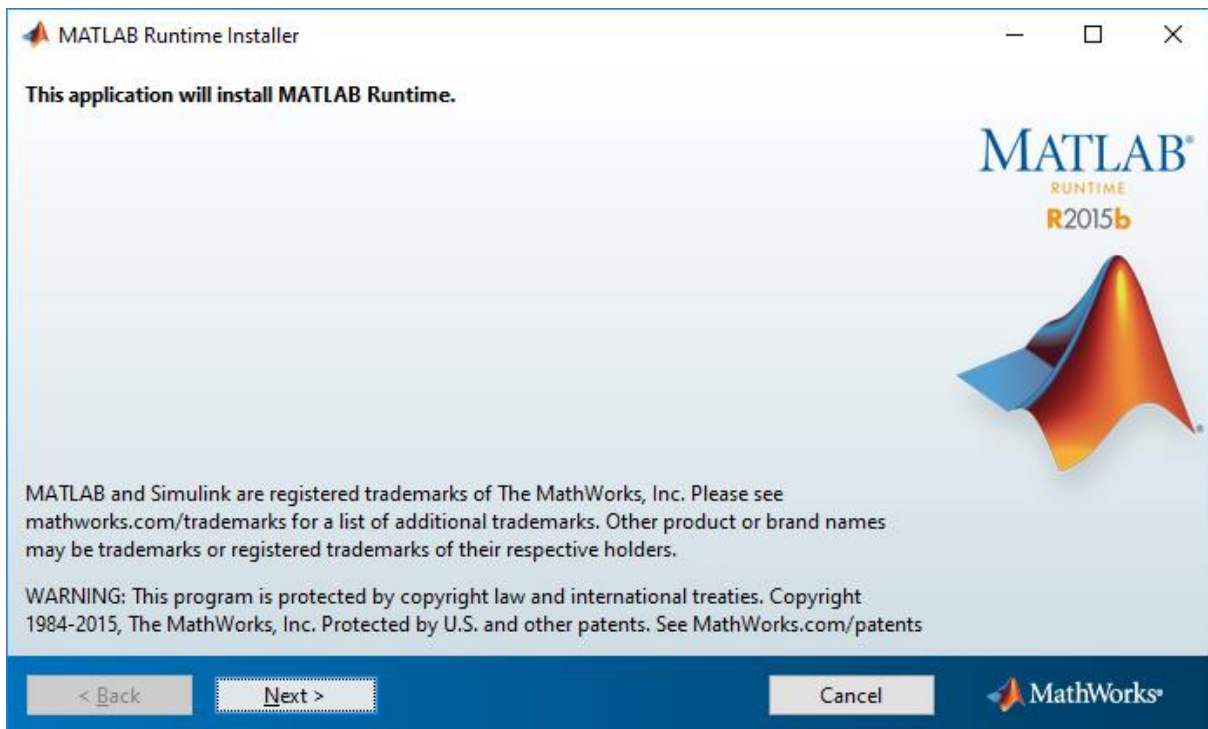
8. After clicking Install, following window will show the progress of the installation process. It should take about 1 min.



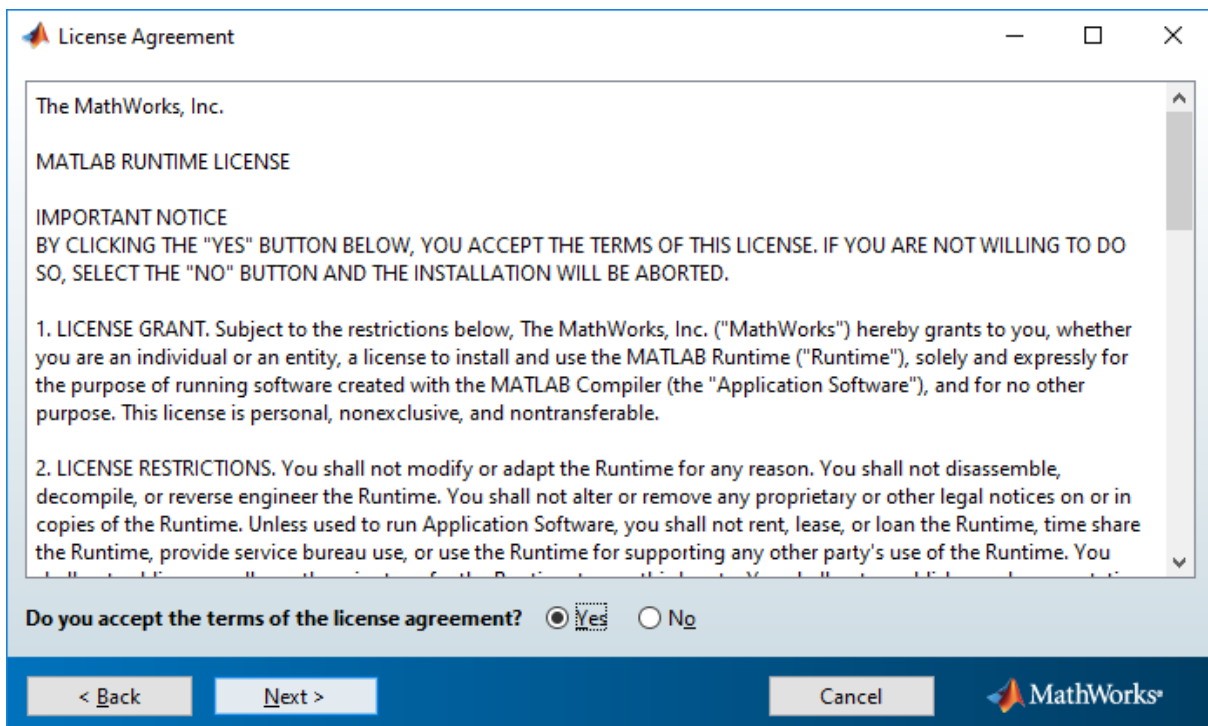
9. Further, the MATLAB Runtime Component is extracted (unzipped):



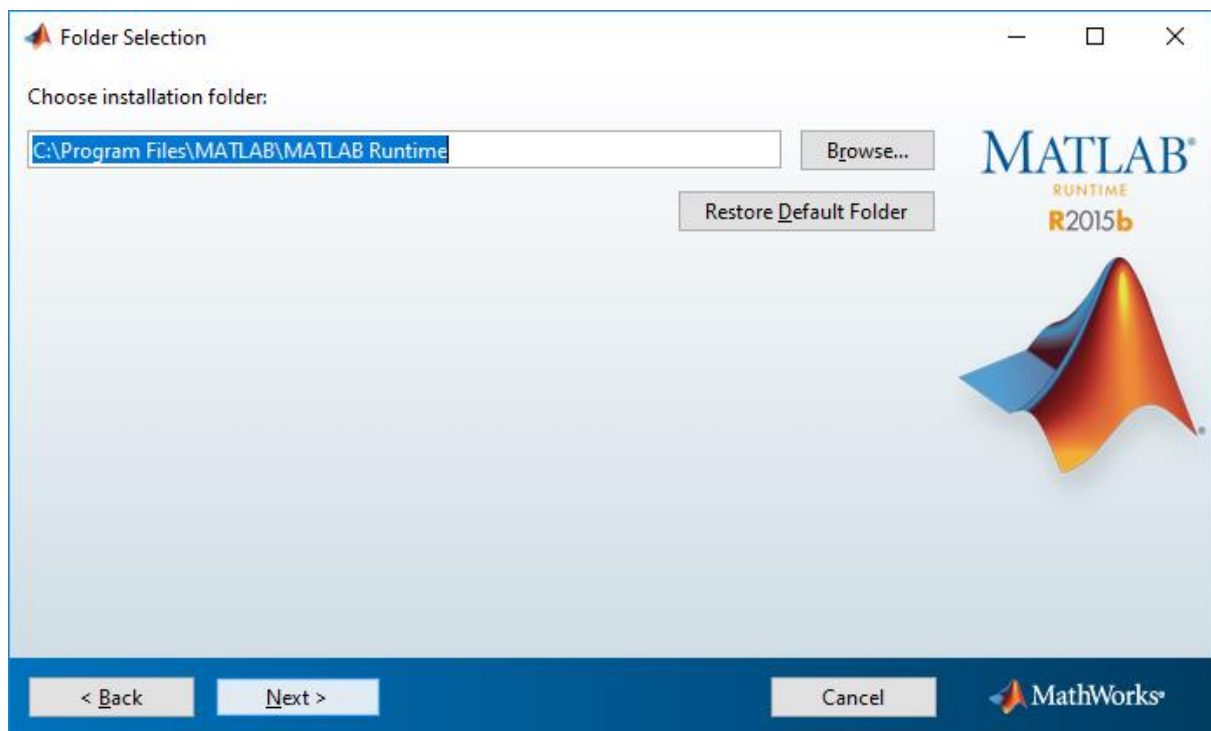
10. and then installed:



11. After accepting the terms for license agreement:

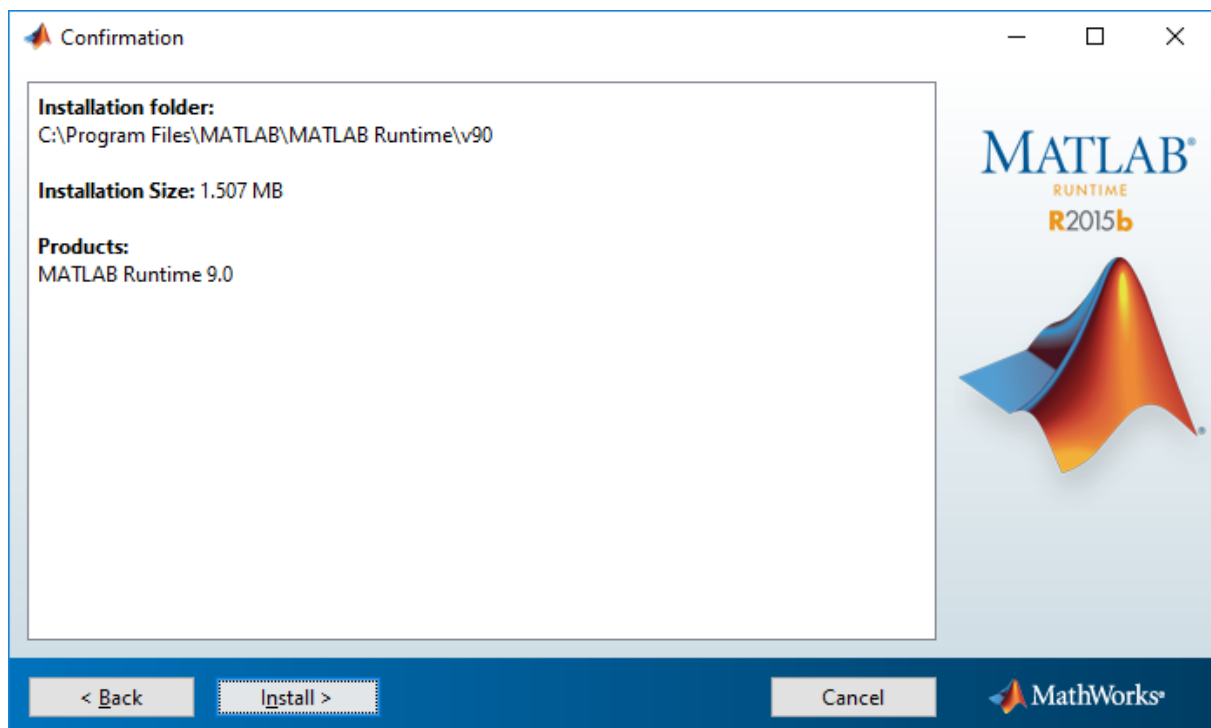


12. the folder for the installation of MATLAB Runtime component is asked to be chosen:

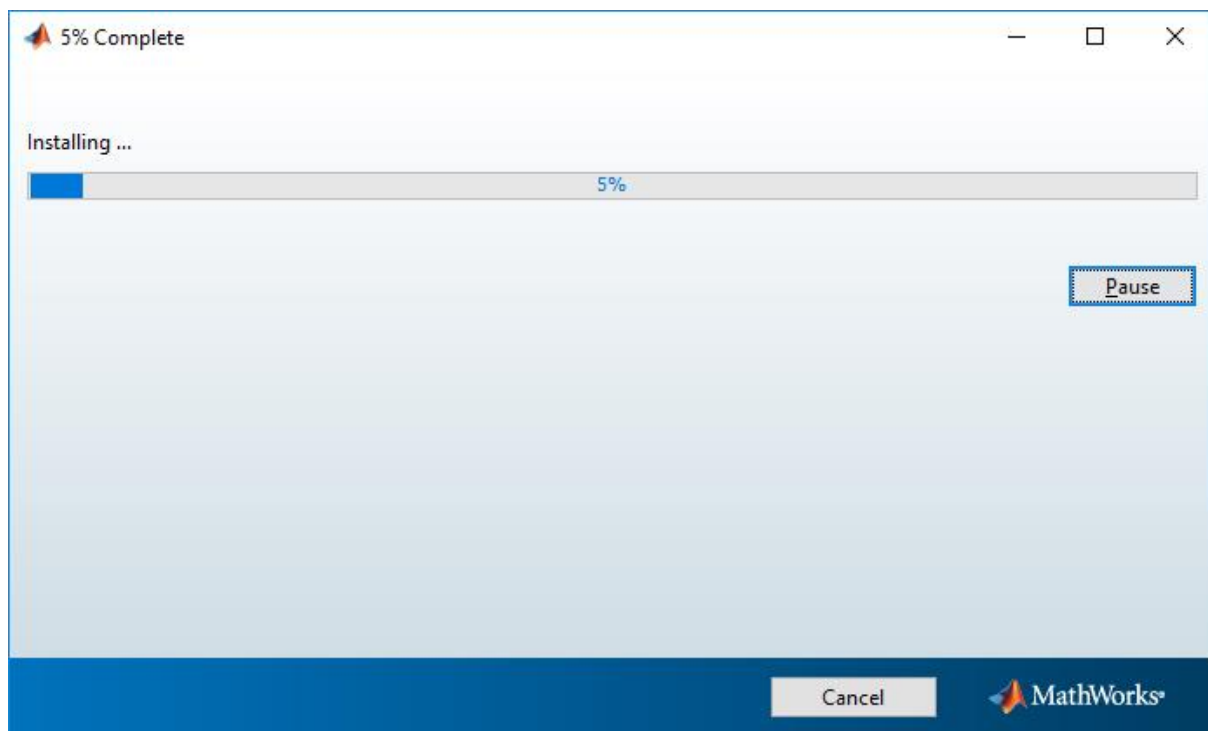


It is recommended to stay with the proposed folder.

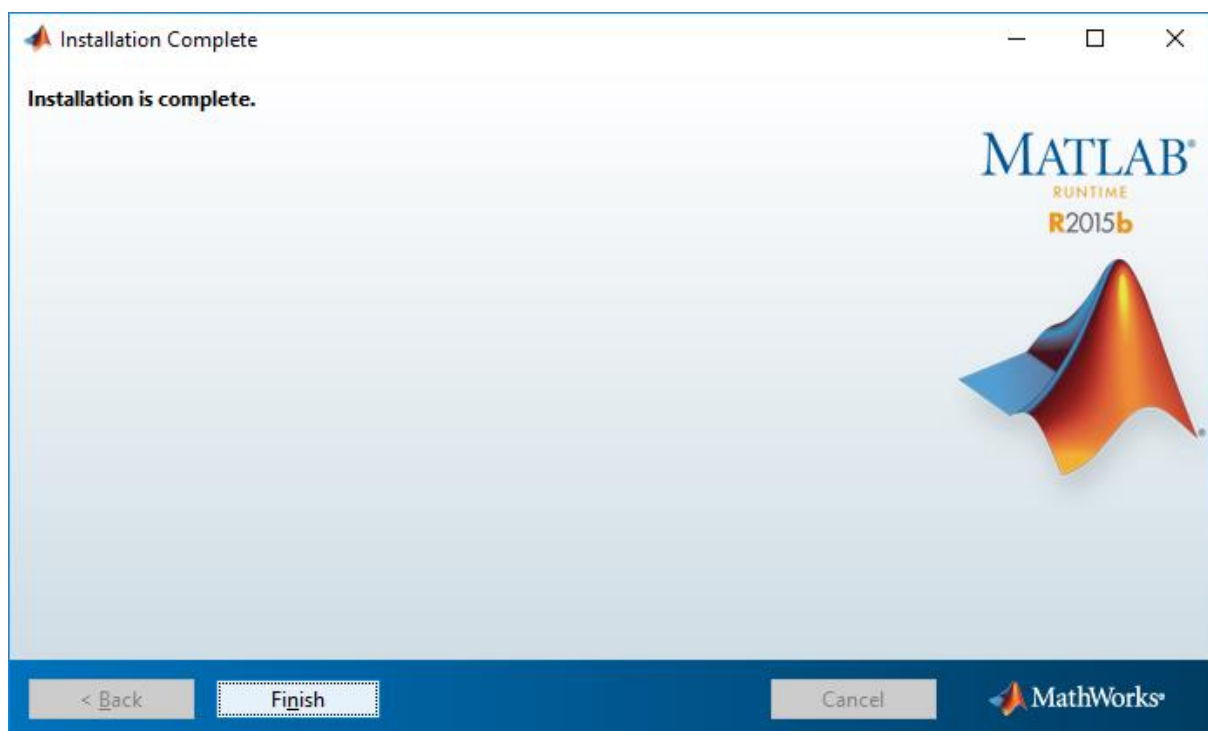
13. A confirmation window will follow:



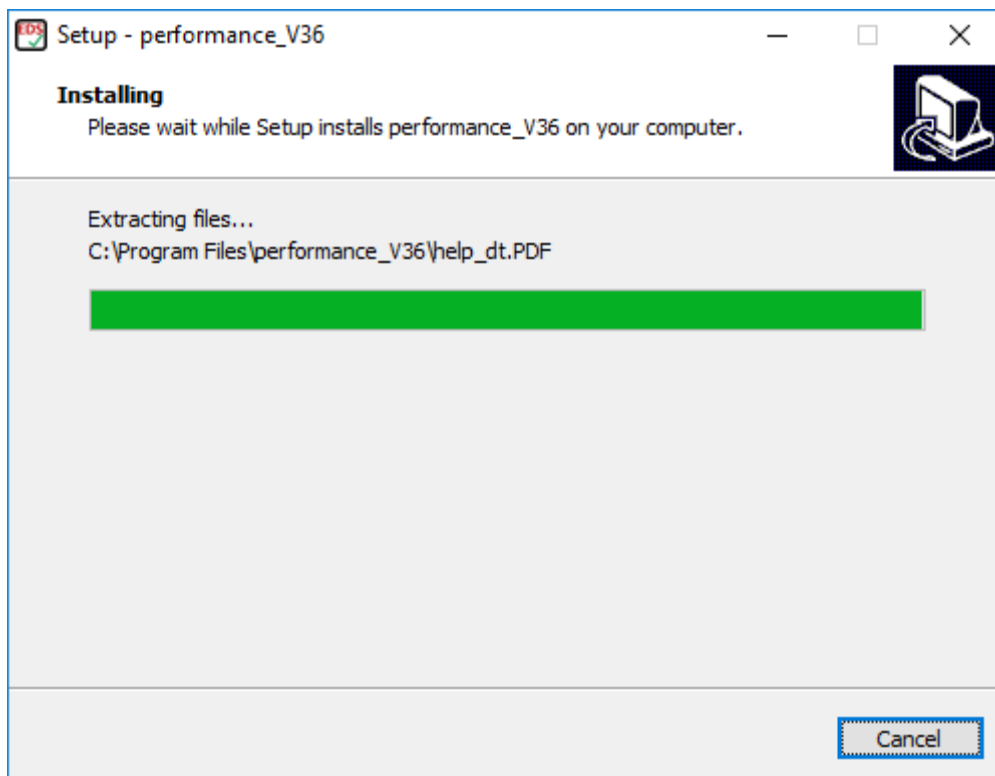
and installation will run for some minutes:



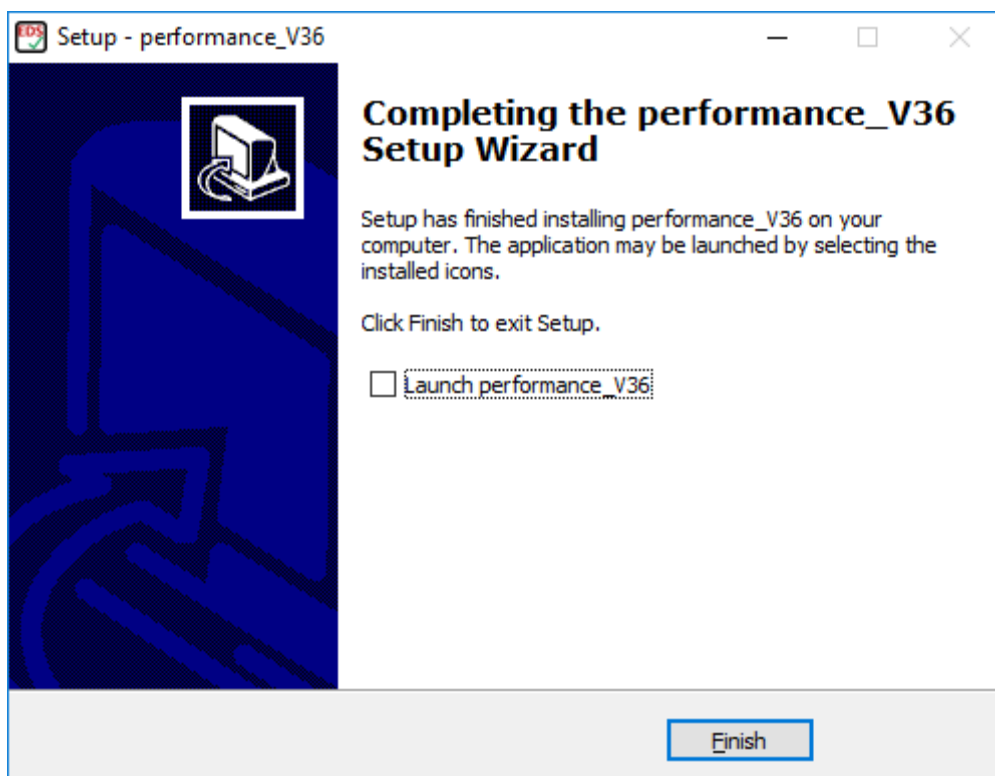
14. followed by the successful confirmation:



15. After pressing Finish button, the installation of performance_V36 program will follow (for about max. 1 min):



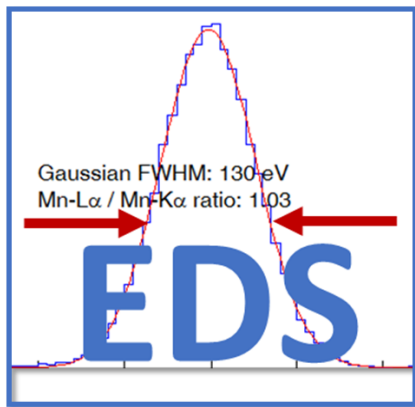
16. and the final confirmation of completing successfully the installation of performance_V36 will come:



For the case that an older program version is already installed (e.g. version 3.4), it is recommended to uninstall the older Performance program version as well as the MATLAB Runtime Component (v7.8) and install the new Performance_V36.exe as described above starting with step #1.

3. Running and setup of the program

By double-click on *Performance_V36.exe* (in Windows-Explorer or the related icon on the desktop) the program „EDX Spectrometer Test“ gets started. During the extraction of the archive file a start 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. This extraction procedure will be executed every time when the computer was re-started.

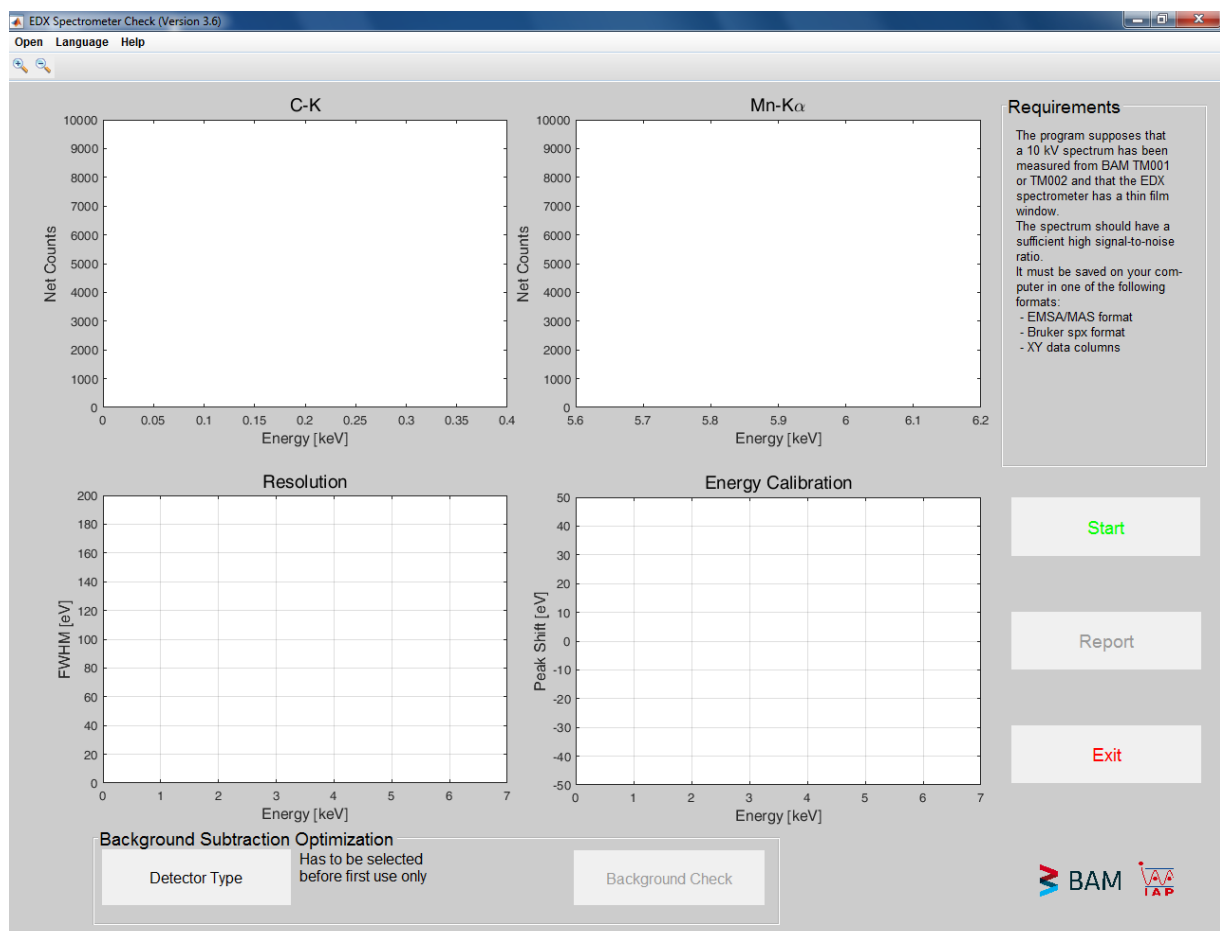


Fig. 2 Graphical user interface after starting the program

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 is necessary to select the detector type. Detector types are Si(Li) with either Ni or Au front contact, as

well as SDDs with Moxtek AP windows, Si3N4 window or windowless SDDs. In case the detector and window type are unknown, the spectrometer manufacturer shall be contacted. When clicking the button „detector type“, a window opens up for the corresponding selection (see Fig. 3).

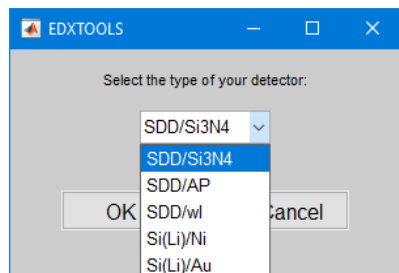


Fig. 3 Selection of the detector type. The window opens up by click on the button „detector type“.

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 in the SEM and the surface of the specimen shall be positioned at 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-K α 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 file formats that can be opened with EDX Performance Check are specified later.

The spectrum shall be acquired until the intensity of the Mn-K α peak - measured as net peak area! - exceeds 100.000 counts. When the counting statistics does not meet this criterion a warning message will appear (see Fig. 4). Note that non-consideration of this recommendation may affect significantly the results.

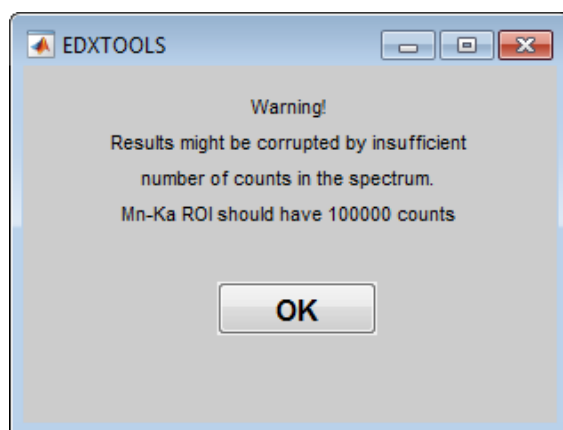


Fig. 4 Warning message for a spectrum with insufficient counting statistics.

The third step implies the evaluation of the spectrum. This is automatically carried out by the software, after loading the spectrum (Fig. 5). This takes place when clicking the START button.

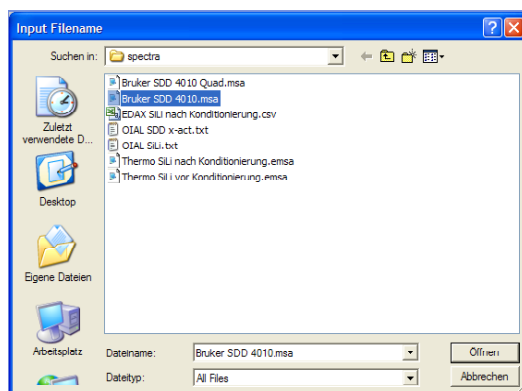


Fig. 5 Loading the spectrum by click on one of the fields under „Open“

By default, the software assumes the saved spectrum in the EMSA/MAS format independent of the extension (e.g. *.emsa, *.msa, *.mas, *.txt). Spectra in Bruker's spx-format or simply in two columns for energy and counts are also accepted. In these cases, the spectrum file must be opened via the Open button in the menu row (see Fig. 6). The default can also be set to Bruker-spx format, i.e. by clicking the START button an spx-spectrum is opened. This possibility was added because the export to EMSA/MAS is an option of Bruker's software, which is not available to all users.

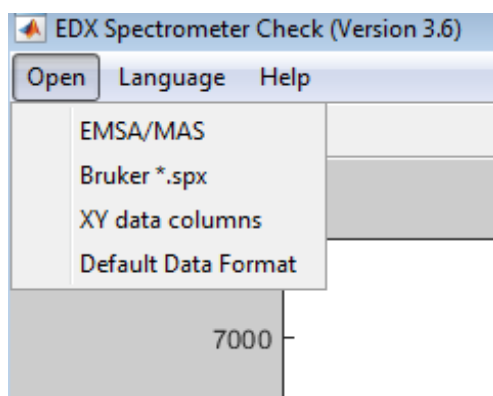


Fig. 6 Menu with the file formats that can be opened with EDX Performance Check

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-K α (see Fig. 7). They should be compared with the manufacturer specifications. Moreover, the Mn-L α /Mn-K α intensity ratio is provided as an indicator for a possible detector contamination. For the case that the values of the Mn-L α /Mn-K α intensity ratio decrease steadily one should contact the service.

The bottom left diagram shows the energy resolution of the EDS in dependence on the X-ray energy. The red, continuous line was calculated from the FWHM of the Mn-K α 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.

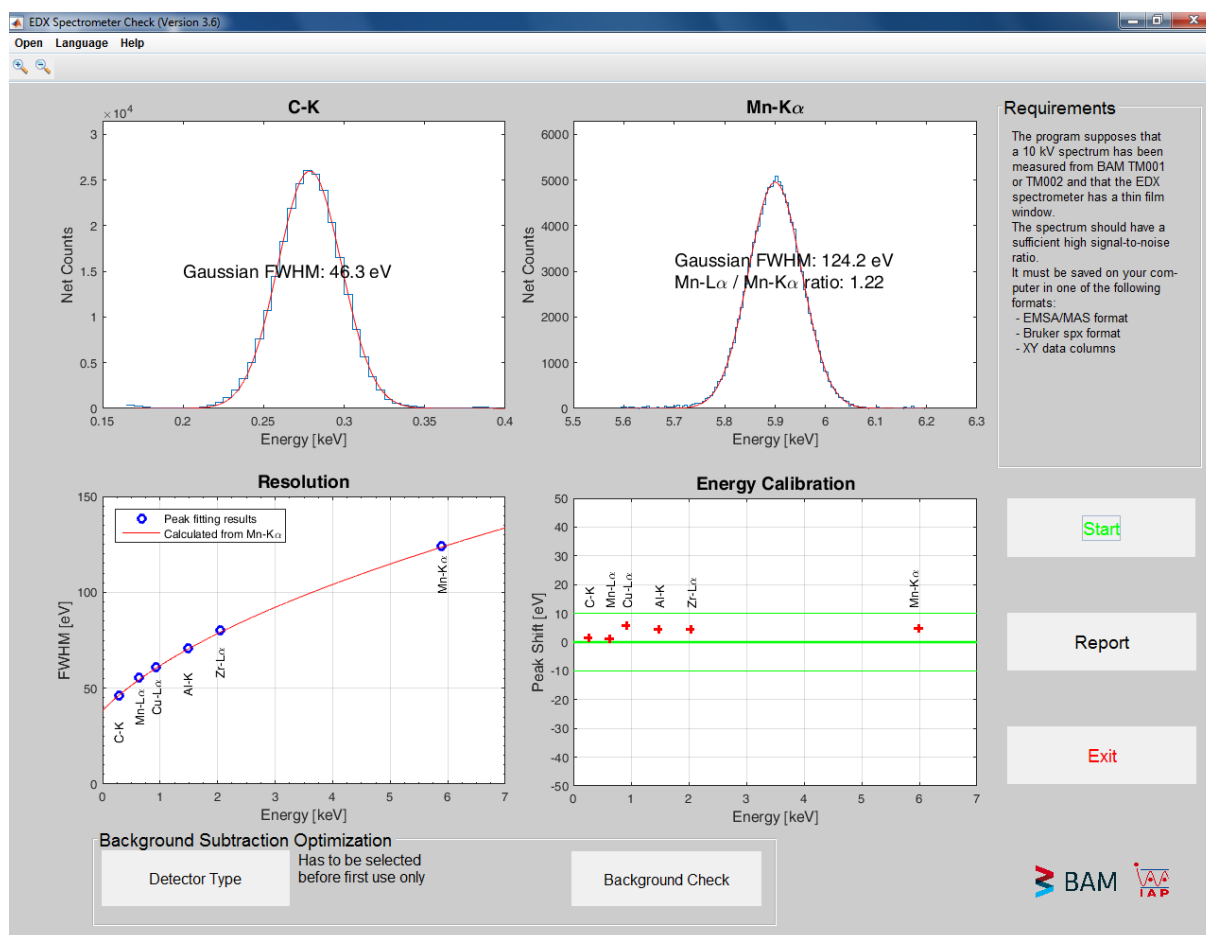


Fig. 7 Result of the EDS performance check

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. 8).

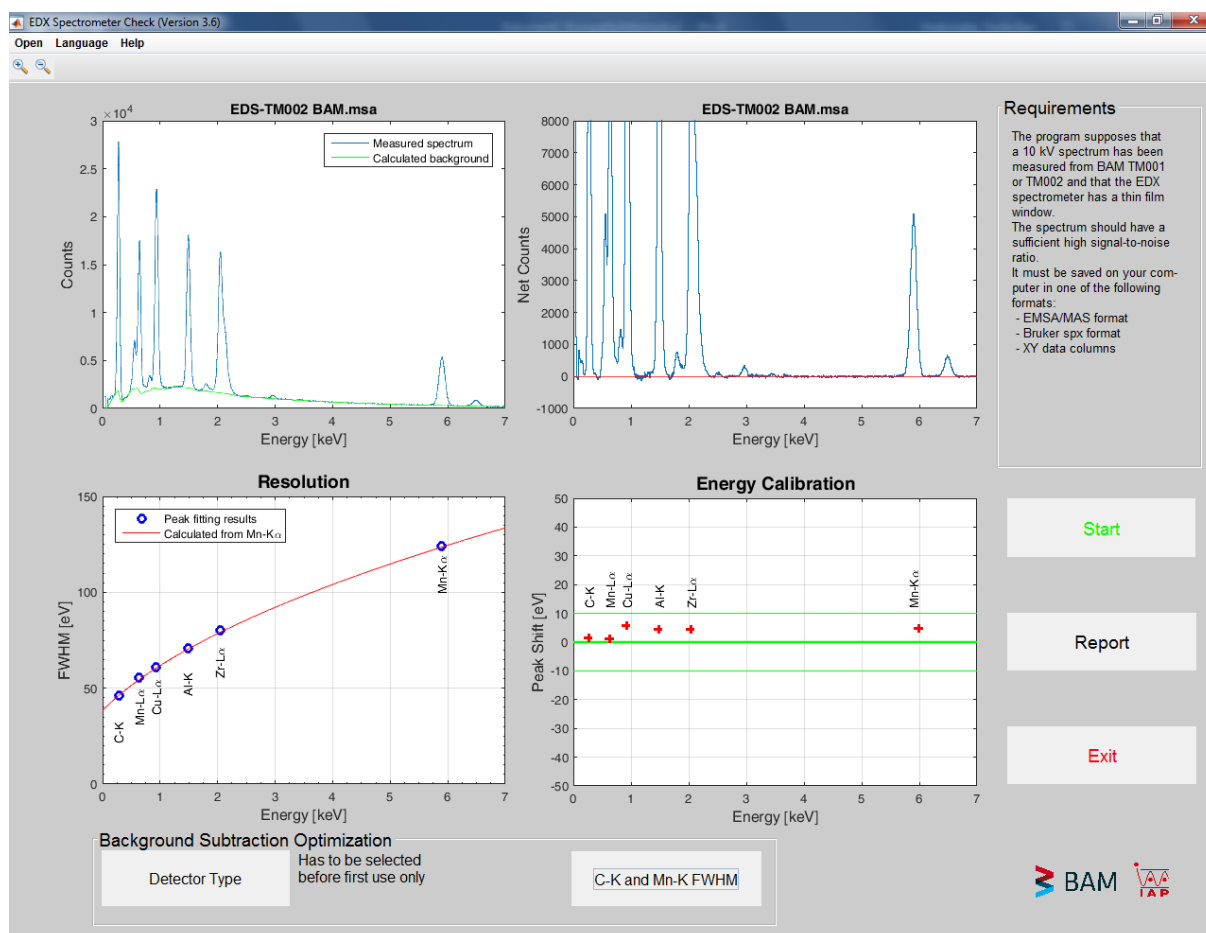


Fig. 8 Check for the background subtraction

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.

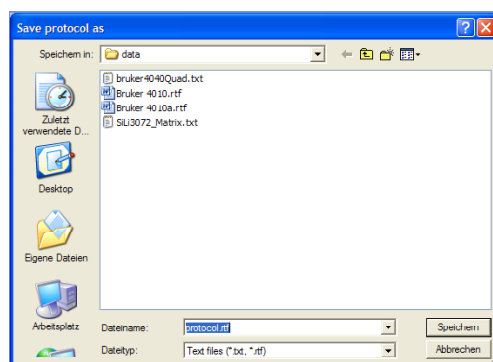


Fig. 9 Saving the results of the performance check as *protocol.rtf*

When saving the data as a text file (*protocol.rtf* in the example of Fig. 9) 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.docx“:

Protocol performance check, Date: 28-Sep-2018

Filename: EDS-TM002 BAM.msa

FWHM Mn-Ka : 124.19 eV
 Position Mn-Ka : 5.900 keV
 Shift Mn-Ka: 4.87 eV
 Intensity Mn-Ka : 1.311036e+05 counts

FWHM C-K : 46.32 eV
 Position C-K : 0.279 keV
 Shift C-K : 1.61 eV
 Intensity C-K : 2.567584e+05 counts

Mn-La/Mn-Ka: 1.22

