

**MODERNIZATION OF CHEMICAL ANALYSER EDS TO
SEM 525M MICROSCOPE**

TECHNICAL-MOTIVE DOCUMENTATION

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1. How it works.

1.1. Characteristic X-ray radiation

The characteristic spectrum is the result of the interaction between the electron beam and the electrons from the inside coat of the atoms in the sample. Usually it is the case that K and L coats with the atomic numbers higher than 10 are filled. If one of the K electrons is removed, the atom can 'experience' transmission, in which L or M electron 'jumps' into an empty space and the potential energy released during that process is emitted as a quantum of electromagnetic radiation. Typical values for such transitions are within the X-ray range of the electromagnetic radiation spectrum. Quantum energy is precisely specified for all the transition types in a given element and therefore it is the diagnostic feature.

X-ray spectrometer registers characteristic X-ray radiation. Its task is to calculate all the impulses from the X-ray radiation and to segregate them. There are two methods of doing X-rays spectroscopy:

- Energy Dispersive Spectrometry – EDS. It uses the detectors whose initial signal intensity is proportional to the impulse energy, in example: scintillation meters, proportional flow meters, Li/Si detectors.
- Dispersive Spectrometry – WDS. It uses X-ray spectrometer with analysing crystal.

1.2. Lines of characteristic X-ray radiation

In order to **make** characteristic X-ray radiation the gap in the inside electron coats of the atom is necessary. Particular series of lines can be distinguished dependently on the coat on which the gap appeared.

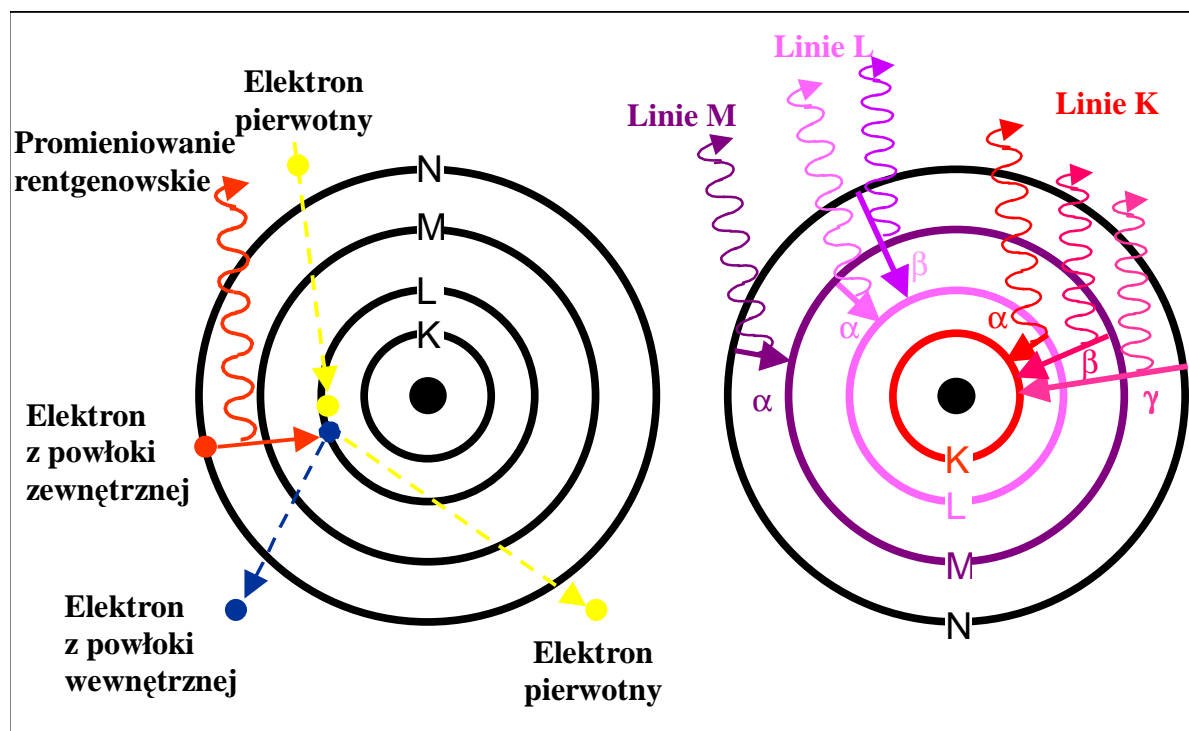
If the electron on K envelope is knocked-out then lines observed in the characteristic X-ray radiation spectrum responsible for energy emission, accompanying transition of the electron in order to fill in the gaps, are to be called K lines:

- $K\alpha$ - in case of transition from L coating,
- $K\beta$ - in case of transition from M coating,
- $K\gamma$ - in case of transition from N coating.

If the electron is knocked-out from L coating we deal with L lines:

- $L\alpha$ - in case of transition from M coating,
- $L\beta$ - in case of transition from N coating.

If the electron is knocked-out from M coating we can observe M lines (see: pict.):



General rules concerning characteristic X-ray radiation:

For a given element lower lines have higher energy than higher ones :
 $E_K > E_L > E_M$

Within a given series lines of the elements with low atomic number have low energy, for instance: carbon's K line has lower energy than oxygen's K line.

Lines of lower series (K) are distinct and have simple structure while lines of higher series (L and M) have complex structure and overlap.

In X-ray micro-analyser the radiation is still the background for the lines of characteristic X-ray radiation. It is very important to know the intensity of this background when determining the limits of detection for a given element.

1.3. X-ray maps

X-ray radiation produces the shadows of much worse quality than 'electron' shadows. One of the reasons for that is wide interaction range from which recorded X-ray radiation comes, which consequently results in worse resolution. Another one is constant X-ray radiation (Bremsstrahlung) which combined with usually low level of characteristic radiation impulses makes the signal - noise proportion disadvantageous.

Usually, X-ray shadows are presented as maps. Analytical beam scans the analysed range point after point. Spectrometer is set in such a way so as to register the point within the analysed range when it detects an X-ray impulse with energy characteristic for a given element. As a consequence we obtain a map imaging distribution of a given element within the analysed range. Modern EDS systems can create maps in grey shadows showing relative intensity of the impulse in each point. Yet, it requires keeping the beam in each of those analytical points long enough to achieve proper results. One use of the analytical beam can register distribution maps for several different elements.

1.4. X-ray analyses

Because of low resolution the X-ray impulses are more useful for the analytical purposes rather than for sample imaging. Qualitative analysis aims at determining whether researched sample range contains given elements – taking into consideration presence or lack of their characteristic peaks within the spectrum. The objective of the quantitative analysis is to determine the proportion of elements' contents on the basis of comparison between the intensity of particular peaks with other peaks or with the standards. Quantitative analysis is quite a complicated process due to the fact that various interactions between the atoms of the sample and characteristic X-ray radiation may appear.



2. Construction and extent of chemical analyser modernization.

The modernized system consists of:

- detector;
- chemical analyzer, PC and 'ISA' software;
- set of cables.

While modernizing we did not introduce any modifications either into the detector itself or preliminary amplifier built onto it.

By means of three cables the signal from the detector enters modernized case of the analyzer.

The whole transformation process takes place inside that module.

There is also a CD with software inside the case. It allows to analyse the measurements made.

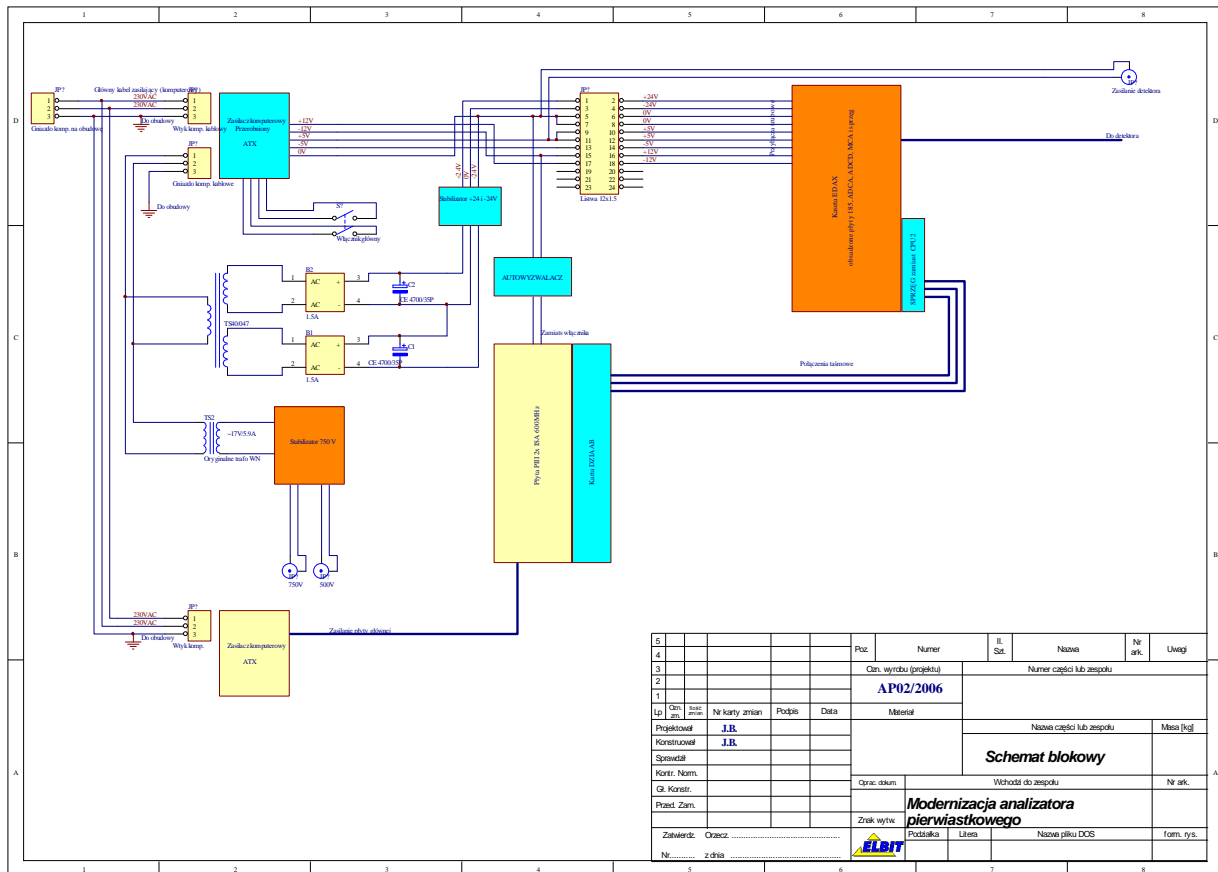
While modernizing it was decided to leave four original boards with electronic elements (amplifier '185', converters 'ADC A' and 'ADCD' and memory board 'MCA'). The reason for that – financial reasons – savings.

The other blocks were made or replaced by modern electronics and software.



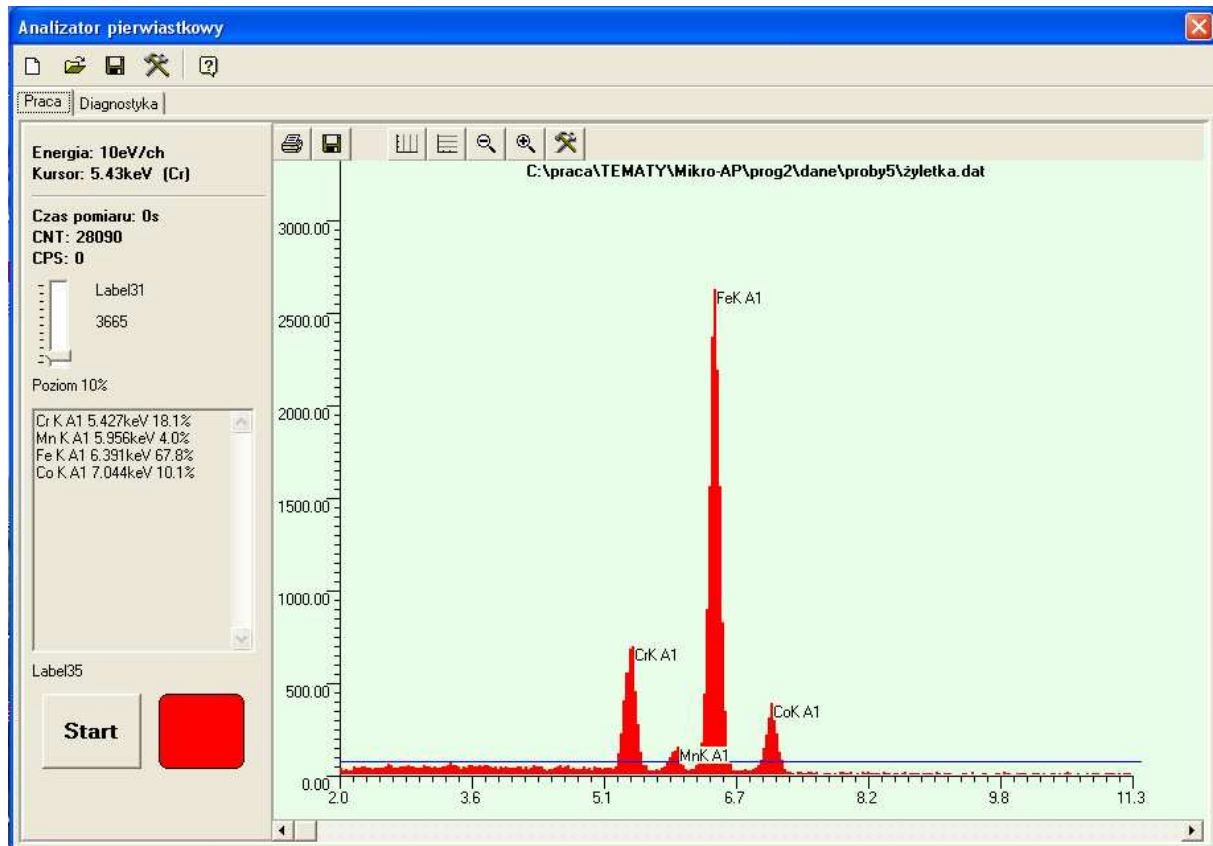
The block diagram below shows current connections in the module.

Red represents original boards, yellow – commercial elements and blue – electronics made by Elbit company as a part of modernization process



3. Software.

The programme operating modernized chemical analyzer in its basic form can be seen below:







The whole window is divided into three functional parts:

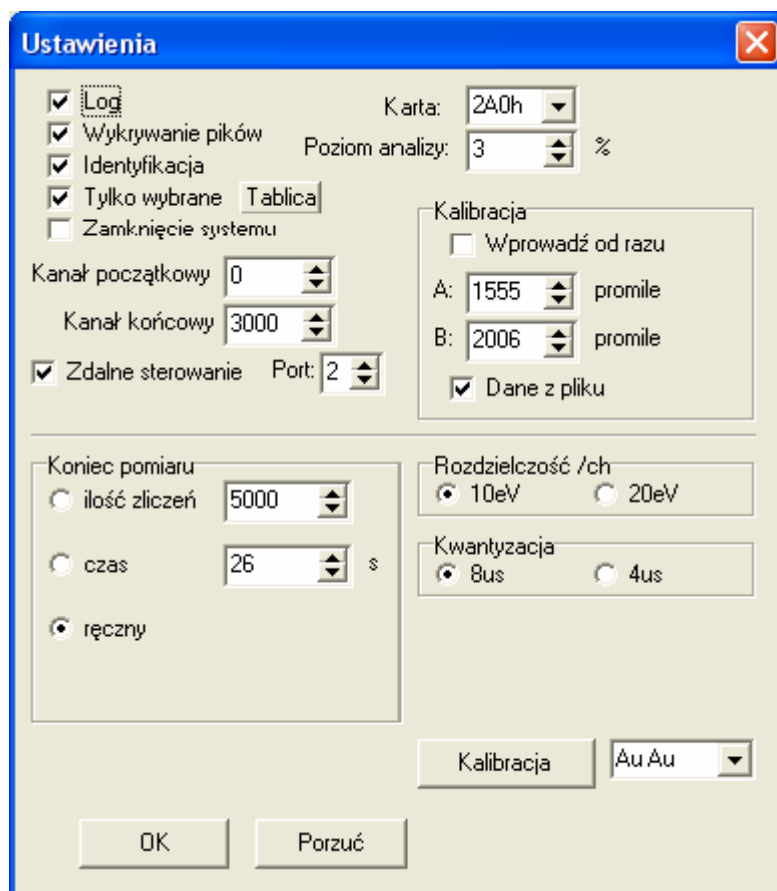
- icon toolbar;
- 'Work' bookmark – two parts can be distinguished here: information panel and graphic part (diagram)
- 'Diagnostics' bookmark

3.1. Icon toolbar

Thanks to icon toolbar it is possible to execute some of the commands connected with the data very quickly as well as to set the measurement parameters easily. The data can be saved and read from the hard disc freely. The functions to be found in the toolbar are as follows:

-  **New measurement** – activation of this function clears all the data and the programme is ready to make new measurements;
-  **Load the data** – activation of this function opens new popup window where the name of the file is to be selected/chosen from the list in order to be opened.
-  **Save the data** – activation of this function saves all the data in the current catalogue.
-  **Programme configuration** – opens the popup window with the settings of the programme.

This is how the window looks like:



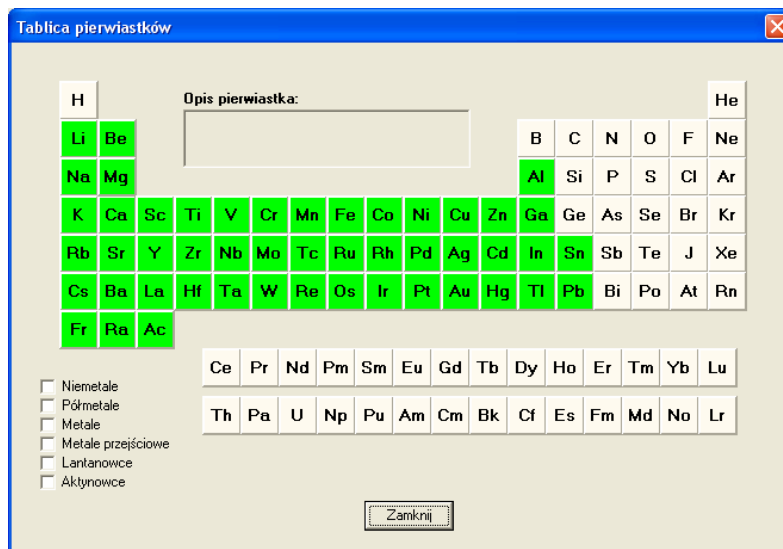
Some fields need to be discussed.

Log – service function. When initialized, the programme creates a file with the description of every action taken as far as the access to the electronics of the analyser is concerned.

File detection – after activation the functions detecting local maximums within the spectrum are on, thanks to which it is possible to determine how many and what kind of characteristic peaks measured the spectrum has. Each detected peak is described with either energy of its maximum or with the element corresponding to this energy.

Identification – this option switches on the function which assigns corresponding pathways for given elements to the detected energies.

Selected only – due to the fact that most of the elements in a given sample is to be found in trace quantities generating only interfering noise and increasing the risk of mistakes, identification algorithms can be supported by prior determination of the set of elements that can be found in a given sample and that are to be detected. Switching on that very parameter limits the set of elements. The set is to be defined in a popup window activated with ‘table’ button. It looks like that:



System shut-down – selecting this option closes the system and shuts down the computer at the moment of closing ‘ISA’ programme. Starting the system and the ‘ISA’ programme takes place automatically after switching on the power with the main switch.

Initial channel and Final channel – The measurement range of the analyzer is about 140 keV. For most microscopes it is too much – they are not able to generate secondary radiation with such energy. Therefore, in order to make the scanning faster it is possible to select limits of the measured spectrum. The values are given as the number of the measurement channel but the channel with the lowest energy has got number 0 and the channel with the highest energy has got the number 4096.

Remote control – ‘ISA’ programme can transmit steering commands and send the results to the superior system. This transmission takes place by means of series connection for which the number of the port is to be defined. With the help of this mechanism a function defining the element’s composition on large spaces was created. The main steering programme here is ‘VID’ programme which controls table’s drive moving it as required, for instance 2µm. and producing analysis for each such point. The results of this analysis (generated in ‘ISA’ programme) are transmitted back to the ‘VID’ programme where two-dimensional map of elements’ distribution in a sample is created.

Card – A computer card with most of the steering functions of the old analyzer was created during modernization process. This parameter defines the access point to it.

Analysis level – This value determines the level below which all the peaks of the spectrum are to be recognized as noise and will not be taken into consideration in algorithms of elements recognition. It is given in percentages (from 1 to 99) of the maximum peak within the spectrum. Such definition (in comparison with the highest peak) has the following advantage – the absolute level increases along with the number of counted points and it does not need to be corrected during measurement process. The current level is marked in the graphic field with the blue line.

Resolution – It defines the resolution of the single channel which is connected with the range of measurement for axis of ordinates (energy).

Quantization – It defines the level of quantization of the measurements which is connected with the exactness of spectrum peaks' mapping.

Calibration – Every analyzer needs calibration. The original analyzer before modernization had to be calibrated every single day. Now, it is enough to proceed one calibration in several days' intervals. The calibration assigns to separate channels recognized by them energies so that the identification function could show and recognize the correct elements. In modernized analyzer the calibration process is fully automated when switched on with proper button. Calibration for three various samples was implemented into the analyzer: brass (CuZn calibration), pure gold (Au Au calibration) and zirconium (Zr Zr calibration). It is possible to introduce manual corrections.

3.2. WORK bookmark

On the left of the window there is an information panel which displays the following information:

- resolution of the device (in keV/channel which means per single memory cell);
- cursor position in the graphic window and the calculation of the position corresponding to that energy as well as the approximate recognized element;
- the time of the measurement given in seconds;
- total number of calculations (CNT);
- the number of calculations per second that is the speed of calculation (CPS);
- analysis level slide;
- the window displaying the amount of a given element in the spectrum given in percentages (not regarding its weight);
- the button and the diode show whether the measurement is in process or not.

The function of the analysis level slide needs some more explanation. It gives the level below which all the peaks of the spectrum are to be recognized as the noise and will not be taken into consideration in algorithms of element recognition. It is given in percentages (from 1 to 99) of the maximum peak within the spectrum. Such definition (in comparison with the highest peak) has the following advantage – the absolute level increases along with the number of counted points and it does not need to be corrected during measurement process. The current level is marked in the graphic field with the blue line.

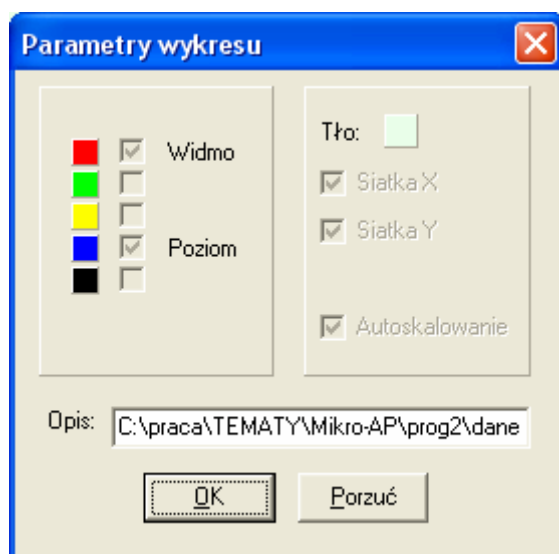
The graphic part, that is the spectrum diagram, is scaled in keV and has got auto-scaling function. It means that during measurement process the programme selects the scale of axis of ordinates automatically.

Depending on the type of the analysis each peak higher than selected level of analysis is identified as an element and its energy is given in keV.

At the top of the diagram there is a space for the description of the measured spectrum to be filled in by the user.

The graphic field has got in its upper part the icon toolbar displaying available functions.

- **Print:** - the function prints current graphic field as a picture;
- **Save the diagram as a bitmap:** - the function saves current appearance of the graphic field as a picture with extension and prints current graphic field as bmp picture in the current catalogue;
- **Axis X:** - switches on and off vertical lines of the graph;
- **Axis Y:** - switches on and off horizontal lines of the graph;
- **Make smaller:** - „tightens” the axis;
- **Enlarge:** - „stretches” the axis;
- **Diagram parameters:** - switches on the popup window with detailed options connected with the graph. This is how the window looks like:



„Clicking” particular spaces allows to change the colours of the diagram as well as the background. It also allows to add description which is later on displayed on the spectrum picture.

At the bottom of the graphic field there is a slide allowing smooth horizontal shift of the diagram.

3.2. DIAGNOSTICS bookmark

Service parameters are to be found in this part of the programme. Changing them may result in the analyser mistuning!