Report

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Project title:

Centre of Excellence for Optical Spectroscopy Applications in Physics, Material Science and Environmental Protection

Appendix C:

Manual instruction for Bomem DA8 FTIR spectrometer and IR-Plan microscope

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A. Manual instruction for Bomem DA8 FTIR spectrometer

1. Description of Bomem DA8 spectrometer at Institute of Physics in Belgrade

The configuration of the instrument is given in Figure 1. It consists of three sections: the upper one containing the source and beamsplitter compartment, the middle section with the beam switching compartment, the sample compartment and the detector modules, and the lower part containing the vacuum leads, the power supplies and the data processing and control electronics. Depending of the moving mirror travel, the resolution ranges from 0.06 to 0.0026 cm^{-1}. The instrument has two focused output beams in a sample compartment and three parallel output beams. In the present configuration our DA8 system operates in the range from 10 to 25 000 cm^{-1} with the maximal resolution of 0.02 cm^{-1}. As sources we use Hg lamp, Globar (SiC) and Quartz lamp. Beamsplitters are 25 μm Mylar film (10-125cm^{-1}), Hypersplitter (40-700cm^{-1}) KBr (500-5000cm^{-1}) and Quartz (4000-25000cm^{-1}). We use the following detectors: DTGS (far IR), MCT (mid infrared), InSb (near infrared) and Si photodiode (visible). The spectrometer is equipped with two cryostats: He flow Janis STDA 100 (LN2 and LHe, 4-400K) and ARS DMX 20 closed-cycle low vibration cryostat (4.5-300K).

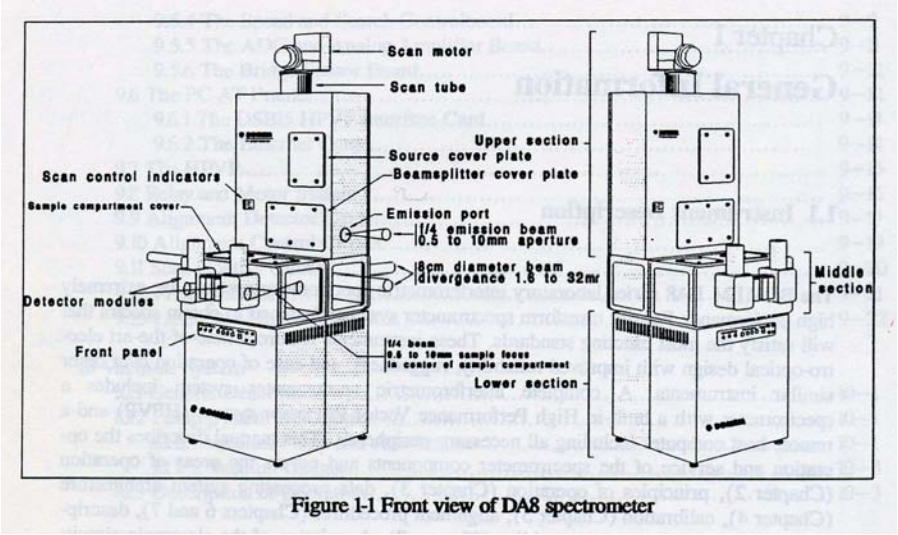


Figure 1. Bomem DA8 FTIR spectrometer configuration

For data acquisition and processing DA8 uses the last working version of Bomem GRAMS/AI 7.0 software. In addition the new DA8 PC.AT Frame Pentium, and the Bomem dedicated IR-PLAN advance analytical microscope were obtained.

2. Standard measuring procedure with Bomem DA8 spectrometer

First, for a desired spectral range it is necessary to choose an appropriate beamsplitter, source and detector. It is worth mentioning that beamsplitters and detectors must be manually replaced and mounted on DA8 system. Usually they are already aligned and do not need further adjustment. Depending on transmission or reflection measurements we choose sample holders. Regarding the

sample opening on the sample holder and desired resolution one chooses a corresponding iris aperture below the sources.
In our case we use two sample holders: a simple one which needs breaking the vacuum between the measurements of the sample and reference or a sample holder on the cold finger of a cryostat which enables vertical movement under the vacuum. In the case of reflection measurements additional optical alignment in the sample compartment is necessary. Namely, the optical beam must be precisely focused on the sample and the detector spot with flat and parabolic mirrors of the reflection box accessory. This normally takes hour or two and when the signal is maximized the system is ready for measuring.

B. IR-Plan analytical microscope 0047-xxx

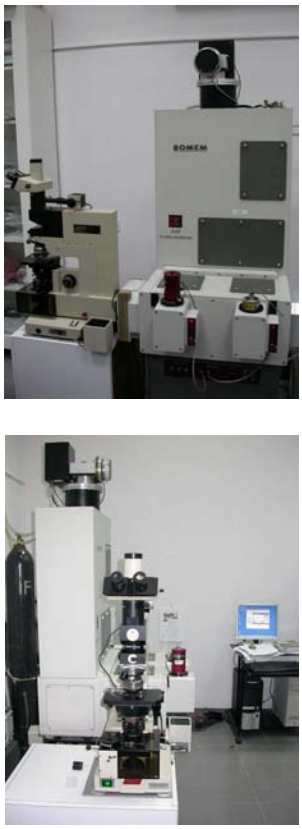


Figure 2. IR Plan microscope mounted on Bomem DA8 spectrometer

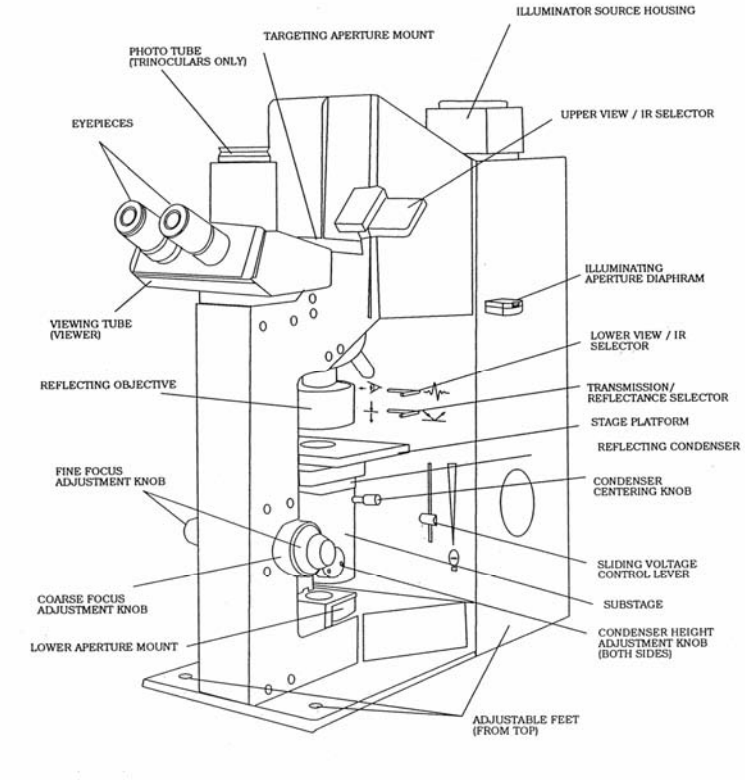


Figure 3 . Schematic diagram of IR-Plan microscope

IR-PLAN analytical microscope is a visible light microscope equipped with a high performance infrared sampling accessory designed for operation with FT-IR spectrometer. The FT-IR measurements with IR microscope can be performed using FT-IR MCT (mercury cadmium telluride) detector or can be equipped with an optional MCT detector. The microscope performance is directly related to the detection system because usually the analyzing samples are small so it is necessary to use MCT detector. IR-PLAN enables viewing of the exact sample area that will be analyzed and offers the best resolution available in FT-IR microscopy in order to obtain the highest possible quality spectra with the least stray light. On Figure 2 is given the front view of the IR-PLAN analytical microscope connected

to our spectrometer BOMEM DA8, whereas Fig. 3 presents the feature diagram with main components of the IR-PLAN.

IR-PLAN can be mounted, depending on the type of the spectrometer, in the primary compartment or in an external sample compartment or alongside of the spectrometer.

1. Instrument specifications

Viewing optics: Standard 15X Reflachromat IR/VIS objective of the Cassegrainian type design for 150X viewing and 10X D-plan achromat glass objective for 100X viewing and visual identification of the sample area with a larger field of view.

Viewing attachments: Binocular viewer has paired 10X eyepieces with crosshair measuring reticle. In combination with the standard 15X objective, provides over 150X visual magnification.

Detection: use of the spectrometer's detector optics and detector or a dedicated MCT detector.

Illumination: High intensity reflected and transmitted light illumination with variable light intensity and field and aperture stops.

Objective positioning: 4 position rotatable nosepiece.

Sample positioning: Standard 2"x3" travel rectangular rotatable manual stage with stage clip.

Sample masking: Two circular variable apertures for masking capable of being used simultaneously to mask the sample, anywhere in the field of view.

Field of view& sampling area: Nominal 1.3mm field of view with a 15x objective. Sampling area depend on the detector, detector optics and the collecting objective being used.

Purge: the spectrometer's purge system can be used or own purge system depending on the instrument and coupling.

Sampling mode: Transmission or reflectance.

Microscope support: Rolling work station/table.

2. Operating modes

IR-PLAN analytical microscope can operate in two modes:

1. **transmission,** and
2. **reflectance**

Transmitted illumination means that the sample is illuminated from behind the sample (below). Samples which are visible opaque can be analyzed in reflectance mode. In this mode the illumination is brought to the front of the sample (from the top). Switching to one or another mode is accomplished by flipping the rocker switch on the left side of the IR-PLAN. The switch is labelled **EPI** for reflectance mode and **DIA** for the transmission mode. To select visible or reflectance sample illumination the view/IR selector on the operating selection panel should be moved to the **IN VIEW** position. Than transmission/reflectance lever on the operating mode selection panel should be moved to the transmission or reflectance mode.

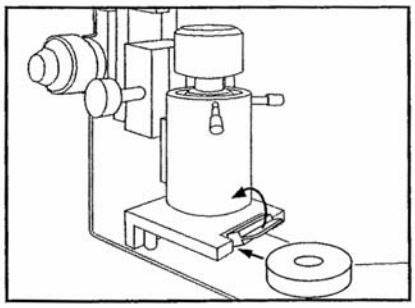
Measurements in reflectance mode: The view/IR selector is in the IR position and the transmission/reflectance selector is in the reflectance position. When these two levers are in the position and the upper view/IR selector is in the IR position the microscope is ready to collect data in the IR reflectance mode

Measurements in transmission mode: The same procedure is valid for transmission measurement when only the transmission/reflectance selector is in the transmission position.

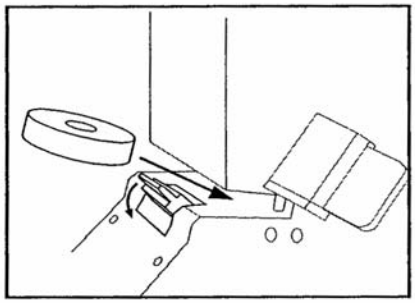
3. Sample position adjustment

IR-PLAN analytical microscope is equipped with a standard 1"x2" manual stage with a stage clip (see Fig. 4) provides the movement of the sample along the x, y and z-axis (focus adjustment). The focus adjustment moves the complete stage and sample up and down. The focus adjustment knobs are located on either side of the microscope stand. These knobs are graduated and procure coarse and fine adjustment. Usage of two circular, rotatable masking apertures above and below the sample reduces the stray light and other unwanted spectral contributions. The upper aperture is located in the infrared path between the infrared source and the sample, whereas the lower aperture is located in the infrared path between the sample and the infrared detector.

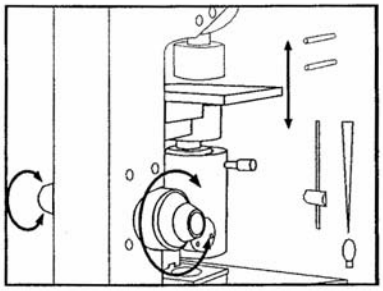
Figure 4. Front view of IR-Plan microscope



lower aperture



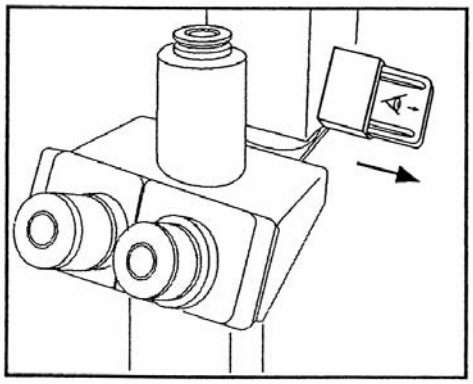
upper aperture



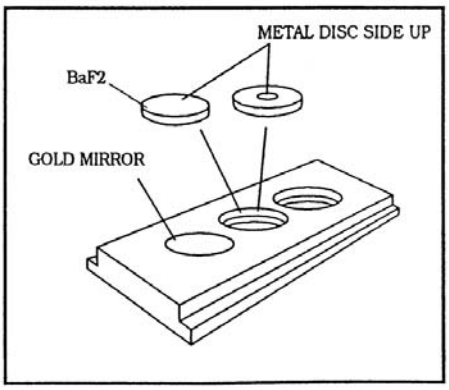
coarse and fine focus adjustment knobs

4. Mounting and focusing the 100µm pinhole

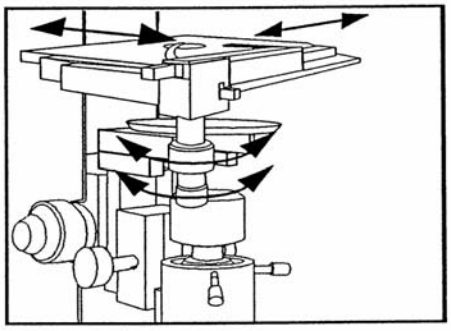
The upper view/IR selector is in the view position. Put the lower view/IR selector in the view position and the transmission/reflectance selector is in the reflectance position. Place the pinhole on the sample slide in such a way that a stainless steel disc is facing upward toward the objective and put it on the stage assembly. Using the x, y movements of the stage and focus adjustment knobs, pinhole should be brought to the focal point of the light. Now the pinhole can be focused and cantered to the cross hair reticle which is mounted on one of the eyepieces (usually in the right hand one).



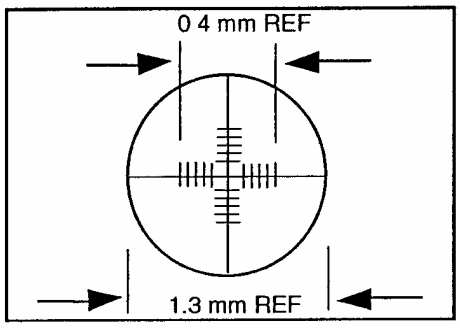
upper view/IR selector



sample holder



x, y, z stage assembly



cross hair reticle

C. Starting measurements

Checking of the alignment: After installation and during the normal operation of the microscope the optical alignment in both transmission and reflection modes should be checked in order to perform accurate sample analysis.

Signal-to-noise ratio: One of the measures of overall performance of the microscope is signal-to-noise ratio test. The procedure involves collection and analysis of a standard 100% line and calculation of the signal to noise ratio value from that spectrum.

Measurements in transmission/reflectance modes

Alignment check

Checking of the alignment in the transmission mode

1. Lower the sample stage and install the slide with a 100 μm pinhole mounted on a stainless steel disc into the stage clip.
2. Slide the upper view/IR selector to the IN VIEW MODE position. Set the operating mode selection knobs to REFL and VISUAL positions.
3. Raise the illuminator intensity to halfway using the slide control on the operating mode selection panel.
4. Using course/fine focus controls, raise the100 μm pinhole until the light from the15X objective focuses on the pinhole surface.
5. Through the viewer locate and center the pinhole relative to the reticle crosshair using x, y position stage adjustments.
6. Adjust the focus in such a way so the pinhole image is in sharp focus.
7. Switch the operation mode selector to TRANSmitted illumination.
8. Using only x adjustment knob, move the pinhole to the side.
9. Install lower aperture below the condenser and focus the image.
10. Using x, y adjustment knobs, center the aperture image with respect to the reticle crosshair.
11. Turn the pinhole back into centred position adjusting only x sample stage.
12. Install the upper aperture.
13. Move the upper VIEW/IR beamsplitter slide to the IN IR MODE position. Switch the operating mode selection panel to IR mode and leave the system in TRANSmission mode.
14. Start with measurements.

Checking of the alignment in the reflection mode

1. Lower the sample stage and install the gold mirror stage plate.
2. Repeat procedure 2-4 for transmission mode.
3. Through the viewer locate and center the mirror relative to the reticle crosshair using x, y position stage adjustments.
4. Adjust the focus in such a way so the mirror image is in sharp focus.
5. Install the upper aperture.
6. Move the upper VIEW/IR beamsplitter slide to the IN IR MODE position. Switch the operating mode selection panel to IR mode and leave the system in REFLected mode.
7. Start with measurements.

Signal-to –noise ratio in transmission mode

1. Place the100 μm pinhole on the sample slide
2. Slide the lower view/IR selector to the view position.
3. Operating mode selector should be switched to REFLected illumination.
4. Raise the sliding illumination voltage control lever to halfway.
5. Using x, y stage and coarse and fine focus knobs raise (or lower) 100 μm pinhole until the light emanating from the reflecting objective focuses on the pinhole surface.
6. Adjust the focus so the centred pinhole image is in sharp focus.
7. Install the 1.5 mm lower aperture, focus and center the image of the lower aperture using only the condenser centring knobs and condense height adjustment knobs. **DO NOT USE COARSE OR FINE FOCUS ADJUSTMENT KNOBS.**
8. Install the upper aperture.
9. To perform signal-to-noise ratio test in a transmission mode a 100 μm pinhole should be aligned with 1.5 mm lower and upper apertures.
10. Operating mode selection panel should be switched to TRANSmitted illumination
11. IR mode and the upper **VIEW/IR** beamsplitter slide to **IN IR MODE.**
12. Acquire the background spectra and the spectra of 100 μm pinhole as a sample.
13. The ratio of these two spectra should give a flat line near 100% transmission. Spectrum should be free of periodic spikes (see Fig. 5).
14. To calculate the signal-to–noise ratio the certain region of the spectra should be expanded. The highest and lowest value of the peaks in the expanded region should be recorded and their difference should be calaculated.
15. 100 divided by the above difference represents the signal-to–noise ratio (see Fig. 6).

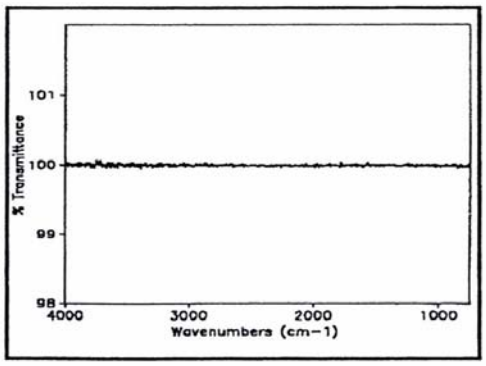


Fig. 5 Standard 100% line in transmission mode.

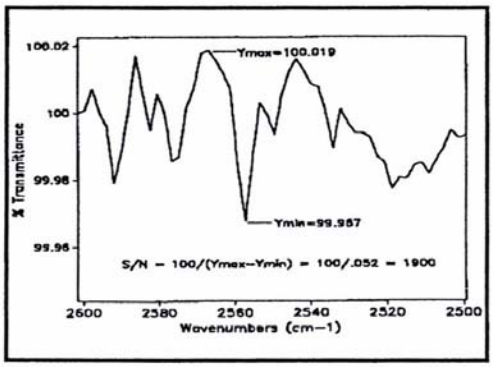


Fig. 6 Calculation of the signal to noise ratio value of the spectrum from Fig. 5

Signal-to –noise ratio in reflection mode

The procedure from 1-5 should be repeated for reflection mode as well but in that case golden mirror is used as a sample, only upper aperture is installed and the operating mode should be adjusted for REFLected illumination mode.

Measurements in transmission/reflection mode

Reflectance measurements

1. Slide the upper and lower VIEW/IR selectors to **view** position.
2. Slide the transmission/reflectance selector to the **reflectance position**.
3. Focus the sample.
4. Install upper aperture.
5. Slide the upper and lower VIEW/IR selectors to the **IR position**.
6. Collect the sample spectrum.
7. Slide the upper and lower VIEW/IR selectors to **view** position.
8. Do not change or adjust the aperture.
9. Using stage x, y controls move the gold mirror until the light strikes the mirror surface.
10. Look through the viewer and adjust the focus knobs so the mirror is in sharp focus.
11. Slide the upper and lower VIEW/IR selectors to **IR** position.
12. Collect the background spectrum.
13. The rationed spectrum is the proper one.

Transmission measurements

1. Slide the upper and lower VIEW/IR selectors to the **view** position.
2. Slide the transmission/reflectance selector to the **reflectance position**.
3. Install the lower and upper aperture.
4. Slide the upper and lower VIEW/IR selectors to the **IR position**.
5. Slide the transmission/reflectance selector to the **transmission position.**
6. Collect the sample spectrum.
7. Slide the upper and lower VIEW/IR selectors to **view** position.
8. Do not change or adjust the aperture.
9. Slide the upper and lower VIEW/IR selectors to **IR** position.
10. Collect the background spectrum.
11. The rationed spectrum is the proper one.

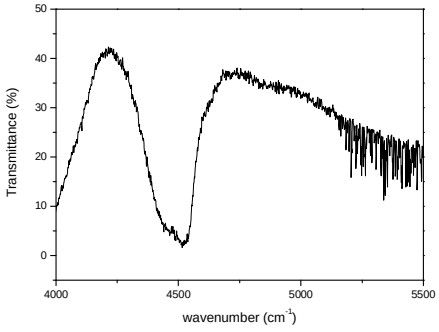


Fig. 5 MIR spectra of the pinhole in transmission mode

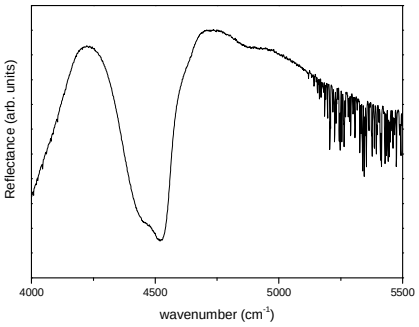


Fig. 6 MIR spectra of the golden mirror in reflectance mode

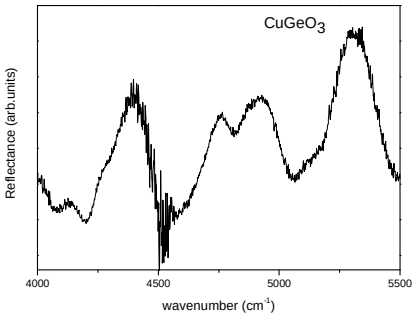


Fig. 7 MIR spectra of the $CuGeO_3$ sample in reflectance mode