This paper describes in detail the concept of an innovative multispectral laboratory developed by "Mir photonics" LLC, as well as a new technique for spectral analysis of liquids, solids, and gases, based on an inter-spectral chemometric approach.

This user manual was developed by "Mir photonics" and is intended for use by researchers of educational and scientific laboratories.

Multispectral Laboratory of Optical Analysis Methods

purpose, methods, and applications

Analytical chemistry is a science that develops the theoretical basis of the chemical analysis of substances and materials and develops practical methods for identifying, detecting, separating, and determining chemical elements and their compounds, as well as methods for establishing the chemical composition of substances. The analysis is usually divided into quantitative and qualitative. The first answers the question "what does the sample consist of?", the second answers the question "what is its quantitative composition, how much of this or that substance does it contain?" The task of an analytical chemist is to develop and improve methods for quantitative and qualitative analysis. For this purpose, knowledge from various fields of science, including modern scientific and technical achievements of chemistry, physics, mathematics, biology, and others are used. The analysis methods being developed are aimed at solving various life problems: ensuring the quality of manufactured products, increasing the efficiency of technological processes, environmental protection, and medical diagnostics. Thus, analytical chemistry is directly related to the solution of real practical problems.

The analytical chemistry today is no longer associated with archaic chemical methods, such as colour reactions, precipitation, gas evolution, and combustion. Laboratory measurement methods based on titration, filtration, distillation, and weighing are becoming a thing of the past and are becoming part of the educational process rather than practical chemical analysis. Modern analytical chemistry is complex, highly automated measuring instruments that use a variety of effects that arise when a substance interacts with a field, wave, or particle flow.

Optical spectral analysis has a special place among physicochemical methods for studying substances and materials. It is based on measuring the result of the interaction of electromagnetic radiation with a sample in a wide range of wavelengths from hard ultraviolet to far infrared light. The result of this interaction, measured by using a spectrometric or sensory analyzer, carries a lot of chemical information about the analysed object. Being extremely flexible in organizing measurements and, not requiring mandatory sample preparation, fast and non-destructive, optical analysis often has no alternatives in solving important problems of ensuring the quality and safety of life in modern society.

One of the most noticeable trends in the development of optical spectroscopy is the increasing use of fiber optic solutions. Fiber optic probes and cells are built into reactors and production lines of ongoing technological processes, allowing for online analysis on site and obtaining results in near real time. Thanks to the achievements of photonics, optics and micromechanical engineering, the development of specialized analyzers of a new type, optical multisensor systems (OMS), is underway. These inexpensive, compact, and portable systems are increasingly being used for field and rapid analysis.

An integral part of modern spectral methods is the mathematical processing of complex multidimensional data in order to extract useful analytical information from them. For this purpose, a special branch of analytical chemistry is used - chemometrics, which provides chemists with a rich toolkit for constructing calibration and classification models, as well as methods for in-depth research data analysis.

This document is devoted to the description of a universal optical multispectral laboratory - a collection of optical spectral equipment specially selected to solve a wide variety of scientific, educational, and industrial problems. The laboratory is focused on modern analytical research, including the online analysis using fiber optic cables and probes. The equipment includes spectrometers for analysis using basic optical methods – absorption in ultraviolet (UV) and visible, as well as in near and mid-infrared (IR) spectral ranges, fluorescence, and Raman scattering. Measurement of all kinds of gaseous, liquid, and solid objects is organized using a variety of interfaces and allows for sample analysis for transmission, diffuse reflection or emission measurements. Appropriate techniques for measuring and analyzing the resulting data are provided, including software, as well as illustrative notes. It is possible to combine two or more spectral methods when necessary to solve particularly complex analytical problems, such as analytical process control and medical diagnostics. The description is provided with typical examples of using the laboratory, including the construction of operational analytical techniques using chemometric software.

2. Purpose of the laboratory

The laboratory is designed to carry out all main types of modern optical spectral analysis. What is the advantage of purchasing an entire spectral laboratory at one- time, compared to purchasing it element by element? To answer this question, we need to consider different situations.

2.1 Use for educational purposes

The optical spectral laboratory can serve to support the educational process, for example, within the framework of undergraduate, graduate, and postgraduate programs in the course of studying courses in analytical chemistry, chemical ecology, pharmaceutical and food production, as well as other disciplines related to chemical analysis. The use of a pre-selected set of equipment, with optimized price, equipped with software and ready-made examples of laboratory works on various topics, significantly facilitates the technical organization of the educational process. Such an educational laboratory can simultaneously be used in scientific development, as shown below.

2.2. Scientific research

The optical spectral laboratory can be used in research conducted at universities and other scientific institutions. The exploratory nature of scientific research does not always allow one to predict in advance which of the existing methods will bring the desired result. The ability to try different optical spectroscopy approaches is important for solving complex analytical problems, such as the analysis of natural and industrial samples of not fully known composition in order to select the most suitable method or combination of them.

The presence of a modern, pre-designed and well-integrated laboratory of spectral analysis methods and pre-proven techniques makes it easier to obtain funded projects from the state and industrial customers.

2.3. Industrial use

A thoughtful combination of spectral equipment and software can significantly facilitate and reduce the cost of equipping of a newly created or updated industrial laboratory. Industrial enterprises and

commercial structures, and sometimes even scientific and educational institutions involved in chemical spectral analysis, may not have sufficient competence to optimally select the necessary spectral equipment, including spectrometers, measurement interfaces and software. In particular, this concerns the creation of multispectral techniques, where two or more spectroscopy methods are optimally combined.

Traditionally, equipping of a spectral laboratory is happening gradually, as the need for a particular analytical method and instrument arises. Often this approach is dictated by the organization's budget and ensures an even distribution of the available funds. However, it is not optimal from the point of view of combining laboratory elements with each other and does not consider the entire scope of practical problems that may arise in the future.

It is important that the integrated approach takes into account in advance the mutual compatibility of instruments and software, the ability to work with data from various spectral formats, thereby facilitating the preparation and analysis of data.

Thus, purchasing an entire laboratory is in many cases more practical than completing it piece by piece. In addition to integrated equipment, software and ready-made techniques, the comprehensive equipment of the spectral laboratory includes the knowledge and experience of many expert analysts.

3. Advantages of the multispectral approach

3.1. Information content of spectral methods

Spectral analysis provides information about the chemistry and morphology of the sample under study: the concentrations of the components it contains, their distribution, and particle sizes. The analytical information provided by different spectral methods can vary greatly because it is closely related to the chemistry of the sample being examined, the nature of the physical phenomena involved, and the method of measurement itself (Table 1).

One of the most informative methods of analysis is molecular vibrational spectroscopy. To record energy transitions between vibrational levels of molecules, three spectroscopy methods are used: mid- or near-infrared (NIR) ranges, as well as Raman spectroscopy.

Despite the theoretical commonality of these methods, their practical instrumental implementation and use in analysis are very different. The difference in operating wavelength ranges - from 360 nm to 25000 nm (400 cm^{-1}) - requires the use of different light sources, optics, and detectors, and therefore, as a rule, is implemented in separate spectral devices. The different absorption coefficients of light in NIR, IR and Raman spectroscopy, as well as the depth of its penetration into a particular dense medium (solid, liquid or powder) require different measurement interfaces and techniques, such as cuvette, probe, reflection measurement, etc. These differences in methods create natural limitations for their use, that is, one or another problem of practical analysis requires the selection of the most optimal method and approach (technique) for solving it. Thus, NIR spectroscopy with its relatively large penetration depth (up to several mm) even into a dense solid sample due to low absorption and strong scattering. This makes it possible to use ordinary glass for optics, as well as standard lamps as light sources, which is ideal for online applications, including distance measurement or through fiber optic probes. In contrast, the strong absorption in the mid-IR region limits the corresponding spectral method to the analysis of only very thin samples (usually less than 50 μm) but opens the possibility of using the attenuated total reflection (ATR) method by connecting appropriate attachments and probes. Measuring the weak Raman effect requires special cells and probes but is recorded in the visible and NIR regions of the spectrum. In this case, the spectra of vibrational transitions in Raman and mid-IR

spectroscopy carry complementary qualitative and quantitative information about the composition of the sample.

UV/Vis spectroscopy is based on electronic transitions between energy levels of a molecule. It has very high sensitivity, especially in the UV region (190-360 nm), but requires the use of special light sources, cuvettes, and optics, including fiber optics. However, the strong overlap of spectral signals in UV spectra complicates the analysis of real samples and makes the use of chemometrics algorithms a necessary component of methods for the qualitative analysis of mixtures.

Another very sensitive method, UV/Vis fluorimetry, is based on the secondary emission of excited molecules of certain substances, most often of biological origin. However, most chemicals do not have this feature. Therefore, fluorimetry is in demand mainly in medicine, biology, and biotechnology.

The spectra of the different methods vary greatly, both in their nature and in the information they contain. Mid-IR band spectra contain well-defined peaks (absorption bands), while NIR, UV, and fluorescence spectra contain broad, highly overlapping signals that form the so-called continuum spectrum. Extracting the information necessary for quantitative and qualitative analysis from optical spectra requires special methods for analyzing multidimensional data, called the above-mentioned term chemometrics. Chemometrics algorithms are implemented in special software that allows preliminary preparation (preprocessing) of data, construction, verification (validation) and application of predictive, namely, calibration or classification models.

For measurements of liquid and soft or powdery solid samples, immersed fiber optic probes are increasingly used, operating in various spectral regions from UV to mid-IR. With their help, one can obtain absorption, diffuse reflection or transmission spectra, as well as ATR. The use of probes may become necessary for in situ analysis - in the field or in a live process environment. Online measurements require high speed spectrum acquisition. Often the analysis result must be obtained in real time in order to use it for making management decisions.

The analysis of individual spectral methods, their capabilities and limitations, shows that in order to develop an optimal instrumental technique for solving an existing analytical problem, it is necessary to take into account many different factors. Thus, choosing a spectral method for a specific analysis problem is not an easy task, requiring special knowledge from the fields of chemistry, physics and mathematics. Moreover, this refer to the equipping an entire laboratory for future needs, since the need for a particular method of optical analysis is difficult to predict in advance. In the set of equipment offered here, called the multispectral laboratory, experts from various fields have already worked hard to select the optimal solutions.

Table 1. Comparative characteristics of spectral methods and measurement techniques.

№	Method	Measurement method	Physical phenomenon	Sample Type	Optical path, depth	Sensitivity	Selectivity	Cost
1	**IR spectroscopy**	ATR- platform	absorption	liquid, powder, paste, soft	3-6 mu	average	average	high
		ATR probe*						
		cuvette		liquid	10-100 mu	high		average
		sample layer		solid film	5-50 mu			
		sample surface	diffuse reflection	solid	10 mu	average		
2	**NIR spectroscopy**	cuvette	transmission	liquid	1-5 mm	low	low	average
		transmission probe		solid, colloidal	2-5 mm			high
		Defuse reflection-probe	diffuse reflection	liquid	0.5-1 mm			
		integrating sphere		solid, powder	5-10 mm			
		surface at a distance		solid, powder	2-5 mm			average
3	**Raman spectroscopy**	cuvette 90°	raman scattering (emission)	liquid	2-5 mm	low	high	high
		Raman-probe		liquid, powder				
4	**UV/Visible spectroscopy**	cuvette	absorption	liquid	1-5 mm	высокая	low	low
		ATR probe			1-5 mu	low		average
		Defuse reflection-probe	diffuse reflection or transmission	solid, pill	1-5 mm	высокая		low
		probe, lens	emission of an external source	radiating sample	any	high		
5	**Fluorimetry**	cuvette 90°	fluorescence (emission)	liquid	10mm	high	average	low
		fluorimetric probe		liquid, solid	1-5 mm			

3.2. Synergy of spectral methods combination

The information supplied by one or another optical spectral method may be insufficient when analyzing real samples. These include complex multicomponent mixtures, most often of natural origin, such as oil, milk, process medium, fermentation medium, biological tissue. Often, the analysis of such objects is complicated by the overlap of several phenomena at once: absorption, Rayleigh, and Raman scattering, as well as fluorescence. Morphological factors, such as inhomogeneity of the sample (for example, biological tissue) or turbulence of the process, should also be taken into account when choosing a measurement technique.

In the analysis of such systems, a combination of two or even more spectral methods may be required. This often gives a synergistic effect, allowing you to solve a problem with which each method individually does not cope or does not cope fully. Thus, the combination of NIR-band spectroscopy with fluorescence spectroscopy has shown that it is possible to determine the biomass content during yeast fermentation with higher accuracy, as well as to distinguish their intracellular and extracellular metabolites. The combination of NIR and IR spectroscopy proved being beneficial determining the

tumor boundary in medical diagnostics. The combination of fluorescence and IR spectroscopy also proved to be necessary for the classification of biological tissues. There are many other examples of a successful combination of various spectral methods in the problems of quantitative determination or classification of objects. This approach is called multispectral optical analysis, and it is increasingly used in practice.

The basis of the synergetic effect observed when using these two or more methods is the combination of heterogeneous information about the object. This can be chemical information of various kinds, as with the combination of complementary vibrational IR and RAMAN spectra of molecules. It can also be morphological information about the size of scattering particles, as in the analysis of milk [14,15]. Information can also be obtained from different depths, as in the analysis of biological tissue samples by IR and NIR spectroscopy using fiber-optic probes.

The data from the above publications [9-15] was obtained using equipment (spectrometers, power supplies and probes) included in the equipment of this multispectral laboratory.

3.3. Obtaining and analyzing multispectral data

To maximize the benefits of the multispectral approach, it is necessary to correctly collect and analyze the data obtained, sometimes very different in their properties, form and content. Special measurement interfaces, such as probes, have been developed and continue to be developed for the collection of multispectral data, and special chemometrics algorithms have been developed for their effective analysis [6]. Below are the main problems of the multispectral analysis and their solutions.

3.3.1 Simultaneous measurement by two or more spectral methods

The analysis of solid inhomogeneous samples, such as biological tissues, is often carried out using fiber-optic probes. At the same time, it is very important that the spectral information obtained by various methods, namely, IR, NIR, Raman and fluorescence spectra, was obtained from the same small, and even point, portion of the sample. This is difficult to achieve by using two probes alternately for measurements. High accuracy can be achieved using specially designed multispectral probes. One of these probes, optionally supplied with a multispectral laboratory, allows to combine measurements of the IR spectra of an object by the ATR method with measurements of fluorescence spectra [12] and even Raman spectra [16]. An additional measurement takes place through the ATR crystal of zirconium oxide, which has the shape of a truncated cone. To do this, an additional pair of quartz light guides is connected to the base of the crystal, conducting in the range of UV and visible radiation. Another multispectral probe is also under development, combining four spectral methods: IR, NIR, RAMAN, and fluorescence [17].

The problem of simultaneous measurement also arises in the multispectral study of fast processes. In this case, two or more probes belonging to different methods can be used for measurements, but the data of each method must be obtained at the same time. The use of a combined probe facilitates measurements and allows you to analyze a smaller sample volume. There may also be situations where problems of inhomogeneity and rapid changes of the sample exist simultaneously. In this case, the use of multispectral probes becomes a necessity.

3.3.2 Multispectral data analysis features

With simultaneous analysis of processes by different methods, there may be a problem of data synchronization, since the rates of taking different spectra can vary greatly – from milliseconds to minutes, while the interval between measurements may also vary. Ultimately, the measurement data should be brought to a common step in time. Successful synchronization of methods is achieved, first, by the correct choice of conditions for obtaining spectra. Since perfect synchronization is often difficult to achieve, post-synchronization of measurements is usually required by selecting the optimal

time step and selecting suitable data, as well as processing them, for example, averaging. This function should be performed by software algorithms for data processing.

After the data of various methods is synchronized and combined into one matrix, it is not always possible to use such combined data "as is". The variables obtained from different spectral methods are unequal. If the variables differ in units of measurement, then the data should be normalized in one way or another, for example, using the auto-calibration algorithm. But rationing may not eliminate all problems. So, variables of different methods can vary greatly in quality. That is, one part of the data matrix may contain more useful information (higher variance) than the other. Variables of different methods may differ in the level of noise contained or irrelevant variance. In addition, methods containing a larger number of variables have a greater impact on the model. In order to effectively analyze multispectral data, avoiding the above problems, and maximize the benefits of combining spectral methods, special data fusion algorithms are being developed [18].

3.3.3 Instrument execution/implementation of multispectral analysis

One of the problems of multispectral analysis is the need to simultaneously connect at least two independently operating measuring instruments. Often these are universal laboratory spectrometers in desktop design, taking up quite a lot of space. Each spectrometer is usually connected to a separate personal computer or laptop. The cost of such spectrometers is also very high.

There is only one way to get away from cumbersome and expensive solutions in multispectral analysis. It is necessary to develop specialized analyzers – optical multisensory systems (OMS) focused on a specific analytical task. The growing use of OMS is a trend in the development of analytical chemistry. The following section is devoted to the use of a multispectral laboratory in the development of an OMS.

4. Multispectral laboratory in the development of optical multisensor systems (OMS)

The development of OMS begins with studies of the object of analysis by standard laboratory spectrometers. This is necessary in order to calculate the optimal configuration of the future multispectral analyzer in order to select the necessary elements – LEDs, filters, etc.

It is important to understand that, unlike classical spectral methods, such as UV and visible, NIR and mid-IR spectroscopy, OMS are not limited to the same spectral ranges. For example, LED analyzers can simultaneously contain radiation sources from any of the above areas. An ordinary blue LED at a wavelength of 400 nm can be adjacent to an LED in the near-infrared region, having a maximum intensity at 1940 nm, and even with an LED in the middle IR range, for example, 3 microns (about 3300 cm-1). At the same time, the combination of several spectral regions at once does not lead to a significant complication of the design.

5. Hardware and software

5.1. IR spectrometer

Vibrational spectroscopy of the mid-IR range (4000-400 cm-1) is one of the most informative methods of chemical analysis. Due to the high extinction coefficients of various molecules in this area, the method has a high sensitivity and can be used to analyze a wide variety of samples: solid, liquid, and gaseous. However, different samples require different measurement interfaces – special accessories supplied with the spectrometer.

Fig. 1 shows a universal FTIR spectrometer - Simex FT–803, equipped with a set of consoles for various applications (Fig. 1). The measured spectra are displayed on a computer connected to the device using ZAIR's own instrument software (Fig. 1.2). The software is designed for controlling the spectrometer, taking spectra, their primary processing and preservation, as well as for identifying substances using the built-in spectral database. The set-top boxes are easily replaced thanks to the design of the spectrometer, which has two mounting rails (metal guides in Fig. 1.1), onto which it is easy to install one of the attached set-top boxes.

The ATR set-top box (Fig. 1.3) is intended for the analysis of liquid, as well as soft or powdery samples that give a tight fit to the ATR crystal, on the surface of which the interaction of IR radiation with the sample material occurs. The set-top box includes interchangeable tables with diamond and zinc selenide crystals. The choice of crystal is determined by the practical task and the properties of the sample. To ensure the tight fit of solid samples, a clamping mechanism with a spring and a pointed tip is installed on the attachment. The set-top box has a built-in monitor for monitoring the enlarged area of analysis on the crystal surface. Crystal tables have the option of heating and maintaining the desired temperature in a wide range (up to 220 ° C). The heating is controlled by a separate module (Fig. 1.6).

The analysis of liquids can also be carried out using a special accessory (Fig. 1.4) containing replaceable cuvettes with a configurable optical path length (layer thickness). One of the cuvettes has 4 preset precisely calibrated thicknesses. The second allows you to smoothly adjust the thickness in a wide range – from several to tens of microns (microns).

The PRIZE device (Fig. 1.5) allows IR spectral analysis of solid and powdery samples in the mode of diffuse transmission (reflection), using the scattering effect of electromagnetic radiation to deliver light that has undergone interaction with the sample to the detector.

Figure 1. (1) Simex FT-803 infrared Fourier spectrometer with (2) a control computer and a set of set-top boxes: (3) a set-top box with replaceable pads equipped with ZnSe crystals or diamond, (4) cuvettes for liquid samples with an adjustable optical path length, (5) a PRIZE set-top box for analysis solid samples by diffuse reflection/transmission method, (6) temperature control unit for the ATR accessory ; (7) a coupler for connecting fiber-optic probes; (8) set-top box for analyzing films; (9) a gas cell.

The fiber probe coupler (Fig. 1.7) allows the analysis of liquids and solid elastic samples, such as biological tissue, using special probes with high-transmittance optical fiber in the IR region. Such immersible probes work according to the principle of ATR, but they allow analysis to be carried out at a considerable distance from the spectrometer, and without sampling. Fiber probe analysis is becoming widely used in online analysis, for example, in the flow, in the environment of an ongoing process (chemical, biotechnological, natural), as well as for on-site measurements, for example, for field tests or for spectral diagnostics during surgery.

The gas cell (Fig. 1.9) allows obtaining the spectra of gaseous samples injected into a special transparent chamber with an optical path length of 120 mm.

In general, a complete set of set-top boxes and fiber probes supplied with the Simex IR spectrometer allows to analyze almost any samples, including online measurements.

5.2. OceanOptics NIR spectrometer

Near-infrared vibrational spectroscopy (NIR spectroscopy) is the most common method of online analysis carried out using fiber-optic probes. The NIR spectra (780-2500 nm) contains mainly overtones and composite bands of fundamental vibrations of molecules and groups of atoms, such as -OH, -NH2 =NH, -CH3, =CH2, ⩾CH, etc., which are part of many organic and inorganic compounds. Unlike mid-range IR spectroscopy, measurements in the NIR region are facilitated by a number of factors: the transparency of plain and quartz glass (which means the ability to use standard optics and probes), low molar absorption coefficients that allow measurements to be carried out in conventional 1-, 5-, and 10-millimeter cuvettes, high light scattering by solid and dispersed samples, which allows measurements to be carried out by diffuse reflection, including without direct contact with the sample, at a distance. The wide absorption bands observed in this region overlap with each other, which suggests the use of chemometrics for quantitative and qualitative analysis from spectral data. There are several detectors for recording NIR spectra. In modern devices, diode-matrix detectors (DMD) are often used, which are a line of photodiodes for recording the intensities of dispersed light at various wavelengths.

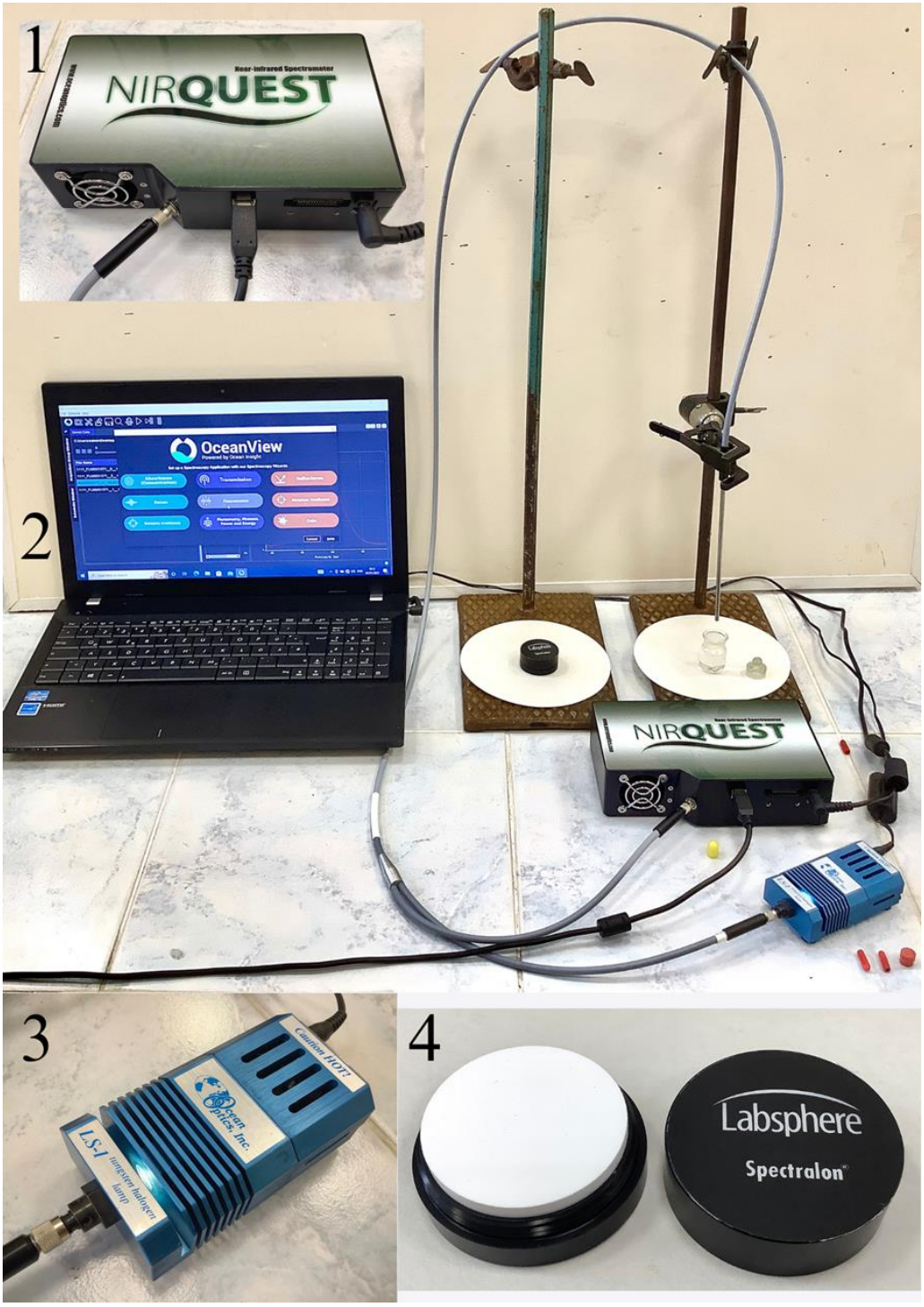

Figure 2. (1) NIRQuest near-infrared spectrometer (NIR) by Ocean Optics with a control computer and OceanView software, (2) a fiber probe for measurement in diffuse reflection mode, (3) an LS-1 light source using a tungsten-halogen lamp and (4) a standard Spectralon ® sample for measuring diffuse reflection spectra.

The NIRQuest spectrometer (OceanOptics Inc., USA), which is part of the multispectral laboratory (Fig. 2), is a striking representative of modern NIR spectrometers with DMD. The working area of NIRQuest (780-1800 nm) contains signals of the main groups and is universal. The spectrometer is structurally designed to work with fiber-optic equipment, primarily with probes (Fig. 2.2), therefore it has an input for a fiber-optic cable in the SMA standard (Fig. 2.1) and an external radiation source (Fig. 2.3). To measure solid samples in the diffuse reflection mode, a standard Spectralon type sample is attached (Figure 2.4), which can be used to obtain a comparison spectrum in absorption measurements. To work with cuvettes, instead of a fiber probe, an external cuvette compartment with a fiber-optic input-output will be required. The OceanView software supplied by the manufacturer allows you to control the device and receive digital spectra in one of the standard formats.

The NIRQuest spectrometer with accessories is a universal solution for conducting NIR spectral analysis both in the laboratory and in online measurement conditions.

5.3. Raman spectrometer

The measurement of the Raman scattering effect, also called Raman scattering, also refers to vibrational spectroscopy. The basis of RAMAN spectroscopy is the registration of weak radiation signals observed when a sample is irradiated with a laser or a powerful LED, usually in the visible region of light. Some of these signals are observed with a shift towards longer wavelengths, compared with the working wavelength of the excitation (Stokes shift). The difference between the observed frequencies and the laser frequency is called the Raman shift, expressed in frequency units – wave numbers (cm-1). The Raman shift is used as the horizontal (spectral) axis in RAMAN spectroscopy, while the vertical axis shows the absolute intensity of the recorded signals (photon count).

Due to the fact that Raman spectroscopy reflects energy transitions between vibrational levels of molecules and groups of atoms, the peaks of the same fundamental frequencies in the IR and RAMAN spectra are in the same positions on the wave number scale. However, their intensities can vary greatly to the point that some bands may not appear in one of the spectra (symmetry prohibition). This feature makes the mid-IR and RAMAN spectra complementary, that is, complementary to each other. That is, the use of these methods in tandem gives more structural information about the studied molecules than each method separately.

From a technical point of view, the measurement of the Raman spectrum is complicated by the low intensity of Raman signals, which are several orders of magnitude weaker than the IR absorption peaks in the IR range (if we compare these spectra in the initial photon counting units). In addition, the observation of RAMAN bands is often complicated by the scattering of light from an irradiating laser, and for many organic compounds by the imposition of a luminescence effect (fluorescence), which has a much higher intensity. The use of special optical filters and specially designed probes makes it possible to eliminate these effects. The detector of the RAMAN spectrometer must be highly sensitive and have a wide dynamic range. Due to these features, the RAMAN spectrometer has significant structural differences from the spectrometer for recording absorption spectra operating in the same spectral region (400-1100 nm) of visible and short-wave NIR radiation.

Figure 3. (Left) Ventana Raman spectrometer; (Left) Raman fiber optic probe.

The Ventana Raman spectrometer from Ocean Optics (Fig. 3), supplied as part of a multispectral laboratory, is a typical representative of its class of spectrometers designed for both laboratory measurements and online analysis of various processes and environments. The device is designed to use fiber-optic interfaces and has an SMA connector for signal registration. A typical measurement interface in this case is a specially designed fiber-optic probe for Raman spectroscopy. If necessary, measurements can be carried out in a cuvette (supplied separately) installed in a special holder with a fiber-optic input-output and special optics. Registration of the RAMAN spectrum of the sample in the cuvette is usually carried out at an angle of 90 ° to the exciting radiation, thus minimizing the effect of elastic scattering of laser radiation (the "tail" of the emission peak). A powerful laser (400 MW) with a working wavelength of 785 nm, supplied by a separate unit, serves as a source of excitation of RAMAN spectra.

The Raman spectrometer supplied as part of this multispectral laboratory is designed for recording Raman spectra of various samples, such as liquid mixtures containing organic and inorganic substances, biological tissue, powdered materials of various origin, etc. The resulting spectra can be used for both qualitative and quantitative analysis of mixtures. Measurement of IR and RAMAN spectra of the same sample can help to interpret complex organic molecules. The dependence of the RAMAN signal on the concentration is linear in a wide range of intensities [24]. Therefore, Raman spectra can (as well as IR absorption spectra) be used to construct chemometric models – calibration or classification.

5.4. Spectrometer for fluorescence analysis

To measure fluorescence spectra in this multispectral laboratory, a mini-Flame spectrometer from OceanOptics is supplied (Fig. 4.1) with a working area of 400-1100 nm (visible and short-wave NIR radiation). It can also be used to obtain absorption spectra in this spectral range. The spectrometer has a single input for optical fiber (SMA), that is, it assumes work with fiber-optic probes (or other fiber-optic interfaces). Fluorimetry is based on the measurement of a wide emission signal observed when irradiating some samples in the UV and visible regions. In this case, the peak of fluorescence is shifted to the long-wavelength region by comparison with the exciting radiation of the source (laser or LED). The method is often used in biology and biotechnology. Recently, it has become one of the main methods of online analysis of biotechnological processes [9,11,12].

The spectrometer is supplied with a computer with control software (Fig. 4.2), which allows to receive spectra and save them in one of the standard digital formats. The source of exciting radiation is a powerful LED included in the device with a working wavelength of 365 nm (Fig. 4.3), the brightness of which can be varied using the control unit (Fig. 4.4). Optical filters are used to suppress the scattered signal of the source (Fig. 4.5 and 4.6 at the corresponding wavelength).

The method of fluorescence spectroscopy can be used for multispectral measurements, for example, in combination with IR [12] or NIR spectroscopy [9]. The Flame mini spectrometer, despite its small size (it fits in the palm of your hand), gives the fluorescence and absorption spectra of sufficiently high quality for quantitative and qualitative analysis of various samples. The spectrometer with accessories can be used for both laboratory and online analysis of technological processes.

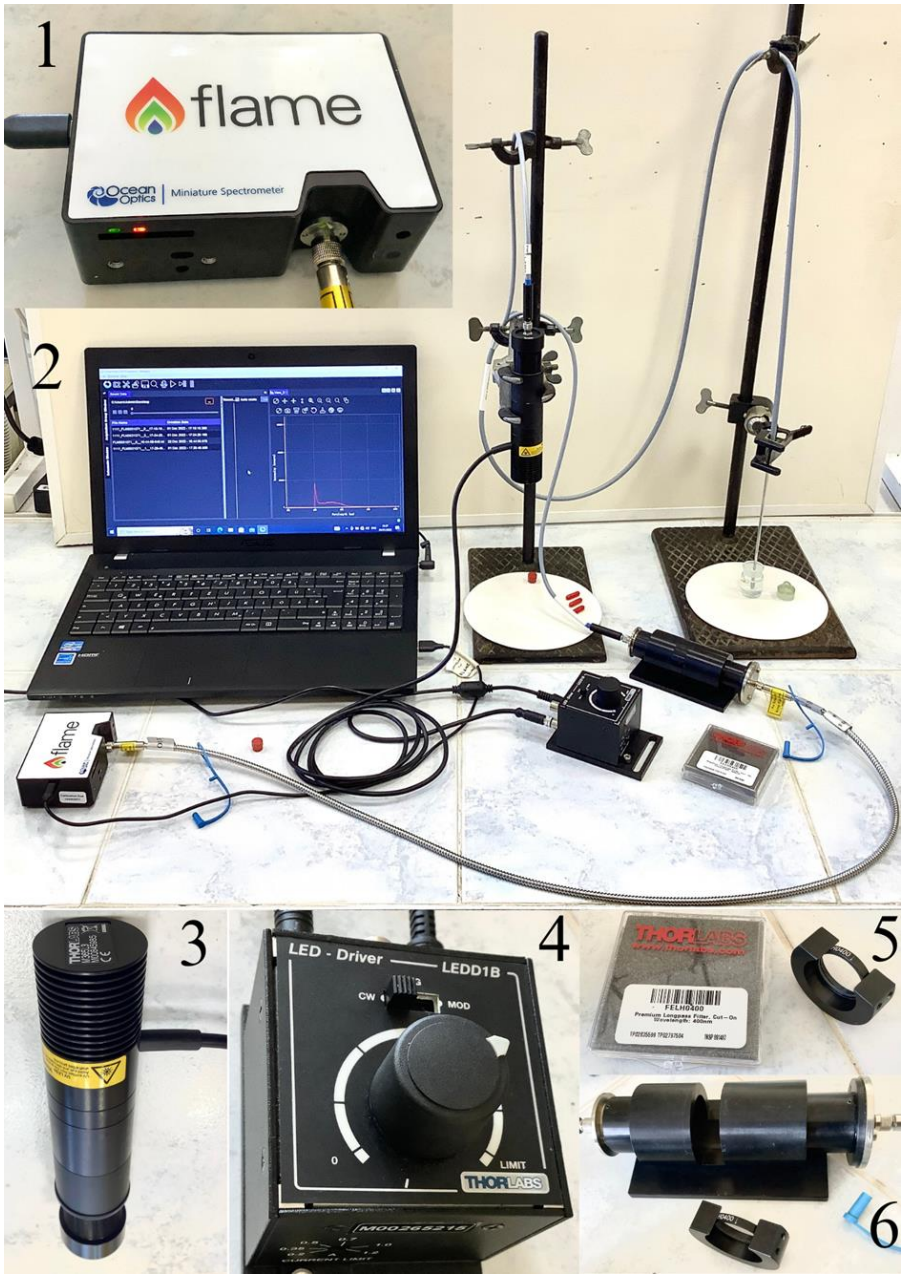

Figure 4. (1) Ocean Optics Flame fluorimetry spectrometer with fiber optic probe and accessories: (2) a computer with OceanView control software, (3) an exciting LED with (4) a control unit, and (5) an optical filter.

5.5. FSD-10 – UV and visible spectrometer

The mini-spectrometer FSD-10 of a wide range manufactured by "STC Fiber-Optic Devices" LLC is designed to register spectra in the UV and visible, as well as in the NIR spectral region (180-1080 nm). The spectrometer is equipped with a fiber-optic SMA input, which allows the use of fiber-optic probes for recording spectra. Reflex type probes with different characteristics for solving various analytical tasks are supplied with the device. The device can be equipped with a cuvette compartment for recording transmission spectra. The spectrometer has 2 built-in radiation sources (on demand): a diode and an incandescent lamp. To register UV spectra, the device includes an external radiation source - a mercury lamp. The device is powered by a USB cable connected to a computer (Fig. 5). Through it, the received spectral data is transmitted for processing. Control of the device and primary data processing is performed by its own instrument software FSD Soft v.6.

The UV spectral range (180-360 nm) contains signals of absorption of electronic transitions of most organic and inorganic molecules. The high molar absorption coefficients of molecules characteristic of this region allow the analysis of small concentrations of substances, at the level of tens of mg/l. The device can also be used to register fluorescence spectra as an alternative to the Flame mini spectrometer (see section 5.4).

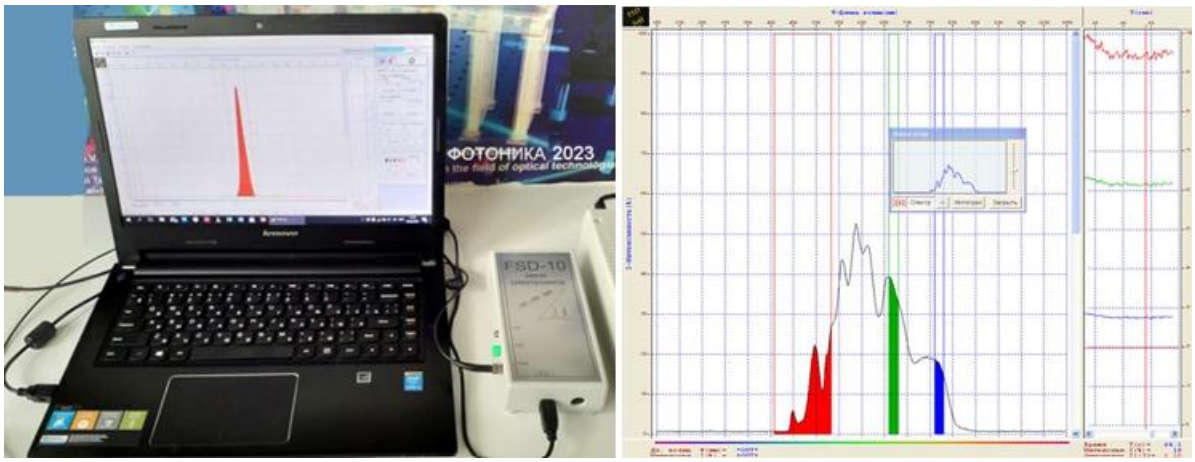

Figure 5. UV spectrometer FSD-10.

The FSD-10 spectrometer has a high spectral resolution (2.5 - 10 nm, depending on the spectral region), which allows it to be used in solving a wide variety of practical tasks, both in laboratory analysis and in online control at production or in the field.

5.6. Thermogravimetric humidity analyzer

Scales-moisture analyzer with sample size up to 120 g. The main method of operation of the hygrometer is heating and drying the sample with a halogen lamp, to a constant weight until the moisture evaporates. In this case, the moisture meter can compare the initial weight and the final one, thereby calculating the amount of moisture that was originally in the sample, as a percentage or as a mass. The device can be used to heat the sample up to 160 °C, and then can hold this temperature with an accuracy of + / - 1 ° C. In this controlled environment at a constant temperature, which will increase the drying speed, the sample can be dried without damaging it.

In addition to measuring humidity, the device can also simply weigh any sample weighing up to 120 g with an accuracy of ± 0.001 g. When working with a moisture meter, almost any type of sample can

be used, from food to plastics and semi-solid substances. Maximum drying time: 10 hours; drying resolution: 0.01%; weighing resolution: 0.001 g; weighing accuracy: ± 0.003 g; analysis accuracy (> 5 g): ± 0.04%; heater: two halogen lamps of 75 mm; overall dimensions: 290 x 185 x 170 mm; weight of the device: ~ 3.8 kg.

Figure 6. Thermogravimetric humidity analyzer PCE-MB 120 C.

5.7. Software tptcloud.com

Software for creating predictive models based on spectral data using chemometrics methods and algorithms is a necessary component of a modern analytical laboratory.

Within the framework of the multispectral laboratory, it is proposed to use the unique chemometric software TPT-cloud (Fig. 7) [25]. A modern "cloud" solution for analyzing spectroscopy data has several advantages over traditional software running locally. The application does not require installation; it is accessible via any Internet browser from a computer, tablet, or smartphone. For analysis, the data is uploaded to a special remote server, where they are processed, and models are created. A centralized repository of data and models organized into projects with the possibility of assigning user roles facilitates teamwork with them. If necessary, experts can be connected to the work, as well as additional design capacities are allocated. The latter may be relevant when analyzing large amounts of information ("big data").

The "cloud" organization is a necessary element of distributed analyzers, such as optical multi-sensor systems. The presence of hundreds and thousands of similar devices operating far from each other makes the usual installation and updating of the model from a local computer impossible.