Automatic Standard of Refraction Index Measurement

The refractive index is an important characteristic of a substance found in any of three aggregate states, i.e., solid, liquid, and gas. High accurate refractometric measurements are required in the optical industry in order to increase the quality and reliability of optical instrument, and in the chemical industry for monitoring substance composition. Refractometry is used extensively in the food industry in the production of sugar, juices, wine, alcoholic drinks, beer, fats, oils, etc., and also in pharmacological plants, in medical establishments and pharmacies carrying out analyses and monitoring the quality of medicinal preparations.

System requirements

Angles measurements requirements

Estimates shows that to measure a refractive index with an error of ±1·10-6 requires primarily the measurement of prism angles and deviations angle at the level of tenths of an angular second.

High Precision Dynamic Goniometer-Spectrometer is based on a dynamic goniometer containing a ring laser, which provides the necessary accuracy characteristics in angle measurement (better then ± 0,4”).

However, there is a difference from an ordinary goniometer in that all the measurements with a goniometer containing a ring laser are made under dynamic conditions with the object stage rotating continuously. That imposes limitations on the minimum deviation method. A novel method has been developed to determine εmin automatically.

The essence of this method is that one first performs a series of automatic measurements of the deviation angle ε with various angular positions of the prism relative to the immobile beam, i.e., for various angles of incidence φi, i = 0, 1, 2, ..., K, where K is the total number of measurements. Then the experimental ε(φi) is fitted to a polynomial of second degree and εmin is calculated from it.

Sample quality

The following are the main error sources in measuring a refractive index: error in measuring angles on the goniometer, errors of manufacture in the working measure (prism), namely deviation from perpendicularity and planarity in the working surfaces.

The pyramidal error consists in the working faces not being perpendicular to the base of the prism. As a result, a beam reflected from a face is deviated with respect to the optic axis of the collimator in the vertical direction, which leads to vignetting in the reflected beam and influences its intensity. The effects of this pyramidal error in the prism faces on the measurement accuracy decrease as the refractive index increases. In the standard equipment, the prism pyramidality should not exceed 5”.

Nonplanar working faces lead to the refracting angle of the prism being different from the nominal value. Consequently, the angle of deviation will also differ from the calculated value. Nonplanarity tests have shown that a deviation of λ/10 leads to the angle changing by 0,4”.

Environmental conditions

The air and sample temperatures should be kept at constant values during the measurement, while the temperature of the prism and the surrounding parameters (air temperature and atmosphere pressure) must be measured with high accuracy.

Using A further feature of such a Goniometer is that it can perform measurements automatically without the presence of the operator in the measurement zone, i.e., the measurements may be made remotely. This also serves to resolve the problems of thermal stabilization for the measurement volume.

The necessary part of the measuring apparatus is placed in an insulated chamber, while the other units, which produce the main heat (recording equipment, spectral lamps, power supplies, and so on) are kept outside it.

Light sources

To measure refractive index on the different wave lengths we apply different light sources – He-Ne laser, Ar-Kr laser with changing wave lengths.

Detector

In High Precision Dynamic Goniometer-Spectrometer applied integral detector (for ex. photomultiplier tube) instead matrix detector (for ex. CCD). This allows to expand spectral range from Ultra-Violet to Infra-Red using different integral (point) detectors (for ex. PbS detectors for IR and photomultiplier tube for visual and deep UV spectral ranges).

System description

Hardware

The High precision dynamic goniometer-spectrometer includes the following:

1) Dynamic goniometer with ring laser for reproducing and storing the unit of refractive index for solid and liquid substances and for transmitting the unit;
2) a climatic chamber with feedback thermal stabilization and a multichannel digital thermometer with separate temperature sensors;
3) a barometer to measure the atmospheric pressure in the chamber;
4) a hygrometer to measure the humidity of the air in the chamber;
5) a system for acquiring and processing the data on the basis of a personal computer.

The main measuring equipment is located within a special chamber of volume 18 m3. The walls, floor, and ceiling of the chamber are made of special thermal insulation panels faced by sheet metal to reduce the temperature gradients. The chamber has a thermally insulated door for access and a window for visual monitoring. It is equipped with systems for humidifying and cleaning the air, and also with the hygrometer and barometer.

A basic element in the primary standard suite is the goniometric spectrometer, which provides a standard error in angular measurements of 0,03” and a systematic error not more than 0,2”.

The light source is provided either by an He–Ne laser and/or Ar-Kr laser. The radiation enters the climatic chamber via an optical fiber bundle.

Constant temperature within the chamber is maintained by a precision air conditioning system: a split system of inverter type. It provides temperatures of the air constant to
20,0 ± 0,1°C in the internal volume with active thermal stabilization. This system is disconnected during the measurements, which last several minutes.

To reduce the temperature gradients, the climatic chamber is located in a room also equipped with an air-conditioning system that maintains a temperature of 20 ± 1°C.

The measurements are completely automated. The operator remains outside the chamber during the measurements. All the heat-producing units in the monitoring and measuring equipment, power supply sources, sources of optical radiation, and the processing computer are placed outside the chamber.

In precision measurements, a optical bench equipment provides reliable protection of the refractometer from the vibration. Therefore, the High Precision Dynamic Goniometer-Spectrometer can be located in any building without special measures for vibrational protection.

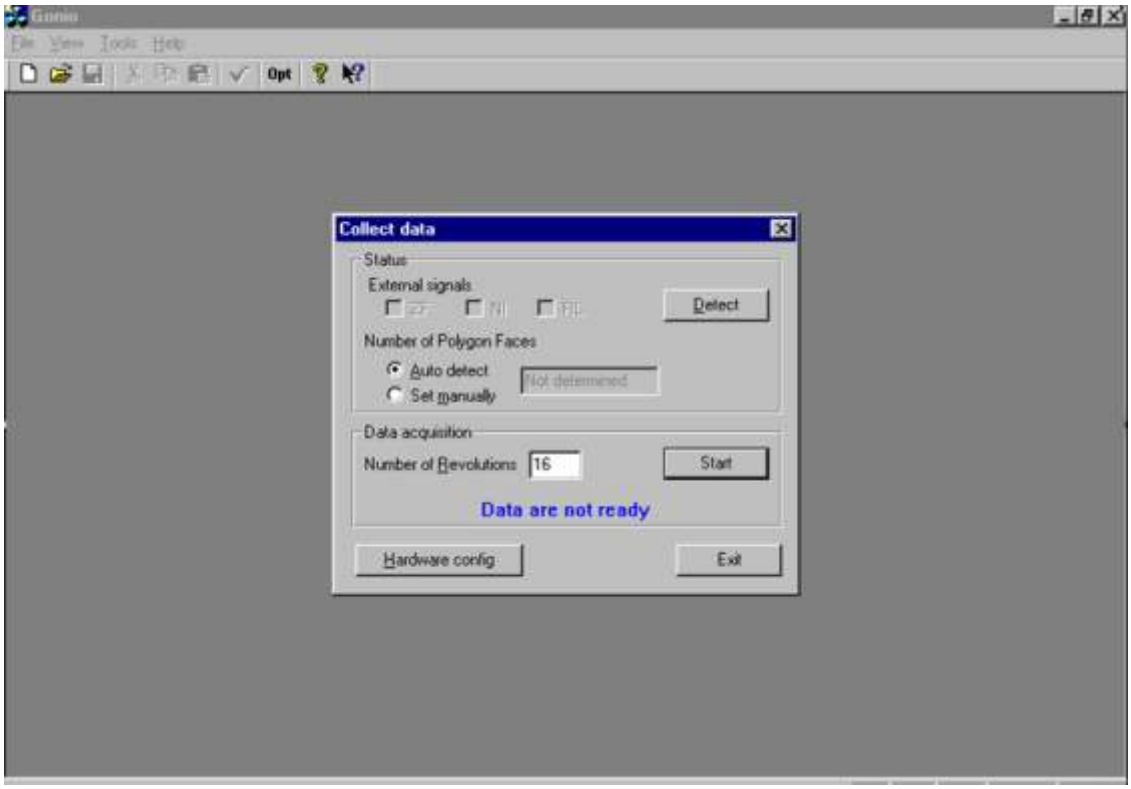
The chamber is equipped with a multichannel digital thermometer for measuring the temperatures of the standard measure and the air within the chamber. The limit to the permissible error in the thermometer measurements is ± 2 mK. The temperature measurement system includes three separate detectors, two of which are placed in the air near the measurement volume and one contact detector is placed on the sample. The readings from all three detectors pass in real time to the computer and are displayed on the screen.

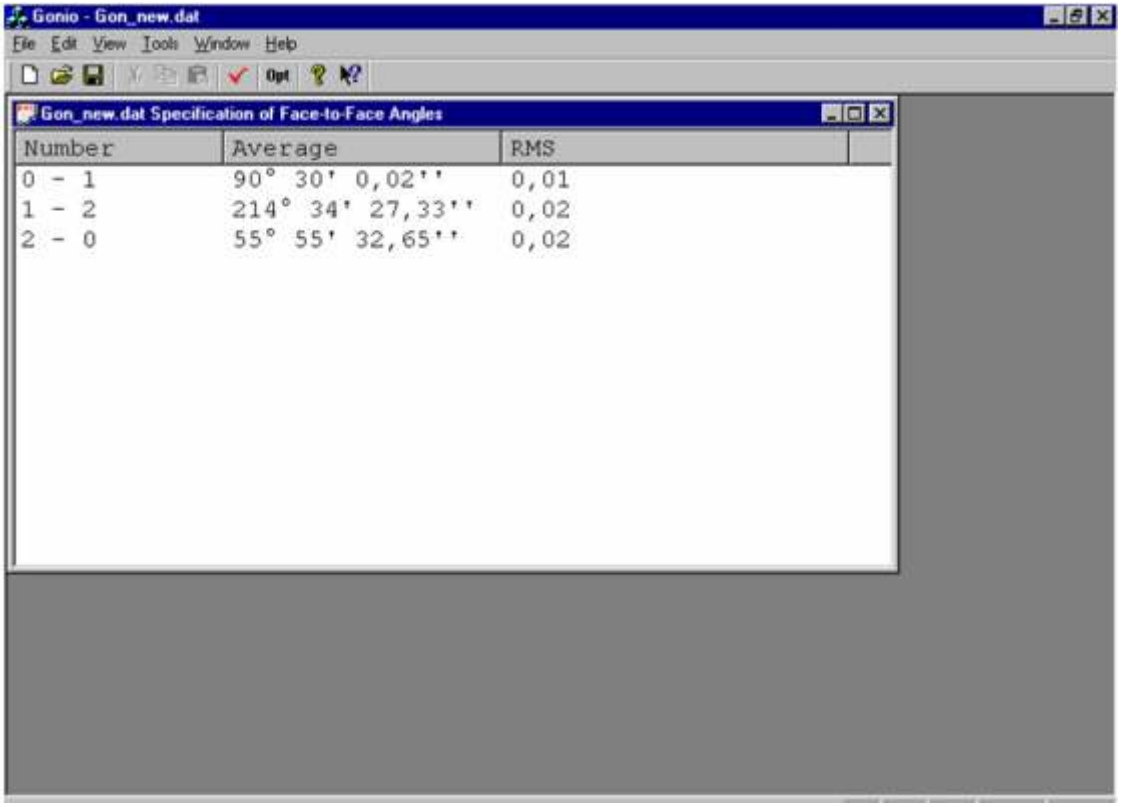
The data-acquisition and processing system consists of a personal computer, interfaces for collecting the measurement data from the spectrometer and the thermometers, together with software for processing the angular measurements and calculating the refractive index.

Software

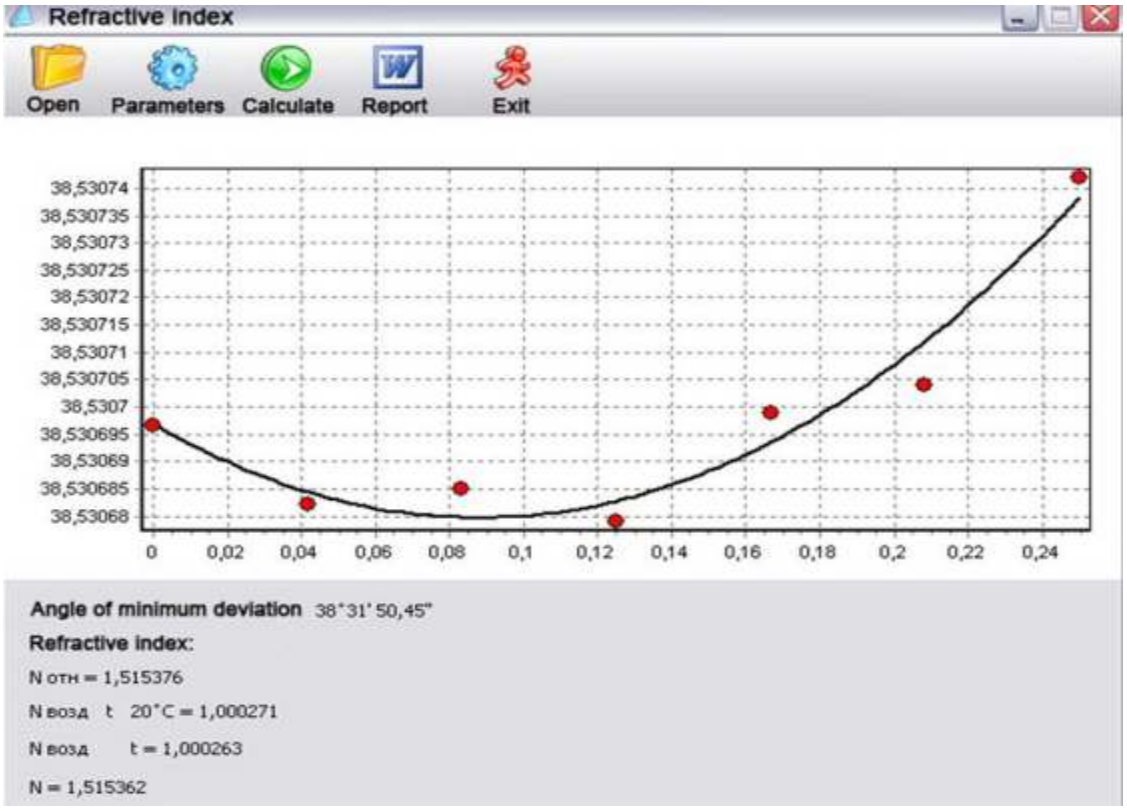
All measurement operations carried out from personnel computer using original software. Software consists of two parts: software for angle measurements and software for refractive index calculation.

Angle measurements carried out in automatic mode.





The second part of software applied for refractive index calculations. Using measured data special algorithm calculates angle of minimum deviation and refractive index of the sample.



Specification	
Apex angle measurement accuracy	0,4”
Refractive index measurement range	1,0 … 3,0
Light sources	He-Ne laser, Ar-Kr laser
Detector	PbS, Photomultiplier tube, solid-state photodiode
Spectral range	193 nm … 2300 nm
Refractive index measurements accuracy:	
Standard uncertainty type A	$1{,}0 \cdot 10^{-6}$
Standard uncertainty type B	$1{,}1 \cdot 10^{-6}$
Total combined standard uncertainty	$1{,}5 \cdot 10^{-6}$
Expanded uncertainty (*k*=2)	$3{,}0 \cdot 10^{-6}$

Advantages

- Fully automated remote measurement method
- Application null-indicator instead autocollimator
- Expanded spectral range from DUV to IR through the use of:
  - integral detector instead matrix detector
  - mirror optic elements (mirror null-indicator, two-sided mirror)
- Increase accuracy by averaging the results of angle measurement and data approximatio

References

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