

Original Contribution

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METHOD AND SPECTROPHOTOMETRIC EQUIPMENT FOR WATER RESEARCH

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Abstrakt: Modem measurement equipment must feature high measurement precision, swift operation, high sensitivity and reliability. The designing of such equipment includes coordination of the operation of optical, mechanical, and electronic assemblies and units, introduction of microprocessor systems and communication interface equipment etc. Therefore, the design, implementation, calibration etc. requirements for such equipment feature an ever growing variety strictness.

Key words: spectrophotometer, water characteristics

While implementing its research and research- application tasks, the Space Research Institute and the Central Solar-Terrestrial Influences Laboratory at the Bulgarian Academy of Sciences collected experience in this field, particularly concerning various-purpose spectrophotometric equipment [1-6]. Here, particular attention is paid to the equipment's automation, application of discrete positioning systems and feedback circuits.

An overview of the spectral analysis equipment and techniques [7] defines the following major requirements for the designed instrumentation:

1. Spectral range varying from 275 to 720 nm, which necessitates two light sources, one for the ultraviolet and one for the visible part of the optic spectrum, providing for their switching over in the operation process. The required positioning precision for a definite wavelength is 1,0 nm.

2. Provision of high monochromeness. The monochromer's spectral bandwidth depends on the width of the input and output prism slot. To provide for reproducable wavelength \pm 1,0 nm, the input and output prism slot must be regulated withinn the range of 0,01 to 1,0 mm.

3. Uniform sensitivity throughout the overall spectral range. The photosensitive receiver must provide uniform sensitivity throughout the equipment's overall spectral range, which is accomplished by using a programmable amplifier and a compensation system.

4. Four-digit indication. It provides data for the positioning along the

wavelength and the signal amplitude at the photoreceiver's output.

5. The use of dedicated microprocessor systems provides for the operations' automation during the calibration and measurement process.

Here, a spectrophotometer is presented intended to measure water characteristics. It was designed on a concrete technical mission and is intended for mass use.

The overall block diagram is shown in Fig. 1. The main blocks are: 1 - optic-spectral block, 2 - cuvette-bearing block, 3 - light-protection unit, 4 - electronic-registration block, 5 - microprocessor system, 6 - electro-mechanical system, 7 - energy sources' block, 8 - power-supply block, 9 - printing unit, 10 - indicating block with digital and monitor indication, 11 - control panel, 12 - storage unit.



In Fig. 2, the operational diagram of the Spectrophotometer for Measurement of Water Characteristics is shown. The two sources of visible - 1 and ultraviolet - 2 emissions are positioned in front of concave glass 3. Glass 4 is positioned along the optical way between glass 3, slot 5, and concave glass 6. Prism 7 is used as dispersing element. Along the optical; axis between output slot 5 and photo-electronic multiplier (PEM) 10, cuvette-bearer 8 with cuvettes, and mechanical blind 9 are positioned. Through amplifier-former 11 and analogue-to-digital converter (AGC) the electrical output 12 of PEM 10 is connected to microprocessor system (MPS) 13. Electric motor 15 is connected mechanically to position sensor 14 and concave glass 3, and electrically through control unit 16 to MPS 13. Dispersing prism 7 is coupled with sine mechanism 17, electric motor 18 and photo-screen converter 19, the latter being connected through control block 20 to MPS 13. Slot 5 and convex glass 6 are coupled with tuning assemblies 21 µ 22. Cuvette-bearer 8 is connected mechanically through reducer 23 and position

sensor 24 to electric motor 25. Directly to MPS 13, mechanical shutter 9 is connected. The safety block is connected, on the one hand, to low-voltage and high voltage blocks 30 and 32, accordingly, and on the other hand, to power block 31. All of them are stabilized to provide for the equipment's normal operation, including and the input power supply - block 29.

The spectrophotometer's water analysis proceeds in the following way.

Upon charging cuvette-bearer 8 with the water-sample-containing cuvettes, through reducer 23, the cuvette-bearer 8 shifts and positions position sensor 24, motor 25, control unit 26, and control panel 27. Then, assisted by motor 15, position sensor 14, and control unit 16, glass 3 is swivelled towards ultraviolet emission source 1 (or visible emission 2, accordingly), enabling the measurement process to start. The position of prism 7 is fixed by MPS 13, sine mechanism 17, motor 18, photo-screen converter 19, and control unit 20. The emission flux aimed at the photo-cathode of PEM 10 is let through mechanical shutter 9, controlled by block 28 only provided a specific cuvette is fixed at the optic way. The output information signal from PEM 10, amplified and formed by block 11, is fed to the input of ADC 12, connected through a data bus to MPS 13.

The course of the optical systems rays is shown in Fig. 3. The optical system is tuned using mechanical assembles 21 and 22 in laboratory conditions only if so required.



Fig. 2 Operational diagram



Fig. 3 Optical sistems

By the use of spectral techniques the designed spectrophotometer can reveal available contents of:

- 6. Sulphates
- 7. Silicon acid
- 8. Nitrites
- 9. Nitrates
- 10. Copper
- 11. Iron
- 12. Phosphates
- 13. Ammonia

The theoretical resolution of prism spectral equipment is:

$$R = \frac{\lambda}{\delta_{\lambda}} = t \frac{dn}{d\lambda} , \qquad (1)$$

where: κ - the average wavelength value of two still separable spectral lines; dk - the difference of the wavelength values; t - the slot's effective width.

The spectral equipment's actual resolution depends on the equipment's function and its width:

$$\delta_{\lambda} = \alpha_{ap} \, \frac{\alpha_{\lambda}}{dl} \, , \tag{2}$$

where: δ_{λ} - the interval between wavelengths recorded separately;

 a_{ap} - the width of the equipment's function.

In case of very thin slot, the analytical expression of the equipment function is the

following:

$$a(l) = \frac{1}{S_0} \left(\frac{\sin \frac{nl}{S_0}}{\frac{nl}{S_0}} \right)$$
(3)

where: l - coordinate of the spectrum plane;

 S_0 - normal slot width:

$$S_0 = \frac{\lambda f}{D} ,$$

where: λ - wavelength;

f- lens focal distance;

D - lens diameter.

The maximal value of the equipment function is:

$$a(l)_{\max} = \frac{D}{\lambda f} . \tag{4}$$

The values of the equipment function at level 0,5 a (where the equipment function's width a_{ap} is determined) and values of resolution *R* for the operation wavelengths are shown on Table 1.

Table 1

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λ[nm]	$0,5a(l)_{\rm max}.10^5$	a_{ap}	<i>R'</i> [nm]
215	4,3046944570	1,0.10 ⁶	0,0010362
225	4,4219609245	1,0.10 ⁶	0,0012986
257	3,577698060	1,2.10 ⁻⁶	0,0028076
275	2,840878	1,4.10 ⁶	0,0042971
313	3,1481270646	1,4.10-6	0,0060493
325	2,5711060664	1,6.10^	0,0089136
335	2,6386689657	1,6.10 ⁴⁵	0,0113757
395	2,4229348593	1,8.10 ⁶	0,0185471
405	2,4566494025	1,8.10 ⁻⁶	0,0199888
420	2,11586228	2,0.10-6	0,0247066
435	2,1687216229	2,0.10'6	0,0273224
440	2,1839196212	2,0.10-6	0,0282485
450	2,2109844623	2,0.10 ⁶	0,0300978
510	1,7789542116	2,4.1 O' ⁶	0,0510095
530	1,8239440553	2,4.10"	0,056872
550	1,6366437915	$2,6.10^{-6}$	0,0675324
570	1,6764350641	$2,6.10^{-6}$	0,074074
578	1,6899596741	2,6.10 ⁶	0,076923
600	1,5338897164	$2,8.10^{-6}$	0,0910569
625	1.5727224561	$2.8.10^{6}$	0.101083

The Spectrophotometer for Measurement of Water Characteristics, described briefly above, which was developed at tie Space Research Institute of the Bulgarian Academy of Sciences, was implemented successfully at the *Shoumensko Pivo* Brewery.

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