# DETERMINATION OF HEAVY METALS IN ENVIRONMENTAL WATER BY TOTAL REFLECTION X-RAY FLUORESCENCE METHOD USING OPTIMIZED ROENTGEN OPTICS CUT-OFF FILTER

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The total reflection X-ray fluorescence (TXRF) method of analyzing elemental contents is based on small angle irradiation of thin samples placed on a total reflected backing with a narrow photon beam. Two instrumental problems are to be solved here. The first one is to form the narrow beam with a small angular deviation.

To reach a sufficient intensity of the beam one must use powerful X-ray generators producing also high energy bremsstrahlung. The total reflection critical angle for the high energy exciting spectrum tail is very small. The result is elastic and inelastic scattering of this part of the spectrum causing the increase of the background. The second problem is to reject this high energy tail. One can use a cut-off filter which improves the spectral distribution of the exciting radiation by filtering out the unwanted high-energy components of the bremsstrahlung.

A new roentgen optic cut-off adjustable filter for total-reflected X-rays has been developed in LNPI. The main advantage of the filter is rather large input solid angle, 7—10 times as large as the published instruments have [1,2]. This became possible due to using a bent reflecting mirror instead of a plain one usually used. The law of bending is a logarithmic spiral. In this case all the beams beginning in the focus of the spiral are falling on the mirror surface at the same angle. This angle in our case is just the critical angle of the total reflection.

The bent mirror works also as an effective cut-off filter because X-rays with energy higher than critical one (corresponding to the total reflection critical angle) are not reflected but scattered when falling on the mirror surface and they never reach the target with a sample. The critical cut-off energy can be changed with variation of the logarithmic spiral curvature.

The filter realized has a mirror of 123 *mm* length bent as it was described above. The mirror is made of polished glass covered with nickel using evaporating procedure. The distance between the X-ray source

position and the center of the sample equals to 280 *mm*, the input solid angle is 16.5 min of arc, the output one is 5.5 min of arc. The input slit of the filter has size of $10\times0.01\ mm^2$. The cross-section of the output beam is $20\times0.18\ mm^2$.

The backing of the sample is also made of the same glass but without the nickel cover.

On the basis of the filter an instrument is designed for the determination of concentration of heavy metals in environmental waters. Copper and molybdenum X-ray tubes (30 *kV*, 10 *mA*) with linear $11\times1.2\ mm^2$ anode were used as an X-ray source.

The microsamples are prepared by evaporating several calibrated drops of water of interest on the surface of a reflecting glass in the form of a spot of 15 *mm* diameter.

Special standard samples were prepared for calibration of the system. They were made of standard solution containing chemical elements in the range of atomic numbers 19 through 39 with equal concentration 1 *mcg/ml*.

The peaks on the spectrum show a regular behaviour increasing with atomic number. The area of every peak can be expressed as

$$A^Z_{q,i} = Q \cdot \Omega \cdot \frac{S^Z_q - 1}{S^z_q} \cdot \tau^z_q \cdot \omega_{q,i} \cdot \varepsilon(E^z_{q,i}) \cdot C^Z,$$

where Q — a constancy proportional to the intensity of X-ray source, $\Omega$ — geometry factor, $S^z_q$ — g-shell edge step ratio, $\tau^Z_q$ — total cross section for q-shell of an element, $\omega_{q,i}$ — fluorescence yield for q-shell, $\varepsilon(E)$ — energy dependence of the detector efficiency, $C^Z$ — concentration of the element Z.

An efficiency curve was measured using calibrated $\gamma$-ray sources. All the atomic data was taken from the X-Ray Data File XRDF [3].

The constancy Q must be independent of Z if the mathematical model of measuring process is right and there are no chemical elements escaping during the sample preparation. In table 1 the calculated values of Q for several elements are presented. All the values are normalized relative Q for Ga chosen as an internal standard.

Table 2 presents as an example the results of measurements of metals in waste water. The sensitivity of such measurements is of order of mcg/ml.

*Table 1*

| Element | $Q/Q_{Ga}$ | Element | $Q/Q_{Ga}$ |
|---|---|---|---|
| Ti-19 | 1.190 ± 0.040 | Ni-28 | 1.061 ± 0.016 |
| V-23 | 1.050 ± 0.024 | Cu-29 | 1.062 ± 0.014 |
| Cr-24 | 1.025 ± 0.025 | Zn-30 | 0.887 ± 0.018 |
| Mnb-25 | 1.021 ± 0.015 | Ga-31 | 1 |
| Fe-26 | 1.060 ± 0.021 | Rd-37 | 0.883 ± 0.015 |
| Co-27 | 1.019 ± 0.014 | Y-39 | 0.635 ± 0.013 |

*Table 2*

| | SL2-3 | |
|---|---|---|
| Mn | 0.023 ± 0.010 | 0.028 ± 0.015 |
| Fe | 0.050 ± 0.008 | 0.174 ± 0.012 |
| Ni | 0.033 ± 0.008 | 0.053 ± 0.008 |
| Cu | 0.025 ± 0.007 | 0.055 ± 0.007 |
| Zn | ≤ 0.021 | 0.061 ± 0.006 |
| Ga | 1. | 1. |
| Br | 0.052 ± 0.004 | 0.029 ± 0.003 |
| Sr | 0.042 ± 0.004 | |

**References**


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