QUANTITATIVE DETERMINATION OF BRONZE CONSTITUENTS BY THIN-LAYER CHROMATOGRAPHY AFTER ANODIC SAMPLING

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In this work Cu-Sn alloys were investigated by thin-layer chromatography after anodic dissolution of samples. Method involves anodic sampling, TLC separation on precoated cellulose layers and quantification by videodensitometry.

Thin-layer chromatography with anodic sampling is almost non-destructive method of metal determination. Sampling is preformed *in situ* with minor visible destruction. It does not require any special sample pretreatment.

For reproducible quantification of the separated and identified metal constituents after anodic sampling, it is necessary to determine and strictly obey the conditions of anodic dissolution. The quantity of metal constituents in a chromatographic spot depends upon sampling duration, volume of electrolyte applicated and dissolution potential. Sampling conditions applicated were: potential difference (4,5 V d.c.), volume of electrolyte in the electrode fiber (20 μL), duration of sampling (20 s) and duration of the solution transfer to the layer (10s).

As a mobile phase in TLC separations mixture of 2-buthanol, HCl and water was chosen (60:35:5, v/v). After development the spots were visualized by spraying of chromatograms with quercetine and dimethylglyoxim reagent solutions.

Calibration standards were certified spectrographic bronze reference materials and/or laboratory analyzed bronze samples, in range for each element of interest: 79-100 % for Cu, 7-21% for Sn and 1-10% for Pb.

The chromatograms were captured under white light by highly sensitive 3CCD color video camera; image acquisition, processing and archiving were controlled by Video Store 2 documentation software and data evaluation was performed from the stored chromatograms via Vista Scan program. The method was validated by determination of precision, limit of detection (LOD) and limit of quantification (LOQ).

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