VALIDATION OF UV-SPECTROPHOTOMETRIC METHOD OF DOXYLAMINE QUANTITATIVE DETERMINATION IN BLOOD: PRECISION

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The purpose of this paper is to test the approaches to the determination procedure and acceptability estimation of precision when validating UV-spectrophotometric methods of quantitative determination for forensic and toxicological analysis, which have been offered previously, by the example of UV-spectrophotometric method of doxylamine quantitative determination in blood.

Precision of UV-spectrophotometric method of doxylamine quantitative determination in blood has been estimated according to the following order:

- precision confirmation of the method is carried out in two directions – by model solutions (without matrix) and by matrix samples;
- verification of the method precision by model solutions is carried out by calculation of their concentrations using the respective linear dependence;
- estimation of the method precision by matrix samples is carried out at two levels – within-run and between-run – using calibration and model samples;
- determination of within-run precision is carried out in the way of calculating the concentrations of calibration samples for each run by individual values of absorbance using the linear dependence obtained for this run;
- determination of between-run precision is carried out in three stages – by calculation of the difference between the mean «found/spiked» values in different days, and also by calculation of the concentrations of model samples and mean concentrations of calibration samples using the linear dependence obtained by the mean values of parallel runs.

It is noted that the methods with preliminary TLC-purification have better intermediate precision and worse repeatability, than the methods without TLC-purification, that is explained by carrying out the additional stages of sample preparation, which worsen naturally within-run precision (estimates, in the first place, random error associated with sample preparation), but level the influence of changing the origin source of matrix on the results of analysis due to sample purification of high quality (improve between-run precision).

Thus, the offered approaches to precision determination have been tested by the example of UV-spectrophotometric method of doxylamine quantitative determination in blood. The results have shown the adequacy of formed approaches.