VALIDATION OF SPECTROPHOTOMETRIC TECHNIQUES FOR QUANTITATIVE DETERMINATION OF PREDNISOLONE

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Prednisolone is a medical drug of corticosteroids group marked by a relatively active pharmacological effect, and so requires meticulous quality controlling. The Quantitative determination of prednisolone may be done by spectrophotometric technique based on Standard Method (SM), as recommended by the International Pharmacopeia; or, by the Absorption Index Method (AIM) as recommended by the European Pharmacopoeia and the State Pharmacopoeia of Ukraine (SPU). Since the named methods are relatively suitable for application in laboratory conditions, we decided to validate those methods and used the validation results for subsequent development of a technique for quantitative determination of prednisolone in officinal medical drugs.

Validation was performed by use of the spectrophotometers Specord-200 and Specol-1500. The equipment, chemical reagents and volumetric glassware were compliant with SPU requirements. Statistic processing of data was done on the basis of standardized procedures for the minimal range 80-120% of technique application, for 9 precise concentration values within the entire range. When assessing linear dependence we determined that at 120% concentration the spectrum properties undergo changing, where by linearity is not observed; therefore, having completed the assessment of sample homogeneity we decided to conduct all further estimations for 8 concentration values in the range of 80-115%.

As part of determining the precision of technique, we calculated a one-sided confidence interval for the probability of 95%, which totaled in 1.78 and 1.35 for the MS (Specord-200 and S pecol-1500 respectively), and in 1.86 and 1.49 respectively for the SPU; the above values exceed the value of maximum acceptable uncertainty for analysis. However, the values of systematic error in MS are relatively low (δ=0.02 and δ=0.20), contrasted by those of SPU (δ=4.50 and δ=9.78). The above clearly proves that the MS for prednisolone was prone for in correct results, which prevents the MS from being used in routine analysis. Therefore, all further measurements (robustness and interlaboratory accuracy) were only done for the MS. Adding 2 drops of 0.01 M solution of NaOH or HCl gave the value $\Delta_{pH,}$%=0.31, which did not exceed the value of maximum systematic error (δmax=0.31). The value of technique reproducibility was also appropriate for applying the technique in various laboratories and on various types of equipment, which is compliant with the SPU requirements.