26th Congress of SCTM

Sept. 20-23, 2023, Metropol Lake Resort, Ohrid, N. Macedonia

Development of Analytical Method for Quantitative Determination of Propyphenazone Residues on Manufacturing Equipment

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The analytical method is one of the deciding factors in establishing the cleanliness of pharmaceutical manufacturing equipment.[1] The purpose of this research is to develop and validate a sensitive and accurate HPLC method, suitable for quantitative determination of propyphenazone residues on manufacturing equipment.

The method has been validated to show specificity, linearity and range, accuracy, precision, the limit of quantification (LOQ) and limit of detection (LOD), as per ICH guideline Validation of analytical procedures: Text and Methodology Q2(R1).[2,3] Accuracy is measured through the recovery of samples from the equipment surface and extraction of the recovered samples into a testing solution. It is reported as % recovery of the amount of analyte in the recovered samples measured against the amount of analyte spiked onto the sample recovery surface. In our case, the recovery for propyphenazone was 98.59%. The precision of the method was evaluated using six samples and the result for RSD was 0.6%. Using diluent, blank, standard and spiked samples it was determined that the method was specific. It exhibited good linearity between the responses of propyphenazone related to the concentrations of standard with correlation coefficient r=1.00. The LOD and LOQ were determined at a signal-to-noise ratio of 3:1 and 10:1, respectively. The limit of detection was 0.03 µg/mL and the limit of quantification was 0.10 µg/mL.

From the obtained results, it was concluded that the method is appropriate for determining the amount of propyphenazone residues on manufacturing equipment. In view of the obtained data, the developed method can be applied for routine control of pharmaceutical equipment cleanliness.

Keywords: method validation, method development, equipment cleanliness, recovery

References

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