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DEVELOPMENT AND VALIDATION OF SAMPLING PROCEDURES AND QUANTITATIVE DETERMINATION HPLC METHODS OF ACTIVE PHARMACEUTICAL INGREDIENT-ALPRAZOLAM RESIDUES ON PHARMACEUTICAL TECHNOLOGICAL EQUIPMENT

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The aim of this study was to validate direct swab and indirect rinse sampling procedures and demonstrate the applicability of developed HPLC method for quantitative estimation of residues of active pharmaceutical ingredient-alprazolam residues as a high potent and practically insoluble compound in water in cleaning control samples collected from pharmaceutical equipment surfaces after manufacturing of alprazolam 1 mg uncoated tablets. The swab and rinse sampling procedures were developed in order to obtain a suitable and good recovery (> 90%). The sampling procedures were qualified in respect to the validation parameters. The known amounts of alprazolam at three different concentration levels are spiked onto representative surfaces, which are disinfected and cleaned, then dried, sampled using swabbing and rinsing and analysed using the validated HPLC method. Additionally, the robustness of sampling procedures was assessed. For swab sampling the surface (sampling area-25 cm^2) was successively wiped with one micro polyester swab (3×2.5×10 mm) moistened with diluent methanol. The influence of swab material on quantitative determination of alprazolam was checked as well. The method for quantitative determination of alprazolam residues was developed using LC system Ag 1260 Infinity and Prodigy C8(2) 250×4.0 mm, 5 µm column with a mobile phase: a mixture of methanol, phosphate buffer pH 3.0 and acetonitrile (10:45:45 v/v); the flow rate-1.4 mL/min; the detector wavelength-220 nm; the injection volume-20 µL; the column temperature-300 °C. The method was validated with respect to robustness, system suitability test, specificity, linearity-range, accuracy, precision (intra-day and inter day), limit of detection (LOD) and quantitation (LOQ). The stability of alprazolam sample solutions and 0.45 µm membrane filter compatibility were studied as well. These studies were performed in accordance with established ICH Q2 guideline and USP requirements. The calibration curve is linear (r^2 =1.00000) over a wide concentration range of 0.0075-10 µg/mL; LOQ-0.0075 µg/mL and LOD-0.005 µg/mL. The method can be applied to determine quantitatively alprazolam residues in test solutions with very low concentrations below the acceptable concentration of the cross-contamination limit.

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