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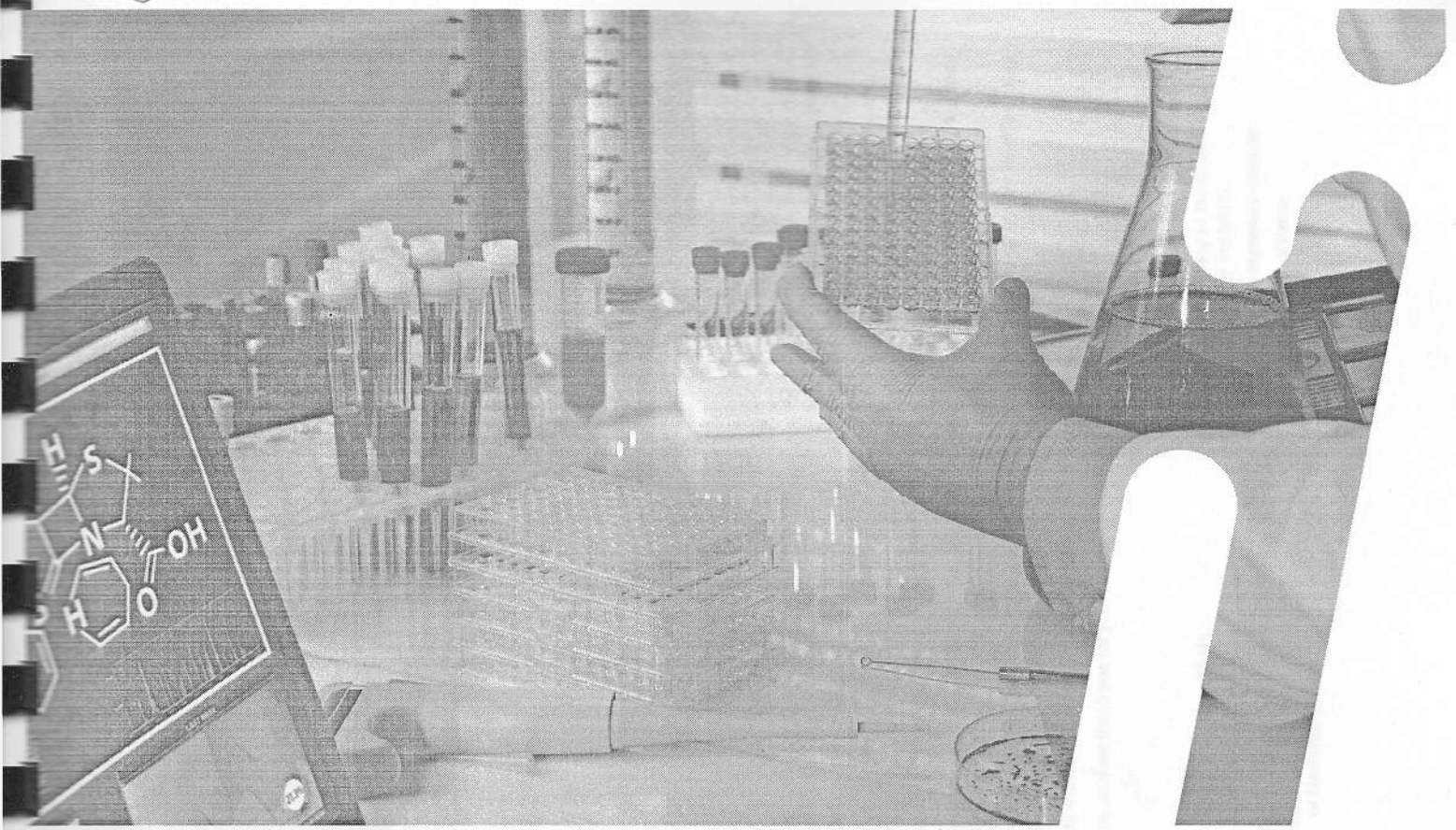

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POSTER020

NOVEL HPLC METHOD FOR QUANTIFICATION OF ATORVASTATIN AND ITS IMPURITIES IN TABLETS ON DIFFERENT CHROMATOGRAPHIC COLUMNS

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This new method we developed appears to be incomparably greener than the pharmacopoeial and all the other previously published methods for testing of atorvastatin impurities, since it is one of the shortest, applicable for routine analyses in quality control laboratories in pharmaceutical companies. The USP method for testing of impurities of atorvastatin is quite different than the EP method, using (L1) C18 column with mobile phase composed of tetrahydrofuran, acetonitrile and combined phosphate-acetate buffer, with gradient mode of elution, with variable mobile phase flow rate, during the period of 65 minutes, for separation of 11 impurities.

The new method we developed, not only meets the system suitability requirements, but also easily achieves the required sensitivity for analysis of impurities that are present in very low percentage in atorvastatin as active substance, or in tablets containing atorvastatin. Beside the selected Shim-Pack XR-ODS II 75 mm x 3 mm, 2.2 µm column, one other column Agilent Poroshell C18ec 100 mm x 4.6 mm, 2.7 µm, also showed very good results in separation of atorvastatin and its impurities. This column also yields very good chromatogram separations which accomplish required system suitability parameters, resolution and peak symmetries. Mentioned column can be considered as alternative to the first proposed Shim-Pack XR-ODS II because the sensitivity that offers the 3 mm internal diameter is superior in comparison to 4.6 mm internal diameter. The method run time in this case is increased for about 20%-25% due to the bigger column length and volume. It can be concluded that method is suitable for determination of lower concentrations of impurities, since the main peak of atorvastatin in the solution corresponding to 0.1 %, with concentration of 1 µg atorvastatin/ml is detected with S/N about 85. This suggests that the method limit of quantification (LOQ) can be at least about 7 times lower, and the limit of detection (LOD) at least about 20 times lower, using injection volume of only 2 µl. This was confirmed by the performed method validation in accordance to the ICH guideline for Validation of analytical procedures Q2(R1), where the selectivity, linearity, accuracy from the aspect of analytical recovery, precision from the aspect of system repeatability and limit of quantification and limit of detection were tested and confirmed, as presented below. Additionally, the method can be simply readapted and used in cases where faster quantification of atorvastatin is needed, for example for determination of average API content in pharmaceutical dosage forms, content uniformity and dissolution tests for tablets. This can be accomplished in even simpler and shorter way, without switching gradient and waiting for re-equilibration.