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IMPURITIES RELATED SULFAMETHOXYPYRAZINE, CHARACTERIZATION AND QUANTITATIVE DETERMINATION OF PROCESS BY SOME ANALYTICAL TECHNIQUES

MUKESH KUMAR GUPTA[1] & MANOHAR V. LOKHANDE[2]

[1]Department of Chemistry, Shri Jagdish Prasad Jhabarmal Tibrewala University, Vidya Nagari, Jhunjhunu, Chudela, Rajasthan, India

[1]IPCA Laboratories Ltd, Chemical Research Division, Kandivli Industrial Estate, Kandivli (W), Mumbai, Maharashtra, India

[2]Department of Chemistry, Sathaye College, Vileparle (E), Mumbai, Maharashtra, India

ABSTRACT

Four impurities in Active pharmaceutical ingredient (API) Sulfamethoxypyrazine were detected by a newly developed gradient reverse phase high performance liquid chromatographic (HPLC) method. These impurities were identified by LC/MS/MS. Three of the impurities were unknowns having not been reported previously. Structural assignment of these impurities was carried out by LC/MS/MS using electro spray ionization source and an ion trap mass analyzer. Structural elucidation using nuclear magnetic resonance (NMR) and infrared (IR) spectroscopy was facilitated by newly developed preparative isolation method. These impurities were characterized as 4-amino-N-(6-hydroxypyrazin-2-yl) benzene sulfonamide (SMP-I), 4-amino-N-(pyrazin-2-yl) benzene sulfonamide (SMP-II) & 4-amino-N-(6-methoxypyrazin-2-yl) benzene sulfonamide (SMP-III). The synthesized /isolated reference samples of the impurity compounds were used for the quantitative HPLC determination. The method was validated according to ICH guidelines with respect to specificity, precision, accuracy, linearity and robustness. Forced degradation studies were also performed for Sulfamethoxypyrazine bulk drug sample to demonstrate the stability indicating power of the newly developed HPLC method.

KEYWORDS: Sulfamethoxypyrazine, Impurities, HPLC, LCMS/MS/MS, Validation, Forced Degradation