Quantitative thin-layer chromatographic method of analysis of azithromycin in pure and capsule forms.

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A validated stability-indicating thin-layer chromatographic (TLC) method of the analysis of azithromycin (AZT) in bulk and capsule forms is developed. Both AZT potential impurity and degradation products can be selectively and accurately estimated in both raw material and product onto one precoated silica-gel TLC plate 60F254. The development system used is n-hexane-ethyl acetate-diethylamine (75:25:10, v/v/v). The separated bands are detected as brown to brownish red spots after spraying with modified Dragendorff's solution. The Rf values of AZT, azaeerythromycin A, and the three degradation products are 0.54, 0.35, 0.40, 0.20, and 0.12, respectively. The optical densities of the separated spots are found to be linear in proportion to the amount used. The stress testing of AZT shows that azaeerythromycin A is the major impurity and degradation product, accompanied by three other unknown degradation products. The stability of AZT is studied under accelerated conditions in order to provide a rapid indication of differences that might result from a change in the manufacturing process or source of the sample. The forced degradation conditions include the effect of heat, moisture, light, acid-base hydrolysis, sonication, and oxidation. The compatibility of AZT with the excipients used is also studied in the presence and absence of moisture. The amounts of AZT and azaeerythromycin A are calculated from the corresponding linear calibration curve; however, the amounts of any other generated or detected unknown impurities are calculated as if it were AZT. This method shows enough selectivity, sensitivity, accuracy, precision, linearity-range, and robustness to satisfy Federal Drug Administration/International Conference of Harmonization regulatory requirements. The method developed can also be used for the purity testing of AZT raw material and capsules, content uniformity testing, dissolution testing, and stability testing of AZT capsules. The potential impurity profiles of both active AZT material and capsule forms are found comparable. The linear range of AZT is between 5 and 30 mcg/spot with a limit of quantitation of 2 mcg/spot. The intraassay relative standard deviation percentage is not more than 0.54%, and the day-to-day variation is not more than 0.86%, calculated on the amounts of AZT RS recovered using different TLC plates.