Azithromycin

The method for a 250 mg azithromycin tablet published in the Minilab manual, Volume II, Supplement 2011, pages 8-11, was modified by simple heating of the plate to cause the azithromycin spots to be visible in daylight, quench fluorescence at 254 nm, and fluoresce at 366 nm so that applications of the sulfuric acid and iodine staining methods were not necessary for detection. Users may consider that elimination of the detection reagents makes this method safer and more convenient, especially for use in the field.

In the modified method, the exact procedures published in the Minilab manual were carried out with two exceptions. Instead of a 250 mg reference tablet for the standard, 250 mg of commercial analytical grade standard (azithromycin, USP, No. 1046056) was used. Also, instead of exposing the plate to iodine vapor or dipping it in methanolic sulfuric acid staining solution followed by heating on a hotplate to detect the azithromycin as colored spots in daylight as shown in the photograph on page 11 of the Minilab manual, the drug was detected as yellow spots in daylight, fluorescence quenching spots under 254 nm UV light, and fluorescent spots under 366 nm UV light, as shown in the photographs of the three plates below, by heating on a hotplate. The 100% working standard solution and 100% working sample solution were 5 mg/mL, and 2 μL volumes were spotted on the plates. The mobile phase was methanol-ethyl acetate-concentrated ammonia solution (20:5:0.5).

The detection of azithromycin as fluorescence quenched zones under 254 nm UV light on silica gel glass plates with a fluorescent indicator (F plates) by reagent free thermochemical activation (heating at 180°C for 5 minutes) was first reported in the literature by D. Zhang, J. Strock, and J. Sherma (Journal of Liquid Chromatography & Related Technologies, 2016, Vol. 39, No. 5-6, pp 277-280).

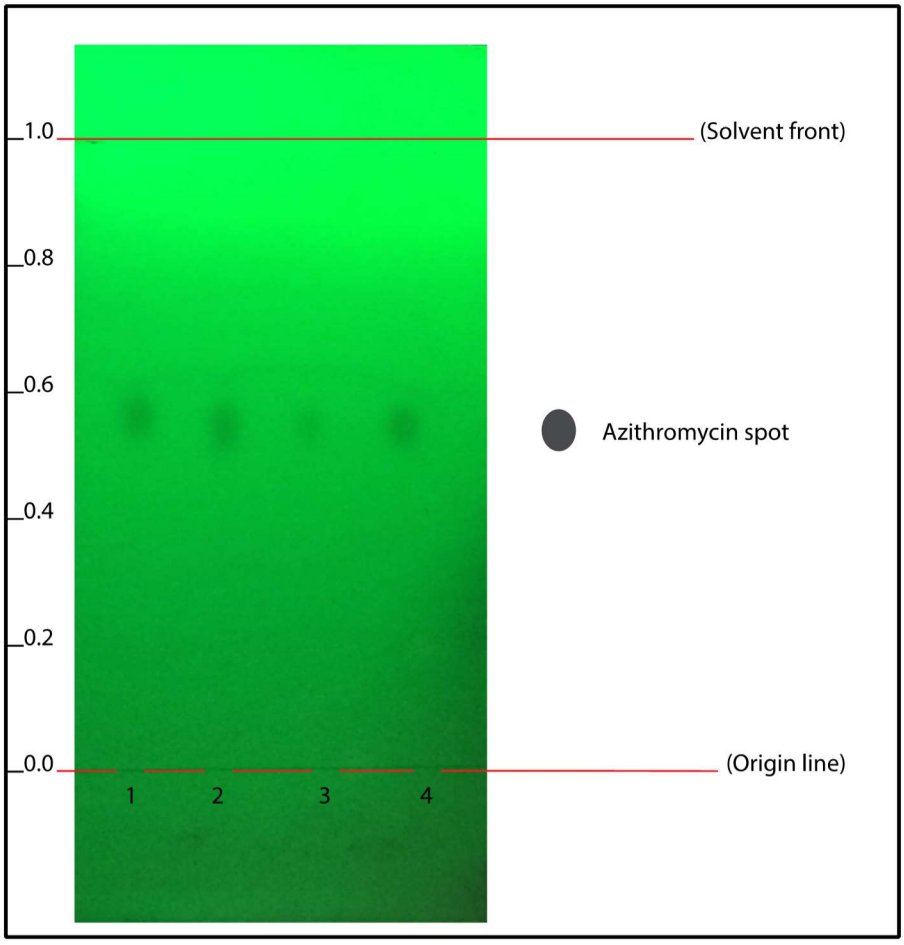
XI. CHROMATOPLATE OBSERVED UNDER 254 NM UV LIGHT AFTER HEATING

Run No.1:
Upper working standard representing 100% of total anhydrous azithromycin

Run No.2:
A drug product of good quality with acceptable drug content

Run No.3:
A drug product of poor quality with unacceptable low drug content*

Run No.4:
Lower working standard representing 80% of total anhydrous azithromycin



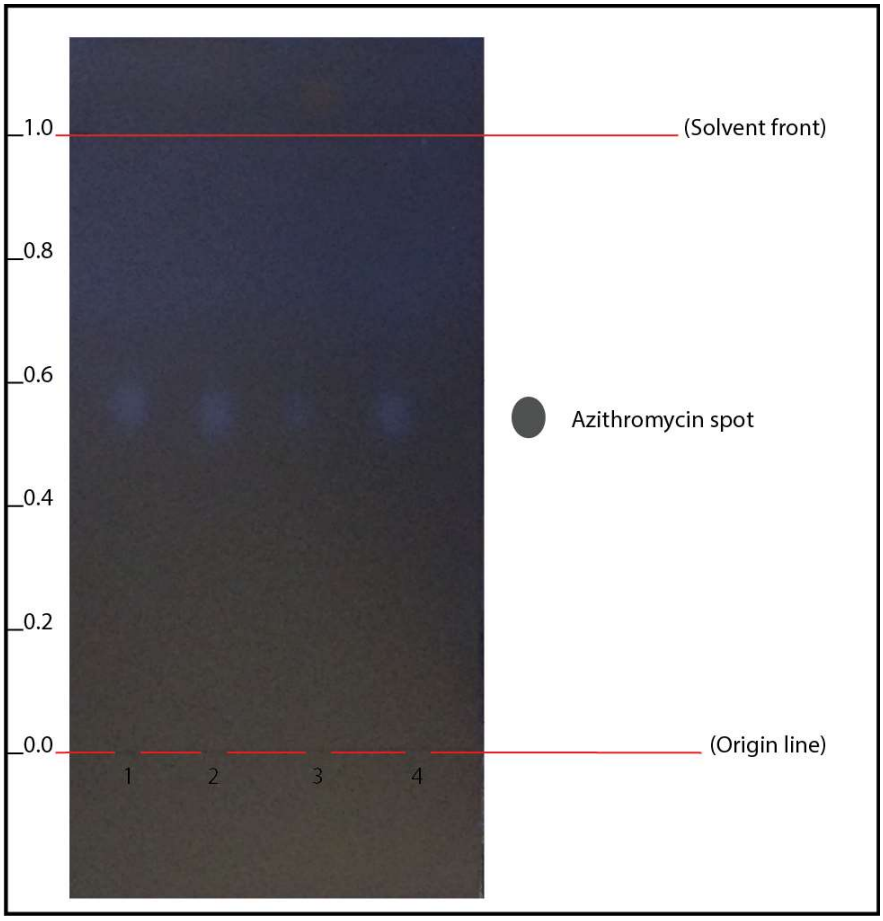
XI. CHROMATOPLATE OBSERVED UNDER 366 NM UV LIGHT AFTER HEATING

Run No.1:
Upper working standard representing 100% of total anhydrous azithromycin

Run No.2:
A drug product of good quality with acceptable drug content

Run No.3:
A drug product of poor quality with unacceptable low drug content*

Run No.4:
Lower working standard representing 80% of total anhydrous azithromycin



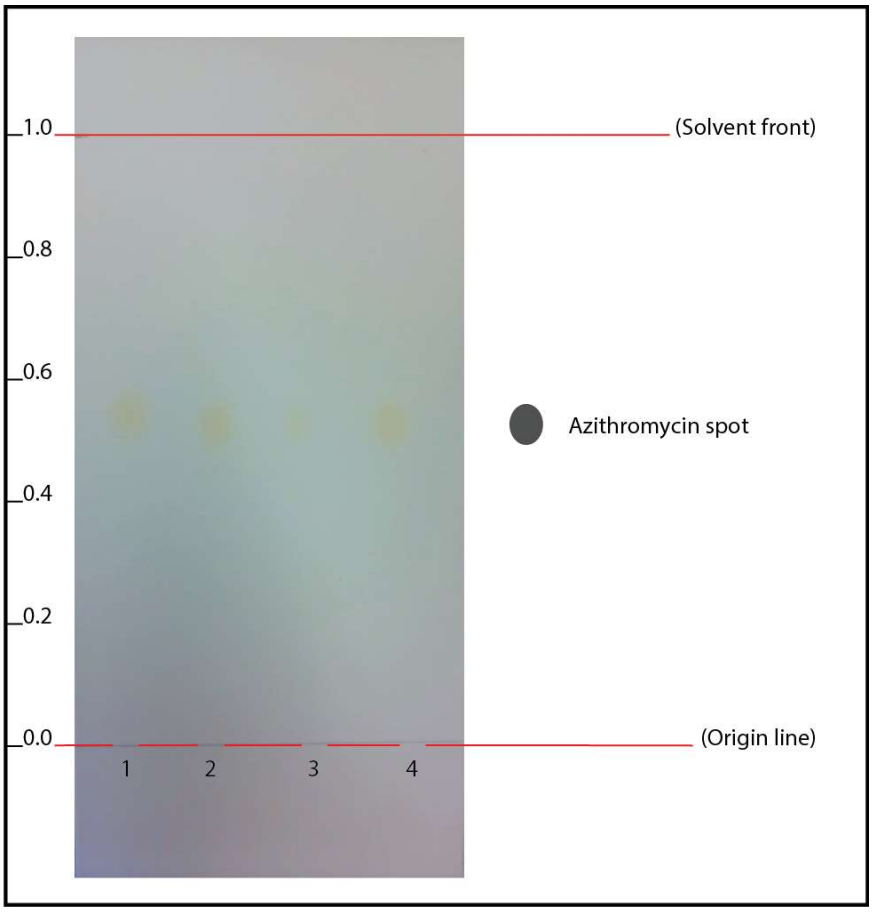
XI. CHROMATOPLATE OBSERVED IN DAYLIGHT AFTER HEATING

Run No.1:
Upper working standard representing 100% of total anhydrous azithromycin

Run No.2:
A drug product of good quality with acceptable drug content

Run No.3:
A drug product of poor quality with unacceptable low drug content*

Run No.4:
Lower working standard representing 80% of total anhydrous azithromycin



(*A drug product of poor quality was simulated by diluting the 100% working sample solution of a drug product of good quality with methanol to one-third of the theoretical value.)

This modified method was developed and tested by Ellen Armour and Joseph Sherma, Department of Chemistry, Lafayette College, Easton, PA, USA., June, 2016. Ellen Armour's EXCEL Scholar research was supported by a Camille and Henry Dreyfus Foundation Senior Scientist Mentor Program award to Professor Sherma.