Artesunate

The method for a 100 mg artesunate tablet published in the Minilab manual, Volume II, pages 56-59, was modified by simple heating of the plate to cause the artesunate spots to be visible in daylight, quench fluorescence at 254 nm, and fluoresce at 366 nm so that application of the sulfuric acid staining method was not necessary for detection. Users may consider that elimination of the detection reagent 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 a few exceptions. Instead of a 50 mg reference tablet for the standard, 50 mg of commercial analytical grade standard (artesunate, Sigma-Aldrich, No. A3731) was used. Instead of a pure 100 mg artesunate tablet, a combination tablet of artesunate and amodiaquine, 100 mg and 270 mg respectively was used. Also, instead of exposing the plate to methanolic sulfuric acid staining solution followed by heating on a hotplate to detect the artesunate as colored spots in daylight as shown in the photograph on page 59 of the Minilab manual, the drug was detected as grey 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 ethyl acetate-acetone-glacial acetic acid (18:4:0.1).

The detection of artesunate 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^{o}C for 5 minutes) was first reported in the literature by M. Nguyen and J. Sherma (Journal of Liquid Chromatography & Related Technologies, 2014, Vol. 37, No. 20, pp 2956-2970).

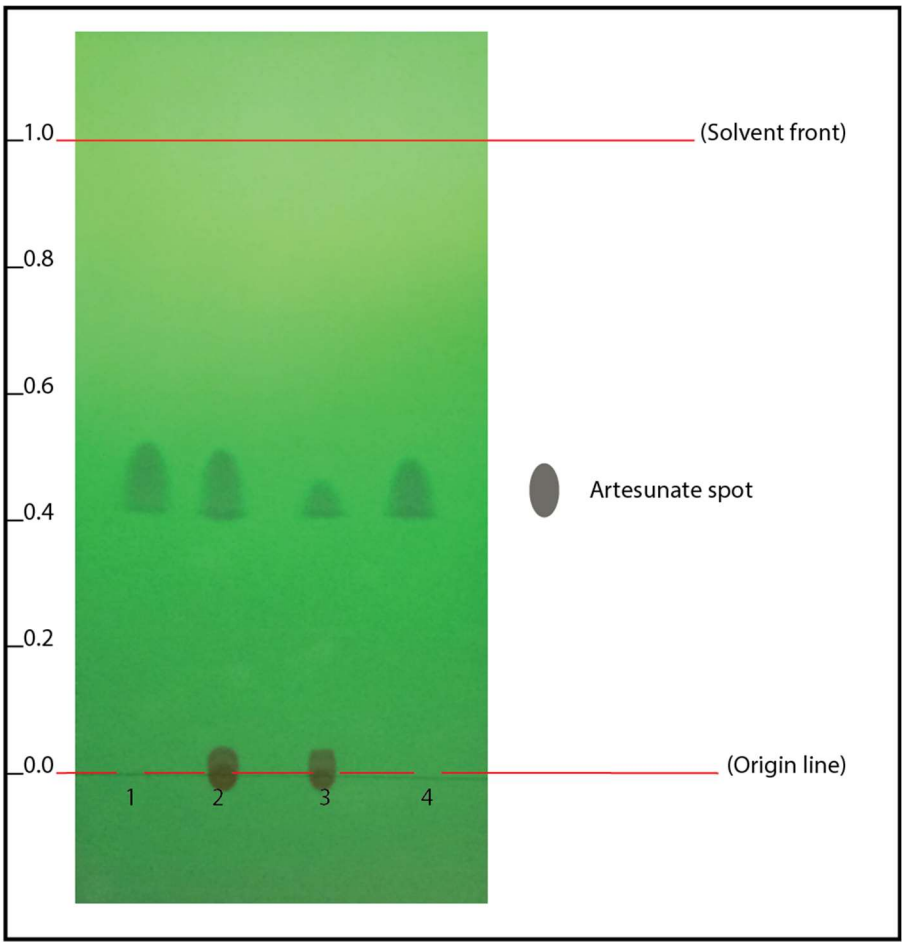
XI. CHROMATOPLATE OBSERVED UNDER 254 NM UV LIGHT AFTER HEATING

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

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 artesunate



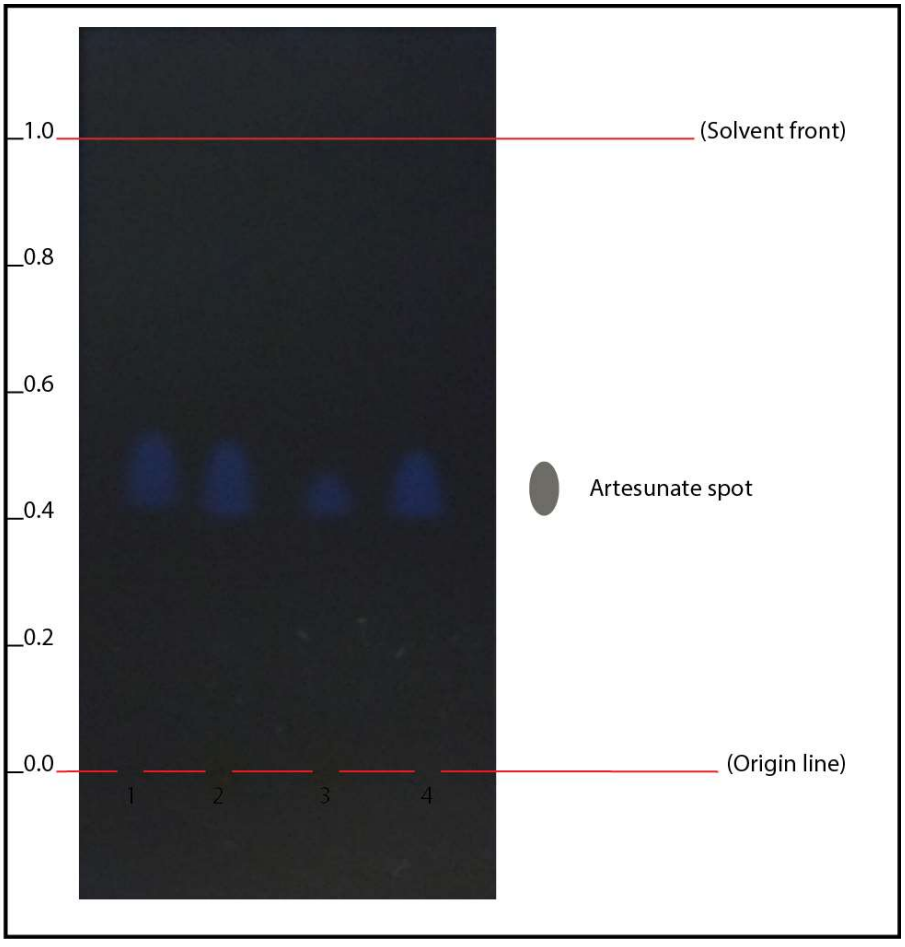
XI. CHROMATOPLATE OBSERVED UNDER 366 NM UV LIGHT AFTER HEATING

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

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 artesunate



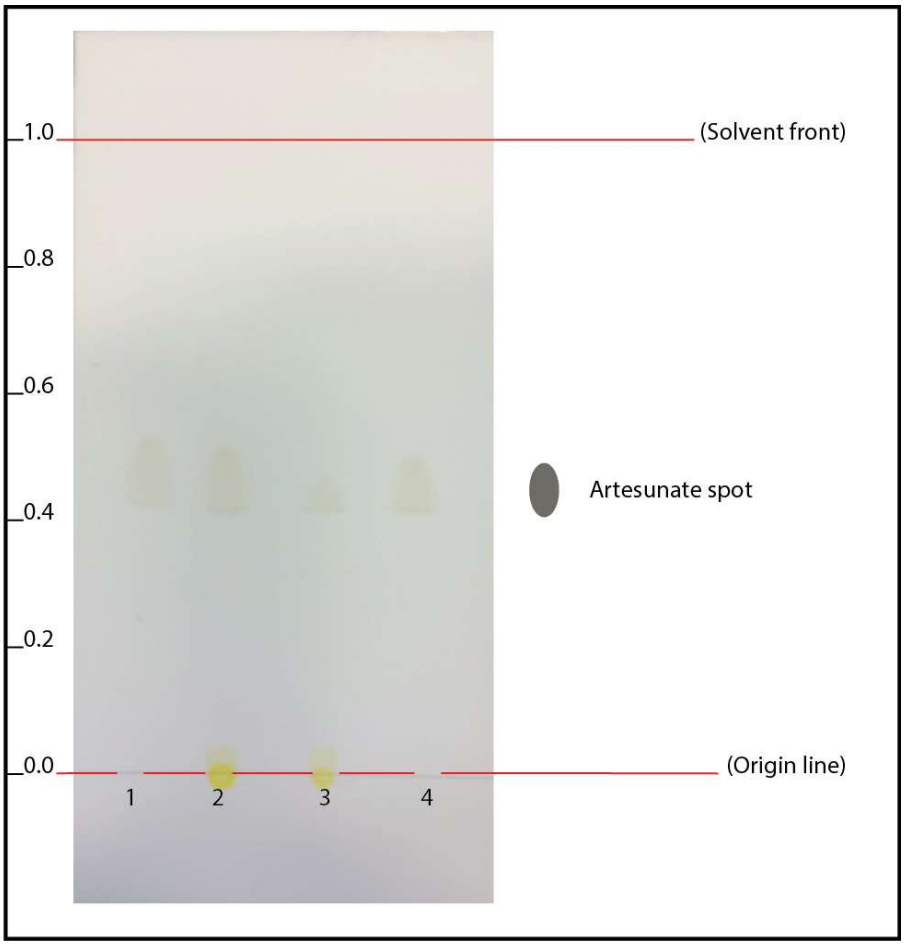
XI. CHROMATOPLATE OBSERVED IN DAYLIGHT AFTER HEATING

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

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 artesunate



(*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., July, 2016. Ellen Armour's EXCEL Scholar research was supported by a Camille and Henry Dreyfus Foundation Senior Scientist Mentor Program award to Professor Sherma.