

Quantitative and Qualitative TLC Analysis of 5meo DMT

By Clear Light and Desert Sun

We are reporting here our method for qualitative and quantitative analysis of the titled compounds using prior hive posts in addition to a unique photometric method for quantitative analysis of UV and visualized chromaphores.

Using TLC plates we have determined the rF values of the titled compounds and identified their wet and dry chromaphores using the modified Van Urk/Salkowski reagent system as well as their respective concentrations.

Methods:

Silica Gel 60 F254 hard surface(polymer) 2.5x7.5 cm plates were used as the TLC sorbent system.

Reagent System:

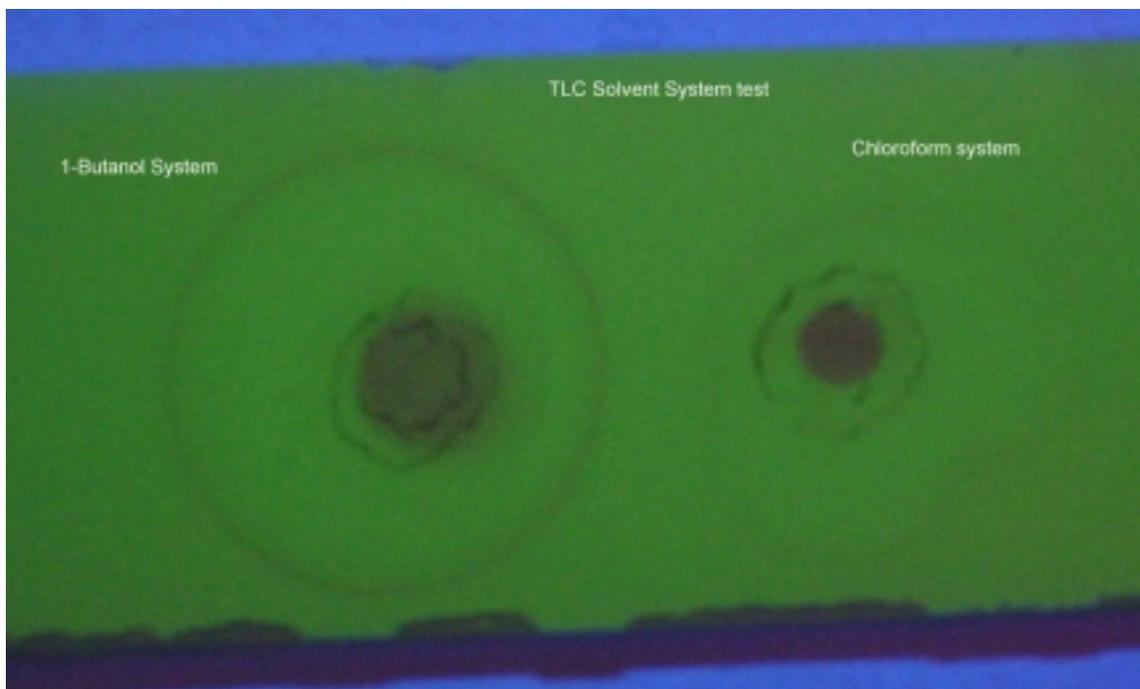
The reagents were made a follows.

- (A) van Urk³⁷ reagent: 1 g p-dimethylaminobenzaldehyde was dissolved in 50 ml conc. HCl (specific gravity 1.190) and 50 ml absolute ethanol was added;
- (B) (B) Salkowski⁶ reagent 2.03 g FeCl₃ · 6 H₂O were dissolved in 500 ml water and 300 ml conc. H₂SO₄ (specific gravity. 1.840); this reagent is stable indefinitely.

Spray reagent. The new TLC spray reagent used, was made up of reagent A and B (1 :3).

We also tested the method previously published of using a cotton ball to swipe the plate rather than spraying. The method that was found most efficacious was to hold the saturated cotton ball in one hand and the top of the plate(hard polymer, gypsum will not work with this method) and swipe the plate vigorously in one stroke. Some gray artifacts may appear on the plate (see Fig. 5 below) but these disappear upon drying and do not affect the analysis. This method avoids the need for a sprayer and fume hood.

A single plate was spotted with DMT 2x and the chloroform/methanol/water (84:14:1) solvent system was tested as was the Propanol/Water/Amm Hydroxide (8:1:1). As you can see from the following photograph under short-wave u.v. the Propanol system provided an expanded spot size and concentric rings while the chloroform spot stayed relatively intact. Based upon this analysis we selected the Propanol system. (Thx to Lugh for the article on this method of solvent system determination)



2 samples of DMT were available. 25 mg of each sample was diluted in 5 ml of Methanol. A 5 λ pipette was used, which gave 25 μ grams of material per spot. Spotting had to be done repeatedly to avoid spot from growing too large. In our new photographic method, it becomes very important to mark the exact center of the loading spot at the start point of the plate, as measurements are in pixels and often times it is difficult to get the spot right on the cross hairs that are drawn with pencil.

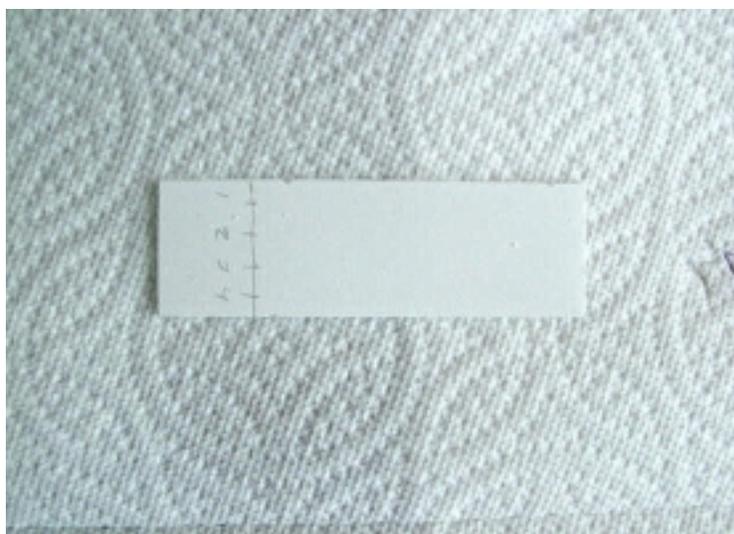


FIG 1: Example of unloaded plate marked for spotting

Based upon the solvent front length average measurement of 47.7 mm and the front spot edge average of 30.2 mm we get an average r_f from the two measurements of .63

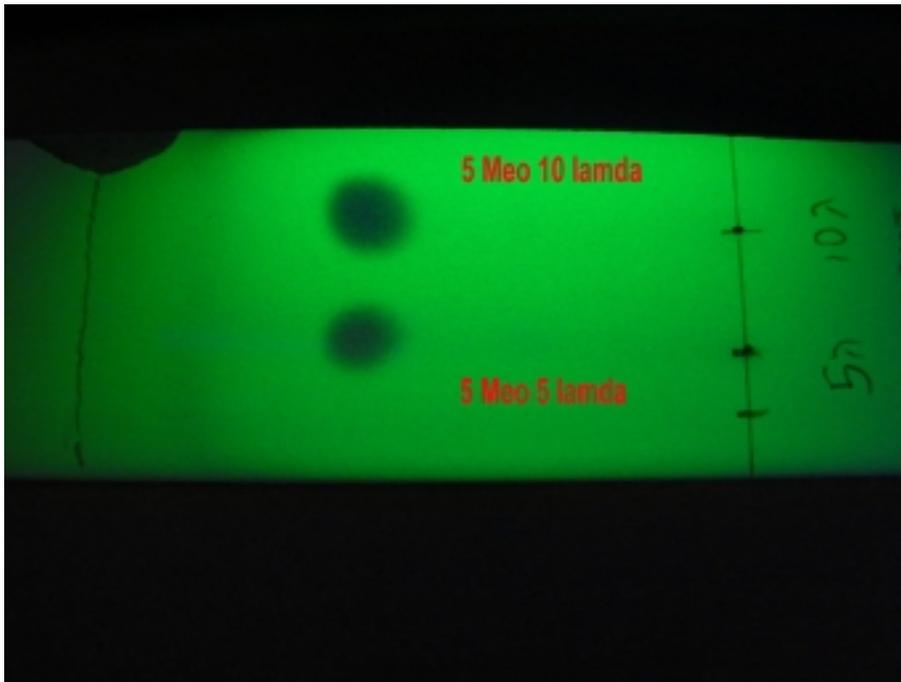


Fig: 3 Short-wave UV illumination of 5meo DMT TLC plate

Using the Background subtracted integrated density (BSID) measurement in the image tool, we find that the absolute value for the 10 lambda spot equals 659578. This is the average of two measurements.

The average abs value for the 5 lambda spot = 331232. Dividing the 10 lambda measurement by the 5 lambda measurement we get a value of **1.99**. Thus by using two or more measurements of the area (these are drawn by hand so multiple measurements are suggested) we get a result of 1.99. Thus we have proven that the Image tool can accurately measure the relative concentration of the 5MEO DMT on the TLC plate and therefore is an accurate tool for qualitative measurement of TLC plate concentrations!

Now we have, with a simple digital camera, Short-wave U.V. lamps and fluorescent TLC media, the equivalent of \$20-30k lab quality analytical instruments. The UTHSCSA Image tool can be downloaded for free from the site

<http://ddsdx.uthscsa.edu/dig/download.html>

. Attempts to analyze the wet image, show below were not successful as the wet plate does not have the fully developed density of the dry plate and the measurements were in error.

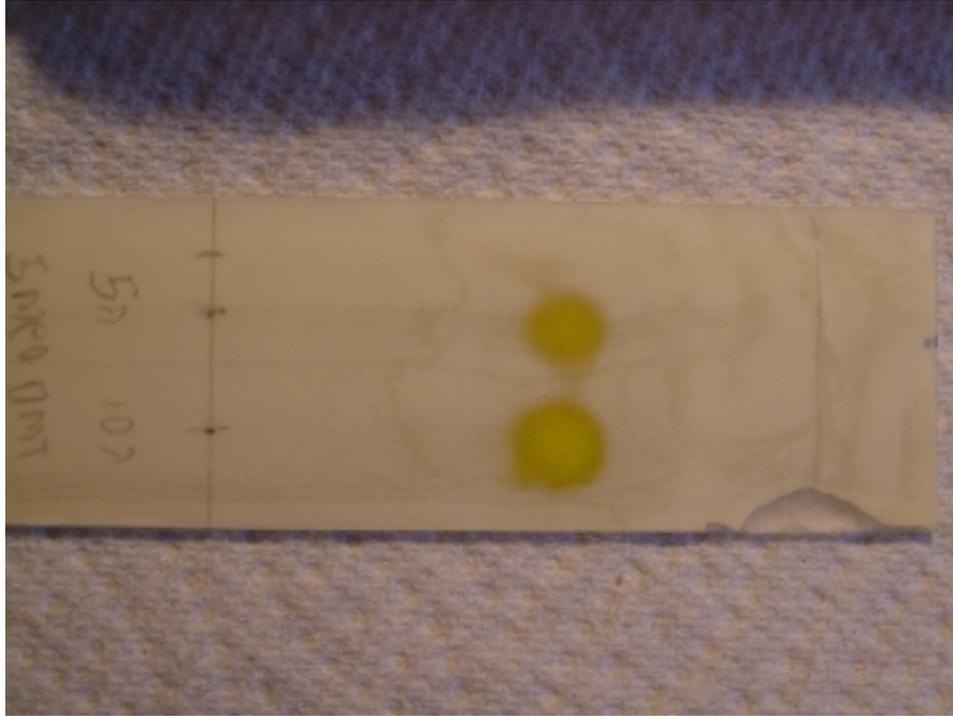


Fig 5. Wet plate of 5meo DMT showing typical colors prior to drying. Note light center on 10 lamda spot which skews BSID measurements.

We now measure the results of the colorization reagent applied to the tryptamine spots on the following image:

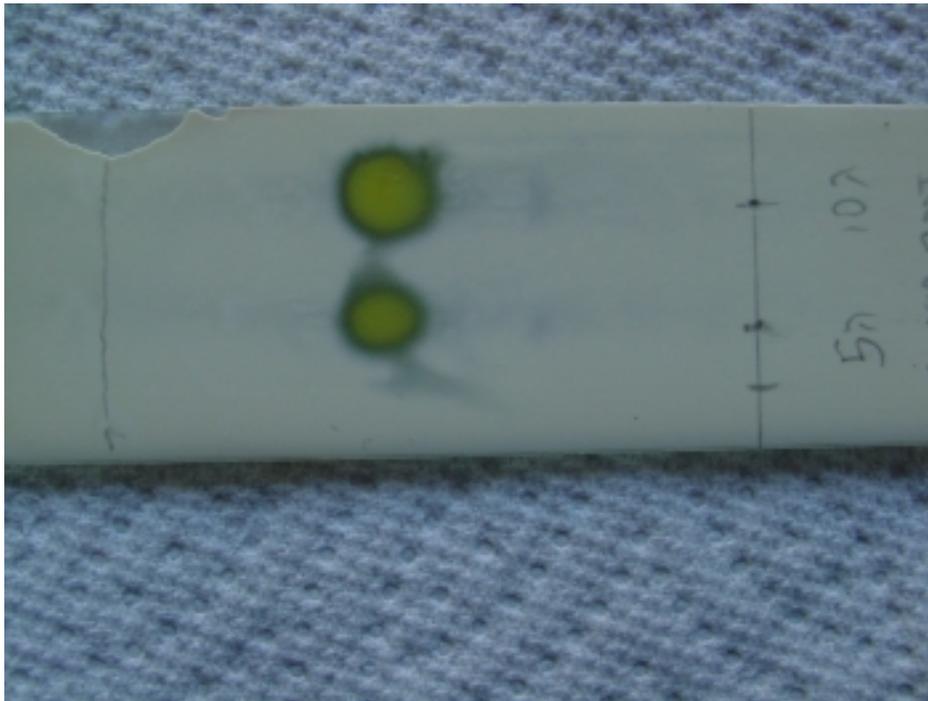


Fig. 4: 5meo DMT visualized with Van Urk/Salkowski reagent system.

Note the typical dry colors of the 5meo DMT as olive green with a dark circle around the spot. This is the standard color for this compound.

Average BSID measurements of these two spots gave the following results:

236272 = Average 10 lambda BSID measurement

112994 = Average 5 lambda BSID measurement

2.09 = Ratio of 10 lambda/5 lambda spots

Thus we have demonstrated the accuracy of the Image tool in analyzing dry plates when plates have been visualized using the Van Urk/Salkowski reagent system.

Conclusions:

Qualitative measurements of TLC plates are now possible using simple, home equipment. Utilizing a free program, Image, Background subtracted image density (BSID) measurements, when averaged, correlate very well with the loadings on the plate. Use of a wet plate will not provide accurate measurements but will provide some qualitative information as to the nature of the materials under analysis. A 5 microliter spot containing 25 micrograms of the title compound was compared to a 10 microliter spot containing 50 micrograms of the title compound. The results for both U.V and visualized BSID measurements were 1.99 and 2.09 respectively.

Short Wave UV measurements of the image as well as dry visualized measurements provide accurate information as to the total amount of compound present when compared to a standard. This method now provides the experimenter with a rapid, easy way to quantify the amounts of compounds present in a sample and may be correlated to other compounds of interest to provide rapid, accurate measurements of your materials. This system compares favorably to analysis done using \$20k –30k systems.