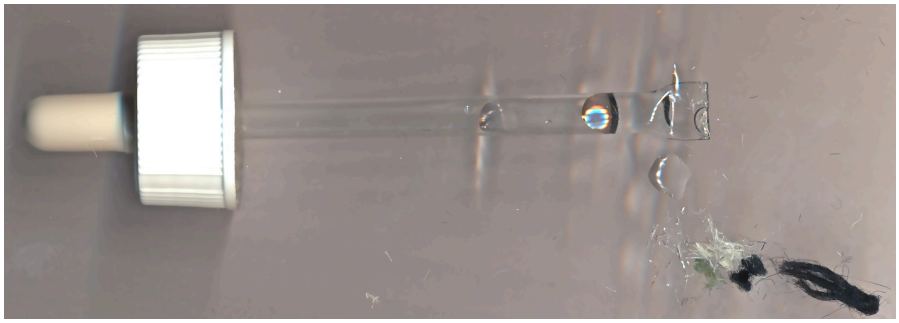
HPLC identification of all the dyes utilized in a supposedly «natural-dyed» rug, is much too complicated and costly for any private citizen, but TLC is a reasonably cheap and uncomplicated way to answer the following limited question: **«Is the red dye madder ?**»

To answer the question «**Is the blue dye indigo ?»,** one does not even need a TLC test, all one needs is the hardware shown below: a) One drop of diluted dithionite (or dithionite -ammonia mix) and indigo-dyed wool turns greenish white, b) Dry. c) A drop of diluted H2O2 and wool recovers its blue shade.



One implicitly assumes that, if the red and the blue are natural dyes, any other dye on the rug is also quite likely to be natural.
This assumption may still be wrong (yellow and orange might be the worst acid dyes mankind ever synthesized and indigo might be synthetic too), but it is a reasonable first approximation and this hobby can be as much fun as watching baseball, at least from a french perspective).

For any fan of natural dyes bitten by the TLC bug, Dr. H. Schweppe's book « Handbuch der Naturfarbstoffe. Niko Verlag. 1993» is a good companion. There is an English translation. German and English editions are quite difficult to find.

What does it take to run a TLC test:

1. A small sample of red wool. This will probably difficult to obtain, but the seller's reaction to this request might tell nearly as much as the TLC test itself.
2. A small glass vessel with cap («the separating chamber»), a few glass micro-pipettes, 1 glass funnel, 1 porcelain cup, a makeshift steam-bath, protecting glasses and gloves (not overly friendly acid and solvents are used), a well aired room (smelly, mildly toxic solvents) and a locked place to keep solvents and acids away from kid's initiative.
3. Small quantities of chemicals, available on internet:

- Diluted sulfuric acid (10% in water), to extract the dye from the fibre.
- Ethyl-acetate and iso-amyl alcohol, the solvents in which the extracted madder- and insect reds will be transferred from the sulfuric acid .
- A few packs of polyamide-layered TLC plates (so-called stationary phase).
- The solvents (Toluene, butanone, acetic acid, formic acid, methanol) which will be selectively mixed for creating the so-called mobile phase which will rise on the TLC plates, carrying the dyes and separating them.
- A color-enhancer (0,5% uranyl acetate solution in a 50:50 mix of water and methanol). Sprayed on the TLC plate at the end of the separating phase, it will identify the dye spots. (Needed since these dyes have a fully different shade on TLC plates and might be nearly colorless.
- A few grams of madder powder (perhaps also insect reds) obtained from commercial sites catering to hobby dyers. Used as reference samples, to be run together with unknown dyes in every TLC run.

How to run a TLC test (quoted from Dr Schweppe's «Practical informations for the identification of historic textile material. Pages 11-14. 1988»)**:**

A. How to extract the madder from wool sample and prepare a sample for TLC comparison:
Madder- and insect dyes are extracted from the wool with boiling 10% sulfuric acid solution.
The extract is then shaken with ethyl acetate.
A yellow- (madder) or orange- (kermes) dye separates in the upper layer (ethyl acetate) which forms when one stops shaking.
If the lower layer (acid solution) is still colored orange, it might still contain a dye, namely the insect reds cochineal or Lac. In this case, one adds some iso-amyl alcohol and shakes again.
The upper layer of solvent(s) and dyes is separated from the lower layer (sulfuric acid and water) in a funnel .
The dye-containing solvent layer is shaken several times with fresh water, to eliminate any residual trace of acid in it.
The dye solution is evaporated in a porcelain dish on a steam-bath (not on open flame!).
The solid residue is dissolved in a few drops of methanol.
This methanol solution can be used for the TLC test.

B. How thin layer chromatography is carried out.
On a TLC plate mark a series of dots with a soft pencil, starting about 2 cm from each side of the plate, at about 1.5 cm from its bottom and with at least 1 cm between dots.
With a micro-pipette, apply solutions of the dye to be investigated and of the reference dye (madder in this case) on the dots. The dye spots should have a diameter of no more than 2-3 mm.
After drying in the air, place the plates in the separating chamber, which had been filled, at least two hours earlier, with the chosen mobile phase (solvent mix) to a level of about 1 cm (thus below the dots). Close the chamber with cap.
The mobile phase rises slowly on the TLC plate. When it has risen about 10cm, mark the level to which it has risen on the plate with a soft pencil and let the plate dry in the air.
Then, spray the plate with the color-enhancer. Press the plate between layers of filter paper and let it dry in the air.
Compare the profiles of unknown dye and of reference dye(s).

Suitable mobile phases for TLC of madder red, on polyamide-layered TLC plates:
. Toluene-acetic acid 90:10 or,
. Butanone-formic acid 95:05 or,
. Chloroform-methanol 95:05

Suitable mobile phases for TLC of insect reds on polyamide-layered TLC plates:
. Butanone-formic acid 70:30 or,
. Butanone-methanol-formic acid 65:30:05

Polyamide layered-TLC plates: Micro-polyamide type F 1700 from Schleicher & Schüll or similar.