EXPERIMENTATION IN ORGANIC CHEMISTRY

LESSON 7. ANALYSIS OF ANALGESICS BY TLC (THIN LAYER CHROMATOGRAPHY)

OBJECTIVE:

The main goal of the practice consists on the determination of the composition of some common analgesics, using the Thin Layer Chromatography (TLC) technique.

REAGENTS:

Samples for the first part – Pure species:

Ibuprofen analgesic

Naproxen analgesic

Paracetamol (*p*-acetamido phenol) analgesic

Acetyl salicylic acid analgesic

Caffeine stimulant, usually accompanying analgesic drugs

Reference sample mixture of the previous five

Samples for the second part – Commercial Analgesics:

Aspirine, Bayer

Cafiaspirina, Bayer

Actron, Bayer

Termalgin, Novartis

Antalgin, Roche

Nurofen, Boots Healthcare

Reference sample: Same as in the first part

MATERIALS:

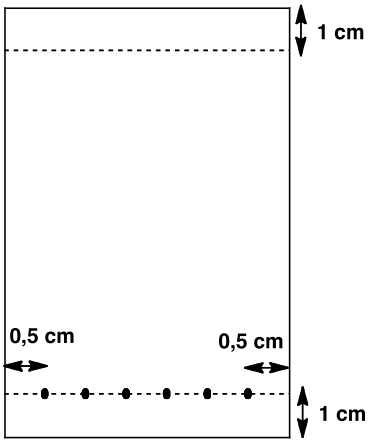
Chromatography jar; 2 TLC Chromatography plates; capillaries; filter paper; ethyl acetate; hexane; UV lamp; iodine; 6 test tubes; chloroform; absolute ethanol; heater with stirring system; water bath

PROCEDURE:

1st part: Analysis of the active species (drugs)

In the first part of the practice, the R_f values of the different active compounds are measured. We will use those values for the analysis of the mixtures during the second part.

Take a TLC plate carefully (do not touch the layer directly (take it from the edges) and draw two straight lines with a pencil (never use a pen), like in the figure, around 1 cm away from the lower and upper edges. With a ruler, mark in the lower line so many points as necessary for the different compounds, leaving around 0.5 cm free from the right and left sides of the layer.



For each commercial analgesic, take one pill and grind it up with the help of a spatula. Introduce the powder in a sample tube (conveniently labelled) and dissolve it with a mixture of 1:1 chloroform-ethanol (5 mL). Heat the suspension slightly in a water bath for a few minutes (part of the mixture will remain insoluble, excipients and other additives). After cooling down to room temperature, the mother liquor only contains the soluble active species.

Then, spot the samples on each mark with a capillary, carefully, taking care not to make holes in the silica. The amount of product placed on each mark can be visualized with the aim of a UV lamp.

Then, pour the eluent (ethyl acetate) into the chromatography jar, circa 0.5 cm high, introducing also a filter paper strip vertically, reaching almost the edge of the jar.

The TLC plate is introduced into the jar, taking special care that the bottom of the plate is horizontal, and that the eluent does not cover the spots. The jar is covered, and we let it run (or develop) **WITHOUT MOVING THE JAR AT ALL**. When the eluent reaches the top line (the one marked with the pencil) of the plate, take it out from the jar and let it dry on the air.

Next, put the TLC plate under the UV light and mark with a pencil the spots we see on the layer. Then, insert the plate into a smaller jar with some crystals of iodine, and wait until the spots get colored. As before, mark these spots with a pencil. The measure of the fraction of the distances that the spots moved comparing to the eluent front is the R_f of each compound.

2nd part: Analysis of the composition of the commercial analgesic mixtures

In the second part of the practice, a set of commercially available analgesic mixtures is analyzed. They are composed of mixtures of the previous pure active species. The students will be provided with six unknown samples (numbered 1 to 6) that they have to identify.

Spot the samples prepared from the blind commercial analgesics and also the samples from the active species as a reference, and proceed as in the first part. Use the UV light and iodine to determine the composition of the commercial analgesics by comparing their Rf values.

Indicate:

- R_f for each reference analgesic compound
- Identify the blind samples 1-6 with the commercial analgesics, indicating the R_f value for each component.

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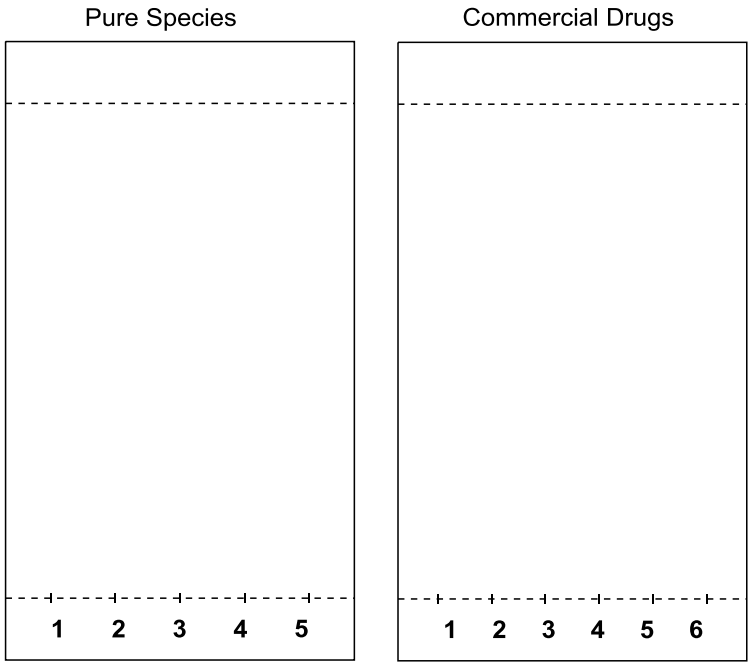
Experimentation in Organic Chemistry	**Practice #:**
NAME:	**DATE:**

A	**Characteristics of the Practice**
A.1	Main Goals:
A.2	Theoretical Basis of the Practice. How is the separation possible?

B	RESULTS
B.1	Draw the TLCs you got. Calculate the Rf values of each compound



B.2	Discussion of the results. - Order the pure compounds according to their polarity. - Find the names of the commercial drugs in the unknown samples
B.3	Physical-chemical properties of the analyzed pure compounds:

Compound	Chemical Structure	Molecular Formula	Molecular weight	State	Color

B.4	Conclusions:
B.5	Special Events: