

Passage VII

Students were given 12 known compounds and 4 unidentified compounds (Unknowns A–D) to analyze.

Experiment 1

Students placed a solid sample of each known compound in a separate *capillary tube* (a thin glass tube). Samples were then heated, 4 at a time, in a *melting point apparatus* (see Figure 1).

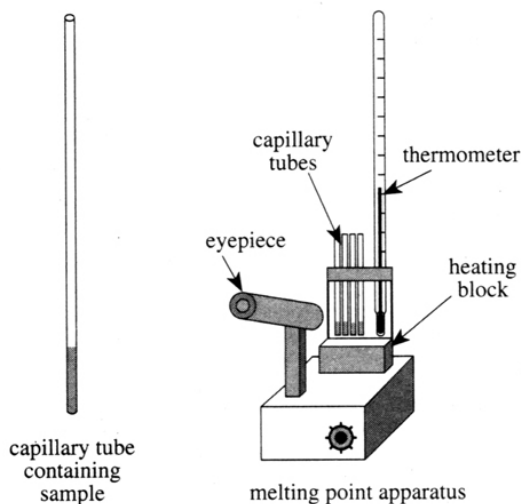


Figure 1

The samples were monitored through the eyepiece. When liquid first appeared in a sample, the temperature was recorded. When no solid remained in the sample, the temperature was again recorded. The compounds were placed in Groups I–IV based on similarity in melting point (MP).

Experiment 2

For each compound in a group, students dissolved a sample and placed a small spot of the solution 2 cm from the bottom of a 13 cm tall and 7 cm wide *thin-layer chromatography* (TLC) plate. Each spot was circled with a pencil and allowed to dry. The plate was placed in a tank containing a small amount of a particular solvent. When the solvent had moved up to 1 cm from the top of the plate, the plate was removed and allowed to dry. Four distances were measured, in cm, under UV light: distance *a* for each spot and distance *b* (see Figure 2).

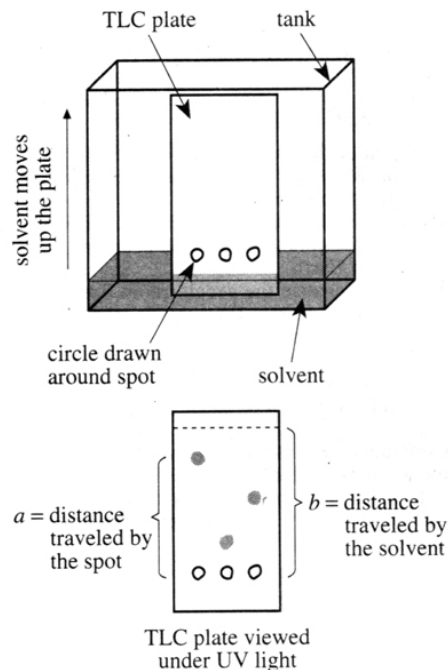


Figure 2

The R_f value of each compound was then calculated by dividing distance *a* by distance *b*. The results of Experiments 1 and 2 are shown in Table 1.

Group	Compound	MP range (°C)	R_f value
I	biphenyl	69–72	0.82
	2,5-DMP	68–71	0.24
	4-NBC	70–73	0.40
II	PNCB	83–84	0.67
	naphthalene	80–82	0.85
	vanillin	81–83	0.05
III	acenaphthene	93–95	0.78
	<i>m</i> -toluamide	94–96	0.02
	methyl 4-nitrobenzoate	94–96	0.51
IV	benzoic acid	122–123	0.19
	<i>trans</i> -stilbene	122–123	0.77
	succinimide	122–124	0.00

Experiment 3

The techniques from Experiments 1 and 2 were used to analyze Unknowns A–D (see Table 2).

Unknown	MP range (°C)	R _f value
A	93–95	0.02
B	123–125	0.74
C	68–71	0.85
D	80–82	0.06

Figures adapted from Samuel G. Levine, "Identification of Unknowns by Melting Point and Thin-Layer Chromatography in Combination." ©1990 by Division of Chemical Education, Inc., American Chemical Society.

35. In Experiment 1, liquid first appeared in the biphenyl sample at which of the following temperatures?
- 69°C
 - 72°C
 - 82°C
 - 100°C
36. The spot for which of the following samples traveled farthest up the TLC plate?
- 2,5-DMP
 - PNCB
 - acenaphthene
 - benzoic acid

37. One of the students calculated the R_f value for Unknown C as 1.18. There had to be an error in the student's calculation because:
- distance *a* cannot be longer than distance *b*.
 - distance *b* cannot be longer than distance *a*.
 - distance *a* must be equal to distance *b*.
 - R_f values must be less than or equal to 1.10.
38. A chamber contains samples of each of Unknowns A–D maintained at 150°C. Suppose the temperature in the chamber is slowly cooled to 40°C. Based on the results of Experiment 3, which of the samples would most likely start to solidify first?
- Unknown A
 - Unknown B
 - Unknown C
 - Unknown D
39. Based on the results of Experiments 1–3, Unknown A is most likely which of the 12 known compounds listed in Table 1?
- biphenyl
 - vanillin
 - acenaphthene
 - m*-toluamide
40. Which of the following diagrams best represents how the TLC plate appeared under UV light for Group II?

