InkChemistry

ANSWER BOOK

Chromatography: TLC, column and gas

Organic analysis · Unit C19

AQA 3.3.16 · OCR A 6.3.1

15 questions · 47 marks

NAVY EDITION

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SECTION A · Short, 1 to 2 marks

A1 CTC-Q1 — [2]

B1 the stationary phase is the solid layer that does not move, silica or alumina on the plate. **B1** the mobile phase is the solvent that moves up the plate, carrying the components with it.

A2 CTC-Q6 — [2]

B1 pencil is graphite, which does not dissolve in the solvent or move up the plate, whereas ink would. **B1** above the solvent level the spots are not dissolved off the plate into the solvent in the tank.

A3 CTC-Q7 — [2]

B1 the lid keeps the air in the tank saturated with solvent vapour. **B1** so the solvent does not evaporate from the plate as it rises and the front moves evenly.

A4 CTC-Q9 — [2]

B1 spray with ninhydrin and warm, which turns amino acids purple. **B1** use a plate containing a fluorescent dye and view it under ultraviolet light, where the spots appear dark; or expose the plate to iodine vapour.

A5 CTC-Q19 — [2]

B1 the detector does not respond equally to every compound, so equal amounts of two compounds can give different peak areas. **B1** calibration with standards of the same compound at known concentrations relates that compound's own peak area to its concentration.

SECTION B · Standard, 3 to 5 marks

B1 CTC-Q8 — [4]

B1 GC identifies a component only by matching its retention time to a standard run under the same conditions. **B1** different compounds with similar properties can have the same retention time. **B1** a compound for which no standard was run, or a new one, cannot be identified at all. **B1** in GC–MS each separated component passes into a mass spectrometer, and its mass spectrum is matched against a database of known spectra.

B2 CTC-Q15 — [4]

B1 in TLC the mobile phase is a liquid solvent; in GC it is an unreactive carrier gas. **B1** in TLC the stationary phase is a solid such as silica; in GC it is usually a liquid on a solid support in a column. **B1** TLC identifies components by Rf value; GC by retention time, each compared with standards. **B1** GC gives the relative amounts of the components from peak areas, which TLC does not, but GC works only for compounds that vaporise without decomposing.

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B3 CTC-Q16 **B1** the equal Rf values are consistent with the spot being leucine. **B1** Rf depends on the solvent, the plate and the temperature, which may have differed between the two days. **B1** other amino acids can have the same Rf as leucine in a given solvent. **B1** a sound identification runs the standard on the same plate as the unknown, and confirms it in a second solvent or by another technique. [4]

B4 CTC-Q20 **B1** sugars do not vaporise without decomposing, so they cannot be carried through a GC column as a gas. **B1** HPLC uses a liquid mobile phase, so the sugars stay in solution and need not be vaporised. **B1** the stationary phase is very small particles packed tightly, so the solvent would barely flow under gravity. **B1** the small particles are used because their large surface area gives a much sharper separation. [4]

B5 CTC-Q5 **B1** silica is polar, with O–H groups on its surface. **B1** the polar compound is adsorbed more strongly onto the silica, so it spends more time on the stationary phase and moves a shorter distance. **B1** the non-polar compound is held weakly and spends more time dissolved in the mobile phase, so it moves further and has the higher Rf. [3]

B6 CTC-Q10 **B1** run the two on the same plate in a different solvent, or on a different stationary phase. **B1** different compounds will usually give different Rf values in at least one of the new conditions. **B1** if they still match in every system, and give a single spot when spotted together, they are very likely the same compound. [3]

B7 CTC-Q11 **B1** the sample is injected and vaporised, and an unreactive carrier gas such as helium carries it through a long coiled column in an oven. **B1** the column holds a liquid stationary phase with a high boiling point, coated on a solid support. **B1** components more soluble in the liquid, or less volatile, take longer to reach the detector and so have longer retention times. [3]

B8 CTC-Q13 **B1** a column is packed with a slurry of silica or alumina in the solvent, and the mixture is added at the top. **B1** eluent is run through continuously and the components move down as separate bands at different rates. **B1** each band is collected as a separate fraction at the tap and the solvent is evaporated to leave the component. [3]

B9 CTC-Q14 **B1** propanone is more volatile, so it spends more of its time as vapour in the carrier gas and is carried through faster. **B1** butan-1-ol forms stronger intermolecular forces, including hydrogen bonds, so it dissolves more in the liquid stationary phase and is held back. **B1** a higher oven temperature shortens both retention times, as both spend more time in the gas phase. [3]

SECTION C · Long, 6 marks or more

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C1 CTC-Q17 **Indicative content** [6]

B1 a pencil baseline is drawn about 1 cm from the bottom of a silica plate, handled only by its edges. **B1** small spots of the mixture and of each standard are placed on the baseline with capillary tubes and allowed to dry. **B1** the plate stands in a covered tank of solvent whose level is below the baseline, until the solvent nears the top. **B1** the solvent front is marked in pencil as soon as the plate is removed, and the plate is dried. **B1** the plate is sprayed with ninhydrin in a fume cupboard and warmed, so the amino acids show as purple spots. **B1** the Rf of each spot is calculated and compared with the standards on the same plate; a match identifies the amino acid.

Levels

Level 3 (5–6 marks) all stages are present in order: the pencil baseline, small spots of the mixture and each standard, the covered tank with the solvent below the baseline, marking the solvent front, developing with ninhydrin in a fume cupboard, and the Rf comparison with standards on the same plate.

Level 2 (3–4 marks) the plate is spotted, run and compared with the standards; the solvent level below the baseline, the marked solvent front, the ninhydrin step or the Rf values are not there.

Level 1 (1–2 marks) the mixture is spotted and run, often with the baseline drawn in ink or the solvent above the spots, and the colourless amino acids are never made visible.

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