

2816/03 Experimental Skills 2

January 2003

Mark Scheme

A2 Practical Test 2816/03

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Skill P 16 marks (out of 18 maximum available)

Analytical tests - 6 marks available

- A1 Test for Fe^{2+} :
add sodium hydroxide solution **and** green precipitate/solid formed [1]
Potassium hexacyanoferrate(III) gives a blue coloration
- A2 Precipitate identified **and** a correct equation for the reaction given [1]
 $\text{Fe}^{2+} + 2\text{OH}^- \rightarrow \text{Fe}(\text{OH})_2$
- A3 Test for sulphate ion:
add aqueous barium chloride/nitrate **and** white precipitate formed [1]
- A4 Precipitate identified **and** equation given for reaction [1]
 $\text{FeSO}_4 + \text{BaCl}_2 \rightarrow \text{BaSO}_4(\text{s}) + \text{FeCl}_2$
- A5 Test for hydration: neat diagram of heating/distillation to drive off the water of crystallisation and then collect it. [1]
- A6 Water identified by boiling point of 100°C
or by turning anhydrous CuSO_4 [from white to] blue
or by turning cobalt(II) chloride paper [from blue to] pink [1]

Titration procedure - 5 marks available

- T1 Known mass of iron tablets crushed
and water added to dissolve them (if titrated directly) [1]
Marks T1 and T2 are alternatives
- T2 Known mass of tablet dissolved in water in a volumetric flask [1]
- T3 Known volume of iron(II) salt solution removed by pipette into a conical flask
and [excess] dilute H_2SO_4 added before titration [1]
- T4 KMnO_4 of known concentration is used in the burette [1]
- T5 No indicator is required (must be stated **or** clearly implied)
and end-point colour change is to pale purple/ pink [1]
- T6 Trial **and** two consistent titres obtained [1]
"Within 0.1 cm^3 " implies consistent
The word "accurate" alone does not earn this mark

Quantities - 4 marks

- Q1 1.5 mole reacting ratio for $\text{MnO}_4^-:\text{Fe}^{2+}$ explained or used in a calculation giving ionic half-equation(s), oxidation states **or** a full balanced equation. [1]
- Q2 Calculation to show what a suitable mass of tablet would be to use with the concentration of KMnO_4 as stated. [1]
Correct M_r of hydrated iron(II) sulphate must be used (278)
- Q3 Specimen calculation of % purity of tablet shown clearly [1]
Dilution procedure described in T1 or T2 must be allowed for
- Q4 Assumption is made in the calculation that binder material in the tablets is not an oxidising/reducing agent, so that it does not alter the mean titre. [1]

Safety and Sources - 3 marks

- S1 Hazards researched for **two** chemicals used in the titration procedure [1]
No detailed precautions are needed.
No mark for a gross overstatement of the hazard.
- S2 References to two secondary sources quoted as footnotes **or** at end. [1]
Books must have chapter or page numbers
Internet reference must go beyond the first slash of web address
Accept one specific reference to "Hazardcards" without any qualification
- S3 Good QWC: between 500 and 1100 words **and** with word count given. [1]
Accept a word count given in multiples of 200
QWC: accept no more than three or four errors in spelling or grammar
Treat ICT skills in text (eg "cm³") as an error
Poor scientific language should be penalised eg "weak" for "dilute".

A2 Practical Test (Part B) Mark Scheme

Page 3 Mainly Skill I - [16 marks]

Note that the page total is out of 16, as it includes 2 "Skill A" marks.

Two mass readings clearly listed **and** both quoted to 2 or 3 d.p. [1]
Masses unexplained or simply "mass = " does not earn this mark

Subtraction to give mass of **E** is correct to 2 d.p. **and** unit is given. [1]
*If **only** the mass of H_2X is given, then award **neither** of these marks*

Correctly labelled table (initial, final and difference) used to record titration data
and units are given (somewhere) **and** a trial titre is shown (somewhere) [1]
The trial must be clearly labelled somehow (eg by "T" or "R")

Data from two or more accurate titrations are given by candidate,
and all burette data to at least 0.1 cm^3 **and** all subtractions are correct [1]
*The 1 d.p. stipulation does **not** apply to trial but **does** apply to " 0.0 cm^3 "*

Candidate's ticked/indicated titres (two or more) agree within 0.15 cm^3 . [2]
*Give **one** mark if both/all ticked titres are consistent within 0.25 cm^3*

Mean titre correctly calculated **and** clearly stated **and** with unit cm^3 . [1]
Candidate must not include the trial or deviant readings (outside 0.2 cm^3).

Accuracy and Safety – 8 + 1 marks are available

The accuracy of the titration is assessed by comparing the candidate's calculated M_r of hydrated NiSO_4 with the M_r obtained by the supervisor.

The M_r is calculated using the formula below

$$M_r \text{ of hydrated } \text{NiSO}_4 = \frac{100 \times \text{mass used}}{(2.75 - 0.08 \times \text{mean titre})}$$

Use the conversion chart below to decide the mark out of 8 for accuracy.

If both M_r values are within 1.5	→	[8 marks]
If both M_r values are within 2.5	→	[7]
If both M_r values are within 4.0	→	[6]
If both M_r values are within 5.5	→	[5]
If both M_r values are within 7.0	→	[4]
If both M_r values are within 8.5	→	[3]
If both M_r values are within 10.0	→	[2]
If both M_r values are within 12.0	→	[1 mark]

Put the mark awarded, written as "+6" etc, in the margin.

Safety precaution stated **and** explained briefly [1]

Accept any sensible idea, such as safety specs/ pipette filler.

This must be specifically related to the hazard (harmful/irritant) of the chemicals.

Pages 4 and 5: Skill A (14 marks)

Award error carried forward marks between parts of the question.

One mark is given if the method is correct, but the answer calculated is wrong.

- (a) $n(\text{MgSO}_4)$ used in mean titre, correctly calculated [2]

One mark given for correctly quoting or using the $\frac{MV}{1000}$ formula

Answer to 3 or 4 sig fig: otherwise give one mark

- (b) $n(\text{edta})$ left over [1]

Answer to "b" should be the same as "a".

- (c) $n(\text{edta})$, total, added to nickel salt = $0.025 \times 0.110 = 0.00275$ [2]

Working correct scores one mark.

- (d) $n(\text{edta})$ reacting with nickel salt, correctly calculated [2]

Answer = "c" – "b". (approx 0.001 mol).

*Working correct (ie correct subtraction idea) scores **one** mark*

- (e) $n(\text{nickel salt})$ in 250 cm^3 of solution [1]

Answer = $10 \times \text{"d"}$. No sf penalty

- (f) $M_r = \frac{\text{mass}}{\text{moles}}$ [1]
*Formula in this format **or** appropriate figures from working quoted*

M_r correctly worked out [2]

*Award **1** mark if answer not quoted to nearest integer or to 1 d.p.*

*Award **1** mark only if ecf is being applied.*

M_r of anhydrous nickel(II) sulphate = 155 [1]

Mass of water present = $\frac{M_r - 155}{18}$ (this mark is for the method) [1]

x (correctly worked out **and** quoted to nearest integer **or** one d.p.) [1]

Award this mark only if $x > 1$ and $x < 12$

True value: $x = 6$

Skill E (14 marks)**Question (a) (4 marks)**

- (a) Mass of anhydrous salt = 0.77 g [1]
 $n(\text{NiSO}_4) = \frac{0.77}{154.8} = 0.00497 \text{ (0.00498) mol (no sf penalty)}$ [1]
- (b) M_r of hydrated salt = 263 **or** $n(\text{H}_2\text{O}) = 0.0300 \text{ mol}$ [1]
Mass of water = 108.3 **or** $x = \frac{0.030}{0.00498} \therefore x = 6$ [1]

Question (b) (maximum 10 marks)

- A1 **Use a lid** on the crucible [1]
A2 Lid avoids spitting/escape of solid when heating [1]
A3 Lid is removed during [later] heating to let water vapour escape [1]
A4 Lid reduces absorption/condensation of water during cooling [1]
- B1 **Dehydration might have been incomplete** (*owtte*) [1]
B2 Heat for a longer period **or** more strongly [1]
B3 Re-heat and re-weigh until mass is constant **or** heat to constant mass [1]
Mark B3 automatically earns B2
- C1 **Mass of hydrated salt used was too small** [1]
C2 Use a larger mass of salt [1]
Mark C2 automatically earns C1
- C3 This results in a greater % accuracy in the weighing [1]
- D1 **Heating might have been too strong** [1]
D2 Anhydrous NiSO_4 might undergo thermal decomposition [1]
D3 Oxide(s) of sulphur would be produced **or** recorded loss in mass would be too great [1]
- E1 **Experiment was only carried out once** [1]
E2 Take mean/average of readings **or** repeat until consistent [1]

F1	Don't use yellow flame because of soot content	[1]
F2	Ensure crucible is cool before weighing + reason	[1]
F3	Cool in a desiccator	[1]
F4	This contains a drying agent/dry atmosphere	[1]
F5	Prevents anhydrous residue from re-absorbing water vapour	[1]

Question (c) **(4 marks, maximum)**

G1	Titration involves burette/pipette which is/are accurately calibrated	[1]
G2	Titration was repeated	[1]
G3	Consistent titres are evidence of <u>reliability</u> (<i>ora</i>)	[1]
G4	No evidence that salt was completely dehydrated in gravimetric method	[1]
G5	Gravimetric procedure involves fewer measurements/operations	[1]
G6	Gravimetric is less likely to have experimental/cumulative error	[1]

A – E marks not awarded in (b) can be awarded for answer to part (c) (up to maximum 10)