Candidate 1: Identification of an Unknown

<u>Results</u>

		Repeats	Replicate 1	Replicate 2	Control
					using
					butanone
	Colour		clear,	clear,	clear,
			colourless	colourless	colourless
	State at 25 °C		liquid	liquid	liquid
	Smell		solvent smell	solvent smell	solvent
					smell
	рН		7	7	7
	Saturation		Saturated	Saturated	Saturated
	Polarity		Immiscible in	Immiscible in	Immiscible
			water	water	in water
	Flammability		burns with	burns with	burns with
			clear flame,	clear flame,	clear flame,
			vapour	vapour	vapour
(0			flammable	flammable	flammable
ties	Density:				
bei	Mass of beaker (g)	1	55.3662	52.3475	52.2249
prc		2	55.3623	52.3212	52.2753
ical		3	55.3765	52.3560	52.2249
syr	Mass of beaker and	1	95.4832	92.4396	92.2318
E	50ml of organic	2	95.5323	92.3129	92.3249
	compound (g)	3	95.4832	92.4612	92.2138
	Difference in mass	1	40.1170	40.0921	40.0069
	(g)	2	40.1700	39.9917	40.0496
		3	40.1067	40.1052	39.9889
					
		1	802.3	801.8	800.1
	(mass/volume (0.051))	2	803.4	799.8	801.0
	(Kg/m ³)	3	802.1	802.1	799.8
		Average	802.6	801.3	800.3
	Reaction with codium		no reaction	no reaction	no reaction
	metal		THO TEACTION	no reaction	no reaction
sts	Reaction with Tollens'		Silver mirror	Silver mirror	Silver mirror
te	solution		forms	forms	forms
lica			445	444	444
lem	derivative (%)		115	114	114
ర		1	116	115	117
			110		117
		2	116	11/	116

Melting point of	3	115	117	115
purified derivative	Average	115.7	116.3	116.0
(°C)				

Density Calculations:

The mass of the compound was found using weighing by difference. The mass of the empty beaker was taken away from the mass of the beaker containing the compound to find the mass of the compound. Then the density was calculated by dividing the mass by the volume of the compound. Example calculation using data from the 1st repeat of 1st replicate:

Mass of beaker and compound – Mass of beaker = Mass of compound 95.4832 g - 55.3662 g = 40.1170g Mass of compound \div Volume of compound = Density 40.1170 g \div 0.05000 m³ = 802.3 kg/m³

IR Spectra



Candidate 2: Iron tablets

Experimental Procedure

<u>Kit List</u>

- 4 different types of ferrous sulphate tablets
- 1.5 moll⁻¹ sulfuric acid
- 0.01 moll⁻¹ potassium manganate (VII)
- Deionised water
- Pestle and mortar
- Weighing boat
- Electronic balance
- 250 cm³ beaker
- Filter funnel and paper
- 250cm³ volumetric flask
- 25cm³ pipette
- 50cm³ burette
- 250cm³ conical flask
- A white tile
- Wash bottle
- 100cm³ conical flask and stopper
- 100cm³ measuring cylinder

Stage 1- Preparation of Iron Tablet Solutions

5 iron tablets of one brand were weighed using an electronic balance. These tablets were then transferred into a mortar and were crushed carefully making sure not to lose any of the crushed tablets. Once the tablets were crushed into a fine powder they were transferred into a 100cm³ conical flask along with 50cm³ sulfuric acid. Some of the sulfuric acid was used to rinse the mortar and pestle. These rinsings were also transferred into the conical flask. The flask was stoppered and inverted multiple times before being put aside for 24 hrs to allow the tablets to dissolve fully. The following day the solution was filtered into a 250cm³ volumetric flask using filter paper and filter funnel. Deionised water was used to rinse the original conical flask and these rinsings were also filtered. Deionised water was used to make the solution up to the mark on the 250cm³ volumetric flask. The flask was inverted several times to ensure the solution was mixed well. This whole process was repeated for each brand of tablet.

Stage 2- Redox Titration

The pipette, burette and conical flask were all washed with deionised water. Then the burette was rinsed with potassium manganate (VII) and the pipette was rinsed with the iron tablet solution. Using the pipette filler, the pipette was filled with iron tablet solution and this was transferred to a 250cm³ conical flask. The solution was acidified by adding about 25cm³ sulfuric acid to the conical flask. The burette was then filled with potassium manganate (VII) solution and allowed to slowly run into the burette. While the potassium manganate (VII) ran into the conical flask the contents of the flask was swirled. The titration was carried out until the colour changed from pink/purple to colourless. The potassium manganate (VII) is so intense that the top of the meniscus was used to measure the volumes. For each brand of tablet a rough titration was carried out and then further titrations were done until 2 concordant results were achieved.

Results

Type 1 – Actavis

First Titration:

	Titre (cm ³)			
	Rough	First	Second	Third
Initial burette reading (cm ³)	0.6	12.4	24.0	36.1
Final burette reading(cm ³)	12.4	24.0	36.1	48.0
Volume of KMnO ₄ (cm ³)	11.8	11.6	12.1	11.9
Average volume of concordant results (cm ³)	12.0			
Replicate:				

		Titre (cm³)			
	Rough	First	Second		
Initial burette reading (cm ³)	0.2	12.2	24.4		
Final burette reading(cm ³)	12.2	24.4	36.5		
Volume of KMnO ₄ (cm ³)	12.0	12.2	12.1		
Average volume of concordant results (cm ³)	12.2				

Type 2- Actavis

First Titration:

		Titre (cm³)			
	Rough	First	Second		
Initial burette reading (cm ³)	27.7	11.8	24.0		
Final burette reading(cm ³)	39.7	24.0	36.0		
Volume of KMnO ₄ (cm ³)	12.0	12.2	12.0		
Average volume of concordant results (cm ³)	12.1				

Replicate:

		Titre (cm ³)		
	Rough	First	Second	
Initial burette reading (cm ³)	13.7	25.8	23.0	
Final burette reading(cm ³)	25.8	38.3	35.3	
Volume of KMnO₄ (cm³)	12.1	12.5	12.3	
Average volume of concordant results (cm ³)	12.4			

Type 3- Actavis

First Titration:

	Titre (cm ³)			
	Rough	First	Second	
Initial burette reading (cm ³)	0.2	12.0	23.8	
Final burette reading(cm ³)	12.0	23.8	35.7	
Volume of KMnO₄ (cm³)	11.8	11.8	11.9	
Average volume of concordant results (cm ³)	11.9			

Replicate:

		Titre (cm ³)			
	Rough	First	Second		
Initial burette reading (cm ³)	12.9	25.2	1.2		
Final burette reading(cm ³)	25.2	37.7	13.7		
Volume of KMnO₄ (cm³)	12.3	12.5	12.5		
Average volume of concordant results (cm ³)	12.5				

Type 4- Wockhardt

First Titration:

		Titre (cm ³)			
	Rough	First	Second		
Initial burette reading (cm ³)	0.3	12.4	29.2		
Final burette reading(cm ³)	12.4	24.1	40.8		
Volume of KMnO ₄ (cm ³)	12.1	11.7	11.8		
Average volume of concordant results (cm ³)	11.8				

Replicate:

	Titre (cm ³)			
	Rough	First	Second	Third
Initial burette reading (cm ³)	2.2	14.3	25.9	1.0
Final burette reading(cm ³)	14.3	25.9	38.0	12.9
Volume of KMnO₄ (cm³)	12.1	11.6	12.1	11.9
Average volume of concordant results (cm ³)	12.0			

Calculations
Example Calculation for Type 1 – Actavis
Average volume KMnO ₄ = 12.0 cm ³
Concentration KMnO ₄ = 0.01 moll ⁻¹
Volume Fe ²⁺ solution = 25 cm ³
Moles MnO ₄ = C x V
= 0.01 x 0.012
= 1.2x10 ⁻⁴ moles
5 moles Fe ²⁺ : 1 mole MnO ₄ -
So,
Moles Fe ²⁺ (in 25cm ³) = 5 x 1.2 x10 ⁻⁴ = 6x10 ⁻⁴ moles
Moles Fe ²⁺ (in 250cm ³) = 10 x 6x10 ⁻⁴ = 6x10 ⁻³ moles
Atomic mass Fe = 55.8
Mass of iron (in 5 tablets) = n x gfm
= 6x10 ⁻³ x 55.8
= 0.3348 g
Mass of iron (in one tablet) = 0.3348 /5 = <u>66.96 mg</u>
Replicate
Mass of iron (in one tablet) = 67.80 mg
Average mass of iron = (66.96 + 67.80) / 2 = <u>67.38 mg</u>
Type 2- Actavis
Mass of iron (in one tablet) = <u>67.52 mg</u>
Replicate –
Mass of iron (in one tablet) = <u>69.19mg</u>
Average mass of iron = (67.52 + 69.19) / 2 = 68.36 mg

Type 3- Actavis

Mass of iron (in one tablet) = 66.12 mg

Replicate -

Mass of iron (in one tablet) = 69.75 mg

Average mass of iron = (66.12 + 69.75) / 2 = 67.94 mg

Type 4- Wockhardt

Mass of iron (in one tablet) = 65.57 mg

Replicate -

Mass of iron (in one tablet) = 66.96 mg

Average mass of iron = (65.57 + 66.96) / 2 = 66.27 mg

Summary of Results :

Tablet Type	Average mass of iron (mg)
1	67.38
2	68.36
3	67.94
4	66.27



Candidate 3: Ink analysis



Replicate 1:

Fig.6 Replicate 1 Thin Layer Chromatography Plates

Distance travelled by solvent in control plate (spots 1-3)= 60mm Distance travelled by solvent in unknown plate (spots 4-7)= 62mm

Rf Calculation:

Spot 1:distance traveled by substance= 26mm Rf= 26/60 Rf=0.43

Number corresponding to spot	Distance travelled by substance (mm)	Retardation factor (Rf) Value
1	26	0.43
2	48	0.80
3	41	0.68
4	23	0.37
5	47	0.76
6	24	0.39
7	43	0.69

Replicate 2:





Distance travelled by solvent in control plate (spots 1-3) = 65 Distance travelled by solvent in unknown plate (spots 4-7) = 64

Number corresponding to spot	Distance travelled by substance (mm)	Retardation factor (Rf) Value	Average Rf (Rep.1+2)
1	6	0.09	0.26
2	47	0.72	0.76
3	32	0.49	0.59
4	6	0.09	0.23
5	52	0.81	0.79
6	5	0.08	0.24
7	29	0.45	0.57

Spots 1, 4 and 6 are all the same peach colour and similar average Retardation factor values ranging from 0.23 to 0.26. This suggests that control B is in both samples X and Y. Spots 2 and 5 could be argued to be the same since its is previously suggested that control B is in sample Y therefore spot 5 is a mixture of spot 2 and the pale pink colour at the similar point. Spots 2 and 5 also have similar averageRf values with a difference of 0.03. Spots 3 and 7 are the same dark yellow colour and have similar average Retardation factor values with the difference of 0.02 so could be considered the same substance.

Candidate 4: Calcium in milk

5. <u>Results:</u>

Raw Data:

Skimmed Milk

Trial 1

	Initial Reading	Final Reading	Titre Volume
Rough	1.1	11.2	10.1
1	13.9	24.0	10.1
2	24.0	34.1	10.1
3	34.1	44.3	10.2

Trial 2

	Initial Reading	Final Reading	Titre Volume
Rough	2.2	13.1	10.9
1	21.3	32.6	11.3
2	32.6	43.9	11.3
3	8.5	19.9	11.4

Semi skimmed Milk

Trial 1

	Initial Reading	Final Reading	Titre Volume
Rough	2.5	9.1	6.6
1	10.5	20.0	9.5
2	20.0	29.5	9.5
3	29.5	39.0	9.5

Trial 2

	Initial Reading	Final Reading	Titre Volume
Rough	3.5	13.5	10.0
1	15.2	25.9	10.7
2	26.4	37.1	10.7
3	13.2	23.9	10.7

Whole Milk:

Trial 1

	Initial Reading	Final Reading	Titre Volume
Rough	5.7	15.0	9.3
1	15.0	24.4	9.4
2	24.4	33.8	9.4
3	33.8	43.2	9.4

Trial 2

	Initial Reading	Final Reading	Titre Volume
Rough	6.3	15.8	9.5
1	15.8	25.0	9.2
2	25.0	34.2	9.2
3	34.2	43.5	9.3

Calcium Chloride Control:

Trial 1

	Initial Reading	Final Reading	Titre Volume
Rough	1.0	12.5	11.5
1	12.5	24.0	11.5
2	24.0	35.5	11.5
3	35.5	47.0	11.5

Trial 2

	Initial Reading	Final Reading	Titre Volume
Rough	2.4	14.2	11.8
1	14.2	25.6	11.4
2	25.6	36.9	11.3
3	7.2	18.6	11.4



Processing Results:

Table shows the Calcium Ion Concentration in different Milk and Calcium Chloride control:

	Trial 1	Trial 2	Average
Milk Type	Calcium ion concentration (moll ⁻¹)	Calcium ion concentration (moll-1)	Calcium ion concentration (moll ⁻¹)
Whole	0.0376	0.0369	0.0373
Semi skimmed	0.0380	0.0428	0.0404
Skimmed	0.0405	0.0453	0.0429
Control	0.0460	0.0454	0.0457



Graph 1 -- The average calcium ion concentration in different milk samples

Candidate 5: Piperine in pepper

Results:	
Deve Devela	

caw Results:							
Experiment	Mass of Weighing Boat (g)	Mass of Weighing Boat + Pepper (g)	Initial Mass of Pepper (g)	Mass of Weighing Boat (g)	Mass of Weighing Boat + Mass of Piperine (g)	Final Mass of Piperine (g)	Melting Point (°C)
1	1.6118	11.6158	10.0040	1.6118	2.1357	0.5239	120-
2	1.6132	11.5992	9.9860	1.6132	2.5247	0.9115	122-

Percentage Mass Calculations:

Experiment 1:

$$\% Mass = \frac{Mass of Piperine}{Mass of Pepper} \times 100$$
$$= \frac{0.5239}{10.004} \times 100$$

= 5.24%

Experiment 2:

$$\% Mass = \frac{Mass of Piperine}{Mass of Pepper} \times 100$$
$$= \frac{0.9115}{9.9860} \times 100$$

= 9.13%

Final Results:

Experiment	Percentage Mass of Piperine in Black Pepper (%)	Percentage Uncertainty (± %)	Melting Point (□C)
1	5.24	0.002	120-123
2	9.13	0.001	122-125

Candidate 6: Aspirin

2.2 Results

Table 1 – Values from titration of Flask #1

	1 st Titre	2 nd titre	3 rd titre
Start volume of	0.1	9.3	17.6
sulphuric acid (cm ³)			
End volume of	9.3	17.6	25.9
sulphuric acid (cm³)			
Volume of sulphuric	9.2	8.3	8.3
acid added (cm ³)			

Mass of aspirin in the 3 tablets = 1.503g

Total mass of the 3 tablets = 1.7531g

Mean titre volume = 8.3cm³

Percentage of aspirin in the aspirin tablets = 85.7%

Table 2 – Values from titration of Flask #2

	1 st Titre	2 nd titre
Start point (cm ³)	0.3	8.7
End point (cm³)	8.7	17.0
Volume (cm³)	8.4	8.3

Mass of aspirin in 3 tablets = 1.4985g

Total mass of the 3 tablets =1.8192g

Mean titre volume = 8.35cm³

Percentage of aspirin in the aspirin tablets =82.4%

Example calculations

- Flask #1 titration calculations

 $n \text{ of sulfuric acid} = cv = 0.0083 \times 0.050 = 0.000415 \text{ mol}$

n of NaOH (left in 25ml of the hydrolysed solution) = $2 \times 0.000415 = 0.00083$ mol

(1 mole of sulfuric acid reacts with 2 moles of NaOH)

 $n \text{ of } NaOH \text{ in } 250ml = 10 \times 0.00083 = 0.0083 \text{ mol}$

n of NaOH added to the aspirin tablets originally = $0.025 \times 1 = 0.025$ mol

n of NaOH that reacted with the aspirin tablets = 0.025 - 0.0083 = 0.0167 mol

(2 moles of NaOH react with 1 mole of aspirin)

n of aspirin in sample = $\frac{0.0167}{2}$ = 0.00835 *mol*

 $m \ of \ aspirin \ in \ sample = 0.00835 \times 180 = 1.503g$

% by mass of aspirin in the tablets = $\frac{1.503}{1.7531} \times 100 = 85.7\%$