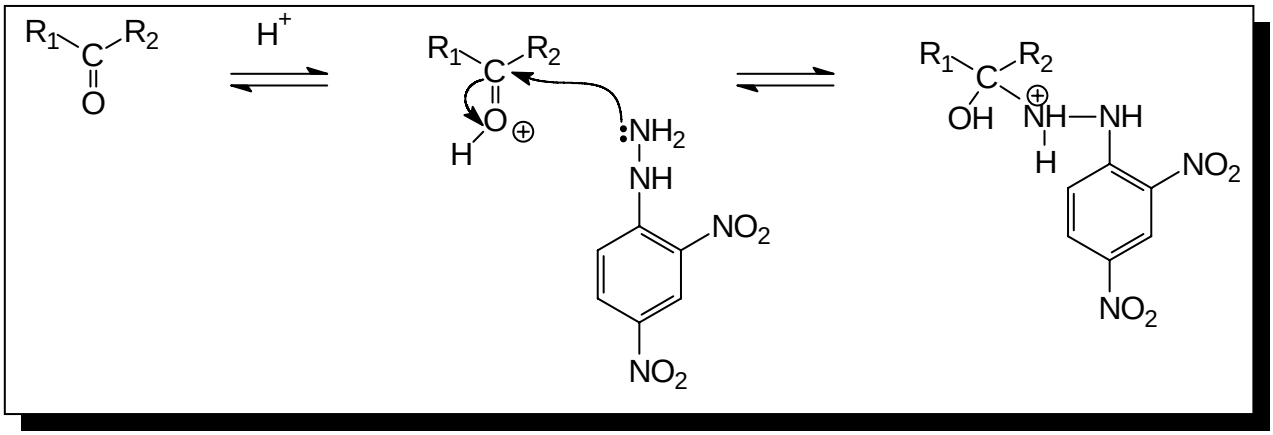
JAMES COOK UNIVERSITY
OF NORTH QUEENSLAND

Department of Chemistry
Cairns Edition



CH1012
REPORTS
2007

Name:

Partner:

REPORT

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CH1012 ASSIGNMENT 1
LABORATORY SAFETY

Date -_____

1. INTRODUCTION

Most laboratory work involves the use of chemicals at some stage. This practical will involve a familiarisation with the typical safety equipment in the laboratory, the use of Material Safety Data Sheets to look at the risks associated with the use of chemicals and the basic steps involved with working safely in a laboratory situation.

2. RESULTS AND DISCUSSION

2.1 Material Safety Data Sheets

Compound 1.

How would you deal with the situation where a large drum of this chemical had broken while putting it down in a Chemical Store room?

An accident occurs during the clean-up and the chemical is splashed into the eyes of one of the people who is assigned to the clean-up duty. What should be done next?

Compound 2.

A fire occurs in a fumecupboard which contains 2 kg of this compound sitting in a bottle at the back of the cupboard. You are the only one in the room what should you do?

In the clean up after the fire the labels on all the bottles in the fumecupboard are found to have vanished. What is an appropriate method for dealing with this situation?

2.2 Obtaining Safety Information

From the library obtain the names of two books concerned with Chemical Laboratory Safety. Provide the proper reference for these books using the system of referencing used in the chemical literature.

1.
2.

From the CRC Handbook of Chemistry and Physics in the laboratory obtain the names of chemicals with the formulae :

$C_6H_{12}O_2$ Name:
$C_5H_{11}Br$ Name:

Search for these compounds using the MSDS database on the WEB (**http://siri.org/msds**), you wish to find information on the health hazards associated with using these compounds.

Health hazards associated with the use of :

$C_6H_{12}O_2$

$C_5H_{11}Br$

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CH1012 ASSIGNMENT 2
COLLOIDS

Date -_____

Experiment	Observation	Explanation
2.1 Dialysis		
2.2 Slime Preparation		

Possible structure of slime :

Question 1 :

2.3 Addition of various solutions to $Fe(OH)_3$ colloid		
2.4 Protective Colloid		
2.5 Flocculation of a clay mineral colloid.		

2.6 Preparation of an emulsion		

Question 2 :

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CH1012 ASSIGNMENT 3
CHEMICAL EQUILIBRIA

Date -_____

Unknown sample number:
Qualitative Examination of Equilibrium

$$2(dmgH) + Ni^{2+} \rightleftharpoons 2H^{+} + Ni(dmg)_{2(aq)}$$
$$Ni(dmg)_{2(aq)} \rightleftharpoons Ni(dmg)_{2(s)} \text{ (red ppt)}$$

TEST	OBSERVATION	EXPLANATION/EQUATIONS
2.1.1 Suspension: + H_2SO_4		
2.1.2 Filtrate:		
(i) + dmg solution		
(ii) + ammonia		
(iii) + sodium acetate		

/4

2.1.3 Metal ammine complexes

METAL ION	OBSERVATION ON NH_3 ADDITION Small	Large	EXPLANATION(S)/EQUATION(S)
Zn^{2+}			
Cu^{2+}			
Fe^{3+}			

GENERAL EXPLANATION

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2.1.4 Silver complexes

(a) What ions are present in the silver nitrate solution in distilled water?
.....

What ions are present in the silver nitrate solution containing excess ammonia?
.....

(b)

TEST	OBSERVATION	EXPLANATION(S)/EQUATION(S)
<u>Addition of NaCl solution</u> $AgNO_3$/distilled water		
$AgNO_3$ in ammonia		

TEST	OBSERVATION	EXPLANATION(S)/EQUATION(S)
<u>Addition of KI solution</u> $AgNO_3$/distilled water		
$AgNO_3$ in ammonia		

/4	

2.2 Quantitative Analysis of a Nickel Salt Solution

mass of crucible =

mass of crucible + precipitate =

mass of precipitate =

∴ mass of Ni in original 25 mL aliquot =

∴ Molarity of Ni^{2+} solution =

WORKING SPACE - show calculation method:

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CH1012 ASSIGNMENT 4
THE COPPER CYCLE

Date -_____

Reclamation of copper

mass of copper wire	=	
mass of crucible	=	
mass of crucible & recovered copper	=	
mass of recovered copper	=	
% recovery	=	

/2	

Step	Observation & Class of reaction	Balanced net ionic equation
2.1 Dissolving copper wire in concentrated nitric acid		
2.2 Addition of ammonia solution		
2.3 Addition of sodium hydroxide solution		

2.4 Heating		
2.5 Addition of sulphuric acid		
2.6 Addition of zinc mesh		
2.8 Treat solid with dilute hydrochloric acid		

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C. Why do you wash the precipitate as described in 2.9?

D. Why do H_2SO_4 and HCl react with zinc but not the precipitated copper?

E. What other metals could be used in step 2.6 instead of zinc?

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CH1012 ASSIGNMENT 5
PREPARATION OF POTASSIUM TRIS(OXALATO) FERRATE(III) HYDRATE

Date -_____

2.1 Preparation of potassium tris(oxolato)ferrate(III)

(*i*) Preparation of Ferrous Oxalate

Equations for reaction(s):

Explanation:

/3	

(*ii*) Oxidation with Hydrogen Peroxide

Equations for reaction(s):

Explanation:

/3	

(*iii*) Crystallization

Equations for reaction(s):

Explanation:

/3	

3. Calculations

3.1 Theoretical yield

Equations for reaction(s):

Calculation:

3.2 Percentage yield

Calculation:

/4	

3.3 Answer to question

/2	

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CH1012 ASSIGNMENT 6
SPECTROSCOPY

Date -_____

At ___ nm the absorption of light by _____ is negligible compared to that of _____. This allows the equilibrium concentration of _____ to be measured spectroscopically, in the presence of _____, at ____. From this the equilibrium constant for the chromate/dichromate equilibrium in aqueous solution was determined to be _____.

/3	

1. Introduction

Concentrations of substances in solution may be found spectrophotometrically[1] using the Beer-Lambert law[1] for absorption of electromagnetic radiation,

$$A = log_{10}(I_0/I_t) = \varepsilon.c.l \qquad (1)$$

where A is the absorbance of the solution, I_0 is the incident intensity of light of a particular wavelength, I_t is the transmitted intensity at that wavelength, ε is the molar absorption coefficient at that wavelength, c is the concentration of the absorbing substance, and l is the path length. The application of this technique is illustrated in a study of the chromate/dichromate equilibrium

$$2CrO_4^{2-} + 2H^+ \rightarrow Cr_2O_7^{2-} + H_2O \qquad (2)$$

in aqueous solution, since it is possible to find a wavelength at which the concentrations of the dichromate ions may be measured with minimal interference from the chromate ion. If the H^+ concentration and the total chromate/dichromate concentration is known (or can be calculated from the "initial" concentrations) then the equilibrium constant

$$K_c = ([Cr_2O_7^{2-}]/[CrO_4^{2-}]^2\,[H^+]^2) \qquad (3)$$

can be evaluated. A convenient way of obtaining a solution of known pH is to use a buffer solution such as sodium (or potassium) acetate and acetic acid.

2. Experimental

Absorbances were measured using a _____spectrophotometer. Correction for the cell and contents other than the species of interest was achieved by zeroing the meter for the appropriate reference solution. For potassium chromate in 1 mol dm^{-3} sodium hydroxide this was a 1 mol dm^{-3} sodium hydroxide solution; and for the potassium dichromate in 2 mol dm^{-3} sulphuric acid a 2 mol dm^{-3} sulphuric acid solution was used. The experiments were carried out at an ambient temperature of __ °C. All dilutions were carried out using B grade volumetric apparatus.

As described below, a suitable wavelength for the measurement of the absorbance of dichromate, in the presence of chromate, was found to be ____nm. Plotting the absorbances of four different dichromate solutions (0.4000, 0.8000, 1.200, and 1.600 mmol dm^{-3}) in 2 mol dm^{-3} sulphuric acid against concentration allowed determination of the molar absorption coefficient at the chosen wavelength. This plot was used as a calibration curve for determination of unknown concentrations from absorbance measurements.

/1	

3. Results

The absorption spectrum of dichromate, from 400 to 600 nm (Table 1 and Figure 1), was obtained by using a strong acid solution (2 mol dm^{-3} H_2SO_4) since the concentration of chromate in such a solution is negligible. To obtain the absorbance spectrum of chromate, from 400 to 600nm (Table 1 and Figure 1), a strongly basic solution (1 mol dm^{-3} NaOH) was used since the concentration of dichromate is negligible in this case.

It is possible to measure the concentration of dichromate, independent to that of chromate, if a wavelength can be found at which dichromate absorbs strongly while the absorbance due to chromate is negligible. Inspection of Figure 1 shows that ____nm is a suitable wavelength. In order to use absorbance measurements, at the chosen wavelength, it is necessary to know the molar absorption coefficient **ε**, of the dichromate solution. This can be found by plotting the absorbance of the solution against dichromate concentration, use the Beer-Lambert Law ($A = \varepsilon.c.l$) to calculate ε (where the path length l = 1cm). Such a plot is shown in Figure 2 (data Table 2). It can be seen that this plot **does/does not** obey the Beer-Lambert law (equation 1).

The value for the molar absorption coefficient ε was found to be _____.

To obtain the equilibrium constant K_c (equation 3), the equilibrium concentration of dichromate was found for a solution by mixing 5 cm^3 of 3.200 mmol dm^{-3} potassium chromate, in 1 mol dm^{-3} NaOH, with 5 cm^3 of 1.23 mol dm^{-3} acetic acid. It was found by measuring the absorbance of the solution at _____ nm which was found to be:

A =

Inverse interpolation from the graph gives the dichromate concentration of :

$[Cr_2O_7^{2-}]$ =

Since the original concentration of chromate in the mixture was: _____ _____
(value + units) ,

the equilibrium concentration of chromate is

$[CrO_4^{2-}]_{equil}$ = _____ _____ (see Appendix 1).

The solution is buffered by NaOH and acetic acid to a pH of 5.4, so that the H^+ concentration is $[H^+]$ = _____ _____

Using these concentrations in equation (3) gives the value for the equilibrium constant to be

K_c = _____ _____ at ____^oC

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Table 1. Absorbance values at different wavelengths for 0.0016 mol dm^{-3} potassium dichromate in 2 mol dm^{-3} H_2SO_4 solution and of 0.0004 mol dm^{-3} potassium chromate in 1 mol dm^{-3} NaOH solution.

λ/nm	$A(Cr_2O_7^{2-})$	$A(CrO_4^{2-})$	λ/nm	$A(Cr_2O_7^{2-})$	$A(CrO_4^{2-})$
400			465		
410			470		
415			475		
420			480		
425			490		
430			500		
435			510		
440			520		
445			530		
450			540		
455			550		
460			560		

Table 2. Absorbance of $K_2Cr_2O_7$ in 2 mol dm^{-3} sulphuric acid solution at _____nm.

$[Cr_2O_7^{2-}]$/ mol dm^{-3}	0.0000	0.0004	0.0008	0.0012	0.0016
A at _____nm					

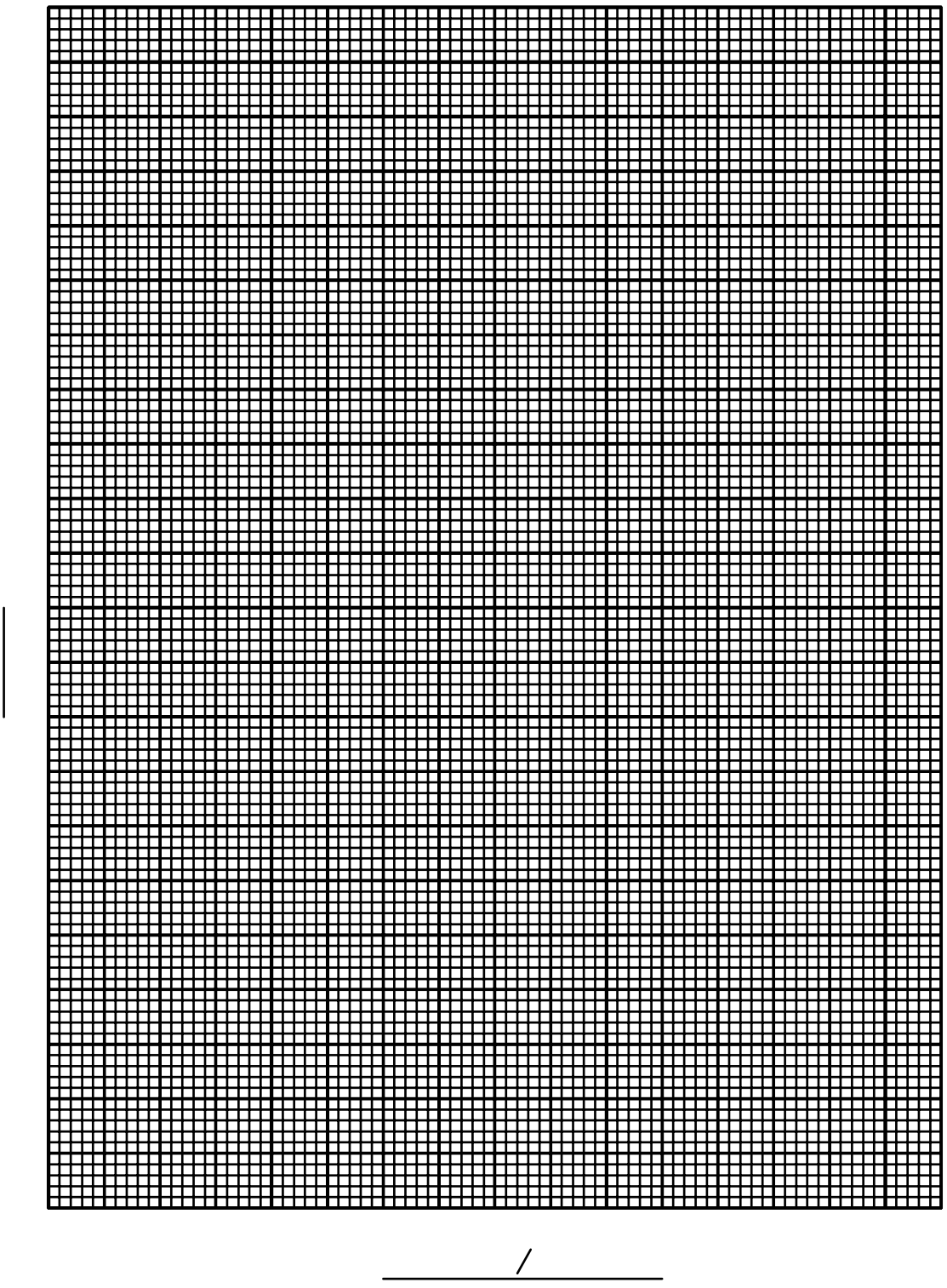

Figure 1. Absorbance of a) _____ M dichromate solution and b) _____ M chromate solution, as a function of wavelength at _____ ^{o}C

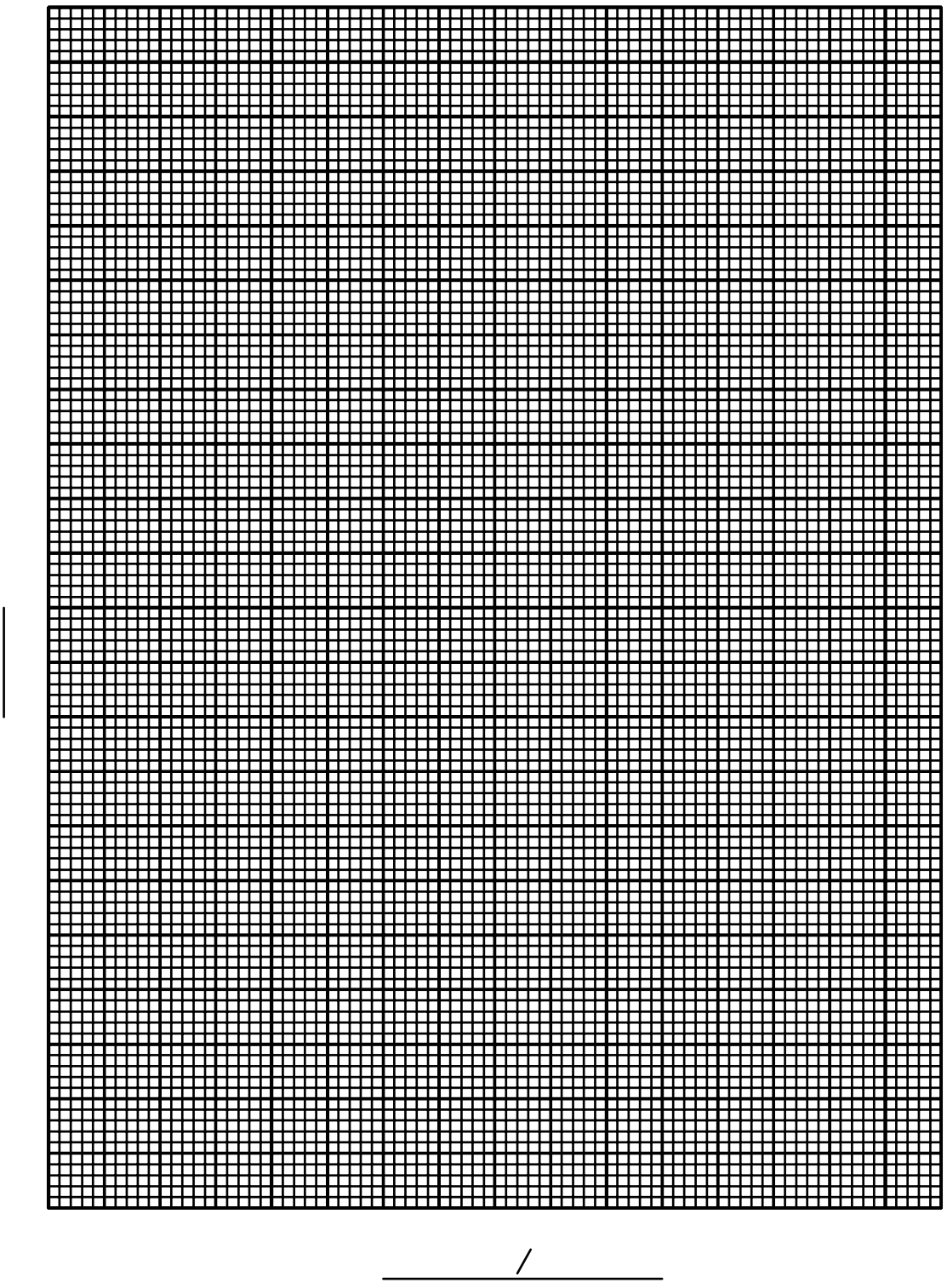

Figure 2. Absorbance (A) of dichromate as a function of concentration at _____ ^{o}C

4. Discussion

/4	

5. References

1. I.N. Levine, "Physical Chemistry", 4th edition, McGraw-Hill, New York, 1995.

2. G.H. Aylward and T.J.V. Findlay, "SI Chemical Data", third edition, John Wiley and Sons, Hong Kong, 1994.

3. "Handbook of Chemistry and Physics", 76th. edition, Ed. D.R. Lide, Chemical Rubber Co., New York, 1996.

4. _____.

/1	

Appendix 1. Equilibrium concentration of chromate

Initial and final (equilibrium) concentrations of chromate and dichromate are given by:

$2CrO_4^{2-}$ + $2H^+$ ---> $Cr_2O_7^{2-}$ + H_2O

initial concentrations a 0

final concentrations a - 2c c

where a = _____._____ (amount + units)

and c = _____._____

so $[CrO_4^{2-}]_{equil}$ = (a - ____) (general expression)

= (_____ - _____) (expression in values + units)

= _____ _____ (result + units)

K_c = _____ (general expression)

= _____ (expression in values + units)

= _____ (results + units)

ε = _____ (general expression)

= _____ (expression in values + units)

= _____ (result + units)

/3	

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CH1012 ASSIGNMENT 7
QUALITATIVE ANALYSIS OF ORGANIC COMPOUNDS

Date -_____

The relevant equations should be written on the last two pages of this report.

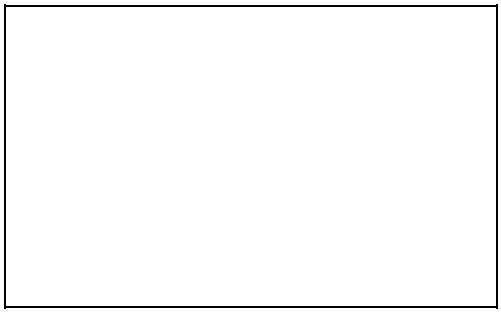
Structure of aniline

Solubility of aniline

Solvent	Observation	Explanation / Equation
water		
5% HCl		

/2	

Structure of benzoic acid

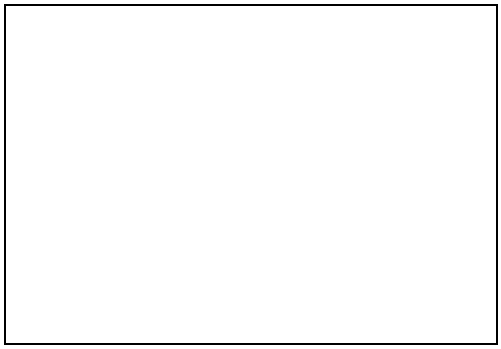


Solubility of benzoic acid

Solvent	Observation	Explanation / Equation
water		
$NaHCO_3$ solution		

Multiple Bond Test

Structure of cholesterol



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Bromination of Cholesterol and Aniline with Br_2 in CH_2Cl_2

Compound	Observation	Explanation / Equation
Cholesterol Br_2 / CH_2Cl_2		
Aniline Br_2 / CH_2Cl_2		

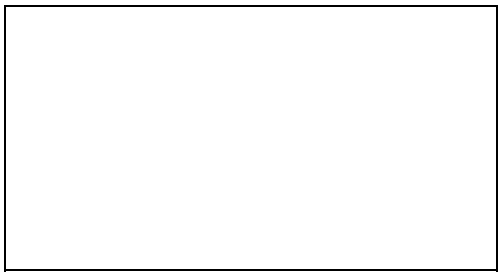
Structure of *o*-methoxyphenol

Phenol test on *o*-methoxyphenol

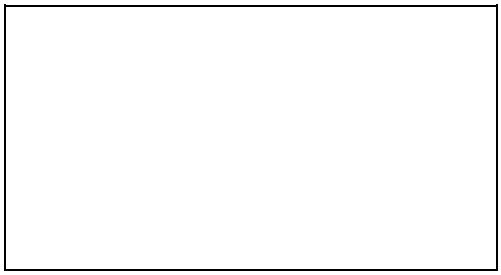
Solvent	Observation	Explanation / Equation
water		
ethanol		

/2

Structure of acetone



Structure of benzaldehyde



DNP test for carbonyls

Compound	Observation	Explanation / Equation
Acetone		
benzaldehyde		

/2

Schiff's test for aldehydes:

Compound	Observation	Explanation / Equation
Acetone		
benzaldehyde		

Structure of ethyl acetate

Ester test on ethyl acetate

Reagent	Observation	Explanation / Equation
5% $H_2NOH.HC1$		
KOH/MeOH, boil, cool		
dilute HCl solution		
$FeC1_3$ solution		

/2

Structure of acetamide

Amide test on acetamide

Reagent	Observation	Explanation / Equation
3M NaOH solution		
3M HCl solution		

/2

QUALITATIVE ANALYSIS OF ORGANIC COMPOUNDS - UNKNOWNS

unknown ☐

Preliminary Tests:

1. Melting point / boiling point

2. Description:

3. Solubility group:

4. Functional group present:
(from IR absorption)

5. Elements present:

The above information indicates that the unknown is likely to be one of the following compounds:

Confirmatory Tests:

6. Alkenes: Observation / Conclusion

7. Phenols: Observation / Conclusion

8. Carbonyl groups: DNP reagent

   Schiff's reagent

9. Amines: Observation / Conclusion

10. Carboxylic Acids: Observation / Conclusion

11. Esters: Observation / Conclusion

12. Amides: Observation / Conclusion

The above tests confirm that the unknown compound is most likely to be:

/3

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CH1012 ASSIGNMENT 8
ELECTROPHILIC AROMATIC SUBSTITUTION
& NUCLEOPHILIC SUBSTITUTION AT SATURATED CARBON

Date -_____

2.1 2,4,6- Tribromoaniline: Electrophilic substitution on an activated aromatic ring

Reaction (equation):

Description of experiment:

Description of product:

m.p. range

Comparison with Literature:	
Yield	g
% Yield*	

*Show calculations below

Answer to question 1. Equations for brominations.

% yield calculation:

/7	

4.1 Preparation of *t*-butyl chloride

Reaction:

Description of experiment:

Description of product:

b.p. range

Comparison with Literature

% Yield

% yield calculation:

Answer to question 2. Relative ease of reactions.

/8	

/20	

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CH1012 ASSIGNMENT 9
REACTIONS OF CARBONYL COMPOUNDS

Date -_____

Identification of an unknown carbonyl compound

2.1 Boiling point determination

unknown

b.p.

2.2 DNP derivative

Reaction:

Description of experiment:

Description of product:

m.p. range

Identification of unknown

identity of unknown

Question 1. Three <u>other</u> nucleophilic addition reactions (with different nucleophiles).

1.

2.

3.

/7

Esterification and hydrolysis

Starting compound **salicylic acid**

5.1 Esterification

Reaction:

Description of experiment:

Description of product:

5.2 Hydrolysis

Reactions:

Description of experiment:

Description of product:

m.p. range	
Comparison with Literature	
Yield	g
% Yield*	

*Show calculations below.

% yield calculation:

5.3 Answers to questions 2. - 4.

Question 2.

Question 3.

Question 4.

/8	

/20	