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Instructions

- Ensure that your name and student code are written in the spaces provided at the top of each page.
- You have 5 hours to work on the problems. Begin only when the START command is given.
- Use only the calculator provided.
- All results must be written in the appropriate boxes. Anything written elsewhere will not be marked. Use the reverse of the sheets if you need scrap paper.
- Write any relevant calculations in the appropriate boxes when necessary. If you provide no working and only the correct result for a complicated calculation, you will receive no marks.
- **Numerical answers are meaningless without the appropriate units. You will be heavily penalised if units are not given where required.** You should also take care to report answers to an appropriate number of significant figures.
- Treat all gases as ideal.
- You must stop work immediately when the STOP command is given. A delay in doing this may lead to your disqualification from the exam.
- When you have finished the examination, you must put your papers into the envelope provided. Do not seal the envelope.
- Do not leave the examination hall until instructed by the supervisors.
- This examination has **42** pages.
- The official English version of this examination is available on request only for clarification.

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Physical constants

Name	Symbol	Value
Avogadro constant	N_A	$6.0221 \times 10^{23} \text{ mol}^{-1}$
Boltzmann constant	k_B	$1.3807 \times 10^{-23} \text{ J K}^{-1}$
Gas constant	R	$8.3145 \text{ J K}^{-1} \text{ mol}^{-1}$
Faraday constant	F	96485 C mol^{-1}
Speed of light	c	$2.9979 \times 10^8 \text{ m s}^{-1}$
Planck constant	h	$6.6261 \times 10^{-34} \text{ J s}$
Standard pressure	p°	10^5 Pa
Atmospheric pressure	p_{atm}	$1.01325 \times 10^5 \text{ Pa}$
Zero of the Celsius scale		273.15 K
Standard acceleration of free fall	g	9.807 m s^{-2}
Bohr magneton	μ_B	$9.274015 \times 10^{-24} \text{ J T}^{-1}$

Provide standard sheet of equations



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Periodic table with relative atomic masses

1																	18														
1 H 1.008																	2 He 4.003														
3 Li 6.94	4 Be 9.01											5 B 10.81	6 C 12.01	7 N 14.01	8 O 16.00	9 F 19.00	10 Ne 20.18														
11 Na 22.99	12 Mg 24.31											13 Al 26.98	14 Si 28.09	15 P 30.97	16 S 32.06	17 Cl 35.45	18 Ar 39.95														
19 K 39.102	20 Ca 40.08	21 Sc 44.96	22 Ti 47.90	23 V 50.94	24 Cr 52.00	25 Mn 54.94	26 Fe 55.85	27 Co 58.93	28 Ni 58.71	29 Cu 63.55	30 Zn 65.37	31 Ga 69.72	32 Ge 72.59	33 As 74.92	34 Se 78.96	35 Br 79.904	36 Kr 83.80														
37 Rb 85.47	38 Sr 87.62	39 Y 88.91	40 Zr 91.22	41 Nb 92.91	42 Mo 95.94	43 Tc	44 Ru 101.07	45 Rh 102.91	46 Pd 106.4	47 Ag 107.87	48 Cd 112.40	49 In 114.82	50 Sn 118.69	51 Sb 121.75	52 Te 127.60	53 I 126.90	54 Xe 131.30														
55 Cs 132.91	56 Ba 137.34	57 La*	58 Ce 140.12	59 Pr 140.91	60 Nd 144.24	61 Pm	62 Sm 150.4	63 Eu 151.96	64 Gd 157.25	65 Tb 158.93	66 Dy 162.50	67 Ho 164.93	68 Er 167.26	69 Tm 168.93	70 Yb 173.04	71 Lu 174.97	72 Hf 178.49	73 Ta 180.95	74 W 183.85	75 Re 186.2	76 Os 190.2	77 Ir 192.2	78 Pt 195.09	79 Au 196.97	80 Hg 200.59	81 Tl 204.37	82 Pb 207.2	83 Bi 208.98	84 Po	85 At	86 Rn
87 Fr	88 Ra	89 Ac ⁺																													

*Lanthanides	58 Ce 140.12	59 Pr 140.91	60 Nd 144.24	61 Pm	62 Sm 150.4	63 Eu 151.96	64 Gd 157.25	65 Tb 158.93	66 Dy 162.50	67 Ho 164.93	68 Er 167.26	69 Tm 168.93	70 Yb 173.04	71 Lu 174.97
⁺ Actinides	90 Th 232.01	91 Pa	92 U 238.03	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	103 Lr

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Problem 1

9% of the total

Estimating the Avogadro constant

1a	1b	1c	1d	1e	1f	1g	1h	1i	1j	1k	Total
4	4	4	2	1	2	3	6	4	3	3	36

Many different methods have been used to determine the Avogadro constant. Three different methods are given below.

Method A – from X-ray diffraction data (modern)

The unit cell is the smallest repeating unit in a crystal structure. The unit cell of a gold crystal is found by X-ray diffraction to have the face-centred cubic unit structure (i.e. where the centre of an atom is located at each corner of a cube and in the middle of each face). The side of the unit cell is found to be 0.408 nm.

- a) Sketch the unit cell and calculate how many Au atoms the cell contains.

Unit cell:

Number of Au atoms in the unit cell:



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b) The density of Au is $1.93 \times 10^4 \text{ kg m}^{-3}$. Calculate the volume and mass of the cubic unit cell.

Volume:

Mass:

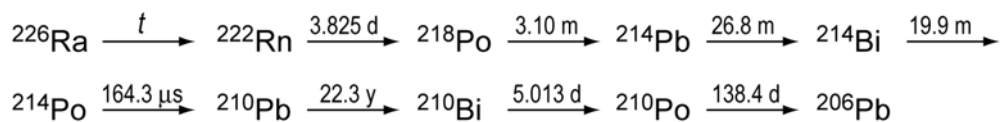
c) Hence calculate the mass of a gold atom and the Avogadro constant, given that the relative atomic mass of Au is 196.97.

Mass of Au atom:

Avogadro constant:

Method B – from radioactive decay (Rutherford, 1911)

The radioactive decay series of ^{226}Ra is as follows:



The times indicated are half-lives, the units are y = years, d = days, m = minutes. The first decay, marked t above, has a much longer half-life than the others.



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- d) In the table below, identify which transformations are α -decays and which are β -decays.

	α -decay	β -decay
$^{226}\text{Ra} \longrightarrow ^{222}\text{Rn}$		
$^{222}\text{Rn} \longrightarrow ^{218}\text{Po}$		
$^{218}\text{Po} \longrightarrow ^{214}\text{Pb}$		
$^{214}\text{Pb} \longrightarrow ^{214}\text{Bi}$		
$^{214}\text{Bi} \longrightarrow ^{214}\text{Po}$		
$^{214}\text{Po} \longrightarrow ^{210}\text{Pb}$		
$^{210}\text{Pb} \longrightarrow ^{210}\text{Bi}$		
$^{210}\text{Bi} \longrightarrow ^{210}\text{Po}$		
$^{210}\text{Po} \longrightarrow ^{206}\text{Pb}$		

- e) A sample containing 192 mg of ^{226}Ra was purified and allowed to stand for 40 days. Identify the first isotope in the series (excluding Ra) that has not reached a steady state.
- f) The total rate of α -decay from the sample was then determined by scintillation to be 27.7 GBq (where 1 Bq = 1 count s^{-1}). The sample was then sealed for 163 days. Calculate the number of α particles produced.



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g) At the end of the 163 days the sample was found to contain 10.4 mm^3 of He, measured at 101325 Pa and 273 K. Calculate the Avogadro constant from these data.

h) Given that the relative isotopic mass of ^{226}Ra measured by mass spectrometry is 226.25, use the textbook value of the Avogadro constant ($6.022 \times 10^{23} \text{ mol}^{-1}$) to calculate the number of ^{226}Ra atoms in the original sample, n_{Ra} , the decay rate constant, λ , and the half-life, t , of ^{226}Ra (in years). You need only consider the decays up to but not including the isotope identified in (e).

$n_{\text{Ra}} =$

$\lambda =$

$t =$



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Method C – dispersion of particles (Perrin, 1909)

One of the first accurate determinations of the Avogadro constant was carried out by studying the vertical distribution under gravity of colloidal particles suspended in water. In one such experiment, particles with radius 2.12×10^{-7} m and density 1.206×10^3 kg m⁻³ were suspended in a tube of water at 15 °C. After allowing sufficient time to equilibrate, the mean numbers of particles per unit volume observed at four heights from the bottom of the tube were:

height / 10 ⁻⁶ m	5	35	65	95
mean number per unit volume	4.00	1.88	0.90	0.48

- i) Assuming the particles to be spherical, calculate: the mass, m , of a particle; the mass of the water it displaces, $m_{\text{H}_2\text{O}}$; and the effective mass, m^* , of the particle in water accounting for buoyancy (i.e. taking account of the upthrust due to the displaced volume of water). Take the density of water to be 999 kg m⁻³.

$m =$

$m_{\text{H}_2\text{O}} =$

$m^* =$



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At equilibrium, the number of particles per unit volume at different heights may be modelled according to a Boltzmann distribution:

$$\frac{n_h}{n_{h_0}} = \exp\left[-\frac{E_h - E_{h_0}}{RT}\right]$$

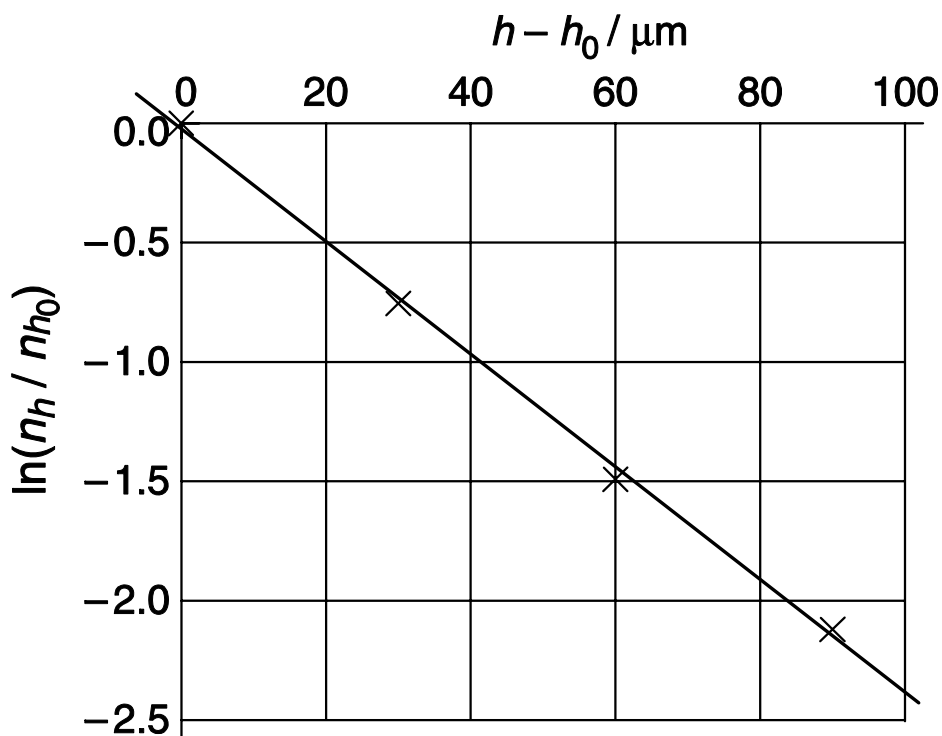
where n_h is the number of particles per unit volume at height h ,

n_{h_0} is the number of particles per unit volume at the reference height h_0 ,

E_h is the gravitational potential energy per **mole** of particles at height h relative to the particles at the bottom of the tube,

R is the gas constant, $8.3145 \text{ J K}^{-1} \text{ mol}^{-1}$.

A graph of $\ln(n_h / n_{h_0})$ against $(h - h_0)$, based on the data in the table above, is shown below. The reference height is taken to be $5 \mu\text{m}$ from the bottom of the tube.





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j) Derive an expression for the gradient (slope) of the graph.

k) Determine the Avogadro constant from these data.

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Problem 2

9% of the total

Interstellar production of H₂

2a	2b	2c	2d	2e	2f	2g	2h	2i	Total
2	2	4	2	6	6	3	2	6	33

If two atoms collide in interstellar space the energy of the resulting molecule is so great that it rapidly dissociates. Hydrogen atoms only react to give stable H₂ molecules on the surface of dust particles. The dust particles absorb most of the excess energy and the newly formed H₂ rapidly desorbs. This question examines two kinetic models for H₂ formation on the surface of a dust particle.

In both models, the rate constant for adsorption of H atoms onto the surface of dust particles is $k_a = 1.4 \times 10^{-5} \text{ cm}^3 \text{ s}^{-1}$. The typical number density of H atoms (number of H atoms per unit volume) in interstellar space is $[\text{H}] = 10 \text{ cm}^{-3}$.

[Note: in the following, you may treat numbers of surface-adsorbed atoms and number densities of gas-phase atoms in the same way as you would normally use concentrations in the rate equations. As a result, the units of the rate constants may be unfamiliar to you. Reaction rates have units of numbers of atoms or molecules per unit time.]

- a) Calculate the rate at which H atoms adsorb onto a dust particle. You may assume that this rate is constant throughout.



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Desorption of H atoms is first order with respect to the number of adsorbed atoms. The rate constant for the desorption step is $k_d = 1.9 \times 10^{-3} \text{ s}^{-1}$.

- b) Assuming that only adsorption and desorption take place, calculate the steady-state number, N , of H atoms on the surface of a dust particle.

The H atoms are mobile on the surface. When they meet they react to form H_2 , which then desorbs. The two kinetic models under consideration differ in the way the reaction is modelled, but share the same rate constants k_a , k_d , and k_r , for adsorption, desorption, and bimolecular reaction, as given below.

$$k_a = 1.4 \times 10^{-5} \text{ cm}^3 \text{ s}^{-1}$$

$$k_d = 1.9 \times 10^{-3} \text{ s}^{-1}$$

$$k_r = 5.1 \times 10^4 \text{ s}^{-1}$$



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Model A

Reaction to form H_2 is assumed to be second order. On a dust particle the rate of removal of H atoms by reaction is $k_r N^2$.

- c) Write down an equation for the rate of change of N , including adsorption, desorption and reaction. Assuming steady state conditions, determine the value of N .

$N =$

- d) Calculate the rate of production of H_2 per dust particle in this model

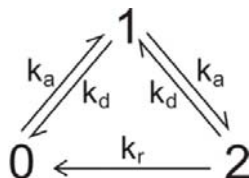


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Model B

Model B attempts to analyse the probability that the dust particles carry 0, 1 or 2 H atoms. The three states are linked by the following reaction scheme. The assumption is made that no more than 2 atoms may be adsorbed simultaneously.



x_0 , x_1 and x_2 are the fractions of dust particles existing in state 0, 1 or 2, respectively. These fractions may be treated in the same way as concentrations in the following kinetic analysis. For a system in state m with fraction x_m , the rates of the three possible processes are

Adsorption ($m \rightarrow m + 1$): rate = $k_a[H]x_m$

Desorption ($m \rightarrow m - 1$): rate = $k_d m x_m$

Reaction ($m \rightarrow m - 2$): rate = $\frac{1}{2} k_r m(m-1)x_m$

- e) Write down equations for the rates of change, dx_m/dt , of the fractions x_0 , x_1 and x_2 .



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- f)** Assuming steady-state conditions, use the above rate equations to find expressions for the ratios x_2/x_1 and x_1/x_0 , and evaluate these ratios.

- g)** Evaluate the steady state fractions x_0 , x_1 and x_2

[If you were unable to determine the ratios in **(f)**, use $x_2/x_1 = a$ and $x_1/x_0 = b$ and give the result algebraically].



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h) Evaluate the rate of production of H₂ per dust particle in this model

i) It is currently not possible to measure the rate of this reaction experimentally, but the most recent computer simulations of the rate give a value of $9.4 \times 10^{-6} \text{ s}^{-1}$. Which of the following statements apply to each model under these conditions? Mark any box you consider to be appropriate.

Statement	Model A	Model B	Neither model
The rate determining step is adsorption of H atoms.			
The rate-determining step is desorption of H ₂ molecules.			
The rate determining step is the bimolecular reaction of H atoms on the surface.			
The rate determining step is adsorption of the second H atom.			
The implicit assumption that reaction can take place regardless of the number of atoms adsorbed leads to substantial error (at least a factor of two).			
Limiting the number of atoms adsorbed on the particle to 2 leads to substantial error (at least a factor of two).			

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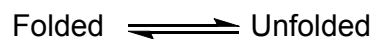
Problem 3

11% of the total

Protein Folding

3a	3b	3c	3d	3e	3f	3g	3h	Total
2.5	3.5	1	6	2	4	2	2	23

The unfolding reaction for many small proteins can be represented by the equilibrium:



You may assume that the protein folding reaction takes place in a single step. The position of this equilibrium changes with temperature; the melting temperature T_m is defined as the temperature at which half of the molecules are unfolded and half are folded.

The intensity of the fluorescence signal at a wavelength of 356 nm of a 1.0 μM sample of the protein Chymotrypsin Inhibitor 2 (CI2) was measured as a function of temperature over the range 58 to 66 $^{\circ}\text{C}$:

Temp / $^{\circ}\text{C}$	58	60	62	64	66
Fluorescence intensity (arbitrary units)	27	30	34	37	40

A 1.0 μM sample in which all of the protein molecules are folded gives a fluorescence signal of 21 units at 356 nm. A 1.0 μM sample in which all of the protein molecules are unfolded gives a fluorescence signal of 43 units.



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- a) Assuming that the fluorescence intensity from each species is directly proportional to its concentration, calculate the fraction, x , of unfolded molecules present at each temperature.

Temp /°C	58	60	62	64	66
x					

- b) Give an expression for the equilibrium constant, K , in terms of x , and hence calculate the value of K at each temperature.

Temp /°C	58	60	62	64	66
K					



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- c) Estimate the value of T_m for this protein (to the nearest 1°C).

$$T_m =$$

Assuming that the values of ΔH° and ΔS° for the protein unfolding reaction are constant with temperature then:

$$\ln K = -\frac{\Delta H^\circ}{RT} + C$$

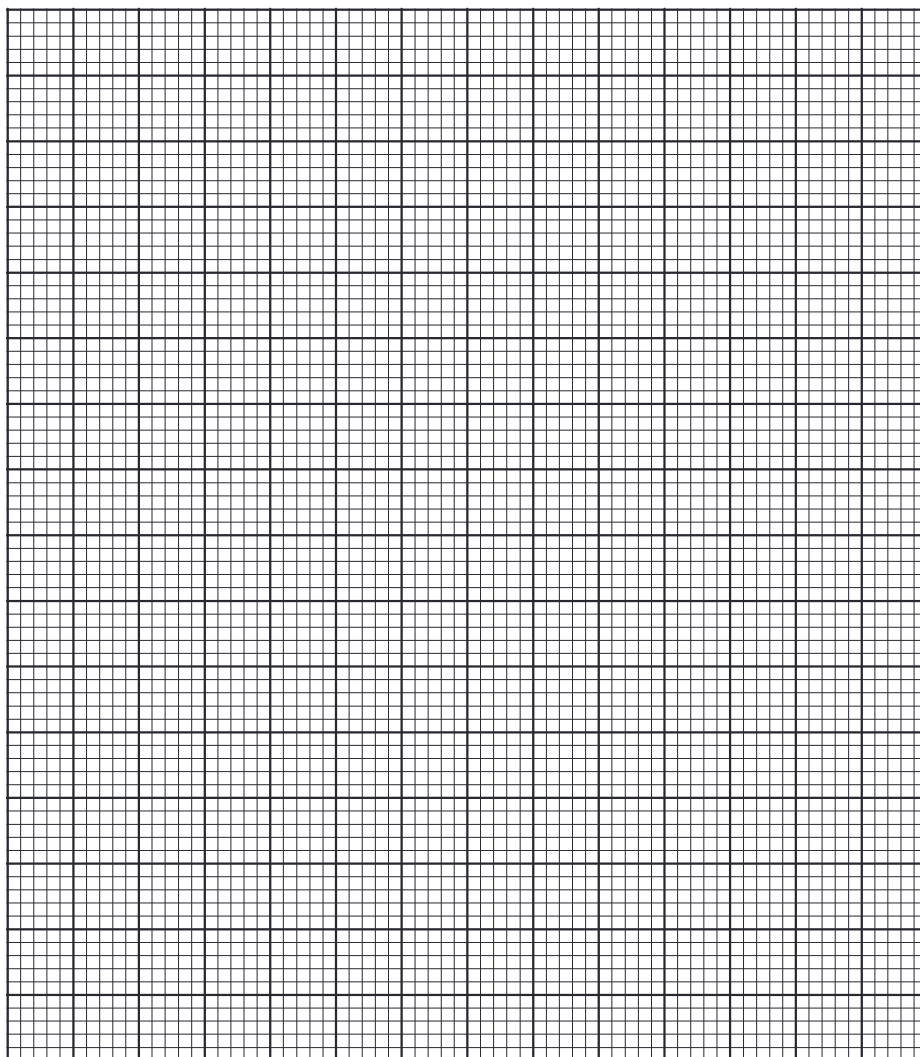
where C is a constant.

- d) Plot a suitable graph and hence determine the values of ΔH° and ΔS° for the protein unfolding reaction.



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$\Delta H^\circ =$

$\Delta S^\circ =$

If you have been unable to calculate values for ΔH° and ΔS° , you should use the following incorrect values for the subsequent parts of the question:

$\Delta H^\circ = 130 \text{ kJ mol}^{-1}$

$\Delta S^\circ = 250 \text{ J K}^{-1} \text{ mol}^{-1}$



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- e) Calculate the equilibrium constant for the unfolding reaction at 25 °C.

$K =$

If you have been unable to calculate a value for K , you should use the following incorrect values for the subsequent parts of the question: $K = 3.6 \times 10^{-6}$

The first order rate constant for the CI2 protein folding reaction can be determined by following the fluorescence intensity when a sample of unfolded protein is allowed to refold (typically the pH of the solution is changed). The concentration of protein when a 1.0 μM sample of unfolded CI2 was allowed to refold was measured at a temperature of 25 °C:

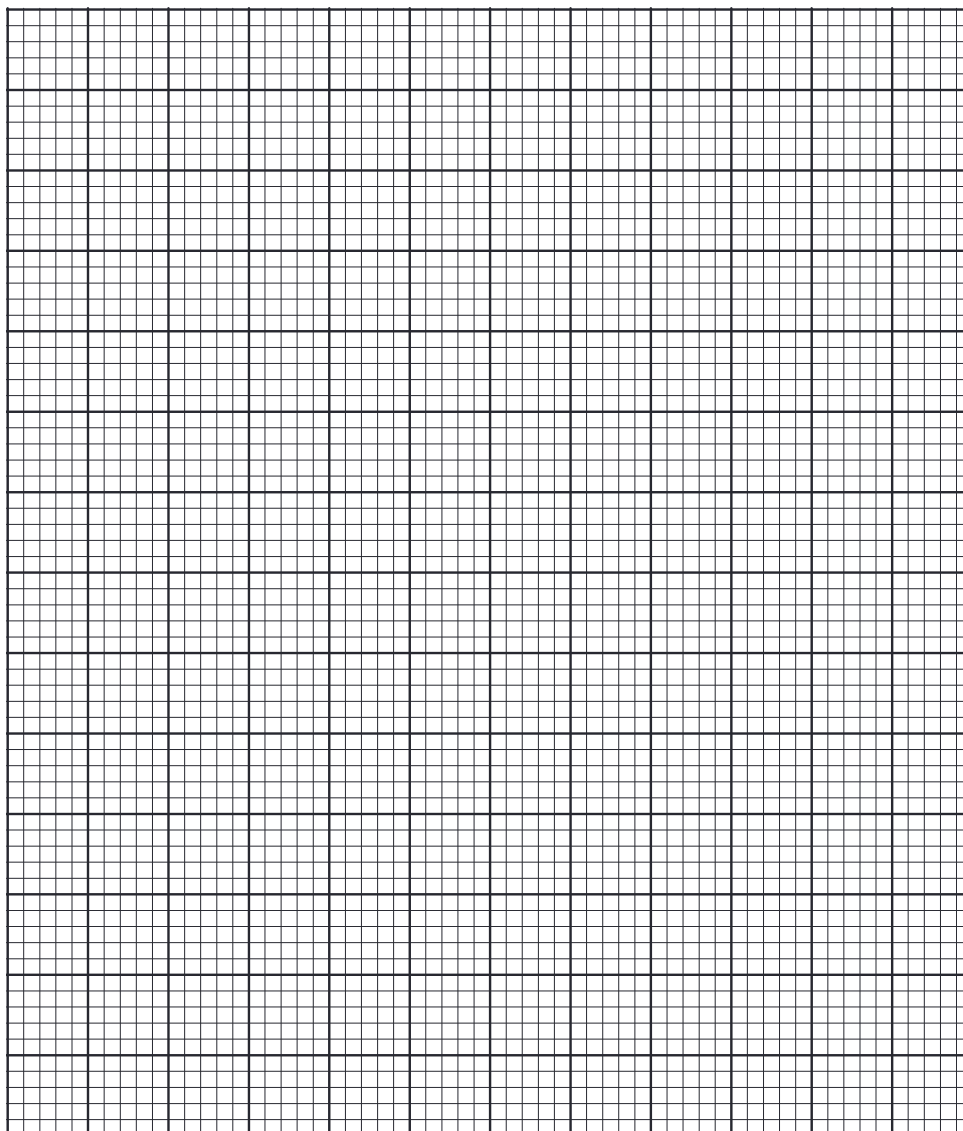
time / ms	0	10	20	30	40
concentration / μM	1	0.64	0.36	0.23	0.14

- f) Plot a suitable graph and hence determine the value of the rate constant for the protein folding reaction, k_f , at 25 °C.



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$k_f =$

If you have been unable to calculate the values for k_f , you should use the following incorrect value for the subsequent parts of the question: $k_f = 60 \text{ s}^{-1}$.



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- g)** Determine the value of the rate constant for the protein *unfolding* reaction, k_u , at 25 °C.

$k_u =$

- h)** At 20 °C the rate constant for the protein folding reaction is 33 s^{-1} . Calculate the activation energy for the protein folding reaction.

Activation energy =

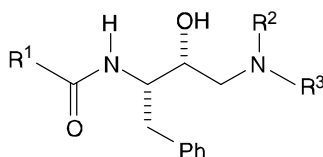
Problem 4

9% of the total

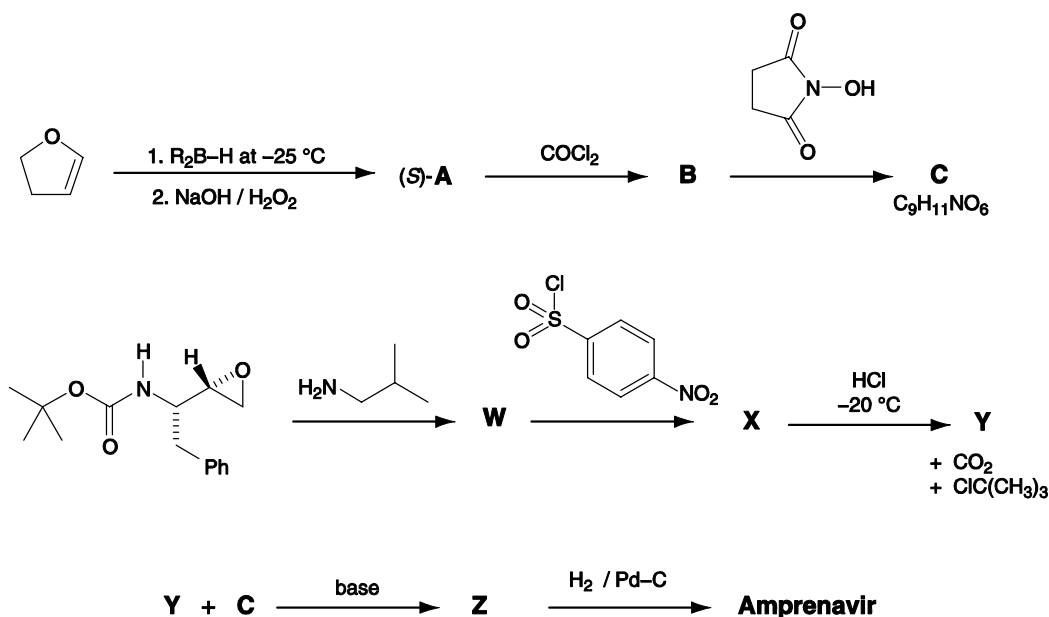
Synthesis of Amprenavir

4a A	4a B	4a C	4a W	4a X	4a Y	4a Z	4b	Total
4	3	2	3	3	2	3	3	23

One class of anti-HIV drugs, known as *protease inhibitors*, works by blocking the active site of one of the enzymes used in assembly of the viruses within the host cell. Two successful drugs, *saquinavir* and *amprenavir*, contain the structural unit shown below which mimics the transition state within the enzyme. In the structure, R^1 , R^2 and R^3 may represent any atom or group other than hydrogen.



Amprenavir may be synthesised as shown in the convergent scheme below.



The reagent R_2B-H used in the first step is chiral. Product **A** is formed as the (S)-enantiomer.

3 of the signals in the 1H NMR spectrum of Amprenavir disappear on shaking with D_2O : δ 4.2 (2H), δ 4.9 (1H) and δ 5.1 (1H).



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Suggest structures for **a)** the intermediates **A, B, C, W, X, Y** and **b)** for *Amprenavir*. Your answers should clearly show the stereochemistry at each centre.

A

B

C

W



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X

Y

Z



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Amprenavir

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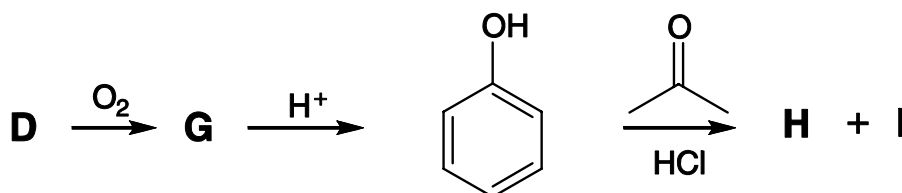
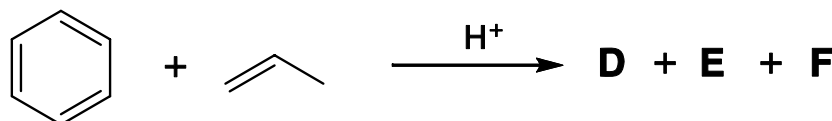
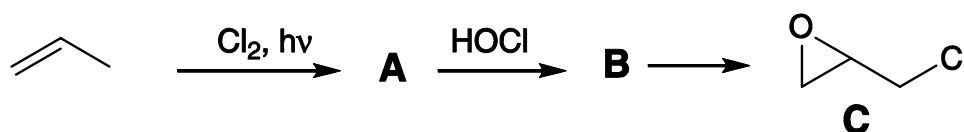
Problem 5

10% of the total

Epoxy resins

5a A	5a B	5b	5c D	5c E	5c F	5d G	5e H	5f	5g I	5h J	5h K	5h L	5i M	5j N	5k O	Total
2	2	1	2	2	2	3	3	1	2	2	2	2	2	4	3	35

The synthesis of epoxy resins is a multi-billion dollar industry worldwide. Epoxy resins are high performance adhesives synthesised from the reaction of a bis-epoxide with a diamine. The bis-epoxide is made from **H** and epichlorohydrin, **C**. **C** and **H** can be synthesised according to the schemes below.



The synthesis of epichlorohydrin **C** begins with the reaction of propene with chlorine in the presence of light.

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a) Draw the structures of **A** and **B**:

A

B

b) Give the formula of a suitable reagent for the conversion of **B** into epichlorohydrin **C**:

The synthesis of **H** commences with the reaction of benzene with propene in the presence of an acid catalyst which gives **D** as the major product and **E** and **F** as minor products.

c) Draw the structures of **D**, **E**, and **F** from the following data:

D: Elemental composition: C 89.94%, H 10.06%; 6 signals in the ^{13}C NMR spectrum

E: Elemental composition: C 88.82%, H 11.18%; 4 signals in the ^{13}C NMR spectrum

F: Elemental composition: C 88.82%, H 11.18%; 5 signals in the ^{13}C NMR spectrum

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D

E

F

Bubbling oxygen through a hot solution of **D** gives **G** which on exposure to acid gives phenol (hydroxybenzene) and acetone (propanone).

G turns starch iodide paper from white to dark blue. **G** has 6 signals in the ^{13}C NMR spectrum and the following signals in the ^1H NMR spectrum: δ 7.78 (1H, s), 7.45-7.22 (5H, m), 1.56 (6H, s); addition of D_2O results in the disappearance of the signal at $\delta = 7.78$.

d) Draw the structure of **G**.

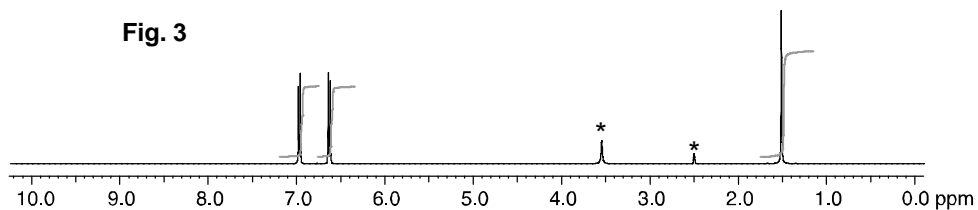
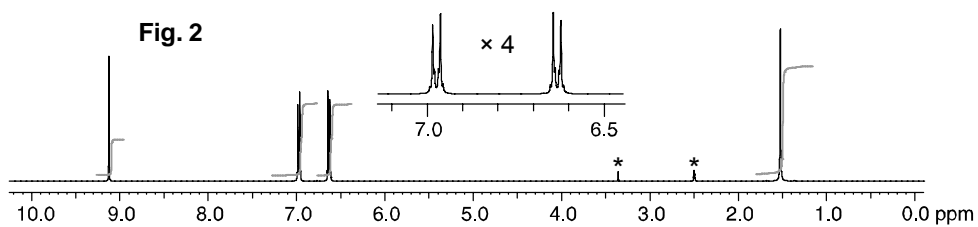
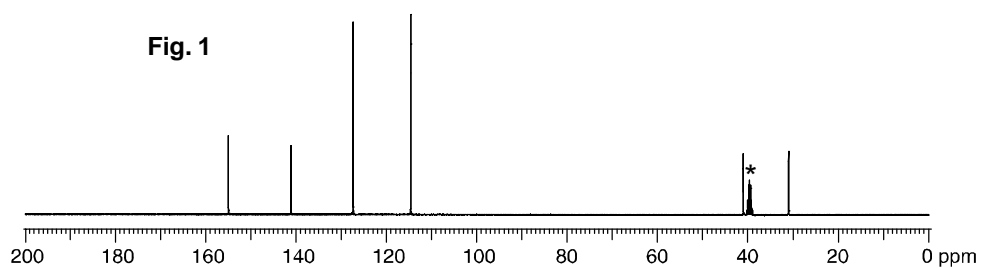
G

Exposure of phenol and acetone to hydrochloric acid gives compound **H**. The ^{13}C NMR spectrum for **H** is shown in Fig. 1. The ^1H NMR spectrum is shown in Fig. 2 together with a four-fold expansion of the region 6.5 – 7.1 ppm. The ^1H NMR spectrum after the addition of a drop of D_2O , is shown in Fig. 3. Peaks due to the solvent are marked with an asterisk (*).



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e) Draw the structure of H.

H



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f) Draw one resonance structure of phenol which explains the regioselective formation of **H**.

A second compound, **I**, is also formed in the reaction of phenol with acetone. The ^{13}C NMR spectrum of **I** has 12 signals. The ^1H NMR spectrum has the following signals: δ 7.50-6.51 (8H, m), 5.19 (1H, s), 4.45 (1H, s), 1.67 (6H, s); addition of D_2O results in the disappearance of the signals at $\delta = 5.19$ and 4.45

g) Draw a structure for **I**.

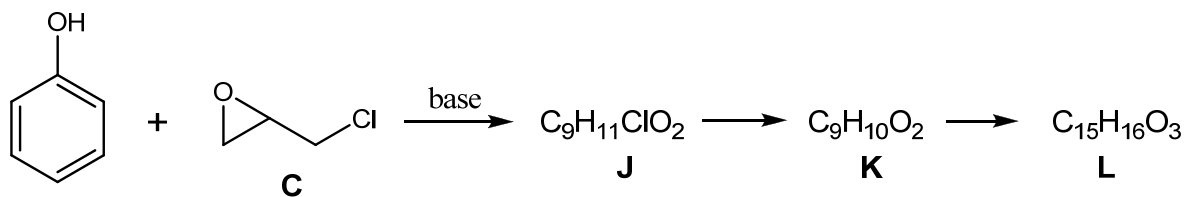
I

Excess phenol reacts with epichlorohydrin **C** in the presence of base to give compound **L** which has 6 signals in its ^{13}C NMR spectrum. If the reaction is stopped before completion compounds **J** and **K** can also be isolated. Compound **L** is formed from compound **K** and compound **K** is formed from compound **J**.



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h) Draw the structures of **J**, **K** and **L**.

J

K

L

Treatment of **H** with a large excess of epichlorohydrin **C** and base gives a monomeric bis-epoxide **M**. **M** contains no chlorine atoms or OH groups.



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- i) Draw the structure of **M**.

M

Treatment of **H** with a small excess of epichlorohydrin and base gives **N**. **N** has the form: **endgroup1-[repeat unit]_n-endgroup2** where *n* is approximately 10 – 15. **N** does not contain chlorine atoms and contains one OH group per repeat unit.

- j) Draw the structure of **N** in the form indicated above (**endgroup1-[repeat unit]_n-endgroup2**).

N

- k) Draw the repeat unit of the polymeric epoxy resin **O** formed from the reaction of the bis-epoxide **M** with ethane-1,2-diamine.

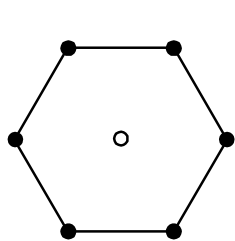
Problem 6

12% of the total

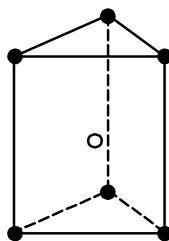
Transition metal complexes

6a	6b	6c	6d	6e	6f	6g	6h	6i	6j	6k	6l	Total
18	5	4	6	5	2	3	2	4	4	2	6	61

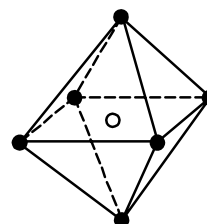
Alfred Werner used the technique of 'isomer counting' to deduce the structure of metal complexes with coordination number six. Three of the shapes he considered are shown below.



X



Y



Z

In each structure, the empty circle shows the location of the central metal atom and the filled circles show the location of the ligands. Structure X is hexagonal planar, structure Y is trigonal prismatic and structure Z is octahedral.

For each of the three shapes, there is just one structure when all of the ligands are the same, i.e. when the complex has the general formula MA_6 where A is the ligand. However, when achiral ligands A are substituted by one or more achiral ligands, it may be possible for each structure to form geometrical isomers. It might also be possible for one or more of the geometrical isomers to be optically active and exist as pairs of enantiomers.

- a) Fill in the table below to indicate how many geometrical isomers may be formed for each structure X, Y, and Z as the monodentate ligands A are substituted by monodentate ligands B or by symmetrical bidentate ligands, denoted C—C. Bidentate ligand C—C can only link between two atoms on adjacent positions, i.e. those positions connected by a line in the structures X, Y, and Z.

In each case write the number of geometrical isomers in the space provided. If one of the isomers exists as a pair of enantiomers, include an asterisk, *, in



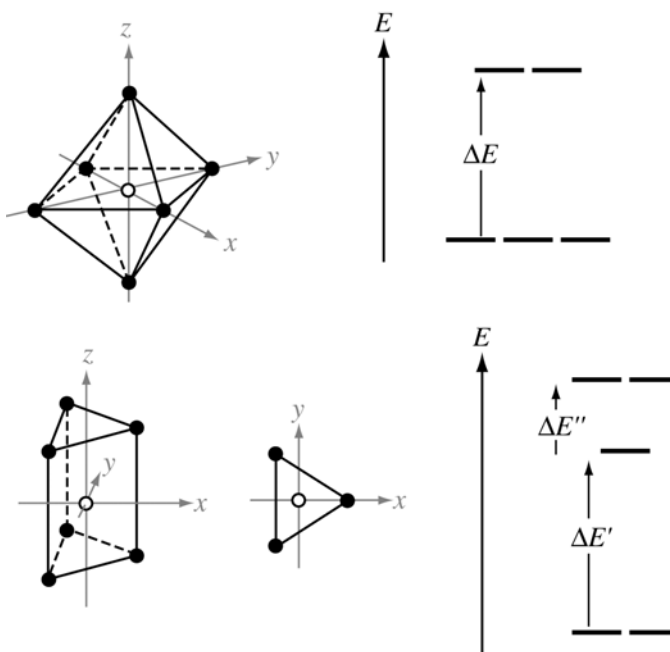
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the box. If two exist as two pairs of enantiomers, include two asterisks and so on. For example, if you think there are five geometrical isomers of a particular structure, three of which exist as pairs of enantiomers, write 5***.

	Number of predicted geometrical isomers		
	Hexagonal planar X	Trigonal Prismatic Y	Octahedral Z
MA_6	1	1	1
MA_5B			
MA_4B_2			
MA_3B_3			
$MA_4(C-C)$			
$MA_2(C-C)_2$			
$M(C-C)_3$			

There are no known complexes that adopt the hexagonal planar geometry **X**, but structures are known for both the trigonal prismatic geometry **Y** and the octahedral geometry **Z**. In these complexes, the orbitals derived from the metal d orbitals have different energies depending on the geometry of the complex. The splitting patterns for the trigonal prismatic geometry and for the octahedral geometry are shown below.



The separations in energy, ΔE , $\Delta E'$ and $\Delta E''$ depend on the particular complex.



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- b) For each of the splitting patterns shown below label which d orbitals are which.



The two complexes $[\text{Mn}(\text{H}_2\text{O})_6]^{2+}$ and $[\text{Mn}(\text{CN})_6]^{2-}$ are both octahedral. One has a magnetic moment of 5.9 BM, the other has a magnetic moment of 3.8 BM but you must decide which is which.

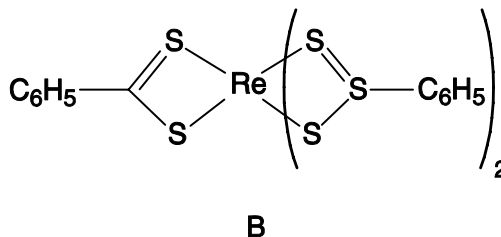
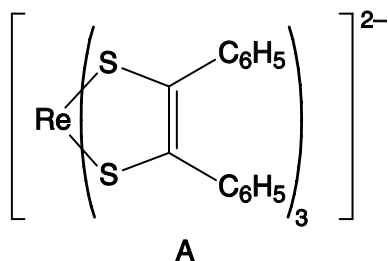
- c) On the diagram below, draw the electronic arrangements for each of the complexes.



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The magnetic moments of complexes **A** and **B** shown below have been measured and found to be 1.9 and 2.7 BM but you must decide which is which.



- d) Draw the orbital splitting diagrams for the two complexes, including the arrangements of the electrons.

A

B

Octahedral complexes are far more common than trigonal prismatic. Werner isolated five compounds **C – G** containing Co(III), Cl, and NH₃ only, each of which contained one octahedral complex. (There is actually a sixth compound but Werner could not isolate it). Werner's five compounds had the molar conductivities shown below. The conductivities are extrapolated to infinite dilution and are expressed in arbitrary units. Compound **G** does not react with aqueous AgNO₃; compounds **C**, **D**, and **E** react with different stoichiometric ratios of aqueous AgNO₃; **E** and **F** react with the same stoichiometric ratio of aqueous AgNO₃.

	C	D	E	F	G
molar conductivity	510	372	249	249	~0



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e) As far as you are able, suggest a structure for each of the compounds **C – G**.

C

D

E

F

G

Werner was also the first person to separate the enantiomers of an octahedral compound, **H**, which contained no carbon atoms. The compound, **H**, is composed of only cobalt, ammonia, chloride and an oxygen species which could be either H_2O , or HO^- or O^{2-} . The compound contains octahedrally coordinated cobalt ions. All of the chloride is easily removed from the compound by titration with aqueous silver nitrate. A 0.2872 g sample of **H** (containing no water of crystallization) required 22.8 cm^3 of 0.100 M silver nitrate to exchange all of the chloride.



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- f) Calculate the percentage, by mass, of chloride in **H**.

H is stable to acids, but is hydrolysed in alkali. A 0.7934 g sample of **H** (containing no water of crystallization) was heated with excess aqueous sodium hydroxide. Cobalt(III) oxide was formed and ammonia gas given off. The ammonia produced was distilled off and absorbed into 50.0 cm³ of 0.500 M aqueous HCl. The residual HCl required 24.8 cm³ of 0.500 M aqueous KOH to be neutralized.

The remaining suspension of cobalt(III) oxide was allowed to cool, approximately 1g of potassium iodide was added, and then the mixture was acidified with aqueous HCl. The liberated iodine was then titrated with 0.200 M aqueous sodium thiosulfate and required 21.0 cm³ for complete reaction.

- g) Calculate the percentage, by mass, of ammonia in **H**.



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- h)** Give the equation for the reaction of cobalt(III) oxide with potassium iodide in aqueous acid.
- i)** Calculate the percentage, by mass, of cobalt in **H**.
- j)** Calculate the identity of the oxygen species contained in **H**. Show your working.



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k) Give the empirical formula of **H**.

l) Suggest a structure for the chiral compound **H**.



Exam Procedure

- **The time allowed** for the exam is **5 hours**. There will be an additional **15 minutes reading time** at the start. **DO NOT** begin practical work until the **START** command is given.
- **When the announcement is given to stop working at the end of the 5 hours you must do so immediately. A delay in doing this may lead to your disqualification from the examination.**
- After the signal to stop working has been given, **wait in your lab space**. A supervisor will come to you and check off the following items as being **left behind**:
 - These 'Practical Exam' instructions.
 - All your answer sheets *plus* the sheet of graph paper from Task 3 in the envelope labelled with your student code. Do not seal the envelope.
 - Your chosen TLC plate in the Ziploc bag labelled with your student code.
 - The sample labelled 'RPA' from Task 1.
- **Do not leave** the examination hall until instructed to do so by the supervisors.



General Information

- **Safety** is of paramount importance in the laboratory. You should be following the safety rules given in the IChO regulations. **Safety glasses** and **lab coats** must be worn at **ALL TIMES**. **Gloves** should be worn during Task 1.
- If you behave in an unsafe way, you will be warned **once** before you are asked to leave the laboratory. You will not be allowed to return and will receive a score of zero for the entire experimental examination.
- The **problem booklet** is made up of 14 pages, within which there are **3 tasks**. You may do the tasks in any order you choose.
- The **answer booklet** is made up of 11 pages. You should write **your name and student code onto every answer sheet**. Do not attempt to separate the sheets.
- Your answers and working must **only be written in the spaces provided for them**. Anything written elsewhere will not be marked. Any calculations must be shown (calculators may be used). If you need to do **rough working**, use the back of the sheets.
- Numerical answers are meaningless without the appropriate units. You will be heavily penalised if units are not given where required.
- Use only the pens, pencil, rubber, ruler and calculator provided.
- If you make an error or break something and require **extra equipment, or chemicals**, ask a supervisor. Whatever you have requested will be provided, but after the first request **there will be a penalty of 1 of the 40 available practical points for each subsequent request**. Additional sheets of graph paper will be provided on request with no penalty.
- If you have any **questions** regarding the tasks or require a **refreshment/toilet break**, ask a member of staff.
- If you need to re-use glassware during the exam, clean it carefully at the closest sink to you.
- Solutions may be disposed of into the sink, **except EDTA and those containing copper or silver**. Please leave these on your bench or dispose of into the containers provided.
- The official English version of this examination is available on request if clarification is required.



Periodic table with relative atomic masses

1																	18
1 H 1.008																	2 He 4.003
3 Li 6.94	4 Be 9.01											5 B 10.81	6 C 12.01	7 N 14.01	8 O 16.00	9 F 19.00	10 Ne 20.18
11 Na 22.99	12 Mg 24.31	3	4	5	6	7	8	9	10	11	12	13 Al 26.98	14 Si 28.09	15 P 30.97	16 S 32.06	17 Cl 35.45	18 Ar 39.95
19 K 39.102	20 Ca 40.08	21 Sc 44.96	22 Ti 47.90	23 V 50.94	24 Cr 52.00	25 Mn 54.94	26 Fe 55.85	27 Co 58.93	28 Ni 58.71	29 Cu 63.55	30 Zn 65.37	31 Ga 69.72	32 Ge 72.59	33 As 74.92	34 Se 78.96	35 Br 79.904	36 Kr 83.80
37 Rb 85.47	38 Sr 87.62	39 Y 88.91	40 Zr 91.22	41 Nb 92.91	42 Mo 95.94	43 Tc	44 Ru 101.07	45 Rh 102.91	46 Pd 106.4	47 Ag 107.87	48 Cd 112.40	49 In 114.82	50 Sn 118.69	51 Sb 121.75	52 Te 127.60	53 I 126.90	54 Xe 131.30
55 Cs 132.91	56 Ba 137.34	57 La*	72 Hf 178.49	73 Ta 180.95	74 W 183.85	75 Re 186.2	76 Os 190.2	77 Ir 192.2	78 Pt 195.09	79 Au 196.97	80 Hg 200.59	81 Tl 204.37	82 Pb 207.2	83 Bi 208.98	84 Po	85 At	86 Rn
87 Fr	88 Ra	89 Ac ⁺															

*Lanthanides	58 Ce 140.12	59 Pr 140.91	60 Nd 144.24	61 Pm	62 Sm 150.4	63 Eu 151.96	64 Gd 157.25	65 Tb 158.93	66 Dy 162.50	67 Ho 164.93	68 Er 167.26	69 Tm 168.93	70 Yb 173.04	71 Lu 174.97
⁺ Actinides	90 Th 232.01	91 Pa	92 U 238.03	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	103 Lr



Apparatus per Student

Apparatus	Number
Task 1:	
Glass beaker (25 cm ³)	1
Large metal spatula	1
Small metal spatula	1
Flat ended glass rod	1
Suction pump	1
Buchner flask (250 cm ³)	1
Rubber ring seal for Buchner flask	1
Hirsch funnel	1
Vial for crude product A, labelled 'CPA'	1
TLC jar and lid with filter paper inside	1
TLC plates (in Ziploc bag labelled with student code)	3
TLC spotters	6
Conical flask (100 cm ³)	3
Magnetic stirring bar	1
Stirrer hotplate	1
Glass funnel (75 mm)	1
Spring test tube holder	1
Buchner funnel	1
Polystyrene tray (ice bath)	1
Vial for recrystallised product A, labelled with student code and 'RPA'.	1
Ziploc bag containing:	1
• pH paper and chart	1
• Filter paper for Hirsch funnel	2
• Filter paper for hot filtration	2
• Filter paper for Buchner funnel	2
Task 2:	
Burette (50 cm ³)	1
Measuring cylinder (25 cm ³)	1
Conical flask (250 cm ³)	4
Plastic funnel (40 mm)	1



Task 3:	
Tall plastic vessel	1
Conductivity meter	1
Rubber Pipette bulb (50 cm ³)	1
Pipette (50 cm ³)	1
Volumetric flask (250 cm ³)	1
Burette (50 cm ³)	1
Plastic funnel (40 mm)	1
Sheet of graph paper with labelled axes	1
For use in more than one task:	
Pencil	1
Marker pen	1
Envelope labelled with student code	1
Wash bottle containing distilled water (500 cm ³)	1
Bosshead	4
Clamps	4
Retort stand and rod (Zoology only)	3
Measuring cylinder (10 cm ³)	1
Tissue paper for cleaning	
Disposable plastic pipette (3 cm ³)	8
Shared equipment:	
UV lamp	
Balance (to 3 decimal places)	
Labelled waste containers for EDTA, copper, and silver waste	
Purple nitrile gloves in all sizes	



Chemicals on Each Desk

Chemical	R phrases	S phrases
Task 1:		
3,4-dimethoxybenzaldehyde: 0.50 g pre-weighed in vial labelled 'DMBA 0.5 g'.	22-36/37/38	22-24/25
1-indanone: 0.40 g pre-weighed in vial.	22	–
NaOH: 0.10 g pre-weighed in vial.	34-35	26-36-37/39-45
HCl (3.0M aqueous): 10 cm ³ in a 30 cm ³ bottle.	34-37	24-26-36-45
Diethyl ether:Heptane (1:1): 25 cm ³ in a 30 cm ³ bottle labelled 'Et ₂ O:Heptane (1:1)'.	Diethyl ether: 12-19-22-66-67; Heptane: 11-38-50/53-65-67	Diethyl ether: 9-16-29-33; Heptane: 9-16-23-29-33-60-61-62
Ethyl ethanoate: 1 cm ³ in a small vial.	11-36-66-67	16-26-33
Sample of 1-indanone dissolved in ethyl ethanoate: 1.0 cm ³ in small vial labelled '1-indanone in ethyl ethanoate'.	See above	See above
Sample of 3,4-dimethoxybenzaldehyde dissolved in ethyl ethanoate: 1.0 cm ³ in small vial labelled 'DMBA in ethyl ethanoate'.	See above	See above
Ethyl alcohol (9:1 mixture with H ₂ O): 100 cm ³ in a 125 cm ³ bottle labelled 'EtOH:H ₂ O (9:1)'.	11	7-16
Task 2:		
Inorganic complex: three samples of approximately 0.1 g, accurately pre-weighed in vials labelled 'Sample 1', 'Sample 2', 'Sample 3'.	22-25-36/37/38	26-28-37/39-45
Inorganic complex: three samples of approximately 0.2 g, accurately pre-weighed in vials labelled 'Sample 4', 'Sample 5' and 'Sample 6'.	22-25-36/37/38	26-28-37/39-45
pH 10 ammonia buffer: 10 cm ³ in a 30 cm ³ clear glass bottle labelled 'pH 10 ammonium buffer'.	20/21/22-36/37/38	26-36
Murexide indicator (solution in H ₂ O): 10 cm ³ in a 30 cm ³ clear glass bottle.	–	24/25
EDTA disodium salt (0.0200 M solution in H ₂ O): 150 cm ³ in a 250 cm ³ clear glass bottle.	22	36
Ethanoic acid: 10 cm ³ in a 30 cm ³ clear glass bottle.	10-35	23-26-45
2,7-Dichlorofluorescein indicator (solution in 7:3 EtOH:H ₂ O): 10 cm ³ in 30 cm ³ clear glass bottle.	36/37/38	26-36-37/39
Dextrin (2% in H ₂ O): 25 cm ³ in a 30 cm ³ bottle.	–	24/25
Silver nitrate (0.1000M solution in H ₂ O): 150 cm ³ in a 250 cm ³ brown glass bottle.	8-34-50/53	26-36-45-60-61
Task 3:		
Sodium dodecyl sulfate (99%): approximately 4.3 g, accurately pre-weighed in vial labelled 'SDS'.	22-36/37/38	26-36/37
Conductivity solution 'HI 70031': 20 cm ³ in pouch.	Non hazardous product	Non hazardous product



Risk Phrases

Indication of Particular Risks

R Number	Meaning
8	Contact with combustible material may cause fire.
10	Flammable.
11	Highly flammable.
12	Extremely flammable.
19	May form explosive peroxides.
22	Harmful if swallowed.
25	Toxic if swallowed.
34	Causes burns.
35	Causes severe burns.
36	Irritating to eyes.
37	Irritating to the respiratory system.
38	Irritating to skin.
65	Harmful: may cause lung damage if swallowed.
66	Repeated exposure may cause skin dryness or cracking.
67	Vapours may cause drowsiness and dizziness.

Combination of Particular Risks

R Numbers	Meaning
20/21/22	Harmful by inhalation, in contact with skin and if swallowed.
36/37/38	Irritating to eyes, respiratory system and skin.
50/53	Very toxic to aquatic organisms; may cause long term effects in the aquatic environment



Safety Phrases

Indication of Safety Precautions Required

S Number	Meaning
7	Keep container tightly closed.
9	Keep container in a well ventilated place.
16	Keep away from sources of ignition. No smoking.
22	Do not inhale dust.
23	Do not inhale gas/fumes/vapour/spray.
24	Avoid contact with the skin.
26	In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
28	After contact with skin, wash with plenty of water.
29	Do not empty into drains.
33	Take precautionary measurements against static discharges.
36	Wear suitable protective clothing.
45	In case of accident or if you feel unwell, seek medical advice immediately (show label where possible).
60	This material and/or its container must be disposed of as hazardous waste.
61	Avoid release to the environment.
62	If swallowed, do not induce vomiting: seek medical advice immediately and show this container or label.

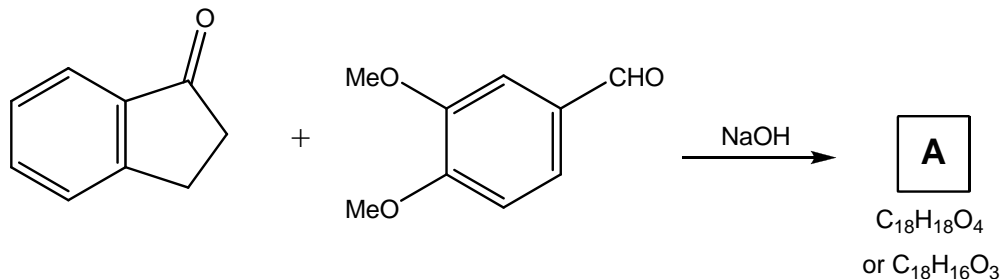
Combination of Safety Precautions Required

S Numbers	Meaning
24/25	Avoid contact with skin and eyes.
36/37	Wear suitable protective clothing and gloves.
36/37/39	Wear suitable protective clothing, gloves and eye/face protection.
37/39	Wear suitable gloves and eye/face protection.



Task 1 – An Environmentally Friendly Aldol Condensation

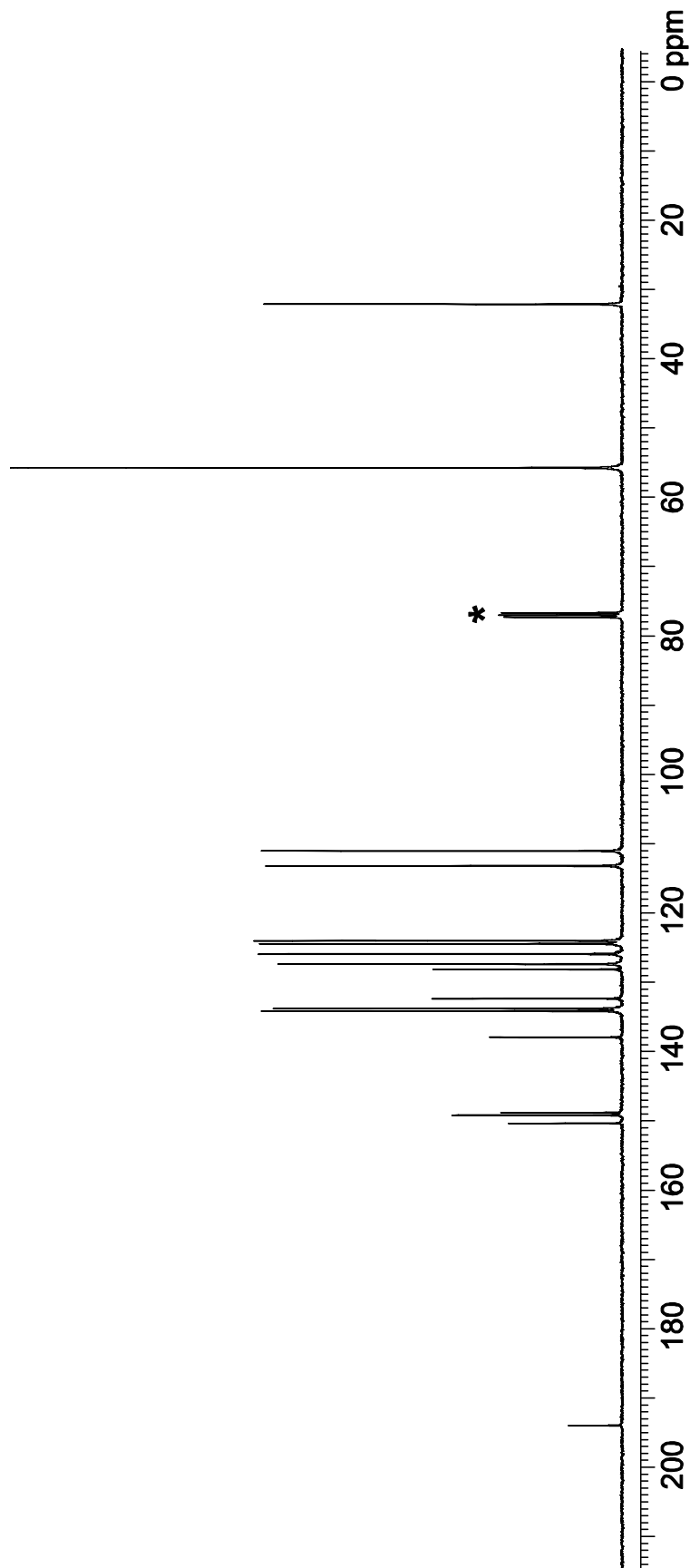
In attempts to become more environmentally friendly, increasing attention is being paid to minimising the large amounts of solvents used in chemical reactions. In the following experiment, an aldol condensation reaction is carried out in the absence of solvent.



1. Add 3,4-dimethoxybenzaldehyde (DMBA 0.50 g, 3.0 mmol) and 1-indanone (0.40 g, 3.0 mmol) to a 25 cm³ beaker. Use a metal spatula to scrape and crush the two solids together until they become a clear oil.
 2. Add NaOH (0.1 g, 2.5 mmol) to the reaction mixture, crush any lumps formed and continue scraping and crushing until the mixture becomes solid.
 3. Allow the mixture to stand for 20 minutes. Then add 4 cm³ HCl (3 M aqueous) and scrape around the beaker so as to dislodge all product from the walls. Use a flat-ended glass rod to crush any lumps present.
- a) Measure and record the pH of the solution.
4. Isolate the crude product using vacuum filtration through a Hirsch funnel. Rinse out the beaker with 2 cm³ HCl (3 M aqueous) and pour over the crude product in Hirsch funnel to wash, continuing to pull air through the solid for 10 minutes to facilitate drying.
- b) Report the mass of the crude product (which may still be a little wet), using the vial labeled 'CPA' as a container.



5. Take a TLC to assess whether the reaction is complete, using Et₂O:heptane (1:1) as the eluant. Solutions of both starting materials in ethyl ethanoate are provided. The crude product is soluble in ethyl ethanoate. [Note: three TLC plates are provided. You may use them all, but you must only submit *one* in your labelled Ziploc bag. This should be the plate that you draw in your answer booklet.]
- c) Using UV light to visualize, draw around the spots on the plate in pencil to show where they are, copy your plate onto the answer sheet, and place your plate in the Ziploc bag labeled with your student code. Determine and record the relevant R_F values.
6. Using a 100 cm³ conical flask with a stir bar in the bottom, recrystallise the product from 9:1 EtOH:H₂O (N.B. A hot filtration, using the glass funnel provided, is required as part of this process to remove small amounts of insoluble impurities). Any lumps may be crushed using the flat-ended glass rod. Allow the conical flask containing the filtered solution to cool to room temperature and then cool in an ice bath (use the polystyrene tray to make the ice bath in) for one hour before filtration through a Buchner funnel to collect your product. Suck air through for 10 minutes to dry the product. Place your product in the vial marked with your code and labeled 'RPA'.
- d) Report the mass of the purified product.
- e) Determine the potential structures for Product **A**, using the information on the answer sheet.
- f) The ¹³C NMR spectrum for **A** is shown on the next page. Peaks due to the solvent, CDCl₃, are marked with an asterisk. With the aid of the spectrum, decide which is the correct formula for **A**. Mark your answer on the answer sheet.
- g) Calculate the percentage yield of the purified product, based on the formula you gave for its structure.





Task 2 – Analysis of a Copper(II) Complex

You are provided with a sample of an inorganic copper(II) complex, the anion of which is made from copper, chlorine, and oxygen. The counter ion is the tetramethyl ammonium cation. There is no water of crystallisation. You are required to determine proportions of copper ions and chloride ions by titration and hence determine the composition of the complex

Titration to determine the proportion of copper ions

1. You are provided with three accurately pre-weighed samples of copper complex, each of approximately 0.1 g. These are labeled "Sample 1", "Sample 2", "Sample 3", together with the exact mass of the copper complex. Take the first of these, note down the mass of the sample and quantitatively transfer the contents to a 250 cm³ conical flask using approximately 25 cm³ of water.
2. Add pH 10 ammonia buffer solution until the precipitate which forms initially just redissolves (about 10 drops).
3. Add 10 drops of the murexide indicator.
4. Titrate with the 0.0200 mol dm⁻³ EDTA solution until the solution turns violet and the colour persists for at least 15 seconds. Record the volume of solution used in the titration.
5. Repeat if necessary with samples 2 and 3.

Note: you will be marked only on a single value you report in the answer booklet. This may either be an average value, or a single value you feel most confident in.

- a) Calculate the volume of EDTA solution needed to react completely with 0.100 g of complex.
- b) Give an equation for the titration reaction.
- c) Calculate the percentage by mass of copper in the sample.

You will need to wash out your burette before you start the titration for the determination of chloride ions. Any remaining EDTA solution may be disposed of into the waste containers labelled 'EDTA'.



Titration to determine the proportion of chloride ions present

1. You are provided with three accurately pre-weighed samples of copper complex each of approximately 0.2 g. These are labeled "Sample 4", "Sample 5", "Sample 6", together with the exact mass of the copper complex. Take the first of these, note down the mass of the sample and quantitatively transfer the contents to a 250 cm³ conical flask using approximately 25 cm³ of water.
2. Add 5 drops of ethanoic acid, followed by 10 drops of dichlorofluorescein indicator and 5 cm³ dextrin (2% suspension in water). N.B. Shake the bottle well before adding the dextrin suspension.
3. Titrate with the 0.1000 mol dm⁻³ silver nitrate solution, swirling constantly until the white suspension turns pink and the colour does not disappear after swirling.
4. Repeat if necessary.

Note: you will be marked only on a single value you report in the answer booklet. This may either be an average value, or the value you feel most confident in.

- d) Calculate the volume of silver nitrate solution needed to react completely with 0.200 g of complex.
- e) Give an equation for the titration reaction.
- f) Calculate the percentage by mass of chloride ions in the sample.

The percentage of carbon, hydrogen and nitrogen in the complex was determined by combustion analysis and found to be as follows:

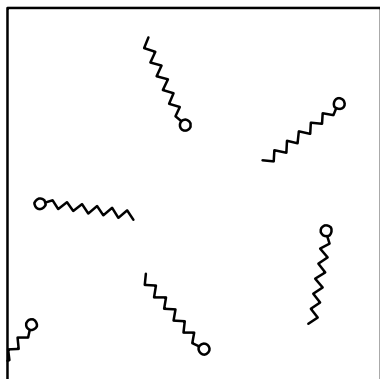
Carbon: 20.87 %	Hydrogen: 5.17 %	Nitrogen: 5.96 %
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- g) Mark in the answer booklet, which element in the complex has the greatest percentage error in the determination of its proportion.
- h) Determine the formula of the copper complex. Show your working.

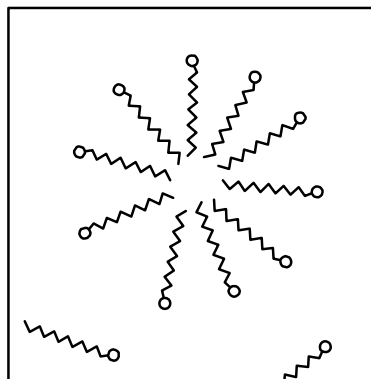
Task 3 – The Critical Micelle Concentration of a Surfactant

Surfactants are used extensively in many everyday cleaning products, such as shampoos or detergents for washing clothes. One such surfactant is SDS, sodium *n*-dodecyl sulfate, $\text{CH}_3(\text{CH}_2)_{11}\text{OSO}_3\text{Na}$ (Relative Molecular Mass: 288.37).

Very dilute aqueous solutions consist of solvated individual molecules of SDS. However, if the concentration is gradually increased beyond a specific concentration, the concentration of monomeric SDS does not change, but instead the surfactant begins to form clusters known as *micelles*. It is these micelles that assist in the removal of grease and dirt. The concentration at which the micelles form is called the *critical micelle concentration*. This process is shown schematically in the figure below.



low SDS concentration
free monomer only



high SDS concentration
micelles and some free monomer

In this experiment, you will determine the critical micelle concentration of SDS by measuring the conductivity of different concentrations of SDS.

1. You are provided with approximately 4.3 g SDS, accurately pre-weighed in a vial, a 250 cm³ volumetric flask, a 50 cm³ burette, 50 cm³ bulb pipette, a conductivity meter, conductivity solution (used only for calibration), and a tall plastic vessel.
2. You need to measure the conductivity (σ , in $\mu\text{S cm}^{-1}$) of various concentrations of aqueous SDS (c , up to 30 mmol dm⁻³). [Note: you may assume all volumes are additive.]



- a) Give the concentration of your stock SDS solution.
- b) Use the table given in the answer booklet to record your results and plot a suitable graph to determine the critical micelle concentration (CMC) on the paper provided.
- c) State the concentration at which micelles begin to form (the critical micelle concentration).

Notes

- 1) Solutions of SDS readily form bubbles if shaken.
- 2) The conductivity meter needs at least 50 cm³ of solution to be inside the plastic vessel in order to work correctly.
- 3) To calibrate the meter:
 - Switch the meter on by pressing the ON/OFF button once.
 - Press and hold the ON/OFF button again, this time for about 3 seconds, until you see the letters 'CAL' on the screen, indicating that the calibration mode has been entered. Let go of the ON/OFF button and '1413' will start blinking on the display. To calibrate, carry out the next step immediately, before the meter has reverted back to reading '0' on the screen (meaning you have exited the calibration mode)
 - Immerse the probe in the pouch containing the 'HI 70031' calibration solution, without exceeding the maximum immersion level.
 - Stir gently and wait for about 20 seconds to confirm the reading.
 - Once the display stops blinking, the meter is calibrated and ready for use.
 - Rinse the meter with distilled water and dry before making measurements.
- 4) To record the reading:
 - Switch the meter on by pressing the ON/OFF button
 - Immerse the probe in the sample without exceeding the maximum immersion level and being above the minimum immersion level.
 - Stir gently and wait for the reading to stabilize. The meter automatically compensates for temperature variations.
 - The conductivity value of the sample will be shown on the LCD.