



NTNU – Trondheim
Norwegian University of
Science and Technology

Department of Chemistry

Examination paper for KJ2073, Analytical Environmental Chemistry

Academic contact during examination: Øyvind Mikkelsen

Phone: 92899450

Examination date: 11.06.2016

Examination time (from-to): 09:00 – 13:00

Permitted examination support material: (D) pocket calculator

Other information: All questions/tasks should be answered, however in task 2 select one of the two eligible EITHER/OR alternatives.

Language: English

Number of pages (front page and enclosed pages included): 5

Number of pages enclosed: 2 (Annex 1 and Annex 2)

Checked by:

Date

Signature

Task 1. (3p + 3p + 2p + 2p = 10p total)

- a) Given that we have a hydrodynamic system and a redox active compound present, explain why the resulting voltammogram (current plotted against the applied potential) in linear scan voltammetry get a characteristic waveform. Explain what is meant by limiting current is in linear scan voltammetry.
- b) Explain how we through the methods differential pulse voltammetry and square wave voltammetry respectively are able to achieve a removal of the charging current.
- c) Describe briefly (one to two sentences) the main difference between a voltammetric and a potentiometric system.
- d) Make a sketch of an ion selective electrode for calcium.

Task 2. (4p + 4p + 2p = 10p total)

EITHER

- a) Describe the theoretical principle for a potentiometric pH sensor based on the glass electrode. Include a sketch showing the construction of the glass electrode and the potentiometric system.
- b) Describe sources of errors associated with the potentiometric glass electrode for pH determination, and describe how to perform a calibration.
- c) Suggest, in brief, an alternative analysis methodology / sensor for pH determination in water.

OR

- a) Describe the theoretical principle for an amperometric oxygen sensors (oxygen saturation in water) based on the Clark electrode. Include a diagram showing the construction of the Clark electrode and the amperometric system.
- b) Describe sources of errors associated with the amperometric Clark electrode for oxygen determination, and describe how to perform a calibration.
- c) Suggest, in brief, an alternative analysis methodology / sensor for determining the oxygen saturation in water.

Task 3. (9p + 6p + 6p + 3p + 3p + 3p = 30p total)

- a) Describe the physical-chemical principle for spectroscopy. In the answer, emphasize in particular the analytical use of Beers law and what's behind it. Sketch, with figures, how different components relative to each other are installed in the instruments for the following techniques; emission spectroscopy, absorption spectroscopy and fluorescence spectroscopy.
- b) Discuss different limitations in Beers law.
- c) Describe, in more detail, different wavelength selectors in spectroscopy, and specify which applications (area of use) they have.
- d) Select a radiation source for use in spectroscopy; explain how it is constructed and how it works. Specify which applications (area of use) it has.

e) Describe the difference between IR spectrometers with a dispersing unit and Fourier spectrometers (Fourier Transform Infrared Instruments - FTIR). Include also differences in the components that the instruments consist of.

f) For Inductively Coupled Plasma Mass Spectrometry (ICP-MS), explain what is meant by the resolving power (R). What is typical resolution for a standard quadrupole mass spectrometer used in the ICP - MS? How is it possible to attain an increase in the resolution?

Task 4. (5p + 2p + 3p = 10p total) – use Annex 1 for this task.

a) A classification of copper and zinc pollution in a river (dissolved phase) is to be performed. Describe how you will proceed to carry out sampling, what equipment you will use and how to preserve and store the samples. Recommend an instrumental method of analysis for the metals, and describe how you would secure quality assurance of analyzes.

b) Analyzes shows that the concentrations of copper and zinc in the possible contaminated area is 50 ppb and 15 ppb respectively. Use the classification system set out in Annex 1 to characterize the river water with respect to these two metals in the actual area.

c) The river ends in a fjord. It is of interest to investigate whether the sediments, nearby and at approximately 50 meters deep, can have been affected by the relevant metals. Explain how you will conduct sampling, and how to treat the sample prior to analysis by ICP -MS.

Task 5. (10p)

Indicate right or wrong on these statements (*Answer on attachment in Annex 2*, marked with your candidate number and page number, and handed inn along with the examination papers)

	Correct	Wrong
Water vapor and CO ₂ absorbs only the energy in the range of 109 J/mole and therefore has no importance in IR spectroscopy		
IR spectroscopy is a less suitable method for quantitative analysis compared with visible / UV spectroscopy because of lower sensitivity, and frequent deviations from Beers Law		
IR spectroscopy is a suitable technique for identifying pure organic and inorganic compounds, except homonuclear molecules such as O ₂ , N ₂ and Cl ₂ , because most molecules absorb energy in the IR range		
10 ³ - 10 ⁵ J/mole is the energy area corresponding to changes in configuration and used in IR analysis		
Saturated bonds often provide good absorption peaks in UV-VIS molecular spectroscopy		
Dissolved organic matter (DOC) in water can be determined IR spectroscopy.		
In ICP - MS the ions formed from the ICP discharge are typically negatively charged		
Detectors used in ICP-MS can be sensitive to light; therefore, they must be stored in the dark		
In polarography, electrode processes consuming hydrogen ions could produce significant change pH if buffer a solution is not used		
Cyclic voltammetry can be used for kinetic studies		

Annex 1, Classification system for pollution classes, metals in water ($\mu\text{g/L} = \text{ppb}$, $\text{mg/L} = \text{ppm}$)

Parameter:	Classes				
	I	II	III	IV	V
	"Very good"	"Good"	"Less good"	"Bad"	"Very bad"
TOC, mgC/l	<2,5	2,5 - 3,5	3,5 - 6,5	6,5 - 15	>15
pH	>6,5	6,0 - 6,5	5,5 - 6,0	5,0 - 5,5	<5,0
Oxygen saturation %	>80	50 - 80	30 - 50	15 - 30	<15
Iron, $\mu\text{g Fe/l}$	<50	50 - 100	100 - 300	300 - 600	>600
Manganese, $\mu\text{g Mn/l}$	<20	20 - 50	50 - 100	100 - 150	>150
Copper $\mu\text{g Cu/l}$	<0,6	0,6 - 1,5	1,5 - 3	3 - 6	>6
Zinc $\mu\text{g Zn/l}$	<5	5 - 20	20 - 50	50 - 100	>100
Cadmium $\mu\text{g Cd/l}$	<0,04	0,04 - 0,1	0,1 - 0,2	0,2 - 0,4	>0,4
Lead $\mu\text{g Pb/l}$	<0,5	0,5 - 1,2	1,2 - 2,5	2,5 - 5	>5
Nickel $\mu\text{g Ni/l}$	<0,5	0,5 - 2,5	2,5 - 5	5 - 10	>10
Chromium $\mu\text{g Cr/l}$	<0,2	0,2 - 2,5	2,5 - 10	10 - 50	>50
Mercury $\mu\text{g Hg/l}$	<0,002	0,002 - 0,005	0,005 - 0,01	0,01 - 0,02	>0,02

Annex 2, To be submitted along with the examination papers KJ2073 spring 2016

Candidate number: _____

Page number / total number of pages: ____/____

Select correct or wrong for the following statements

	Correct	Wrong
Water vapor and CO ₂ absorbs only the energy in the range of 109 J/mole and therefore has no importance in IR spectroscopy		
IR spectroscopy is a less suitable method for quantitative analysis compared with visible / UV spectroscopy because of lower sensitivity, and frequent deviations from Beers Law		
IR spectroscopy is a suitable technique for identifying pure organic and inorganic compounds, except homonuclear molecules such as O ₂ , N ₂ and Cl ₂ , because most molecules absorb energy in the IR range		
10 ³ - 10 ⁵ J/mole is the energy area corresponding to changes in configuration and used in IR analysis		
Saturated bonds often provide good absorption peaks in UV-VIS molecular spectroscopy		
Dissolved organic matter (DOC) in water can be determined IR spectroscopy.		
In ICP - MS the ions formed from the ICP discharge are typically negatively charged		
Detectors used in ICP-MS can be sensitive to light; therefore, they must be stored in the dark		
In polarography, electrode processes consuming hydrogen ions could produce significant change pH if buffer a solution is not used		
Cyclic voltammetry can be used for kinetic studies		