



MASINDE MULIRO UNIVERSITY OF SCIENCE AND TECHNOLOGY
(MMUST)

UNIVERSITY EXAMINATIONS 2022/2023 ACADEMIC YEAR

FIRST YEAR SEMESTER 2 MAIN EXAMINATIONS
FOR THE DEGREE
OF
MASTERS OF SCIENCE CHEMISTRY

COURSE CODE: SCH 821 TITLE: ADVANCED ANALYTICAL TECHNIQUES
II

DATE: 11th April 2023

TIME: 2 PM to 5 PM

INSTRUCTIONS TO CANDIDATES

1. Answer ALL questions
2. This is an open book exam

Sewage sludge is a by-product of wastewater treatment processes, and may be employed in agriculture as a fertilizer or in forestry for land reclamation. It is an important source of nutrients but its reuse can arouse concern on account of the wide range of contaminants that are retained and may persist during treatments. The Ministry of Environment in Kenya has communicated that information on the emerging contaminants (ECs) in sewage sludge in Kenya is limited. It has put up a call for the development of Analytical Methods to provide information on ECs from 12 waste water treatment plants spread across the country. It is desirable that the method performance be good, with recoveries higher than 70%, good repeatability (< 20%) and sensitivity in the low ng g⁻¹ range, allowing measurement of the analytes selected. This challenge required the following to be determined in sewerage sludge:

- (i) Over 42 pharmaceuticals belonging to 12 therapeutic categories ranging from antibiotics, anti-inflammatories estrogens, lipid regulators and, antihypertensives amongst others;
- (ii) Perfluorinated substances;
- (iii) Pesticides; and
- (iv) Nutrients applicable to improving agribusiness of low income farmers. Partial results as shown in the table below

CLASS	COMPOUND	CONCENTRATION (ng/l)
Sweeteners	Sucrose	1032
	Cyclamate	991
	Saccharin	1908
	Lauramidopropylbetaine	8924
	Nonylphenol isomers	379
	Lauryl diethanolamide	23836
	Dodecyl sulfate	11820
	Tetradecyl sulfate	11,3958
	Lauric isopropanol amide	572
	Decyl sulfate	15711
Rubber additives	2-(methylthio)benzathiole	738
	2-hydroxybenzothiazole	239
	Benzothiazole	1756
Plastic additives	Diethyl phthalate	23125
	Bis-(2-thyhexy) phosphate	11817
pharmaceuticals	Trimethoprim	2135
	Diclofenac	1695
	Sulfamethoxazole	4208
	10,11-dihydro-10,11-didyroxy Carbamazepine	1778
	Losartan	920

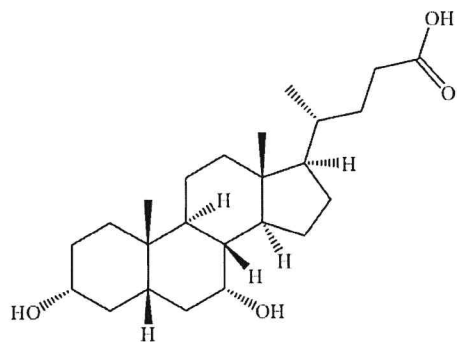
	Fluconazole	2273
	Metformin	19,499
	Ibuprofen	15,338
	Cetirizine	79189
	Dehydroabiatic acid	3499
	Acetyl-sulfamethoxazole	2161
Pesticides	Imidacloprid	557
	Carbaryl	687
	Dichlorvos	76
	Propoxur	168
	4-isopropyl aniline	49
	2,4-DB	518
Industrial	Bisphenol	1401
	2,4-dichlorophenol	772
	2-oxindole	38,305
	Methyl3(3,5-di-tert-butyl-4-hydroxy phenyl) propionate	1764
	Triethylene glycol monobutyl ether-M+ NH ₄	1013
	Bisphenol A	2313
	Bisphenol E	834
	3,5-di tert-butyl-4'-hydroxyacetophenone	859
Cyclohexylamine	1756	
Human metabolite	Chenodeoxycholic acid M+ NH ₄	8483
	Deoxycholic acid	35,6471
	Cholic acid	211,414
	7-oxolithocholic acid	72585
	Hexadecyl pyridinium	1268
	Propylparaben	584
	Diuron	211

QUESTION ONE

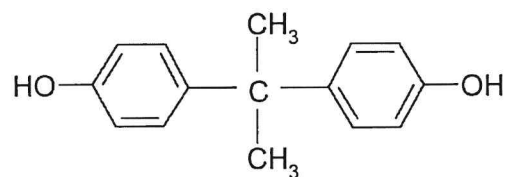
- (a) What challenges are associated with separation of these ECs in waste waters (5 marks)
- (b) Discuss modern trends in analytical separation of the above ECs (10 marks)

QUESTION TWO

- (a) Chenodeoxycholic acid M+ NH₄ is one of the human metabolites detected in waste water alongside Bisphenol A. Design a potentiometric method for selective determination of Bisphenol A from Chenodeoxycholic acid M+ NH₄ (10 marks)



Chenodeoxycholic acid $M^+ NH_4$



Bisphenol A

- (b) Discuss the type of seiderophores you will use to reduce the matrix effects from heavy metals present (5 marks)

QUESTION THREE

- (a) Choose one of the compounds in the table above and describe a mass spectroscopy-based hyphenated technique for characterizing it amidst other compounds (8 marks)
- (b) Describe a Surface Enhanced Raman Spectroscopy (SERS) sensor for detecting Dichlorvos in the matrix (7 marks)

QUESTION FOUR

Describe a pumice-biochar-based composite for removing these compounds from waste waters detailing

- (i) Method of pumice-biochar composite formulation (5 marks)
- (ii) Surface characterization techniques of the adsorption process (5 marks)
- (iii) Analytical methods providing proof of removal of contaminants (5 marks)