Class IV Wastewater Laboratory Analyst Examination Study Guide

Specific laboratory analyses included on Class IV Exam:

All from Class I, Class II, and Class III, in much greater depth.
Plus the following -

Biological Examination
Bioassay/Toxicity
Cyanide
Metals
Organics by Gas Chromatography (GC)
Total Phenols
Salinity
TOC (Total Organic Carbon)
TOX (Total Organic Halogen)

Also include the following subjects -

Activated Sludge
Biology
Sampling
General Laboratory Information and Procedures
Apparatus and Reagents
Chemistry
Mathematics
Safety
Quality Assurance / Quality Control (QA/QC)
Compliance Rules and Regulations
WWTP Operational Information
Laboratory Management and Laboratory Supervision

REFERENCES:

- 18th Edition of the Standard Methods for the Examination of Water and Wastewater
- 19th Edition of the Standard Methods for the Examination of Water and Wastewater
- 20th Edition of the Standard Methods for the Examination of Water and Wastewater
- U. S. EPA Approved Methods for Wastewater Analysis
- 40 CFR Part 136
- Water Environment Federation [WPCF] Manual of Practice No. 11
- Operation of Wastewater Treatment Plants, California State University, Sacramento [Sacramento Manual]

Microscopic Biological Examination

Be familiar with all information covered under both the Class I, II and Class III NTK.

Be able to categorize the condition of a given sludge from a description of the Microscopic Biological Examination results.

Know the family names of all of the activated sludge microorganisms.

Know the general life cycles for the main activated sludge microorganisms.

Bioassay/Toxicity

Be familiar with the different forms of the Bioassay test including Acute, and Chronic.

Be familiar with the different organisms used in Bioassay testing including when and where each would be used.

Be familiar with the sampling requirements, procedures and practices for Bioassay.

Be able to define the following terms;

1. Chronic Toxicity
2. Acute Toxicity
3. Acclimate
4. Response (with respect to toxicity)
5. Control
6. Definitive test
7. Range finding test
8. Screening test

Cyanide

Be familiar with the distillation preparatory step for the cyanide test. This includes all reagents and their purpose, as well as the procedures and apparatus used.

Be familiar with the different analytical methods for detecting cyanide including colorimetric, titrimetric, and selective ion electrode.

Be familiar with the difference between Total Cyanide and Cyanide Amenable to Chlorination, and how both of them are determined.

Be familiar with the chemistry of the analytical methods for detecting cyanide.

Be familiar with the sampling requirements for all cyanide analyses.

Be able to define the following terms;

1. Total Cyanide
2. Amenable Cyanide
3. complexed cyanide
4. free cyanide

Metals

Be familiar with the different types of Atomic Absorption (AA) methods, the instruments and apparatus associated with each including direct aspiration AA, cold vapor AA, hydride generated AA, and electrothermal graphite furnace AA.

Be familiar with Atomic Emission (AE) methods, the instruments and apparatus associated with it including when it is appropriate to use, and what elements can and cannot be analyzed by Atomic Emission.

Be familiar with the different preparatory methods for the analysis of metals by AA and AE.

Be familiar with the most common problems and interferences associated with running AA samples.

Be able to define the following terms;

1. Atomic Absorption Spectroscopy
2. Atomic Emission Spectroscopy
3. Acid digestion
4. Inductively Coupled Plasma (ICP)
5. Absorption line
6. Emission line
7. Hollow Cathode Lamp (HCL)
8. Dissolved metals content
9. Total Recoverable metals content

Organics by Gas Chromatography (GC)

Be familiar with the different parts of a GC and how they are used. This should include the reason(s) for each component, it's operating conditions and the reason for those conditions.

Be familiar with the different types of sample introduction to the GC, the preparation of the samples, and the purpose of each.

Be familiar with the different analytical methods and what they test for.

Gas Chromatograph (Separates mixtures of compounds in the gas phase using the solubility of each compound in a liquid phase or "packing", as well as boiling or vaporization points)

Delivering sample to the GC

a. Purge / Trap
   Sample is purged with an inert gas, which strips any volatile organics out. This gaseous mixture is then moved through a "trap" that retains the organics. The trap is then thermally desorbed onto the head of the GC column.
b. Liquid Injection
   Sample is either injected "neet" into the injector or a liquid extraction of the sample is injected into the injector.

The injector vaporizes the sample to deliver it to the column as the "gas" phase. There are three main types of injection.

a. Split injector / injection
   A percentage of the vaporized sample is vented or purged away from the column.
b. Splitless injector / injection
   Purge flow is stopped for a time to allow as much as possible of the analytes to reach the column before venting the solvent.
c. On column injection
   Liquid sample is injected directly onto the head of the column.

Columns are what hold the "packing" or what causes the separation of the mixtures. There are two types.

a. Packed Short (1 to 2 m), liquid phase (packing) is coated onto particles (support) that is "packed" into the column.
b. Capillary Long (15 to 75 m), liquid phase is coated directly onto the inside wall of the capillary column.

Detectors are what allow the GC to "see" the compounds that are separated. There are seven major types of GC detectors used in wastewater work.

a. FID Flame Ionization Detector
   General organic compounds
b. ECD Electron Capture Detector
   Generally halogenated compounds, but also O, N, P, and S
c. Hall (Electrolytic Conductivity)
   Halogenated compounds
d. PID Photo Ionization Detector
   Aromatic compounds and unsaturates
e. NPD Nitrogen Phosphorous Detector
   Nitrogen, and Phosphorous compounds
f. TCD Thermal Conductivity Detector
   Detects anything
g. Mass Spectrometer
   Detects any compound with mass within the range set.
   Gives structural data as well as mass data.

Be familiar with how to calculate a response factor, and how that factor is used in both internal standard calibrations and external calibrations.

Internal Standard Method:

Response Factor for Internal Standard

$$RF_{is} = \frac{\text{Peak Response}_{is}}{\text{Concentration}_{is}}$$

Relative Response Factor for an Analyte

$$RRF_x = \frac{\text{Peak Response}_x}{(RF_{is})\,(\text{Concentration}_x)} = \frac{(\text{Concentration}_{is})\,(\text{Peak Response}_x)}{(\text{Concentration}_x)\,(\text{Peak Response}_{is})}$$

II. **GC Methods**

1.	601 / 602	Volatiles by Purge / Trap 601 - Halogenated purgeables on Hall detector 602 - Aromatic purgeables on PID
2.	603	Acrolien, Acrylonitrile, Acetonitrile by Purge / Trap on FID
3.	604	Phenols by extraction and GC FID
4.	605	Benzidines by HPLC
5.	606	Phthalate Esters by extraction and GC FID
6.	607	Nitrosamines by extraction and GC FID
7.	608	Chlorinated Pesticides by extraction and GC ECD
8.	609	Nitroaromatics, & Isophorone by extraction and GC FID
9.	610	PolyNuclear Aromatic Hydrocarbons (PNA's) by HPLC
10.	611	Haloethers by extraction and GC Hall
11.	612	Chlorinated Hydrocarbons by extraction and GC ECD
12.	624	Volatiles by Purge / Trap GC/MS
13.	625	Semivolatiles by extraction and GC/MS
14.	8150	Chlorinated Acid Herbicides by extraction, esterification and GC ECD
15.	8140	Organophosphorus Pesticides by extraction and GC NPD
16.	Total Petroleum Hydrocarbons (both ranges) Gasoline range - Purge / Trap and GCFID Diesel range - extraction and GC FID	

By studying Methods 624 and 625 you will gain a good overview of organics methods in general.

Total Phenols

Be familiar with the preliminary distillation step, the apparatus, reagents, and procedures.

Be familiar with the colorimetric (4AAP) detection method, it's apparatus, reagents, and procedures.

Be familiar with the common interferences of this method and how to overcome them.

Salinity

Be familiar with the procedures, apparatus and units used in the analysis for salinity.

Understand the difference between Practical Salinity and NaCl Equivalent Salinity.

Be able to explain the difference between the conductivity method and the density method, including the strengths and weaknesses of each.

Total Organic Carbon (TOC)

Be able to explain the different methods of oxidation used in TOC, including when each is appropriate.

Be familiar with how TOC is measured, what type of detection is used, and how the carbon is delivered to the detector.

Be able to explain the following terms;
- Total Organic Carbon
- Dissolved Organic Carbon
- Purgeable Organic Carbon
- Inorganic Carbon

Be familiar with the apparatus, reagents and procedures for the three methods for TOC.

Be familiar with the calibration method(s) used for this analysis.

Total Organic Halogens

Be familiar with what Total Organic Halogens are and how they are measured.

Microorganisms

Be familiar with the different types of microorganisms in activated sludge, what are considered normal, and what happens to them when the sludge is out of balance.

Be familiar with the different types of microorganisms in anaerobic sludge digestion.

Biology

Be familiar with the biological testing on wastewater including coliforms (total and fecal), and microscopic examination, and how these results affect effluents, and plant operations.

Be familiar with the effect of Nitrifying bacteria on BOD determinations.

Sampling

Be familiar with the sampling information covered in both the Class I, II and Class III NTK documents and expand it to include the specific analyses listed on page 1 of this NTK. Pay particular attention to organics and bioassay sampling requirements as they have some unusual aspects.

GENERAL LABORATORY INFORMATION AND PROCEDURES

Know the names of the instrumentation, major instrument components and/or apparatus and operational theory used to conduct analyses for the parameters listed in the Class I, II and Class III study material.

Be familiar with and be able to use the method of standard additions.

Understand why the method of standard additions is used.

Be familiar with the troubleshooting procedures for any of the methods in the Class I, Class II, and Class IV NTK.

Be familiar with all general laboratory calibrations and calibration procedures.

Know how to verify and measure separation of components in gas chromatography.

Be familiar with and be able to use the method of internal standard calibration.

Be familiar with and be able to use the method of external standard calibration.

Apparatus / Reagents

Be familiar with the equipment, apparatus, and reagents for all of the tests covered in the Class I, Class II, and Class IV NTK. You should be able to describe, or recognize the description of any of the apparatus. You should also be able to explain the operating conditions and the reason for those conditions.

Know the names and major components names of all apparatus used in instrumental testing.

Be familiar with procedures for preparation of common laboratory reagents including lab pure waters.

Chemistry

Be familiar with the chemistry information covered in the Class I, II and Class III NTK documents and expand it to include the specific analyses listed on page 1 of this NTK.

Be familiar with the nomenclature of organic chemicals and their identifying functional groups.

Be able to balance both a chemical equation, and a chemical formula.

Be able to determine the normality of a reagent given its standardization data.

Be familiar with the detection chemistry for each parameter covered in the Class I, II and Class III NTK. This means what chemicals are used, and what each of their roles are in the method.

Mathematics

Be able to work problems covering at least the following;

- Pump rates
- Units conversions
- Standard preparation
- Preparation of a reagent from a non-pure source
- Specific gravity
- Concentration determination

Standardization
Chemical dosage

Be able to use set up algebraic equations from word problems.

Be able to calculate a result for any of the tests covered in the Class I, Class II, and Class IV NTK given the raw data.

HINT: There is a LOT of math on the Class IV !!!

Safety

Be familiar with the Safety information covered in both the Class I, II and Class III NTK documents and expand it to include the specific analyses listed on page 1 of this NTK.

Be familiar with what odor Hydrogen Cyanide has, and how it is formed.

Be familiar with basic radiation safety.

Know the hazardous properties of common laboratory chemicals and the safety measures associated with them. This should include but not be limited to ;
Sulfuric Acid, Nitric Acid, Hydrochloric Acid, Hydrofluoric Acid, Perchloric Acid,
Sodium Hydroxide, Ammonium Hydroxide, and flammable gases

Quality Assurance / Quality Control (QA/QC)

Be familiar with the QA/QC information covered in both the Class I, II and Class III NTK documents and expand it to include the specific analyses listed on page 1 of this NTK.

Be familiar with the statistics involved in setting control limits (ie. Standard Deviation, Mean, Confidence windows).

Compliance Rules and Regulations

Be familiar with the Compliance information covered in both the Class I, II and Class III NTK documents and expand it to include the specific analyses listed on page 1 of this NTK.

WWTP Operational Information

Be familiar with the Operational information covered in both the Class I, II and Class III NTK documents and expand it to include the specific analyses listed on page 1 of this NTK.

Understand Food to Mass ratios, how to calculate them, and their significance to operations.

Be able to perform rate/dosage calculations.

Understand “Jar” tests and their significance to operations.

Laboratory Management and Laboratory Supervision

Know the four levels of supervision.

S1 = "working foreman" (does the same work with the workers but also reviews others)

S2 = "coaching" (has active involvement in work activities)

S3 = "advisor" (has no active participation in the work, but works with policy)

S4 = "CEO" (is a hands off manager)

Know the Precision / Accuracy (P/A) minimum requirements for qualifying an analyst for a given parameter.

Know the minimum requirements to qualify a laboratory manager, and the responsibilities of that position.

Know the minimum requirements to qualify a laboratory supervisor, and the responsibilities of that position.

Class IV Potpourri

Be familiar with specific indicators used in various analyses including the tests in which the following can be used:

- Methyl orange
- Methyl purple

Definitions to Know

- Total Toxic Organics

Be familiar with the penetration power of the following types of radiation:

- Alpha
- Beta
- Gamma

Describe the differences between the total cyanide test and the cyanide amenable to chlorination test.

Be able to recognize/name functional groups in organic chemical diagrams e.g. alcohol, aldehyde, alkane, alkene, ester, ketone

Describe how surfactants are classified.

Given appropriate data be able to use the pounds formula to solve for any component.

Perchloric acid may react explosively with what types of substances?