

Class III Wastewater Laboratory Analyst Examination Study Guide 2008

Specific laboratory analyses included on Class III Exam:

All Information and tests from Class I and Class II

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
- Supervisory/Management Information

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 and Class II 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 and the conditions under which certain families of microorganisms flourish, in particular those such as filaments which can cause treatment plant problems.

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.

Why are daphnia often chosen for Whole Effluent Toxicity testing?

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

Be able to define the following terms;

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

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 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

Describe appropriate sample preservation for metals samples.

Describe the sample pretreatment procedure for obtaining dissolved 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
- 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 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 (including filter type and size)
9. Total Recoverable metals

Be familiar with the following concerning AA:

- use of matrix modifiers
- Deuterium arc
- Why nitrous oxide is used in AA
- Why flame AA standard is measured every 10 samples

Be familiar with the following concerning ICP

- Each ICP sample run should begin with the analysis of what?
- In ICP the argon is ionized by?
- Why ICP standard is measured every 10 samples

Organics by Gas Chromatography (GC)

Describe the appropriate preservation techniques for various types of organics analyses.

The term "Total Toxic Organics" as defined by EPA includes which chemicals?

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.

What is the appropriate preservation for purgeable aromatic hydrocarbons?

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 Acrolin, 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

Be able to recognize specific organic chemicals from the following lists:

- Total Toxic Organics
- Purgeable Aromatic hydrocarbons

- Volatile organic compounds
- Semi-volatile organic compounds

Why are surrogate compounds used in organic analysis methods?

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.

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.

Total Organic Halogens

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

Activated Sludge

Be familiar with the different types of microorganisms in activated sludge, what are considered normal, and what symptoms the activated sludge will exhibit when there is a problem.

Given appropriate data, be able to calculate the Food to Mass ratio.

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.

Sampling

Be familiar with the sampling information covered in both the Class I and Class II 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.

When shipping a wastewater sample by common carrier, the regulations of what Federal agency apply to the packaging and shipping of the sample?

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 and Class II 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 III NTK.

Be familiar with all general laboratory calibrations and calibration procedures.

Describe how to prepare the following types of laboratory water:

- organic free water
- Ammonia free water
- Carbon dioxide free water

Be familiar with the following: specifications and uses of:

- specifications and uses of Type I, Type II, Type III and Type IV laboratory water
- use and maintenance of mixed bed resin cartridges in laboratory water treatment

Be familiar with the relationship between absorbance and transmittance as it relates to spectrophotometric analyses as well as how to express the relationship mathematically

Describe how to separate components with different boiling points.

What is the definition of MDL, what is the MDL determination procedure and how many analyses are required?

What is the definition of PQL?

What does NELAC stand for?

What is the National Institute of Standards and Technology (NIST) and what role does it play in the wastewater laboratory?

Describe the relationship between absorbance and transmittance when using laboratory instrumentation.

Apparatus / Reagents

Be familiar with the equipment, apparatus, and reagents for all of the tests covered in the Class I, Class II, and Class III 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 name's 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 both the Class I and Class II 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.

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

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 III NTK given the raw data.

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

Safety

Be familiar with the Safety information covered in both the Class I and Class II 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, carbon dioxide, and flammable gases

Describe how to safely move gas cylinders.

Describe the types of circuit breakers typically used in a laboratory.

What toxic fumes are emitted in the digestion of TKN?

How should fecal coliform dishes be disposed of after counting?

Describe the specifications for the Hazardous Characteristic of Ignitability

Be familiar with the following documents and regulations:

1. Chemical Hygiene Plan
2. Material Safety Data Sheets
3. Hazard Communication Standard and Right-to-Know regulations

Know what to do if an employee is injured on the job.

Be familiar with typical fume hood requirements for air exchange.

Quality Assurance / Quality Control (QA/QC)

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

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.

Calibration standards and reference materials must be traceable to what entity?

Control limits are set at the average of the data set plus or minus how many standard deviations?

In a data set for developing a control chart what percent of values fall within 2 standard deviations?

Define the following terms as they relate to QA/QC: random error, bias, PQL, MDL, IDL, control limits

Where does the SOP for each analytical procedure originate?

Given appropriate data be able to determine the control limits on a control chart.

Describe the difference between quality control and quality assurance and know the purpose of each.

Compliance Rules and Regulations

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

Resource Conservation and Recovery Act (RCRA) waste material are defined by what properties?

WWTP Operational Information

Be familiar with the Operational information covered in both the Class I and Class II 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 wastewater plant operations.

Be familiar with common filamentous bacteria found in activated sludge and the characteristics of each type.

Supervisory/Management Information

Be familiar with what an organization chart shows

What is the purpose of good public relations?