UNCONTROLLED

WEST VALLEY NUCLEAR SERVICES CO., INC.

ANALYTICAL CHEMISTRY METHOD
ANALYTICAL AND PROCESS CHEMISTRY

ACM-NO_3-3701, Rev. 3
Effective Date: 06/28/89

NITRATE - PHOTOMETRIC (UV)

Approved by:

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Analytical and Process Chemistry

Part I

1.0 PURPOSE

Provide a method for the determination of nitrate in samples of melter feed using UV absorption.

2.0 APPLICATION

This technique is very sensitive, but is subject to interference from organic material present in the sample.

3.0 DISCUSSION

Measurement of UV absorption at approximately 200 nm enables rapid determination of NO_3^-. Because dissolved organic matter also may absorb at 200 nm and NO_3^- does not absorb at 275 nm, a second measurement made at 275 nm may be used to correct the NO_3^- value. The method is not recommended for samples requiring a significant correction. Sugar, which is often added to the melter feed does not interfere with this method.

4.0 REFERENCES

Standard Methods for the Examination of Water and Wastewater, 16th Edition, 1985, Method 418A, p.392.

Part II

5.0 EQUIPMENT

5.1 UV Spectrophotometer Calibrated

5.2 Quartz Absorption Cells

5.3 Glass Volumetric labware

6.0 REAGENTS AND STANDARDS

6.1 Refer to ACP 8.1 prior to preparing any standard solutions.

6.2 Nitrate-free water: Use distilled, deionized water.

> 6.3 Calibration stock nitrate solution (1000 ppm NO_3): Dissolve 0.163 g of potassium nitrate (KNO_3) in 100 mL nitrate-free water. This solution is stable for one month.

6.4 Calibration working standard nitrate solution (1 mL = 100μg NO_3): Dilute 10.0 mL of the stock nitrate solution (6.3) to 100 mL with nitrate free water. This solution should be made fresh when needed.

> 6.5 QC stock (1,000 ppm) dissolve 0.1371 g of sodium nitrate ($NaNO_3$) in 100 mL of nitrate-free water. This solution is stable for one month.

> 6.6 QC working standard nitrate solution (5 μg NO_3): Dilute 0.5 mL of stock nitrate solution (6.5) to 100 mL with nitrate free water. This solution is made fresh when analysis is performed.

7.0 SAFETY

7.1 Use normal laboratory safety precautions as stated in ACP 7.2.

8.0 RECORDS

8.1 All relevant measurements and sample identification shall be recorded on the nitrate work sheet (attachment A). The final result is then recorded on the Analytical Request Sheet (ACP 5.1).

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9.0 CALIBRATION AND CONTROL

9.1 Calibration curve: Prepare NO_3^- calibration standards in the range of 0 to 6 ppm NO_3 by diluting the following volumes of standard nitrate solutions to 100 mL: 0, 1.0, 2.0, 3.0, 4.0, 5.0, and 6.0 mL

> 9.2 The calibration curve shall be determined at a frequency of every 12 months.

9.3 Continue as in step 10.2.

> 9.4 Control is verified by the use of Quality Control (QC) sample whose results are plotted on a quality control chart. The QA sample is repeated every 20 samples in the set. If results deviate by more than 3σ (Sigma), the known will be reanalyzed, and the QC sample repeated. See ACP 8.2.

10.0 PROCEDURE

10.1 Prepare dilutions of the samples to bring the nitrate level into the calibration range. Two dilutions will be necessary. Pipet 0.1g into 100 mL volumetric flask. Record weight on worksheet. Dilute to 100 mL mark. Record total weight. Pipet 2.0g of first dilution into another 100 mL flask. Record weight. Dilute to 100 mL mark and record total weight of second dilution on worksheet also. Typical dilutions for melter feed would be 1:50,000 prepared on a weight basis.

10.2 Determine cuvette correction of two quartz cells at 200 nm and 275 nm using nitrate free water and record on worksheet.

10.3 Using quartz cuvettes, measure the absorbance at 200 nm, then at 275 nm (using nitrate free water to set the zero at each wavelength). Subtract 2x the absorbance found at 275 nm from that measured at 200 nm. If greater than a 10 percent correction, filter the test solutions and repeat the test. An alternate procedure must be used if interferences remain at levels greater than indicated above. Rinse cell used to measure sample absorbance with aliquot of sample, then refill cell with second aliquot of sample being measured.

11.0 CALCULATIONS

11.1 $\text{ABS } NO_3^-\text{ (net)} = \text{ABS } NO_3^-\text{ (200 nm)} - 2 \times \text{ABS } NO_3^-\text{ (275 nm)}$

11.2 Using equation obtained from calibration curve, determine ppm NO_3 (net)

11.3 $\text{ppm } NO_3^-\text{ (final)} = \text{ppm } NO_3\text{ (net)} \times \text{Dilution Factor D. F.}$

11.4 D.F. = $\frac{\text{sample and diluent wt(g)}}{\text{sample aliquot wt(g)}}$ =

> $\frac{\text{(Sample Aliquot+Diluent Wt of Dilution 1)X(Sample Aliquot+Diluent Wt of Dilution 2)}}{\text{(Sample Aliquot Wt of Dilution 1) X (Sample Aliquot Wt of Dilution 2)}}$

> Where ALIQ1= Sample Aliquot Wt of Dilution 1

ALIQ2= Sample Aliquot Wt of Dilution 2

ALlQ1 + Diluent 1 = Sample Aliquot + Diluent Wt of Dilution 1

ALlQ1 + Diluent 2 = Sample Aliquot + Diluent Wt of Dilution 2

12.0 ATTACHMENTS

12.1 Attachment A: Nitrate - Photometric Work Sheet

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ATTACHMENT A

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NITRATE - PHOTOMETRIC WORK SHEET

SAMPLE NAME _____ LOG NUMBER _____

SPECIAL INSTRUCTIONS UV Spec. Serial # _____

(*) Using equation from calibration curve in QC book

CUVETTE CORRECTION: @ 200 nm @ 275 nm

ALL WEIGHT IN GRAMS

SAMPLE ID				
ALIQ1 Wt- D				
ALIQ2 Wt- E				
D x E- F				
ALIQ1 + Diluent 1 Wt- G				
ALIQ2 + Diluent 2 Wt-H				
G x H- J				
ABS AT 200 nm				
ABS AT 200 nm - CUVETTE CORR.(A)				
ABS AT 275 nm				
ABS AT 275 nm - CUVETTE CORR.(B)				
C - B x 2				
CORRECTED ABS - A-C				
PPM (*)				
DILUTION FACTOR $\frac{J}{F}$				
SAMPLE PPM ($\mu g/g$) OF NO_3				

ANALYST _____ DATE _____

APPROVED _____ DATE _____