

APPENDIX D-3
Analytical Procedure for Total Kjeldahl Nitrogen (TKN):
Lachat Method

QuikChem METHOD 13-107-06-2-D

DETERMINATION OF TOTAL KJELDAHL NITROGEN IN SOILS
AND PLANTS BY FLOW INJECTION ANALYSIS

(Block Digester Method)

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Applications Group

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QuikChem Method 13-107-06-2-D

Total Nitrogen in Kjeldahl Digests of Soils and Plants

(Block Digester Method)

1.0 to 100 mg N/L
0.03 to 2.50%N in Plant Tissue
0.01 to 1.25% N in Soil

--Principle--

Samples are digested with sulfuric acid in 75 mL tubes in a block digester. With a copper sulfate catalyst, the samples' Kjeldahl nitrogen is converted to the ammonium cation. Potassium sulfate is also added to raise the boiling temperature of the digestion and speed the conversion to ammonium. The digest is diluted to a final volume of 50 mL with DI water.

Approximately 0.06 mL of the digested sample is injected onto the chemistry manifold where its pH is controlled by raising it to a known, basic pH with a concentrated buffer. This in-line neutralization converts the ammonium cation to ammonia, and also prevents undue influence of the sulfuric acid matrix on the pH-sensitive color reaction which follows.

The ammonia thus produced is heated with salicylate and hypochlorite to produce blue color which is proportional to the ammonia concentration. The color is intensified by adding sodium nitroprusside. The presence of tartrate in the buffer prevents precipitation of calcium and magnesium.

--Special Apparatus--

1. Heating Unit
2. Block Digester/75 mL tubes (Lachat Part No. 1800-000)
3. Vortex Mixer

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QuikChem Method 13-107-06-2-D

DETERMINATION OF TOTAL KJELDAHL NITROGEN BY FLOW INJECTION ANALYSIS COLORIMETRY

1. SCOPE AND APPLICATION

- 1.1. This method covers the determination of nitrogen in dried, ground plant or soil samples. Since acid consumption during digestion is proportional to organic matter content, highly organic materials may require less sample. If there is a doubt about the best sample weight, preliminary experiments should be run.
- 1.3. The applicable range is 1.0 to 100 mg N/L. The method detector limit is 1.0 mg N/L. The method throughput is 72 injections per hour.

2. INTERFERENCES

- 2.1. Samples must not consume more than one fifth of the sulfuric acid during the digestion. The buffer will accommodate a range of 5.6 to 7% (v/v), H₂SO₄ in the diluted digestion sample with no change in signal intensity.

3. SAFETY

- 3.1. The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.
- 3.2. Each laboratory is responsible for maintaining a current awareness file of the Occupational Health and Safety Act (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.
- 3.3. **Always** wear a full face shield, gloves, and a lab coat when working with hot digest samples.
- 3.4. The following chemicals have the potential to be highly toxic or hazardous, for detailed explanation consult the MSDS.
 - 3.4.1. Sodium Hydroxide
 - 3.4.2. Sulfuric Acid
 - 3.4.3. Sodium Nitroprusside

- 3.4.4. Sodium salicylate
- 3.4.5. Clorox bleach (5.25% sodium hypochlorite)
- 3.4.6. Copper sulfate
- 3.4.7. Ammonium chloride
- 3.4.8. Hydrochloric acid

4. EQUIPMENT AND SUPPLIES

- 4.1. Balance -- analytical, capable of accurately weighing to the nearest 0.0001 g.
- 4.2. Glassware -- Class A volumetric flasks and pipettes or plastic containers as required. Samples may be stored in plastic or glass.
- 4.3. Flow injection analysis equipment designed to deliver and react sample and reagents in the required order and ratios.
 - 4.3.1. Autosampler
 - 4.3.2. Multichannel proportioning pump
 - 4.3.3. Reaction unit or manifold
 - 4.3.4. Colorimetric detector
 - 4.3.5. Data system
- 4.4. Special Apparatus
 - 4.4.1. Heating unit
 - 4.4.2. Block Digester/75 mL tubes (Lachat Part No. 1800-000)
 - 4.4.3. Vortex Mixer

5. REAGENTS AND STANDARDS

5.1. PREPARATION OF REAGENTS

Use deionized water (10 megohm) for all solutions.

Degassing with helium:

To prevent bubble formation, degas all solutions except the standards with helium. Use He at 140kPa (20 lb/in²) through a helium degassing tube (Lachat Part No. 50100.) Bubble He through the solution for one minute.

Reagent 1. Buffer

By Volume: In a 1 L volumetric flask dissolve **65 g sodium hydroxide** (NaOH), **50.0 g sodium potassium tartrate** (potassium sodium tartrate, d,1-NaKC₄H₄O₆·H₂O) and **26.8 g sodium phosphate dibasic heptahydrate** (Na₂HPO₄·7H₂O), and **950 g water**. Dilute to the mark and invert to mix. Stir or shake until dissolved.

By Weight: To a tared 1 L container add **65 g sodium hydroxide** (NaOH), **50.0 g sodium potassium tartrate** (potassium sodium tartrate, d,1-NaKC₄H₄O₆·H₂O), **26.8 g sodium phosphate dibasic heptahydrate** (Na₂HPO₄·7H₂O), and **950 g DI water**. Stir or shake until dissolved.

Reagent 2. Salicylate Nitroprusside

By Volume: To a tared 1 L volumetric flask dissolve **150.0 g sodium salicylate** [salicylic acid sodium salt, C₆H₄(OH)(COO)Na], **1.00 g sodium nitroprusside** [sodium nitroferricyanide dihydrate, Na₂Fe(CN)₅NO·2H₂O] and about **800 mL DI water**. Dilute to the mark and invert to mix. Store in a dark bottle and prepare fresh monthly.

By Weight: To a tared 1 L dark container, add **150.0 g sodium salicylate** [salicylic acid sodium salt C₆H₄(OH)(COO)Na], **1.00 g sodium nitroprusside** [sodium nitroferricyanide dihydrate, Na₂Fe(CN)₅NO·2H₂O] and **908 g water**. Stir or shake until dissolved. Store in a dark bottle and prepare fresh monthly.

Reagent 3. Hypochlorite Solution (0.3% NaOCl)

By Volume: In a 1 L volumetric flask, dilute **60.0 mL Regular Clorox Bleach** (5.25% sodium hypochlorite, The Clorox Company, Oakland, CA) to the mark with **DI water**. Invert to mix. Prepare fresh daily.

By Weight: To a tared 1 L container, add **64 g of Regular Clorox Bleach** (5.25% sodium hypochlorite, The Clorox Company, Oakland, CA) and **936 g DI water**. Shake to mix. Prepare fresh daily.

Reagent 4. Matrix Blank/Diluent/Digestion Solution

NOTE: Prepare three liters of this solution.

By Volume: In a 1 L volumetric flask, add approximately 700 mL DI water, then add 70 mL concentrated sulfuric acid (H_2SO_4). Add 30 g potassium sulfate (K_2SO_4). Add 2.5 g copper sulfate ($CuSO_4 \cdot 5H_2O$) and dilute to the mark with DI water. Mix with a magnetic stirrer and allow the solution to cool. Dilute to the mark with DI water after the solution has cooled. Prepare fresh monthly.

By Weight: In a tared 1 L container, add 915 g DI water, then add 128.1 g concentrated sulfuric acid (H_2SO_4). Add 30 g potassium sulfate (K_2SO_4). Add 2.5 g copper sulfate ($CuSO_4 \cdot 5H_2O$). Mix with a magnetic stirrer, or invert to mix, and allow the solution to cool. Prepare fresh monthly.

5.2. PREPARATION OF STANDARDS

Standard 1. Stock Standard 1000 mg N/L

By Volume: In a 1 L volumetric flask dissolve 4.716 g ammonium sulfate $(NH_4)_2SO_4$ primary standard in about 800 mL DI water. Dilute to the mark with DI water and invert to mix.

Standard 2. Working Stock Standard 100 mg N/L

By Volume: In a 1 L volumetric flask, add 100.0 mL Standard 1, 30 g potassium sulfate (K_2SO_4), 2.5 g copper sulfate ($CuSO_4 \cdot 5H_2O$), and 70 mL sulfuric acid (H_2SO_4). Dilute to the mark with DI water. Invert to mix.

Working Standards (Prepare Daily)	A	B	C	D	F
Concentration mg N/L	100	75.0	50.0	25.0	0.00

By Volume

Volume (mL) of working stock standard 2 diluted to 100 mL with reagent 4	100	75.0	50.0	25.0	0.0
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By Weight

Weight (g) of stock standard 2 diluted to final weight (~100 g) multiplied by factor below with reagent 4	100	75.0	50.0	25.0	0.0
Division Factor Multiply exact weight of the standard by this factor to give final weight	1.00	0.75	0.50	0.25	0

6. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 6.1. Plant and soil samples are dried overnight at a temperature less than 100°C. The dried soil is then ground to pass a 20 mesh screen and plant tissue is ground to pass a 40 mesh screen. If this fineness of grind is not achieved, samples may not be homogenous. To verify homogeneity, several of each sample should be digested. Digests may be covered tightly and stored for one week.

7. PROCEDURE

7.1. DIGESTION PROCEDURE

NOTE: Calibration is performed using standards in the digest matrix, i.e., NOT digested. Standards are not digested but are instead synthetic solutions of ammonium-nitrogen prepared in the digest matrix. Instructions for preparing standards in the digest matrix are given in section 5 of this method.

CAUTION: Always wear safety goggles, a complete face shield, a labcoat, and acid resistant rubber gloves when carrying out the following procedure. It is also important to follow the safety procedures described in the block digester manual.

- 7.1.1. Since standards are not carried through the digestion procedure, a sample with known concentration of total nitrogen should be included with each digestion set to verify complete digestion.
- 7.1.2. Start with a clean, dry set of digestion tubes. To each tube, add 0.2 g of plant tissue or 0.4 g of soil. If weighing papers are used, a blank should be carried through the digestion and the sample results should be corrected for the blank. If the complete set of tubes is not being used, remove the empty tubes prior to digestion.
- 7.1.3. To each tube add 1.50 g of potassium sulfate (K_2SO_4) and 0.125 g of copper sulfate Pentahydrate ($CuSO_4 \cdot 5H_2O$). This can be accomplished by adding a commercially available salt catalyst mixture in tablet form. (Available from SCT Sales, Inc. Littleton, CO., (303-730-0084, cat no. KC-C1).
- 7.1.4. Add 2-4 boiling stones to each tube. Hengar (Alundum) granules are effective for smooth boiling. They are available from Fisher Scientific, cat. no. S145-500.
- 7.1.5. To each tube add 3.5 mL of concentrated sulfuric acid (H_2SO_4). This is efficiently accomplished using an acid resistant repipet device (EM Science, 108033-1).
- 7.1.6. Place tubes in block digester which has been preheated to 160°C. On the block digester controller, set Temp 1 to 390°C and Time 1 to 180 minutes. If the block temperature is greater than 180°C, cool the block before inserting tubes. If using the Lachat BD-46 or BD-26, the entire digestion can be done with cold fingers in place.

3. Add 10 ml of 1N $K_2Cr_2O_7$ with a volumetric pipette. Swirl the flask gently to disperse the soil in the solution.
4. Rapidly add 20 ml concentrated sulfuric acid, directing the stream into the suspension. Immediately swirl the flask gently until soil and reagents are mixed, then more vigorously for a total of 1 minute.
5. Allow the flask to stand on a heat-impervious surface for about 30 minutes.
6. Add 200 ml water to the flask, and filter the suspension if experience with the particular soil shows that the endpoint of the titration cannot be otherwise be clearly discerned.
7. Add three drops o-phenanthroline indicator and titrate the solution with 0.5N $FeSO_4$. As the endpoint is approached, the solution takes on a greenish cast and then changes to a dark green. At this point, add the ferrous sulfate solution drop by drop until the color changes sharply to blue to red (maroon in reflected light against a white background.)
8. To standardize the dichromate, make a blank determination without soil.
9. Repeat the determination with less soil if greater than 75% of the dichromate is reduced.
10. Calculate the results as follows:

$$\text{Organic C \%} = (\text{meq } K_2Cr_2O_7 - \text{meq } FeSO_4)(0.003)(100)(1.30)/(\text{g water-free soil})$$

$$= (10.0 - \text{meq } Fe SO_4)(0.003)(100)(1.30)/(\text{g water-free soil})$$

Note: 1.30 is an empirically obtained correction factor.

11. Calculate the normality of the ferrous sulfate solution as follows:

$$\text{Normality} = 10/(\text{vol})$$

where vol is the volume of ferrous ion solution required to titrate 10.0 ml 1 N $K_2Cr_2O_7$.

Note: Ferrous ammonium sulfate may be substituted for ferrous sulfate in this procedure.

- 7.1.7. Continue to digest for three hours. During the first two hours the temperature will ramp to 390°C and then during the third hour the temperature should hold at 390±5°C. It is critical that the digestion's remain at 390°C for one full hour.
- 7.1.8. Remove the samples from the block and allow about 10 minutes for cooling.
- 7.1.9. Add 46.5 mL of DI water to each tube. Carefully vortex to mix, pointing the tube away in case of splashing. The final volume should be 50 mL.
- 7.1.10. If digests are not run immediately they should be covered with Parafilm or capped tightly.

7.2. SYSTEM START-UP AND CALIBRATION PROCEDURE

- 7.2.1. Prepare reagent and standards as described in section 5.
- 7.2.2. Set up manifold as shown in section 11.1.
- 7.2.3. Input peak timing and integration window parameters as specified in section 11.
- 7.2.4. Pump DI water through all reagent lines and check for leaks and smooth flow. Switch to reagents and allow the system to equilibrate until a stable baseline is achieved.
- 7.2.5. Place standards in the autosampler, and fill the sample tray. Input the information required by data system, such as concentration, replicates and QC scheme.
- 7.2.6. Calibrate the instrument by injecting the standards. The data system will then associate the concentrations with responses for each standard.
- 7.2.7. After a stable baseline has been obtained, start the sampler and perform analysis.

7.3. SYSTEM NOTES

- 7.3.1. Allow at least 15 minutes for the heating unit to warm up to 60°C.
- 7.3.2. Upon system start-up it is crucial to establish good flow before the salicylate reagent is added. If the salicylate reagent merges with the acid sample prior to neutralization, it will precipitate. Always add the salicylate reagent last. When in doubt, check that the flowcell waste stream is alkaline (with litmus paper) before adding that salicylate reagent.
- 7.3.3 If baseline drifts, peaks are too wide, or other problems with precision arise, clean the manifold by the following procedure:

Place all reagent transmission lines in water and pump to clear reagents (2-5 minutes).

Place reagent lines and carrier in a 1 N hydrochloric acid (1 volume of HCl added to 11 volumes of water) and pump for several minutes.

Place all transmission lines in water and pump for several minutes.

Resume pumping reagents.

At the end of the run place all transmission lines **except the buffer** in water and flush system for two minutes. Place buffer transmission in water, flush system, then pump all lines dry.

- 7.3.4. In normal operation nitroprusside gives a yellow background color which combines with the blue indosalicylate to give an emerald green color. This is the normal color of the solution in the waste container.
- 7.3.5. With most block digesters, about 3% of the original concentration of sulfuric acid is lost during digestion. However, large variations in residual acid concentration will result in poor accuracy and abnormal peak shapes.
- 7.3.6. Digestion efficiency may be better with a mercury catalyst.
- 7.3.7. The percent nitrogen can be calculated by the formula:

$$\%N = [(V_D/W_S) \times C_D]/10,000$$

where:

V_D = Total digest volume (mL), Default = 50 mL

W_S = Weight of sample (g), Default = 0.2 g (Plant), 0.4 g (Soil)

C_D = Concentration in the digest (mg N/L)

8. DATA ANALYSIS AND CALCULATIONS

- 8.1. Calibration is done by injecting standards. The data system will the prepare a calibration curve by plotting response versus standard concentration. Sample concentration is calculated from the regression equation.
- 8.2. Report only those values that fall between the lowest and highest calibration standards. Samples exceeding the highest standard should be diluted with matrix blank and reanalyzed.
- 8.3. Report results in % nitrogen.

9. METHOD PERFORMANCE

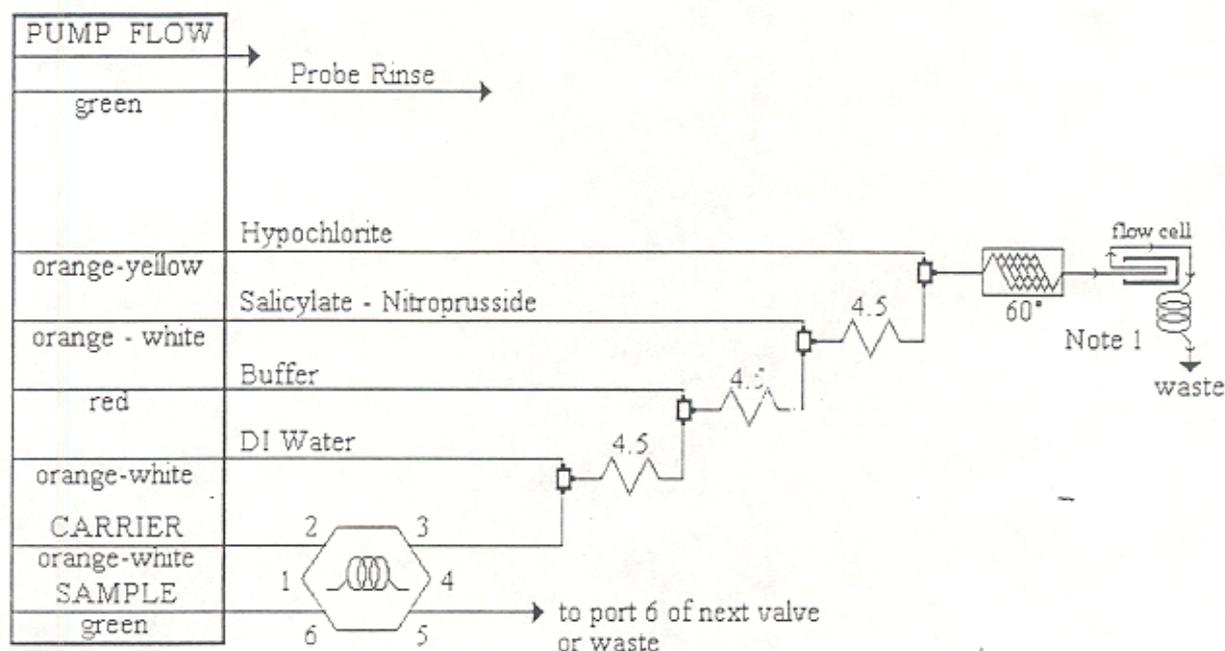
- 9.1. The method support data are presented in section 11. This data was generated according to Lachat Work Instruction J01002, Procedure for Generating Method Support Data on the QuikChem 8000.

10. REFERENCES

- 10.1. Lachat Instruments Inc., QuikChem Method 13-107-06-2-D written by David Diamond on 28 Dec 1992.
- 10.2. Correspondence, Allen Doyle, University of Alaska, Fairbanks, Institute for Arctic Biology, 4/20/92.
- 10.3. Jones, N.M. and H.D. Bradshaw, Copper: An Alternative to Mercury; more effective than zirconium in Kjeldahl Digestion of Ecological material. *Communications in Soil and Plant Analysis*, 20:1513-1524, 1989.
- 10.4. Kaltra, Y.P. and D.G. Maynard, *Methods Manual for Forest Soil and Plant Analysis*, Information Report NOR-X-39, Forestry Canada, Ontario Canada, 1991.

11. TABLE, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

11.1. TOTAL KJELDAHL NITROGEN MANIFOLD DIAGRAM



Sample Loop = Microloop Interference Filter = 660 nm

CARRIER is DI Water.

Manifold tubing is 0.5 mm (0.022 in) i.d. This is 2.5 uL/cm.

4.5 is 70 cm of tubing on a 4.5 cm coil support

APPARATUS: An injection valve, a 10 mm path length flow cell, and a colorimeter detector module are required. The  shows 650 cm of tubing wrapped around the heater block at the specified temperature.

Note 1: 200 cm back pressure loop, 0.5 mm (0.022 in) i.d. tubing

11.2. DATA SYSTEM PARAMETERS FOR QUIKCHEM 8000

The timing values listed below are approximate and will need to be optimized using graphical events programming.

Sample throughput: 72 samples/h, 50 s/sample
Pump Speed: 35
Cycle Period: 50

Analyte Data:

Concentration Units: mg N/L
Peak Base Width: 17.2 s
% Width Tolerance: 100
Threshold: 20000
Inject to Peak Start: 45 s
Chemistry: Direct

Calibration Data:

Level	1	2	3	4	5
Concentration mg N/L	100	75.0	50.0	25.0	0.00

Calibration Fit Type: 2nd Order Polynomial
Calibration Rep.Handling: Average
Weighting Method: None
Concentration Scaling: None
Force Through Zero: No

Sampler Timing:

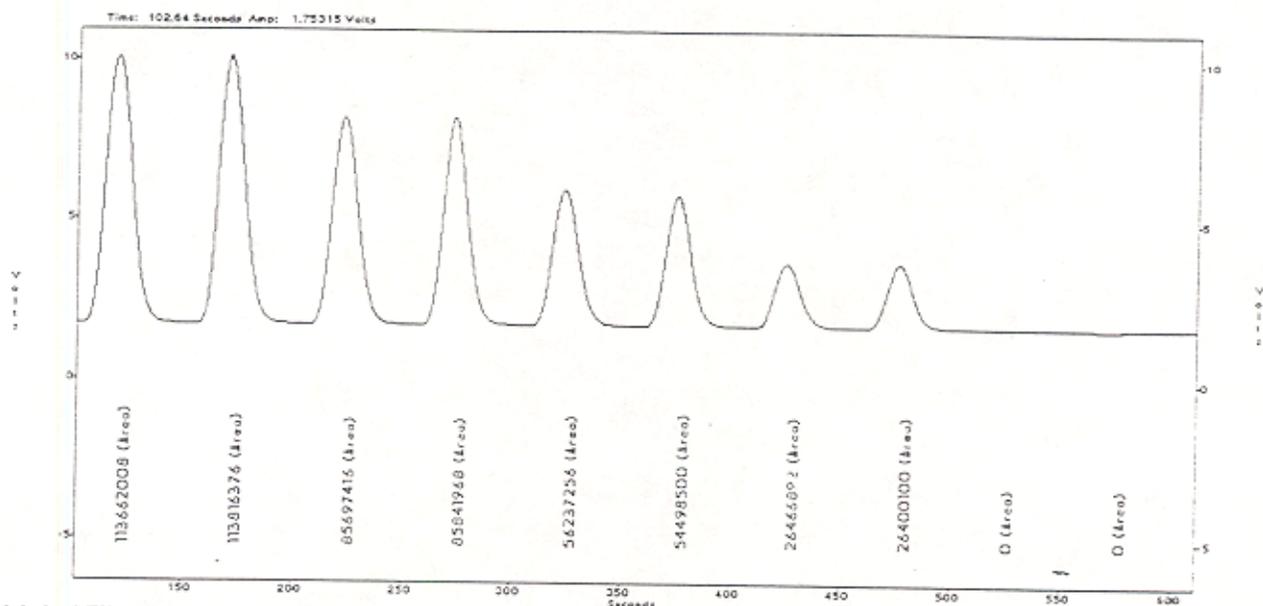
Min Probe in Wash Period: 5 s
Probe in Sample Period: 30 s

Valve Timing:

Load Time: 0.0 s
Load Period: 20 s
Inject Period: 30 s

11.3. SUPPORT DATA FOR QUIKCHEM 8000

Calibration Data for Total Kjeldahl Nitrogen

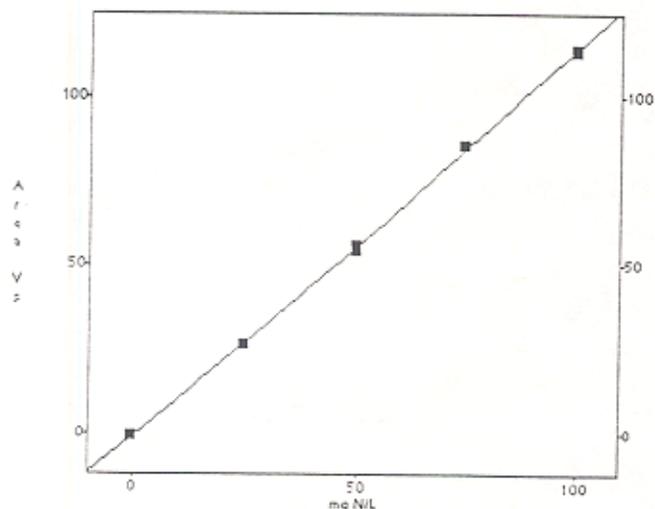


Method File Name: 961203e1.fdt

Acq. Date: 03 December 1996

Calibration Graph and Statistics

Level	Area	mg N/l.	Determined	Replicate %RSD	% residual
1	113739192	100.	99.5	0.1	0.5
2	85769696	75.0	76.0	0.1	-1.3
3	55367880	50.0	49.8	2.2	0.4
4	26433496	25.0	24.3	0.2	3.0
5	0	0.0	0.0	0.0	—



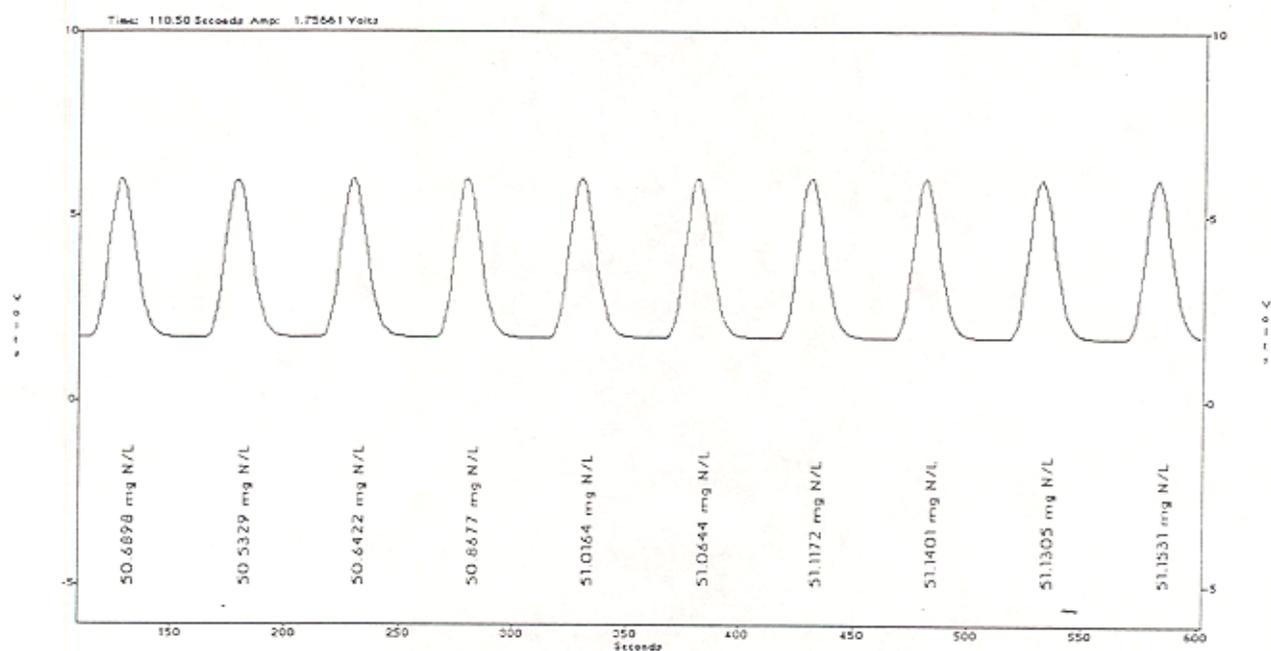
Scaling: None

Weighting: None

2nd Order Poly

Conc = $-3.473e-016 \text{ Area}^2 + 9.108e-007 \text{ Area} + 4.259e-001$

$R^2 = 0.9997$



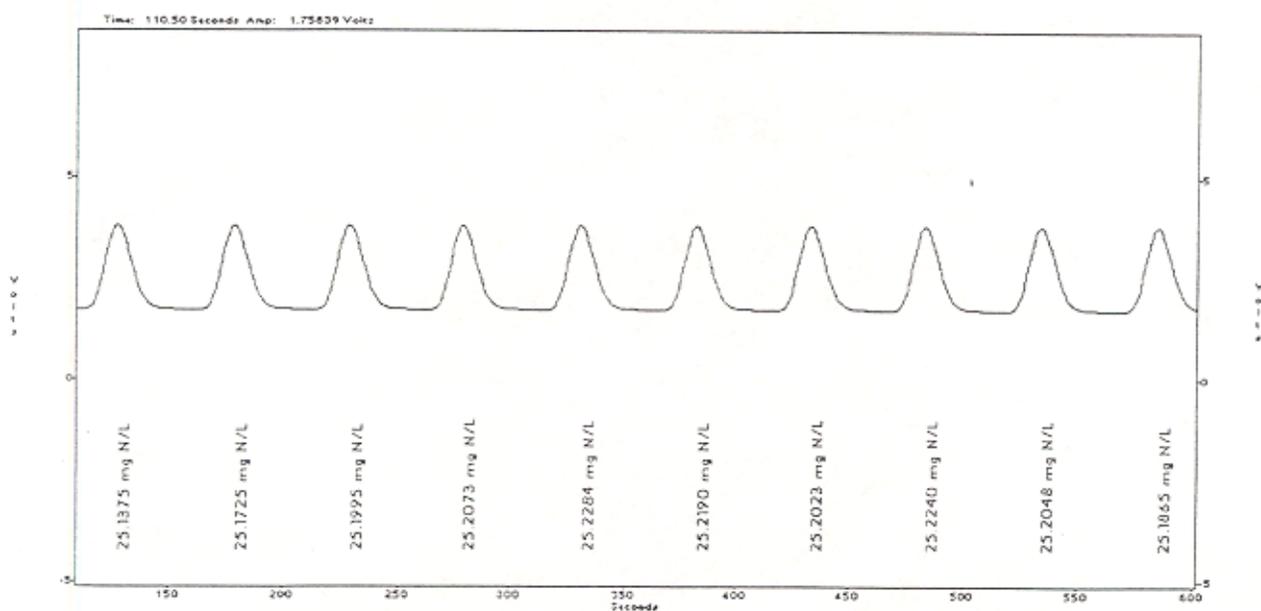
Precision data for total kjeldahl nitrogen using 50.0 mg N/L standard

%RSD = 0.46

Standard Deviation (s) = 0.235, Mean (x) = 50.9 mg/L, Known value = 50.0 mg/L

Data File name 961203p.fdt

Acq. Date: 03 December 1996



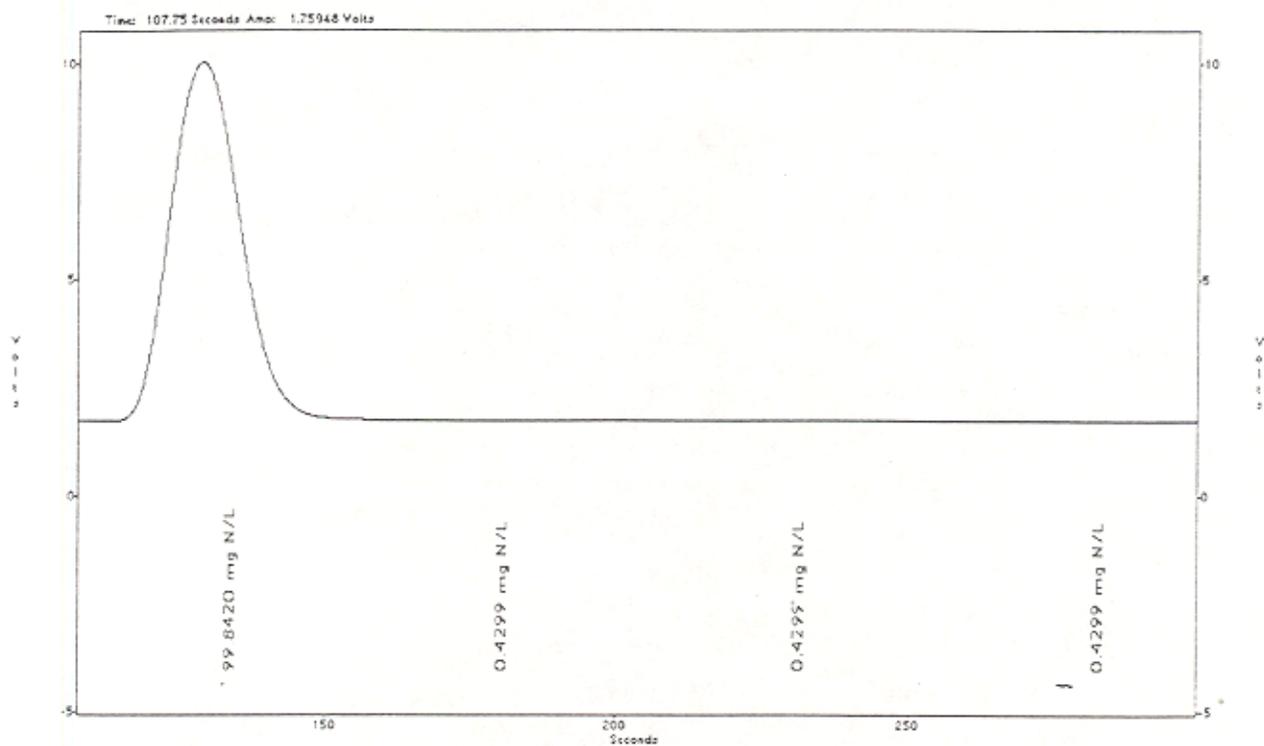
Precision data for total kjeldahl nitrogen using 25.0 mg N/L standard

% RSD = 0.11

Standard Deviation (s) = 0.027, Mean (x) = 25.20, Known value = 25.0 mg/L

Data File name 961203m1.fdt

Acq. Date: 03 December 1996



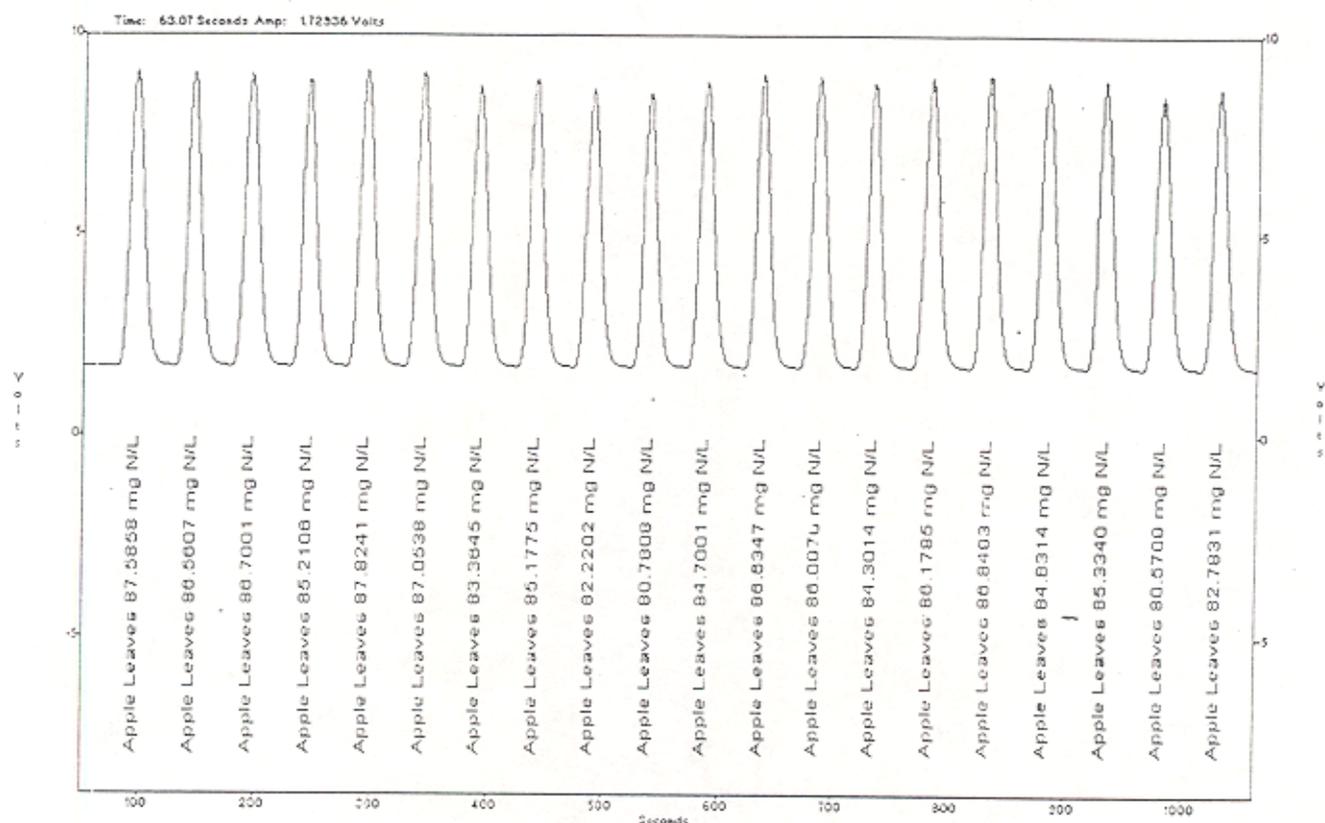
Carryover Study: 100 mg/L standard followed by 3 blanks

Carryover Passed

Data File name 931203r1.fdt

Acq. Date: 03 December 1996

APPLE LEAVES: National Institute of Standards and Technology Certified Standard



Ten digested samples of NIST certified apple leaves, run in duplicate. Each duplicate pair represents a separate weighing and digestion.

Digestion %RSD = 1.40, n = 10

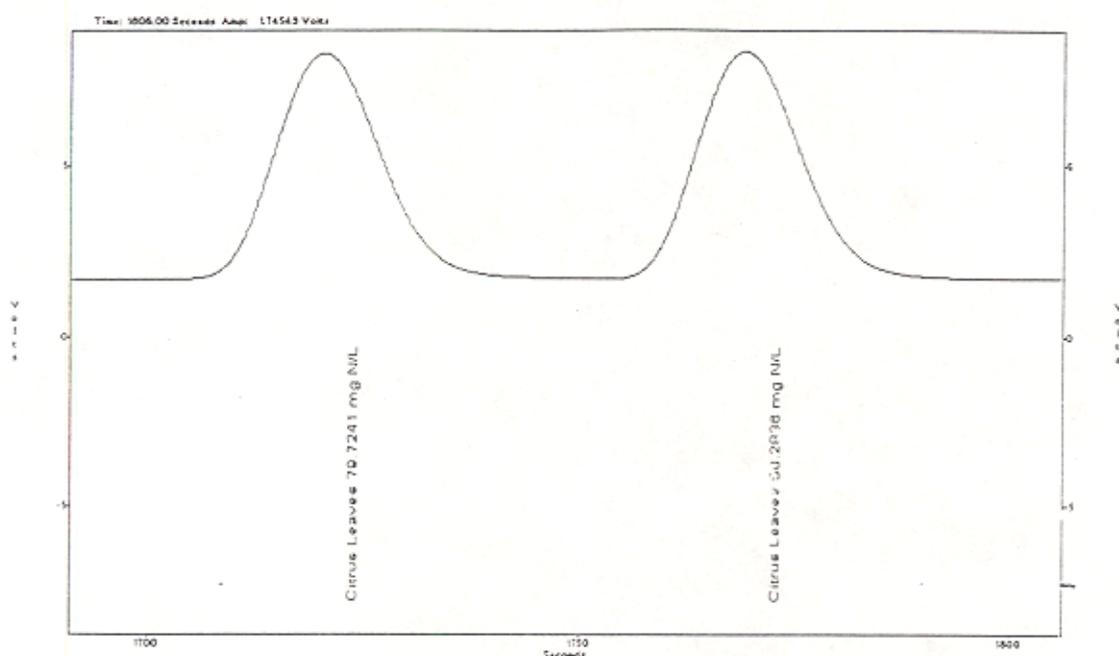
Mean (\bar{x}) = 2.09 % N, Standard Deviation (s) = 0.0293; Known Value = 2.31 % N

Datafile Name: 961205s2.fdt

Acq. date: 5 December 1996

Tube Number	Mean conc. of 2 reps (mg N/L)	Mean conc. of 2 reps (% N)	Recovery (%)
1	87.1	2.05	88.8
2	86.0	2.11	91.5
3	87.4	2.13	92.0
4	84.3	2.08	89.9
5	81.5	2.08	90.0
6	85.7	2.09	90.6
7	85.2	2.10	90.8
8	86.5	2.10	91.1
9	85.1	2.12	91.9
10	81.7	2.04	88.2

CITRUS LEAVES: National Institute of Standards and Technology Certified Standard



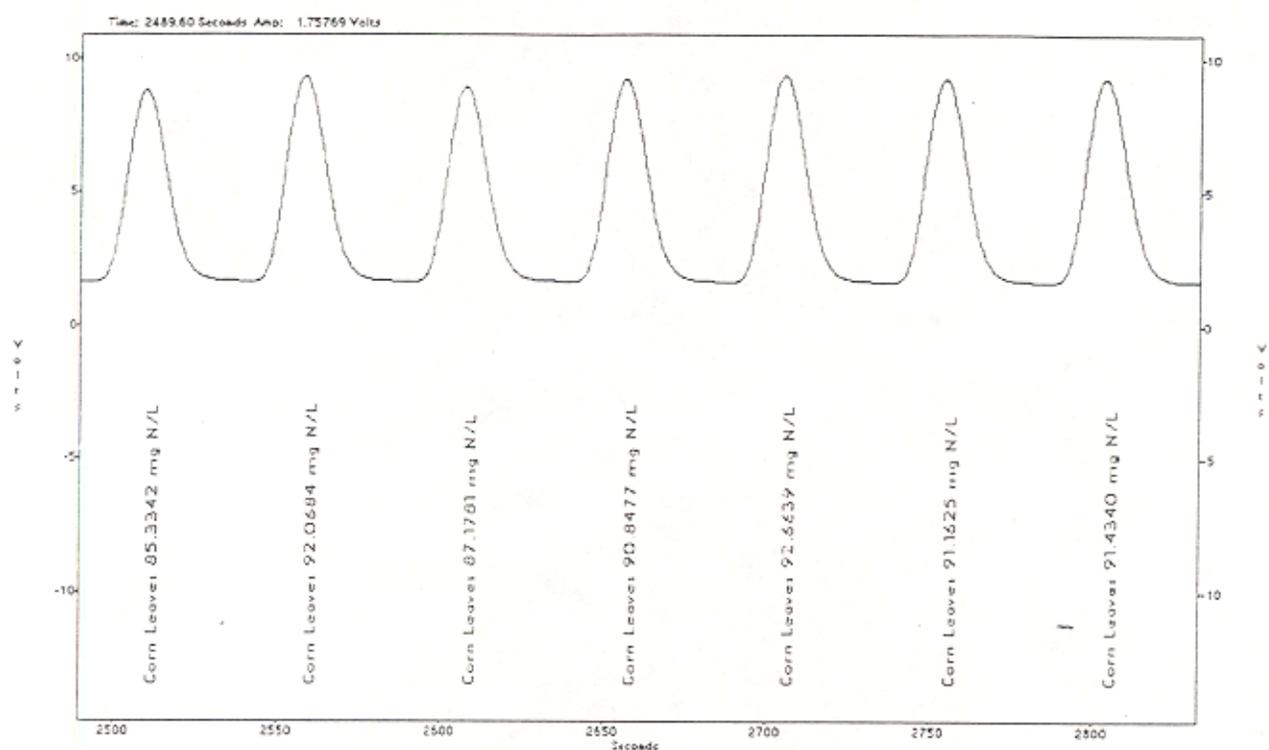
One digested sample of NIST certified citrus leaves, run in duplicate.

Mean (\bar{x}) = 2.63 % N, Known Value = 2.86 % N, Standard Deviation (s) = 0.0129

Datafile Name: 961205s2.fdt

Acq. date: 5 December 1996

CORN LEAVES



Four digested samples of corn leaves, run in duplicate. Each duplicate pair represents a separate weighing and digestion.

Digestion % RSD = 3.03, n = 4

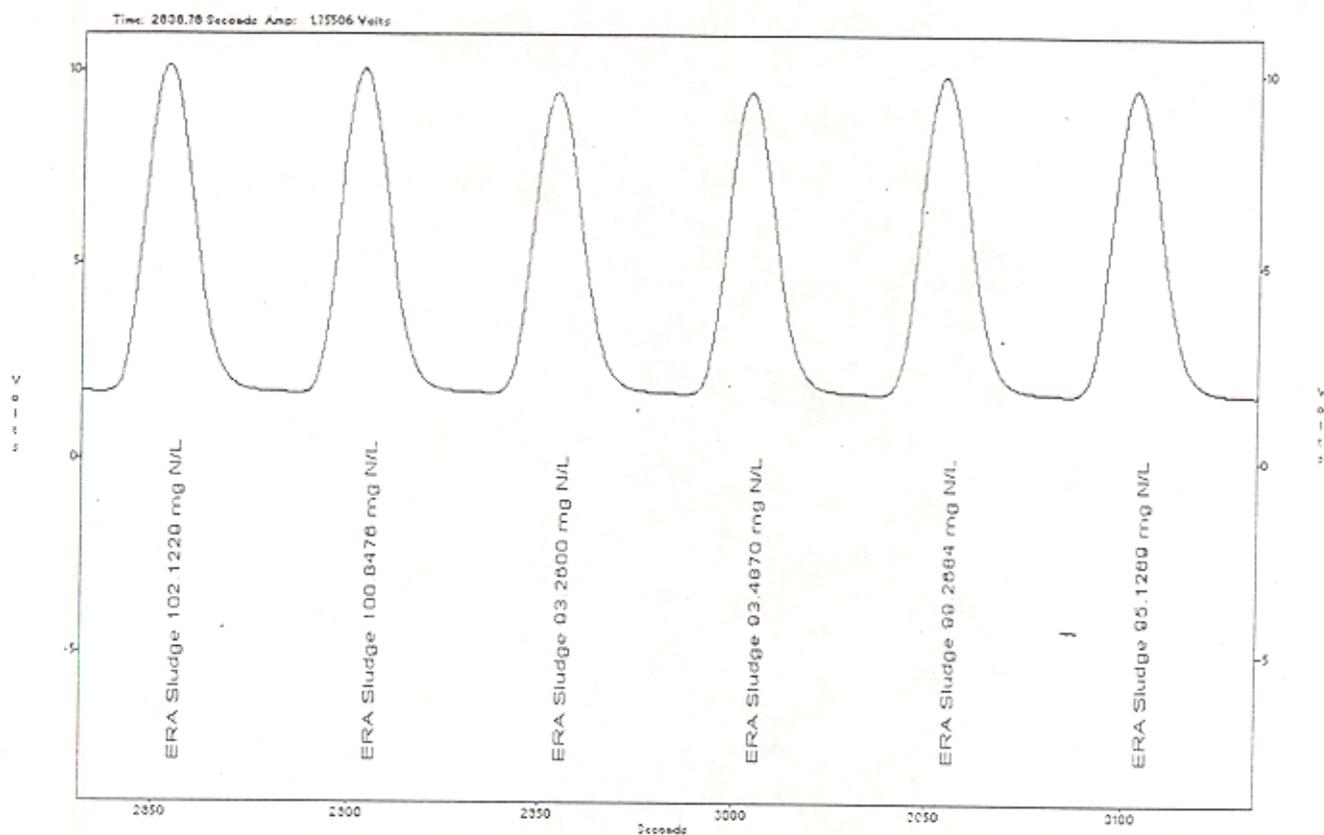
Mean (\bar{x}) = 2.48 % N, Standard Deviation (s) = 0.0754, Known Value = 2.71 % N

Datafile Name: 961205s2.fdt

Acq. date: 5 December 1996

Tube Number	Mean Conc. of 2 reps (mg N/L)	Mean Conc. of 2 reps (% N)	Recovery (%)
1	85.8	2.38	87.7
2	89.6	2.49	91.9
3	91.8	2.54	93.6
4	91.3	2.53	93.5

ERA SLUDGE



Three digested samples of ERA* Sludge, run in duplicate. Each duplicate pair represents a separate weighing and digestion.

Digestion %RSD = 1.41, n = 3

Mean (\bar{x}) = 4.77 % N, Standard Deviation (s) = 0.0673, Known Value = 4.75 % N,

Acceptable range = 3.04 - 6.46 % N

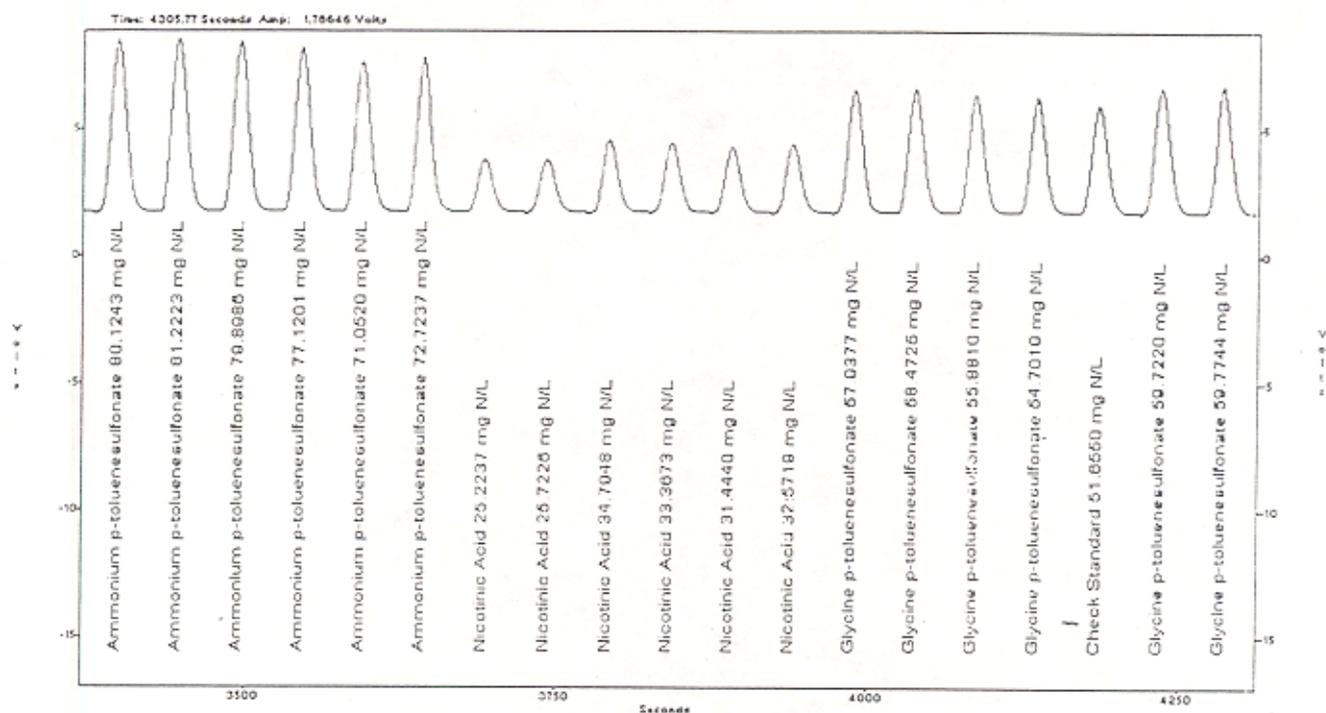
Datafile Name: 961205s2.fdt

Acq. date: 5 December 1996

Tube Number	Mean Conc. of 2 reps (mg N/L)	Mean Conc. of 2 reps (% N)	Within Acceptable Range (Y/N)
1	101.5	4.70	Yes
2	93.4	4.83	Yes
3	97.2	4.77	Yes

* Environmental Resource Associates, Arvada Colorado, 303-431-8454. Catalog no. 545, lot. no. 23016

PRIMARY STANDARDS

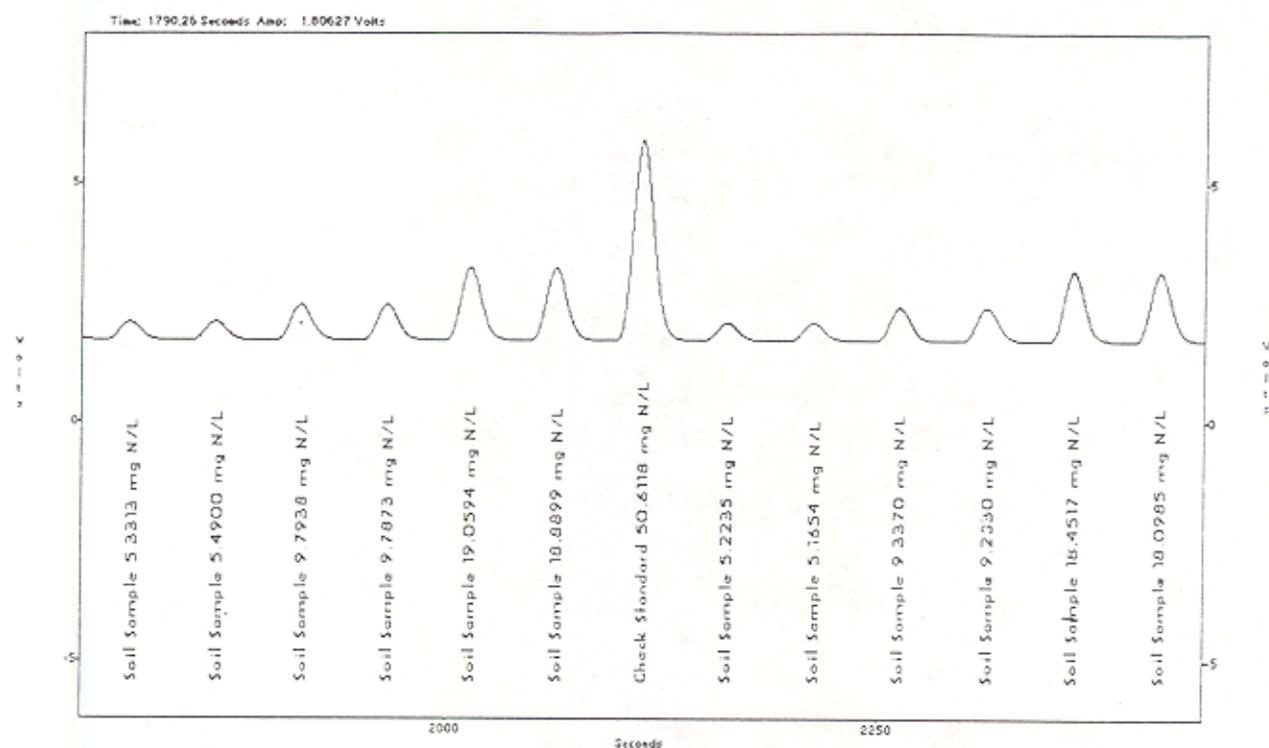


Three sets of digested primary standards, run in duplicate. Each duplicate pair represents a separate weighing and digestion.

Datafile Name: 961205s2.fdt
 Acq. date: 5 December 1996

Primary Standard	Known Value (% N)	Mean (x) (% N)	Standard Deviation (s)	Digestion % RSD, n = 3
Ammonium p-toluenesulfonate	7.40	7.27	0.144	1.98
Nicotinic acid	4.74	1.50	0.234	15.6
Glycine p-toluenesulfonate	5.67	5.56	0.180	3.24

UNKNOWN SOIL SAMPLE



Six unknown soil samples, digested using different starting weights: 0.1, 0.2, and 0.4 g, run in duplicate. Each duplicate pair represents a separate weighing and digestion. Results show a digestion precision of 5.91%. The determined concentration is independent of sample weight from 0.1 to 0.4 g.

Digestion %RSD = 5.91, n = 6

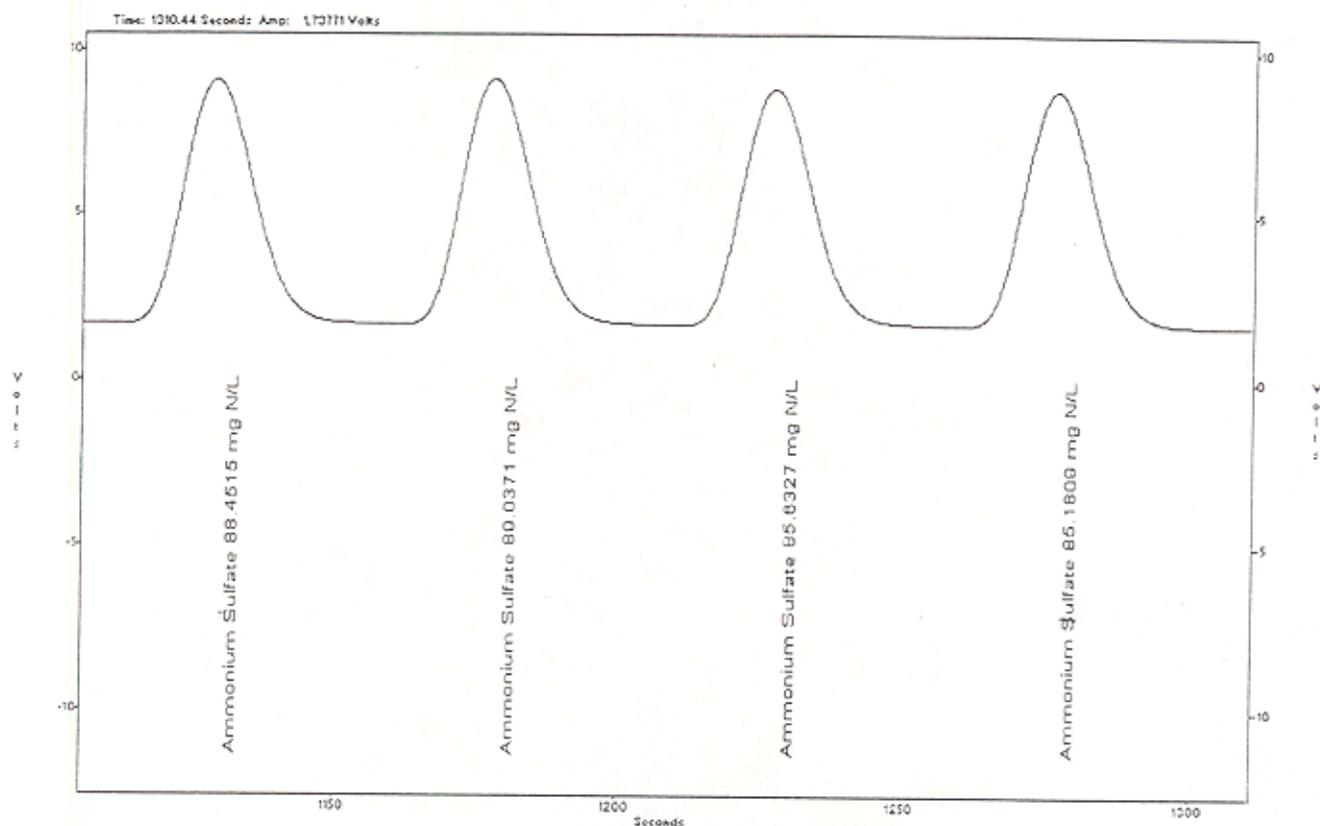
Mean (x) = 0.243 % N, Standard Deviation (s) = 0.014

Datafile Name: 961205s2.fdt

Acq. date: 5 December 1996

Tube Number	Sample Weight (g)	Mean conc. of 2 reps (mg N/L)	Mean conc. of 2 reps (% N)
1	0.1	5.41	0.27
2	0.2	9.79	0.24
3	0.4	18.97	0.24
4	0.1	5.19	0.25
5	0.2	9.31	0.23
6	0.4	18.28	0.23

AMMONIUM SULFATE RECOVERY



Two digested samples of primary standard ammonium sulfate, run in duplicate. Each duplicate pair represents a separate weighing and digestion.

Digestion % RSD = 0.97, n = 2

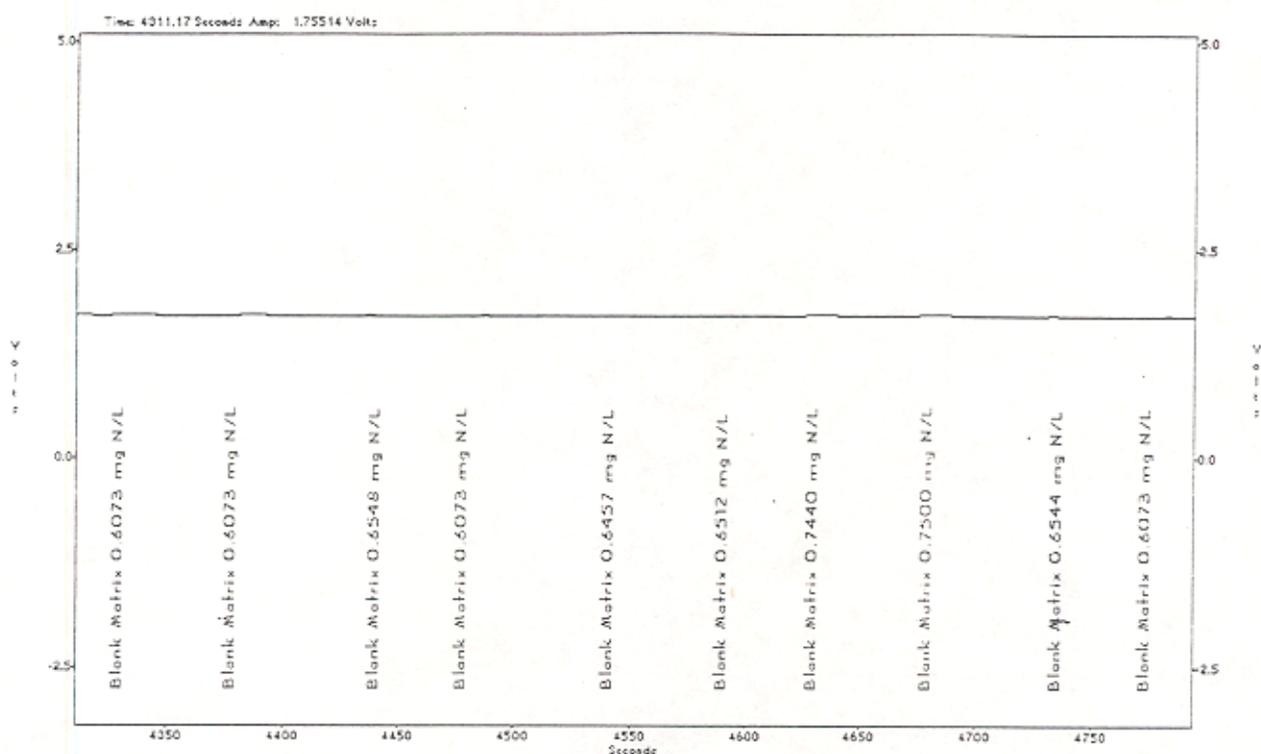
Mean (x) = 20.59 % N, Standard Deviation (s) = 0.199, Known Value = 21.26 % N

Datafile Name: 961205s2.fdt

Acq. date: 5 December 1996

Tube Number	Mean Conc. of 2 reps (mg N/L)	Mean Conc. of 2 reps (% N)	Recovery (%)
1	88.74	20.45	96.2
2	85.41	20.73	97.5

DIGESTION BLANKS



Five digestion blanks containing the weighing paper, copper sulfate, potassium sulfate, and sulfuric acid only, digested and run in duplicate. Each duplicate pair represents a separate digestion. All results are less than 1 mg N/L.

Datafile Name: 961205s2.fdt

Acq. date: 5 December 1996