

**EPA-600/4-75-015**  
**November 1975**

**Environmental Monitoring Series**

# **NATIONAL EUTROPHICATION SURVEY**

## **Data Acquisition and Laboratory Analysis System for Lake Samples**



**Environmental Monitoring and Support Laboratory  
Office of Research and Development  
U.S. Environmental Protection Agency  
Las Vegas, Nevada 89114**

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EPA-600/4-75-015  
November 1975

NATIONAL EUTROPHICATION SURVEY

Data Acquisition And Laboratory Analysis System For Lake Samples

by

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ROAP 25ALD  
Program Element No. 1BA029

U.S. ENVIRONMENTAL PROTECTION AGENCY  
OFFICE OF RESEARCH AND DEVELOPMENT  
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Effective June 29, 1975, the National Environmental Research Center-Las Vegas (NERC-LV) was designated the Environmental Monitoring and Support Laboratory-Las Vegas (EMSL-LV). This Laboratory is one of three Environmental Monitoring and Support Laboratories of the Office of Monitoring and Technical Support in the U.S. Environmental Protection Agency's Office of Research and Development.

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## INTRODUCTION

On December 15, 1971, the U.S. Environmental Protection Agency (EPA) established the National Eutrophication Survey (NES) to identify and evaluate water bodies in the continental United States which have actual or potential eutrophication problems resulting from phosphorus effluent from municipal sewage treatment plants. The specific objective of the survey is to determine if phosphorus removal from municipal wastes would be expected to benefit the trophic state of the receiving waters.

Part of the survey consists of characterizing the present trophic state of the lakes of interest. In situ measurements of depth, temperature, dissolved oxygen, conductivity, turbidity, and pH are made at selected points on each water body. A Secchi disk reading is also obtained.

Discrete samples are obtained at different depths at each sampling station. A portion of each sample is filtered in the field, producing a filtered and an unfiltered portion for each sample. These samples are preserved with mercuric chloride and shipped to the Environmental Monitoring and Support Laboratory in Las Vegas, Nevada, (EMSL-LV). There they are analyzed for total phosphate, dissolved phosphate, ammonia nitrogen, nitrite plus nitrate nitrogen, Kjeldahl nitrogen, and alkalinity. A special sample is also obtained for chlorophyll-a analysis which is performed in the field. Other special samples are obtained for phytoplankton counts which are performed under contract. All data are entered in the Water Quality Control Information System (STORET) for future retrieval and evaluation.

## GENERAL SYSTEM DESCRIPTION

Operationally, the processing of samples and data is complex. Several processes occur simultaneously and are sometimes mutually dependent. Successful processing of samples requires procedures that extend across operational or organizational functions. Therefore, description of the system is based on processing functions rather than operational units. The functional operations are developed as follows:

1. Sample Control Function
2. Laboratory Analysis Function
3. Data Management Function

## SAMPLE CONTROL

Sample control personnel record, prepare, and distribute for appropriate analysis all samples received. Each incoming sample is identified by a unique laboratory number assigned by the field personnel. Filtered and unfiltered

portions are identified by color code. For new sampling stations, location description information is transcribed from the field data form to a station description coding record.

Longitude and latitude are determined from U.S. Geological Survey quadrangle maps and demographic codes are determined. This coding record is forwarded for keypunching and entry of a STORET station description record. A four-part sample control form is generated in Sample Control and accompanies the samples for analysis. A copy of the field data form, station description coding form and sample control card is presented in Appendix A.

## LABORATORY ANALYSIS

Samples are analyzed using three Technicon Industrial System Autoanalyzers. One system is utilized for Kjeldahl nitrogen on the unfiltered portion of the sample, one for dissolved and total phosphorus using both portions of the sample, and one for dissolved ammonia nitrogen, nitrite plus nitrate nitrogen, and alkalinity. Specific procedures for these analyses are presented in Appendix B.

## DATA MANAGEMENT

The STORET system is utilized for the storage and retrieval of NES data. No attempt is made here to describe the STORET system. Input format is described on the sample control form, station description coding form, and the field data form. A list of STORET parameter codes is shown in Appendix C as is a sample of STORET output report. The integration of these elements is shown in detail in Figure 2.

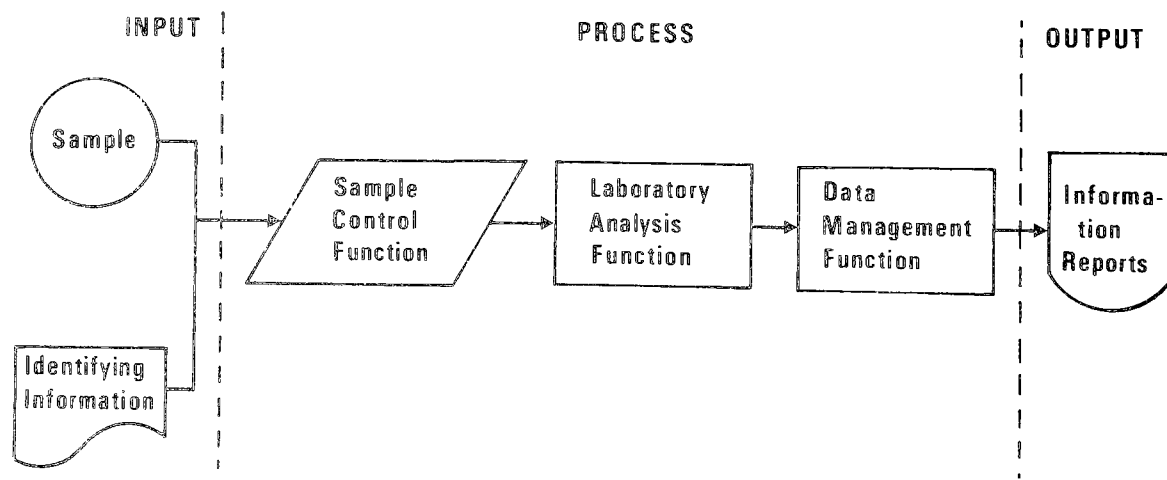


Figure 1. Data acquisition and analysis processing functions



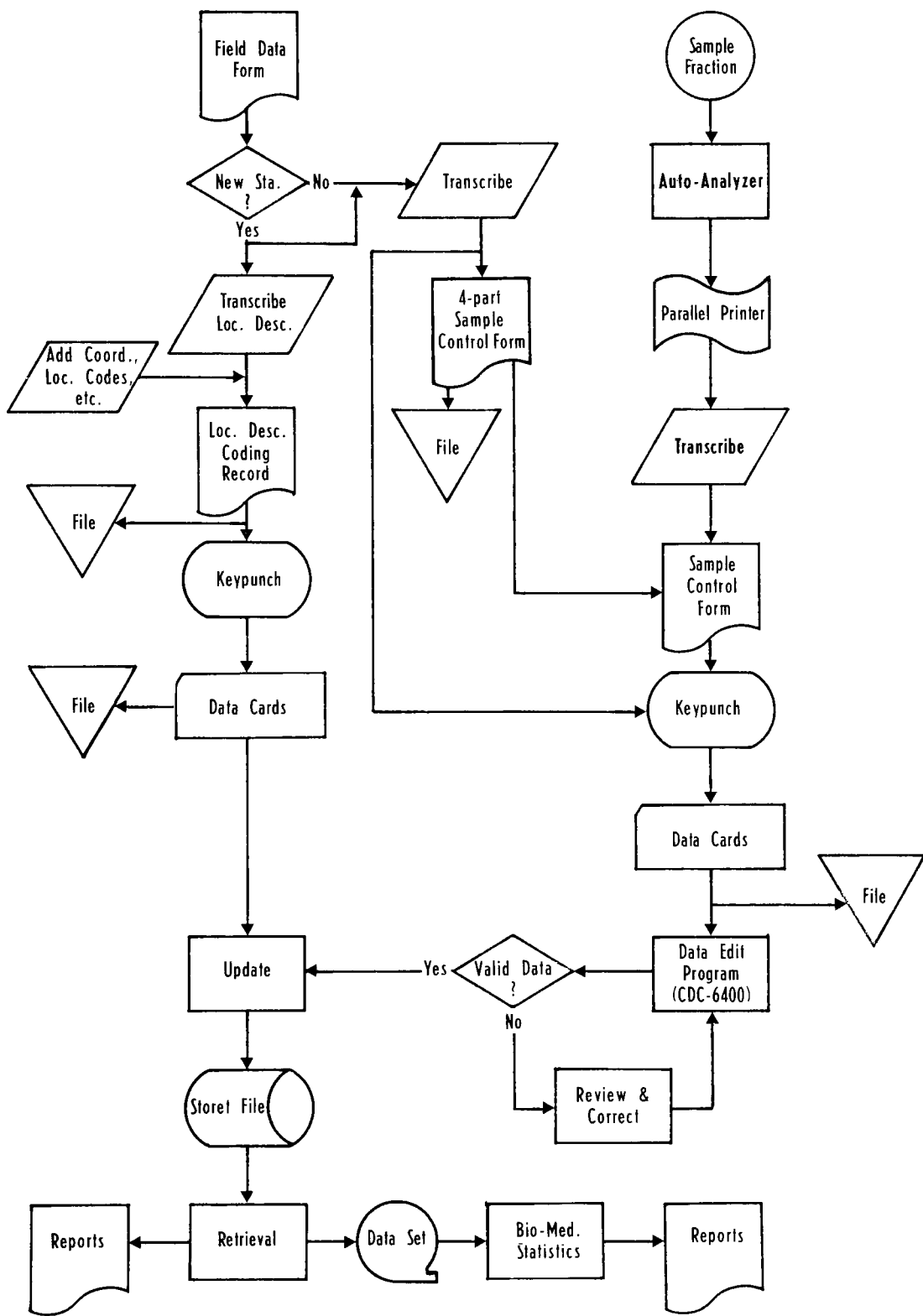


Figure 2. National Eutrophication Survey - Data Flow

## APPENDIX A. DATA FORMS

# NATIONAL EUTROPHICATION STUDY

STATION DESCRIPTION (MUST BE COMPLETED FOR NEW STATIONS)

STATION CODE 4      9

TOTAL DEPTH OF WATER (FT) 30

NEW STATION ☐

STATE

COUNTY

LAKE NAME

---

WORD DESCRIPTION

---



---



---



---



---



---

DATA CODING RECORD

CARD	LOCATION	YR	MO	DY	HR	MN	24	25	TOTAL DEPTH, FT	CHLOR-A, $\mu$ G/L	SECCHI, IN
D											

DEPTH, FT	SAMPLE #	LIGHT, %	COND, $\mu$ MHO	TURB, %TRANS	TEMP, °C	DO, MG/L	PH, STD UNITS		BOD, #
x x x x	x x x x x x	x x x . x	x x x x	x x x x	x x . x	x x . x	x x . x		
1	0								
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
AIR READING									

COMMENTS

---



---



---

ADDITIONAL SAMPLES TAKEN

SEDIMENT CORE ☐

PAP 5 GAL CONTAINER ☐

QUAL CONTROL REPLICATES ☐

OTHER (SPECIFY)

INITIALS

LIMNOL

TECH

HELICOPTER

NO

Figure A-1. Field data form

၆

[illegible]

Figure A-2. Station description coding record

Speediset® Moore Business Forms, Inc. m  
MCP Moore Business Forms, Inc. Patents 3,016,308 3,459,877

SAMPLE IDENTIFICATION														SAMPLE NUMBER			
CARD		LOCATION				COLLECTION				DEPTH							
						YR	MO	DY	HR	MN							
'	D	'	,	'		,					,						
ANALYSIS RESULTS																	
00665 PHOS T MG/L		00671 PHOS D MG/L		00610 NH3 - N MG/L		00630 NO2+NO3 MG/L		00410 TALK MG/L		00625 TKN MG L							
X	X	.	X	X	X	X	X	.	X	X	X	X	X	X	X		
COMMENTS														SEQ			

(P) M

Figure A-3. Sample control card

## APPENDIX B. AUTOANALYZER PROCEDURES

### TECHNICON TRIPLE CHANNEL AAI AUTOANALYZER

#### General

Channel A -- In the nitrate plus nitrite procedure, the nitrates are reduced to nitrites using a copper-cadmium reductor column. The nitrites are reacted with sulfanilamide to form the diazo compound. The diazo compound is coupled under acidic conditions with N-1-naphthylethylenediamine dihydrochloride to form a reddish purple azo dye. The azo dye intensity, which is proportional to the nitrate concentration, is then measured.

Channel B -- The method for total alkalinity uses methyl orange as an indicator because its pH range is in the same range as the equivalence point for total alkalinity. The color transition is extended by the use of a buffer and alkalinity is read as being proportional to the loss of color.

Channel C -- The method for ammonia uses the Berthelot reaction in which the intensity of an indophenol blue color formed by the reaction of ammonia with alkaline phenol hypochlorite, is measured. Sodium nitroprusside is used to intensify the blue color.

#### Operating Notes

<u>Channel</u>	<u>Parameter</u>	<u>Range of Operation</u>
A	nitrate + nitrite	0.02 — 1.0 mg/liter N
B	alkalinity	10 — 200 mg/liter CaCO <sub>3</sub>
C	ammonia	0.02 — 1.0 mg/liter N
50 samples/1 hour		3:1 sample/wash ratio

Standards -- Prepare standards fresh when need is indicated; keep refrigerated when not in use.

Set 1 - Nitrate plus carbonate plus small amount of ammonia standard (full-scale standard only) to allow channel C printer to be started at the proper time

Set 2 - Ammonia

Spiking solution -- The spiking solution will give a theoretical increase of 0.1 mg/liter for nitrate and ammonia and 20 mg/liter for alkalinity when added at a concentration of 0.1 ml per 10 ml of sample. This solution is prepared weekly and kept refrigerated.

Reagents -- The color reagent for nitrate should be clear and colorless; refrigerate it. While washing the system, isolate the ammonium chloride reagent line to avoid contamination of ammonia system. Alkalinity requires a single reagent - methyl orange. On aging, the Brij tends to deteriorate and must be replenished when debubbling becomes erratic.

Method control measures -- Condition of cadmium reductor column is checked daily by running a freshly prepared 0.5-mg/liter nitrite standard. Peak height should be 50% (replace column if peak height exceeds 65% when instrument is calibrated to read 100% with the regular standard containing 1.0-mg/liter nitrate).

Condition of ammonia standard is checked daily by running a freshly prepared 0.5-mg/liter ammonia standard. Peak height should be 50% when instrument is calibrated in the regular manner.

A log is kept of the "Standard Calibration Potentiometer" settings for each day. When there is a significant change in this setting the cause should be determined. General trends in this setting are also useful for diagnosis of problems.

A maintenance log is kept along with the "Standard Calibration" settings to enable correlation of these two factors. Items to be recorded are reagent changes, standard changes, lamp replacement, etc.

Quality control measures -- A record is kept on results for replicate and spiked samples. This record is made available to the Quality Assurance Branch. Every twentieth sample is run in duplicate and is also spiked and the spike is run in duplicate.

Routine operation -- The first run of each day consists of a series of ammonia, nitrate, and alkalinity standards. The 0.5-mg/liter ammonia and 0.5-mg/liter nitrite standards are also included in this run. The full-scale standards are repeated at the end of the run. The balance of the tray is filled in with regular samples.

All subsequent runs begin with a 100% standard for nitrite plus nitrate and alkalinity followed by a 100% standard for ammonia. The standards are followed by two blanks of deionized water, then a series of regular samples. The run is terminated with both 100% standards and then allowed to print three times during the final wash to establish a stable baseline. Baseline and standard calibration settings are not to be changed after the beginning of the run.

Data handling -- Recorder charts for each run are examined for proper peak shape, washout, and location of printout point. Digital printer tapes are examined to assure that the proper result is being printed for the corresponding peak. If standards, replicates, and spikes appear to be satisfactory the results are transcribed onto sheets to allow results for each individual lake to be compared.

After all samples for a particular lake are completed and any questionable results have been rechecked, the results are entered on data cards. The completed cards are returned to Sample Control for processing before entry into the STORET system.

General notes -- The cadmium reductor column is removed after flushing following the last run and kept inverted in a beaker of water.

The cadmium column is reinstalled after the pump is started and all extraneous air bubbles are out of the system. A bypass is installed when the column is removed.

The 90° heating bath on the ammonia manifold is unplugged after the last run. Allow warm-up time of 30 minutes before running samples.

Water supply to wash reservoir is shut off after the instrument is shut down. The Ascarite gas trap is to be kept in place on the water supply vessel at all times to prevent contamination by ammonia and carbon dioxide.

Sensitivity -- For nitrate plus nitrite the minimum detectable concentration is 0.002 mg/liter. The standard deviation is 0.002 mg/liter below 0.05 mg/liter and 4% above 0.05 mg/liter.

For alkalinity the minimum detectable concentration is 10 mg/liter. The standard deviation is 10% from 10 to 50 mg/liter, 5% from 50 to 100 mg/liter, and 2.5% above 100 mg/liter.

For ammonia the minimum detectable concentration is 0.002 mg/liter. The standard deviation is 0.002 mg/liter below 0.10 mg/liter and 2% above 0.10 mg/liter.

#### Reagents For Nitrate Plus Nitrite In Water

##### Ammonium chloride --

Ammonium chloride ( $\text{NH}_4\text{Cl}$ ): 10 g  
Alkaline water: dilute to 1000 ml  
Brij-35\* (30% wt/vol solution): 0.5 ml

Dissolve 10 g of ammonium chloride in alkaline water and dilute to 1 liter. Add 0.5 ml of Brij-35 per liter. Alkaline water is prepared by adding sufficient ammonium hydroxide to distilled water to attain a pH of 8.5.

##### Color reagent ---

Sulfanilamide ( $\text{C}_6\text{H}_8\text{N}_2\text{O}_2\text{S}$ ): 20 g  
Concentrated phosphoric acid ( $\text{H}_3\text{PO}_4$ ): 200 ml  
N-1-naphthylethylenediamine dihydrochloride ( $\text{C}_{12}\text{H}_{14}\text{N}_2 \cdot 2\text{HCl}$ ): 1.0 g  
Distilled, deionized water: dilute to 2000 ml  
Brij-35\* (30% wt/vol solution): 1.0 ml

To prepare this color reagent, add 200 ml of concentrated phosphoric acid and 20 g of sulfanilamide to approximately 1500 ml of distilled, deionized

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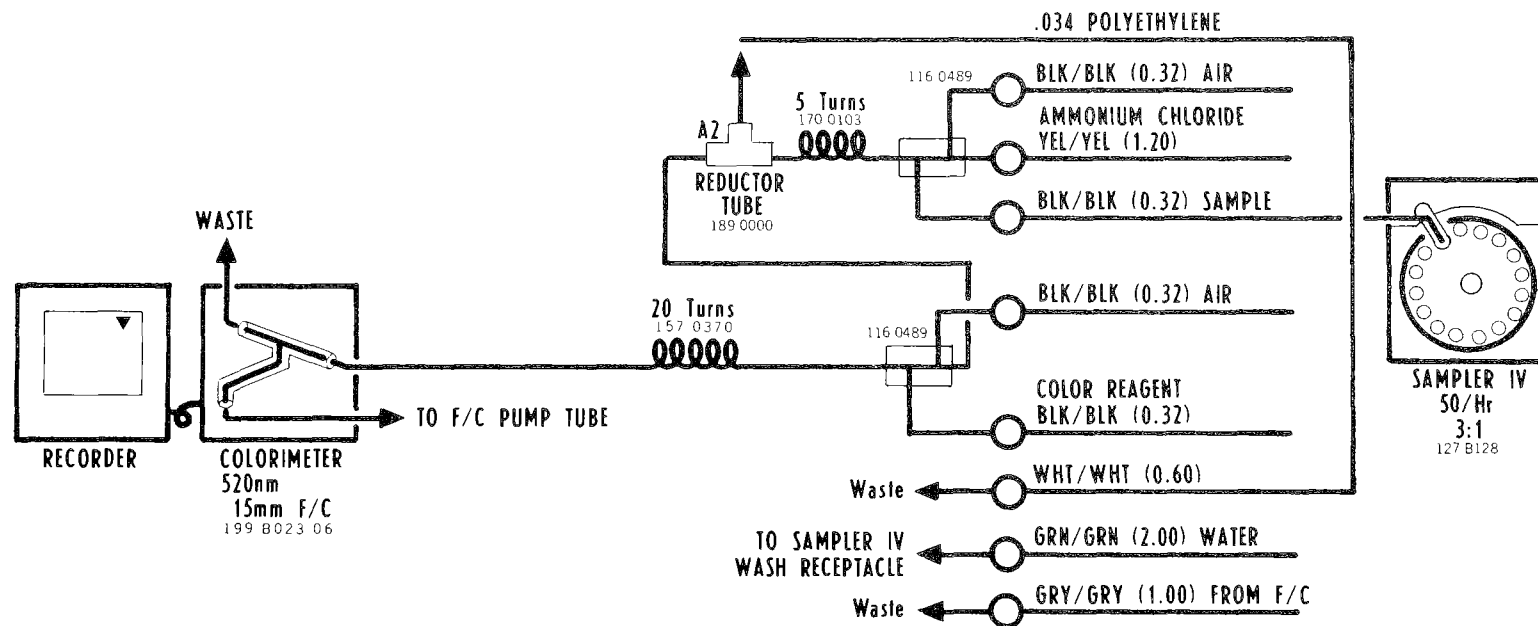
\* Manufactured by Technicon Industrial Systems, Tarrytown, New York 10591



# NITRATE AND NITRITE IN WATER AND WASTE WATER AAI

(RANGE: 0—1.0 ppm N)

MANIFOLD No. 116-D049-01\*



NOTE: Figures in parentheses represent flow rates in ml/min.

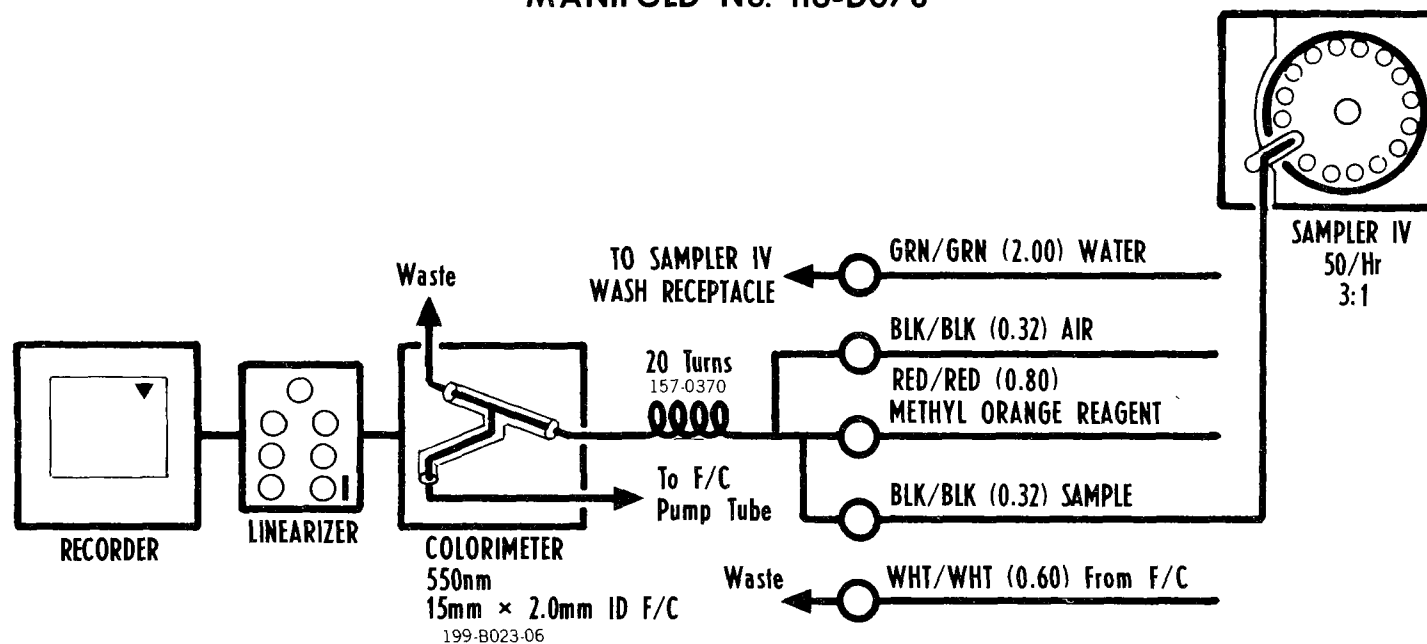
\* TECHNICON INDUSTRIAL SYSTEMS / TARRYTOWN, NEW YORK 10591

Figure B-1. Nitrate and nitrite in water and waste water

# METHYL ORANGE ALKALINITY

(RANGE: 0–200 ppm  $\text{CaCO}_3$ )

MANIFOLD No. 116-D078\*



Linearizer can be incorporated in this system.

NOTE: Figures in parentheses represent flow rates in ml/min.

\* TECHNICON INDUSTRIAL SYSTEMS / TARRYTOWN, NEW YORK 10591

Figure B-2. Methyl orange alkalinity

water. Dissolve completely (heat if necessary). Add 1.0 g N-1-naphthylethylene-diamine dihydrochloride and dissolve. Dilute to 2 liters with distilled, de-ionized water. Add 1.0 ml of Brij-35. Place in brown plastic bottle and refrigerate.

#### Reagents For Methyl Orange Alkalinity In Water

##### Buffer, pH 3.1 --

Potassium acid phthalate ( $C_8H_5KO_4$ ): 10.2 g  
0.1N hydrochloric acid (HCl): 175 ml  
Distilled, deionized water

Dissolve 10.2 g of potassium acid phthalate in 500 ml of distilled water. Add 175 ml of 0.1N hydrochloric acid (8.3 ml hydrochloric acid, sp. gr. 1.19, per liter of distilled, deionized water) and dilute to 1 liter with distilled, deionized water. The pH of this solution should be 3.1. If necessary, adjust with 0.1N hydrochloric acid.

##### Methyl orange (0.05%) --

Methyl orange ( $C_{14}H_{14}N_3NaO_3S$ ): 0.5 g  
Distilled, deionized water

Dissolve 0.5 g methyl orange in 500 ml of distilled, deionized water and dilute to 1 liter. Filter this solution through glass wool if necessary.

##### Working buffer indicator --

Methyl orange (0.05%): 85 ml  
Buffer, pH 3.1: 250 ml  
Brij-35 (30% wt/vol solution): 0.5 ml  
Distilled, deionized water

Add 85 ml of 0.5% methyl orange solution to 250 ml of buffer and dilute to 1 liter with distilled, deionized water. Add 0.5 ml Brij-35 per liter.

#### Ammonia Reagents

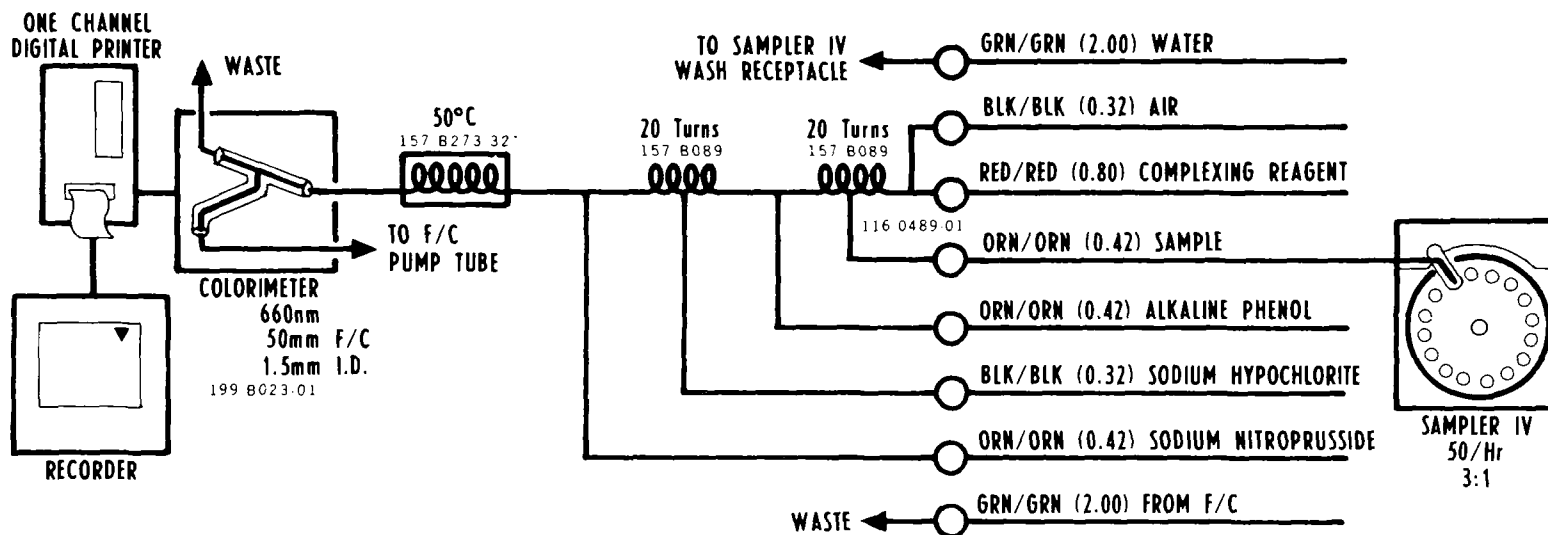
##### Complexing reagent --

Potassium sodium tartrate ( $KNaC_4H_4O_6 \cdot 2H_2O$ ): 33 g  
Sodium citrate ( $HOC(COONa)(CH_2COONa)_2 \cdot 2H_2O$ ): 24 g  
Distilled water

Dissolve 33 g of potassium sodium tartrate and 24 g of sodium citrate in 950 ml of distilled water. Adjust the pH of this solution to 5.0 with concentrated sulfuric acid. Dilute to 1 liter with distilled water. Add 0.5 ml of Brij-35.

# LOW LEVEL AMMONIA IN FRESH AND ESTUARINE WATER

(RANGE 0—0.5 ppm N)  
MANIFOLD No. 116-D082\*



\* Proportional control needed for heating bath.

NOTE: Figures in parentheses represents flow rates in ml/min.

\* TECHNICON INDUSTRIAL SYSTEMS / TARRYTOWN, NEW YORK 10591

Figure B-3. Low level ammonia in fresh and estuarine water

### Alkaline phenol --

Phenol ( $C_6H_5OH$ ): 83 g  
Sodium hydroxide, 20% w/v (NaOH): 180 ml  
Distilled water

Using a 1-liter Erlenmeyer flask, dissolve 83 g of phenol in 50 ml of distilled water. Cautiously add, while cooling under tap water, in small increments with agitation, 180 ml of 20% NaOH. Dilute to 1 liter with distilled water.

Sodium hypochlorite (stock) -- Any good commercially available household bleach having 5.25% available chlorine may be used.

Sodium hypochlorite (working) -- Dilute 200 ml of stock sodium hypochlorite to 1 liter with water.

### Sodium nitroprusside --

Sodium nitroprusside ( $Na_2Fe(CN)_5NO \cdot 2H_2O$ ): 0.5 g  
Distilled water

## AUTOMATED PHOSPHORUS ANALYSIS-TECHNICON AAI AUTOANALYZER

The total phosphorus method utilizes an initial persulfate digestion in a pressure cooker, followed by the formation and reading of intensity of the blue-colored complex formed when ascorbic acid reacts with an antimony-phosphomolybdate complex. The color is proportional to the phosphorus concentration.

### Operating Notes

General information -- The manifold is set up for orthophosphate; 0-0.5 mg/liter phosphorus; 60 samples per hour; 6:1 sample to wash ratio. All samples and standards are autoclaved with persulfate digestion reagent prior to analysis. Results are reported as dissolved phosphorus for filtered samples and as total phosphorus for unfiltered samples.

Standards -- The standards are preserved with mercuric chloride at a concentration of 40 mg per liter, stored at room temperatures and prepared fresh from stock solution monthly.

Spiking solution -- The spiking solution is calculated to give an increase of 0.1 mg/liter phosphorus when added at a concentration of 50  $\lambda$  to 10 ml of sample. This solution is added with a repeating microliter dispenser.

Reagents -- The digestion reagent is prepared fresh daily. The molybdate working reagent is usable for up to 48 hours. The NaCl-Levor IV\* (diluent water) reagent is stable for extended periods.

\*Manufactured by Technicon Industrial Systems, Tarrytown, New York 10591

Method control and quality control -- A record is kept of the "Standard Calibration Potentiometer" settings for each day. When there is a significant change in this setting the cause should be determined. General trends in this setting are also useful for diagnosis of problems.

A maintenance log is kept along with the "Standard Calibration" settings to enable correlation of these two factors. Items to be recorded are reagent changes, standard changes, lamp replacement and other routine maintenance.

A record is kept of results for replicate and spiked samples. This record is made available to the Quality Assurance Branch.

Every twentieth sample is run in duplicate and also spiked, and the spike run in duplicate.

Standard reference samples of known concentration are occasionally run to check overall performance of the system.

Routine operation -- The first run of each day begins and ends with a series of standards and blanks. The balance of the tray is filled with regular samples.

All subsequent runs begin with a 100% standard and a blank followed with regular samples. All runs end with a 100% standard and are allowed to print three times in the final wash to establish a baseline.

Data handling -- Recorder charts are examined for peak shape, washout, and location of printout point. Printer tapes are examined to assure that the proper result is being printed for the corresponding peak.

If standards, replicates, and spikes appear to be satisfactory the results are transcribed onto sheets to allow the results for each lake to be compared.

After all samples for a particular lake are completed and any questionable results have been rechecked, the results are entered on data cards which are sent to Sample Control for entry into the STORET system.

Phosphorus digestion procedure -- Ten ml of sample or standard and 0.5 ml of digestion solution are pipetted into a 16-mm by 125-mm screw cap culture tube. The samples are placed in an autoclave and digested at 250° F for thirty minutes. At the end of thirty minutes the autoclave is turned off and allowed to return to room temperature. The samples at this point are ready for phosphorus analysis. The culture tubes are added directly to the sample changer carousels. The sampling arm is inverted on the sampler to allow clearance.

#### Reagents For Phosphorus In Water

##### Sulfuric acid 3.5 N --

Sulfuric acid ( $H_2SO_4$ ), conc.(sp. gr. 1.84): 179 g  
Distilled, deionized water: dilute to 1000 ml

Add 179 g of concentrated sulfuric acid to 800 ml of distilled, deionized water while stirring slowly. Cool the solution and dilute to 1 liter with distilled, deionized water.

Ammonium molybdate --

Ammonium molybdate  $((\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O})$ : 27 g  
Distilled, deionized water: dilute to 1000 ml

Place 27 g of ammonium molybdate in a glass, 1-liter volumetric flask and dilute to 1 liter with distilled, deionized water. Visually inspect the solution for any trace of molybdate's blue coloration. If any blue color is evident, check the water and glassware for phosphate contamination and make up new reagent solution.

Ascorbic acid --

Ascorbic acid  $(\text{C}_6\text{H}_8\text{O}_6)$ : 18 g  
Distilled, deionized water

Dissolve 18 g of ascorbic acid in 800 ml of distilled, deionized water. Dilute to 1 liter with distilled, deionized water. Refrigerate in brown plastic reagent bottle.

Antimony potassium tartrate --

Antimony potassium tartrate  $(\text{KOOCCCHOHCHOHCOO}(\text{SbO}) \cdot \frac{1}{2}\text{H}_2\text{O})$ : 2.0 g  
Distilled, deionized water

Dissolve 2.0 g of antimony potassium tartrate in 800 ml of distilled, deionized water. Dilute to 1 liter with distilled, deionized water.

Diluent water -- Add 5.0 g of sodium chloride to a 1-liter volumetric flask. Dilute to 1 liter with distilled, deionized water. Add 2.0 ml of Levor IV.

Combined working reagent --

Sulfuric acid  $(\text{H}_2\text{SO}_4)$ , 3.5N: 100 ml  
Ammonium molybdate  $((\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O})$ , 27 g/liter: 30 ml  
Ascorbic acid  $(\text{C}_6\text{H}_8\text{O}_6)$ , 18 g/liter: 60 ml  
Antimony potassium tartrate  
 $(\text{KOOCCCHOHCHOHCOO}(\text{SbO}) \cdot \frac{1}{2}\text{H}_2\text{O})$ , 2.0 g/liter: 10 ml

Digestion solution --

Sulfuric acid  $(\text{H}_2\text{SO}_4)$ , conc., (sp. gr. 1.84): 36.4 g  
Ammonium persulfate  $((\text{NH}_4)_2\text{S}_2\text{O}_8)$ : 32.0 g  
Distilled, deionized water

Add 36.4 g of concentrated sulfuric acid to 125 ml of distilled, deionized water with stirring. Permit the solution to cool, add 32.0 g of ammonium persulfate and dilute to 200 ml with distilled, deionized water.



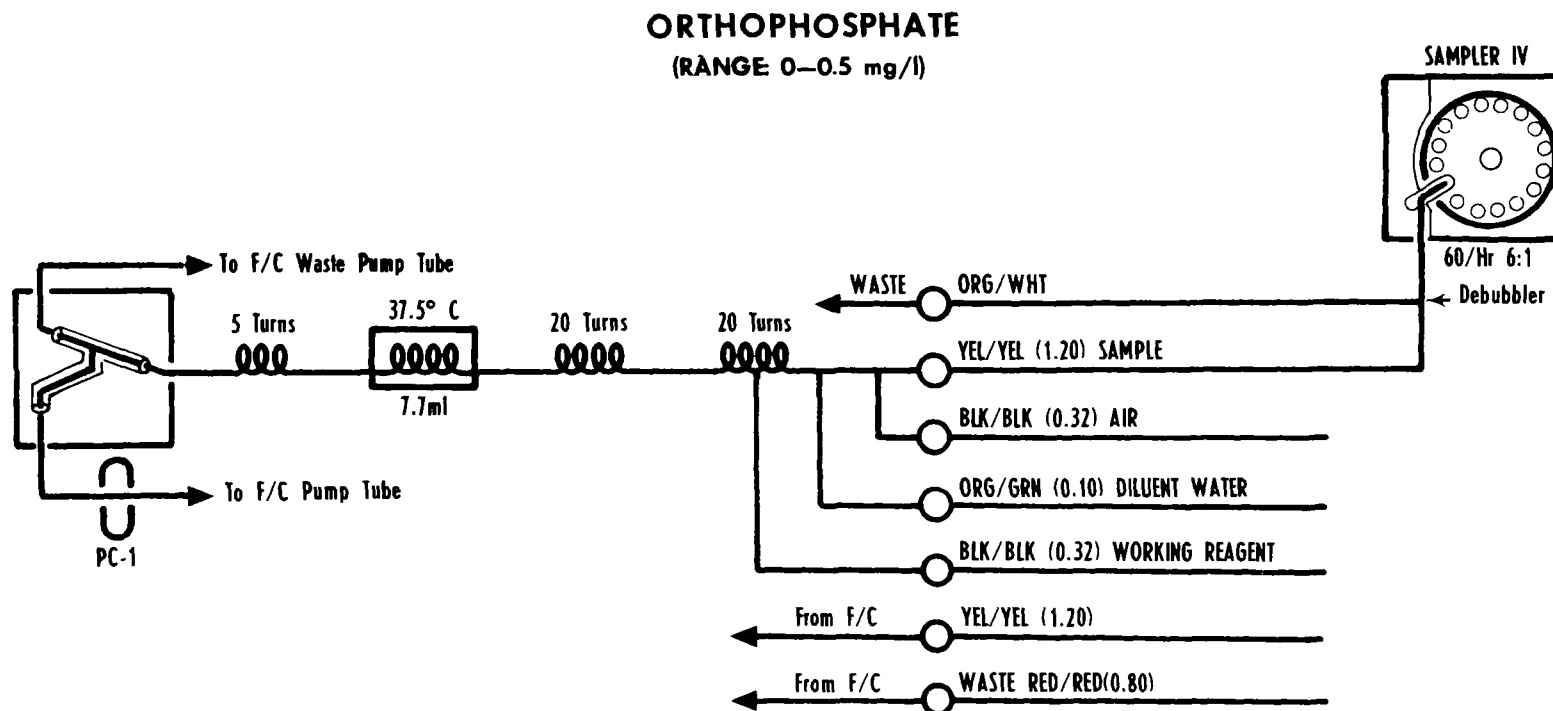


Figure B-4. Orthophosphate

## TOTAL KJELDAHL NITROGEN-TECHNICON AAI AUTOANALYZER

The total Kjeldahl method consists of digesting the organic material with acid in the continuous digester, thereby forming ammonium sulfate. The ammonia is then determined by measuring the color of the indophenol blue complex when ammonia reacts with alkaline phenol hypochlorite. Sodium nitroprusside is added to intensify the color which is proportional to the total organic nitrogen present in the sample.

### Operating Notes

This method involves digestion of the unfiltered sample in a helix type continuous digester followed by colorimetric treatment as in the automated ammonia procedure. The instrument is operated in the range of 0.2 to 5.0 mg/liter nitrogen at 30 samples per hour with a 4:1 sample to wash ratio.

Standards -- The standards are prepared from glutamic acid to be certain the digester is operating properly. 500 mg/liter nitrogen stock solution is prepared by dissolving 5.255 g of glutamic acid in water, adding 1 ml of 4%  $\text{HgCl}_2$  and diluting to 1 liter.

Spiking solution -- The spiking solution is calculated to give an increase of 1.0 mg/liter when added at a concentration of 50  $\lambda$  to 10 ml of sample. The spiking solution contains 200 mg/liter nitrogen and is prepared from the glutamic acid stock solution.

Reagents -- Alkaline phenol: 94 g liquid phenol, 180 ml 20% NaOH, 0.5 ml Brij, dilute to 1 liter. Refrigerate when not in use.

Sodium nitroprusside: 0.5 g/liter, prepared weekly.

Sodium hypochlorite: 200 ml sodium hypochlorite\*/liter.

Sodium hydroxide-tartrate: dissolve 310 g NaOH and 50 g potassium sodium tartrate in 700 ml water, cool, and dilute to 1 liter.

Digestion reagent: dissolve 3.0 g selenium oxide in 50 ml water, add 20 ml perchloric acid, and dilute slowly to 1 liter with conc.  $\text{H}_2\text{SO}_4$ .

Startup procedure -- Reagent lines in water, digest line in air; platens on pumps, pumps off; vacuum pump on, 4 inches of mercury; water aspirator on, cooling water on; distilled water supply on; bath pumps on, start reagents, helix rotor on; turn on digester heaters; invert pulse chamber to fill, bleed A-4 jacket; check manifold bath temperature 55° C; digester temperature - zone 1 360° C, zone 2 320° C; stabilize baseline approximately 20 minutes.

Shutdown procedure -- Heaters off, remove helix cover; reagent tubes in water except digest and NaOH-tart.; when cooled to 150° C, digest line to air; continue sodium hydroxide-tartrate solution 5 min., then water 10 min.; pumps off; break vacuum in waste bottle, vacuum pump off; water aspirator off, cooling water off; distilled water supply off; helix rotor off, replace cover.

\*Any household bleach will suffice.

Emergency shutdown -- Remove helix cover; break vacuum in waste bottle; leave platens clamped on pumps; turn all pump and digester switches off.

Routine operation -- The first run of each day begins and ends with a series of standards and blanks. The balance of the tray is filled with regular samples.

All subsequent runs begin with a 100% standard and a blank followed with regular samples. All runs end with a 100% standard and are allowed to print three times in the final wash to establish a baseline.

Method control and quality control -- A record is kept of the "Standard Calibration Potentiometer" settings for each day. When there is a significant change in this setting the cause should be determined. General trends in this setting are also useful for diagnosis of problems.

A maintenance log is kept along with the "Standard Calibration" settings to enable correlation of these two factors. Items to be recorded are reagent changes, standard changes, lamp replacement and other routine maintenance.

A record is kept of results for replicate and spiked samples. This record is made available to the Quality Assurance Branch.

Every twentieth sample is run in duplicate and is also spiked and the spike run in duplicate.

Data handling -- Recorder charts are examined for peak shape, washout, and location of printout point. Printer tapes are examined to confirm that the proper result is being printed for the corresponding peak.

If standards, replicates, and spikes appear to be satisfactory, the results are transcribed onto sheets to allow the results for each lake to be compared.

After all samples for a particular lake are completed and any questionable results have been received, the results are entered on data cards which are sent to Sample Control for entry into the STORET system.

Sensitivity -- The minimum sensitivity is 0.2 mg/liter. The standard deviation is 0.005 mg/liter below 0.5 mg/liter and 10% above 0.5 mg/liter.

## 20



**NOTE:** Figures in parentheses represent flow rates in ml/min.

Figure B-5. Total nitrogen (KJELDAHL)

## APPENDIX C. STORET PARAMETER CODES

LAKE EUTROPHICATION STUDY				
STORET PARAMETER CODES				
DATA GENERATED	PARAMETER CODE	COMPUTER PRINT-OUT ABBREVIATION	DECIMAL POINT LOCATION	PARAMETER DESCRIPTION
Field Analysis	00008	LAB IDENT NO.	xxxxxx	Sample number
" "	00077	TRANSP. SECCHI INCHES	xxxxxx	Transparency, Secchi disk (inches)
Field Contact Sensor	00400	PH SU	xxxxxx.x	PH (standard units)
"	00010	WATER TEMP.CENT	xxxxxx.x	Temp., water (° C)
"	00300	DO MG/L	xxxxxx.x	Oxygen, dissolved (mg/l)
"	00094	CNDUCTVY AT 25° C MICROMHO	xxxxxx	Conductivity, Field (micromhos)
"	00074	TURB. TRANS. %	xxxxxx	Turbidity, transmissometer (percent transmission)
"	00031	INCDT LT REMNING PERCENT	xxxxx.x	Light, incident, percent remaining at certain depth
Field Lab.	32217	CHLRPHYL-A MG/L	xxxxx.xx	Chlorophyll-A fluorometric method (mg/l)
NERC-LV	00608	NH3-N DISS MG/L	xxxxx.xx	Nitrogen, ammonia
	00630	NO2 & NO3 N-TOTAL MG/L	xxxxx.xx	Nitrite + Nitrate one determ (mg/l as N)
	00665	PHOS-TOT MG/L P	xxxx.xxx	Phosphorus, total, wet method (mg/l as P)
	00671	PHOS-DISS ORTHO MG/L	xxxx.xxx	Phosphorus, diss., wet method (mg/l as P)
	00410	TALK CAC03 MG/L	xxxxxx	Alkalinity, total (mg/l as CaCO <sub>3</sub> )
	00625	TKN MG/L	xxxxx.xx	Kjeldahl nitrogen (mg/l)

**TECHNICAL REPORT DATA**  
(Please read Instructions on the reverse before completing)

1. REPORT NO. EPA-600/4-75-015		2.		3. RECIPIENT'S ACCESSION NO.	
4. TITLE AND SUBTITLE NATIONAL EUTROPHICATION SURVEY Data Acquisition And Laboratory Analysis System For Lake Samples				5. REPORT DATE November 1975	
				6. PERFORMING ORGANIZATION CODE	
7. AUTHOR(S) J. W. Mullins, R. N. Snelling, D. D. Moden and R. G. Seals				8. PERFORMING ORGANIZATION REPORT NO.	
9. PERFORMING ORGANIZATION NAME AND ADDRESS Environmental Monitoring and Support Laboratory Office of Research and Development U.S. Environmental Protection Agency Las Vegas, Nevada 89114				10. PROGRAM ELEMENT NO. 1BA029	
				11. CONTRACT/GRANT NO.	
12. SPONSORING AGENCY NAME AND ADDRESS  Same as 9.				13. TYPE OF REPORT AND PERIOD COVERED 1972-1975	
				14. SPONSORING AGENCY CODE EPA - ORD - Health and Ecological Effects	
15. SUPPLEMENTARY NOTES  Contact J. W. Mullins, (702) 736-2969, extension 326					
16. ABSTRACT A system for data acquisition and laboratory analysis for the National Eutrophication Survey is presented. A description is given of the field measurement and data recording, the sample control process, the laboratory analysis and data management. Flow charts and data forms are given for the field and sample control functions. A description with drawings is given of the Technicon Autoanalyzers used for the laboratory analysis of the samples.					
17. KEY WORDS AND DOCUMENT ANALYSIS					
a. DESCRIPTORS		b. IDENTIFIERS/OPEN ENDED TERMS		c. COSATI Field/Group	
Data acquisition Chemical analysis Water analysis Lakes Phosphorus Nitrogen Laboratory equipment		Eutrophication Autoanalyzers		07B 08H 14B	
18. DISTRIBUTION STATEMENT  RELEASE TO THE PUBLIC		19. SECURITY CLASS (This Report) UNCLASSIFIED		21. NO. OF PAGES 26	
		20. SECURITY CLASS (This page) UNCLASSIFIED		22. PRICE	