ON-LINE ANALYSIS OF NITRATE WITH ULTRAVIOLET ABSORBANCE SPECTROMETRY

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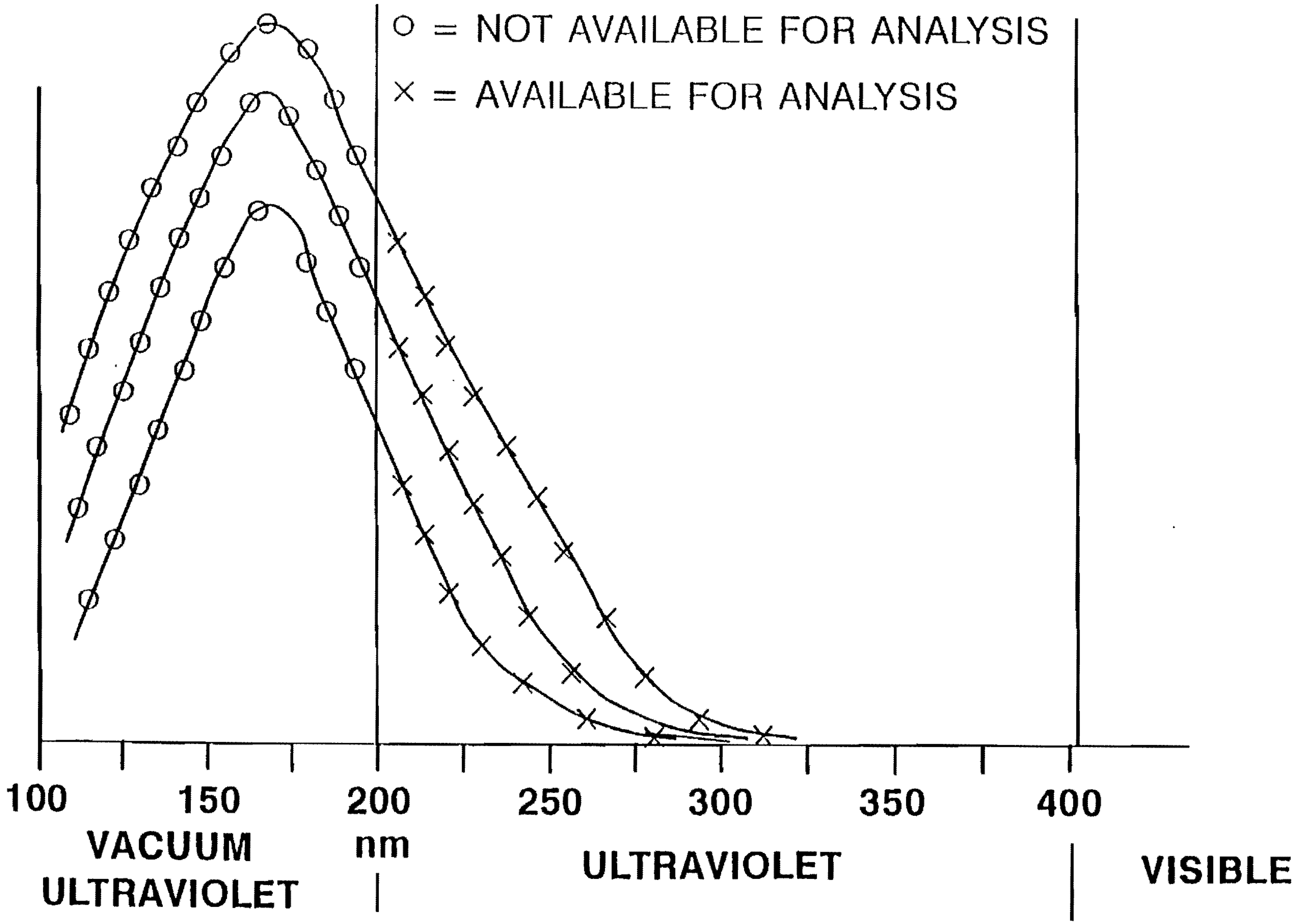
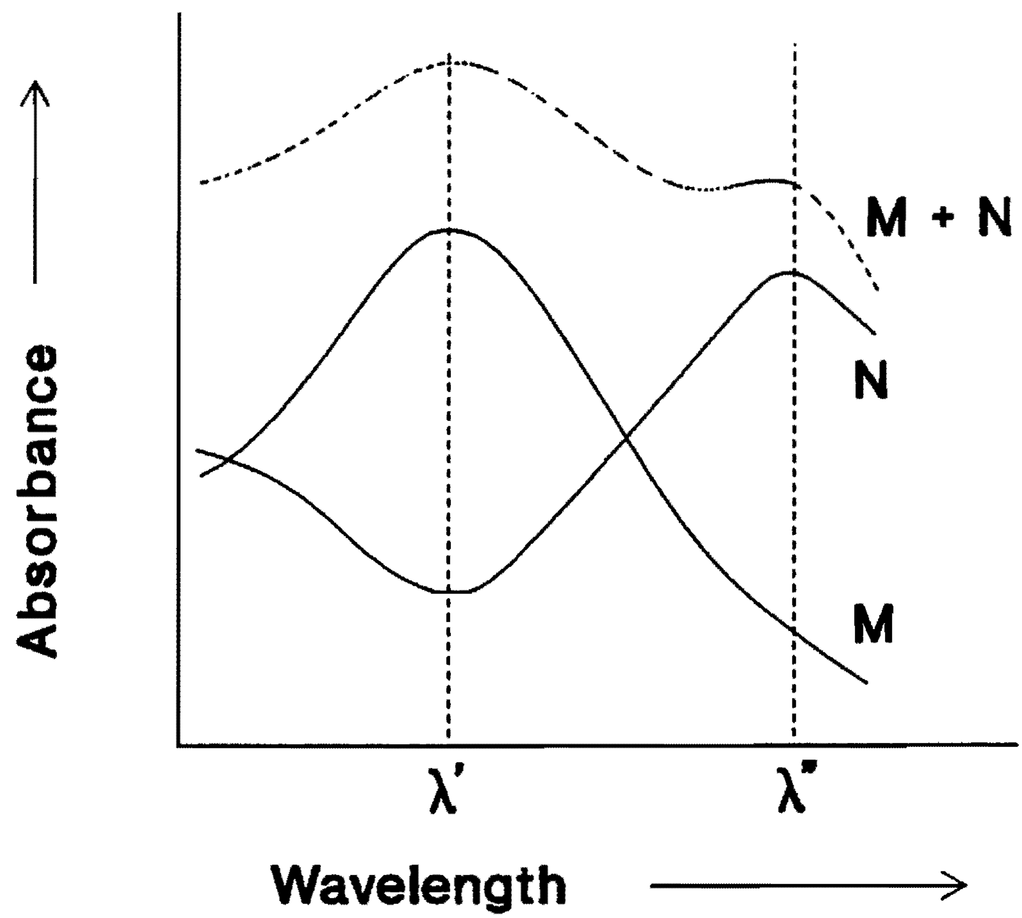
ON-LINE NITRATE ANALYSIS
BACKGROUND

A. BASIC CONCEPTS

1. CHARACTERISTICS OF NITRATE ABSORBANCE SPECTRA
2. MULTIVARIATE ANALYSIS TECHNIQUES
3. UV-6100 SYSTEM DEVELOPMENT
4. IS NOT
    a. AUTOMATED WET CHEMISTRY
    b. ION SELECTIVE ELECTRODE



THE PEAK AND A SUBSTANTIAL PORTION OF THE SPECTRUM IS IN THE VACUUM ULTRAVIOLET AND NOT ABLE TO BE DETECTED USING CONVENTIONAL INSTRUMENTS. THE PORTION OF THE SPECTRUM ABOVE 200nm IS AVAILABLE FOR DETECTION AND ANALYSIS.



Absorption Spectrum of a Two Component Mixture

BB2525

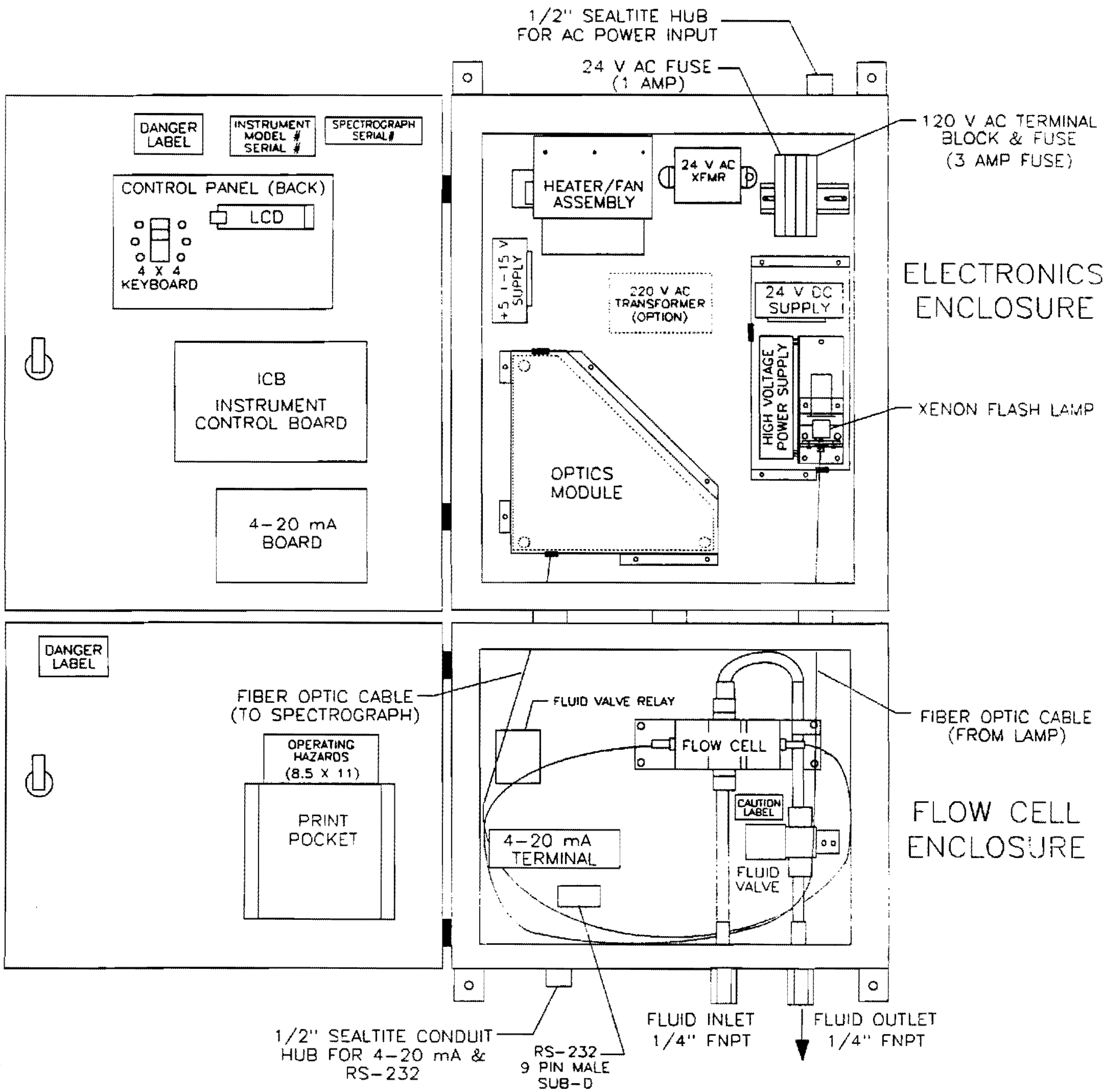


Figure 2. UV-6100 Analyzer Layout

D#CHSANLY1
REV. 2193

ON-LINE NITRATE ANALYSIS
BACKGROUND

B. OCONOMOWOC TEST SITE OBJECTIVES

1. EVALUATE THE ABILITY OF ULTRAVIOLET ABSORBANCE SPECTROMETRY TO ANALYZE NITRATE IN WASTEWATER SECONDARY EFFLUENT WITHOUT THE USE OF REAGENTS.

2. EVALUATE LONG TERM OPERATION OF THE UV-6100 PROCESS ANALYZER IN A WASTEWATER TREATMENT FACILITY.

3. EVALUATE TECHNIQUES TO MINIMIZE THE USE OF LARGE, SITE SPECIFIC CALIBRATION SETS.

4. EVALUATE O&M REQUIREMENTS FOR THE ANALYZER SYSTEM.

OCONOMOWOC TEST SITE
HISTORY

A. INITIAL PERIOD STARTING 9-29-92

1. INITIAL FOCUS ON MECHANICAL AND O&M ISSUES

   a. SAMPLE LINE AND PUMP

   b. INTEGRITY OF ELECTRONICS ENCLOSURE

   c. SAMPLE INTERVALS

   d. DATA COLLECTION MECHANICS

      1) LOG FORMAT AND PROCEDURES

      2) INTERNAL MEMORY MANAGEMENT

OCONOMOWOC TEST SITE
HISTORY

2. FACTORY CALIBRATION (30 POINT) PRIOR TO INSTALLATION

    a. 0 TO 30 ppm NO_3 (6.77 ppm NO_3-N UPPER LIMIT)

    b. 0 TO 5 ppm Fe RANDOM VARIATION IN NO_3 SOLUTION

    c. EVALUATE DYNAMIC RANGE ISSUES

    d. EVALUATE IMPACT OF BACKGROUND VARIATIONS

OCONOMOWOC TEST SITE
HISTORY

3. DEVELOP AND TEST O&M TECHNIQUES

   a. MEASUREMENT OF INSTRUMENT STABILITY

   b. MEASUREMENT OF WINDOW FOULING

   c. VERIFICATION OF BASELINE CORRECTION (INSTRUMENT STANDARD)

   d. PROBE CLEANING PROCEDURES

      1) MECHANICAL

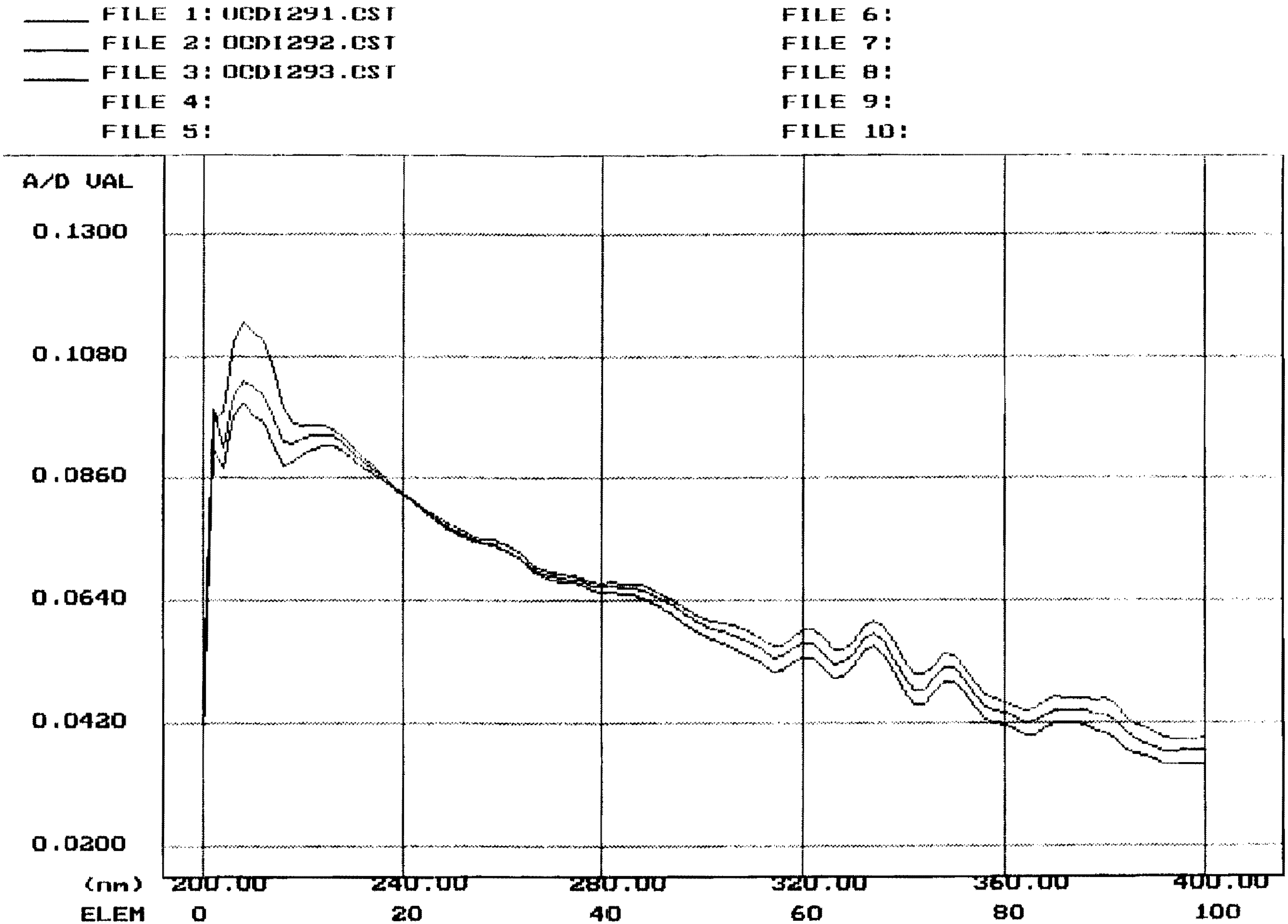
      2) CHEMICAL

   e. MEASUREMENT OF CLEANING EFFECTIVENESS

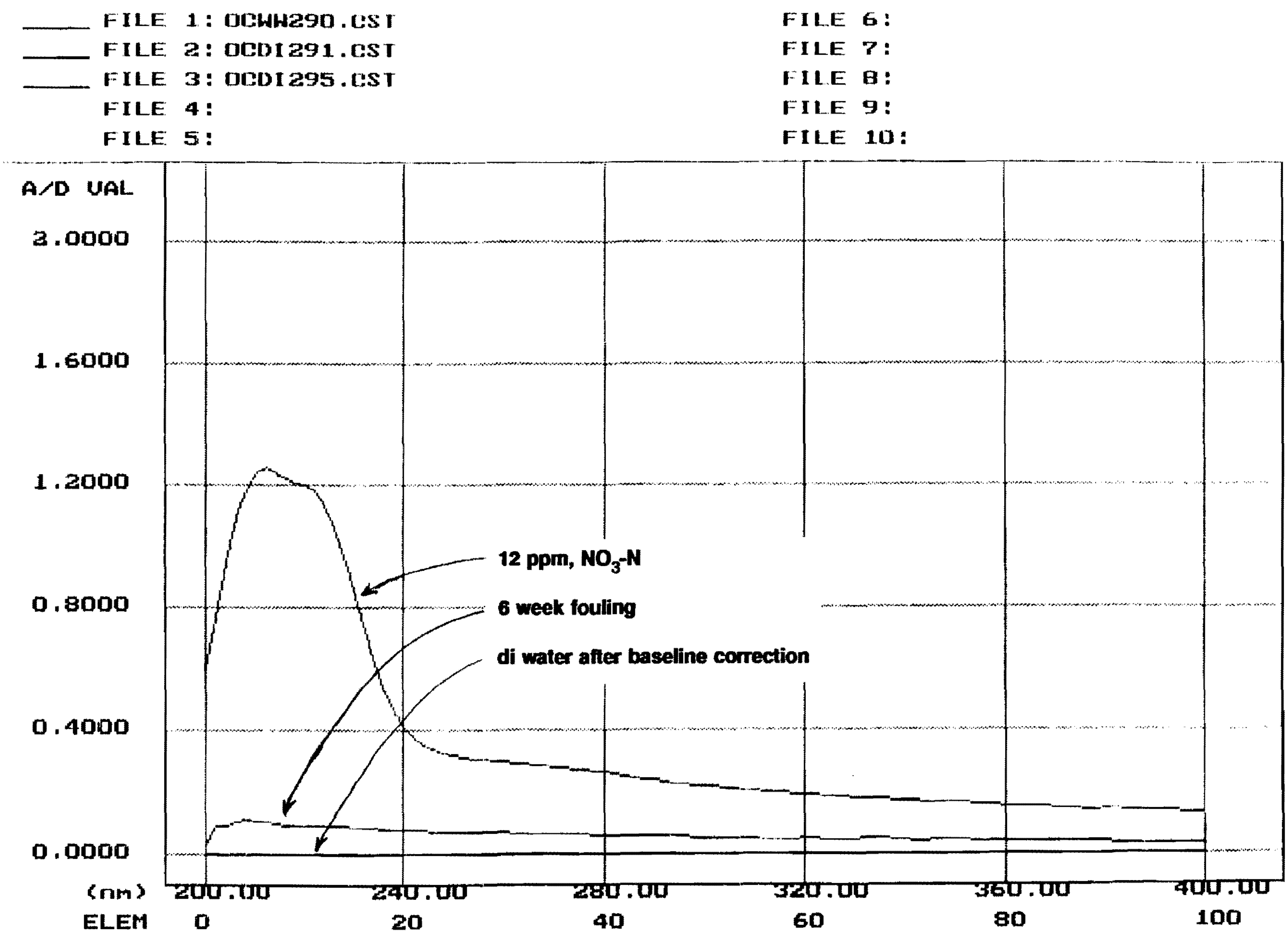
   f. DEVELOPMENT OF TEST PROTOCOL

      1) GRAB SAMPLE HANDLING AND PRESERVATION

      2) LABORATORY ANALYSIS PROCEDURES

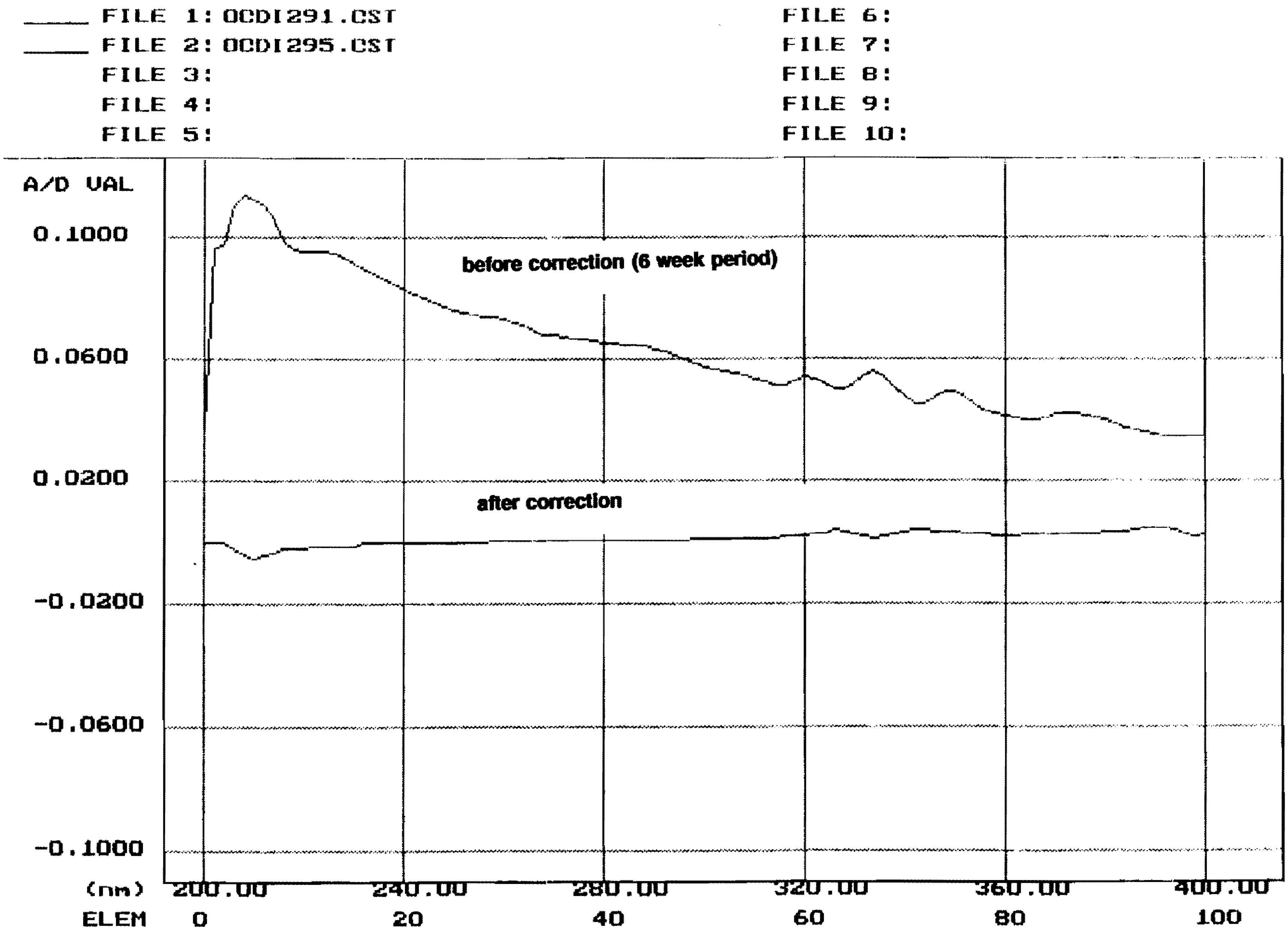


OCONOMOWOC, WI
RELATIVE STABILITY, DI WATER WITH FOULING

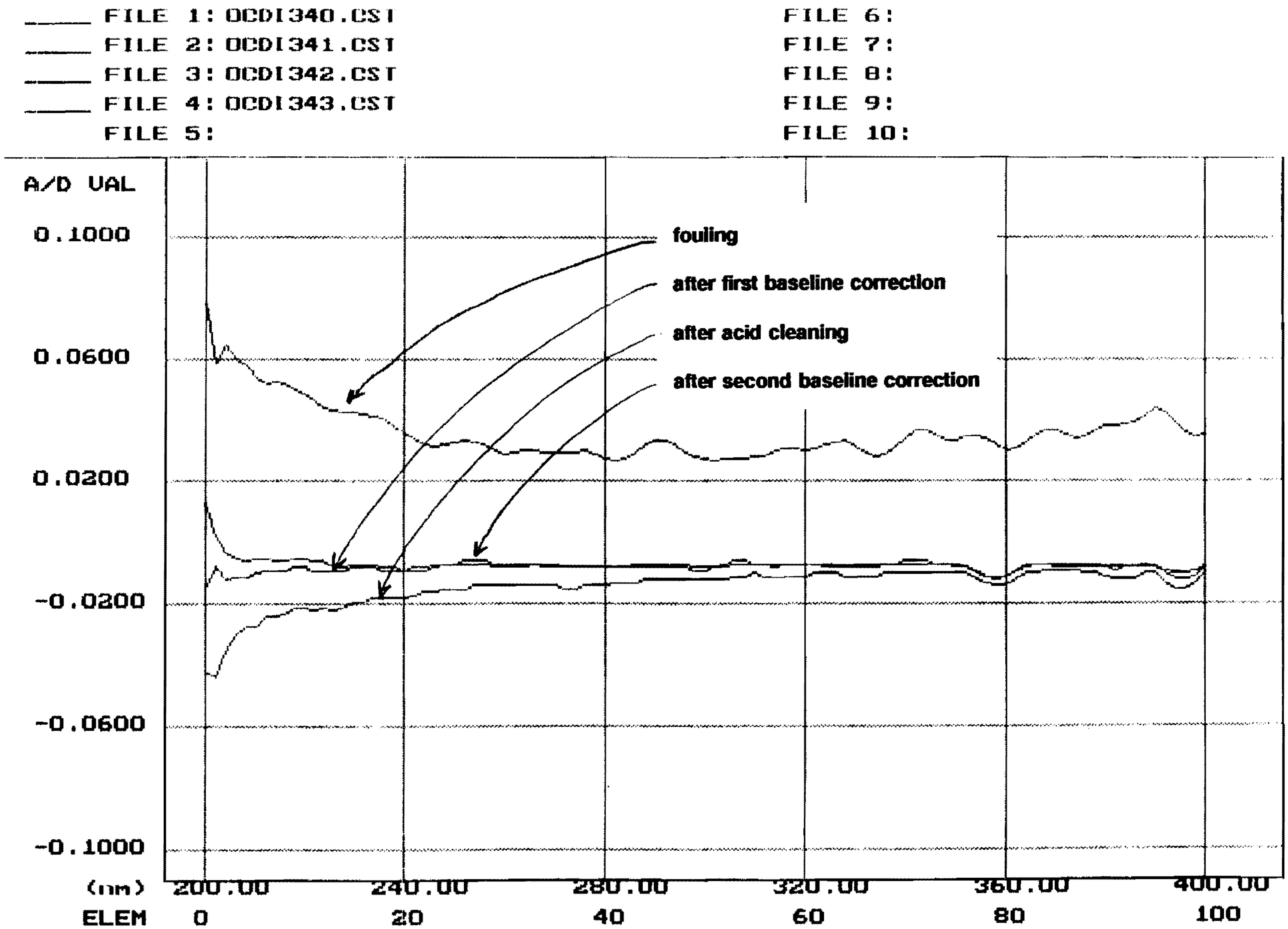


OCONOMOWOC, WI

RELATIVE SCALE, SPECTRA vs MAJOR FOULING



OCONOMOWOC, WI
BASELINE CORRECTION FOR FOULING



OCONOMOWOC, WI
AFTER SECOND BASELINE CORRECTION

OCONOMOWOC TEST SITE
HISTORY

4. DESIGN VERIFICATION

   a. OPERATIONAL SOFTWARE

   b. ANALYTICAL SOFTWARE

   c. OPTICAL "FOCUS"

OCONOMOWOC TEST SITE
HISTORY

B. NEW OPTICS MODULE (SPECTROGRAPH) INSTALLED

1. NEW 30 POINT CALIBRATION USING LABORATORY PREPARED DI STANDARDS

   a. EVALUATE SENSITIVITY TO BACKGROUND VARIATIONS

   b. EVALUATE ABILITY TO ANALYZE SAMPLES THAT HAVE VALUES OUTSIDE OF THE ORIGINAL CALIBRATION RANGE

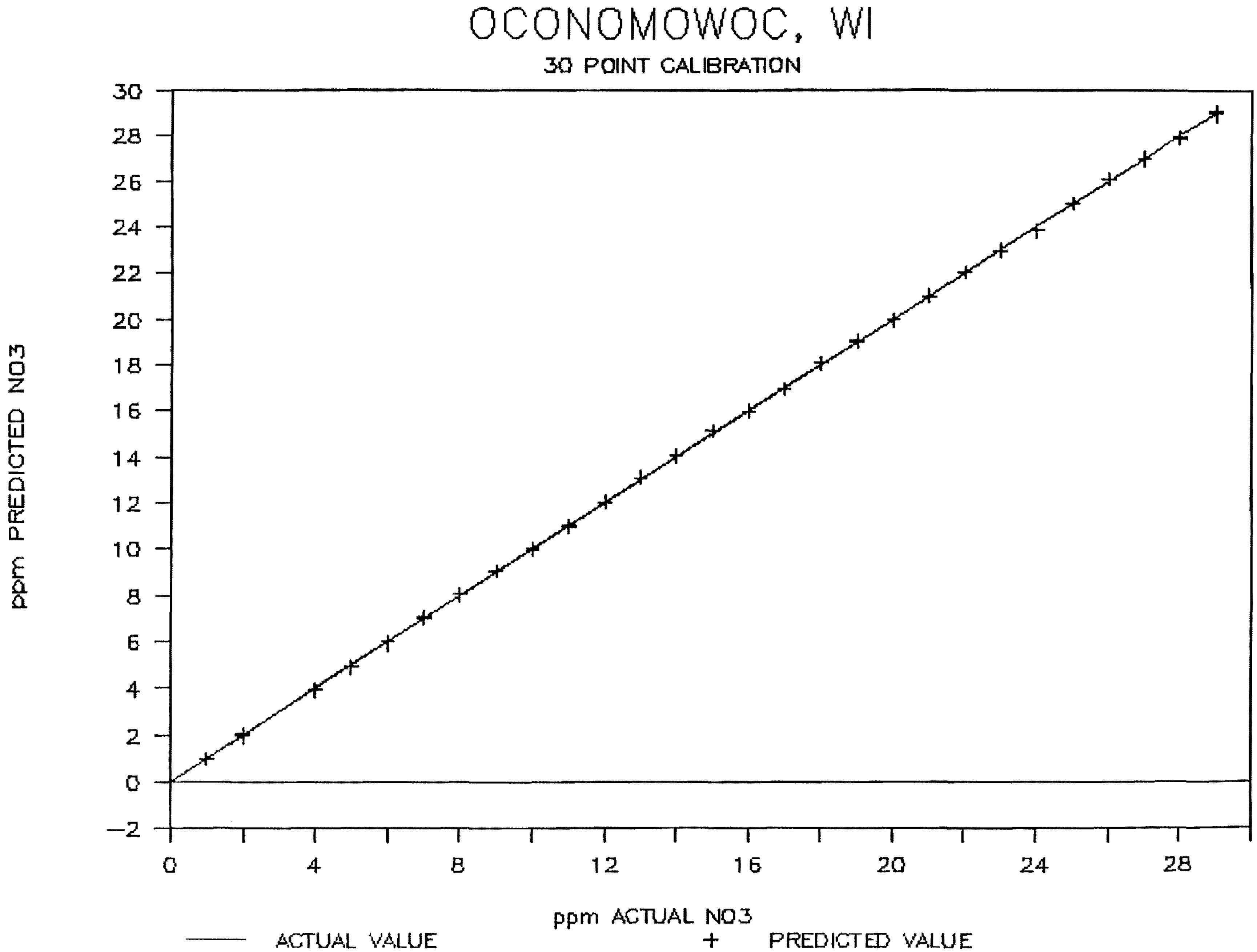
2. RUNNING RECORD MAINTAINED THROUGH USE OF INSTRUMENT INTERNAL DATA LOG FEATURE

   a. OBSERVATION OF TRENDS AND LOAD VARIATIONS

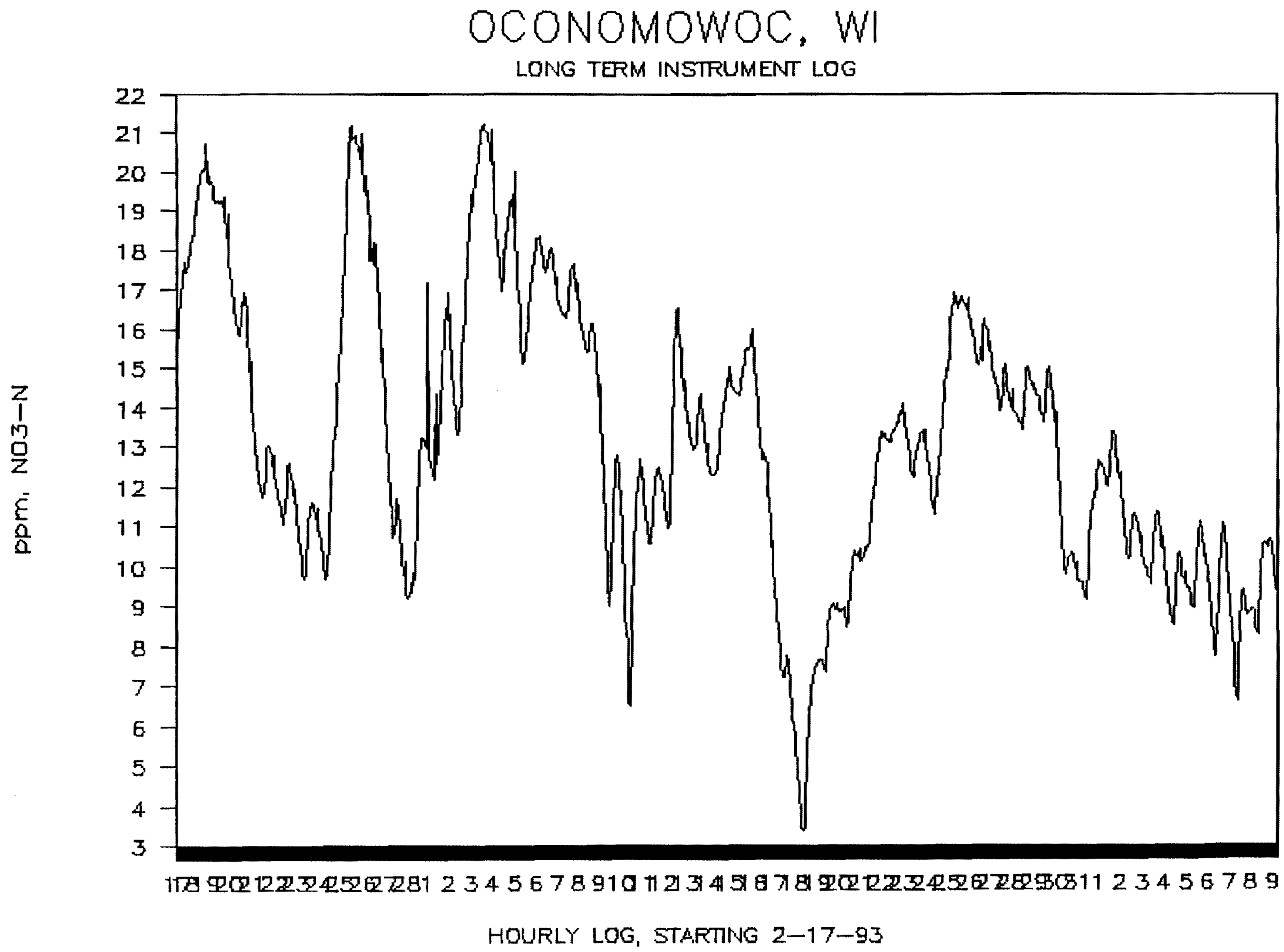
3. REGULAR SAMPLING AND LAB ANALYSIS PROGRAM

   COMPARISON WITH INSTRUMENT READINGS

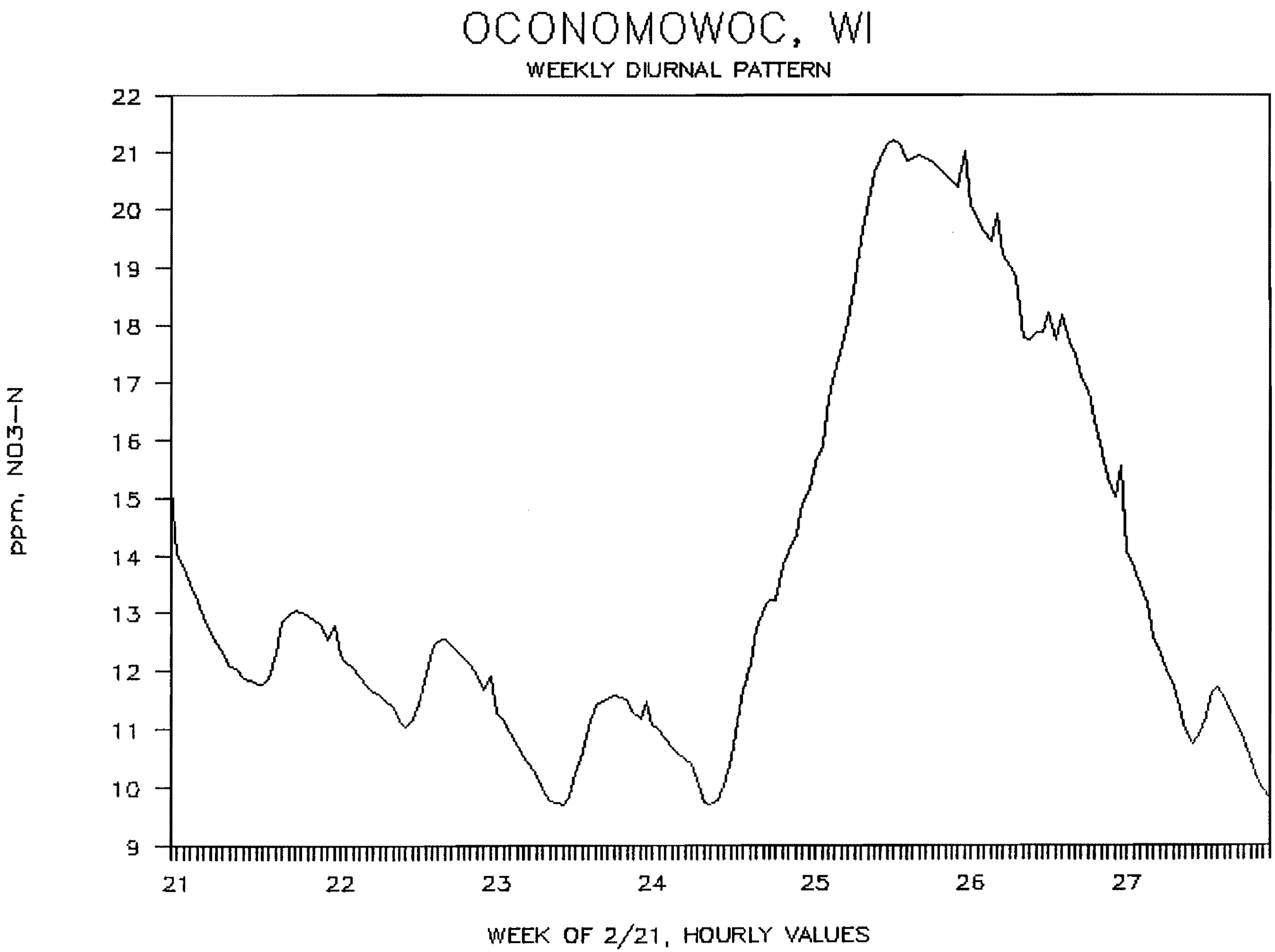
   BUILD A SITE SPECIFIC FILE WITH SPECTRA AND LAB VALUES TO ADD TO CALIBRATION FILE

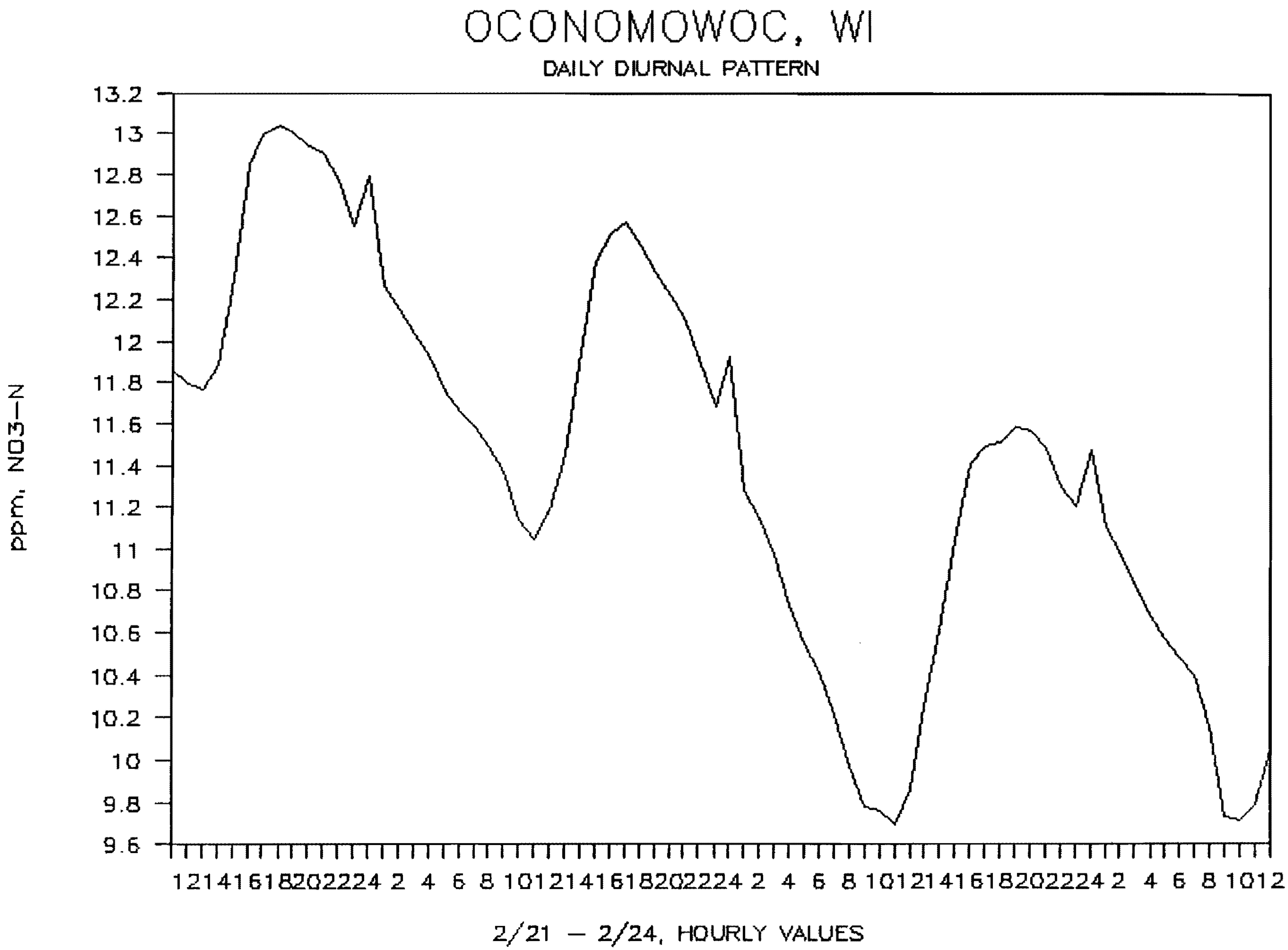


OCONOMOWOC, WI
30 POINT CALIBRATION
ppm PREDICTED NO3
30
28
26
24
22
20
18
16
14
12
10
8
6
4
2
0
-2
0
4
8
12
16
20
24
28
ppm ACTUAL NO3
ACTUAL VALUE
+ PREDICTED VALUE



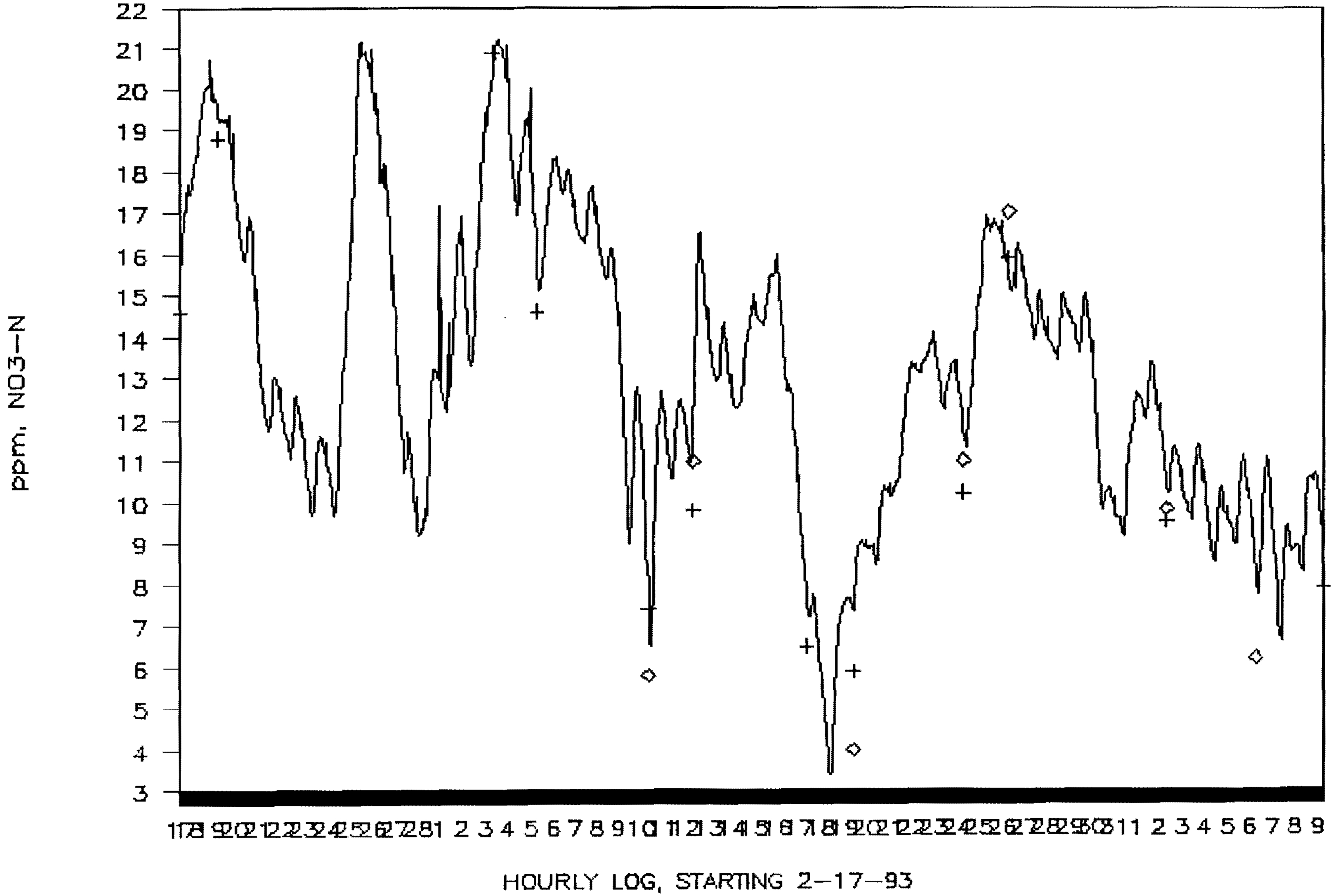
OCONOMOWOC, WI
LONG TERM INSTRUMENT LOG
22
21
20
19
18
17
16
15
14
13
12
11
10
9
8
7
6
5
4
3
ppm, NO3—N
HOURLY LOG, STARTING 2—17—93

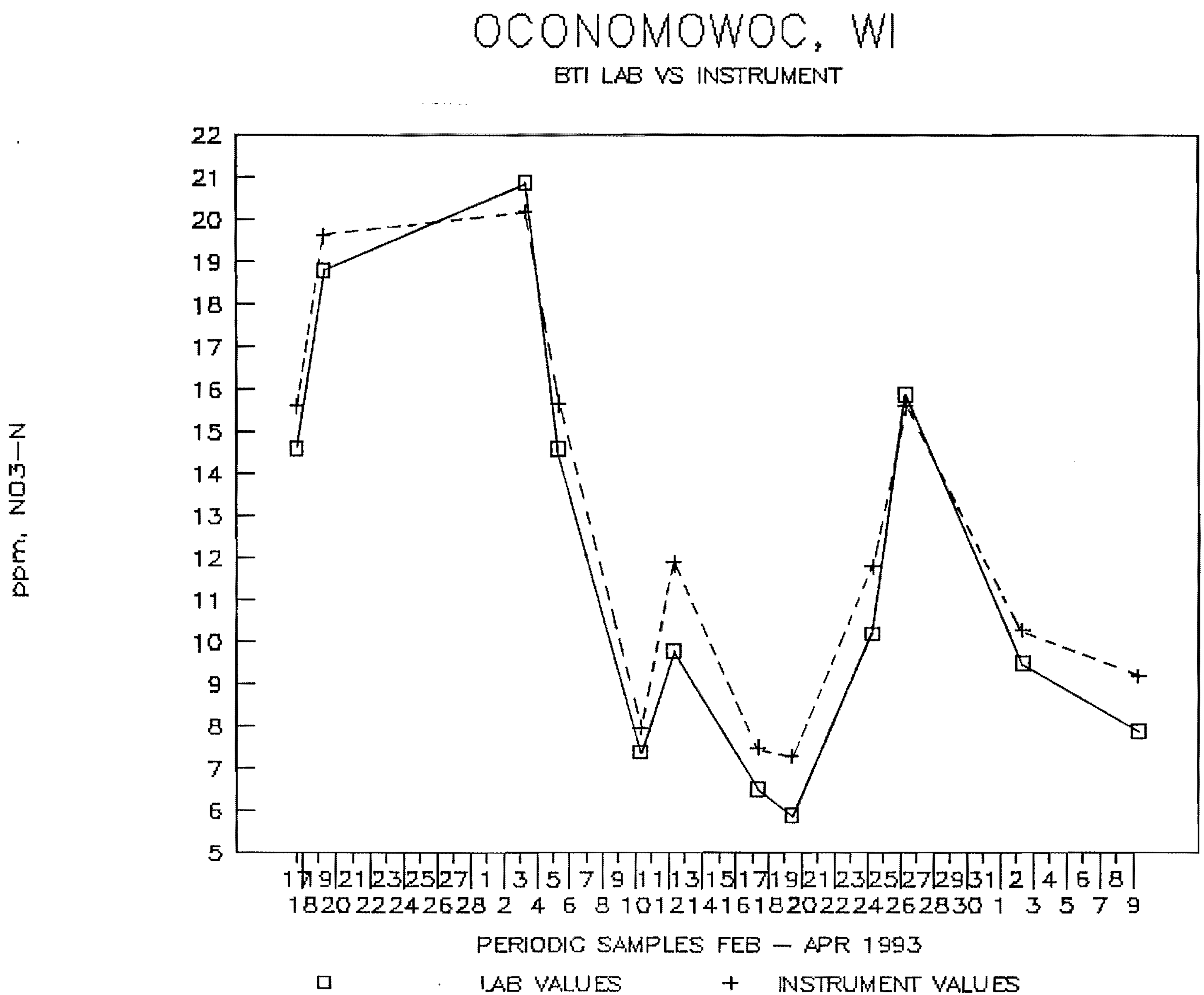

OCONOMOWOC, WI
WEEKLY DIURNAL PATTERN
22
21
20
19
18
17
16
15
14
13
12
11
10
9
ppm, NO3–N
21
22
23
24
25
26
27
WEEK OF 2/21, HOURLY VALUES


OCONOMOWOC, WI
DAILY DIURNAL PATTERN
13.2
13
12.8
12.6
12.4
12.2
12
11.8
11.6
11.4
11.2
11
10.8
10.6
10.4
10.2
10
9.8
9.6
ppm. NO3—N
121416182022 24 2 4 6 8 101214161820222 4 2 4 6 8 101214161820222 4 2 4 6 8 1012
2/21 — 2/24, HOURLY VALUES

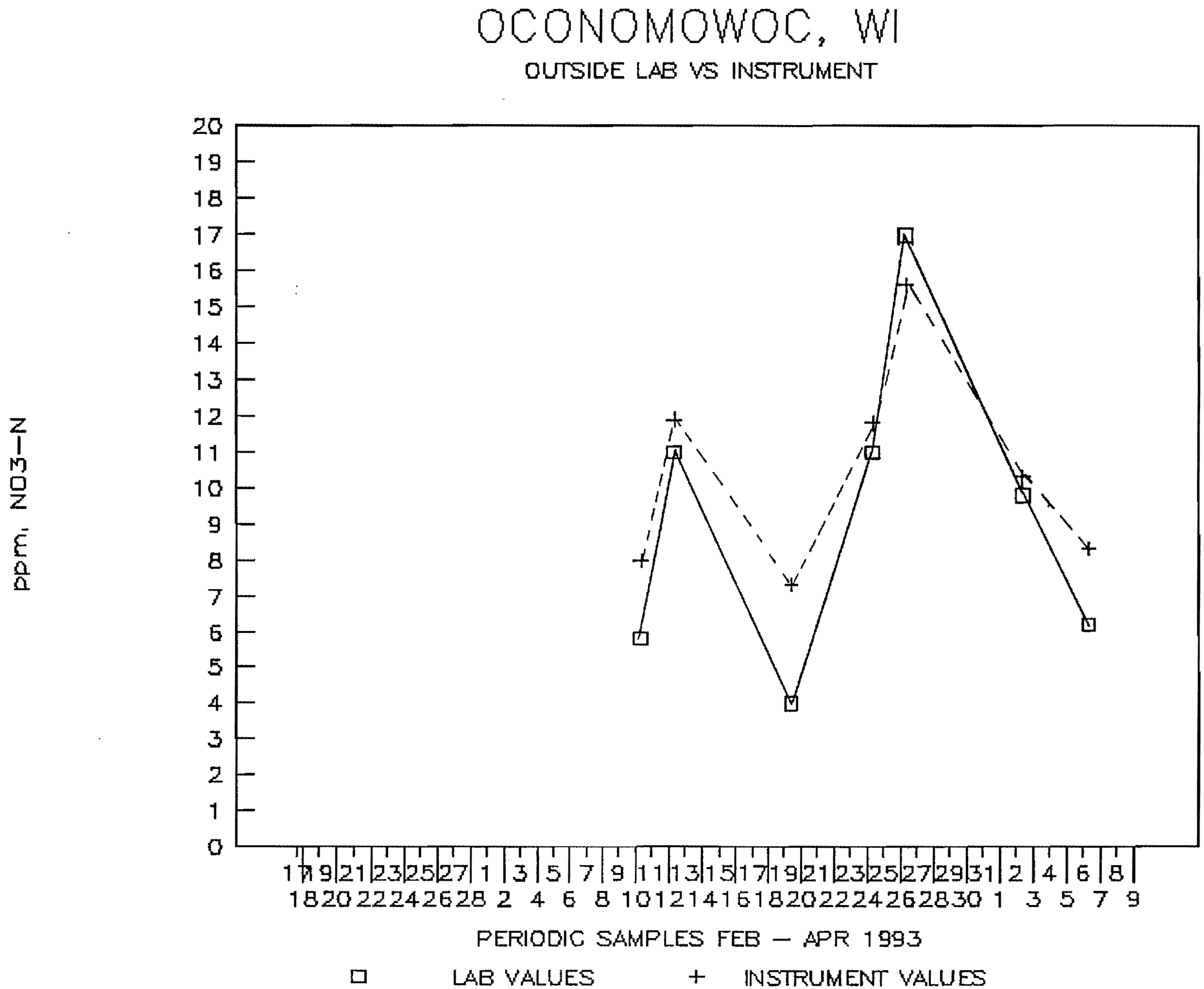
OCONOMOWOC, WI

INSTRUMENT LOG WITH LAB VALUES





OCONOMOWOC, WI
BTI LAB VS INSTRUMENT
ppm, NO3—N
22
21
20
19
18
17
16
15
14
13
12
11
10
9
8
7
6
5
17 19 21 23 25 27 1 3 5 7 9 11 13 15 17 19 21 23 25 27 29 31 2 4 6 8
18 20 22 24 26 28 2 4 6 8 10 12 14 16 18 20 22 24 26 28 30 1 3 5 7 9
PERIODIC SAMPLES FEB — APR 1993
LAB VALUES
INSTRUMENT VALUES



OCONOMOWOC, WI
OUTSIDE LAB VS INSTRUMENT
ppm, NO3-N
PERIODIC SAMPLES FEB - APR 1993
LAB VALUES
INSTRUMENT VALUES

OCONOMOWOC TEST SITE
OBSERVATIONS AND CONCLUSIONS TO DATE

1. BASELINE CORRECTIONS COULD BE USED TO PERIODICALLY ADJUST FOR THE EFFECTS OF FLOW CELL FOULING. THIS PROCEDURE CAN BE PERFORMED IN ABOUT 15 MINUTES USING DI WATER AND THE "INSTRUMENT STANDARD" FEATURE OF THE ANALYZER.

2. THE UV-6100 PROCESS ANALYZER REFERENCE WAVELENGTH FEATURE WILL ALLOW ACCURATE ANALYSIS OVER EXTENDED PERIODS OF TIME, EVEN WHEN ROUTINE MAINTENANCE SUCH AS FLOW CELL CLEANING AND BASELINE CORRECTIONS HAVE NOT BEEN PERFORMED.

3. A 20% HCL SOLUTION COULD BE USED TO CHEMICALLY CLEAN THE FLOW CELL, EXTENDING THE INTERVALS BETWEEN MECHANICAL CLEANING.

4. THE EFFECTIVENESS OF CLEANING AND BASELINE CORRECTION PROCEDURES CAN BE VERIFIED BY RECORDING AND COMPARING SPECTRA BEFORE AND AFTER THE PROCEDURES.

5. THE STABILITY AND REPEATABILITY OF THE ANALYZER CAN BE VERIFIED BY RECORDING AND COMPARING SPECTRA FROM IDENTICAL SOLUTIONS.

6. THE USE OF AN IN LINE FILTER AHEAD OF THE ANALYZER HAS NOT BEEN NECESSARY.

7. SUCCESSFUL LONG TERM MECHANICAL, ELECTRICAL, ELECTRONIC AND OPTICAL PERFORMANCE CAN BE ACHIEVED IN A WASTEWATER TREATMENT PLANT ENVIRONMENT.

8. THE USE OF LABORATORY GENERATED CALIBRATION STANDARDS IN DI WATER CAN PROVIDE A REASONABLY GOOD STARTING POINT FOR THE ANALYSIS OF NITRATE IN A NITRIFIED WASTEWATER EFFLUENT FOLLOWING TERTIARY FILTRATION. THIS CAN REDUCE BUT NOT ELIMINATE THE NEED FOR SITE SPECIFIC CALIBRATION SOLUTIONS IN MANY WATER AND WASTEWATER APPLICATIONS.

9. ULTRAVIOLET ABSORBANCE SPECTROMETRY CAN SUCCESSFULLY DETECT NITRATE ON-LINE IN WASTEWATER WITHOUT REAGENTS.

10. THE UV-6100 PROCESS ANALYZER CAN BE USED TO FAITHFULLY TRACK NITRATE CONCENTRATIONS AT FREQUENT INTERVALS, PROVIDING RELIABLE ON-LINE INFORMATION FOR WASTEWATER TREATMENT PROCESS MONITORING AND CONTROL.