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OPERATION AND ROUTINE MAINTENANCE
OF THE AUTOMATIC MONITOR FOR
HEXAVALENT CHROMIUM IN COLUMBIA RIVER WATER

By

David W. Alton

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AUTOMATIC MONITOR FOR HEXAVALENT CHROMIUM IN COLUMBIA RIVER WATER

By

David W. Alton

Environmental Evaluations
Radiation Protection
TECHNICAL SERVICES DIVISION

January 20, 1969

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TABLE OF CONTENTS

	<u>Page</u>
I. INTRODUCTION	1
II. COMPONENTS OF THE AUTOMATIC HEXAVALENT CHROMIUM ANALYSIS SYSTEM	2
III. START UP PROCEDURE	12
IV. SHUT DOWN OPERATION	14
V. THE MONITORING SYSTEM	16
VI. TROUBLE SHOOTING	19
APPENDIX	23

LIST OF FIGURES

1. Automatic Columbia River Monitoring Station
2. Water Flow for ACRMS Sample Systems
3. Schematic of Analyzer System
- 4.a Upper Half of Analyzer
- b Lower Half of Analyzer
5. Automatic Programmer and Split Cams
6. Programmer Valves and Overflow Cell
7. Continuous Filter
8. Glass Membrane Filter Holder
9. Proportioning Pump
10. Manifold for Cr^{+6} Analysis
11. Flow Diagram for Cr^{+6} Analysis
12. Colorimeter
13. Range Expander
14. Recorder and Controls
15. Power Strip and Voltage Stabilizer
16. Standards Chart
17. Standard Operating Chart.

OPERATION AND ROUTINE MAINTENANCE OF THE
AUTOMATIC MONITOR FOR HEXAVALENT CHROMIUM IN COLUMBIA RIVER WATER

I. INTRODUCTION

A Technicon Autoanalyzer* for hexavalent chromium has been installed at the Automatic Columbia River Monitoring Station (Figure 1), a 12' by 12' metal building located along the west bank of the Columbia River approximately 30 feet above water level. The water intake is located approximately 100' from the PRTR intake structure (DWG No. H-3-9237) in the main channel above the PRTR discharge line. The sample water is delivered to the building by a deep well jet pump for continuous monitoring for gross gamma, ^{131}I , hexavalent chromium, and for emergency water samples. An alarm system indicates unusually high activity levels. The overall sample system diagram is shown in Figure 2.

The unit was installed to detect hexavalent chromium which reaches the river as a result of cooling water treatment for the production reactors upstream, and via groundwater as a result of waste disposal practices at the 300 Area. The detection of the Cr^{+6} ion is based upon its reaction with diphenylcarbazide to produce a reddish-violet color in slightly acid solutions as reported in Standard Methods for the Examination of Water and Wastewater, Twelfth Edition. The degree of color is indicated by light absorption in the colorimeter which is plotted on the recorder and is directly related to the concentration of hexavalent chromium in the river water. Figure 3 shows a schematic of the Autoanalyzer and how the sample is processed.

Although designed to operate continuously and automatically, routine inspection is required to assure proper functioning of all components and to perform the required maintenance and pick up of recorded data. The following data is to provide general information relevant to the operation and maintenance of the system. For additional details and explanation, the "General Operating Instruction Manual for Basic AutoAnalyzer Modules", by Technicon Corporation, is available in the Environmental Evaluations offices.

* Registered trademark.

II. COMPONENTS OF THE AUTOMATED HEXAVALENT CHROMIUM ANALYSIS SYSTEM

The original system was made by Technicon Corporation, Ardsley, New York. Modifications have been made to the flow manifold and to the filtering system to allow continuous operation on river water. A picture of the total system is shown in Figure 4. A condensed maintenance procedure is attached as an Appendix, showing the time sequence for periodic maintenance.

A. Overflow Sampler Cell Sample

This receives a stream of water from the jet pump, a portion of the sample stream is split off as the sample to be analyzed and the rest is wasted as overflow (see Figure 6). This unit will require twice monthly cleaning to remove sand and grit as well as algae accumulation. Cleaning in an ultrasonic bath is recommended. As high a flow rate as the cell can accommodate without leakage should be used. The overflow tubing from the cell and the tubing to the cell need to be replaced periodically due to algae growth.

B. Automatic Programmer

This unit permits the monitoring of one sample stream and one standard solution in a sequence and time interval of the operator's selection (see Figure 4). It also allows adjustment of the automatic baseline standardization cycle to occur at the precise time it is wanted. The programmer operates via a synchronous motor driving a series of two cam-operated microswitches, one to control sample or standard stream, and one to control time of automatic standardization (see Figure 5). These microswitches are adjustable by means of rotation of the split cams to the precise position needed for activation and by the width of opening in the cams for length of activation. The unit also allows operation in either an automatic or manual mode. Under normal conditions, no adjustment of these cams will be necessary. An example and specific detail for adjusting the cams is contained in the Technicon Operating Manual under "Programmer".

C. Programmer Valves

These valves as mentioned previously are operated in time sequence of choice in the automatic mode or manually by the operator's choice (see Figure 5). The valve has 3 nipples on it, one which is normally open, one normally closed, and one common (see Figure 6). The normally closed nipple accepts the tubing from the distilled water jug (baseline), the normally open nipple accepts the tube from the overflow sampler cell (river H₂O), and the common nipple accepts the tubing leading to the continuous filter via the proportioning pump. Periodic cleaning will be required to remove a build-up of fine sand that will cause the valve to malfunction. It is provided with a stand-by valve that can be used in case the valve being used malfunctions.

D. Continuous Filter

This unit serves as a prefilter for the water sample, removing most of the filterable material and thus extending the life of the membrane filter (see Figure 7). This unit has been modified in several ways from the original unit: (a) The motor has been replaced with one which is much slower (4 RPH) and it has been disconnected from the variable speed switch; (b) The original drive roller has been replaced with a smaller one, (1 in. circumference), allowing even slower paper speed; (c) The angle of the unit has been reduced to allow slower movement of the sample water across the filter paper; (d) A paper guide has been added to keep the filter paper being fed the sample from contacting the main filter roll, as the filter paper tends to become wet some distance back from the point of sample contact.

With these modifications, a different and more efficient type of filter paper can be used than that originally provided by Technicon. The filter material now used is S & S #478, a creped filter paper efficient for removal of fine precipitates and with a high wet strength. It is used at the rate of 4 inches/hour or 8 ft/day.

E. Membrane Filter

This filter acts as a finishing filter for the sample water removing the very fine precipitates not removed by the continuous filter (see Figure 8). Removal of these fine materials is necessary to keep them from forming a film on the flow cell causing a rapid drift of the baseline. It is designed to operate as a pressure and suction filter simultaneously. The unit utilizes 99 mm diameter, 0.45 μ pore size membrane filters. It is clamped together with a rubber seal between the two halves of the glass filter holder. A moderate amount of water buildup both above and below the filter is normal, without interfering with the normal response time. The filter requires changing twice weekly unless unusually turbid water is encountered, which may require more frequent changing. This unit will require cleaning in an ultrasonic cleaner utilizing a bath of alcohol and NaOH to remove the sealant grease and other fine materials, especially those in the filter support of fritted glass. The filter was fabricated in the glass shop from a 600 milliliter glass Buchner funnel with a glass frit of medium porosity.

F. Proportioning Pump

This unit is the heart of the system, which with the manifold introduces, proportions, and mixes the sample, reagent (DPC), and air, and moves these fluids to other modules in the system (see Figure 9). Basically the Proportioning Pump consists of two parallel stainless steel roller chains, five stainless steel rollers that connect the chains and a bake-lite platen with springs underneath that exert continuous pressure on the platen when the roller

F. Proportioning Pump (Continued)

head assembly is locked into position. Two stainless steel side rails are placed on the pump platen, one on each side. These keep the pump tubes from having too much pressure exerted on them by the rollers, and thus wearing rapidly. At the point where the stainless steel rollers contact the rails, wear takes place, particularly on the rollers. As this wear increases the pressure on the tubes increases until a point of rapid wear and erratic pulsation of the pump tube liquids occurs. The rollers will wear most rapidly on the drive side of the roller head assembly. When wear becomes too severe the rollers and chain can be separated at any one of the links and the chain reversed, placing the worn spot on the roller on the reverse side. The side rails also will wear and must be switched or replaced depending on their condition. This extends the life of the rollers to approximately four months of continuous useage. Care must be taken in lubricating the roller chain. Only a few drops are needed, on the felt pads, just enough to keep the chain lightly oiled. If the unit becomes over lubricated the rollers become contaminated with oil, as well as the pump tubes and platen, causing rapid wear and snaking of the tubes as well as erratic performance. Cleanliness of the platen, pump tubes and rollers is a must. Routine weekly cleaning with alcohol of the rollers, pump tubes and platen will accomplish the necessary removal of accumulated dirt, grime, oil, etc.

The manifold pump tubes must not bend (snake) during pumping. If this condition occurs, the platen surface should be checked for dirt, oil, particles, or too smooth a platen surface. If these conditions are not present but snaking persists, the pump tubes must be replaced. If replacement tubes are not available, for short periods of time the tubes may be stretched by placing either end block to the next hold position on the platen pins. This procedure changes the relative pumping capacities of the tubes, thus changing the baseline and standard readings. If tubes are stretched it must be indicated on the chart paper, and it should only be done in emergency situations. If the tubes are to be left in a stretched position for any length of time a series of standards must be run. Pump tubes will require replacement after two weeks of continuous operation.

G. Manifold

The manifold holds the pump tubes, and is mounted on the pump and pump platen (Figure 10). It advances various fluids (samples, standards, reagents, gases) through the system in the exact proportions required for analysis of hexavalent chromium. The manifold (including fittings and mixing coils) is secured on a platen to facilitate storage, reduce breakage, protect pump from spillage, and provide for easier and more compact operation of the Autoanalyzer system.

G. Manifold (Continued)

The flow diagram (Figure 11) gives the exact positioning of the pump tubes, and the flow through the entire system. The pump tubes used have the following parameters:

<u>Shoulder</u>	<u>I.D.-in.</u>	<u>Delivery cc/min.</u>			<u>Fluid</u>
		<u>Min.</u>	<u>Nominal</u>	<u>Max.</u>	
Orange-Yellow	0.020	0.13	0.16	0.19	Diphenylcarbazide
Red-Red	0.045	0.73	0.80	0.87	Air
Green-Green	0.073	1.90	2.00	2.10	Sample from flow cell
Purple-Black	0.090	2.77	2.90	3.03	Waste from Cont. filter (others may be substituted)
Purple-Orange	0.100	3.26	3.40	3.54	Membrane filter water
Purple-Orange	0.100	3.26	3.40	3.54	Tape filter water
Purple-White	0.110	3.75	3.90	4.05	Sample water

The air pump tube provides air for bubbles to segment the liquids into small sections providing more efficient mixing of liquids and scrubbing of tubing walls. The scrubbing action prevents build up of contamination. The manifold consists of the following parts:

1. Manifold pump tubing - is supplied in various sizes. Each size delivers a specific amount of reagent or sample into the system, the various pump tubes are color coded.
2. Glass fittings and polyethylene nipples - are used for inter-connecting manifold tubes etc. (see Bulletin G. F. 3 contained in the "Manifold" section of the Technicon Operating Manual which lists the various types of fittings and nipples).
3. Mixing Coils - mix the liquids that are brought together and provide time delay for color development before introduction into the colorimeter flow cell.
4. Transmission Tubing - interconnects modules and components throughout the system. This tubing may be made of glass or plastic, but in most cases is Tygon 1/16" I.D. available from stores.
5. End Blocks - hold the manifold pumping tubes in position on the pump platen with the desired degree of tautness.
6. Platen - holds the above parts in an optimum arrangement.

(a) Construction of a Manifold

For ease of construction, manifolds may be built on the proportioning pump platen.

- 1). With the roller chain assembly tilted back, the end blocks are placed on the platen with the platen pins going through the holes closest to the lip of the end block, with the lips positioned away from the pump.
- 2). The 7-slot bottom section of the end block is placed down on the platen; the 8-slot section should then face upwards.
- 3). The required pump tubes, as indicated by the flow diagram, are laid out on the platen, being stretched between the end blocks with their shoulders on the outside of, and underneath the lips of the end blocks.
- 4). To prevent errors, the tubes should be laid out in the same sequence as suggested by the flow diagram, or as in the old manifold.
- 5). Cut tubing to proper length beyond the shoulders.
- 6). Attach various tubing as indicated by the flow diagram and assemble to other modules with transmission tubing as necessary.
- 7). The reagent lines (distilled H₂O and diphenylcarbazine) should have glass weights attached to the ends by squeeze fit. These glass weights keep the reagent delivery lines on the bottom of the containers.
- 8). The glass-to-glass fittings must be butted together using 1/8" I.D. Tygon tubing. This prevents improper bubble patterns. This tubing is also used for tubing to glass fittings. All nipple to tubing fittings must be properly made, use a hemostat to stretch the tubing, which allows useage of much larger nipples and makes much better, tighter connections.
- 9). The glass coils used on the manifold are #'s 105-83 and 105-84 listed in the Technicon Manual under "Manifold".

Details of construction of a manifold and how the various pieces fit together are covered in the Technicon Operating Manual.

H. Colorimeter

The colorimeter monitors the concentration levels of Cr⁺⁶ samples and standards flowing through it (see Figure 12). The color present in the tubular flow cell (50 mm) increases as the Cr⁺⁶ concentration of the sample or standard increases, resulting

H. Colorimeter (Continued)

in a decrease of transmitted light. The results are transmitted to a recorder, where a permanent record is recorded on a moving chart. The unit is made more sensitive by using 540 mμ light filters. This is equivalent to a Wratten No. 58 filter with 28% of the transmission at 540 mμ $\pm$ 9 mμ at 1/2 of the peak height.

The flow cell is the critical portion of the colorimeter from a routine maintenance standpoint. Its assembly is accomplished using standard transmission tubing to accept the sample from the C-5 debubbler and run to the flow cell inlet. Care must be taken using the U-clip to keep the tubing out of the light pathway. The flow cell must be cleaned weekly. Care must be taken in replacing the flow cell to assure it is properly seated, and is aligned with the red dots on the flow cell pushed as far forward towards the red dots on the flow cell holder as it will go.

With diphenylcarbazide and distilled water being pumped through the system, the unit is zeroed by placing the blank or zero aperture in the aperture slot on the sample side, and replacing the covers. The aperture used with the unit at all times on the reference side is #3.

1. Set zero on the recorder using the colorimeter zero control with all covers in place, then remove the zero aperture.
2. Set the baseline to 96% Transmission on the recorder chart using the potentiometer on the colorimeter.
3. Adjust the gain control in the recorder so that the pen returns to within 0.1% of its original setting when the large "Bull" gear is displaced to the left or right of its null position. The unit is then ready for sample analysis.

Air bubbles are removed from the sample by the C-5 debubbler before it enters the flow cell in the colorimeter. At start up and at other times an air bubble may become trapped in the flow cell, as indicated by an oscillating pen, or a loss of sensitivity. To eliminate an air bubble that is trapped in the flow cell, pinch the outlet line from the flow cell for approximately 5 seconds and then suddenly release pressure. This may or may not suck additional bubbles into the system from the debubbler but in most cases this will eliminate the trapped bubble. A bubble passing rapidly through the flow cell is indicated on the recorder by a sharp drop in %T followed by a fast increase back to approximately the original position, which looks like a straight line drawn across the chart paper.

Loss of pot adjustment on the colorimeter will occur with time. When the 96%T baseline can no longer be reached using the colorimeter pot adjustment, maintenance on the colorimeter is required. Clean the flow cell, optics, filters, light bulb and recheck the flow

H. Colorimeter (Continued)

cell alignment. The light bulb tends to pick up a fine coating of dust, which can be removed by rubbing it with a tissue. If this procedure fails to provide enough potentiometer adjustment to reach 96%T, replace the light bulb. If this fails, see the trouble shooting guide in this paper as well as the Technicon Operation Manual.

Optical alignment of the flow cell and optics may be required periodically, particularly if the flow cell has been forced into position or mishandled. This will result in light leaks and a loss of sensitivity of %T. The procedure for optical alignment is covered in the Technicon Operation Manual under "Colorimeter".

I. Range Expander

The function of the range expander is to improve the readability of test results which fall in a narrow % transmittance range (see Figure 13). The range can be expanded so that a greater amount of scale is used for the same range of %T.

At the present time the range expander is being operated on X1 scale, but future data may indicate it is preferable to operate on the X2 scale, which would in effect spread 50 to 100%T over the full scale width. The procedure for using this module is covered under "Expanding a Range Whose Upper Limit is 100%T" in the Technicon Operation Manual.

As a precautionary note, the range expander is very sensitive to temperature fluctuations, and if used, should be protected adequately from drafts, etc.

J. Recorder

Operation and maintenance of the recorder is fully described in the Bristol Instruction Book PL2058-1, included in the Technicon Operation Manual (see Figure 14).

1)a Zeroing - with the #3 aperture in place and covered on the reference side of the colorimeter, put the blank (0) aperture in the aperture holder on the sample side and replace the cover. The pen on the recorder should now move toward the zero end of the scale. Turn the zero adjustment on the colorimeter to bring the pen exactly to zero on the paper. Once the zero is set, remove the zero aperture from the sample side and replace the cover. It will take from 10 to 15 minutes for the photocells to become "normalized" and some drifting will occur after this zeroing procedure.

After waiting the appropriate time for normalization to occur, if necessary readjust the pen to 96%T on the recorder using the 100%T control on the colorimeter. Reset gain control if necessary.

J. Recorder (Continued)

- b. Setting 96% Transmission - With the appropriate filters, #3 aperture and covers in place, start distilled water and diphenylcarbazide pumping through the system until a stable line is drawn by the recorder with little or no drift. Turn the 100%T control dial on the colorimeter until the pen on the recorder chart reads 96%T. This is the 96%T base line.
- c. Adjusting Amplifier Gain - Adjust the gain control in the back of the recorder so that the pen returns to within 0.1% of its original position when the large "Bull" gear (located just behind the top roller in the Recorder) is displaced to the left or right of its null position. An alternate method of adjusting the gain is to turn the Gain Control clockwise until the stylus hunts (oscillates). Then rotate the control counter clockwise until the oscillation just stops. Finally, rotate the Gain Control further counterclockwise approximately 10°.
- d. Automatic Baseline Standardization - The recorder is provided with a means to compensate for drift in the system. Once the baseline has been set at 96%T the unit automatically resets the baseline at 96%T when the standardization cycle is being run on the hourly sampling cycle controlled by the programmer. The period of time the automatic standardization takes place is controlled by the positions of the last set of cams in the programmer. There are three controls in the recorder which bear directly on the automatic standardization procedure, these are:
 - (1) The Mode Switch - located on the recorder door on the side. It may be placed in the automatic or off position and may be pushed manually to activate an adjustment of the baseline.
 - (2) The Automatic Standardization Position Control - located on the side of the recorder door above the mode switch. This is the control that positions the 96%T baseline adjustment by the programmer.
 - (3) The Automatic Standardization Adjustment Control Pot - located on the back of the recorder door. This control indicates the amount of automatic adjustment available to move the baseline in the appropriate direction to bring it to rest on 96%T.
- e. Procedure for Adjusting Automatic Baseline Setting:
 - (1) Set the programmer on manual and standard. Set the Automatic Standardization Adjustment Control Pot to 1.0 on its scale of 3.0.
 - (2) Set the baseline at 96%T using the procedure previously outlined. Be sure to wait until a constant reading is obtained on the baseline.

e. Procedure for Adjusting Automatic Baseline Setting: (Continued)

(3) Set the Automatic Standardization Position Control to approximately 96%T on its scale of 100%T and then manually push the mode switch to standardize several times, this will move the pen towards 96%T. If not on 96%T readjust the automatic position control and manually standardize. Keep repeating this process until the pen rests exactly on 96%T then lock the setting in place on the automatic position control.

(4) Set the Mode Switch on the automatic position, switch the programmer to the automatic position with the time control dial on the back of the programmer drawer at the upright (0) position. The unit will then proceed through the normal automatic standardization cycle which should be observed to see that it functions properly. If the unit is operating properly, the recorder will trace a line in the vicinity of 96%T and then adjust to 96%T indicated by several pulses of the pen, with the pen finally coming to rest on the 96%T baseline.

K. Multiple Receptacle Power Strip and Voltage Stabilizers

This is a 4 foot long metal strip with 12 electrical receptacles in it. All receptacles provide the same voltage into which the strip is plugged. A manually resettable circuit breaker or fuse protects the system from electrical damage during operation. A neon lamp indicates when power is available from receptacles. The total current of all modules connected to the strip must not exceed 15 amperes. (See Figure 15.)

Procedure and Use

1. Mount the power strip above and to the rear of the Autoanalyzer system. Do not face the receptacles upward in order to avoid short circuits if liquids fall on the power strip.
2. Plug the power strip into the voltage stabilizer providing a constant 115 VAC 60 cycle voltage. This receptacle must be properly grounded.
3. To determine if the Power Strip is properly grounded, plug the recorder into it, open the recorder, a neon test lamp on the terminal strip at the left will glow dimly if a bad ground is present. If the lamp is glowing brightly then the power strip is not receiving the correct polarized voltage. In either case check the voltage supply.
4. The neon test lamp in the recorder should not glow at any time during the operation of the analyzer.

K. Multiple Receptacle Power Strip and Voltage Stabilizers (Continued)

5. In the event that total current drawn through the power strip exceeds its rated 15 amperes, the circuit breaker will open and cut off power to the receptacles. It is necessary to determine which of the modules has caused the excessive power drain. Follow the procedure: (a) Unplug all modules from the strip; (b) Allow 10 seconds for cooling of circuit breaker and reset; (c) Plug modules in one at a time until defective module causes circuit breaker to open.

There are two voltage stabilizers in the system. One provides constant power of 115 VAC 60 cycle for the power strip and was not part of the original system. The other is an integral part of the system and supplies the colorimeter with constant voltage for operating the tungsten light source of the colorimeter. A stabilized lamp voltage of 6.0 volts AC is maintained. The colorimeter is the only module that operates from this stabilizer.

III. START UP PROCEDURE

A. Place the modules in correct sequence in the cabinet. Plug all modules into the power strip which is plugged into the 115 VAC 60 cycle voltage regulator. Plug the male lead on the recorder into the receptacle on the back of the colorimeter. Plug the male lead from the stabilizer unit into the other receptacle on the back of the colorimeter. Plug the telephone jack from the recorder into its receptacle on back of the colorimeter.

B. Determine whether the power strip is receiving a good ground or is properly polarized by checking the neon bulb in the back of the recorder. If the lamp is glowing brightly, then the power strip is not receiving the correct polarized voltage, if it is glowing dimly a bad ground is present. Do not proceed until this situation is corrected.

C. Place manifold on pump and attach modules with transmission tubing as indicated in the flow diagram.

D. Assemble the colorimeter flow cell as shown in the Technicon Operation Manual (except use standard transmission tubing to run from C-5 debubbler to flow cell inlet). Position the flow cell properly with the red dots on the flow cell pushed towards the red dots on the colorimeter.

E. The #3 aperture should be in place on the reference side of the light source, and the reference light and sample light filters (540 mμ) in their proper positions.

NOTE: Steps C to E should include, connection of reagent lines, building of the manifold, installation of chart paper, filling of pen, placement of filters papers, etc. Be sure waste lines lead to a suitable outlet.

F. Turn on the colorimeter - allow 25 minutes for the unit and photocells to come to equilibrium. (Caution - under no circumstances should the light filters be removed with the light source on, as this may damage the photocells, and/or ruin the response, taking several hours to return to normal.)

G. Turn on the pump, and start pumping distilled water and diphenylcarbazide into the system.

H. Turn on the recorder, allowing five minutes for warm up.

I. Operate the range expander at "fixed" and X1.

J. Turn on the continuous filter. Check to make sure no bubbles are entering the system below the tape filter. Adjust filter tape and/or level of filter if necessary.

K. After fifteen minutes, verify that bubble patterns are evenly segmented, connections properly butted, no leaks present, and pumping of liquid adequate, with minimum pulsation and no pump tube snaking. Watch for bubbles in the flow cell, indicated by an oscillating pen. Remove bubbles by pinching the outlet line from the flow cell.

L. When a straight line is being drawn on the recorder with no drift (not greater than 1% per hour), set the base line at 96%T with the colorimeter 100%T control (with the automatic standardization pot on the back of the recorder door set at 1.0.)

M. Once the baseline has been set, zero the recorder, and reset the baseline if necessary.

N. Check recorder gain.

O. Adjust the river sample flow to overflow sampler cell.

P. Turn on the automatic programmer which should have the cams set for the appropriate sampling and calibration time sequence.

Q. Set the programmer time clock (on back of Programmer drawer) to the 0 position, and place the switch on the automatic mode of operation. This will start the standard cycle (distilled H₂O).

R. Turn the Automatic Standardization Mode Switch on the recorder to automatic. Observe the recorder trace as it goes through the standardization cycle for the appropriate adjustment to 96%T baseline. Adjust the Automatic Standardization Position Control if the adjustment is not to 96%T. Then standardize manually until 96%T is properly adjusted.

S. Turn the automatic programmer to manual and standard, develop a consistent 96%T baseline (turn automatic standardization to the off position until a steady 96%T has been reached).

T. Test the response of the system with the standard solution containing 20 parts per billion Cr⁺⁶ in distilled water. The response should be such that the pen has started down scale by nine minutes and should have peaked at 85%T ± 2%T by 30 minutes. It should hold a steady state signal with fluctuations, if any, less than 1/2 %T.

U. Set the automatic programmer timer at the 0 position, switch the unit to automatic, and place the Automatic Standardization Switch on the recorder in the automatic mode. Mark recorder chart with date and time and standard solution concentration. The unit is ready to monitor the river water.

IV. SHUT DOWN OPERATION

The complete shut down procedure in most cases will not be used except during extensive repairs. The first procedure listed below will be that used during normal maintenance or other short (1 day or less) shut down times. The second list gives additional procedures to be used for a longer and complete removal from service.

A. Turn the Pump Off

Caution: DO NOT RELEASE AND RAISE THE ROLLER ASSEMBLY DURING TESTING UNLESS YOU FIRST:

1. Clamp the waste lines from the colorimeter
2. Clamp the line from the diphenyl carbazide bottle
3. Clamp the line from the membrane filter
4. Clamp the line from the common nipple on the programmer valve.

Mixed reagents in the tubing will flow backward into the system, contaminating the reagent bottles and draining the system unless this procedure is followed.

5. ALTERNATE:

If it is necessary to stop pumping for a short period of time (less than 10 minutes) flip the switch to off, and leave the roller head assembly down in operating position; this prevents drainage and contamination of the system.

NOTE: Do not leave the roller head assembly in the down position for long periods when the pump is not operating. The fixed roller pressure on the pump tubes will damage the tubes. Keep the roller assembly in raised position off the manifold tubes when the unit is not in operation. Release one of the manifold end blocks to relax tubing when pump is not in operation for an extended period of time.

B. Proceed to other modules, turning off chart paper feed, filter tape feed, or others as needed for maintenance.

For Longer than One Day Shut Down

1. Prior to following the pump opening procedure previously listed, pump distilled water through all lines leading to the system for fifteen minutes and then clamp them. This leaves water in the flow cell, which is recommended unless very long down times are anticipated (several weeks).

2. If longer down times are anticipated:

- a. Drain all lines and flow cell,
- b. Remove flow cell and rinse under tap (using bent pipe cleaner if necessary). When clean, rinse with distilled water, wipe all light transmitting surfaces with lens paper.
- c. Clean all equipment thoroughly and protect with plastic bags.

V. THE MONITORING SYSTEM

A. Data Interpretation

1. Interpretation of the data is best understood by giving an example of the sensitivity of the unit. If standards in distilled water of 5 and 50 parts per billion Cr^{+6} are placed into the system, responses of approximately 93%T and 70%T [$\pm 2\%$ T] respectively would be expected. The unit responds logarithmically to concentrations, being the most sensitive at the lower range of concentration.
2. At any time a chart reading on river water occurs at less than 85%T (greater than ~ 20 PPB), immediate notification should be made to an exempt staff member of Environmental Evaluations or Environmental Monitoring. Figure 16 indicates the appearance of a normal operation chart. Figure 17 is a picture of a series of standard measurements.

B. Standard Check and Calibration

1. The routine standard check will be made with 20 PPB Cr^{+6} in distilled water. It will be used after any of the following items occur:
 - a. Changing pump tubes
 - b. Changing light bulb in colorimeter
 - c. Cleaning flow cell in colorimeter
 - d. Any major maintenance on recorder, pump, or colorimeter such as changing rollers, etc.
 - e. Any time the performance of the analyzer is in doubt.

It will also be used routinely once per week, in addition to any of the above checks. The standard response with 20 PPB Cr^{+6} in distilled water will be 85%T $\pm 2\%$ T with the baseline at 96%T.

2. Standard Check Procedure:

- a. Zero the recorder and develop a steady state baseline of 96%T with no drift (less than 1%T/hour) on distilled water, before starting standard into the system at the programmer valves. (See the zeroing procedure outlined previously under Recorder).
 - (1) Be sure to use contamination free tubing for the standard bottle.
 - (2) Siphon standard into the tubing before placing it on the stream nipple on the programmer valve to keep air bubbles from entering the system.

- b. Run a known standard of 20 parts per billion in distilled water into unit. It should read $85\%T \pm 2\%T$ within 30 minutes. If it does not, further investigation into the cause is needed. See the trouble shooting guide in this paper or the Technicon Operation Manual.

3. Standardization

During the last week of each month, a 3-gallon grab sample will be obtained from the Priest Rapids Dam Gauge Station for the purpose of making a series of hexavalent chromium standards. River water is used to allow for natural color interferences in the river water which would not be present in distilled water standards. These curves will be used to evaluate readings made on the recorder. The standards will be made up and run the day after the sample is collected if possible. The concentrations to be used are: 10, 20, 50, and 100 parts per billion Cr^{+6} . The river water must be analyzed at the PRTR laboratory for Cr^{+6} to account for any natural Cr^{+6} in the water, so that this may be accounted for while drawing up the standardization curves.

4. Standardization Procedure (River Water)

- a. Zero the recorder and develop a steady state baseline of $96\%T$ with no drift (less than $1\%T/hour$) on distilled water, before starting standard into the system. (See the zeroing procedure outlined previously under Recorder.)
 - (1) Be sure to use contamination free tubing for the standards.
 - (2) Siphon standard into the tubing before placing it on the stream nipple on the programmer valve to keep air bubbles from entering the system.
- b. Run a known standard of 20 PPB in distilled water into the unit. It should read $85\%T \pm 2\%T$ in 30 minutes. If it does not, further investigation into the cause is needed. See the trouble shooting outline in this guide or the Technicon Operating Manual.
- c. Using contamination free tubing, start at the lower concentration of 10 PPB in river water, as the initial standard to be run into the unit. From there proceed to the next higher concentration and so on until the four concentrations have been run.
 - (1) The unit may be run on the automatic programmer, standardizing the baseline on distilled water between each standard, requiring that a person return on the hour to put the next standard on; or,
 - (2) The standards may be run sequentially, requiring the operator to stay and switch to the next standard after the

previous one had peaked (approximately 30 minutes to peak). Be sure to turn off the Automatic Standardization Switch if this method is used.

a.) Using this last method, after all the standards have been run, return to the baseline on distilled water to determine drift, and report the baseline reading along with the standard readings.

d. Record and report readings on river water standards to the Supervisor, Environmental Monitoring.

VI. TROUBLE SHOOTING

The complexity of this system makes it difficult to pin-point trouble spots for one unfamiliar with the system. Problems which have developed during routine operation of this specific system have been addressed in the following suggestions. For additional help in trouble shooting, see the Technicon Operation Manual.

A. Main Trouble Areas

Generally there are three specific potential trouble spots, the pump and manifold, the colorimeter, and the filters. Each has its particular effect when it malfunctions.

1. The Pump and Manifold

This is the area of most frequent trouble, mainly because it is subject to wear and must be continuously kept in a clean and oil-free condition for proper operation.

<u>Trouble</u>	<u>Indicated by</u>	<u>Corrective Action</u>
(a) One or a combination of worn, snaking, oily, dirty, pump tubes, platen and/or manifold.	(a) Improper standard response, poor bubble pattern, slight pulsing of pumped fluid, oscillating pen on recorder on a constant concentration.	(a) Cleaning of all tubes and platen and/or replacement of worn pump tubes. Cleaning and rebuilding of manifold.
(b) Worn pump rollers and/or side rails.	(b) Rapid wear of pump tubes (less than 2 weeks) improper standard response, severe pulsing of pumped fluid, oscillation of recorder pen on a constant concentration, bubbles entering system.	(b) Reverse roller chain and side rails, or replace one or both as indicated.
(c) Improper connections from glass to glass, tubing to glass, or tubing to nipple.	(c) Poor bubble pattern, oscillating pen, poor response.	(c) Butt glass pieces end to end, use proper nipple sizes, make connections tight, use hemostat to stretch tubing to fit connections.

2. The Colorimeter

The light bulb and the flow cell are the two sources of most frequent trouble in this unit.

2. Colorimeter (Continued)

<u>Trouble</u>	<u>Indicated By</u>	<u>Corrective Action</u>
(a) Dirty or weak light bulb, or dirty and/or misaligned flow cell, dirty optics.	(a) Loss of Adjustment for 96%T, loss of sensitivity.	(a) Clean light bulb or replace light bulb. Clean optics and flow cell and/or realign flow cell with red dots pushed as far as possible towards each other. After turning unit back on, allow 25 minutes for stabilization before it is standardized.
(b) Bad light bulb.	(b) Erratic signal to pen of recorder or lack of sufficient light to set 96%T.	(b) Change light bulb.
(c) Misaligned optics.	(c) Loss of adjustment for 96%T, loss of sensitivity.	(c) Realign optics as indicated in Technicon Operation Manual under Colorimeter.
(d) Bubbles and/or particles in flow cell.	(d) Erratic signal to pen of recorder; loss of %T, inability to set 96%T; pen rapidly moves to lesser %T then back again.	(d) Eliminate source of bubbles; check for leaks; pinch waste line from flow cell for 5 seconds, suddenly releasing pressure, until bubbles are removed as evidenced by lack of oscillation by pen, and possible gain in %T.
(e) Diphenylcarbazide not properly made or not being pumped.	(e) Non-fluctuating or inadequate response when instrument is on line.	(e) Make sure fluid is being pumped; replace solution.

3. Filters

Improper flow through pump tubes affecting the filters is the main problem. Air entering the system may occur at the tape or membrane filters interfering with the signal to the recorder from the colorimeter.

<u>Trouble</u>	<u>Indicated By</u>	<u>Corrective Action</u>
(a) Buildup of excess water above and below the milipore filter.	(a) Large volume of water almost completely filling the filter enclosure from top to bottom, causing very slow response to concentration changes, and interfering with automatic baseline standardization.	(a) Measure volume of flow/min of pump tubes to and from the filter, and replace either or both pump tubes if they are not pumping at near their recommended volumes (± 0.2 ml). Replace filter if it is dirty, clean fritted glass filter if it has not been cleaned recently (use ultrasonic bath & a grease solvent).

3. Filters (Continued)

<u>Trouble</u>	<u>Indicated By</u>	<u>Corrective Action</u>
(b) Bubbles entering system.	(b) Buildup of water on top of membrane filter & bubbles entering system at bottom of milipore filter, or bubbles from the continuous filter at the filter block. Ragged signal and bubble pattern, with possibly very large spikes on the recorder.	(b) Replace filter and/or clean membrane filter holder. Adjust flow onto continuous tape, adjust level of continuous filter, check water flow to filter and pump tube.

B. Other Trouble Areas

1. Recorder

<u>Trouble</u>	<u>Indicated By</u>	<u>Corrective Action</u>
(a) Gain adjustment too high.	(a) Oscillating pen signal on steady state concentration.	(a) Adjust gain.
(b) Pen not inking.	(b) No ink trace.	(b) Change ink, clean pen.
(c) Automatic standardization not working.	(c) Lack of adjustment of baseline.	(c) Adjust pot on back of recorder door to 1.0 and reset baseline to 96%T using colorimeter pot. Check switch position for automatic mode operation.

2. Automatic Programmer and Valves

<u>Trouble</u>	<u>Indicated By</u>	<u>Corrective Action</u>
(a) Lack of sample, plugged valves, plugged sample overflow cell.	(a) Pen driving down scale to 0%T during either the stream or standard cycle, and coming back down scale on one or the other; lack of liquid to system.	(a) Plugged valves, or valves inoperative, clean and repair as needed. Unplug sample overflow cell.
(b) Not cycling between standard and stream.	(b) Steady state signal on stream or baseline.	(b) Check switch on Programmer for automatic mode operation.

3. Environmental Conditions

<u>Trouble</u>	<u>Indicated By</u>	<u>Corrective Action</u>
(a) Drifting baseline.	(a) Rapid baseline drift up or down scale or both.	(a) Keep temperature constant. (The unit is very sensitive to change in temperature, particularly rapid changes in either direction.)
(b) System not being maintained in a clean condition and tubing in worn, aged or dirty condition.	(b) Poor response, lack of 96%T adjustment, poor bubble pattern, oscillating signal to recorder.	(b) Clean system, optics, flow cell platen, pump tubes, filter holder, etc. Replace tubing at proper interval, keep in good condition.
(c) Waste lines plugged, pinched, or not properly set.	(c) Erratic signal response, poor bubble pattern, pulsation in system.	(c) Check waste lines, make sure they flow freely. Change tubing as needed.

APPENDIX
INSTRUMENT MAINTENANCE
FOR TECHNICON AUTOANALYZER

PUMP

Each Visit

(1) Clean rollers with tissue and alcohol and clean pump tube platen of dust, etc. by blowing, particularly at wearing edge.

(2) Check rollers for excessive wear. If roller wear is excessive it will interfere with the operation of the system, and will cause pump tubes to wear rapidly. If excessive - flip chain over to place unworn roller edge where worn roller edge was. Also switch the guide bars to opposite sides.

(3) Any time the pump is to be opened prior to opening it clamp the waste tubes from the colorimeter, the line from the DPC bottle, the line from the Millipore filter and the sample line from the Programmer Valve. This reduces down time and erratic behavior. Be sure to remove all clamps when putting the unit back into operation.

(4) See that the pump tubing is not snaking and that pump is preparing a smooth sample delivery with a good even bubble pattern. If tubing is snaking, open pump and stretch tubing to next hole in endblock.

Weekly

(1) Clean pump tube platten with alcohol.

Other

(1) Every two weeks replace all pump tubing in pump manifold.

(2) Every two weeks clean sample over flow cell, removing sand and grit.

(3) Every 3 months, apply oil to pump, to chain and other appropriate points (lightly). See maintenance guide book routine check chart.

Instrument Maintenance (Continued)

Pump (Continued)

Each Visit

- (5) Check to see that platen is free of oil or other residue and clean. Clean platen with alcohol any time pump is opened.
- (6) Check to be sure oil is present on gear chain.
- (7) Check sample overflow cell to see adequate volume is flowing.

Weekly

Other

STANDARDIZATION

Weekly

- (1) Zero the analyzer and develop a good 96%T baseline on distilled water before starting standard into unit. See the "Colorimeter" section in the Technicon manual.
- (2) Run a known standard of 20 PPB in distilled water into unit. It should read $85\%T \pm 2\%T$. If it does not, further investigation into the cause is needed. See trouble shooting guide in Technicon manual. It will take about 30 min. for the standard to peak.

Monthly

- (1) Run a set of standards made with river water collected at the Priest Rapids Gauge Station. These will be in concentrations of 10, 20, 50, & 100 PPB. Record date, time, and %T of baseline and each standard and give the results to Bill Horton who will give them to Peggy Blumer.

Other

- (1) After each of the following procedures the standard check should be made: a) changing pump tubes, b) changing light bulb in colorimeter, c) cleaning flow cell, d) any major maintenance on pump or colorimeter such as changing rollers, e) any time the performance of the analyzer is in doubt.

FILTERS

Each Visit

- (1) Check filter tape to see it is operating properly.
- (2) Check milipore filter to see it is not plugged and is operating properly.

Weekly

- (1) Clean tape filter inlet block, outlet block and tape guides.

Instrument Maintenance (Continued)

RECORDER

Each Visit

- (1) Check chart for proper signal, also to see if it needs to be changed.
- (2) Check ink supply.
- (3) Check to see it is operating in proper concentration range of 5-20 PPB. If above this, immediately contact EMU Supervisor or other appropriate personnel.
- (4) Indicate each check by placing an arrow at exact time of visit and write date, time and initial.
- (5) Check to see if 96%T baseline is being reached by automatic calibration. If not:
 - 1) check pot adjustment on automatic calibration and readjust if necessary, 2) if still unable to attain 96%T clean flow cell optics, filters and light bulb, 3) if not adequate, replace light bulb, 4) if this fails, see trouble shooting guide in Technicon maintenance manual.
- (6) All major maintenance items such as changing light bulbs, replacing pump tubing, standardization, etc. should be indicated on the chart

Twice Weekly

- (1) Readjust automatic calibration pot on Mondays and Fridays. Re-zero and adjust baseline to 96%T on distilled water.

Weekly

- (1) Remove previous weeks' chart and deliver to Bill Horton.

Monthly

- (1) Lubricate and clean as necessary.
- (2) Empty, flush out recorder pen reservoir. Refill with fresh ink.

Instrument Maintenance (Continued)

Recorder (Continued)

<u>Each Visit</u>	<u>Twice Weekly</u>	<u>Weekly</u>	<u>Monthly</u>
(6) along with the time, date, and other pertinent information. Any maintenance that causes a deviation from normal on the chart must be explained.			

COLORIMETER

<u>Each Visit</u>	<u>Weekly</u>	<u>Monthly</u>	<u>Other</u>
(1) Check for leaks and proper operation.	(1) Clean flow cell with cleaning solution and rinse with distilled H ₂ O, wipe dry with tissue.	(1) Replace transmission tubing between modules. (2) Check flow cell alignment in light path. (3) Check drain line from flow cell - be sure it is draining properly.	(1) Change light bulb every 3 months or as needed to attain 96%T.

NOTE: See Technicon Manual for various procedures and methodology.

ENVIRONMENTAL MONITORING MAINTENANCE
FOR TECHNICON AUTOANALYZER

Daily

- (1) Check performance of analyzer to see it is operating properly and is operating in proper range of 85 to 96%T. If out of this range contact EMU Supervisor. Also see sample of chart on wall which indicates what normal operation looks like - if not operating properly contact EMU Supervisor.
- (2) See that tape filter is tracking properly, if not, adjust.
- (3) Remove used tape filter material and dump unless notified otherwise.
- (4) The tape filter will need replacing periodically. It requires 8 ft/24 hrs. (a) shut the pump off when replacing the filter (b) place the filter on the center of the spool and be sure the tape is not touching the main filter roll as this will cause wetting of the filter paper. This is the purpose of the clear plastic guide bar above the filter (c) see the Technicon Manual under Continuous Filter for further details.
- (5) Write date and time of each check on chart and any maintenance performed.

Twice Weekly

- (1) Replenish diphenylcarbazide (DPC) solution as needed. Two 1-liter bottles will be obtained every other week and 3 1-liter bottles on the other weeks from chemical standards lab in 325 Bldg. Bottles not in used must be kept refrigerated until used. Use in sequence.
- (2) The unit uses DPC at the rate of slightly over 1 liter/3 days. When the unit is to be left 3 days or more it will require pouring the DPC into a larger bottle. When only a small amount of DPC is left at the time for addition it should be dumped.
- (3) Turn the pump off when changing bottles.
- (4) Change the milipore filter on Mondays and Thursdays. (a) shut the pump off when changing the filter (b) Caution: the filter is very fragile and requires care in placement (c) wet the filter before placing it in the filter holder, be sure a good seal without air bubbles is obtained between the glass frit and the filter (d) place the rubber gasket over the filter, place the glass top on this and clamp with the 8 clamps provided (e) check the filter after it has been placed in filter holder for any breaks

Weekly

- (1) The distilled water will be replaced on Fridays alternating the two large plastic jugs. When one jug is not being used, it should be rinsed with fresh distilled water and aired upside down.
- (2) Turn the pump off when changing jugs.

Environmental Maintenance (Continued)

Daily

Twice Weekly

Weekly

(4) [cont.] in the filter (f) level the filter and start pump, watch filter operation to be sure it is functioning properly (g) care must be taken not to break the filter holder as it is quite expensive.

FIGURE 1
Automatic Columbia River Monitoring Station

FIGURE 2
Water Flow for ACRMS Sample Systems

FIGURE 3
Schematic of Auto Analyzer System

1. Voltage stabilizer
2. Distilled water
(Chamber)
3. Automatic frequency
4. Control panel

1. Continuous Tape Filter

2. Continuous Filter

3. Continuous Filter

4. Continuous Filter

5. Continuous Filter

6. Continuous Filter

7. Continuous Filter

8. Continuous Filter

9. Continuous Filter

10. Continuous Filter

11. Continuous Filter

12. Continuous Filter

13. Continuous Filter

14. Continuous Filter

15. Continuous Filter

16. Continuous Filter

17. Continuous Filter

18. Continuous Filter

19. Continuous Filter

20. Continuous Filter

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25. Continuous Filter

26. Continuous Filter

27. Continuous Filter

28. Continuous Filter

29. Continuous Filter

30. Continuous Filter

31. Continuous Filter

32. Continuous Filter

33. Continuous Filter

34. Continuous Filter

35. Continuous Filter

Figure 4
see page 20

If alarm-disable is required, there will be a third microswitch connected to the timer.

FIGURE 6
Programmer Valves

ON/OFF POWER SWITCH

APPENDIX 4

PROPOSED POLYMERIZATION

1. Mixing Coils
2. End Blocks
3. Pump Tubes
4. Mixing Cactus Fitting
5. Manifold Tray

FIGURE 11
Flow Diagram for Cr^{+6} Analysis

COLORIMETER SHOWN WITH

FIGURE 13
Range Expander

FIGURE 14
Recorder and Control

Stabilizer Transformer

Multiple Receptacle Power Strip

FIGURE 15
Power Strip and Voltage Stabilizer

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