



## INLAND WATERS BRANCH

DEPARTMENT OF ENERGY, MINES AND RESOURCES

# *An Automated Method for Determining Mercury in Water*

*P. D. Goulden and B. K. Afghan*

TECHNICAL BULLETIN NO. 27



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Mercury in Water*

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DEPARTMENT OF ENERGY, MINES AND RESOURCES  
OTTAWA, CANADA, 1970

## TABLE OF CONTENTS

	Page
SUMMARY . . . . .	v
INTRODUCTION . . . . .	1
EXPERIMENTAL . . . . .	1
Laboratory Apparatus . . . . .	2
Reagents . . . . .	2
Instrument Settings . . . . .	3
Procedure . . . . .	3
Other Equipment . . . . .	4
Instrument Settings - Model 303 . . . . .	4
RESULTS AND DISCUSSION . . . . .	4
Organo-Mercury Compounds . . . . .	6
Preservation of Samples . . . . .	7
Interferences . . . . .	8
REFERENCES . . . . .	8

## TABLES

Table 1. Coefficients of variation at different levels of mercury .	5
Table 2. Apparent mercury content of water from Saskatchewan River, irradiated with ultraviolet light and unirradiated . . .	6
Table 3. Apparent mercury level, per cent of total, in various mercury compound solutions . . . . .	7

# ILLUSTRATIONS

	Page
Figure 1. Manifold arrangement. . . . .	9
Figure 2. Gas separator . . . . .	10
Figure 3. Arrangement of equipment. . . . .	11
Figure 4. Recorder plot with standard solutions of mercury (2 - 32.0 $\mu\text{g/l}$ ) . . . . .	12
Figure 5. Calibration curve 0 - 32 $\mu\text{g/l}$ of mercury. . . . .	13
Figure 6. Recorder plot with standard solutions of mercury (0.5 - 16 $\mu\text{g/l}$ ) . . . . .	14
Figure 7. Calibration curve 0 - 16 $\mu\text{g/l}$ of mercury. . . . .	15
Figure 8. Recorder plot with standard solutions of mercury (0.05 - 2.0 $\mu\text{g/l}$ ) . . . . .	16
Figure 9. Calibration curve 0 - 2 $\mu\text{g/l}$ of mercury . . . . .	17
Figure 10. Recorder plot with standard solutions of mercury (2 - 16 $\mu\text{g/l}$ of $\text{HgCl}_2$ and 1 - 20 $\mu\text{g/l}$ of PMA) . . . . .	18
Figure 11. Calibration curve 0 - 20 $\mu\text{g/l}$ of mercury ( $\text{HgCl}_2$ and PMA solutions). . . . .	19
Figure 12. Recorder plot with standard solutions of mercury (0.1 - 0.4 $\mu\text{g/l}$ ) . . . . .	20
Figure 13. Recorder plot with 0.5, 2 and 8 $\mu\text{g/l}$ of mercury . . . . .	21

## SUMMARY

This report describes a method for determining the mercury content in water with mercury concentrations as low as 0.05  $\mu\text{g}/\text{l}$ . The method has been applied to water samples containing both inorganic-and organo-mercury compounds. It involves the oxidation of organo-mercury compounds by ultraviolet irradiation followed by the reduction of the mercury to the elemental state. The mercury is swept out of the solution by a stream of air and its concentration measured in an absorption cell in an atomic absorption spectrophotometer. For levels of mercury in the range 1.0 to 32  $\mu\text{g}/\text{l}$  the method has been automated to measure 20 samples per hour. For levels of mercury in the range 0.05 to 1.0  $\mu\text{g}/\text{l}$  only 10 samples per hour can be processed because a longer sampling time is required.

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## INTRODUCTION

With the growing concern for the possible effects of mercury contamination in groundwater and surface water, the Water Quality Division has been in search of a method suitable for monitoring the mercury content of water anywhere in Canada.

There are several methods available to measure mercury, for example, neutron activation; spectrophotometric methods with such reagents as dithizone; atomic absorption spectrophotometry; etc. The method that appears most likely to give the desired sensitivity and be best suited to the analysis of water is that described by Hatch and Ott (1968). In this method, the mercury in solution is reduced to elemental mercury; this elemental mercury is swept from the solution in a current of air and the absorption of the mercury-containing air stream is measured at 253.7 m $\mu$  in an absorption cell placed in an atomic absorption spectrophotometer.

The following procedure has been used to analyse water samples containing both inorganic and organo-mercury compounds. Using automated equipment to feed the air stream to the Atomic Absorption Spectrophotometer, 20 samples per hour can be analysed in the range 1.0  $\mu\text{g/l}$  to 32  $\mu\text{g/l}$  and 10 samples per hour, in the range 0.05  $\mu\text{g/l}$  to 1.0  $\mu\text{g/l}$ .

## EXPERIMENTAL

The reducing system used by Hatch and Ott, that is, reduction with stannous sulphate in a hydroxylamine sulphate - sodium chloride solution using a mixture of sulfuric and nitric acid to acidify the solutions, was found to give a satisfactory sensitivity. Other reducing agents such as stannous chloride and hypophosphorous acid were tried but they did not improve the sensitivity of the method.

The effect of changing the flow rate of the reagents by  $\pm 50\%$  was investigated to make sure that the continuous flow system used was a stable one. There were no significant changes in the response when these  $\pm 50\%$  changes were made.

Organo-mercury compounds do not give a response in the method unless they are first broken down to give mercuric ions. Following the suggestion of Armstrong, Williams and Strickland (1966), the organo-mercury compounds are broken down by irradiation with ultraviolet light. This technique has the advantage that it eliminates the need for large amounts of reagents that

would be required to chemically destroy the organo-mercury compounds, and consequently, the need for "blank" corrections; it also eliminates the need for heating the sample in a digestion step with consequent possible volatilization of mercury.

#### Laboratory Apparatus

Atomic Absorption Spectrophotometer: "Perkin-Elmer" Model 403 equipped with "Perkin-Elmer" Model 165 Recorder.

Absorption Cell: Made from borosilicate glass tubing 10 mm. I.D. 100 mm long. The ends were ground square and quartz windows were attached with epoxy cement. Gas inlet and outlet ports were attached approximately 5 mm from the ends of each cell.

Metering Pump: A "Technicon" "AutoAnalyzer" proportioning pump.

Sampler: A "Technicon" "AutoAnalyzer" sampler.

Gas Separator: A gas separator made from borosilicate glass, as shown in Figure 2.

Ultraviolet Irradiation Apparatus: This apparatus is similar to that described by Armstrong and Tibbitts (1968). A 550-watt mercury arc lamp is used; the irradiation tubes are made of silica tubing 23 mm O.D. which allows 12 tubes to be placed around the lamp. (The maximum temperature reached in the tubes is 45°C).

Small Reading Lamp with 60-Watt Bulb: This is arranged to shine on the absorption cell, which maintains the temperature in the cell about 10°C above ambient and prevents condensation of moisture inside the cell.

The equipment is arranged as shown in Figure 1 and in the photograph in Figure 3. It is found convenient to arrange the sampler, pump, reagent bottles, etc. on a small laboratory cart which can be pushed up to the spectrophotometer when required.

The absorption cell is attached to the top of one of the atomic absorption burners in a small wooden frame fixed on the burner with masking tape. The gas connections are made to the cell with "Technicon" transmission tubing.

#### Reagents

- 1) Hydroxylamine Sulphate - Sodium Chloride Solution: dissolve 15 g. of hydroxylamine sulphate and 15 g. of sodium chloride in 500 mls water. (This solution is made up daily).
- 2) Stannous Sulphate: dissolve 25 g. of stannous sulphate and 5 g. sodium chloride in 250 mls of 2N sulfuric acid. Add about 1 g. of mossy tin. (This solution is stable for about two weeks).
- 3) Stock Inorganic Mercury Solution (0.1% Hg): dissolve 0.1354 g. of mercuric chloride in 100 mls of 1N sulfuric acid. Dilute this stock solution with 1N sulfuric acid to prepare appropriate mercury standards. These dilute standard solutions are prepared daily.

- 4) Stock Organo-Mercury Solution (0.01% Hg): dissolve 0.0168 g. of Phenyl Mercuric Acetate in 100 mls of IN sulfuric acid. Dilute this stock solution with IN sulfuric acid to prepare appropriate mercury standards. (These dilute standards are prepared daily).
- 5) Hydrogen Peroxide: 30% solution.
- 6) Wash Solution: to 800 mls of water add 120 mls of sulfuric acid (96%) and 80 mls of nitric acid (70%); add the acids slowly while stirring. Cool to room temperature.

#### Instrument Settings

"Perkin-Elmer" Model 403 Atomic Absorption Spectrophotometer equipped with a "Perkin-Elmer" Model 165 Recorder.

Lamp Current - 10 mA

Wavelength - 253.7 mμ

Slit Setting - 6

Concentration Dial - 50

Recorder Full Scale 4A, 2A, 1A

4A for 5-32 mg/l range

2A for 2-16 mg/l range

1A for 0.05 - 2 mg/l range

Recorder Response - 3

Mode - "Concentration" "10 Average"

Chart Speed - 5 mm/min.

#### Procedure

Prepare standard solutions of inorganic mercury to cover the anticipated range (40 mls of each standard solution are required). To each of these standard solutions add 6.0 mls of sulfuric acid (96%), 4 mls of nitric acid (70%) and 2 drops of hydrogen peroxide (30%). Transfer the solution to the silica tubes and irradiate in the ultraviolet irradiator for two hours. Check that the temperature in the tubes does not exceed 45°C. Cool the solutions to room temperature and load into the "Technicon" sample tray. Treat the samples as above and run with the standards at 20 samples per hour using the manifold shown in Figure 1 and the settings for the 0 to 16 μg/l on the "Perkin-Elmer" Model 403 Atomic Absorption Spectrophotometer. If the samples contain more than 16 μg/l the samples may be re-run with the larger recorder range or the irradiated solution may be diluted and re-run. If the samples contain less than 1 μg/l they may be re-run using the 10 samples an hour cam and the 1A setting on the "Recorder Full Scale" dial. Samples containing up to 2.0 μg/l may be run with the longer sampling time and higher sensitivity if it is desired to obtain higher precision. Periodically, standard organo-and inorganic-mercury solutions are run side by side with an irradiation time of one hour to confirm the proper functioning of the ultraviolet irradiation process.



### Other Equipment

Some mercury analyses were also performed using a "Perkin-Elmer" Model 303 Atomic Absorption Spectrophotometer, equipped with a "Sargent" Model SRL laboratory recorder. An absorption cell 80 mm long and 12 mm in diameter was used. With the settings shown below, measurements of mercury in the range 0.2 to 20  $\mu\text{g/l}$  were obtained with essentially the same equipment as used with the "Model 403". It was found that this particular instrument had an electronic noise level that made it impossible to gain further amplification and greater sensitivity. By using an absorption cell that was 160 mm long and manually feeding the sample to the "Technicon" pump for a five minute sampling period, it was possible to detect 0.05  $\mu\text{g/l}$  of mercury. However, the action being taken in this laboratory is to upgrade the electronics by stabilizing the power source and replacing parts where necessary to approach the same sort of stability obtained with the "Model 403". There is nothing inherent in the instrument design to prevent this.

#### *Instrument Settings - "Model 303"*

##### Spectrophotometer Settings:

Lamp Current 10 mA

Wavelength 253.7 m $\mu$

Slit Setting 5

Scale X3

Noise Suppression 2

##### Recorder Settings:

Linear Mode

Scale Selector mV

Range 10 mV

Chart Speed - Slow - 0.2 inch per min.

### RESULTS AND DISCUSSION

Typical recorder plots of the measurements made on the standard solutions to establish calibration curves are shown in Figures 4, 6, 8 and 10. Typical calibration curves are shown in Figures 5, 7, 9 and 11. Typical plots at 0.5  $\mu\text{g/l}$ , 2  $\mu\text{g/l}$  and 8  $\mu\text{g/l}$  are shown in Figure 13.

The coefficients of variation at different levels of mercury were determined. These results, together with the 10 individual results used in calculating these coefficients are shown in Table 1.

Recovery of mercury "spiked" into river and groundwater samples as mercuric chloride, methyl mercuric bromide and phenyl mercuric acetate solutions, was complete within the experimental errors shown in the following table.

TABLE 1

## Coefficients of Variation at Different Levels of Mercury

Level Hg $\mu\text{g/l}$	Instrument	Range $\mu\text{g/l}$	Results	Mean	Coefficient of Variation %
0.5	"403"	0.05 - 1.0	0.49, 0.48, 0.50, 0.51, 0.51, 0.50, 0.50, 0.56, 0.49, 0.52	0.51	4.4
2.0	"403"	0 - 16	2.00, 1.96, 1.82, 1.90, 1.92, 2.08, 1.80, 2.20, 2.04, 2.01	1.97	6.1
8.0	"403"	0 - 16	8.05, 8.15, 8.20, 7.67, 8.04, 7.76, 7.80, 7.67, 8.57, 7.78	7.97	2.6
7.2	"303"	0 - 20	7.25, 7.35, 7.25, 7.10, 6.75, 7.70, 7.10, 7.25, 7.00, 7.10	7.19	3.4

From these results it can be seen that the method is sufficiently precise for water monitoring purposes.

The detection limit of the method on the 0 - 1  $\mu\text{g/l}$  range is about 0.05  $\mu\text{g/l}$  (where detection limit is defined as the concentration that gives a signal that is twice the level of the baseline "noise").

The greatest problem with the method is the return of the absorbance reading to the baseline between samples. Where deflections of more than 30 scale divisions are seen with the 20 samples per hour cam, the pen does not return to the original baseline before the next sample is seen. The shape of the peaks is quite characteristic at the different mercury levels and it has been found a satisfactory technique to project the "tail" of a peak and measure the next peak height from this "tail" as shown in Figure 13.

A return to the baseline can be achieved if wash solution is alternated with samples in the sample cups, but this reduces the sample rate to 10 per hour. In the Water Quality Division's laboratory, the technique described above is used in preference to accepting the reduction in sample rate. In order to reduce the "tailing", the volume of the absorption cell has been reduced to the minimum, consistent with sensitivity, and the total volume of the gas-containing apparatus also has been reduced to the minimum. The drying column used by Hatch and Ott was eliminated for this same reason; it was found not to be necessary if the absorption cell temperature was maintained above ambient.

Of the water samples obtained throughout Canada, the mercury content in almost all samples has been in the range 0 to 10  $\mu\text{g/l}$  and the technique described here has been adequate to determine that content.

## Organo-Mercury Compounds

Workers in Sweden (Westoo and Norea, 1967; Sernelov, 1969) have shown that although mercury may reach the waters as divalent mercury or phenyl mercury salts, it is quickly converted to methyl mercury compounds, presumably by microbiological action. The organo-mercury compounds do not react in the reduction-reaction process unless they are first oxidized to (presumably) mercuric ion. Table 2 shows the apparent mercury content of water samples from various rivers in Saskatchewan, analyzed both with and without the ultraviolet irradiation step. It is seen that the levels found "with ultraviolet irradiation" are consistently higher than those found "without ultraviolet irradiation" and that approximately 60% of the mercury does not react in the reduction-reaction step. This part of the mercury is presumably present as organo-mercury compounds.

TABLE 2  
Apparent Mercury Content of Water from Saskatchewan Rivers  
Irradiated with UV Light and Unirradiated

Sample	Apparent Mercury Level $\mu\text{g/l}$	
	Irradiated	Unirradiated
1	5.0	2.1
2	5.6	1.8
3	4.5	1.2
4	4.8	0.8
5	8.5	2.1
6	8.6	2.8
7	4.3	2.5
8	7.6	7.6
9	11.1	2.6
10	6.1	3.1
11	3.1	2.5
12	3.3	3.4
13	3.3	2.5
14	3.5	2.8
15	2.8	2.1
16	3.5	1.2
17	2.8	0.0
18	3.2	0.8
19	3.1	0.8
20	3.8	0.5
21	3.5	1.2
22	1.6	0.8
23	1.7	0.9
Average	4.6	2.0

To determine the breakdown rate of specific organo-mercury compounds likely to be present in water, solutions of phenyl mercuric acetate, methyl mercuric bromide, dimethyl mercury, ethyl mercuric chloride and diethyl

mercury (containing about 10 µg/l of mercury) were irradiated for various periods of time and the apparent amounts of mercury present in the solution were measured. These measurements are shown in Table 3. All of these compounds break down to release all of the mercury in a measurable form after irradiation for one hour. Phenyl mercuric acetate (PMA) breaks down directionally more slowly than the other compounds studied and, because of its ready availability as a pure chemical, it is selected as the organo-mercury compound to be used as a continuing check on the effectiveness of the ultra-violet irradiation. Figure 11 shows the results of analyses of mercuric chloride and phenyl mercuric acetate solutions; the results for the two materials lie on the same calibration curve.

TABLE 3

Apparent Mercury Level, Per Cent of Total, in Various  
Mercury Compound Solutions

	Apparent Mercury Level - % of Total					
Irradiation Time-Mins.	0	10	20	30	40	60
Compound						
Phenyl Mercuric Acetate	5	60	75	90	95	100
Methyl Mercuric Bromide	0	70	85	95	100	-
Di - Methyl Mercury	5	70	90	100	-	-
Ethyl Mercuric Chloride	50	80	95	100	-	-
Di - Ethyl Mercury	40	70	90	100	-	-

There may be innumerable organo-mercury compounds present in the samples being analyzed and it is possible that some of these are slower to break down than the particular materials studied. For this reason a safety factor of two is included in the analytical procedure and an irradiation time of two hours is used. Approximately 20 of the water samples in Table 2 were irradiated for four hours; there was no significant increase in the apparent mercury level for this four hours over the normal two hour irradiation time.

#### Preservation of Samples

Most of the water samples are obtained in polyethylene bottles. It has been found that there is a rapid loss of mercury from solution in samples that are stored in these bottles. (At the 10 µg/l level, the loss is 40% in four days). It is presumed that the loss is by adsorption on the plastic, but it has not been possible to recover the mercury from the plastic by acid leaching. In order to avoid this loss and to obtain a valid analysis, it is necessary to acidify the sample to below pH 1 when the sample is taken. A practice found convenient in the Water Quality Division's laboratory is to add 1% by volume of 96% sulfuric acid to the sample.

### Interferences

The common anions and cations found in natural waters have been added to synthetic samples to determine possible interferences with the method. Calcium, magnesium, ammonium, zinc, copper, lead, silver, iron, cadmium, sulphide, cyanide, chromate, thiocyanate, sulphite were added to river water in amounts of about 1000 mg/l. Of these ions, copper and sulphide were the only ones which gave interference. Copper interferes because it is more readily reduced than mercury. However, at the 100 mg/l level of Cu, there is sufficient stannous sulphate added to reduce the copper and the mercury, and at this level, copper does not interfere. Sulphide interferes by the precipitation of mercuric sulphide which does not react in the reduction-reaction step. Sulphide becomes readily oxidized in the ultraviolet irradiation step and, at 100 mg/l levels, becomes completely oxidized using the technique described; it does not then interfere with the mercury determination.

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- Hatch and Ott, 1968. Analytical Chemistry 40: 2085.
- Sernelov, 1969. Vatten 3: 304-309.
- Westoo and Norea, 1967. Van Foda: 135-78.

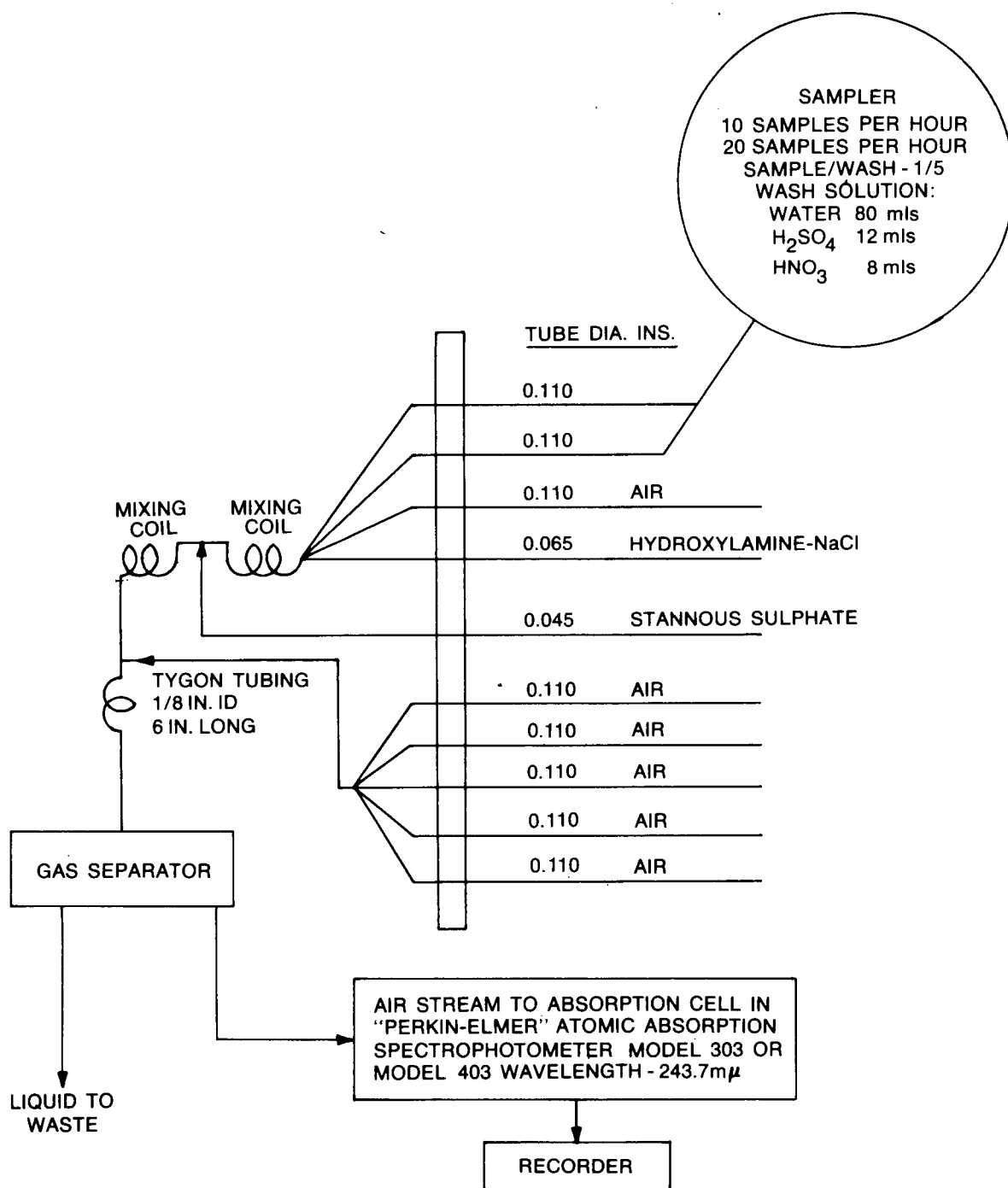


Figure 1. Manifold arrangement.

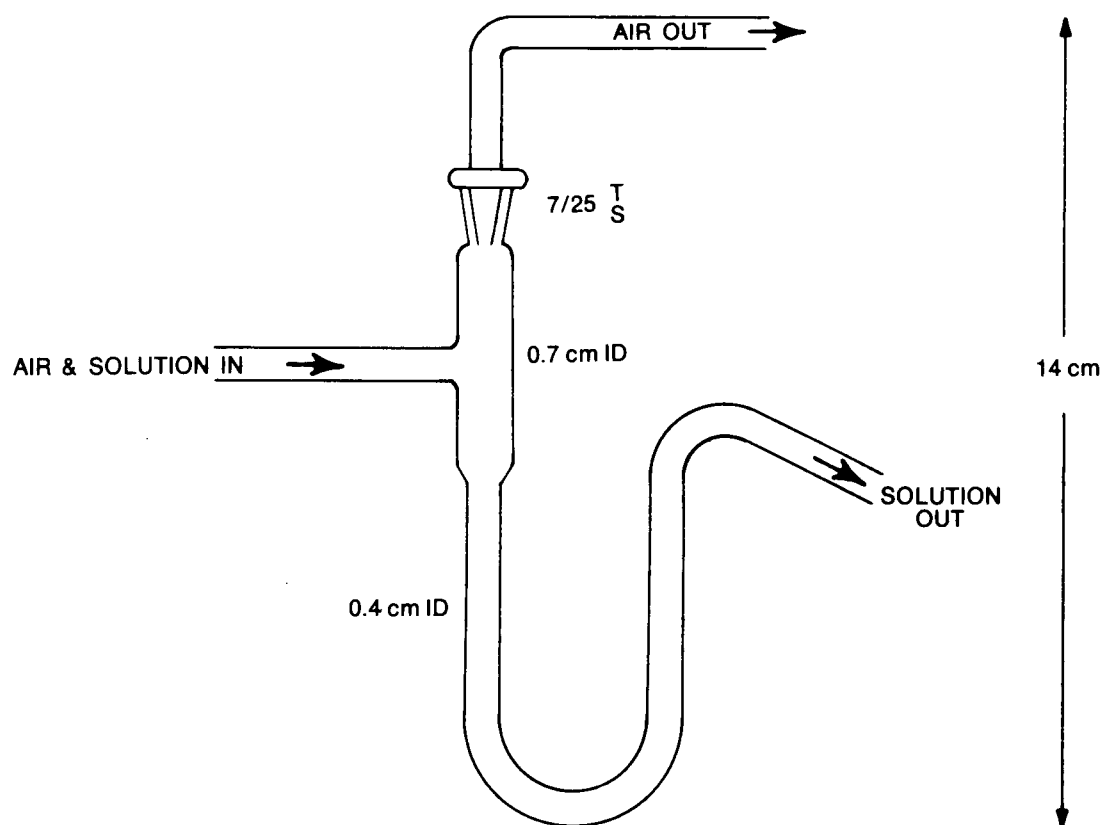


Figure 2. Gas separator.

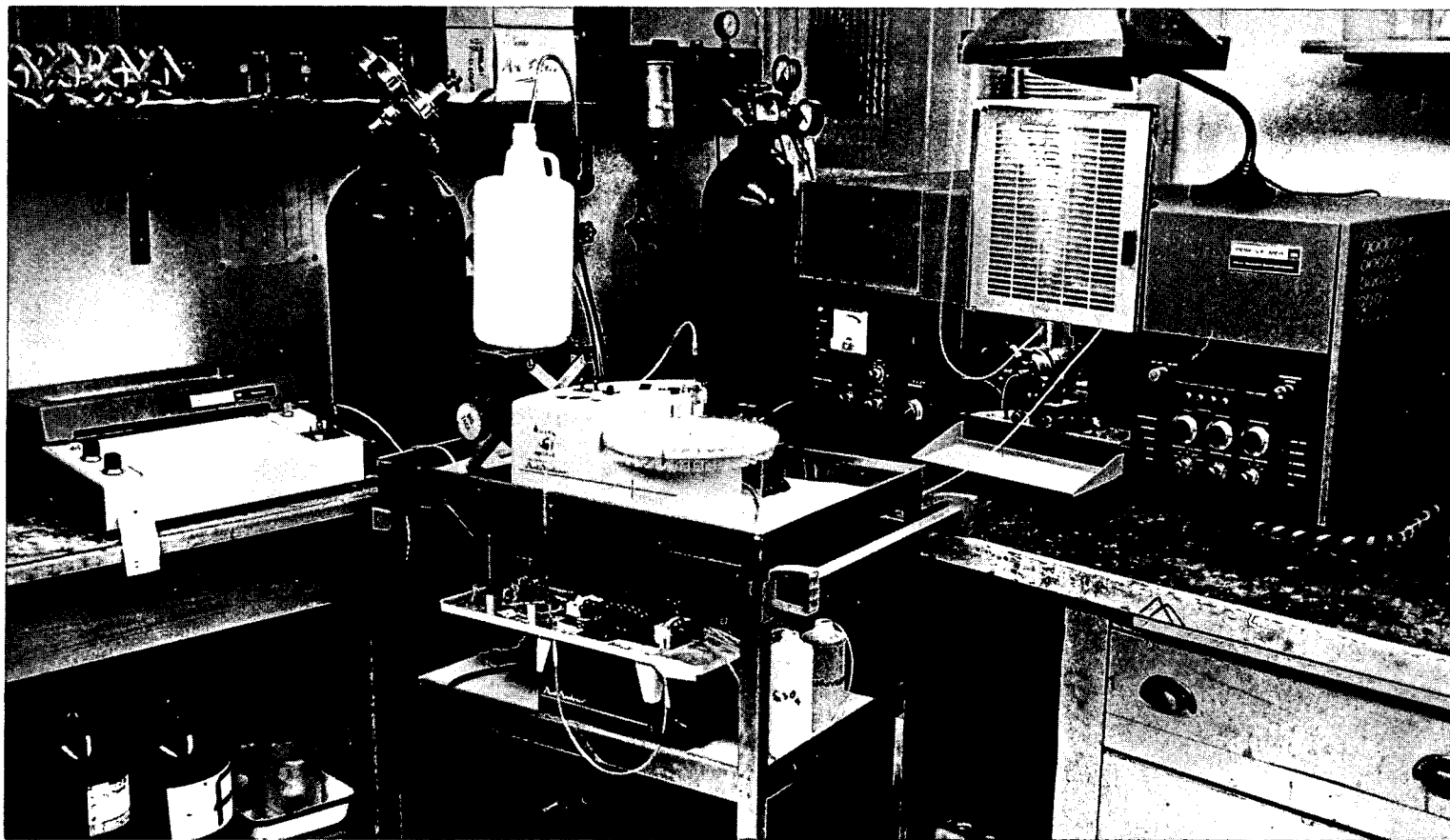


Figure 3. Arrangement of equipment.



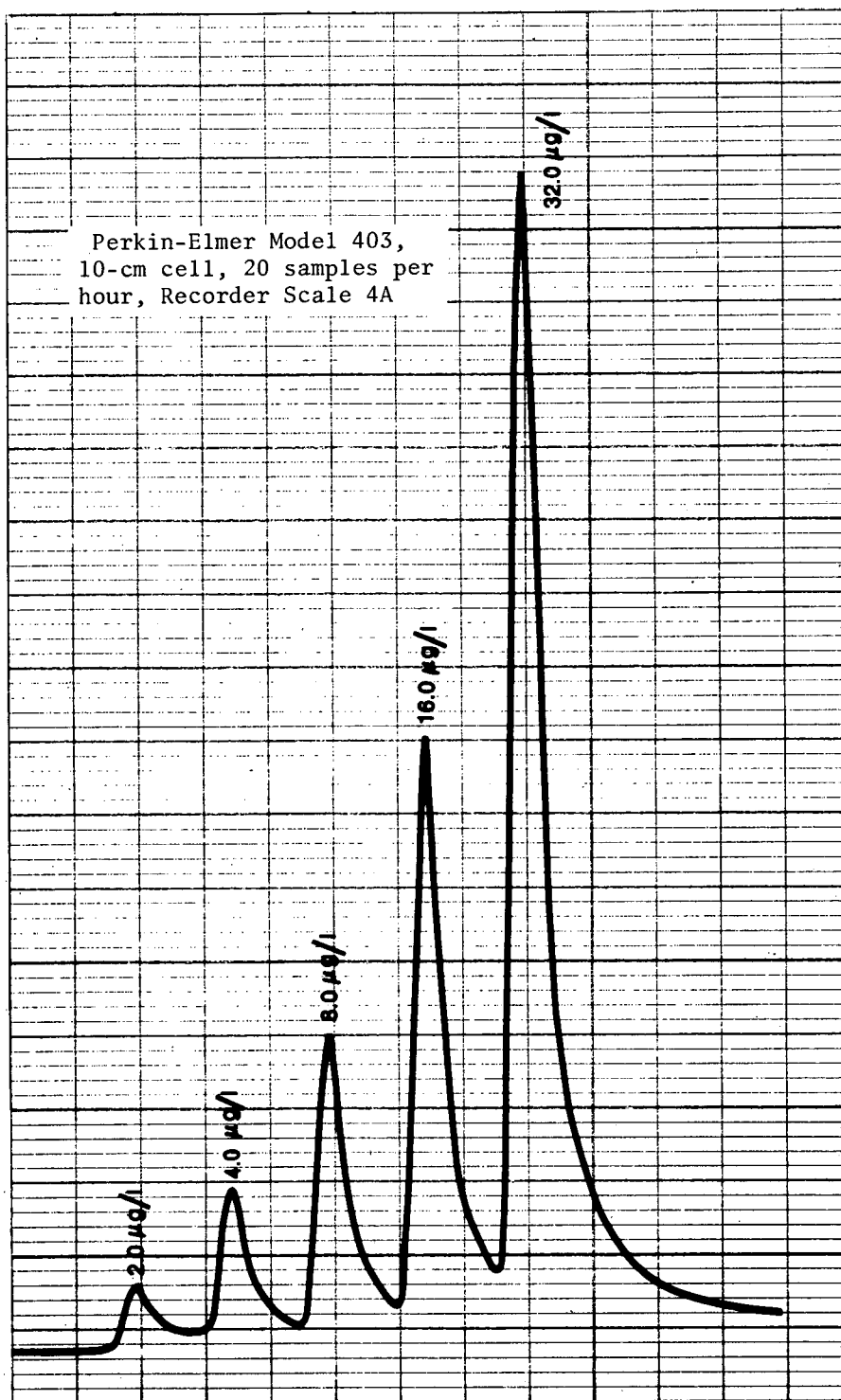


Figure 4. Recorder plot with standard solutions of mercury.

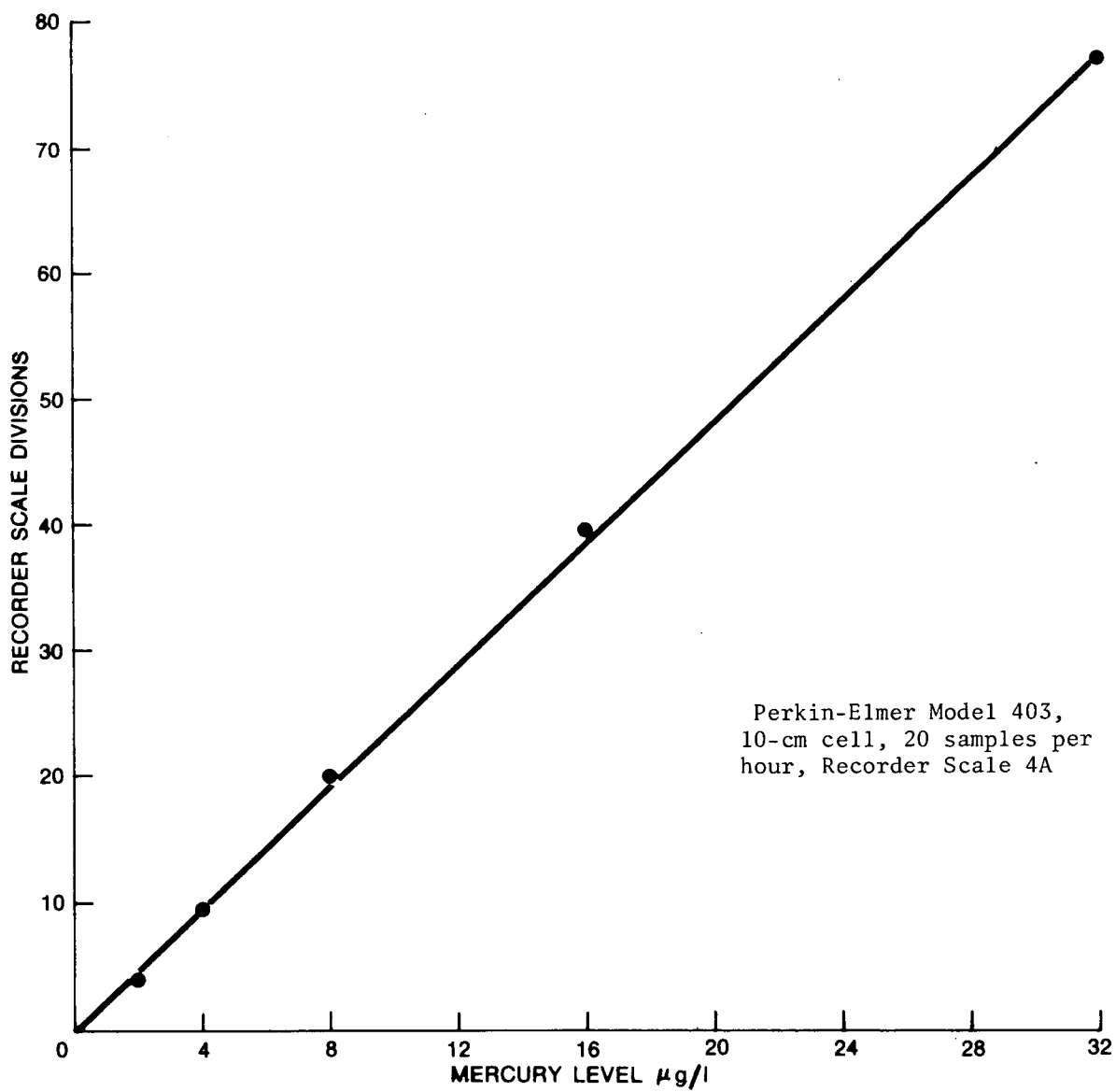


Figure 5. Calibration curve 0 - 32  $\mu\text{g/l}$  of mercury.

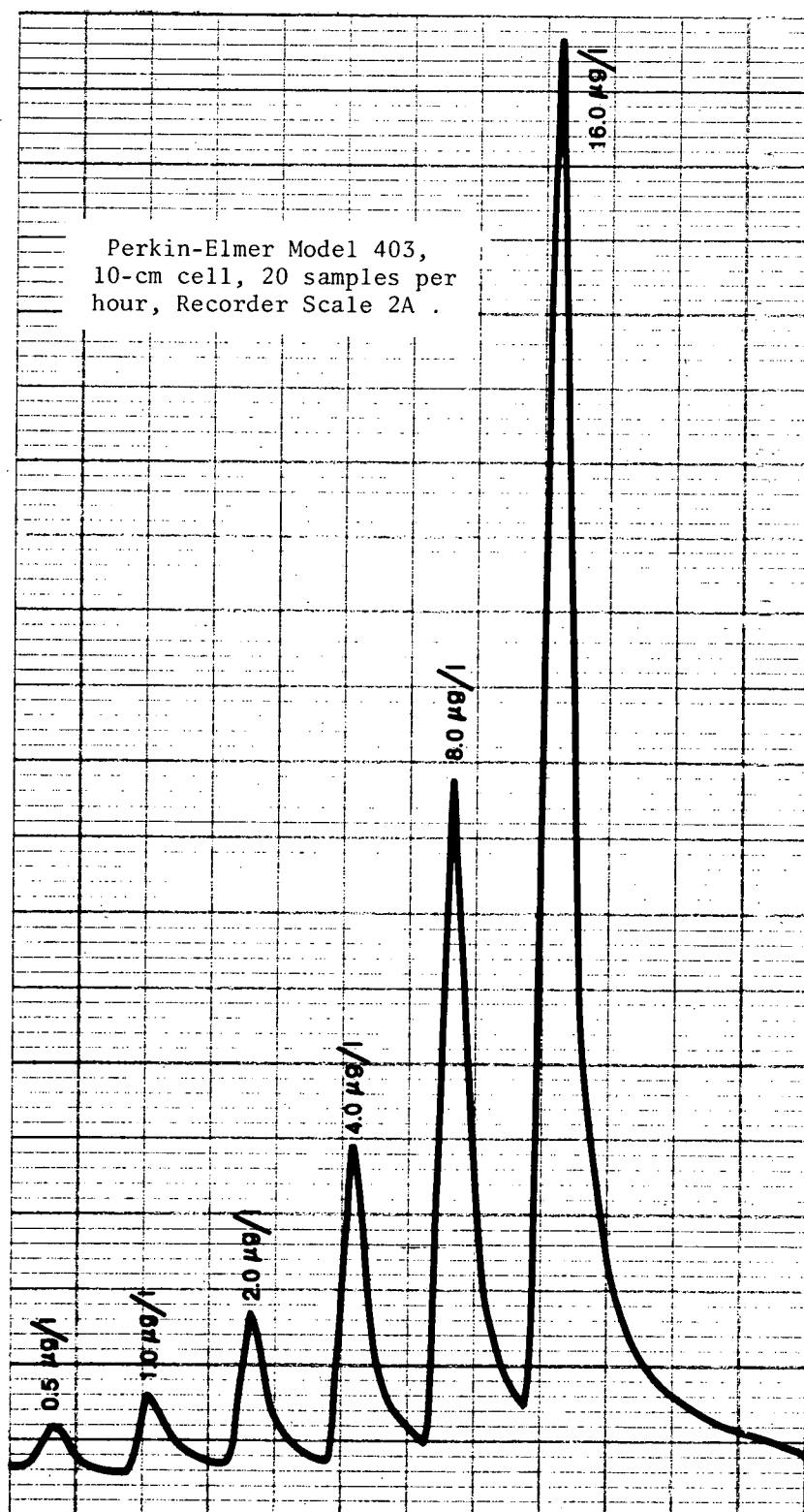


Figure 6. Recorder plot with standard solutions of mercury.

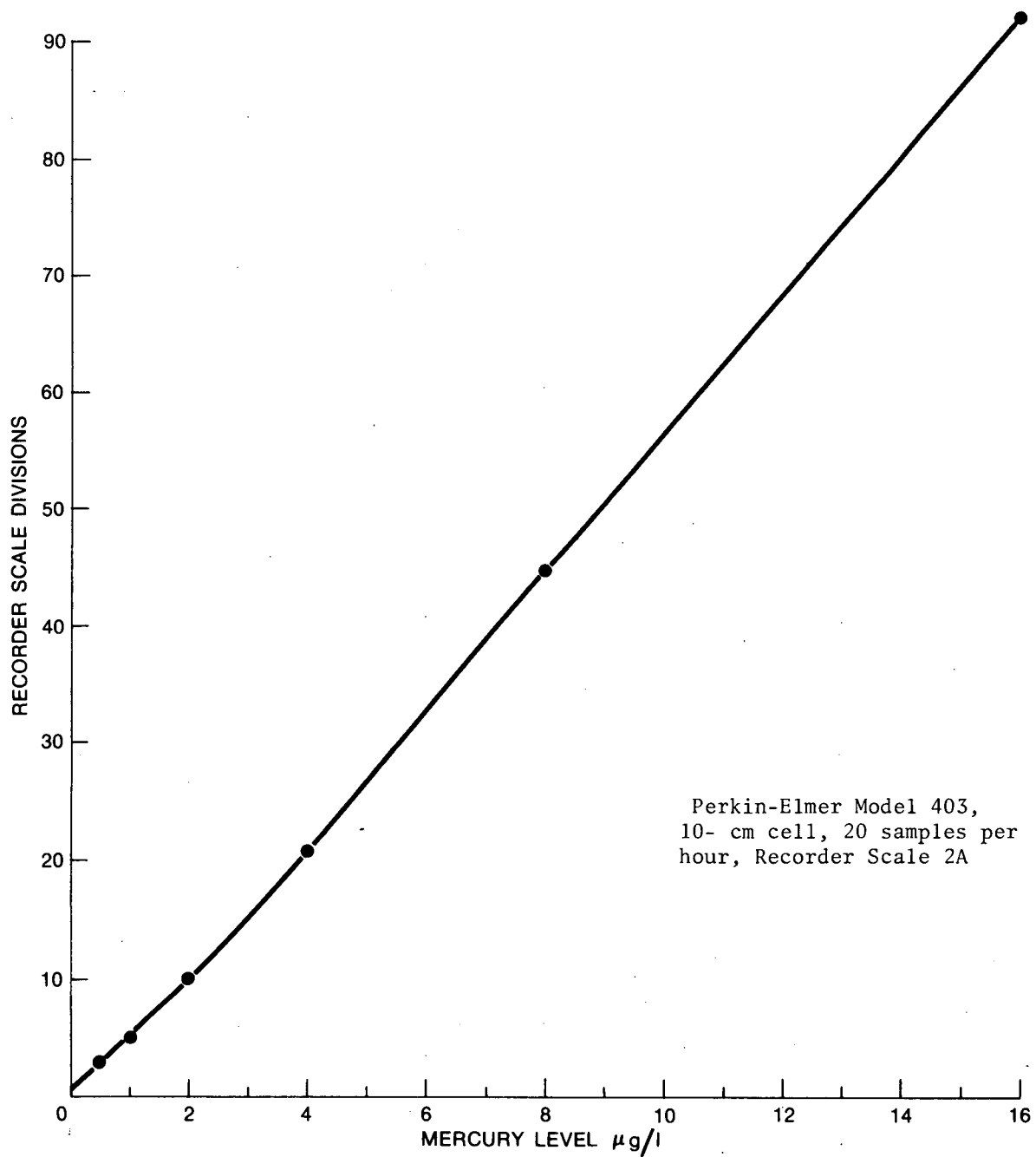


Figure 7. Calibration curve 0 - 16  $\mu\text{g/l}$  of mercury.

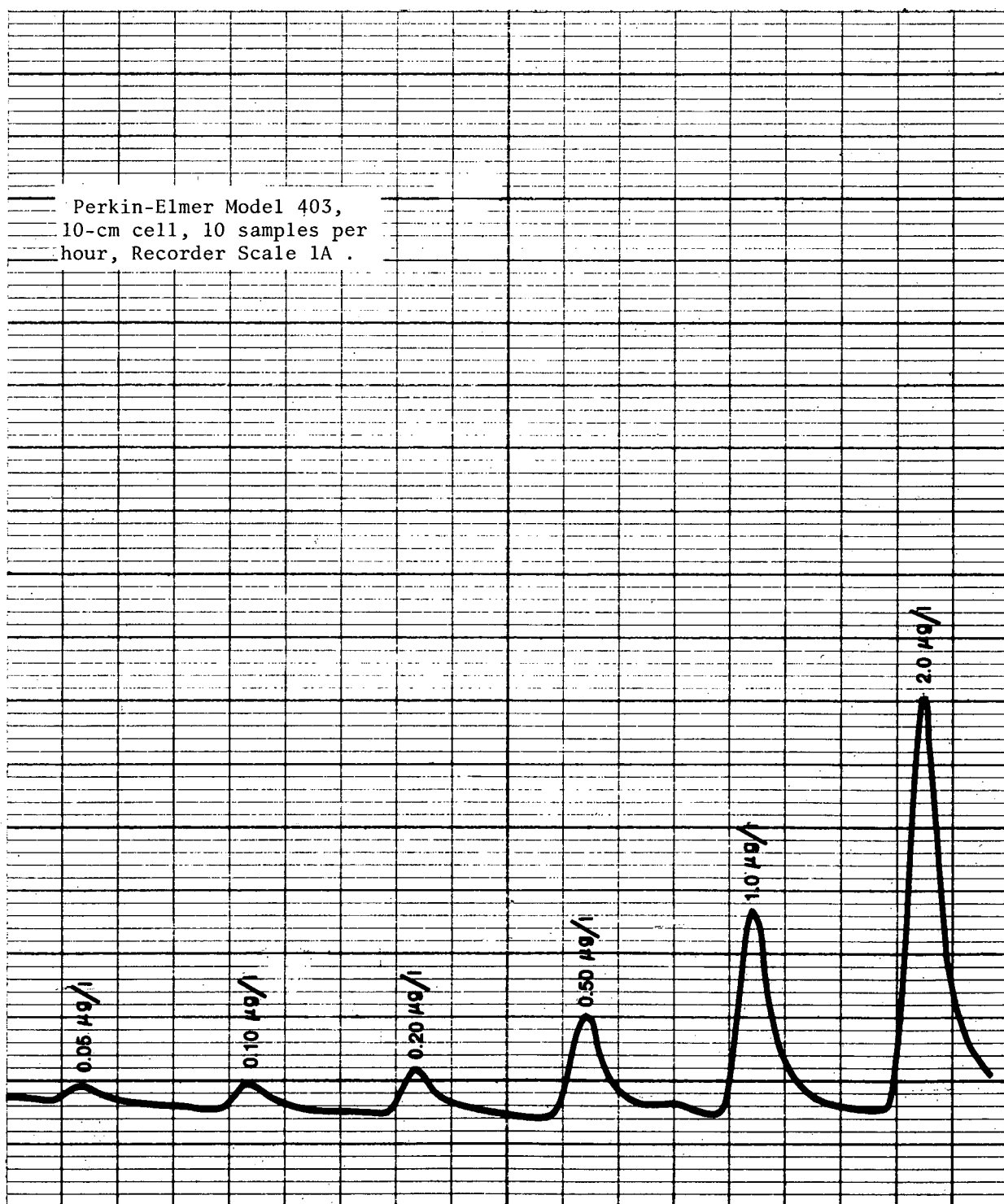


Figure 8. Recorder plot with standard solutions of mercury.

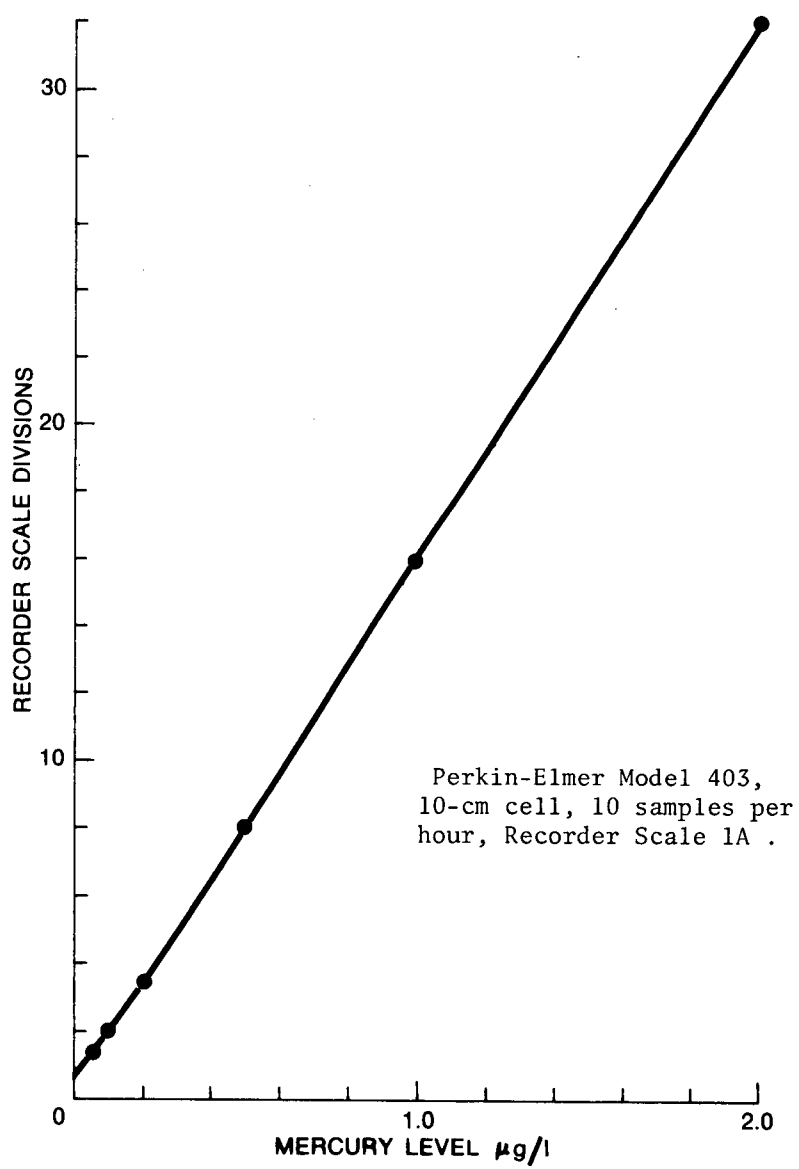


Figure 9. Calibration curve 0 - 2  $\mu\text{g/l}$  of mercury.

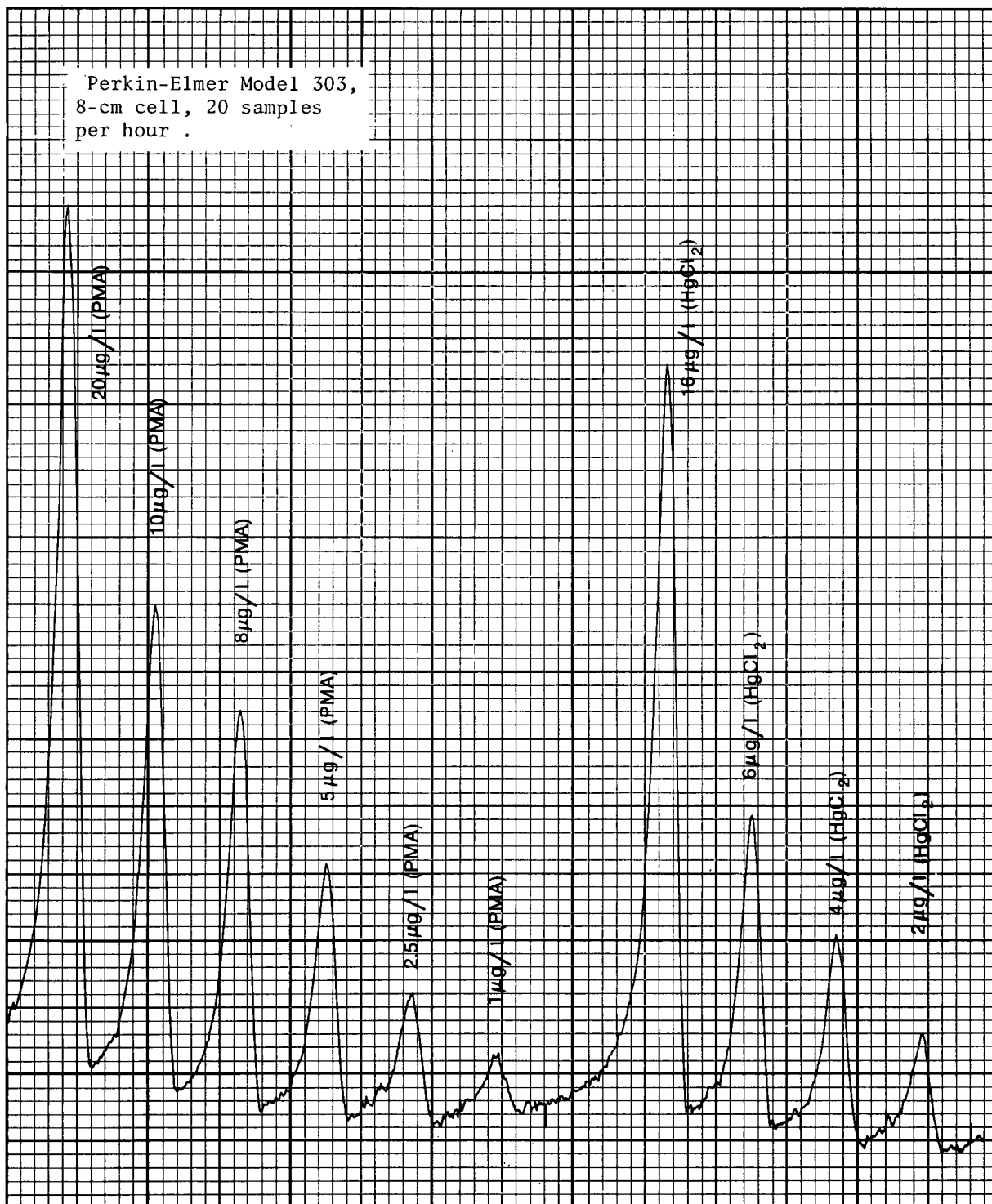


Figure 10. Recorder plot with standard solutions of mercury.

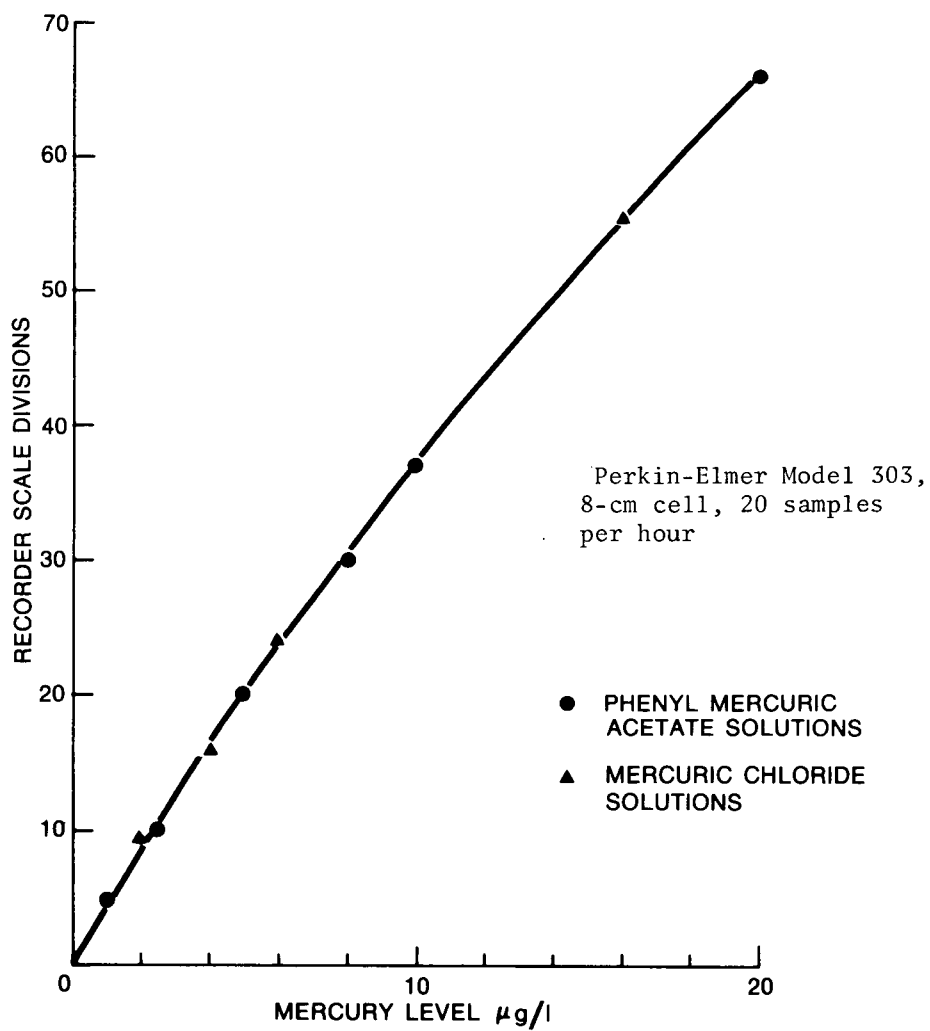


Figure 11. Calibration curve 0 - 20  $\mu\text{g/l}$  of mercury.



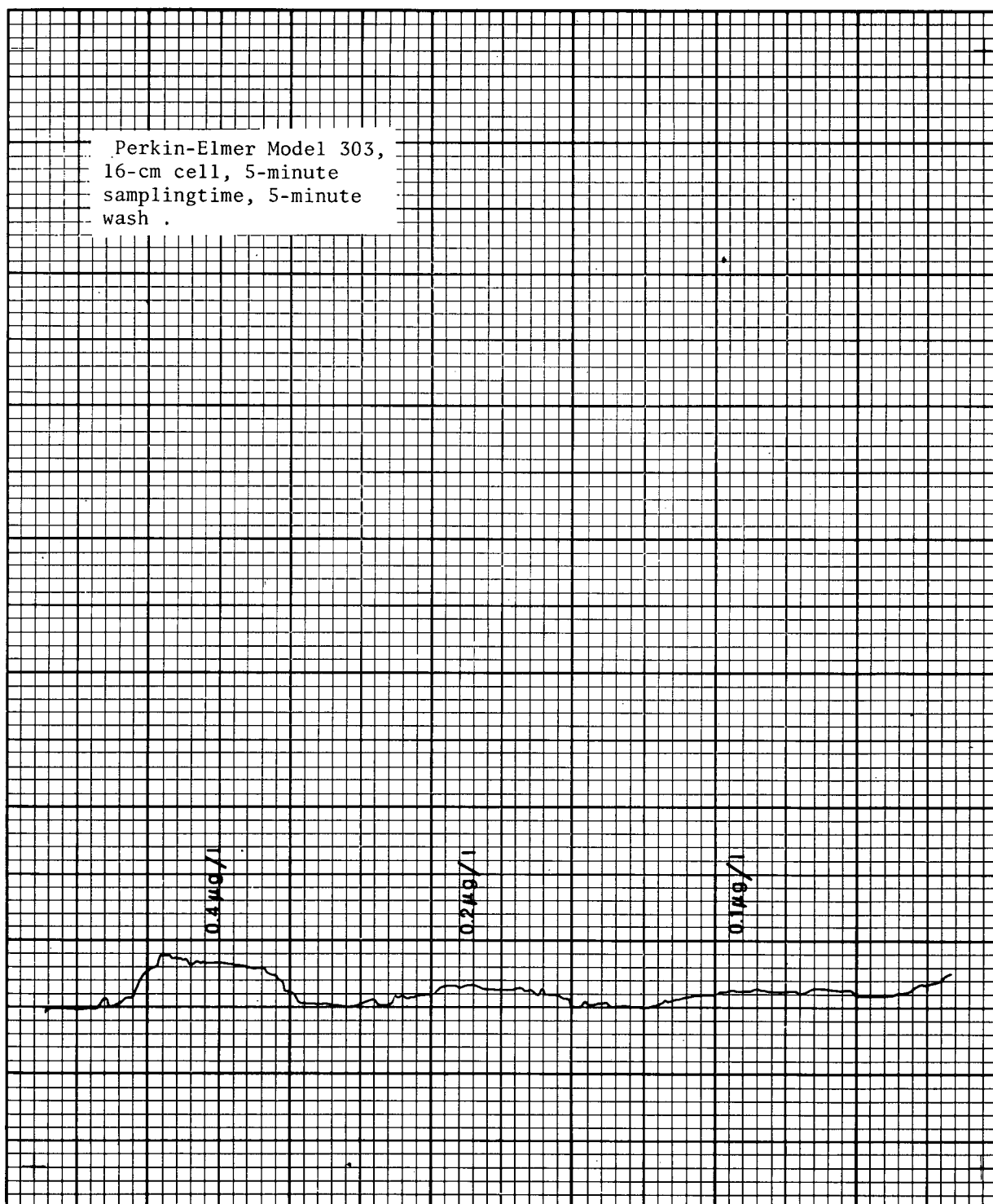
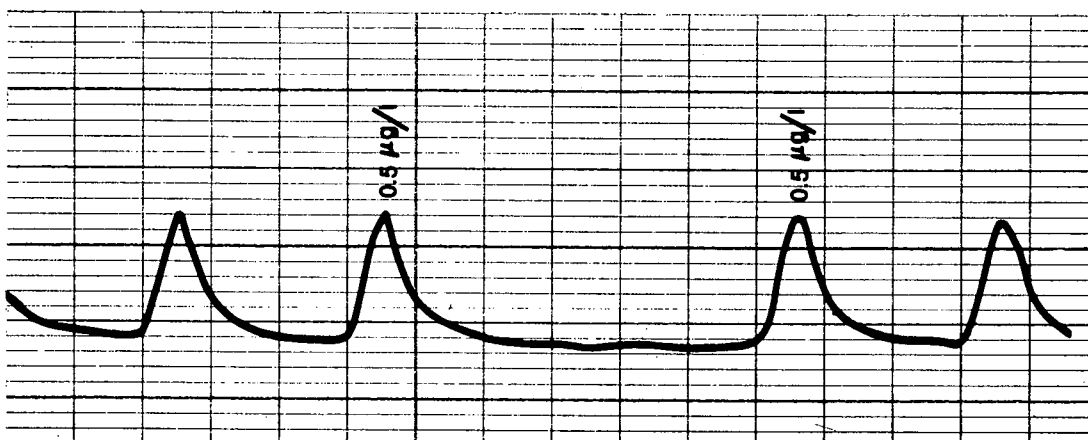
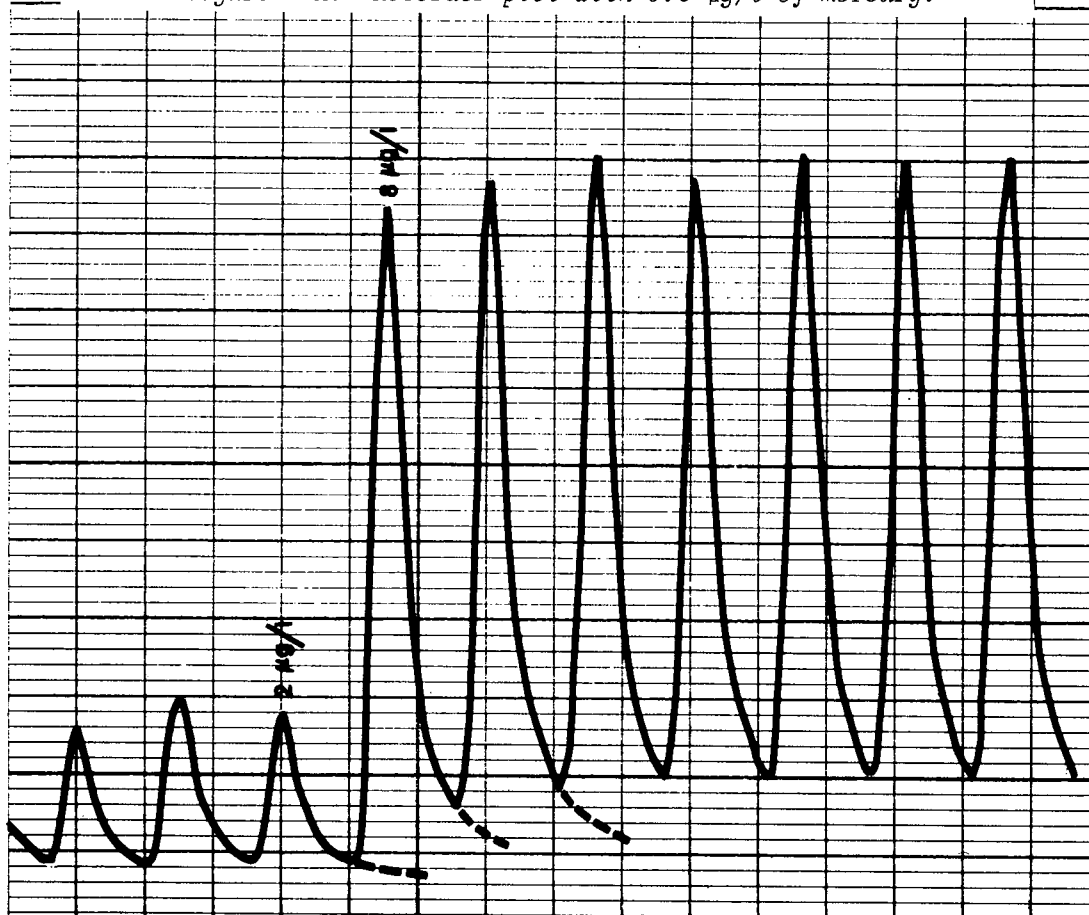


Figure 12. Recorder plot with standard solutions of mercury.



(Perkin-Elmer Model 403, 10 samples per hour, Recorder Scale 1A)

Figure 13A. Recorder plot with  $0.5 \mu\text{g/l}$  of mercury.



(Perkin-Elmer Model 403, 20 samples per hour, Recorder Scale 2A)

Figure 13B. Recorder plot with  $2 \mu\text{g/l}$  and  $8 \mu\text{g/l}$  of mercury.

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*A report on the results of chemical analyses of phosphate content in detergents and washing products. The report traces the development of washing products from organic soaps to modern phosphate-based detergents and describes the relationship between phosphates and the eutrophication process.*
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*A review of streamflow measuring techniques applicable to rivers with peak flows up to 10,000 cfs.*
- No. 26 The Control of Eutrophication. Canada Centre for Inland Waters, Burlington, Ontario; Fisheries Research Board of Canada, Winnipeg; Inland Waters Branch, Ottawa, 1970.  
*A discussion of the respective roles of phosphorus, nitrogen and carbon as critical elements in limiting the eutrophication process.*

A complete list of titles in the Technical Bulletin Series and copies of any of these publications may be obtained from the Director, Inland Waters Branch, Department of Energy, Mines and Resources, Ottawa, Ontario.

