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DETERMINATION OF DYES AND CHLORINATED HYDROCARBONS IN WASTEWATER TREATMENT PLANT INFLUENTS AND EFFLUENTS IN GRANBY AND MAGOG, QUEBEC

by

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ABSTRACT

The influent of a sewage treatment plant in Granby, Quebec, and the influent and effluent of a wastewater treatment plant of a textile mill in Magog, Quebec, were analyzed for dyes, PCB congeners and chlorinated pesticides. No dyes were found in the Granby sewage treatment plant influent. Four orange-red dyes were found in the Magog plant influent, and their ultraviolet-visible spectra were recorded. These dyes were not found in the effluent, indicating efficient removal by the wastewater treatment process.

In the Granby sewage treatment plant influent only three PCB congeners were found, all in the dichloromethane extract of the basic influent which had previously been thoroughly extracted under acidic conditions. The Magog wastewater treatment plant influent was more contaminated, especially with PCB congeners, whose sum of concentrations was about 250 ng/L. The basic extract of this influent after extraction at acidic pH also revealed substantial concentrations of PCBs (36% of the total). Moreover, some chlorinated hydrocarbons were only found in the basic extract. The basic extract of the effluent also contained significant concentrations of some PCB congeners relative to the acid extract. However, this one sample indicated that the wastewater treatment plant removed over 90% of those chemicals found in the influent. The occurrence of neutral lipophilic compounds in basic extracts of water after extraction at acidic pH suggests that concentrations of these compounds have been underestimated using the usual extraction techniques. If this phenomenon is widespread, it will require a change in extraction methods for these chemicals.

RÉSUMÉ

On a analysé les eaux brutes d'une usine d'épuration à Granby (Québec), ainsi que les eaux brutes et les eaux traitées d'une usine d'épuration d'une usine textile à Magog (Québec), en vue de déterminer si ces eaux contenaient des colorants, des congénères de PCB et des pesticides chlorés. Aucun colorant n'a été décelé dans les eaux brutes de l'usine d'épuration de Granby. Par contre, les eaux brutes de l'usine de Magog contenaient quatre colorants oranges-rouges pour lesquels nous avons obtenu le spectre UV-visible. Le procédé d'épuration était efficace, car ces colorants étaient absents des eaux traitées.

On n'a décelé que trois congénères de PCB dans les eaux brutes de l'usine d'épuration de Granby; tous ces congénères se trouvaient dans l'extrait au dichlorométhane d'eaux brutes basiques préalablement extraites en conditions acides. Les eaux brutes de l'usine d'épuration de Magog renfermaient plus de contaminants, en particulier des congénères de PCB, dont la concentration totale était d'environ 250 ng/L. L'extrait basique de ces eaux brutes après extraction en milieu acide renfermait des quantités notables de PCB (36 % de la totalité). De plus, certains hydrocarbures chlorés n'ont été décelés que dans l'extrait basique. Cet extrait contenait aussi, si on le compare à l'extrait acide, des quantités importantes de certains congénères de PCB. Toutefois, l'analyse de cet échantillon indiquait que l'usine d'épuration éliminait plus de 90 % des produits chimiques décelés dans les eaux brutes. Vu la présence de composés lipophiles neutres dans les extraits basiques après extraction en milieu acide, on peut supposer que les techniques d'extraction habituelles

sous-estimaient les concentrations de ces composés. Il faudra, si ce phénomène est généralisé, modifier les méthodes d'extraction de ces produits.

MANAGEMENT PERSPECTIVE

This very limited study has demonstrated the utility of the high performance liquid chromatograph with a diode array spectrophotometric detector in the tentative identification of dyes in environmental samples. In addition, it extends to another geographical area the observations of lipophilic chemicals in basic extracts after prior extraction at lower pH. More work is required to assess the generality of this phenomenon.

PERSPECTIVE-GESTION

Dans cette étude très limitée, on montre dans quelle mesure la chromatographie liquide à haute performance avec détection à l'aide d'un spectrophotomètre à réseau de diodes est utile pour identifier des colorants dans des échantillons environnementaux. De plus, cette étude indique que des produits chimiques lipophiles ont aussi été observés dans des extraits basiques après extraction à faible pH d'échantillons provenant d'une autre région. Il faudra effectuer d'autres travaux pour déterminer s'il s'agit là d'un phénomène général.

INTRODUCTION

Dyes and pigments are high volume chemicals which may enter aquatic environments in significant quantities. The distinction between dyes and pigments is largely made on the basis of solubility. Pigments have extremely low solubilities in water, and are not very soluble in organic solvents either, generally being applied in the solid state. Dyes are generally more soluble either in water or organic solvents. Global production of dyes in 1978 was estimated to be 6.4×10^8 kg of active ingredient (Clarke and Anliker, 1980). About 10^8 kg were produced in the United States in 1986 (Reisch, 1988). It has been estimated that 90% of dyes applied end up in fabrics, with the remaining 10% discharged to waste streams (Porter, 1973). In 1982 about 6.5×10^6 kg of dyes and 2.5×10^6 of pigments were imported into Canada (Statistics Canada, 1982). Most dyes are imported into Quebec and pigments into Ontario.

Interest in the environmental behaviour of dyes arose largely from concerns about carcinogenicity. Some dyes are carcinogens (Searle, 1976) and others after transformation yield compounds such as aromatic amines which may be carcinogenic or otherwise toxic (e.g., Weber, 1988). Very little is known of the environmental occurrence, persistence and fate of dyes, largely because of formidable analytical difficulties (e.g., Maguire, 1989).

We have been interested in determining the occurrence and persistence of dyes in the Yamaska River in the Eastern Townships of Quebec since much of Canada's textile industry is concentrated in the Eastern Townships.

Preparatory to the main part of the study, and at the request of Environnement Quebec, we analyzed the influent and effluent of the sewage treatment plant at Granby on the Yamaska River, and the influent and effluent of a wastewater treatment plant of a textile mill at Magog on the Magog River for dyes. This report gives the results of this study. Results are also presented for the determination of sixty-seven PCB congeners and chlorinated pesticides in these samples.

EXPERIMENTAL SECTION

Materials

Chlorinated pesticide standards were obtained from chromatographic supply companies. Sets of PCB congeners were obtained from the National Research Council of Canada, Halifax. These PCB congeners represent, by weight, 3% of Aroclor 1221, 44% of Aroclor 1242, 48% of Aroclor 1248, 38% of Aroclor 1254 and 60% of Aroclor 1260; therefore, the sum of the PCB congeners reported in this study should not be regarded as "total PCBs". Pesticide grade dichloromethane and other solvents were obtained from different suppliers and their purity (at 1000x concentration) was checked before use. High performance liquid chromatographic grade solvents were obtained from various suppliers. The sodium sulfate, glass fibre filters and disposable pipets were heated to 500 °C for 24 h before use. Hydrochloric acid and sodium hydroxide solutions used for pH adjustment were prepared from reagent grade chemicals, but were extracted with hexane before use to elimination

contamination. Organic-free water was distilled water purified by carbon adsorption and reverse osmosis. Gas chromatography with electron capture detection of hexane extracts of this water revealed no contamination, even with 1000-fold concentration. ACRO LC13 disposable filters with Luer inlets for particle removal before analysis by high performance liquid chromatography were obtained from Gelman Sciences (Montreal).

Methods

A sample (4L) of sewage treatment plant influent from Granby was collected by Environnement Quebec personnel on June 12, 1985. The plant was not operating that day and had not operated for several days, so the influent was presumably identical to the effluent. Samples (4L) of influent and effluent from a wastewater treatment plant associated with a textile mill at Magog were also collected the same day. The whole water samples were acidified with concentrated HCl to pH 1 and preserved with 200 mL of dichloromethane for transport to the laboratory. The dichloromethane layer was colourless in the Granby sample, deep orange-pink in the Magog influent sample and only faintly orange-pink in the Magog effluent sample. The samples were more thoroughly extracted (in triplicate) within one week of collection. After extraction at acidic pH, the aqueous phase was made basic to pH 12 by the addition of 10 N NaoH and the water was extracted three more times with dichloromethane. This procedure was followed since neutral compounds such as chlorinated hydrocarbons have been found in basic extracts of natural water after extraction at acidic pH (Maguire and Tkacz, 1990). The dichloromethane extracts were dried and concentrated to 10 mL. The 10 mL dichloromethane

extracts were solvent-exchanged with hexane by addition of hexane and careful evaporation to 0.5 mL with a gentle flow of nitrogen. This procedure was done three times to remove traces of dichloromethane. The extract was made up to 1 mL. At this point there was a small amount of precipitation in the test tube, but all material was transferred to the cleanup column as described below.

The 1 mL hexane extracts were cleaned up on activated silica gel columns of length 20 cm and i.d. 1 cm, with a layer of sodium sulfate for drying. Four 100 mL fractions were eluted from the columns. Fraction 1 was hexane; fraction 2 was dichloromethane/hexane (20/80, v/v); fraction 3 was dichloromethane/hexane (60/40, v/v); fraction 4 was 50 mL of dichloromethane followed by 50 mL of methanol. At each solvent change, a little solvent was used to rinse the test tubes containing the original extract, and in this way even the precipitated material was transferred to the cleanup column. All four fractions were solvent-exchanged and reduced to 1 mL of isooctane "keeper". Most of the PCBs and chlorinated insecticides eluted in fraction 1, but occasionally traces were found in fraction 2. All fractions of acidic and basic extracts were analyzed as described below.

Analyses of the isooctane extracts for dyes were performed with a Waters Associates high performance liquid chromatograph (HPLC) (Millipore-Waters, Mississauga) and 990+ diode array spectrophotometric detector. Samples of 25 uL volume were injected with a Waters WISP 712 autosampler onto a 15 cm x 4.6 mm i.d. reverse-phase Supelcosil LC-8-DB column preceded by a Supelguard LC-8-DB guard column (Supelco, Oakville). Both guard and analytical columns were thermostatted at 35°C. The diode array detector was used to take 200 - 800 nm

spectra of the HPLC effluent every 200 ms. The gradient conditions are shown in Table 1. The ion-pair procedure (Martin et al., 1978) is appropriate for disperse dyes and those ionic dyes which may be extracted by dichloromethane under acidic conditions. It should be noted that use of the diode array detector alone will likely not be sufficient for a positive identification of a particular dye since few authentic standards of dyes used in the Eastern Townships are available with which to match retention times. Confirmed identification of those dyes detected in this work awaits analysis by liquid chromatography - mass spectrometry.

Analyses for chlorinated hydrocarbons were performed with a Hewlett-Packard 5890A capillary column gas chromatograph with a single injector-dual column-dual electron capture detector technique. One column was Ultra-2 and the other was OV-17. Column dimensions were 0.2 mm i.d. x 25 m in length, with 0.17 um film thickness. Injector and detector temperatures were 250 and 350 °C, respectively. The initial column temperature was 60 °C, and the program rate was 3°/min to 280 °C, with an 8 min. final hold. The analyses were performed in 8:1 split mode. The carrier gas flow rate was 1.0 - 1.5 mL/min (50-80 cm/s) and the make-up gas flow rate was 20 mL/min. Table 2 shows the 67 chlorinated insecticides and PCBs sought. Standard mixtures of all of these compounds in the expected concentration ranges were prepared and used to calibrate retention times and detector responses. Chromatographic "windows" were typically 0.04 min at most at 80 min retention time. The presence of a compound was taken to be tentatively confirmed if (i) it occurred within the appropriate chromatographic window on both columns, (ii) the concentrations determined with each column were within 50% of each other

(in which case the lower of the two concentrations was reported), and (iii) the concentrations were above the limit of quantitation for the particular sample, as opposed to the limit of detection (Keith et al., 1983). Measurement precision near the limit of quantitation was about 15 - 20%. Appropriate solvent and reagent blanks were performed. At no time was contamination evident.

RESULTS AND DISCUSSION

No dyes were found in the Granby sewage treatment plant effluent within the limits of detection of the diode array spectrophotometric detector. Four dyes were found in the Magog wastewater treatment plant influent, and none were found in the effluent of the latter plant.

Three of the dyes found in the Magog wastewater treatment plant influent were in the fourth cleanup fraction of the dichloromethane extract obtained under acidic conditions, which indicates that they were relatively polar. Figures 1-3 show spectra of these dyes which had retention times of 5.12, 5.68 and 5.8 min, respectively. All three dyes had absorbance maxima in the range expected for orange or red dyes. The fourth dye was found in the first cleanup fraction of the dichloromethane extract obtained under basic conditions. Its spectrum, shown in Figure 4, is also consistent with those of orange or red dyes. The fact that it was found in the first cleanup fraction indicated that it was relatively non-polar. Unambiguous identification of these dyes would be extremely difficult without electron impact mass spectra.

Within the limits of detection of the diode array detector, the four dyes appear to be efficiently removed in the Magog wastewater treatment plant since none were detected in the effluent.

Results of the analyses for chlorinated hydrocarbons in Granby sewage treatment plant influent are shown in Table 3, while results for the Magog wastewater treatment plant influent and effluent are shown in Table 4.

In the Granby sewage treatment plant influent only three PCB congeners were found of the sixty-seven compounds sought. Interestingly, they were only found in the dichloromethane extract of the basic influent which had previously been thoroughly extracted under acidic conditions. This observation was first made for PCBs and other lipophilic chemicals in filtered water from the Niagara River and the Burlington Ship Canal (Maguire and Tkacz, 1990), and more recently for PCBs by an independent laboratory (Fox, 1990). This phenomenon may be the result of a strong association between a fraction of the dissolved lipophilic chemicals and dissolved organic matter in fresh water, an association that is resistant to organic solvent extraction at acidic or neutral pH, but which is at least partially disrupted by extraction at high pH. In the present study the influent and effluent samples were not filtered before extraction, and so the results may reflect (i) a strong association with dissolved organic matter, (ii) a strong association with particulate material which is only partially disrupted during the time of contact between organic solvent and suspended solids in the extraction procedure, or both.

The Magog wastewater treatment plant influent was more contaminated, especially with PCB congeners, whose sum of concentrations was about 250 ng/L (cf. Table 4). The basic extract of this influent also revealed substantial concentrations of PCBs (36% of the total). Moreover, p,p'-DDE, beta-endosulfan and PCBs 141, 185 and 196 were only found in the basic extract.

The basic extract of the effluent also contained significant concentrations of some PCB congeners relative to the acid extract. However, this one sample indicated that the wastewater treatment plant removed over 90% of those chemicals found in the influent.

This very limited study has demonstrated the utility of the high performance liquid chromatograph with a diode array spectrophotometric detector in the tentative identification of dyes in environmental samples. In addition, it extends to another geographical area the observations of lipophilic chemicals in basic extracts after prior extraction at lower pH. The occurrence of neutral lipophilic compounds in basic extracts of water after extraction at acidic pH suggests that concentrations of these compounds have been underestimated using the usual extraction techniques. If this phenomenon is widespread, it will require a change in extraction methods for these chemicals. More work is required to assess the generality of this phenomenon.

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Table 1. High performance liquid chromatographic gradient conditions for disperse and some ionic dyes*

Time, min	% Acetonitrile	% 0.01 M KH_2PO_4
0	5	95
25	100	0
30	5	95

*The flow rate was 0.5 mL/min, with linear gradients throughout.

Table 2. Chlorinated hydrocarbons sought and elution order on an Ultra-2 column*

No.	Chemical Abbreviation	Name
1	Lindane	gamma-hexachlorocyclohexane
2	PCB 18	2,2',5-trichlorobiphenyl
3	PCB 15	4,4'-dichlorobiphenyl
4	PCB 54	2,2',6,6'-tetrachlorobiphenyl
5	PCB 31	2,4',5-trichlorobiphenyl
6	Heptachlor	heptachlor
7	PCB 52	2,2',5,5'-tetrachlorobiphenyl
8	PCB 49	2,2',4,5'-tetrachlorobiphenyl
9	Aldrin	aldrin
10	PCB 44	2,2',3,5'-tetrachlorobiphenyl
11	PCB 40	2,2',3,3'-tetrachlorobiphenyl
12	PCB 103	2,2',4,5',6-pentachlorobiphenyl
13	Heptachlor epoxide	heptachlor epoxide
14	PCB 121	2,3',4,5',6-pentachlorobiphenyl
15	PCB 60	2,3',4,4'-tetrachlorobiphenyl
16	o,p'-DDE	o,p'-DDE
17	PCB 101	2,2',4,5,5'-pentachlorobiphenyl
18	alpha-Endosulfan	alpha-endosulfan
19	PCB 86	2,2',3,4,5-pentachlorobiphenyl
20	PCB 87	2,2',3,4,5'-pentachlorobiphenyl
21	Dieldrin	dieldrin
22	p,p'-DDE	p,p'-DDE

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Table 2 cont'd

No.	Chemical Abbreviation	Name
23	PCB 77	3,3',4,4'-tetrachlorobiphenyl
24	PCB 154	2,2',4,4',5,6'-hexachlorobiphenyl
25	o,p'-DDD	o,p'-DDD
26	Endrin	endrin
27	PCB 151	2,2',3,5,5',6-hexachlorobiphenyl
28	beta-Endosulfan	beta-endosulfan
29	PCB 118	2,3',4,4',5-pentachlorobiphenyl
30	PCB 143	2,2',3,4,5,6'-hexachlorobiphenyl
31	p,p'-DDD	p,p'-DDD
32	PCB 114	2,3,4,4',5-pentachlorobiphenyl
33	o,p'-DDT	o,p'-DDT
34	PCB 153	2,2',4,4',5,5'-hexachlorobiphenyl
35	PCB 105	2,3,3',4,4'-pentachlorobiphenyl
36	PCB 141	2,2',3,4,5,5'-hexachlorobiphenyl
37	PCB 137	2,2',3,4,4',5-hexachlorobiphenyl
38	p,p'-DDT	p,p'-DDT
39	PCB 138	2,2',3,4,4',5'-hexachlorobiphenyl
40	PCB 129	2,2',3,3',4,5-hexachlorobiphenyl
41	PCB 159	2,3,3',4,5,5'-hexachlorobiphenyl
42	PCB 182	2,2'3,4,4',5,6'-heptachlorobiphenyl
43	PCB 187	2,2',3,4',5,5',6-heptachlorobiphenyl
44	PCB 183	2,2',3,4,4',5',6-heptachlorobiphenyl

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Table 2 cont'd

No.	Chemical Abbreviation	Name
45	PCB 128	2,2',3,3',4,4'-hexachlorobiphenyl
46	PCB 185	2,2',3,4,5,5',6-heptachlorobiphenyl
47	PCB 202	2,2',3,3',5,5',6,6'-octachlorobiphenyl
48	PCB 171	2,2',3,3',4,4',6'-heptachlorobiphenyl
49	PCB 156	2,3,3',4,4',5-hexachlorobiphenyl
50	PCB 173	2,2',3,3',4,5,6-heptachlorobiphenyl
51	PCB 200	2,2',3,3',4,5',6,6'-octachlorobiphenyl
52	Methoxychlor	methoxychlor
53	PCB 180	2,2',3,4,4',5,5'-heptachlorobiphenyl
54	PCB 191	2,3,3',4,4',5',6-heptachlorobiphenyl
55	Mirex	mirex
56	PCB 170	2,2',3,3',4,4',5-heptachlorobiphenyl
57	PCB 201	2,2',3,3',4',5,5',6-octachlorobiphenyl
58	PCB 203	2,2',3,4,4',5,5',6-octachlorobiphenyl
59	PCB 196	2,2',3,3',4,4',5',6-octachlorobiphenyl
60	PCB 189	2,3,3',4,4',5,5'-heptachlorobiphenyl
61	PCB 208	2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl
62	PCB 195	2,2',3,3',4,4',5,6-octachlorobiphenyl
63	PCB 207	2,2',3,3',4,4',5,6,6'-nonachlorobiphenyl
64	PCB 194	2,2',3,3',4,4',5,5'-octachlorobiphenyl
65	PCB 205	2,3,3',4,4',5,5',6-octachlorobiphenyl
66	PCB 206	2,2',3,3',4,4',5,5',6-nonachlorobiphenyl

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Table 2 cont'd

No.	Chemical Abbreviation	Name
67	PCB 209	decachlorobiphenyl

*The IUPAC numbering scheme for the PCB congeners is given in Ballschmiter and Zell (1980).

Table 3. Concentrations (ng/L) of chlorinated hydrocarbons in extracts of Granby sewage treatment plant influent*

Chemical	Acidic Extract	Basic Extract
PCB 18		3.5
PCB 87		1.8
PCB 171		0.5

*The water was extracted first under acidic conditions, then under basic conditions. The limits of quantitation vary with each chemical and are approximately 0.1 ng/L for 1 mL extracts from a 4 L whole water sample. The compounds are listed in order of elution from an Ultra-2 column. The chemical names for the PCB congeners are given in Table 2.

Table 4. Concentrations (ng/L) of chlorinated hydrocarbons in extracts of a Magog wastewater treatment plant influent and effluent*

Chemical	Influent			Effluent		
	A	B	T	A	B	T
PCB 18	11.5	4.5	16.0	5.7	7.0	12.7
PCB 52	13.5		13.5	2.1	2.0	4.1
PCB 101	12.2		12.2	1.0	0.7	1.7
PCB 87	6.5		6.5		0.6	0.6
p,p'-DDE		1.3	1.3			
beta-Endosulfan		2.5	2.5			
PCB 118	11.3		11.3			
PCB 105	6.1		6.1			
PCB 141		3.8	3.8			
PCB 137	0.7		0.7			
PCB 185		2.2	2.2			
PCB 156	1.6		1.6			
PCB 196		11.8	11.8			
PCB 194	21.5	70.0	91.5			
PCB 205	25.4		25.4			
PCB 206	51.2		51.2			
Sum of PCBs	161.5	92.3	253.8	8.8	10.3	19.1

*A and B refer to extracts made sequentially of whole water under acidic-then-basic conditions, and T is the sum of concentrations in both extracts. The limits of quantitation vary with each chemical and are approximately 0.1 ng/L for 1 mL extracts from a 4 L whole water sample. The compounds are listed in order of elution from an Ultra-2 column. The chemical names for the PCB congeners are given in Table 2.

FIGURE CAPTIONS

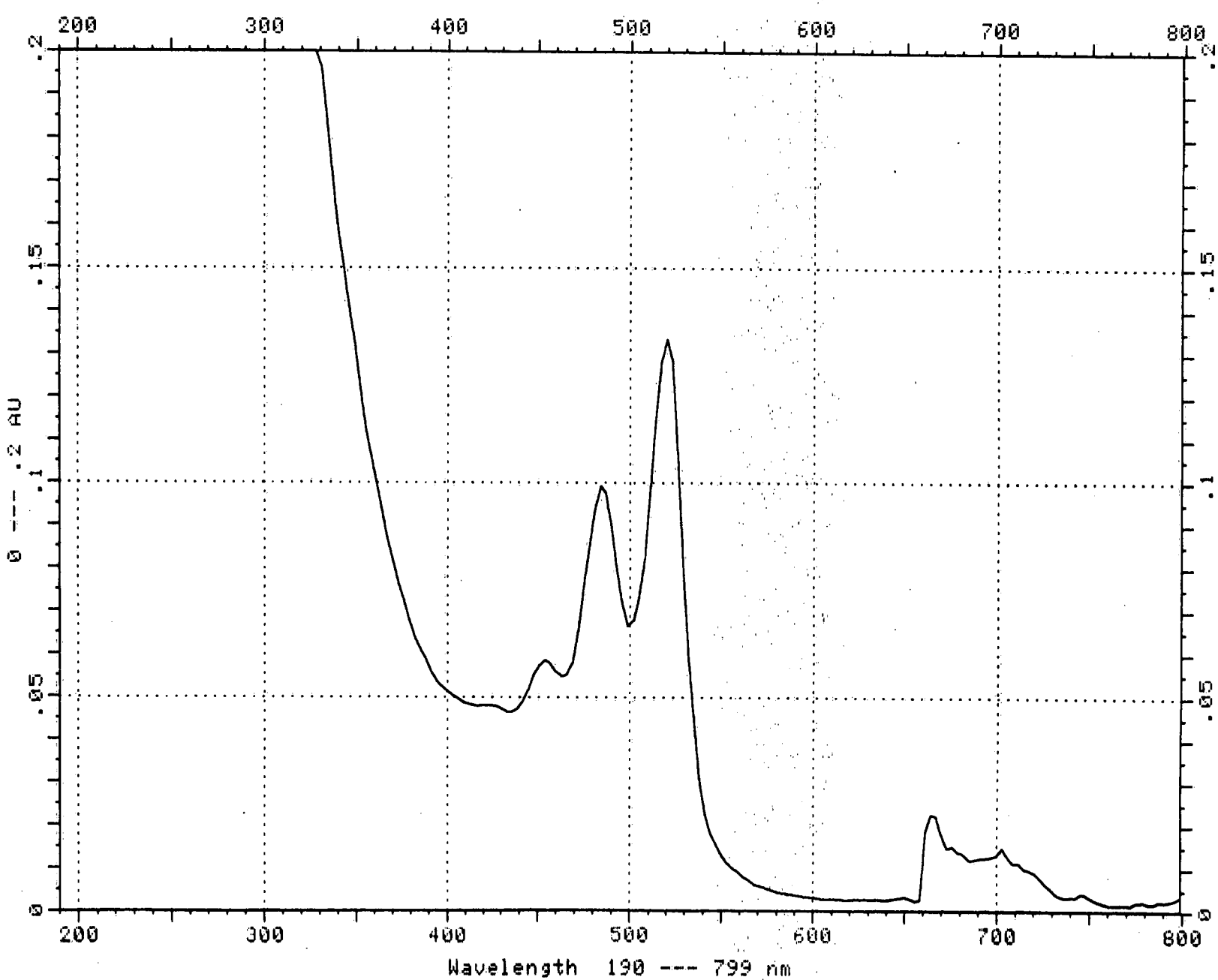
Figure 1. Spectrum of dye of retention time 5.12 min found in fourth cleanup fraction of acidic dichloromethane extract of Magog sewage treatment plant influent.

Figure 2. Spectrum of dye of retention time 5.68 min found in fourth cleanup fraction of acidic dichloromethane extract of Magog sewage treatment plant influent.

Figure 3. Spectrum of dye of retention time 5.90 min found in fourth cleanup fraction of acidic dichloromethane extract of Magog sewage treatment plant influent.

Figure 4. Spectrum of dye of retention time 4.06 min found in first cleanup fraction of basic dichloromethane extract of Magog wastewater treatment plant influent.

Waters 990 SPECTRUM



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Resolution
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0 min
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Column
mmID * mm
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Flow rate
ml/min
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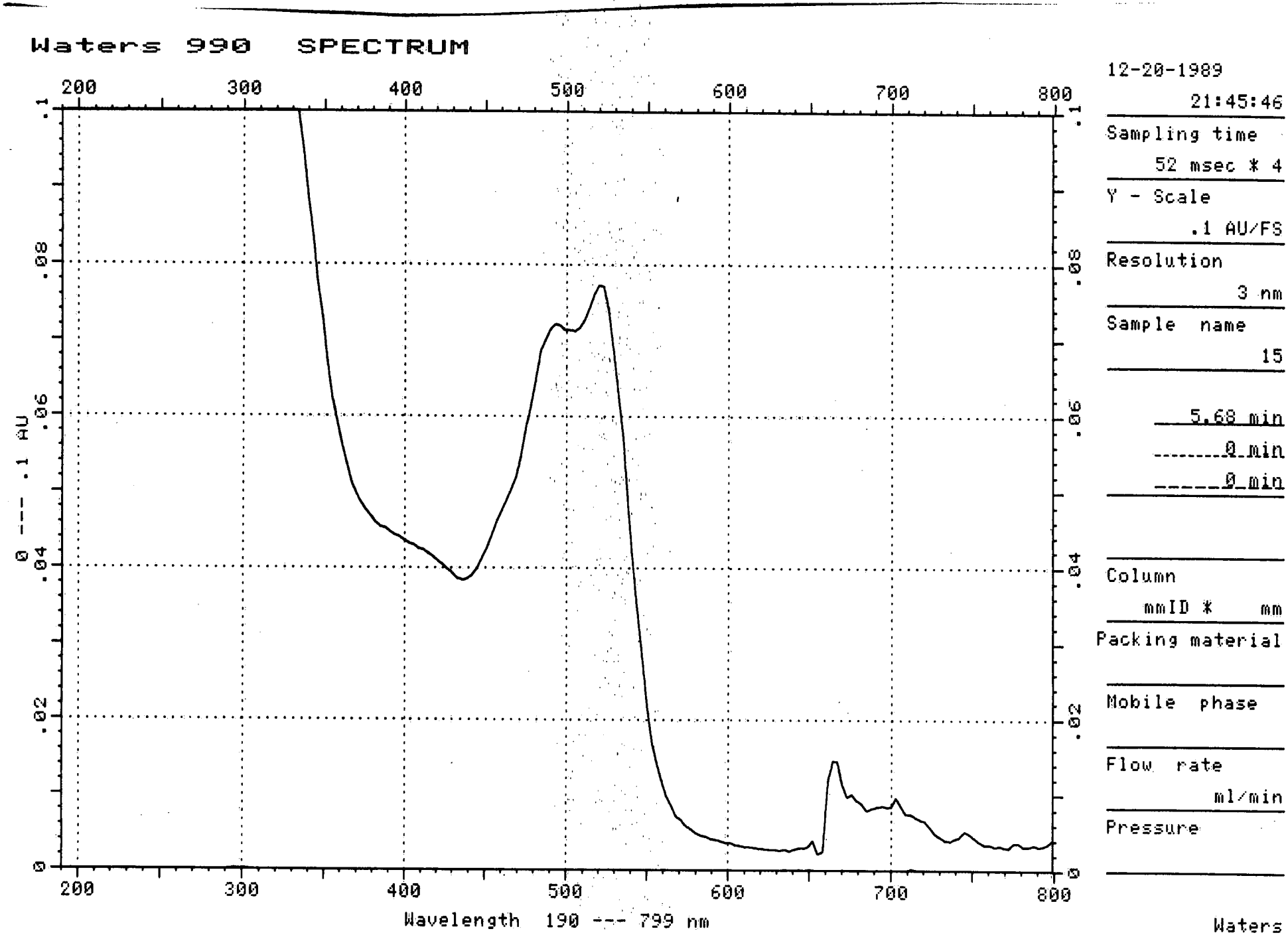
Waters


Waters 990 SPECTRUM
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800
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12-20-1989
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Sampling time
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Y - Scale
.1 AU/FS
Resolution
3 nm
Sample name
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5.68 min
0 min
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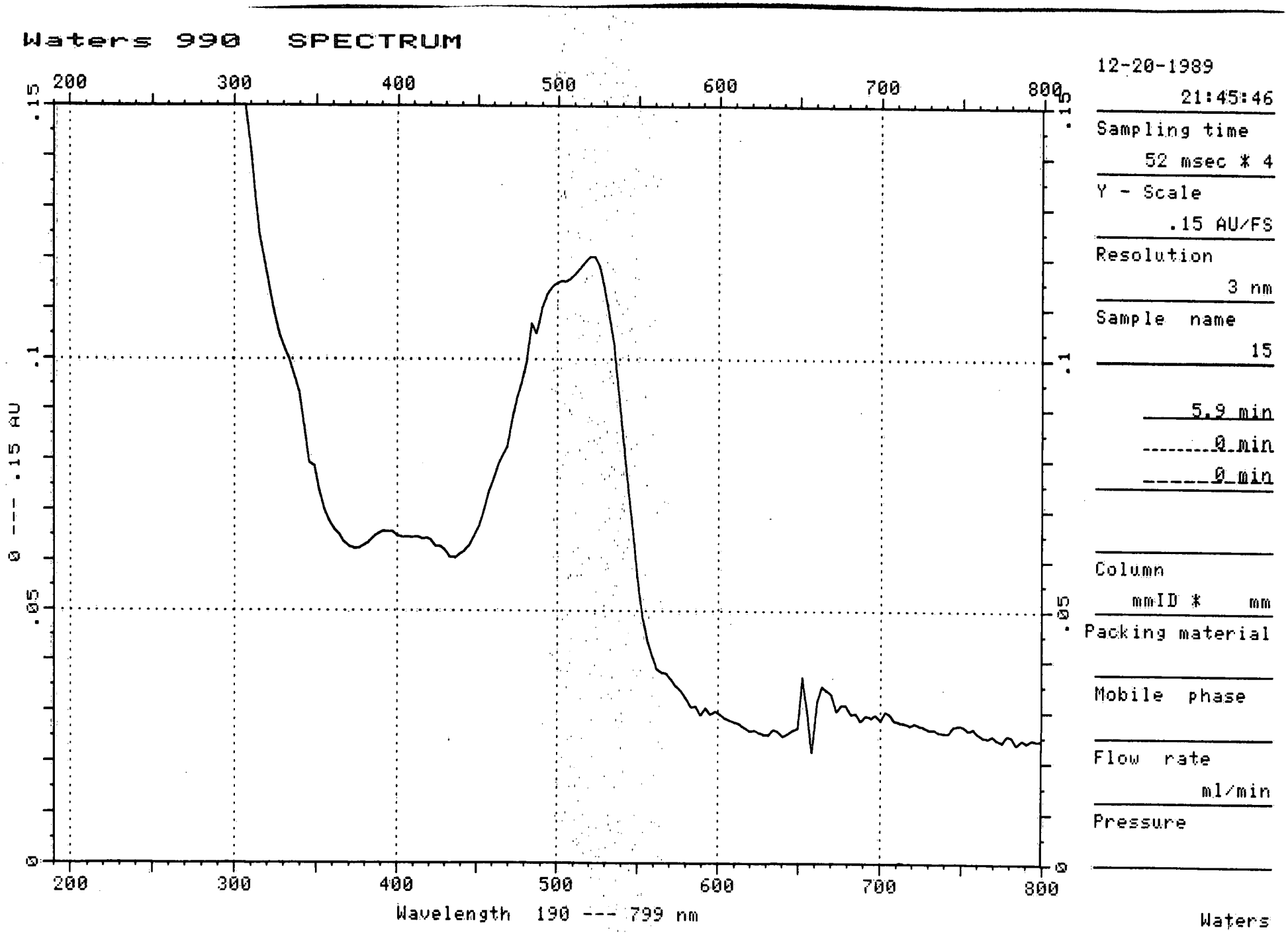
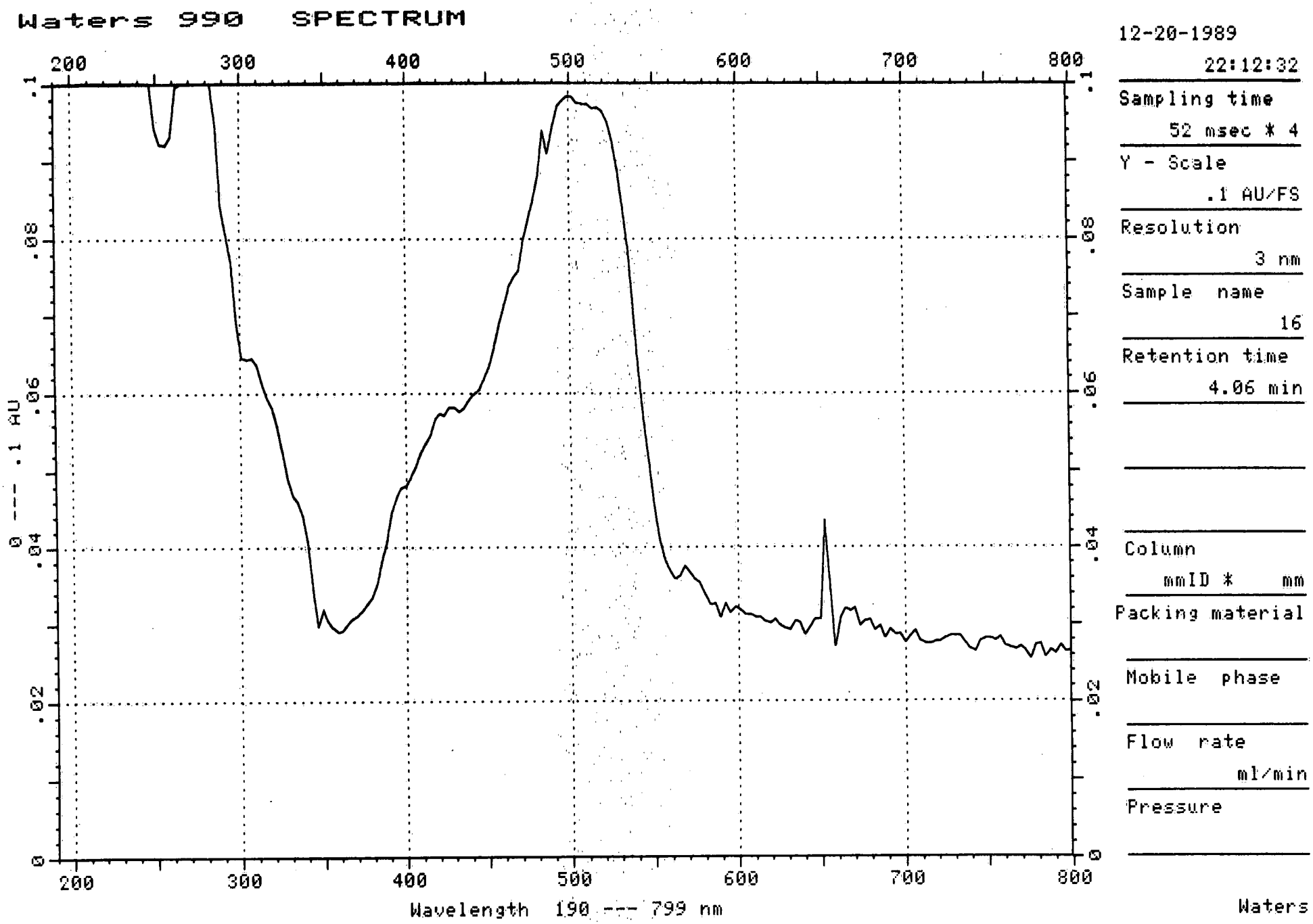
Column
mmID * mm
Packing material
Mobile phase
Flow rate
ml/min
Pressure
Waters


Waters 990 SPECTRUM
12-20-1989
21:45:46
Sampling time
52 msec * 4
Y - Scale
.15 AU/FS
Resolution
3 nm
Sample name
15
5.9 min
0 min
0 min
Column
mmID * mm
Packing material
Mobile phase
Flow rate
ml/min
Pressure
Waters
0 --- .15 AU
Wavelength 190 --- 799 nm
200
300
400
500
600
700
800
0
.05
.1
.15


Waters 990 SPECTRUM
12-20-1989
22:12:32
Sampling time
52 msec * 4
Y - Scale
.1 AU/FS
Resolution
3 nm
Sample name
16
Retention time
4.06 min
Column
mmID * mm
Packing material
Mobile phase
Flow rate
ml/min
Pressure
0 --- .1 AU
Wavelength 190 --- 799 nm
Waters