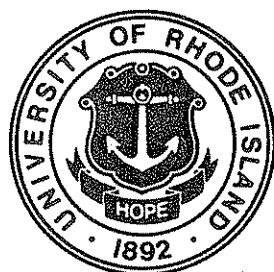


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**UV-OZONE PROCESS FOR THE
CONCURRENT DECOMPOSITION OF HALOGENATED ALKANES
AND ALKANES AND THE DISINFECTION OF WATER**



**Rhode Island
Water Resources Center**

Technical Completion Report

A-076-RI

UV-OZONE PROCESS FOR THE
CONCURRENT DECOMPOSITION OF HALOGENATED ALKANES
AND ALKANES AND THE DISINFECTION OF WATER

by

Calvin P.C. Poon, and Bruno M. Vittimberga

Dept. of Civil and Environmental Engineering/Dept. of Chemistry

Submitted to
Office of Water Policy
United States Department of the Interior
Washington, D.C. 20243

The work on which this report is based was supported in part by funds provided by the United States Department of the Interior as authorized under the Water Research and Development Act of 1978.

Rhode Island Water Resources Center
University of Rhode Island
Kingston, R.I. 02881

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Final Report
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UV-Ozone Process for the Concurrent Decomposition of Halogenated Alkanes and Alkanes and the Disinfection of Water.

Calvin P.C. Poon
Dept. of Civil and Environmental Engineering

Bruno M. Vittimberga
Dept. of Chemistry

INTRODUCTION

Many organic chemical species found in water supply systems are toxic. Among these species are trihalomethanes, trichloroethylene and its related chemicals. All have been linked to animal cancers, and epidemiological data suggest that they may contribute to cancer in humans. Trihalomethanes (THM) can form in drinking water from the chemical interaction between chlorine, used as a disinfectant, and organic substances that occur naturally in drinking water sources. Unprotected watersheds with a "surface system" of wetlands and shallow reservoirs contain more organics and demand more chlorine for disinfection. The chances of having THMs in drinking water from these sources are higher than those from better protected watersheds. The case in point is the finding of unusually high concentrations of total trihalomethane (TTHM) in the Bristol County water system in Rhode Island. Several samples, according to EPA's analysis in November 1977 (ref. 1) showed slightly above to more than 3 times the EPA proposed standards of 100 ppb for TTHM for public water supplies. Like other places having similar problems in their water systems, Bristol County has an unprotected watershed with shallow reservoirs.

One of the major provisions of the National Safe Drinking Water Act of 1974 is the initiation of research on health, economic and technological problems related to drinking water supplies. In partial

response to these needs, a new technology of water treatment enabling the destruction of these organic substances and providing disinfection at the same time is investigated in this research.

The major forms of THM found in water supply systems in the United States are chloroform (CHCl_3), bromodichloromethane (CHB_rCl_2), dibromochloromethane (CHB_rCl) and bromoform (CHB_rCl_3), with CHCl_3 usually amounting to more than 85 percent and CHB_rCl_2 12 percent of the TTHM. In most water systems deriving their water from protected watersheds, the THM concentration is usually very low. At the time of the EPA tests in 1974, the high concentration in the nation was recorded in Miami with 311 ppb. Later tests have shown up to 550 ppb in some water systems. Even these high concentrations are very low in water for any known method of treatment to be effective.

Ozonation is one means of removing trihalomethane, trichloroethylene, carbon tetrachloride and their related compounds from water. Recent studies at the University of Rhode Island show that despite high ozonation using 5 gram/hour application sparging for 5 minutes, only 60 percent of removal can be realized. Activated carbon adsorption also has severe limitations on the removal of these chemicals because their low concentrations in water are not significantly different from the equilibrium concentrations from isotherm studies. Ozonation has an advantage over activated carbon in that ozonation provides disinfection as well as the removal of these toxic organic compounds. The process therefore is more economically justifiable. However, the need exists for a significant improvement of the efficiency of organic removal by ozone. One possible means is to use an ultraviolet light-ozone (UV-ozone) process.

UV-ozone is the strongest physical-chemical oxidation reaction effective with toxic and highly refractory organics. The ultraviolet light provides the bond-breaking energy to excite molecules and to produce free radicals which are highly reactive in the presence of ozone. Ozone alone produces substantially less radicals and oxidizes refractory organics only to intermediates according to Gould (ref. 2). The interpretation of the photochemistry of aqueous solutions of ozone, according to Ashmore (ref. 3) is in the following. At a longer wavelength (600 m μ) the oxygen atom produced in the primary step ($O_3 + h\nu \rightarrow O + O_2$) is in its ground electronic state or $O(^3P)$, and cannot react with water. However, at the two shorter wavelength (310 and 245 m μ), a 1O oxygen (excited or high energy state) atom may be produced (higher efficiency at 245 m μ). This atom reacts with water to give two hydroxyl radicals (-29 kcal/mole) of which the main reaction in solution will be recombination ($O + H_2O \rightarrow 2OH\cdot$; and $2OH\cdot \rightarrow H_2O_2$). The hydroxyl radicals are possibly the active species primarily responsible for the oxidation of THMS with a possible chain reaction.

Many compounds may be economically oxidized using UV-ozone whose reaction rates would otherwise be too low to be processed in other systems. UV and ozone were applied for both sterilization and reduction of organic matter in a wastewater-reuse system (ref. 4). Successful removal of complexed cyanides from wastewater was also reported (ref. 5). In the absence of UV radiation, difficult compounds such as potassium ferricyanide, ethanol and acetic acid are essentially unoxidized by ozone; however, with UV radiation oxidation proceeds rapidly at room temperature (ref. 6).

Recent EPA sponsored studies (ref. 7) have shown that UV-ozone reduces the level of polychlorinated biphenyls (PCB) in industrial

wastewaters to less than $0.01 \mu\text{g/l}$, and in many cases to below detectable limits. Activated carbon adsorption can only reduce PCB levels to $0.1 \mu\text{g/l}$. Consequently, UV-ozone has been specified by EPA as the best practicable control technology currently available for PCB (as well as for photoprocessing wastewater).

It can be stated in summary that UV-ozone is capable of oxidizing most refractory organic and inorganic compounds. Although UV is an effective reagent itself in water disinfection (the possibility of photoreactivation can be a drawback of UV disinfection), the purpose of utilizing UV in the UV-ozone process is to produce radicals which initiates propagation chains and causes the overall reaction to proceed much faster, enhancing the reaction with ozone 10^2 to 10^4 folds. This brings about three results as follow: (1) faster and more complete oxidation of refractory and toxic compounds by ozone; (2) more complete disinfection of water; and (3) very small size reactors are needed which compensates for the higher power cost in operation. The fact that both toxic compound oxidation and disinfection can be accomplished in one step instead of two separate processes is unique and should result in significant saving of capital as well as operational costs in water treatment.

OBJECTIVE OF STUDY

The objective of this study is to determine the effectiveness of the UV-ozone process for concurrent removal of THMs and coliform in water. By taking advantage of the rapid chain reactions of UV-ozone and organic compounds, a simple and compact treatment system can be developed. A laboratory scale column reactor is specifically designed and built for

the study. Individual effects of ozone and UV radiation and their combination are determined. The resulting data are analyzed in terms of kinetic rate expressions. The kinetic rates are used to compare the relative effectiveness of the control variables such as UV intensity and ozone dosing rate.

EXPERIMENTAL PROCEDURES

A specially designed column reactor is built as described in Figure 1. The UV lamp has an output of 13.8 watts and an intensity of 15800 $\mu W/cm^2$ on the surface of the quartz jacket. The liquid volume in the reactor is 1.3-liter. Thus the power input of the UV radiation is 10.6 w/l solution. The four diffusers allow either air or ozone into the reactor in the form of medium size gas bubbles. The rising bubbles provide the mixing of the solution which is critical for photolysis since photolysis is a diffusion control process. Air or ozone supplied is maintained at a steady rate of 0.4 l/min or 0.308 l/min-l solution. The rate is adequate and eliminates any mass transfer limitation in the photolytic reaction.

Stock solutions of THM species are prepared as follow: $CHCl_3$ 4.5 $\mu g/\mu l$, CHB_rCl_2 0.6 $\mu g/\mu l$, mixture $CHCl_3$ 2.6 μg + CHB_rCl_2 0.23 $\mu g/\mu l$. A reservoir holding two liters of distilleu water was spiked with one or both of the standard solutions aforementioned. The mixture was then pumped into the reactor for experiment. Batch experiments are used throughout the study.

Four sets of experiments are conducted as follow:

UV radiation only

Ozone only

Air purging only

UV-ozone (oxyphotolysis)

The UV lamp and the ozonator are warmed up 30 minutes and 3 hours respectively before the experiment. An initial sample is taken directly from the reservoir to establish the THM concentration. Zero time and periodic samples are taken from the reactor during the experiment. Samples taken are refrigerated 24 to 48 hours prior to extraction and gas chromatograph (GC) analysis. Refrigeration is found necessary for all UV exposed samples because sample temperature rises above the boiling point of the extraction solvent, pentane at 36.1⁰C. Extraction vessels are 40-ml in volume. THM extraction follows the liquid/liquid extraction procedure outlined in the Federal Register (Ref. 8). CHCl₃ and CHB_rCl₂ are analyzed with a Perkin-Elmer Sigma 3B gas chromatograph equipped with an electron capture detector. A steel column instead of a glass column is used for the GC.

Ozone, when used in the experiment, is analyzed for its initial and final concentrations in solution following Standard Methods (Ref. 9). Ozone input rate expressed in mg/min-l is determined by measuring the ozone trapped in a potassium iodide solution over a given period of time.

For disinfection study, the distilled water is spiked with a primary effluent from a local sewage treatment plant. Coliform analysis is according to Standard Methods (Ref. 9).

RESULTS

Regardless of which method of treatment is used in this study, all data indicate a straight line obtained from a plot of the logarithmic concentration ratio ($\ln c/c_0$) versus time. This indicates a first order removal kinetic for the concentrations of CHCl₃ and CHB_rCl₂ used in this study. The concentrations used are 199 to 312 ppb for CHCl₃ and 15.3 to 48.6 ppb for CHB_rCl₂, simulating their respective

concentrations found in drinking water supplies in the United States. When all control variables are held constant, i.e., UV power input $I = 10.6$ w/l solution; air or ozone flow rate = 0.308 l/min-l solution; ozone input rate = 8.8 ± 0.2 mg/min-l; the removal kinetic equation for any treatment is represented by $-dc/dt = k.c$ or $k = -\ln(c/c_0) / t$. The slope of the plot, or k , represents the removal rate with a unit of time^{-1} . Four plots, one each representing the typical results of a treatment method, are presented in Figures 2, 3, 4, and 5.

The results of the experiments including time of treatment, residual THM concentration, residual ozone concentration, and the calculated values of removal rate k , percentage of removal, are summarized in Tables 1 through 4.

Disinfection is conducted only for a few selected experiments using UV-ozone treatment. The sole objective for this portion of the study is to see if coliform can be killed effectively at the UV-ozone dose successful in THM removal. The result is recorded in Table 5.

DISCUSSION

Ozone appears to be effective in the removal of CHCl_3 and CHB_rCl_2 , individually or as a mixture. Ozone input at 7.7 to 10 mg/min-l is adequate with sufficient dissolved ozone remained in the solution (0.9 to 3.9 mg/l) after 50 to 60 -minute treatment. Both the percent removal and removal rate are higher for CHCl_3 than CHB_rCl_2 . Table 1 shows the average of 95.4 percent removal and 0.053 min^{-1} removal rate for CHCl_3 versus 87.2 percent removal and 0.042 min^{-1} removal rate for CHB_rCl_2 .

Table 1, Ozone Treatment of chloroform
and Bromodichloromethane
(0.308 l O₃/min-1 solution, Temp. 22-25°C)

Chemical Compound	Time of Reaction	Initial Conc.	Final Conc.	Percent Removal	Removed Rate	O ₃ Input	Residual O ₃
	min.	ppb	ppb	%	min ⁻¹	mg/min-1	mg/l
CHCl ₃	60	170	8	95.3	0.049	7.7	1.9
	60	178	6	96.6	0.054	9.2	1.7
	50	316	18	94.3	0.056	8.1	1.9
				$\bar{x} = 95.4$	$\bar{x} = 0.053$		
CHBrCl ₂	60	28.8	3	89.6	0.044	8.3	1.8
	60	25.4	2.9	88.6	0.043	8.4	2.5
	60	48.6	8.1	83.3	0.039	9.8	0.9
				$\bar{x} = 87.2$	$\bar{x} = 0.042$		
				$S = 0.0036$			
					$S = 0.0026$		
Mixture	60	224 (CHCl ₃)	12	94.6	0.050		
		20.3 (CHBrCl ₂)	3	85.2	0.038	10	1.6
	60	256 (CHCl ₃)	9	96.5	0.057		
		18.8 (CHBrCl ₂)	2.5	86.7	0.040	9.9	3.9

Table 2. Air Purging of Chloroform
and Bromodichloroform
(0.308 l air/min-1 solution, Temp. 23-26°C)

Chemical Compound	Time of Reaction	Initial Conc.	Final Conc.	Percent Removal	Removed Rate
	min.	ppb	ppb	%	min ⁻¹
CHCl ₃	120	299	2	99.3	0.053
	90	199	2	99.0	0.053
	90	203	2	99.0	0.065
				$\bar{x} = 99.1$	$\bar{x} = 0.057$ $s = 0.0069$
CHBrCl ₂	90	42.8	1.1	97	0.040
	90	43.3	1.9	96	0.036
	90	30.0	1.0	97	0.040
	90	28.8	1.1	96	0.035
			$\bar{x} = 96.5$	$\bar{x} = 0.038$ $s = 0.0026$	
Mixture	105	238	1.0	99.6	0.053
		(CHCl ₃) 20.5	1.0	97.0	0.040
	105	(CHBrCl ₃) 236	1.0	99.6	0.059
		(CHCl ₃) 23.9	1.0	96.0	0.036
	90	(CHBrCl ₃) 237	3.3	98.6	0.055
		(CHCl ₃) 21.1	1.0	95.0	0.041
		(CHBrCl ₂)			

Table 3. UV Radiation Treatment of Chloroform
and Bromodichloroform
(10.6-watt/l, Temp initial 27-30°C)
final 39-46°C

Chemical Compound	Time of Reaction Min.	Initial Conc. PPb	Final Conc. PPb	Percent Removal %	Removal Rate Min ⁻¹
CHCl ₃	150	292	211	27.8	0.0020
	150	312	209	33.0	0.0025
	300	290	111	25(150 min)	0.0015
	300	305	112	26(150 min)	0.0018
	150	200	136	32	0.0023
	150	176	136	23	0.0015
				$\bar{x} = 27.8$	$\bar{x} = 0.0019$ $s = 0.0004$
CHBrCl ₂	80	42	1.0	97.6	0.045
	80	46	1.0	97.8	0.050
	80	36	1.5	95.8	0.041
	80	44	1.1	97.5	0.046
	80	28	1.0	96.4	0.053
	70	30.2	1.0	96.7	0.054
				$\bar{x} = 97.0$	$\bar{x} = 0.048$ $s = 0.005$
Mixture	150	227 (CHCl ₃)	183	19.4	0.0014
		22.9 (CHBrCl ₂)	1.0	95.6	0.043

Table 4. UV-Ozone Treatment of Chloroform
and Bromodichloroform
(10.6-Watt/l and 0.308 l O₃/min-1)
(Temp. initial 26-30°C, final 28-34°C)

Chemical Compound	Time of Reaction Min.	Initial Conc. PPb	Final Conc. PPb	Percent Removal %	Removal Rate Min. ⁻¹	O ₃ Input mg/min-1	Residual O ₃ mg/l
CHCl ₃	60	298	3	99.0	0.075	10.0	0
CHBrCl ₂	60	20	1.0	95.0	0.096	9.5	0.1
Mixture	30	197 (CHCl ₃)	16	91.9	0.082	---	0.5
		18 (CHBrCl ₂)	1	94.4	0.102		
	60	228 (CHCl ₃)	2	99.1	0.081	12.3	0.3
		15.3 (CHBrCl ₂)	1	93.5	0.094		

Table 5. UV-Ozone Kill of Coliform
 (10.6-Watt/l or 0.308 l O₃/min.-l)
 (Temp. initial 26-30°C, final 28-34°C)

Exposure Time Min.	Initial Coliform Concentration MPN/100 ml	Final Coliform Concentration MPN/100 ml	Other Chemicals in Solution
60	180	0	CHCl ₃
60	220	0	CHBrCl ₂
60	49	0	CHCl ₃ CHBrCl ₂
30	170	0	CHCl ₃ CHBrCl ₂

In a mixture, each chemical compound is removed at a rate independent of the other, i.e., CHCl_3 is removed faster and has a higher percentage removal than CHB_rCl_2 by the same amount as in experiments with solutions containing only a single compound.

It was observed in all experiments of CHB_rCl_2 removal by ozonation that there was an initial 10 to 15 minutes of lag period during which time the removal was extremely slow. This period as shown in Figure 2 was excluded from the calculation of the removal rate, k . While the mechanism and the pathway of reactions in ozonation was not investigated in this study, the fact that the lag period was never observed in the CHCl_3 treatment indicates possible different reactions involved, at least in the beginning, for the breakdown of the two compounds. When ozone gas is introduced into the column reactor, part of it dissolves into solution to provide a possible oxidation process while the remaining escapes from the reactor, stripping some CHCl_3 and/or CHB_rCl_2 from the solution. Consequently chemical oxidation and physical purging of the two THM species are expected although the relative amount of removal by either process is not known. Nevertheless the overall removal is high and ozonation is considered a successful process for CHCl_3 and CHB_rCl_2 removal.

By comparison, the result of the air purging experiments is as promising as those of the ozone treatment. Chloroform can be removed at a slightly faster rate and CHB_rCl_2 at a slightly slower rate when the air flow rate is at 0.308 l/min.-l of solution. This flow rate is identical to the ozone gas flow rate used in ozone experiments. Since much less air dissolves into water than ozone does in the column reactor, air stripping is expected to be more efficient than stripping with

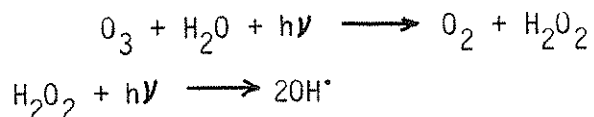
ozone. However the ozone treatment process provides oxidation in addition to the physical stripping of the chemical compounds. The oxidation removal of CHB_rCl_2 more than makes up the decrease in removal due to stripping. The fact that air stripping alone removes CHCl_3 and CHB_rCl_2 at a rate comparable to the ozone treatment process clearly indicates that air stripping is a much more cost-effective process.

Ultraviolet radiation is proven to be ineffective for CHCl_3 removal. With a power input of 10.6-watt/l and an intensity of 15,800 $\mu\text{w}/\text{cm}^2$, only 19.4 percent to 27.8 percent removal is obtained after 150 minutes of exposure, leaving 111 to 209 ppb CHCl_3 in the solution, a concentration exceeding the limit of the drinking water quality standards. The removal rate is 0.0019 min.^{-1} . At this rate, it would take 20 hours of exposure time to reduce 300 ppb CHCl_3 down to 30 ppb. This low photolysis is not unexpected since CHCl_3 does not appreciably absorb radiation in the UV region.

Although CHB_rCl_2 is similar to CHCl_3 in its chemical structure, it behaves significantly different in that CHB_rCl_2 absorbs UV radiation strongly. This leads to a high rate of photolysis at 0.048 min.^{-1} with the same power input of 10.6 w/l and UV intensity of 15,800 $\mu\text{w}/\text{cm}^2$. Ninety-seven percent removal is obtained in 80 minutes of exposure time. In a solution with a mixture of both CHCl_3 and CHB_rCl_2 , each breaks down independantly at its own rate. This finding is similar to that in the ozone treatment process.

In the UV-ozone process, ozone oxidation of CHCl_3 and CHB_rCl_2 is not expected to take place since ozone is breaking down quickly into O_2 and O in the presence of UV. The oxygen atom combines with a water

molecule to form H_2O_2 . UV radiation then converts H_2O_2 into hydroxyl radicals.



Hydroxyl radicals are possibly the active species primarily responsible for the oxidation of CHCl_3 and CHB_rCl_2 with a possible chain reaction. Table 4 shows very low ozone residue concentration in the solution exposed to UV, indicating a rapid breakdown of ozone in the solution by UV. The removal of CHCl_3 or CHB_rCl_2 by ozone oxidation, if there is any, is therefore minimal. On the other hand the hydroxyl radicals are much more effective in oxidizing both CHCl_3 and CHB_rCl_2 . By comparing the results shown in Tables 1, 3 and 4, it is seen that UV-ozone removes CHCl_3 at a rate of 0.075 min.^{-1} , which is 42-percent high than 0.053 min.^{-1} for ozone treatment and 39 times as fast as 0.0019 min.^{-1} for UV treatment. Similarly, UV-ozone removes CHB_rCl_2 at a rate of 0.096 min.^{-1} which is a 2.3 times the rate of 0.042 min.^{-1} for ozone treatment and 2.0 times the rate of 0.048 min.^{-1} for UV treatment. This rate increase is particularly important for CHCl_3 treatment where UV radiation alone is ineffective.

In this study, the CHCl_3 concentration is much higher than the CHB_rCl_2 concentration, mimicking their respective concentrations found in various water supply systems. The ratio of the rates of the chain termination reactions to propagation reactions is smaller when the chain carrier find it easier to locate the substrate CHCl_3 . This explains why the increase of the rate of removal by UV-ozone over that by photolysis alone is much more for CHCl_3 than for CHB_rCl_2 , a finding which is in agreement with the chain reaction hypothesis according to Glaze (ref. 10).

The power input and ozone dose rate in the UV-ozone process effective in the removal of CHCl_3 and CHB_rCl_2 are also effective in coliform kill. Table 5 shows complete kill of coliform with 30 minutes of exposure time. There is no doubt that a UV-ozone system designed for successful removal of THM species will be successful in coliform kill, resulting in a drinking water meeting both the TTHM concentration and coliform concentration standards.

CONCLUSION

Two major species of TTHM, CHCl_3 and CHB_rCl_2 , which often constitute more than 95 percent of TTHM in drinking water supplies can be removed by air purging, ozonation, UV radiation, and UV-ozone. UV radiation alone removes CHCl_3 only at a very slow rate because the chemical does not appreciably absorb UV radiation. Both air purging and ozonation are moderately effective and remove TTHM at comparable rates. UV-ozone is the most effective process attested to by its fastest removal rate of 0.096 min.^{-1} for CHB_rCl_2 (2.3 times the rate of removal by ozonation and 2.0 times the rate by UV radiation) and 0.075 min^{-1} for CHCl_3 (42 percent higher than the rate of removal by ozonation and 39 times the rate of removal by UV radiation). The increase of the removal rate by UV-ozone over that by photolysis alone is much more significant for CHCl_3 than CHB_rCl_2 . Since a much higher CHCl_3 concentration is used in the study, this can be explained by a chain reaction hypothesis involving the active hydroxyl radicals which require higher THM concentrations to propagate the reactions.

UV-ozone process is also found to be very effective in coliform kill. The process therefore can be used for concurrent THM destruction and coliform kill to meet the drinking water quality standards.

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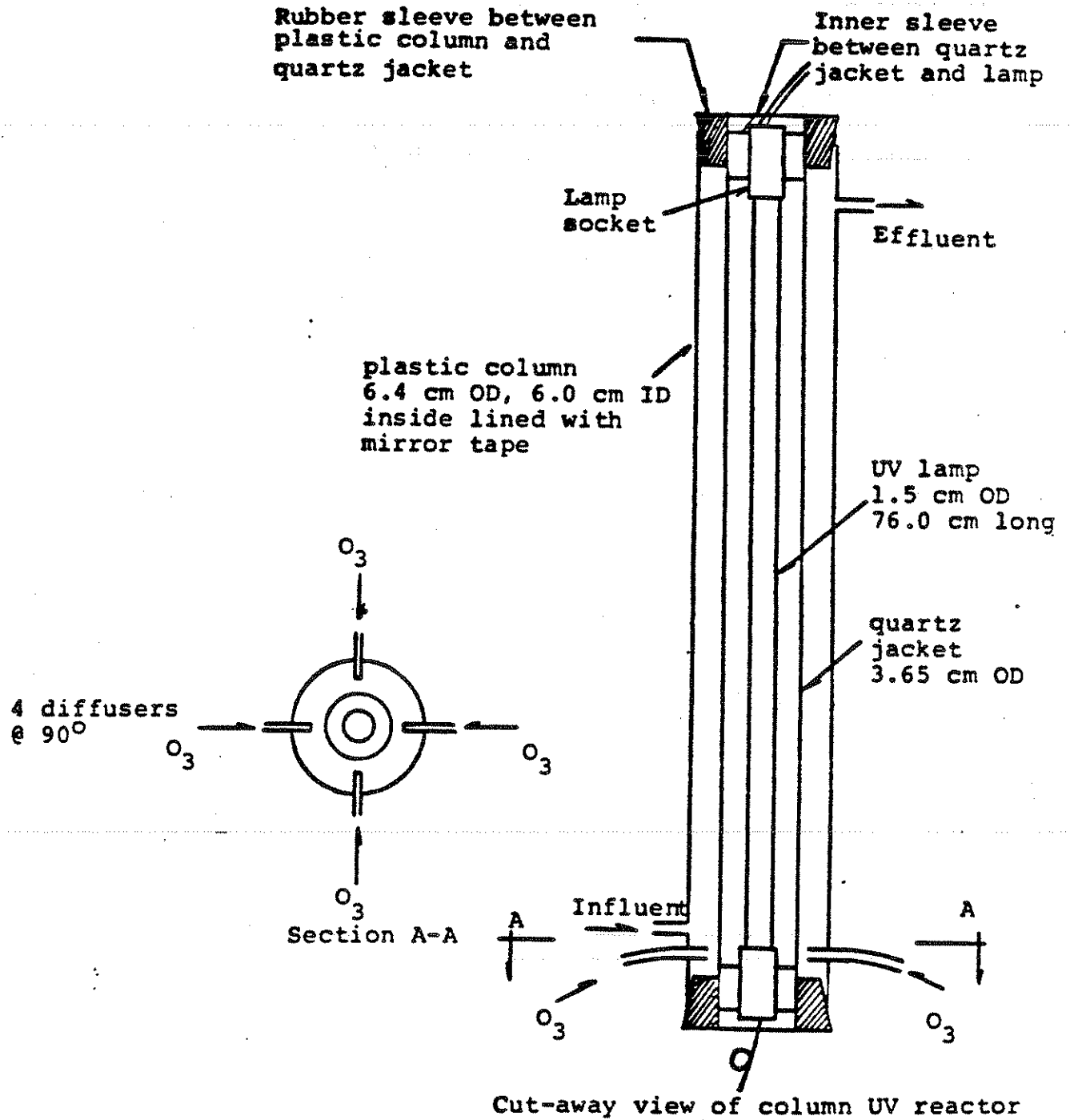


Figure 1

PHOTOCHEMICAL COLUMN REACTOR

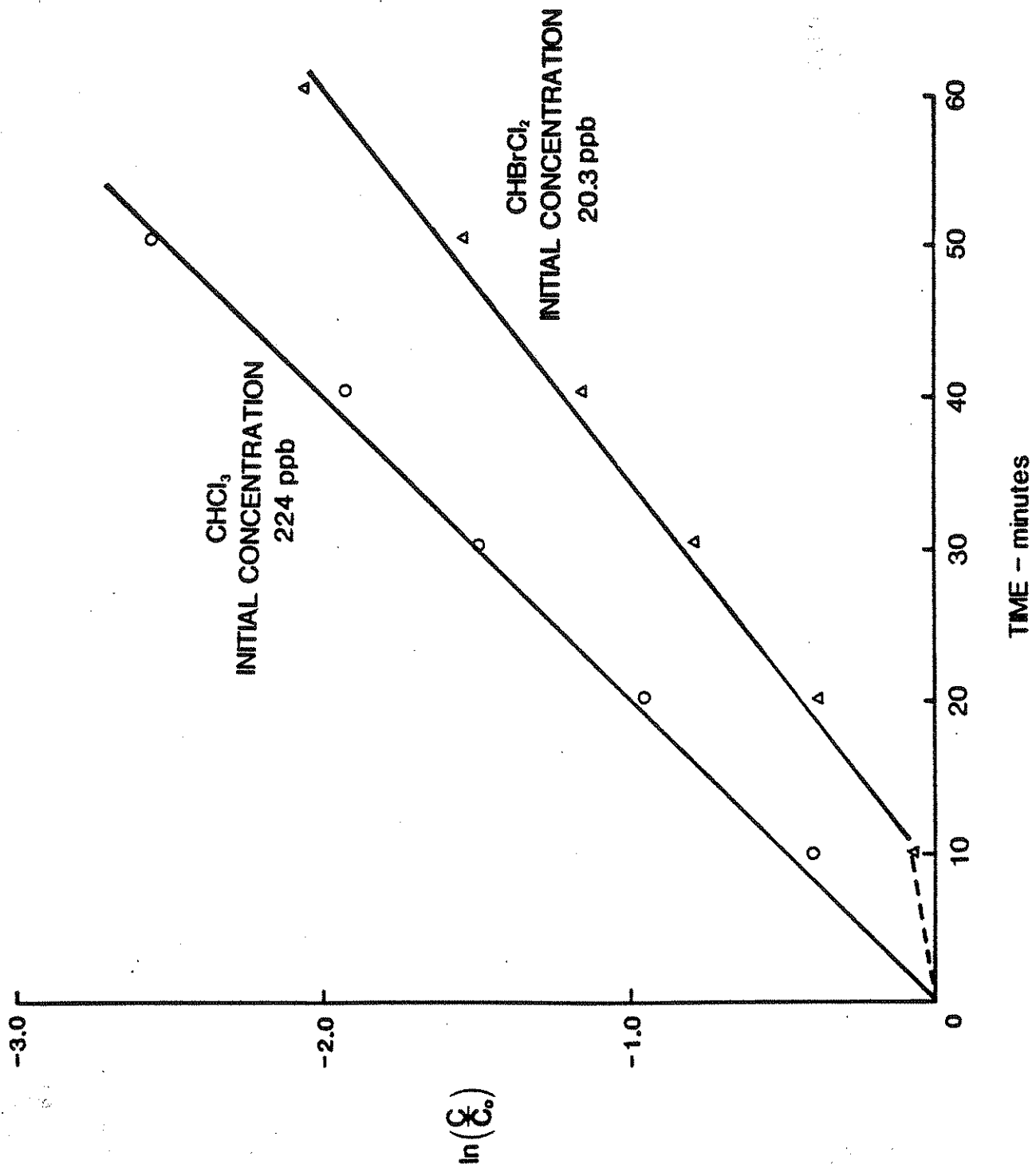


FIGURE 2-- REMOVAL OF CHCl₃ AND CHBrCl₂ BY OZONATION

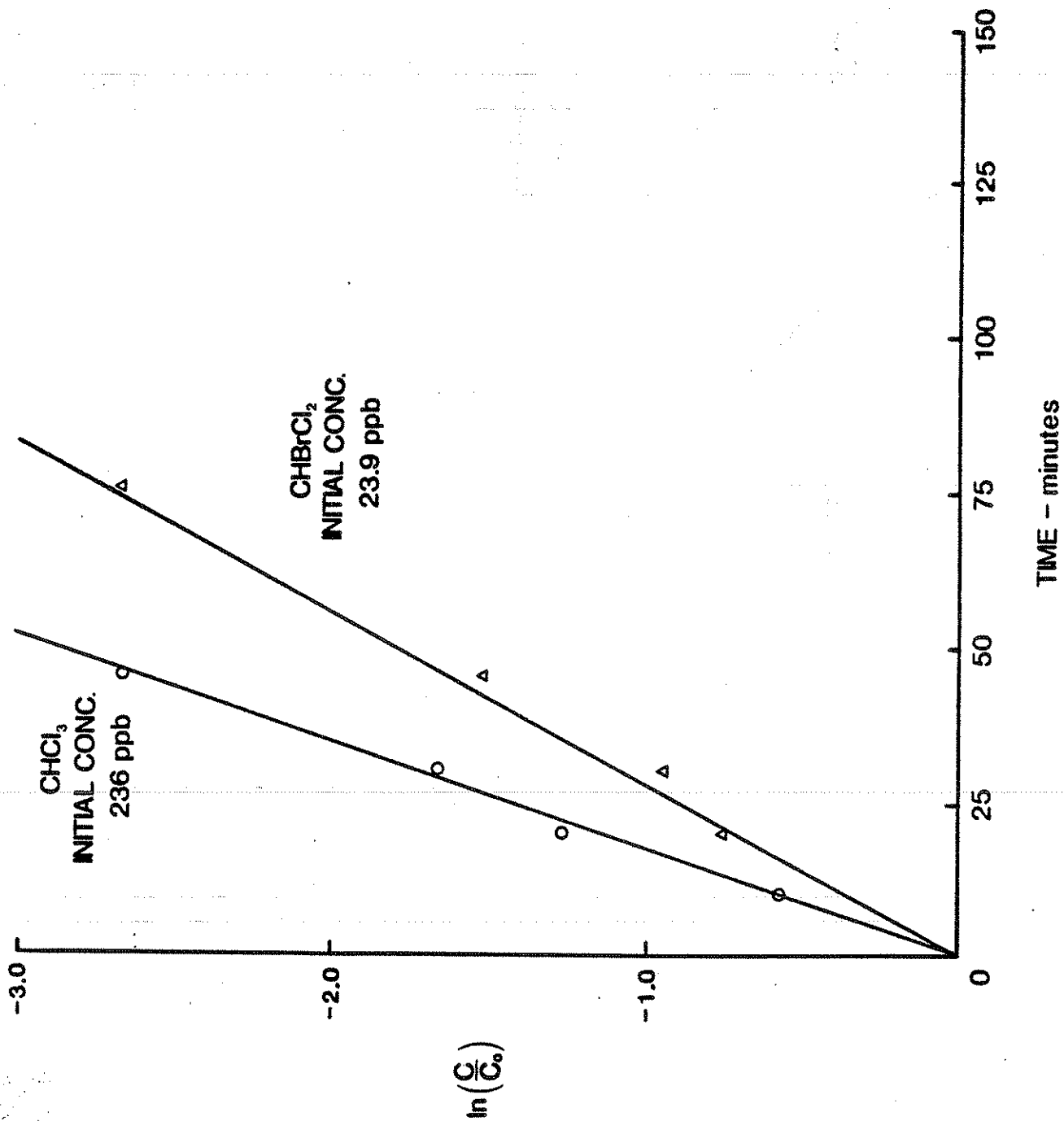


FIGURE 3- REMOVAL OF CHCl₃ AND CHBrCl₂ BY AIR PURGING

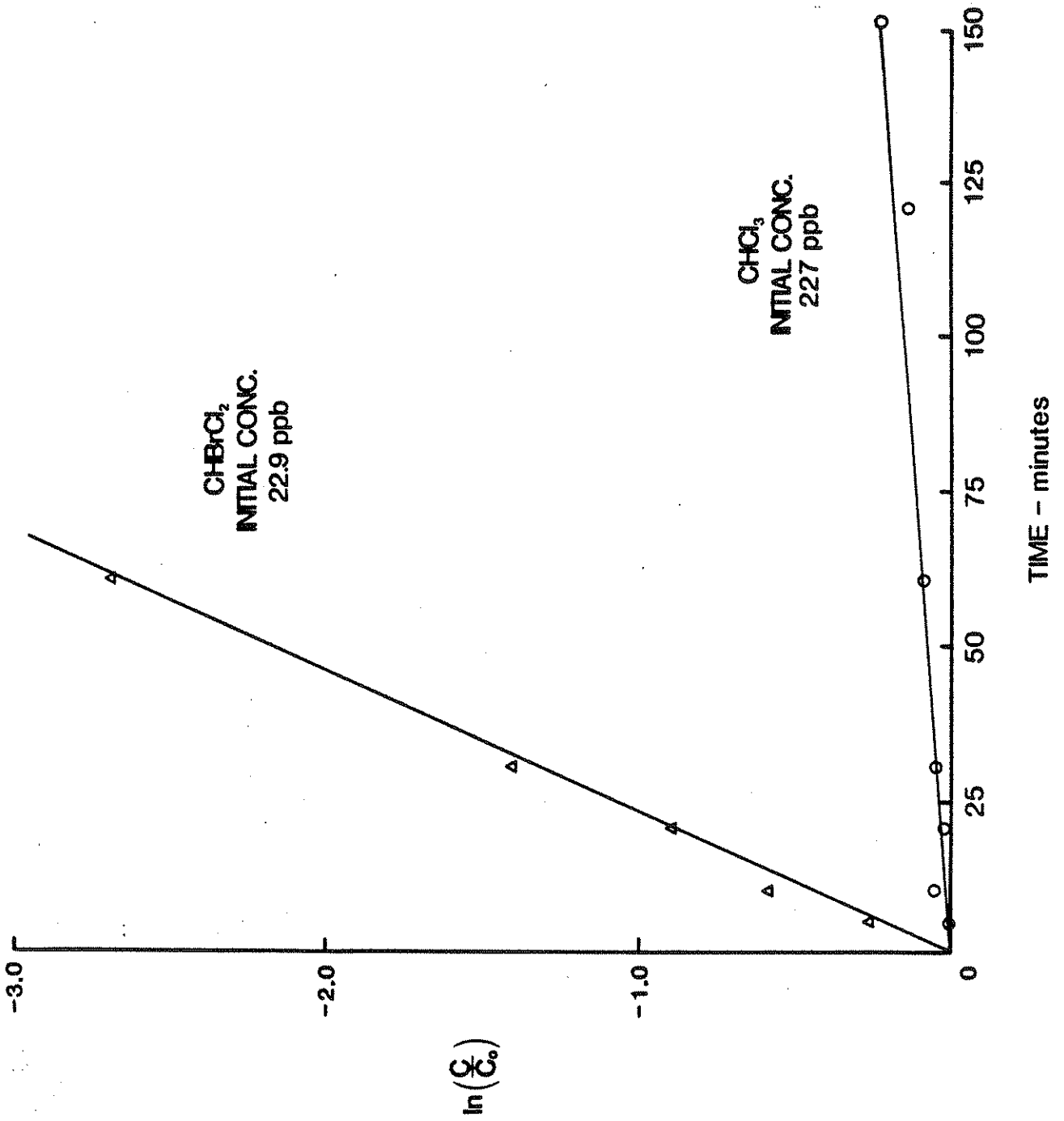


FIGURE 4- REMOVAL OF CHCl_3 AND CHBrCl_2 BY UV RADIATION

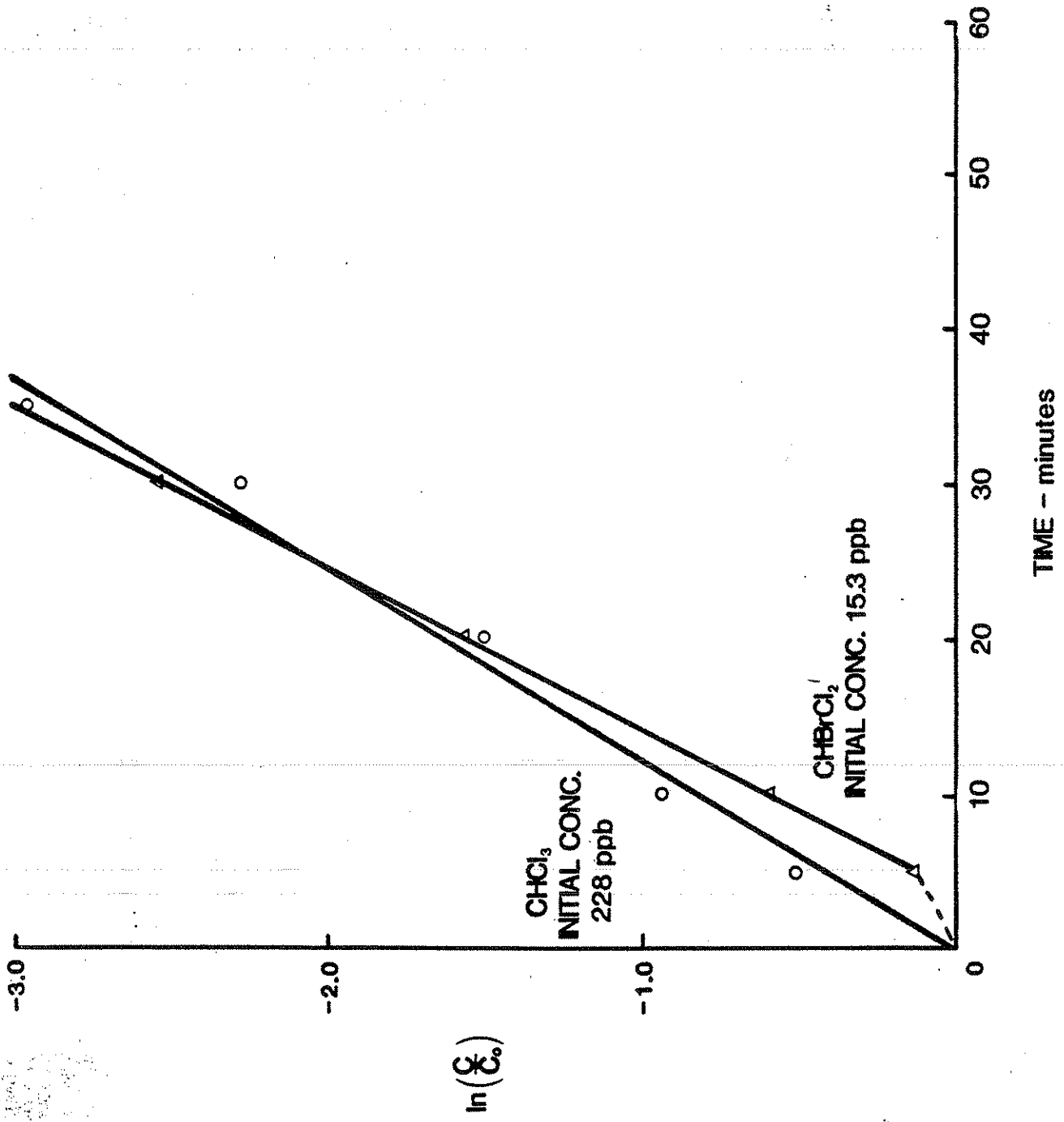


FIGURE 5-- REMOVAL OF CHCl₃ AND CHBrCl₂ BY UV-OZONE

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