The Combustion Products From Vinyl Chloride Monomer

A joint report issued by Task Group "C" of the Safety and Fire Protection Committee of the Manufacturing Chemist Association.

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Objectives

Task Group "C" of the MCA Committee on Vinyl Chloride Transportation Safety was assigned the problem of defining the combustion products from Vinyl Chloride Monomer (VCM). The objectives of this task group were as follows:

(1) Determine the amount of phosgene that may be produced when VCM is burned under known conditions,

(2) Define the total combustion products from VCM,

(3) Develope a mathematical model to simulate the burning of a tank car of VCM and define the concentration of the various combustion products at a given distance from the fire,

(4) Make recommendations as to field testing methods for measuring the concentration of toxic combustion products.

Conclusions

1. Phosgene is a very minor combustion product from VCM. Very small quantities of phosgene (40 - 200 ppm) can be produced when VCM is burning under very specific conditions which involves premixing of VCM with O_2. Without premixing, no detectable amount of phosgene is produced.

   The detection of phosgene, in the presence of the gross quantity of HCl produced from the burning of VCM, is an extremely difficult analytical task.

2. When VCM is burned in the presence of air, an almost quantitative yield of HCl is obtained. The only other combustion products of any consequence are CO, CO_2 and H_2O. Under diffusion conditions, the combustion of VCM produces a very sooty flame. Approximately 10% of the available carbon is converted to carbon black.

3. A computer program has been developed that calculates a profile of combustion product concentrations at ground level at various distances from a VCM fire. Ten windspeeds from 2 to 35 mph are used. The turbulence of the air is also taken into account. The program selects at each of three atmospheric conditions, the maximum ground level concentration of the pollutant and the resulting windspeed at which this concentration took place. Using this windspeed, the program calculates a profile of pollutant concentration, at various distances, to a maximum of 20,000 ft. from the fire.

   The results obtained from this program show that the burning of a single tank car of VCM (200,000#), under the most unfavorable conditions of atmosphere and wind, will produce a ground level concentration of HCl, above the MAC value (5 ppm), up to a distance of 10,000 ft. (approximately 2 miles).

4. From the data collected it appears that carbon monoxide should not be a cause for concern in the vicinity of burning vinyl chloride. Insufficient oxygen and large quantities of HCl seem to be the chief hazards. Hazardous quantities of phosgene may be produced in the immediate vicinity of a vinyl chloride fire.

The Drager tube, when equipped with a zinc scrubber tube, would be valuable for monitoring phosgene levels at the site of a vinyl Chloride Fire.

Introduction

Several recent derailments of tank cars containing VCM have focused a considerable amount of attention on the possible hazards involved in such incidents, particularily when the cars have been ruptured and are subsequently ingnited and burned. From the experienced gain in these incidents, it has become apparent that the VCM industry must be able to respond to emergency situations of this type in a manner that will provide the maximum protection to the general public and will result in a minimum amount of adverse publicity.

The incident at Glendora Mississippi created a panic situation because of the fear that the combustion products from the burning VCM contained gross quantities of phosgene. These fears were not completely without some basis in fact. Phosgene has been described as a major combustion product from VCM in several texts(1)(2)(3) which are considered authoritative in describing the hazards of flammable liquids.

As a result of the joint efforts of the companies involved in the activities of Task Group C we are now able to adequately describe the combustion products from VCM and to place the potential hazards in their proper perspective.

A thorough search of the literature has not revealed a comprehensive study of the combustion products from VCM. Although there are numerous references in well accepted texts on the safe handling of flammable materials that state that phosgene is a combustion product from VCM, these claims are not supported by reference to any research or previous publications on this subject. It is possible that the general association of phosgene in the combustion products from other chlorinated hydrocarbons has been applied to VCM by the authors of these text "just to be on the safe side".

It is well known that phosgene is a combustion product from many highly chlorinated materials. The early history of the toxic nature of these combustion products was reviewed by Sjoberg.(4) The danger of using carbon tetrachloride in fire extinguishers was first demonstrated in 1919 when a fire on board a submarine was extinguished in a closed space. Two members of the crew died with symptoms that resembled phosgene poisoning.

Other publications by Biesolski(5) and Tandberg(6) showed the generation of phosgene when carbon tetrachloride and other chlorinated methanes and ethanes were heated in the presence of air. Among the results obtained, it should be mentioned that carbon tetrachloride yield the highest percentage of phosgene, and that the formation of phosgene decrease with decreasing chlorine content in the chloroethane and chloromethane compounds.

Sjoberg(4) also investigated the formation of phosgene when chlorinated hydrocarbons are brought into contact with hot metal surfaces. Several hundred experiments were run to obtain yields over a wide temperature range for chloroform, carbon tetrachloride, ethylene dichloride, trichloroethylene, perchloroethylene, tetrachloroethane, pentachloroethane and hexachloroethane.

The results of these experiments showed that an insignificant amount of phosgene was formed from the saturated chlorinated ethanes. There was a marked difference between carbon tetrachloride and all of the other materials studied. When CCl_4 is contacted with Fe, the production of phosgene begins at 100°C while the production of phosgene from the other chlorinated compounds does not begin until a temperature of 300°C is reached. The production of phosgene from CCl_4 reaches a maximum at 350°C while the other materials reaches a maximum at 450°C. At 600°C there is little or no phosgene formation from any of these compounds. This has been attributed to the dissociation of phosgene to CO and Cl_2 at this temperature. It is thus evident that, with the exception of carbon tetrachloride, phosgene formation from contacting on hot metal surfaces occurs only within a limited temperature range, approximately

300 - 600°C. The predominating reaction is the formation of hydrogen chloride. As a result ofthis very intensive study of these compounds, Sjoberg came to the conclusion that, with the exception of carbon tetrachloride, the thermal decomposition of chlorinated hydrocarbons produces HCl in sufficient quantities to act as a warning agent (by its odor) and to make the atmosphere insupportable before the phosgene content is high enough to be dangerous.

Additional evidence to support this conclusion by Sjoberg has been reported by Jay[7] as a result of his study of the formation of phosgene from chlorinated hydrocarbons that were decomposed in an electrical arc in the presence of air. Jay made a correlation study in which the number of chlorine, carbon and hydrogen atoms in each molecular structure was related to the generation of phosgene. As a result of this study, the following formula was derived;

$$\alpha = \frac{\text{No. of Cl atoms - No. of H atoms}}{\text{No. of carbon atoms}}$$

Those structures having α values greater than 0.40 gave both phosgene and chlorine as decomposition products while structures with α values less than 0.40 did not. Thus, VCM would have a negative α value and neither chlorine nor phosgene would be expected as degradation products.

Kubler[8], in his investigation of aerosol propellants, reported that trace amounts of phosgene were formed when VCM was sprayed onto a hot surface. The amount of carbon monoxide and phosgene formed between 100°C and 1000°C and the amount of HCl formed at 1000°C were measured. The decomposition of VCM at the latter temperature gave the following results;

110 ppm

1 g VCM – 39.5 mg CO + 0.11 mg $COCl_2$ + 102 mg HCl

Kubler also came to the same conclusion as Sjoberg. The predominate formation of HCl creates an insupportable atmosphere before the level of phosgene becomes dangerous.

All of the previously cited investigators used basically the same analytical techniques for measuring the amount of phosgene formed. This is based on the reaction of phosgene with aniline in water to form diphenylurea and aniline hydrochloride;

$$4\ C_6H_5NH_2 + COCl_2 \rightarrow CO(NHC_6H_5)_2 + 2\ C_6H_5NH_2HCl$$

Sjoberg used two series-coupled scrubbers filled with aniline water and reported that practically all of the phosgene was absorbed in the first flask. After the experiment the diphenylurea was filtered off, dried at 80°C and weighed. The diphenylurea was identified through a nitrogen determination.

The other investigators have used essentially this same technique but have measured the quantity of phosgene using an ultraviolet spectrophotometric technique developed by Crummett.[9] It was found that fairly small quantities of 1, 3-diphenylurea can be determined in the presence of relatively large amounts of aniline in acidic methanol. Although both compounds exhibit absorption maxima at 254.5 $m\mu$, the absorption of diphenylurea on a weight basis is 93.6 times as intense as that of aniline. An excellent review of other analytical methods for the analysis of phosgene in air has also been publish by Lincn,[10] et.al.

Discussion

The analytical results obtained by the four companies participating in this study will be presented here in a rather brief, general fashion. Because of the limited time for these studies, there are still some unanswered questions and some conflicting results. We have, however, been able to provide acceptable answers to the problems set forth in the objectives for Task Group "C".

1. Determine the amount of phosgene produced when VCM is burned under known conditions.

The amount of phosgene produced from burning VCM is an extremely insignificant quantity. Only one of the four laboratories (Dow) involved was able to make a positive identification. In a premixed flame about 40 - 200 ppm could be detected in the total combustion products. Since an almost quantitative yield of HCl is obtained from the burning of VCM, this very trace amount of phosgene is of little toxicological significance.

The fact that little or no phosgene is found in the combustion products from VCM is really not surprising. There is both thermodynamic and experimental data[4] to show that the formation of phosgene cannot take place in a VCM flame. Trace amounts that might be formed by premixing hot VCM vapors with air would also be decomposed at VCM flame temperatures.

When VCM monomer is burned under diffusion conditions (no premixing with air) a yellow, carbonaceous flame is produced. The flame temperature at these conditions is ~950°C. In a premixed flame a clean, yellow-blue flame is produced having a temperature of ~1460°C. These experimentally measured values are in excellent agreement with the calculated flame temperatures for these two conditions (870°C and 1530°C respectively).

While we have learned that it is extremely difficult to burn VCM under conditions so as to produce even very trace quantities of phosgene, we have also learned that the detection and positive identification of very trace amounts of phosgene in the total combustion products is also a difficult task.

One of us has thoroughly explored the aniline water method of detection and other methods[10] described in the literature and found that there are several limiting effects that preclude their use in the analysis of the combustion products from VCM. It was soon learned that all of these methods are extremely sensitive to HCl. The major analytical problem that developed was the analysis of phosgene in the presence of HCl.

We then directed our efforts toward the construction of an apparatus in which the combustion gases from VCM could be directed through a series of absorption trains to remove the HCl and H_2O. The initial equipment design consisted of a total combustion burner into which VCM and air could be fed at measured rates. This burner was fitted into a metal base and covered by a chimney having a side arm leading to a series of traps. By connecting a vacuum pump to the final trap, the total combustion products were pulled through the absorption train containing traps filled with molecular sieves (to remove H_2O), an inorganic base (to remove HCl) and finally into a trap containing a specific detector solution for phosgene.

Designed experiments with this apparatus revealed that molecular sieves and inorganic bases strongly adsorbed phosgene. Indeed, other experiments showed that phosgene has a strong affinity for clean, unseasoned glassware.

It soon became apparent that the phosgene could not be separated from the gross quantity of HCl evolved from burning vinyl chloride and that any analysis of phosgene would have to be carried out in the presence of large amounts of HCl.

A number of techniques were explored for the analysis of phosgene in the presence of HCl.

Linch et.al.[10] have reviewed a number of methods for quantifying phosgene in the low ppm range; unfortunately all of these specific methods are badly interferred with by HCl. One method, a colorimetric determination based on the condensation of phosgene with bis (4,4' diethylamino) benzophenone (yellow) to produce a green product was investigated. Low levels of phosgene (in the absence of HCl) could be readily determined spectrometrically. However, high concentrations of HCl converted the indicator into the amine hydrochloride which was then insensitive to phosgene.

Another method involving the reaction of phosgene with 2,4 dinitrophenyl-hydrazine to produce 2,4 dinitrophenyl hydrazone was also attempted. Again, HCl badly interferred with the analysis.

A method involving the reaction of phosgene (and HCl) with aniline seemed to offer the most hope. Crummett[9] has modified this technique (previously used gravimetrically) for application with U.V. spectroscopy. The overall reaction of phosgene with aniline involves the reaction products diphenylurea (DPU) and aniline hydrochloride. Although both absorb at 254.5 mμ in the U.V. region of the spectrum, aniline hydrochloride has an additional absorption at 260.5 mμ. Since the intensity of the absorption for DPU is ~100 times greater on a weight basis than that of aniline hydrochloride, the concentration of DPU in the presence of large quantities of aniline hydrochloride can be determined.

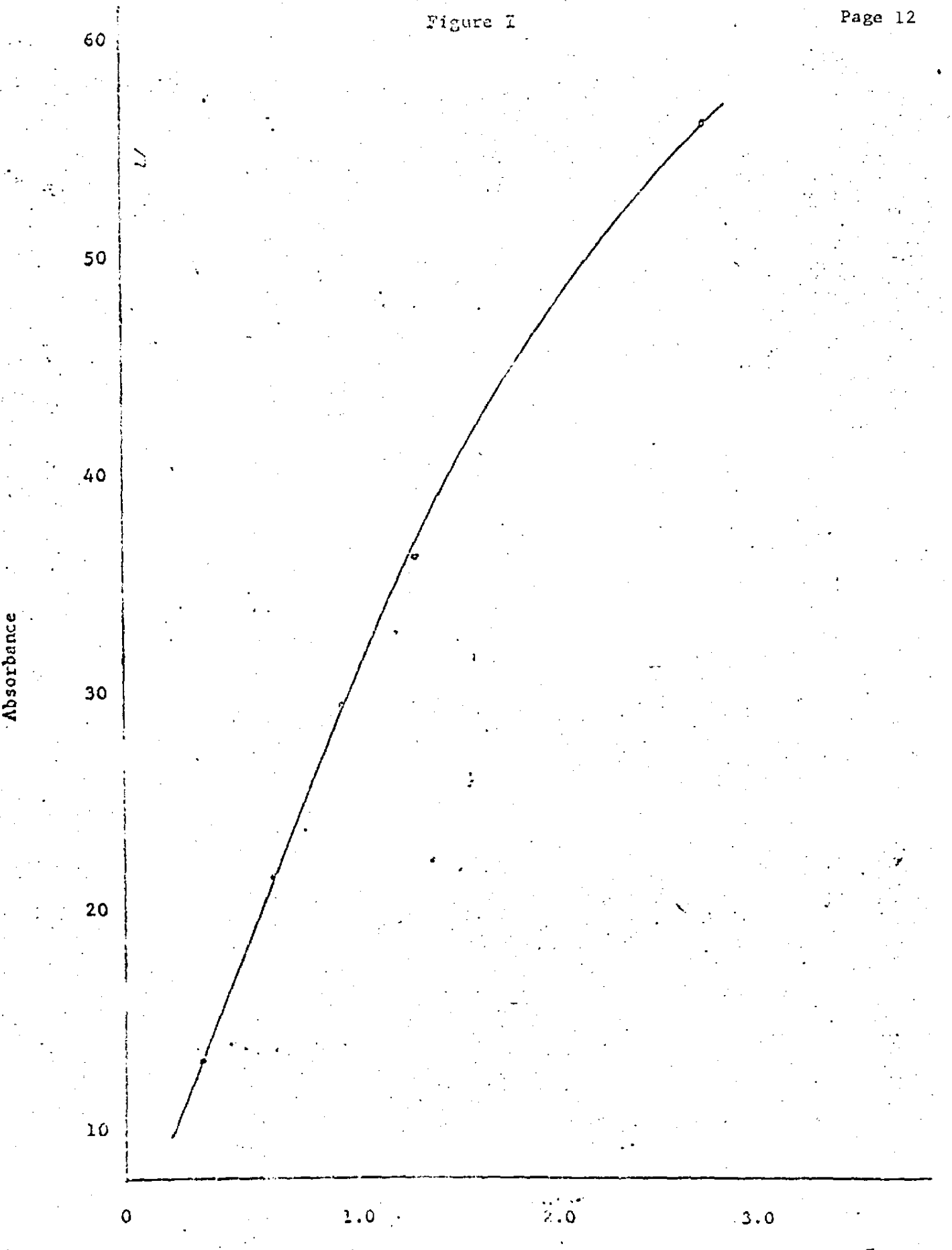
If it is assumed that the major combustion product from vinyl chloride is HCl, a detector solution containing 1.6g aniline/50ml of water (3.2% solution) would be required to neutralize the HCl resulting from the burning of 1.0g of vinyl chloride (~580mg HCl). If it is further assumed that low levels of phosgene (1-10 ppm on a vinyl chloride basis) are produced during the burning of vinyl chloride, it can be seen that the detector solution at the end of the reaction will contain low levels of DPU (2-22 ppm) and large quantities of aniline hydrochloride (>32,000 ppm). Obviously, this amount of aniline hydrochloride negates a spectrophotometric determination of the DPU.

If the decomposition products from vinyl chloride are bubbled through the aniline solution and no precipitate (DPU) forms, the sensitivity of the determination is then dependent upon the solubility of DPU in the aniline hydrochloride solution.

The spectrophotometric response of DPU in solution was determined by preparing an analytical sample of DPU in methanol. The proper dilutions were made and each of the solutions were spectrally analyzed. A plot of concentration vs % absorbance is shown in Figure I.

The solubility of DPU in water (no aniline hydrochloride) was then measured by stirring an excess of DPU in distilled water followed by a spectrophotometric analysis of the resulting solution. It was found that the solubility of DPU in water was 2.07 ppm (W/V). In 50 ml of water, this level of 2.07 ppm would be $1.12 \cdot X\ 10^{-4}$ g of DPU. The amount of phosgene required to produce this amount of DPU would be $5.09X10^{-5}$g ($1.12X10^{-4}$g · DPU/ 2.2). Based on 1 g of vinyl chloride, this amount of phosgene would be 50.9 ppm which would be the limit of detection.

Figure I



It then became important for us to know whether the solubility of DPU in a 3% aniline hydrochloride solution would be lower due to the "salting out" effect of the aniline hydrochloride.

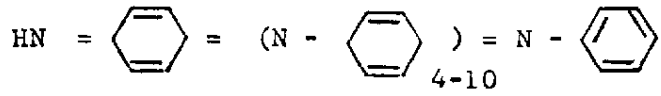
To answer this question, a series of solutions containing various amounts of methanol/water and 3% aniline hydrochloride were saturated with diphenylurea. The concentration of DPU in each of the solution was then determined spectrophotometrically. The results of this study showed that as the % MeOH decreased from 50% to 20%, the concentration of DPU dropped from 116 ppm to less than 1 ppm. The concentration of DPU in a aqueous aniline hydrochloride (0% MeOH) solution would then be well below 1 ppm. To test this, 0.815 mg DPU was stirred into 2000 ml of a 4% aniline HCl solution (0.41 ppm). At the end of a 4 hour stirring period, undissolved DPU was still present in the solution.

As a result of this work, the solubility of DPU in a 3-4% aniline HCl solution was taken to be less than 0.5 ppm (W/V). Based on the concentration of the aniline detector solution and on the amount of vinyl chloride burned, the limit of detection of phosgene is ~9 ppm.

Using the above described method, a number of combustion experiments were run. Since a diffusion flame more closely simulates a VCM fire, all of the phosgene determinations were carried out using a diffuse flame (no premixing of VCM with air). The combustion gases were bubbled directly (no absorption trains) into a 3% aqueous aniline solution.

In a typical experiment, 1 g of VCM yielded 5-15 mg of precipitate. If it is assumed that this precipitate is diphenylurea, the amount of phosgene present in the combustion products would be 2000-7000 ppm. An ultraviolet spectrum of this precipitate showed an absorption at 254.5 mμ,

thereby indicating the presence of diphenylurea. However, a mass spectrum of this precipitate showed a complete absence of diphenylurea. The major volatile components in this precipitate have molecular weights of 182 and 184 and have been previously identify as in the products formed during the oxidative coupling of aniline. In addition to these volatile materials, we could also identify a polymeric material having the following structure;



In summary, the total amount of precipitate formed in the aqueous aniline solution was due to the oxidative coupling of anline.

It was pointed out that the formation of diphenylurea is greatly retarded at low pH's. Since a technique has been developed for removing HCl from phosgene (Dow), the above work was repeated using a pre-scrubber of mossy zinc. This effectively removed the HCl from the combustion products. However, this new approach did not alter the outcome of the experiment... no diphenylurea was formed.

Another approach to the analysis of phosgene in the combustion products from VCM is to use gas chromatography. This is the only method used by the members of Task Group C that resulted in a positive identification. Since the results of this study have been described in an excellent report by Roger Daniel (See Appendix II) it would be redundant to repeat them here.

It is our opinion, that the results obtained by Roger conclusively show that a trace amount of phosgene is produced from the burning of VCM. His plot of VCM rate vs. ppm $COCl_2$ in the combustion products is an excellent illustration of the effect of feed composition and burning rate on phosgene production.

2. Define the Total Combustion Products from VCM

When VCM is burned in the presence of air the combustion products contain 51.9% HCl, base on the weight of the monomer burned. This accounts for 89.1 of the available chlorine in VCM (assuming that 100% of the monomer is burned---which is not very likely). Under diffusion conditions, the combustion of VCM produces a very sooty flame. The amount of carbon black formed is dependant on the rate of burning. One should also keep in mind, that under diffusion conditions the amount of available O_2 does not increase when the burning rate is increased. Consequently, the increased production of soot at higher burning rates is actually due to the limiting of available O_2 for complete combustion.

The amount of carbon black formed from the burning of VCM at two different rates is shown in the following table:

Amount of VCM Burned	4.0 g	8.8 g
Rate	0.133 g/min.	0.088 g/min.
Wt. of Carbon Formed	0.226 g	0.235 g
% of Available Carbon	14.7%	7.0%
% of VCM Burned	5.65%	2.67%

Similarly, if we change the burning rate and consequently the amount of O_2 available for complete combustion, we can expect to see the same effect on the total amount of CO_2 and CO and the ratios of these two components.

A gas chromatogram of the total combustion products was obtained using a dual column system consisting of a molecular sieve column and a Porapak column connected in parallel-series through a 6 part valve. This system

adequately separates all of the combustion products* including CO, CO_2, air and trace amounts of ethylene and unburned monomer. The amount of HCl in the combustion products was measured separately by titration.

To arrive at a complete quantitative description of the total products obtained from the burning of VCM, we have combined the results obtained from these three determinations (weight of carbon black plus HCl measured by titration plus CO_2, CO, ethylene and unburned VCM by gas chromatography).

	Wt. %
HCl	52
Carbon Black	4
CO_2	33.7
CO	10.3
Ethylene	trace
VCM (unburned)	trace
Phosgene	40 - 200 ppm

These are too high because they are not diluted with Air. ÷ by 10

this is conc. immediately above the flame

The ratio of CO_2/CO is 4.27/1.00. This value is reasonably close to results obtained by Coleman and Thomas[11] in their analysis of the combustion products from PVC at about this same temperature. Their measured ratio of CO_2/CO was 5.80/1.00.

It is evident that a study of all of the variables that might have an effect on the yield of combustion products from VCM would require a major research effort and was not within the scope of our present objectives. These variables could include burning rate, available O_2 and flame temperature. These results would probably show that, as with PVC,[12] the production of HCl is independent of O_2 concentration at this temperature (950°C) while the concentration ratio of CO_2/CO is dependent on available oxygen and flame temperature.

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*HCl is poorly resolved on this dual system.

3. Develop a Mathematical Model to Simulate the Burning of a Tank Car of VCM and Define the Concentration of the Various Combustion Products at a Given Distance from the Fire.

A computer program was written (based on equations by Bosanquet and Pearson)[13] that will determine at each of three atmospheric conditions--adiabetic, isothermal and inverson---the maximum ground level concentration of the toxic combustion products (HCl and CO) from the burning of a tank car of VCM. The program first determines the effect of atmospheric wind speed on a fire or venting tank car at a fixed height above ground level. Ten different wind speeds from 2 to 35 mph can be used. It then selects, at each of the atmospheric conditions, the maximum ground level concentration and the resultant wind speed at which this concentration took place. Using this windspeed the program then calculates a profile of pollutant concentration at ground level, at various distances from the fire, to a maximum of 20,000 ft. The turbulence of the air is also taken into account, i.e., whether it is low, average or moderate.

For the purpose of developing this program, we took a hypothetical situation which involved the burning of a 26,000 gal. tank car (~200,000#) of VCM. We assumed that the burning reaction would be as follows.

$$CH_2 = CHCl \xrightarrow{\Delta} HCl + CH \equiv CH$$

$$CH \equiv CH \xrightarrow[1\ 1/2\ O_2]{\Delta} CO + CO_2 + H_2O$$

This would yield the following amounts of combustion products;

		Liq. Vol. %
HCl	116,000#	42.7
CO	44,000#	21.2
CO_2	74,000#	22.6
H_2O	29,000#	13.5
	264,000#	100.0

The contents of a 200,000# car, burning at a constant rate of 6200 CFM through a 1 ft. rupture, will be consumed in 48 hours.

Figure I, Appendix I, shows a plot of HCl concentration (ppm) at ground level vs distance (ft.) from the fire. The effect of turbulence on the ground level concentration of HCl are also shown for a fire burning at a rate of 6200 CFM in an adiabatic atmosphere.

In using the data shown in Figure 1 to establish a safe distance from the fire, one must also consider the maximum allowable concentration (MAC) of the pollutant (long term, short term and lethal).

	Maximum Allowable Concentration	Short Term Exposure	Lethal Concentration
CO	50 ppm	400 ppm	1500 ppm
HCl	5	50	1000-2000
VCM	500	16,000	120,000

Using these values, it is evident that the ground level concentration of HCl (using the wind speed for worst condition) is reduced to the above MAC value at a distance of about 2 miles from the fire.

Figures 1 - 7, Appendix I, show the effects of atmospheric condition, turbulence and emission rate on HCl and carbon monoxide concentrations at ground level at a given distance from the fire. These same variables have also been plotted for VCM venting from a ruptured tank car but not burning (Fig. 8-12).

In general the results of this study shows that the ground level concentration of the combustion products is most sensitive to wind velocity, air turbulence and atmospheric conditions (adiabatic, isothermal or inversion). Increased turbulence and wind speed reduces the concentration of toxic combustion

products. Isothermal and inversion conditions present a spec case where the concentration of combustion products may increase at a distance up to 1200ft from a burning tank car.

Recommendations as to Field Testing Methods for Measuring the Concentration of Toxic Combustion Products

The details of this study are included in a separate report on this subject by J.E.Newell of Uniroyal.

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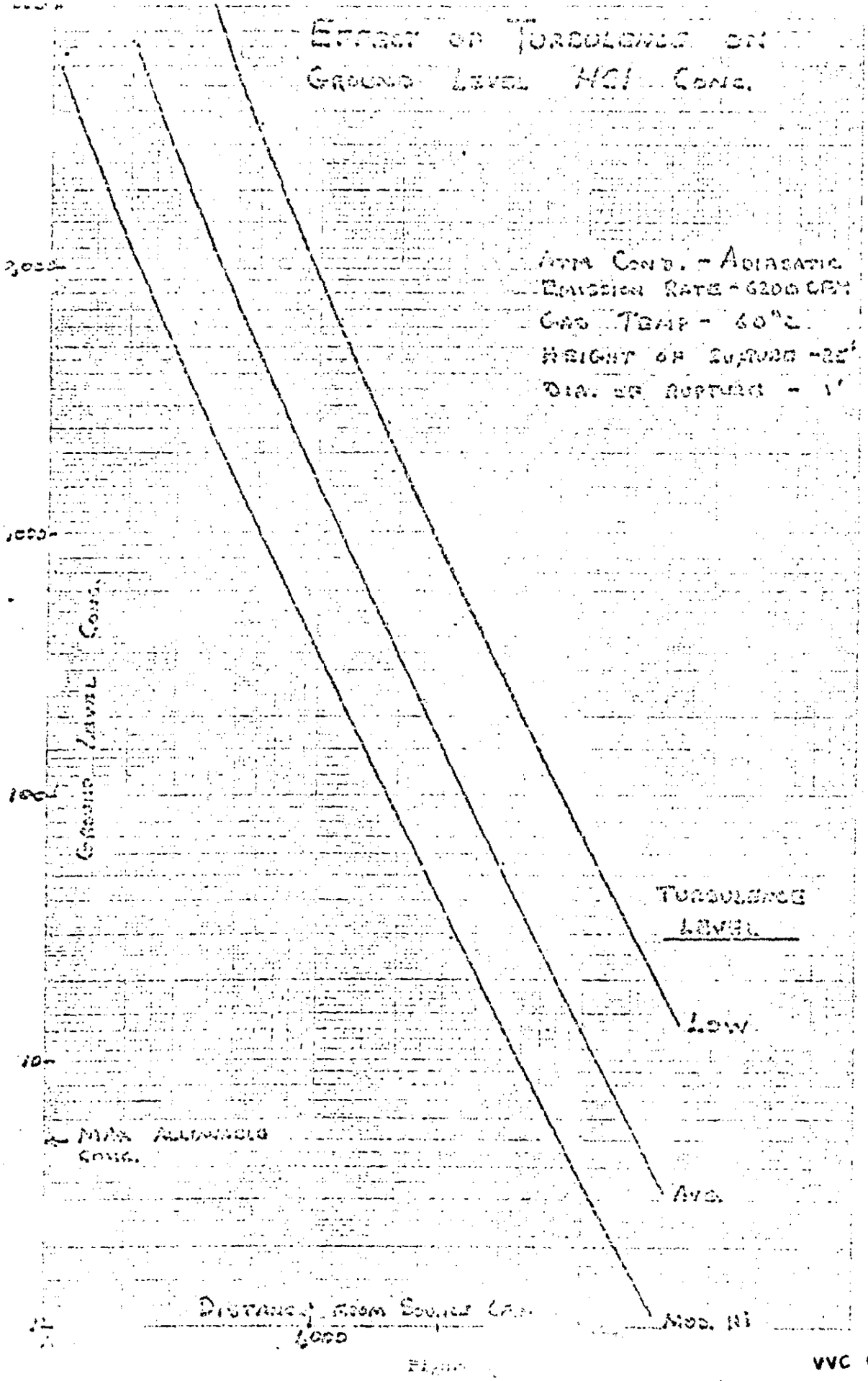
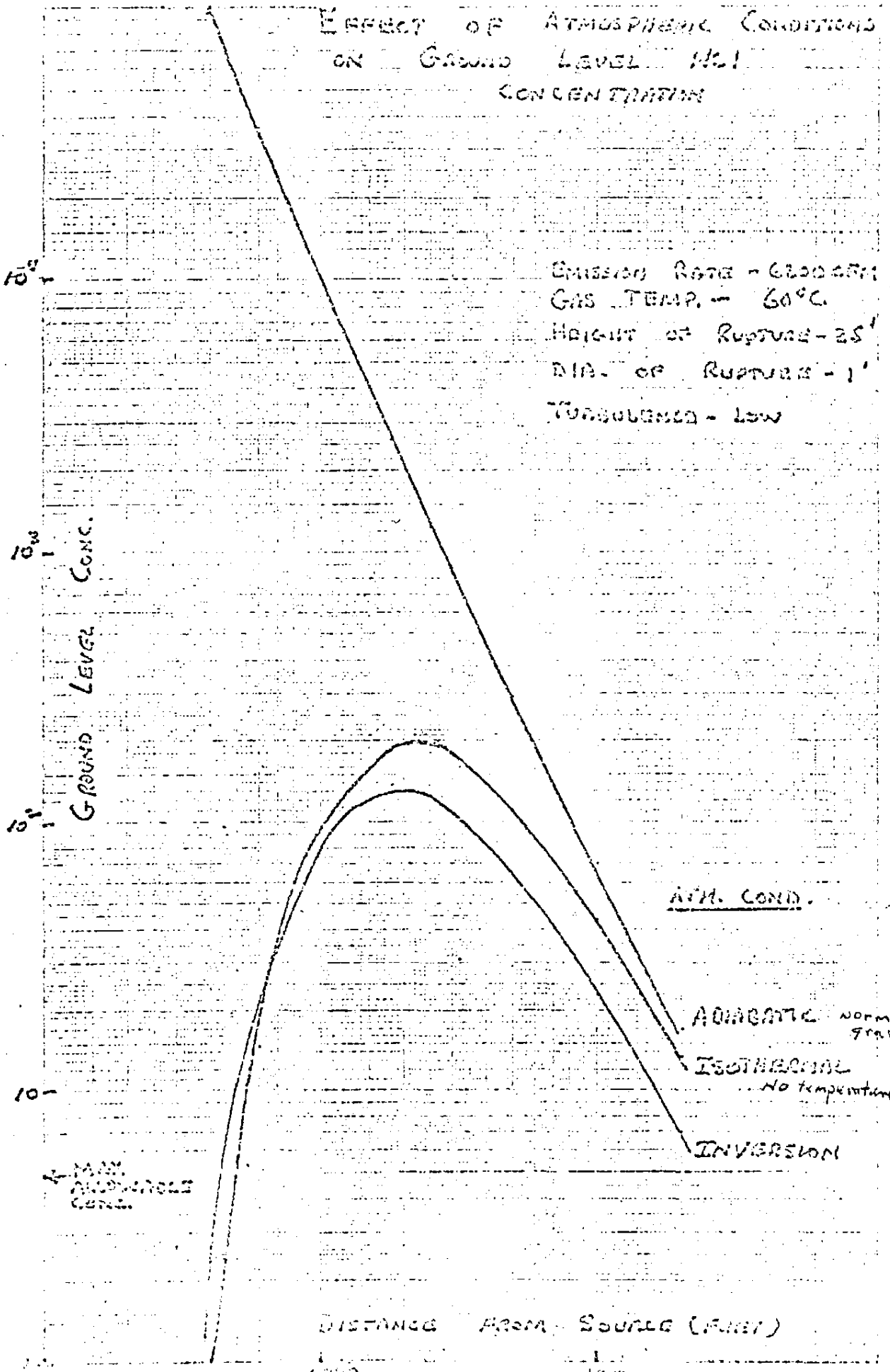
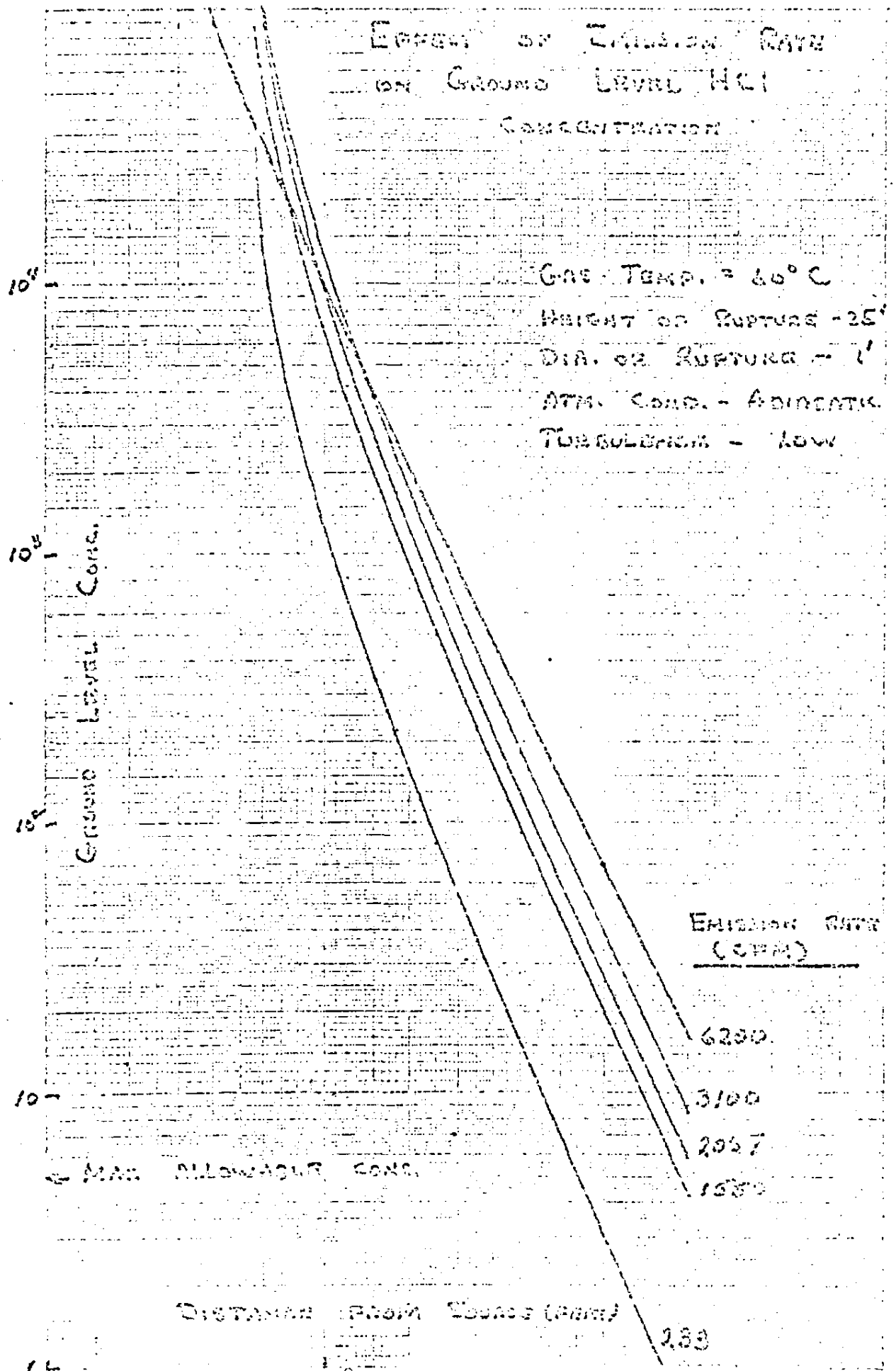
The results of a study to show the effects of turbulence, atmospheric conditions, emission rate and diameter of rupture hole on the ground level concentration of HCl, CO and VCM.

Appendix I



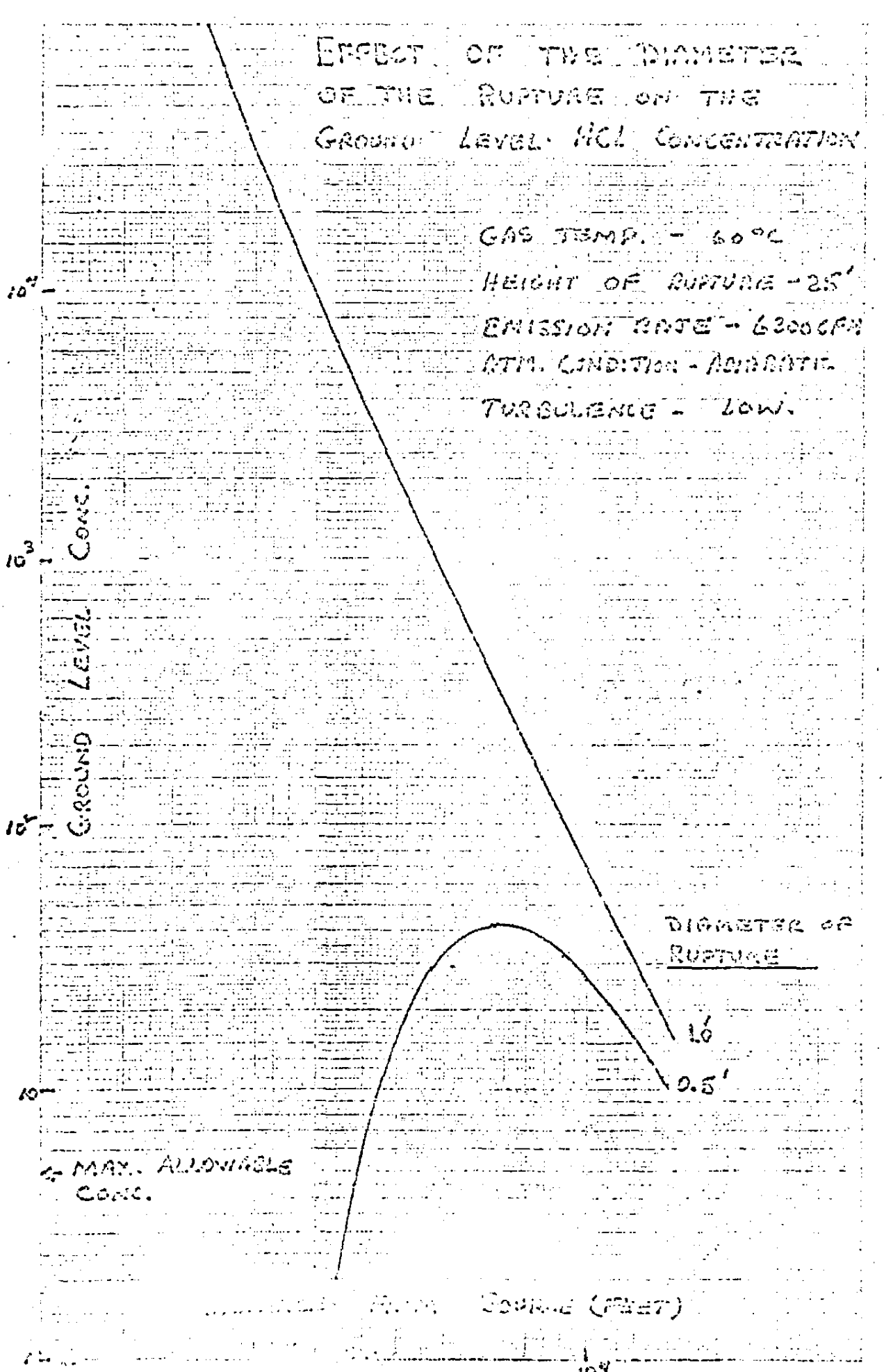
Figure


EFFECT OF ATMOSPHERIC CONDITIONS ON GROUND LEVEL H2S CONCENTRATION
EMISSION RATE - 6200 SCFM
GAS TEMP. - 60°C.
HEIGHT OF RUPTURE - 25'
DIA. OF RUPTURE - 1'
TURBULENCE - LOW
GROUND LEVEL CONC.
ATM. COND.
ADIABATIC normal temperature gradient
ISOTHERMAL ... No temperature gradient
INVERSION
DISTANCE FROM SOURCE (FEET)

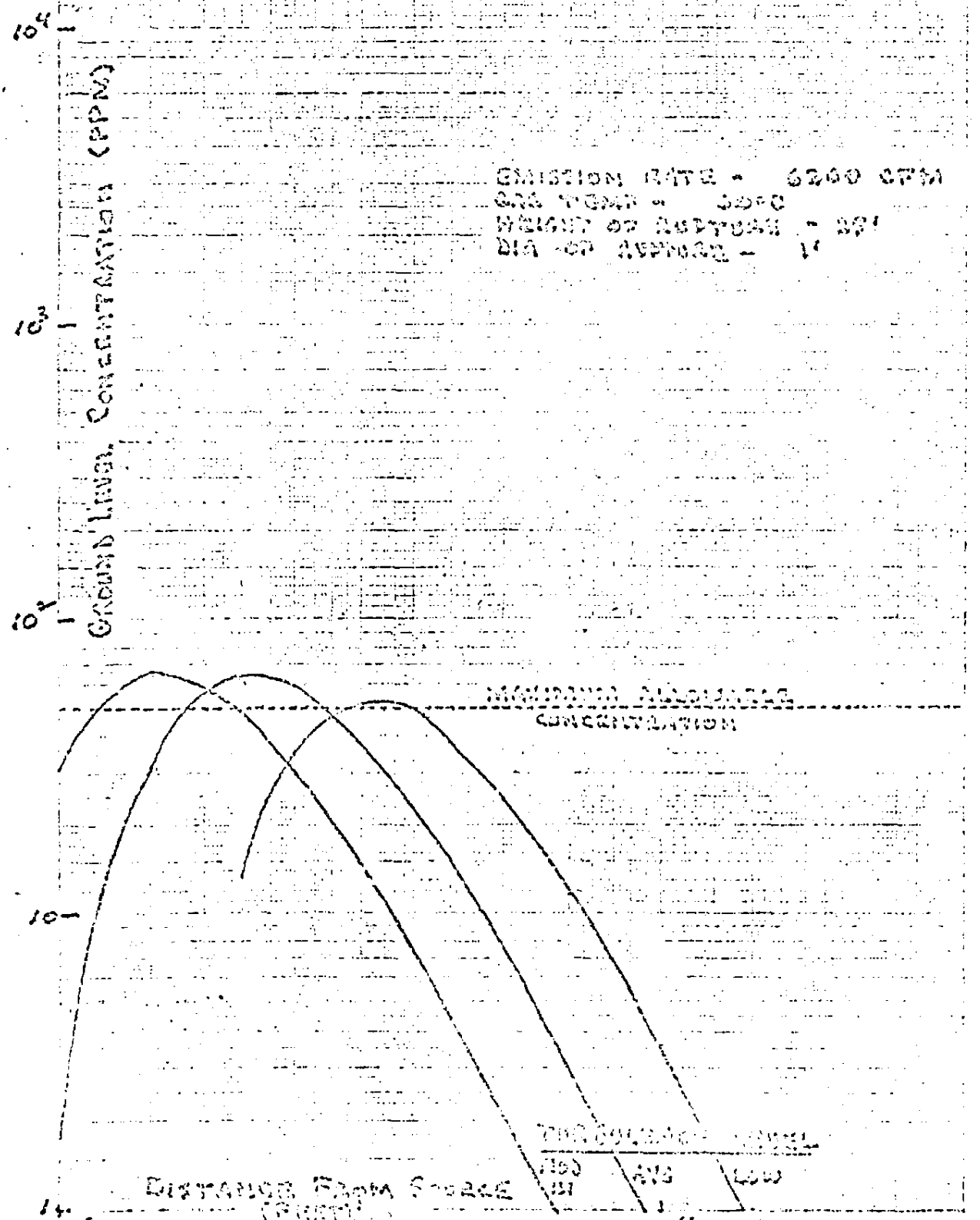

Effect of Emission Rate on Ground Level HCl Concentration
Gas Temp. = 60° C
Height of Rupture = 25'
Dia. of Rupture = 1'
Atm. Cond. - Adiabatic
Turbulence - Low
Ground Level Conc.
Max. Allowable Conc.
Emission Rate (CFM)
6200
3100
2067
1550
Distance from Source (ft.)
250

Effect of the Diameter of the Rupture on the Ground Level HCl Concentration

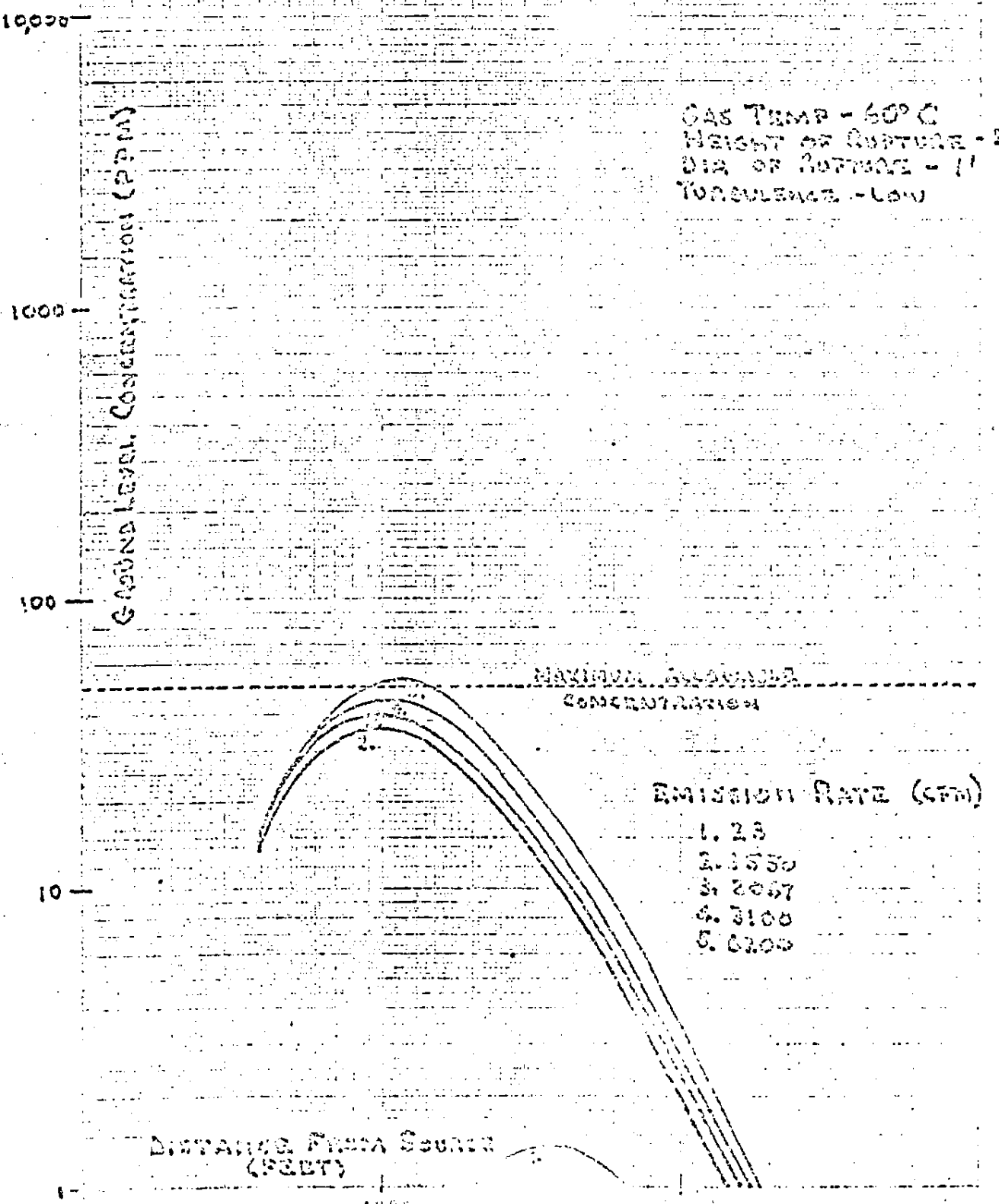
Gas Temp. - 60°C
Height of Rupture - 25'
Emission Rate - 63000 CFM
Atm. Condition - Adiabatic
Turbulence - Low.



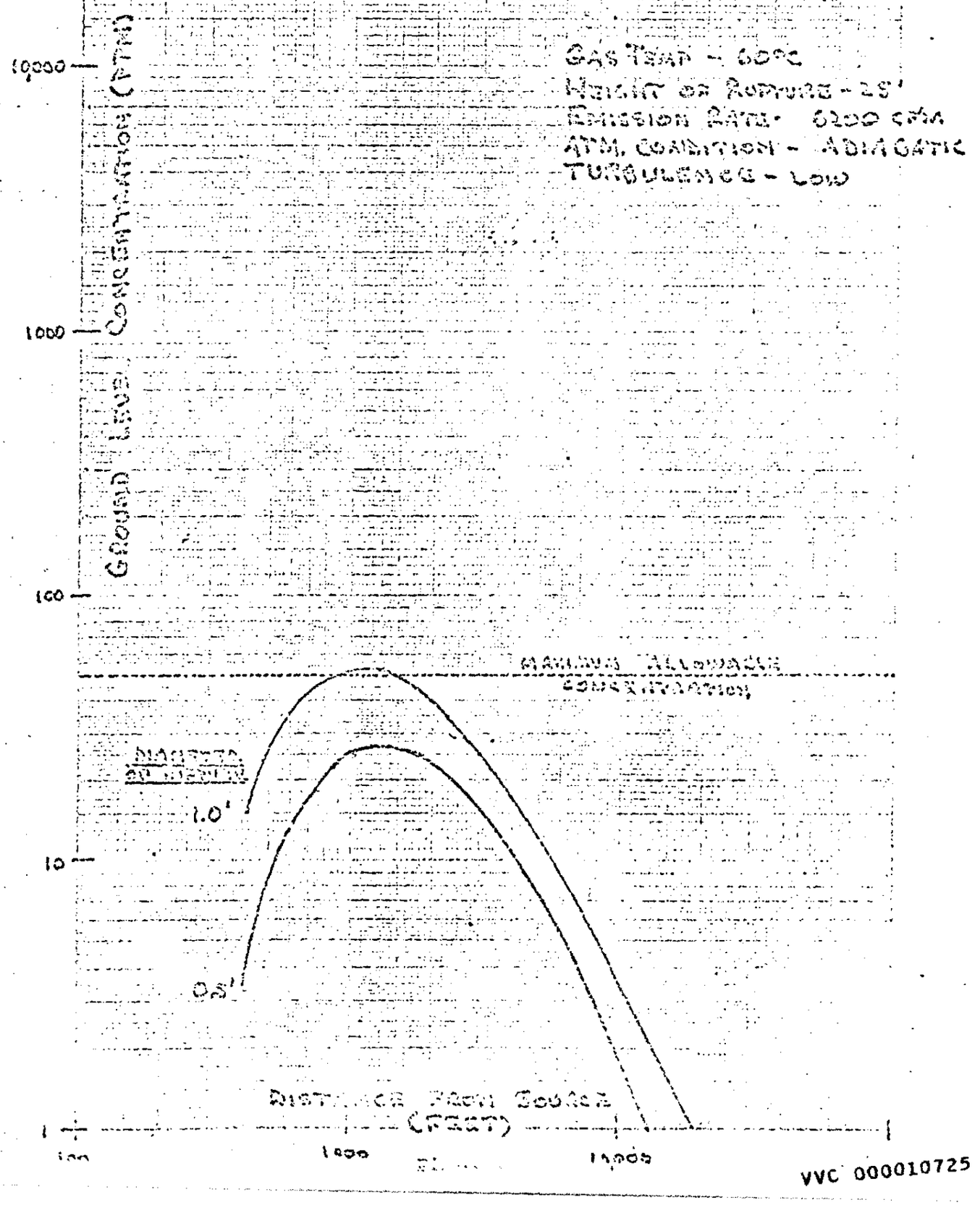
Effect of Turbulence on Ground Level CO Concentration



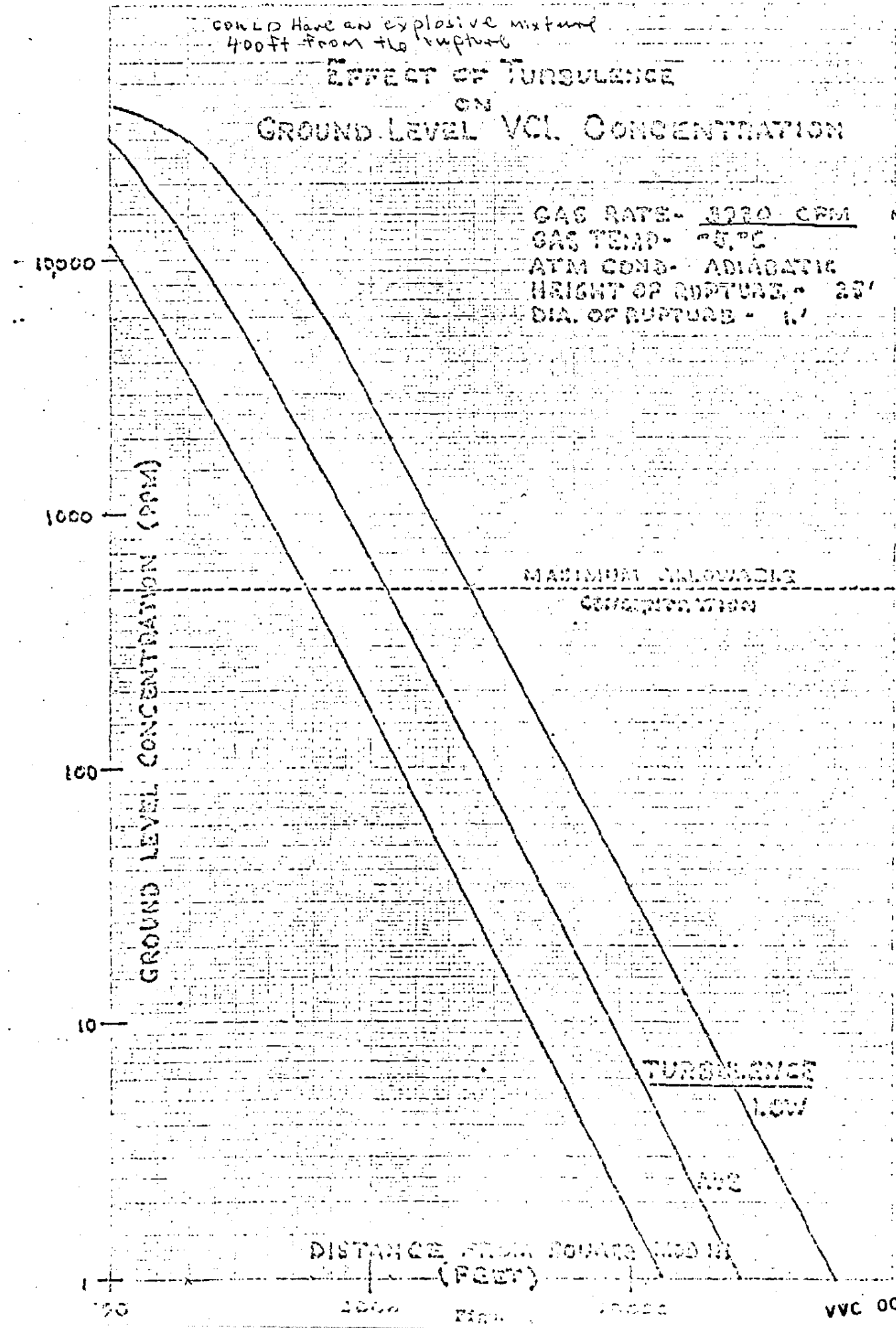
Effect of Emission Rate on Ground Level CO Concentration



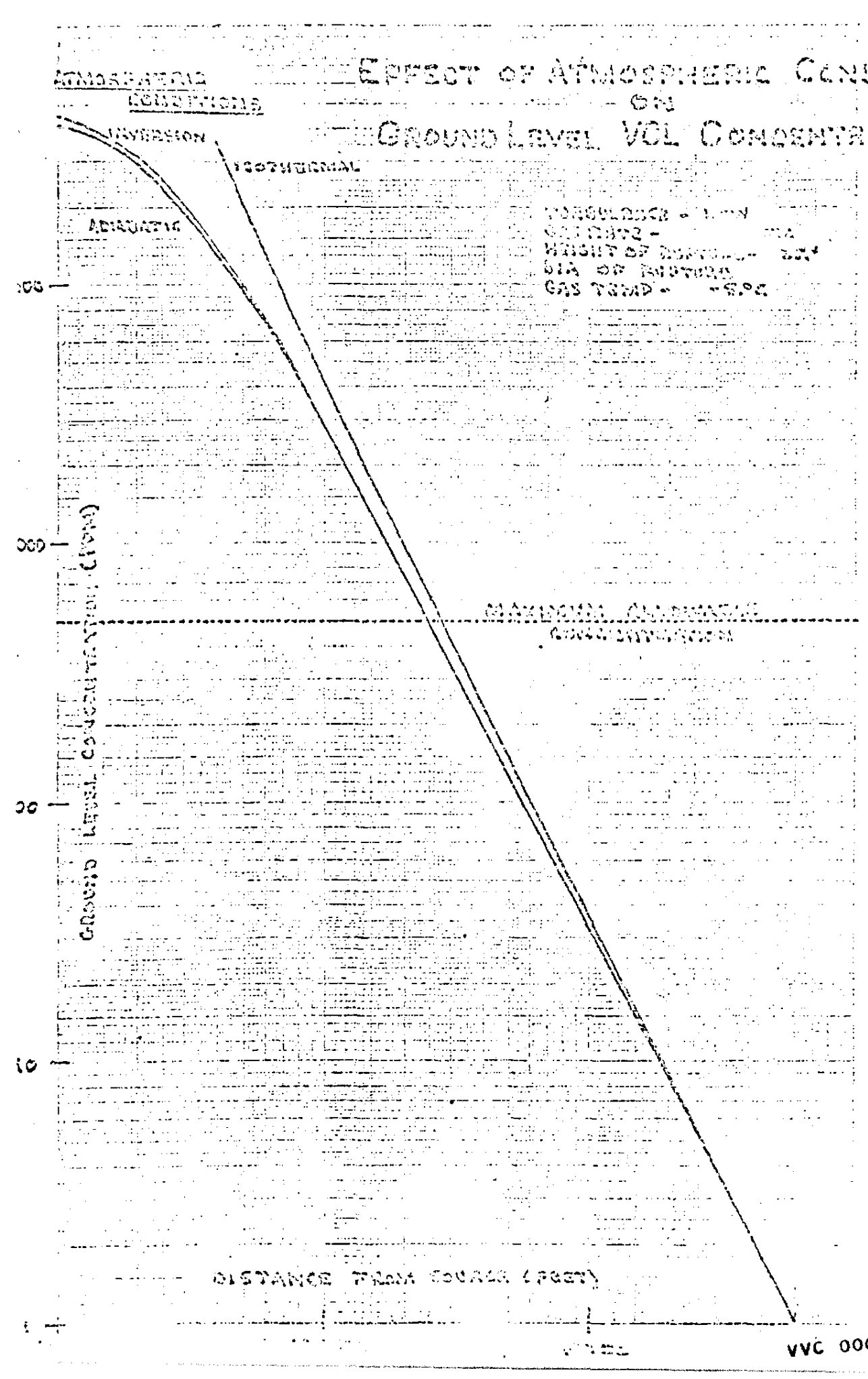
Effect of the Rupture Hole Diameter on Ground Level CO Concentration



could have an explosive mixture 400 ft from the rupture



must look at higher rates of emission.


EFFECT OF ATMOSPHERIC CONDITIONS
ON
GROUND LEVEL VOL CONCENTRATION
ATMOSPHERIC CONDITIONS
INVERSION
ISOTHERMAL
ADIABATIC
MAXIMUM ALLOWABLE CONCENTRATION
GROUND LEVEL CONCENTRATION (PPM)
DISTANCE FROM SOURCE (FEET)
VVC 000010727

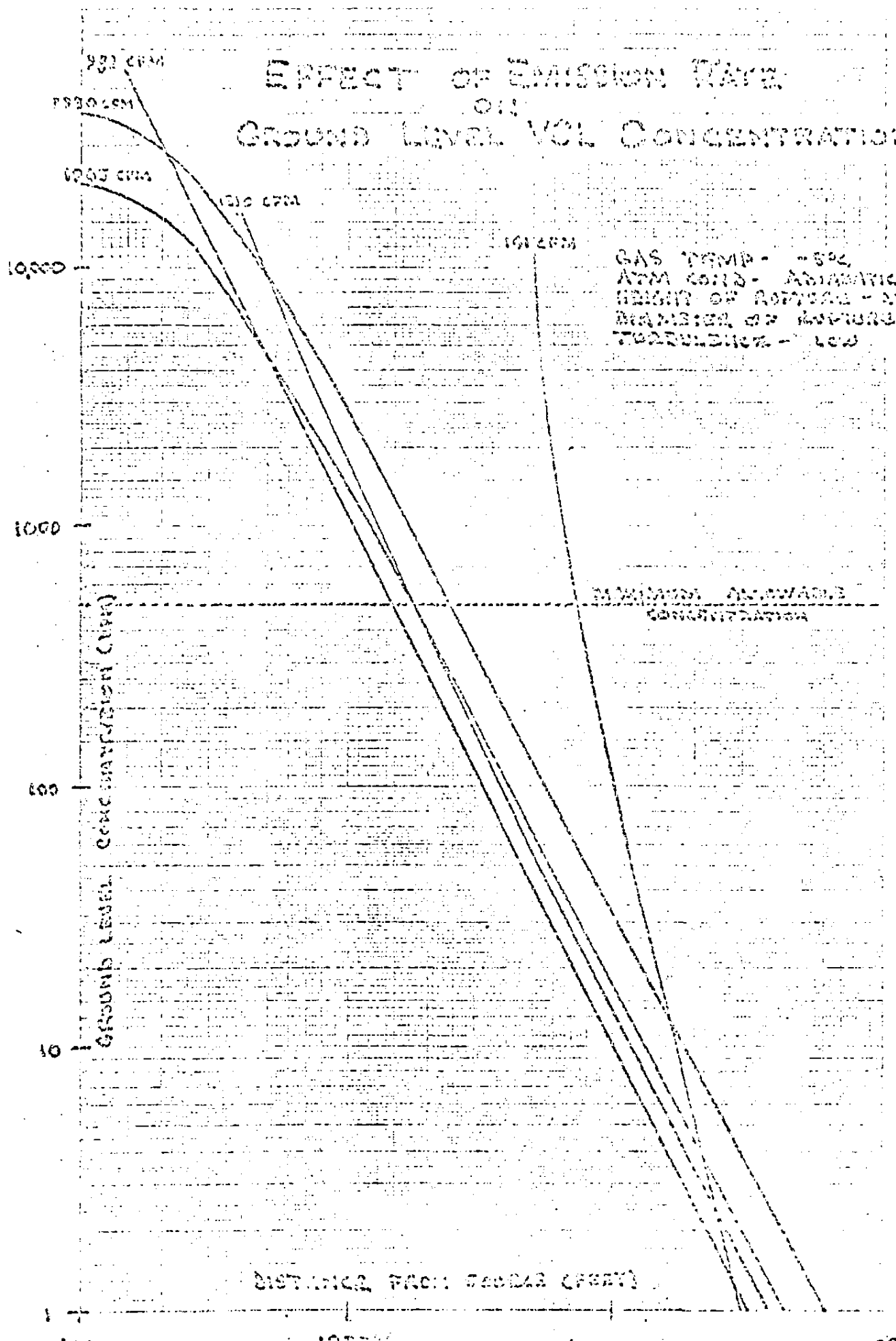
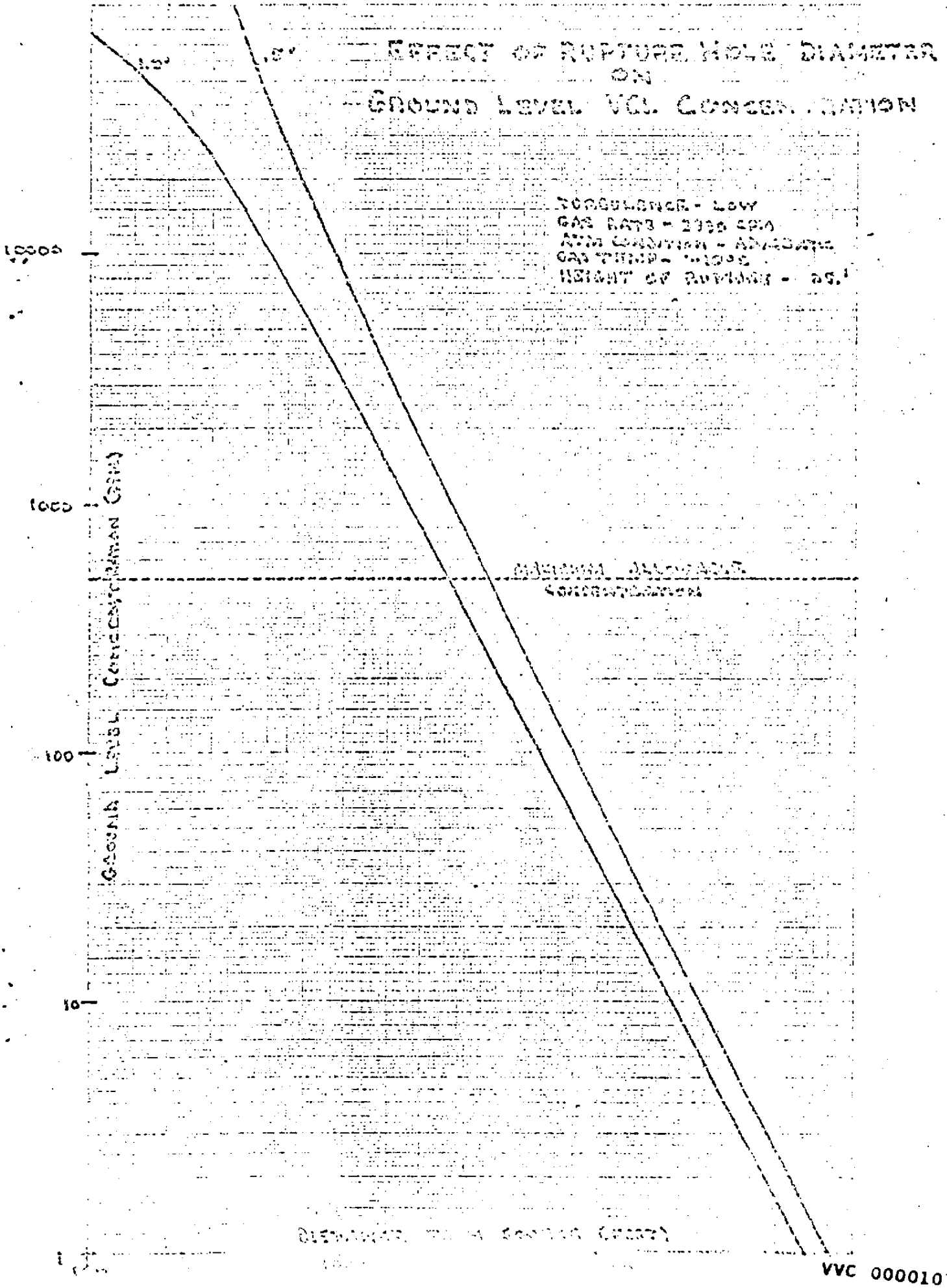


Figure 10.



Effect of Gas Temperature on Ground Level VCL Concentration