

CHLORINE VS. DIOXINS - CONTROL METHODS TO MINIMIZE EMISSIONS

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ABSTRACT

With the enactment of the Boiler and Industrial Furnace (BIF) regulations in August, 1991, hazardous waste combustors were required to sample and demonstrate compliance with Chlorine (Cl_2) emissions. The Resource Conservation and Recovery Act of 1976 required analysis and control of Hydrogen Chloride (HCl) emissions with no requirements for chlorine emissions from combustion systems burning chlorinated organic and organic waste streams. Previous to the BIF regulations, the levels of chlorine emissions from waste combustion systems had not been measured or their impacts on emissions known. Recent studies have demonstrated that the formation of precursors that create the Products of Incomplete Combustion (PICs) such as PCDD/PCDFs is caused by the free Cl_2 , more so than the HCl. Chlorine may be minimized in the combustion process by control of several parameters. These include temperature, excess Oxygen, Hydrogen to Cl_2 ratio, and Sulfur to Cl_2 ratio.

The effect of ash deposition on metallic surfaces such as boiler tubes, heat exchange surfaces, pollution control devices, ductwork and bag filter supports cause cells of activity between the HCl, Cl_2 , iron, and copper which again serve as catalysts for the formation of PCDD/PCDFs. The effect of chlorine concentration in the flue gases, the temperatures of the surfaces allowing the reactions to take place, and the time available for the reaction will be discussed in relationship to the PIC emissions. HCl is easily absorbed in water, whether in a quench tower or spray dryer. Chlorine is much more difficult to neutralize than HCl and requires a basic solution of caustic or lime. Therefore, Cl_2 often persists in those APC systems that are designed for the 99% removal of HCl only.

Emissions of PCDD/PCDFs may be minimized to nondetectable levels with controls applied to combustion zones as well as APC systems knowing the impacts associated with the formation of the precursors and in particular, Chlorine. This paper should allow a better understanding of the conditions which create the problems associated with formation of free chlorine in combustion systems. It should help the designers and operators of these systems understand the potential solutions that can be implemented.

INTRODUCTION

In a 1978 study "Trace Chemistries of Fire", Dow Chemical Company contended that when mythical gods gave man fire, dioxin was thrown into the deal. [Dow Chemical, USA, 1978] Dow theorized that dioxins come primarily from combustion sources, including industrial incinerators, wood burning stoves

and cigarette smoke and are omnipresent in the environment. Dr. Christopher Rappe and a number of other dioxin researchers stated the Dow study was scientifically flawed because it failed to discriminate among the different types of dioxins. [Rappe, Buscr., C. Et al, 1978]

Combustion systems have been found to form dioxins in circumstances of inefficient combustion of chlorinated hydrocarbons. These are the result of insufficient oxygen (reducing atmosphere), poor mixing of the oxygen with the burning fuel containing chlorine, low temperatures ($<800^{\circ}C$) and short residence times. Other mechanisms in the total combustion and APC system which lead to dioxin formation are flame bypassing and carryover, formation from organic precursors, gas phase synthesis in the flame, gas phase synthesis and surface catalyzed formation in the formation temperature window and formation within the bulk of carbonaceous solids. By control of temperature and oxygen, and insuring an adequate ratio of H_2 to Cl_2 , it is possible to find and maintain optimum combustion conditions to minimize formation of free chlorine and unburned hydrocarbons and the resultant PCDD/PCDFs.

Hasselriis has shown results of tests at various types of incinerator applications. Comparisons are made to the combustion zone temperature and CO emission vs. total PCDD/PCDF emissions in ng/m^3 [Hasselriis, 1987]. Even in the most efficient combustion systems, reformation will occur in cooler zones downstream of the combustion zones. A great deal of research has taken place since 1989 to better understand this phenomenon. Critical to the formation is the presence of particulates to provide surface for catalytic reactions (copper chloride and iron chloride provide the most rapid reactions), temperatures of $250^{\circ}C$ to $350^{\circ}C$, precursors such as chlorophenols, and stable compounds of chlorinated hydrocarbons plus residence time.

In this paper, the role of chlorine in the de Novo formation of PCDD/PCDF will be covered. Its release in the corrosion process between ash and boiler/economizer surfaces will be shown.

The effect of typical temperatures of tube surfaces found in the boiler and economizers as well as that on the quenched zones of APC systems in the formation of PCDD/PCDFs will be discussed.

More research is necessary on the effect of cleaning cycles on boiler tube surfaces as well as precipitator plates and fabric filters. Efforts are being made at this time to study these conditions and their effects on formation of PCDD/Fs.

[Fangmark, et al, 1995]; [Milligan, M.S. and Altwicker, E.R., 1995]

COMBUSTION OF CHLORINATED HYDROCARBONS

Since the 1950s, many new products have been developed by the chemical process industry with chlorine as a basic element. These products are in use in our daily lives as pharmaceuticals, solvents, plastics, herbicides, pesticides, etc.. Waste products generated from these processes contain compounds similar to the products manufactured. These wastes are in the form of liquids, tars, sludges, solids and fumes. High temperature incineration has been the most effective method of disposal since it will destroy the compound to 0.01% to 0.0001% of the original input (99.99% to 99.9999% DRE).

In the combustion of waste chlorinated hydrocarbons, hydrochloric acid and chlorine will be present as products of combustion in the exhaust gas. The Resource Conservation and Recovery Act (RCRA) of 1976 required incineration systems to remove 99% of the HCl formed in the system. Chlorine was not a consideration until the Boiler and Industrial Furnace regulation (BIF) of August, 1991 required minimal emissions of chlorine as well as HCl to prevent ground level concentrations to exceed $7 \mu\text{g}/\text{m}^3$ for HCl and $0.4 \mu\text{g}/\text{m}^3$ for Cl_2 . This required development of sampling procedures for Cl_2 by the EPA. These were established and are required in the sampling protocol for BIF compliance tests. States have used omnibus authority for RCRA incinerator trial burns to test for the chlorine as well as metals. These tests include operation at the highest organic chlorine feed with high metal feeds at maximum operating temperatures to determine the removal efficiency of the APC systems for metals and salts.

These tests have shown that chlorine is emitted from combustion systems. Critical to the formation of free chlorine are conditions in the combustion zone. Most important is the combustion zone temperature, excess oxygen, and ratio of Hydrogen to Chlorine. The equilibrium constant, K_p is related to all of these as noted in Figure 1. [Tsuruta, H. 1972] To determine the partial pressure of Cl_2 , the solution is as follows:

$$(\text{P}_{\text{Cl}_2}) = \frac{(\text{P}_{\text{HCl}})^2 (\text{P}_{\text{O}_2})^{1/2}}{K_p (\text{P}_{\text{H}_2\text{O}})} \quad (1)$$

where P is partial pressure, K_p is equilibrium constant.

Note that at low temperatures, K_p is also low. This would result in higher levels of Cl_2 .

This is based on the formation of Cl_2 via the Deacon Reaction as follows:



The Cl_2 partial pressure is plotted as a function of temperature in Figure 2. [Ellis, T.D., et al] The pressure decreases about 4 times from 1490°F (827 °C) to 2030°F (1127 °C). A second equilibrium reaction for monatomic Cl^\cdot can be

written as follows:



Using the JANAF tables, the Gibbs Free Energy of Formation may be found. This allows the calculation of the equilibrium constant at each temperature of concern in the combustion zone.

The Cl^\cdot partial pressure is also plotted as a function of the combustion zone temperature in Figure 2. When the incinerator exhaust gases are rapidly cooled from these temperatures, the total chlorine pressure (as Cl_2) is equal to the sum of $\text{P}_{\text{Cl}_2} + \frac{1}{2} \text{P}_{\text{Cl}^\cdot}$. The combined equivalent pressure is given by the solid line in Figure 2. Note that the condition for formation of minimum Cl_2 occurs at about 2120°F (1177°C). This points out the need to operate incineration systems in the range of 1832°F (1000°C) to 2250°F (1250°C) for a maximum HCl and a minimum Cl_2 level. At lower or higher temperatures, chlorine emission levels will increase and combined with HCl will provide a source for reaction with either copper or iron compounds to form copper chloride and iron chloride which have been found to be catalyst surfaces for the formation of PCDD/PCDFs. Incinerators that have operated at these temperature levels followed by rapid quench to adiabatic temperatures have been found to have the lowest emission levels of PCDD/PCDFs. The advantage of these systems with operation at these temperature levels insures the DRE necessary to destroy the organic compounds, minimize free chlorine and provide little or no time at the critical reformation temperature of 572°F (300°C) for PCDD/PCDFs.

In reviewing data in the recent report published by ASME [ASME - CRTD - Vol. 36 (1995)], Table 1 has been prepared. Note that those units with rapid quench (wet scrubber) are one to two orders of magnitude lower than those with dry quench (spray dryers, baghouses and ESPs). This occurs even at the higher levels of chlorine input. The proper controls of temperature, residence time, excess air and available hydrogen in these incinerators should be very similar. The rapid quenching to low temperatures will absorb HCl rapidly reducing any further formation of Cl_2 . It also minimizes the opportunity for maintaining particulates, chlorine and other PICs at the temperature level of 300° C where the reformation of PCDD/PCDFs would occur.

The reaction of SO_2 and a halogen has also been found to minimize the halogen and formation of the halide. HCl can be easily absorbed in water whereas Cl_2 must be scrubbed using a basic reagent (usually caustic or lime) to be effectively removed. This approach was used by the EPA in reducing the stockpiles of Ethylene Dibromide by incineration. The EDB wastes were burned in the incinerator chambers with sulfur burning wastes. The SO_2 formed as a combustion product reacted with the Br_2 which was the result of EDB combustion. [Ambekar, S.H. and Laseke, B.A., 1989]



Both of the acid gases can be easily removed in caustic scrubbers, eliminating the problem of halogen emissions. A

similar reaction applies to Cl_2 to aid in converting any free Cl_2 formed in the combustion zone to HCl.



In incineration systems where sulfur compounds are burned at the same time as chlorinated hydrocarbons, the resulting exhaust gases will contain less free chlorine gas than that expected through equilibrium reactions. This is another method of reducing the formation of precursors for PCDD/Fs. Firing tests conducted by EPA in their Innovative Furnace Reactor (IFR) Firing Tests conducted with high Cl coal, low sulfur, resulted in HCl concentrations of 163 ppm in the furnace. At this concentration, PCDD/F concentration was negligible. Tests doped with 1308 ppm HCl resulted in increased levels of PCDD/Fs. When levels of S/Cl were increased to 0.8 by addition of SO_2 (tests with coal, MSW ash, HCl and SO_2), the PCDD/F yield dropped dramatically. These laboratory test results demonstrate the ability of sulfur to inhibit PCDD/F formation. [Gullett, B.K., et al, 1992] Full scale demonstration tests are necessary to separate the sulfur effects from the potential of carbonaceous ash forming PCDD/Fs.

RELEASE OF CHLORINE DURING CORROSION PROCESSES

The Deacon reaction described above occurs in flue gases containing free HCl and O_2 to form Cl_2 and H_2O vapor. To understand the theories of HCl corrosion of boiler tubes, laboratory experiments were conducted by passing a mixture of HCl and air in the ratio of 1:1,000 over tube deposits. Tests conducted at BASF in Ludwigshaven, Germany showed that the formation of Cl_2 from HCl takes place starting at 630°F (350°C). [Fassler, K., et al, 1968] The Cl_2 formation mounted sharply to over 1112°F (600°C) stopping precipitously at 1292°F (700°C). At that point the tube deposit serving as a contact had melted. The formation of free chlorine is strongly impeded by water vapor. (See Figure 3). The iron chlorides formed combined with the O_2 present to yield Fe_2O_3 and Cl_2 . Water impeded the Cl_2 formation and SO_2 stopped it almost completely in increasing concentrations. This follows the explanation described above with SO_2 and H_2O converting free Cl_2 to HCl.

Tubes without deposits have shown little or no attack. Since waste heat boilers associated with incinerators will trap ash on the surface of the tubes from ash contained in the waste, the contact of HCl with the deposit and eventually the iron in the tube proper is assured. The attack is strongest if good permeability of the flue gases through the deposit layers is guaranteed. The actual course of the corrosion may be explained as follows [Huch, R., 1966]:

HCl or Cl_2 can diffuse through the deposits of refuse ash to the tube surfaces; there the free hydrogen chloride or the chlorine can react with the surface oxides and metals of the boiler tube with the formation of iron chloride (300 to 400°C). The iron chloride diffuses toward regions of higher temperatures in the tube deposit and is there decomposed to iron oxides and gaseous HCl and Cl_2 (500 - 600°C), which in turn migrate back to the tube surface and react.



This transport property and the reactions described above can explain why very small quantities of chlorine are detected in the deposits. Only small quantities of HCl and Cl_2 are needed to maintain this cyclic process. The result is that HCl will be converted to Cl_2 in this corrosion process providing the conditions necessary for reaction with carbon or other organics to form the precursors for PCDD/Fs. (See Figure 4) [Krause, H.H., 1989]

The buildup on the tube surfaces and the length of time that this buildup remains would have an impact on the ability of these deposits to provide the chlorine addition to the stack gases. The time available for exposure of these deposits to precursors at the temperature levels where de-Novo synthesis reaches peak levels will influence the PCDD/Fs emissions from the system. These temperature conditions (250-350°C) have been found to be the same levels at which the maximum formation of PCDD/PCDFs occurs. One would expect the deNovo synthesis to occur in the later stages of the boiler, economizer and/or air preheater where gas temperatures are too low to cause destruction of PCDD/Fs that may have formed but ash and metal temperatures are closer to 300°C. Almost any combination of C, H, O and Cl can yield some PCDD/Fs under suitable conditions of time and temperature. The quantities formed in incinerators, ng/m³ or ng/g, need to be explained in terms of time/temperature regimes relevant to those units. Current evidence suggests that surface catalyzed reactions at relatively low temperatures (250-400°C) may play an important role. [Altwickler, E.R., et al, 1990]

Plants that have waste heat recovery units have been found to have higher emissions of PCDD/Fs than those with no heat recovery. This phenomenon of releasing Cl_2 into the exhaust gases from the boiler which had initially contained higher levels of HCl may account for these increases. Temperatures of the boiler tubes and the ash deposits on the tube surfaces are also critical to the level of formation. Gullett et al, demonstrated that the amounts of phenol conversion to chlorophenols (a precursor for PCDD/Fs) are at least three orders of magnitude higher with Cl_2 than with HCl. Product formation at gas temperatures of 400°C, near the temperature of maximum formation, is proportional to Cl_2 concentration. (Fig.5) [Gullett, B.K., et al, 1990]

FORMATION CONDITIONS OF PCDD/PCDF DUE TO TIME/TEMPERATURE

The effect of thermal treatment of flyash on the behavior of PCDD/F was studied in the range of 248°F (120°C) to 1112°F (600°C). Annealing of the flyash at 572°F (300°C) resulted in an increase of PCDD/F concentration by a factor of 10 to 15. At 1112°F (600°C), degradation to concentrations below 0.1 ng/g was observed. [Vogg, H., and Stieglitz, L. 1986] [Stieglitz, L. And Vogg, H., 1987]. (See Fig. 6). These conditions will occur downstream of the combustion chamber of incinerators in the cooler zones. These conditions will be found on the surfaces of tubes in boilers, process furnaces, economizers, flue

gas ducts, electrostatic precipitator plates and housings and the surface of fabric filters. Vogg and Steiglitz reviewed data from 25 incinerators. Data related to medium (956 ppb PCDD) to high (2810 ppb) might be explained if they were obtained from incinerators where the flyash had been exposed to temperatures around 572°F (300°C) for a longer time either during sampling or due to a longer residence time in the ESP. [Lustenhow, J.W.A., et al, 1980]

Altwickler et al have reviewed the impact of time on the low temperature (300°C) catalytic formation of PCDD/Fs over flyash in APCs. In determining the relevance of the various mechanisms to incinerator - APC conditions, several parameters, such as the degree of desorption of PCDD/F, extent of reentrainment of collected particles, particle collection efficiency, etc., were studied. [Konduri, R., and Altwickler, E.R., 1994] Three processes which impact on residence time were reviewed. These processes are:

1. Mass transfer of precursor molecules to the particle surface
2. Adsorption of precursor molecules
3. Formation of Product Molecule

The results of their study indicate that levels of 150-300 ng/m³ PCDD/F emissions may be attained from retention times of 15 to 25 minutes on ESPs. These are theoretical analyses but point to the need for more intense research into the area of time/temperature on the rate of formation of PCDD/Fs from precursors on surfaces. More rapid cycling of the cleaning process, whether it be boiler tubes, ESP plates or fabric filters will shorten the exposure time for the reaction to take place.

CONCLUSION

I have described above the potential impact of chlorine as a precursor to the formation of PCDD/Fs in the APC systems. Chlorine may be formed in both the combustion process and the boiler tube corrosion process. This phenomena may have been overlooked in the past as the cause of PCDD/F formation since it has not been the practice to sample for chlorine as an effluent from combustion processes. Hydrogen chloride was selected by U.S. EPA when the RCRA standards were developed as an emission to be controlled. We have since found that the level of HCl in combustion products or stack gases does not correlate to PCDD/F emissions. [ASME, 1995]

The BIF regulations (1991) were the first to require measurement of Cl₂ as part of the compliance tests. Little data is available on the levels of Cl₂ from MSWs or HWIs, since it has not been required as part of the test burn sampling protocol. As more baseline data is developed, we may find a better correlation between Cl₂ entering APC and PCDD/Fs emitted. The ability of SO₂ to reduce Cl₂ to its halide HCl and minimize PCDD/F emissions should be investigated further for all combustion devices burning fuels or wastes containing chlorinated organics.

As described above, more research is needed to determine the relationship between dust collection and the cycle of cleaning on surfaces and PCDD/F emissions. As noted in Table

I and more recent baseline data collected from incinerator tests, units that cool combustion gases from the high temperature combustion zones to temperatures below 212°F (100°C) by rapid quench have the lowest levels of PCDD/F emissions.

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TABLE I

PCDD/PCDF DATA IN TOTAL NG/DSCM FOR COMMERCIAL HW INCINERATOR POPULATION

Company	Location	Report Date	Facility Type	APCS	% Cl Feed	HCl PPM	Cl ₂ PPM	# Points	PCDD/PCDF (ng/dscm @ 7% O ₂) MIN To	PCDD/PCDF (ng/dscm @ 7% O ₂) MAX	ITEQ (ng/dscm @ 7% O ₂) MIN To	ITEQ (ng/dscm @ 7% O ₂) MAX
Aptus, Inc.	Amagonite, UT	March-92	RK	FF/WS/WS	9-20	0.7-1.5	0.3-0.8	9	246.8	915.0	6.83	27.10
Aptus, Inc.	Coffeyville, KS	May-90	RK	FF/WS/WS	12.7-20.9	0.2-91		12	35	206	0.75	4.41
General Electric	Pittsfield, MA	Oct-90	LJ	PS	26-32			5	99	1229	3.8	76.5
Rollins Environmental Services *	Baton Rouge, LA	Apr-87	RK	WS	15-16.4	1.6-1.8		3	1.3	4.4	0.02	0.18
Rollins Environmental Services *	Bridgport, NJ	Dec-86	RK	WS				6	0.3	6.9	0.01	0.15
Rollins Environmental Services *	Deerpark, TX	May-88	RK	VS	18-33.5			5	2.2	3.2	0.1	0.77
Ross Incineration Services *	Grafton, OH	Mar-93	RK	PS/WS	1.1			3	1.0	1.8	0.12	0.13
Waste Tech Industries **	East Liverpool, OH	Jul-93	RK	ESP	10.3-28.3			18	3.3	23.7	0.02	0.45
	# of Facilities											
	# of Units											
	# of Data Sets											
	# of Data Points											

of Facilities
of Units
of Data Sets
of Data Points

Facility Type
RK - Rotary Kiln
LJ - Liquid Incinerator

APCS Type
FF - Fabric Filter
WS - Wet Scrubber
IWS - Ionizing Wet Scrubber
VS - Venturi Scrubber
PS - Scrubber Proprietary
ESP - Electrostatic Precipitator

* Rapid Quench Systems.
** These data do not reflect the results of tests conducted in February 1994. The preliminary draft report on these recent tests indicates that total dioxin/furan emissions were 5 ng/dscm (with a TEQ of 0.065 ng/dscm).

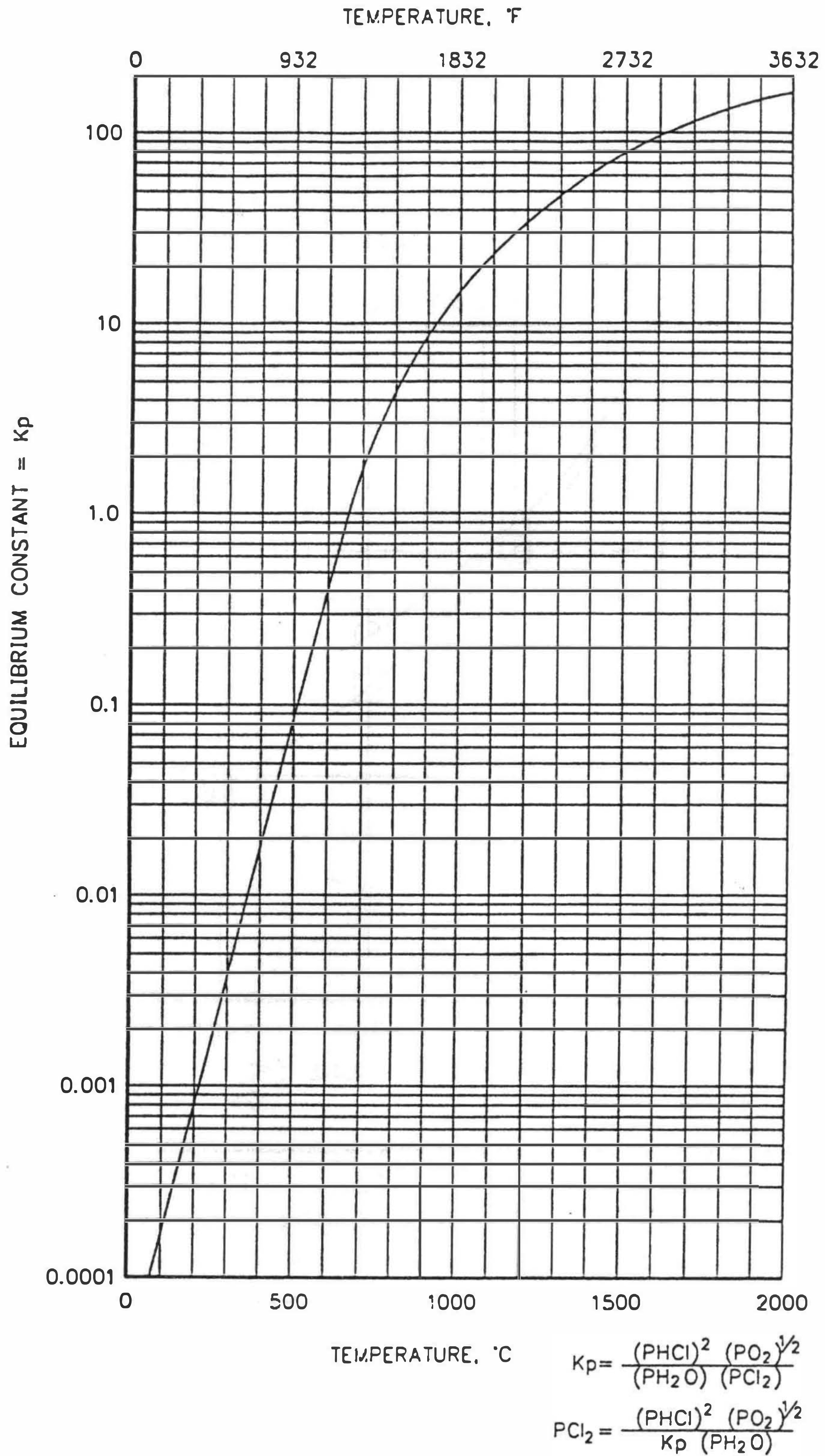


FIG. 1
EQUILIBRIUM CONSTANT VS. TEMPERATURE

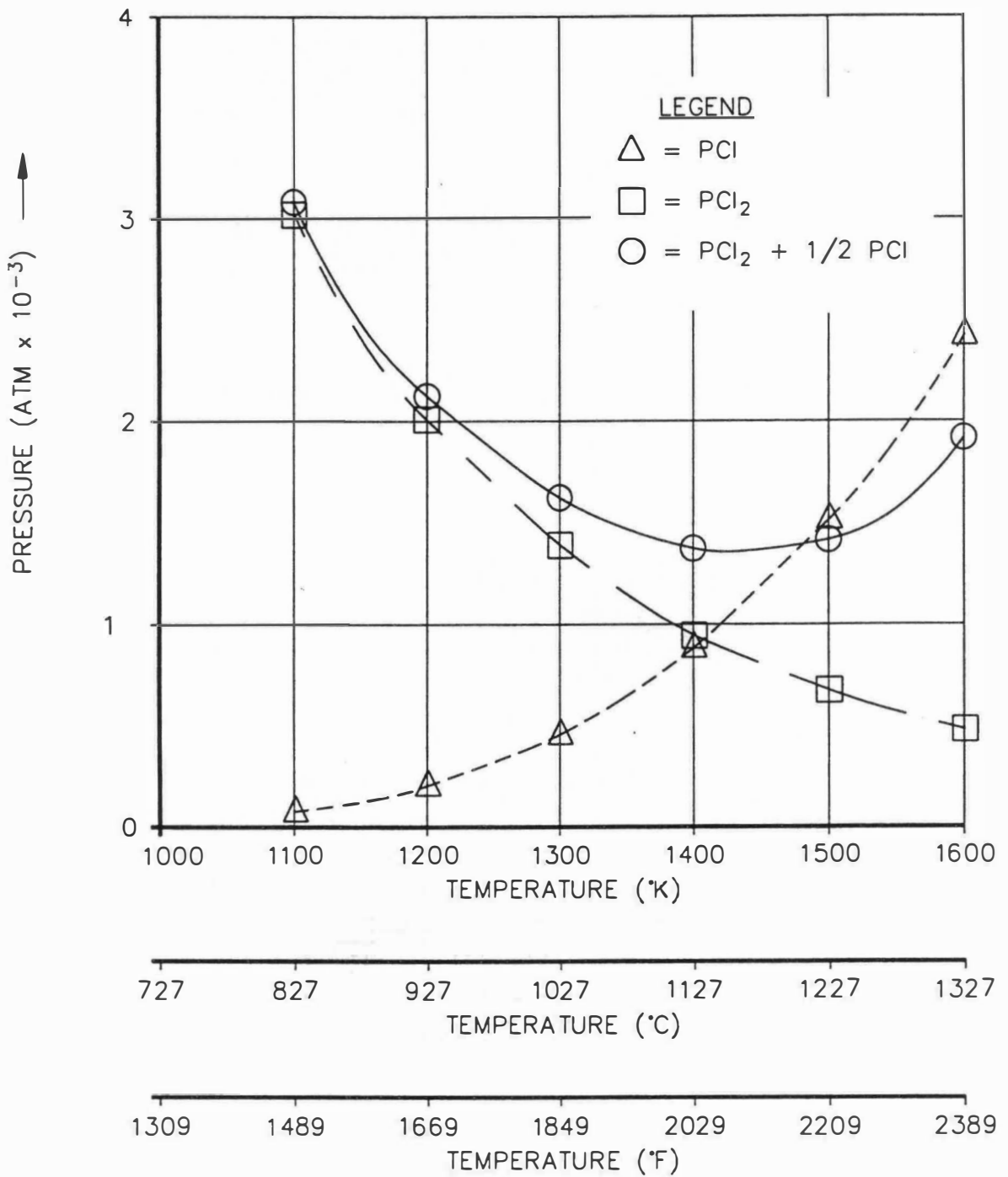


FIG. 2
PARTIAL PRESSURES AS A FUNCTION OF TEMPERATURE

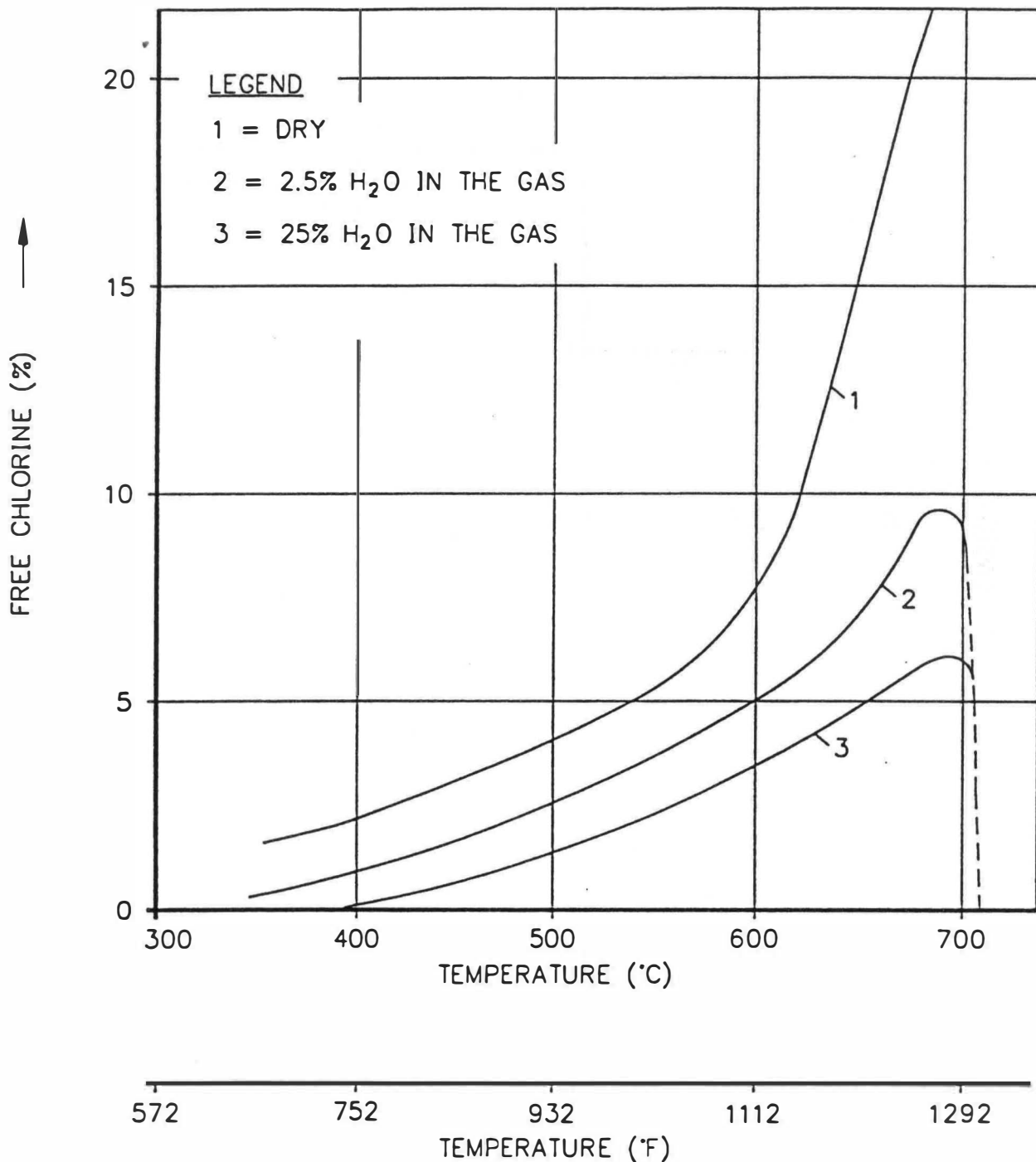


FIG. 3
 PRODUCTION OF FREE CHLORINE FROM HYDROGEN CHLORIDE
 ACCORDING TO THE DEACON PROCESS (REFERRED TO TOTAL CHLORINE).
 [FASSLER, 1968]

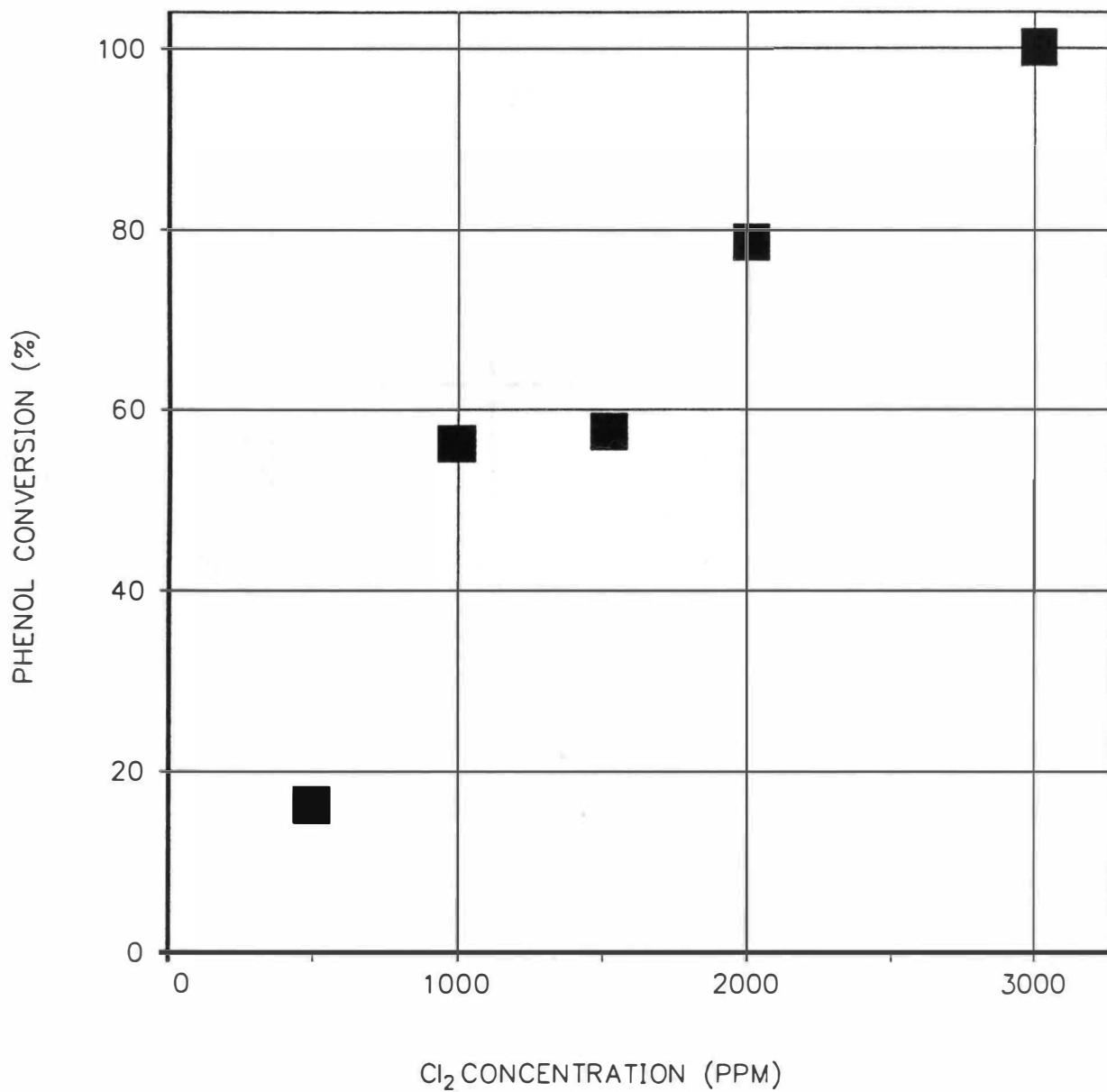


FIG. 4
CL₂ CONCENTRATION DEPENDENCY OF PHENOL
CONVERSION AT 400°C

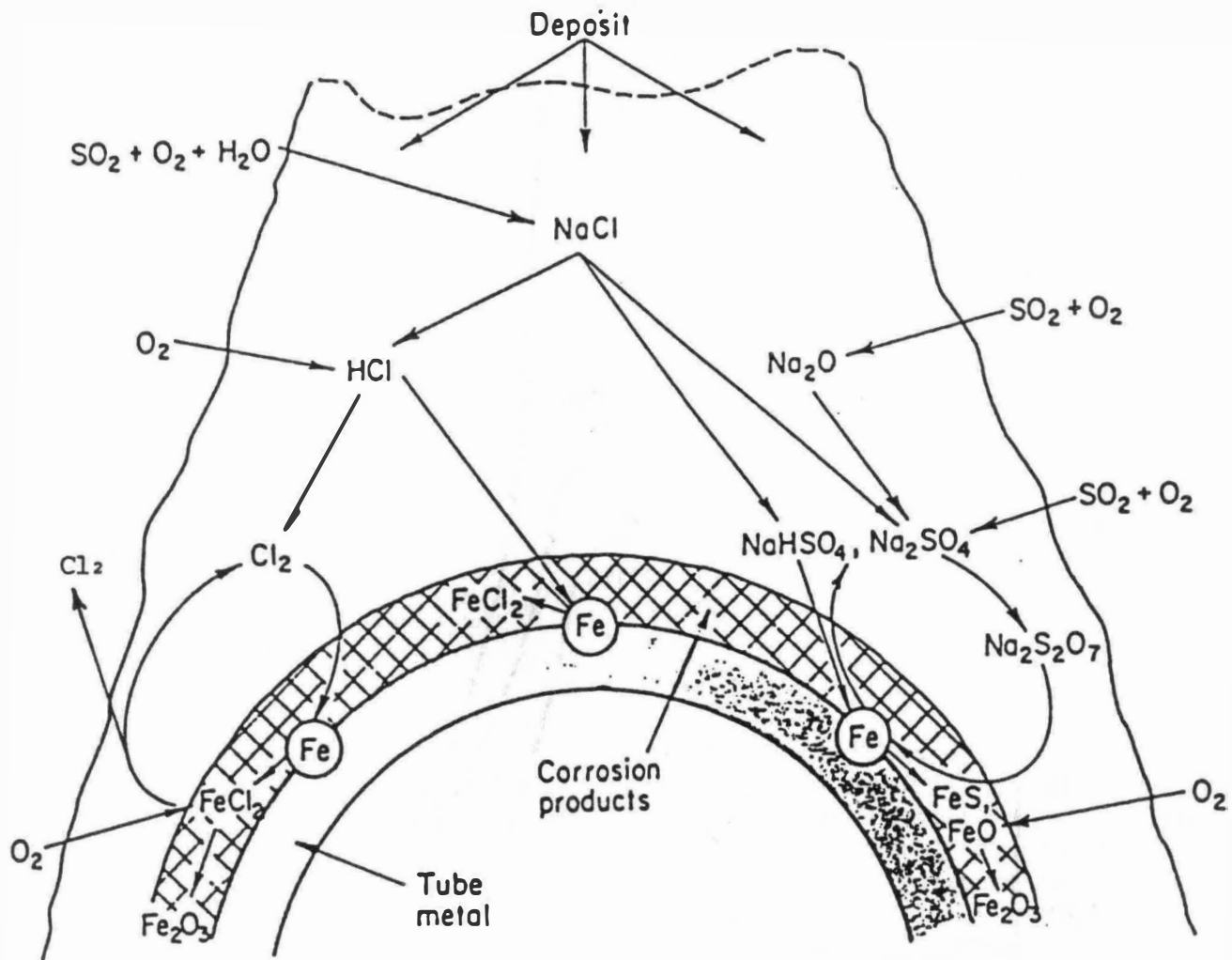


FIGURE 5
Corrosion Chemical Reactions on Incinerator Boiler Tube
[Krause, 1989]

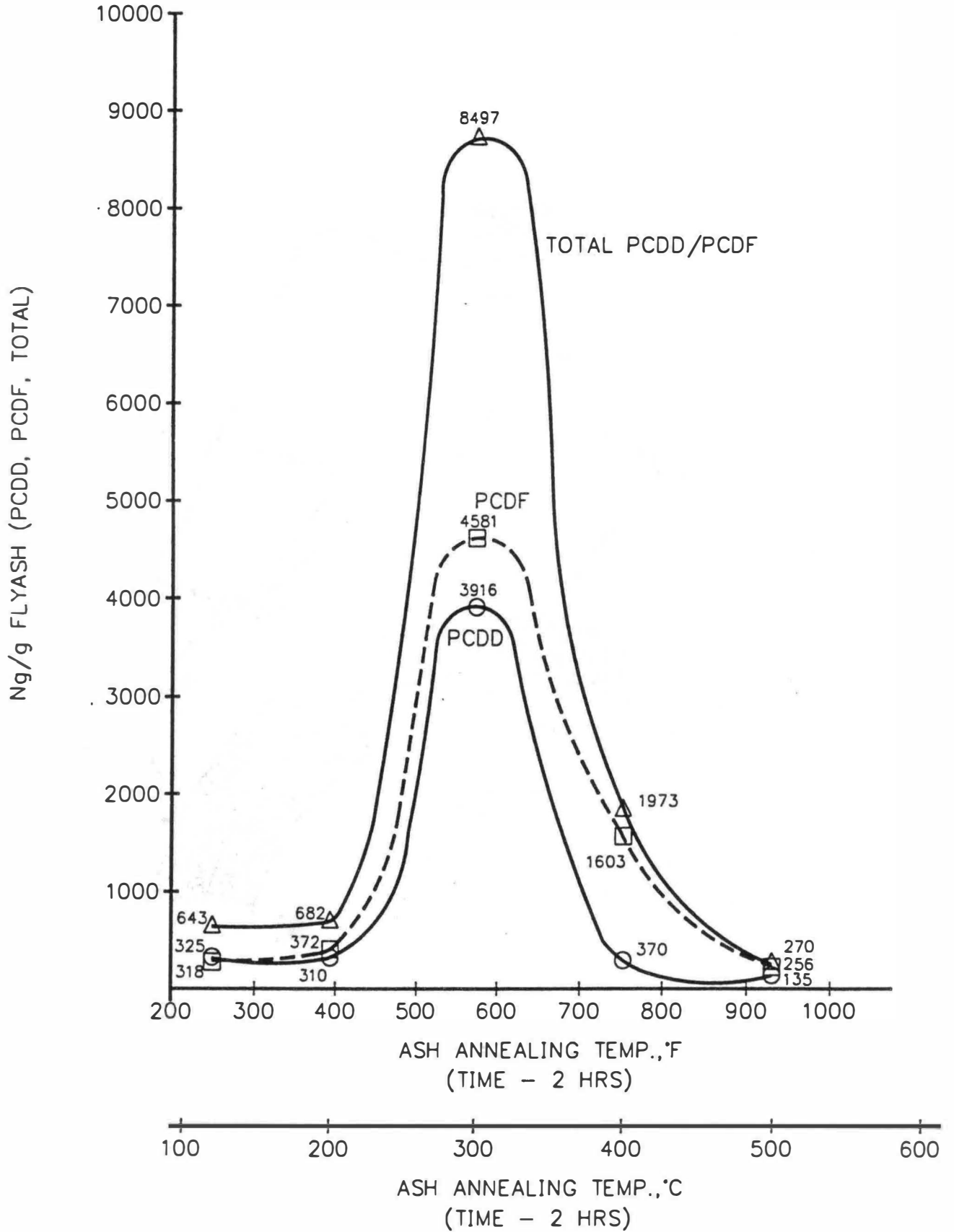


FIG. 6
 THERMAL BEHAVIOR OF DIOXINS/FURANS
 IN FLY ASH
 [VOGG, STIEGLITZ, 1986]