Environment Canada
Environmental Protection Service

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ENVIRO
Technical
Information for
Problem
Spills

FERRIC CHLORIDE

August 1984

Canada

ENVIRONMENTAL AND TECHNICAL INFORMATION FOR PROBLEM SPILLS MANUALS

Environmental and Technical Information for Problem Spills (EnviroTIPS) manuals provide detailed information on chemical substances. This information is intended to assist the reader in designing countermeasures for spills and to assess their impact on the environment. The manual has been reviewed by the Technical Services Branch, Environmental Protection Service, and approved for publication. Approval does not necessarily signify that the contents reflect the views and policies of the Environmental Protection Service. Mention of trade names or commercial products does not constitute endorsement for use.

FERRIC CHLORIDE

ENVIRONMENTAL AND TECHNICAL INFORMATION FOR PROBLEM SPILLS

Technical Services Branch
Environmental Protection Programs Directorate
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FOREWORD

The Environmental and Technical Information for Problem Spills (Enviro TIPS) manuals were initiated in 1981 to provide comprehensive information on chemicals that are spilled frequently in Canada. The manuals are intended to be used by spill specialists for designing countermeasures for spills and to assess their effects on the environment. The major focus of EnviroTIPS manuals is environmental. The manuals are not intended to be used by first-response personnel because of the length and technical content; a number of manuals intended for first-response use are available. The information presented in this manual was largely obtained from literature review. Efforts were made, both in compilation and in review, to ensure that the information is as correct as possible. Publication of these data does not signify that they are recommended by the Government of Canada, nor by any other group.

ACKNOWLEDGEMENTS

The final version of this manual was prepared by the staff of the Environmental Protection Service who wrote extensive revisions to the text, drafted illustrations and incorporated all comments and additions.

The level of detail present was made possible by the many individuals, organizations and associations who provided technical data and comments throughout the compilation and subsequent review. The draft of this manual was prepared under contract to Environment Canada by M.M. Dillon Consulting Engineers and Planners, Concord Scientific Corporation, and Waterloo Engineering Limited.

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1 SUMMARY

FERRIC CHLORIDE ($FeCl_3$)

Brown to black solid, or yellow-brown to reddish brown solution with a slightly acidic odour

SYNONYMS

Ferric Chloride, Solid, Solution, Anhydrous, Hexahydrate; Iron (III) Chloride; Iron Sesquichloride; Iron Trichloride; Chlorure Ferrique (Fr.); Perchlorure de Fer (Fr.)

IDENTIFICATION NUMBERS

UN No. 1773 (solid), 2582 (solution); CAS No. 7705-08-0; OHM-TADS No. 7217242; STCC No. 4944138 (solid), 4932343 (solution)

COMMON GRADES & CONCENTRATIONS

Solid: 96 to 99 percent anhydrous ferric chloride

Solution: 29 to 46 percent aqueous solution

Hexahydrate: equivalent $FeCl_3$, 60 percent

IMMEDIATE CONCERNS

Fire: Not combustible

Human Health: Irritating on contact. Corrosive

Environment: Harmful to some species of aquatic life at concentrations as low as 5 mg/L

PHYSICAL PROPERTY DATA

State: (15°C, 1 atm): solid
Boiling Point: decomposes at 315-319°C
Melting Point: 306°C
Flammability: not combustible
Specific Gravity (water = 1): 2.9 (anhydrous) (25°C/4°C); 1.4 (42°Bé solution)

Solubility (in water): 74.4 g/100 mL (0°C)
Behaviour (in water): sinks and mixes; solid reacts exothermically, producing corrosive HCl fumes

ENVIRONMENTAL CONCERNS

May persist in the environment indefinitely, usually in an insoluble form. Does not biodegrade. Potential exists for accumulation of iron in the aquatic environment for marine and freshwater plants, invertebrates, fish.

HUMAN HEALTH

TLV®: 1 mg/m^3 (as Fe)
IDLH: not established

Exposure Effects

Inhalation: When material is misted or sprayed in air, irritation of upper respiratory tract may occur. Dust is an irritant

Contact: Prolonged contact with solution will cause skin staining and may cause dermatitis similar to that caused by weak acids. Irritating to eyes

IMMEDIATE ACTION

Spill Control

Restrict access to spill site. Issue warning: "CORROSIVE". Notify manufacturer and appropriate environmental authorities. Stop the flow and contain spill, if safe to do so. Avoid contact with solution and solid. Keep contaminated water from entering sewers or watercourses.

Fire Control

Not combustible. Use water spray or fog, foam, dry chemical or carbon dioxide.

COUNTERMEASURES

Emergency Control Procedures in/on

Soil: Construct barriers to contain spill. Remove material by manual or mechanical means. Absorb small amounts of spilled liquid with sand or other absorption materials. Shovel solid into covered containers

Water: Contain by damming or water diversion. Precipitate by treating with sodium bicarbonate. Filter or allow solids to settle. Dredge or vacuum pump to remove contaminated bottom sediments

2 PHYSICAL AND CHEMICAL DATA

Physical State Properties

Appearance	Brown-black solid (CRC 1980), or an orange-brown aqueous solution[1] (CHRIS 1978)
Usual shipping state(s)	Liquid (aqueous solution) or solid (anhydrous or hexahydrate)
Physical state at 15°C, 1 atm	Solid
Melting point	306°C (CRC 1982)
Boiling point	Decomposes at 315-319°C (Hawley 1977; Merck 1983; CRC 1982)
Decomposition temperature	315-319°C

Densities

Specific gravity	Solid: 2.898 (25°/4°C) (CRC 1982) Solution: 1.408 (15.5°C) (42°Bé, 39 percent solution) (CRC 1980)

Fire Properties

Flammability	Noncombustible (CHRIS 1978)
Heat of decomposition	43.76 kJ/mole (332°C) (JANAF 1971)
Decomposition products	Ferrous chloride and chlorine (Merck 1976; Bailar 1973; Kirk-Othmer 1981)

Other Properties

Molecular weight of pure substance	162.21 (CRC 1982)
Constituent components of typical commercial grade	Solid: 96-99 percent $FeCl_3$ Solution: 29-46 percent $FeCl_3$ Hexahydrate: equivalent 60 percent $FeCl_3$, 60 percent equivalent H_2O (Ullmann 1975)
Viscosity	9.655 mPa•s (40 percent aqueous solution 20°C) (CRC 1982)

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1 Solutions containing ferric ion are characteristically yellow-brown due to the presence of $FeOH^{++}$. Ferric ion (Fe^{3+}) is colourless or pale purple but undergoes hydrolysis in aqueous solution: $Fe^{+3} + H_2O \rightleftharpoons FeOH^{++} + H^+$. A red-brown solution indicates the presence of $Fe(OH_3)$ or more correctly $Fe_2O_3 \cdot xH_2O$. Addition of nitric acid (HNO_3) to a ferric solution will make the colour disappear, whereas addition of HCl will form a yellow solution ($FeCl^{++}$ is yellow) (Cotton 1972; Sienko 1961; Hogness 1954). Thus the range of possibilities for a solution colour may be colourless to pale purple to yellow or yellow-brown to reddish-brown.

Hygroscopicity	Very hygroscopic (Merck 1976)
Latent heat of fusion	43.1 kJ/mole (at melting point) (JANAF 1971)
Heat of formation	-362.81 kJ/mole (25°C) (JANAF 1971)
Heat of solution	-132.7 kJ/mole (18°C) (Perry 1973)
Heat capacity	
constant pressure (Cp)	134 J/(mole∘°C) (25°C) (JANAF 1971)
	96 J/(mole∘°C) (25°C) (CRC 1982)
pH of aqueous solution	5 percent solution of hexahydrate has a pH of 1.7 (Kirk-Othmer 1981)

Solubility

In water	Anhydrous: 74.4 g/100 mL (0°C), 535.7 g/100 mL (100°C) (CRC 1982) Hexahydrate: 91.9 g/mL (20°C) (CRC 1982)
In other common materials	Anhydrous: in Acetone, 63 g/100 mL (18°C) (CRC 1980); soluble in ethanol, methanol, diethyl ether (CRC 1982); nitriles and amines (Bailar 1973); glycerol (Hawley 1971); and ketones (Ullmann 1975) Hexahydrate: soluble in ethanol and diethyl ether (CRC 1982)

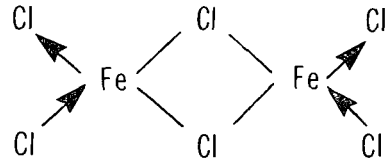
Summary of Ferric Chloride Chemistry

Ferric chloride occurs as hygroscopic, hexagonal, dark (brown-black) crystals. It can be prepared from the reaction of iron with gaseous chlorine at 350°C, from the reaction of ferric oxide (Fe_2O_3) with hydrogen chloride at up to 1000°C, or by other methods such as heating ferric oxide and carbon tetrachloride (CCl_4) under pressure. The thermal stability of the ferric halides decreases from the fluoride to the iodide: ferric fluoride (FeF_3) sublimes at a temperature greater than 1000°C, while the chloride sublimes above 300°C with noticeable decomposition to ferrous chloride ($FeCl_2$) and chlorine:

$$2FeCl_3 \rightleftarrows 2FeCl_2 + Cl_2$$

An excess of chlorine must be present to deter decomposition during the purification (sublimation) process. In the vapour phase (at 400°C), ferric chloride exists as a dimer; at 750°C, it exists as a monomer. In the solid phase, the crystalline material has a semicovalent layer structure with hexagonal packing of chloride ions. Each ion atom is surrounded octahedrally by six chlorine atoms. Ferric chloride is the most commonly encountered of the iron halides; the hexahydrate ($FeCl_3 \cdot 6H_2O$), a yellow, lumpy material,

Fe_2Cl_6



is the next most common. The ferric ion is a relatively strong oxidizing agent, and can also be used as a chlorinating agent and in the manufacture of ink, pigments and dyes. It is also extensively used in the treatment of sewage and industrial waste gases (Bailar 1973; Cotton 1972; Kirk-Othmer 1981).

Ferric chloride is soluble in water, methanol, ethanol, acetone and generally in ethers, ketones, nitriles and amines. It is relatively insoluble in nonpolar solvents such as benzene. It is also insoluble in ethyl acetate. Complexes are formed in donor solvents. The chloride is freely soluble in water; many hydrates (e.g., $FeCl_3 \cdot nH_2O$, where n = 10, 6, 3.5, 2.5 and 2) have been identified. The anhydrous chloride undergoes hydration in moist air, forming the yellow-brown hexahydrate ($FeCl_3 \cdot 6H_2O$ - the actual structure is trans-$(FeCl_2(H_2O)_4)Cl \cdot 2H_2O$). The hexahydrate can also be crystallized from acidified aqueous solution. The solid material is soluble in water, ethanol, diethyl ether and acetone, generally with the formation of solvates, e.g., $FeCl_3 \cdot 2C_2H_5OH$. Hydrolysis and hydroxylation occur in aqueous solution to such an extent that the pH of a 5 percent solution is 1.7. Upon standing, gelatinous hydroxy compounds begin to precipitate. Drying of the compound at selected temperatures produces a series of lesser hydrated species (Bailar 1973; Kirk-Othmer 1981).

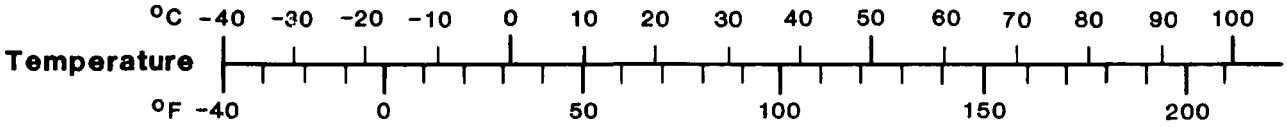
Aqueous solutions of ferric chloride are strongly acidic. They dissolve electropositive metals, such as magnesium, with evolution of hydrogen and liberate carbon dioxide from alkali metal carbonates. In both cases, hydrous iron (III) oxide (FeO(OH)) is precipitated. The hydrolysis of the hexaquoferric ion in dilute solutions can be represented by the following equations:

1. $(Fe(H_2O)_6)^{+3} + H_2O \qquad (Fe(H_2O)_5(OH))^{+2} + H_3O^+$ (yellow)
2. $(Fe(H_2O)_5(OH))^{+2} + H_2O \qquad (Fe(H_2O)_4(OH)_2)^+ + H_3O^+$
3. $(Fe(H_2O)_4(OH)_2)^+ + H_2O \qquad (Fe(OH)_3(H_2O)_3) + H_3O^+$
4. $(Fe(OH)_3(H_2O)_3) \qquad FeO(OH) + 4H_2O\downarrow$

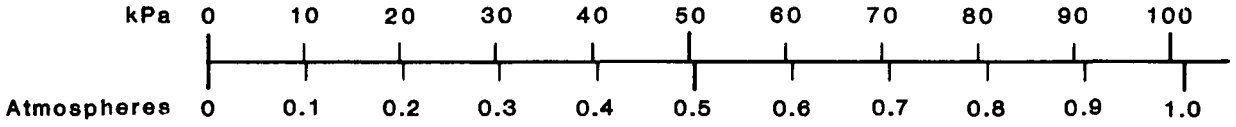
The extent of hydrolysis is very great even at a pH of 2 to 3. In order to have a solution containing primarily the hexaquo ferric ion ($Fe(H_2O_6)^{+3}$), the pH must be around 0. The

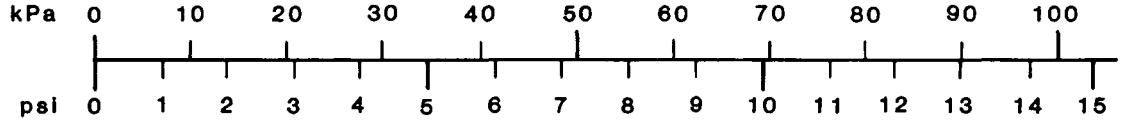
latter ion is pale purple in colour. The various hydroxo species (equations 1 and 2) are yellow; thus, aqueous solutions of ferric salts even with noncomplexing anions are yellow unless strongly acid. As the pH is increased above the 2-3 range, equations 2 and 3 predominate; ultimately, hydrous ferric oxide is precipitated as a red-brown gelatinous mass. In more concentrated solutions of ferric chloride, chloride ions enter the coordination sphere of the iron, and octahedral species such as $(FeCl_4(H_2O)_2)^-$ predominate. If hydrochloric acid is added to a concentrated solution, a polymeric species is formed which consists of alternating $FeCl_4$ (tetrahedral) and $FeCl_4(H_2O)_2$ (octahedral) units with adjacent units sharing a chloride ion. On the other hand, dilute solutions (0.68 M) of ferric chloride in concentrated hydrochloric acid contain primarily the tetrahedral tetrachloroferrate (III) anion ($FeCl_4^-$). Other chloro complexes that occur in crystalline compounds are $FeCl_6^{-3}$ and $Fe_2Cl_9^{-3}$ (Bailar 1973; Cotton 1972).

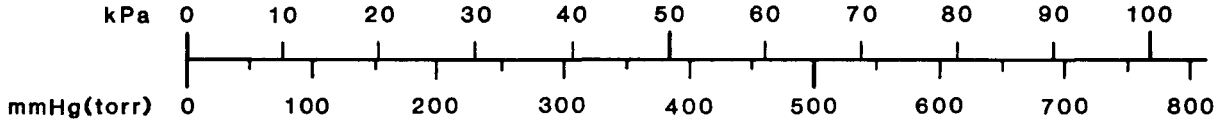
CONVERSION NOMOGRAMS



Pressure 1 kPa = 1 000 Pa







Viscosity

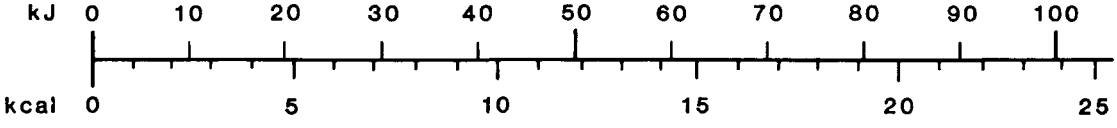
Dynamic 1 Pa·s = 1 000 centipoise (cP)

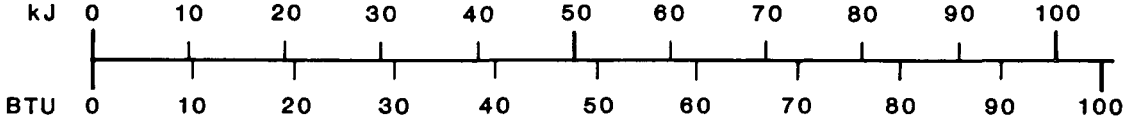
Kinematic 1 m^2/s = 1 000 000 centistokes (cSt)

Concentration (in water)

1 ppm ≅ 1 mg/L

Energy (heat) 1 kJ = 1 000 J





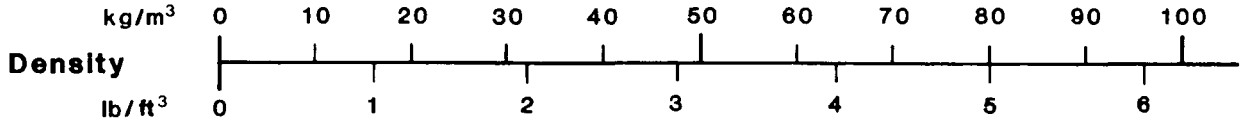


FIGURE 1

FERRIC CHLORIDE

PHASE DIAGRAM OF THE $FeCl_3 \cdot H_2O$ SYSTEM

Reference: LINKE 1958

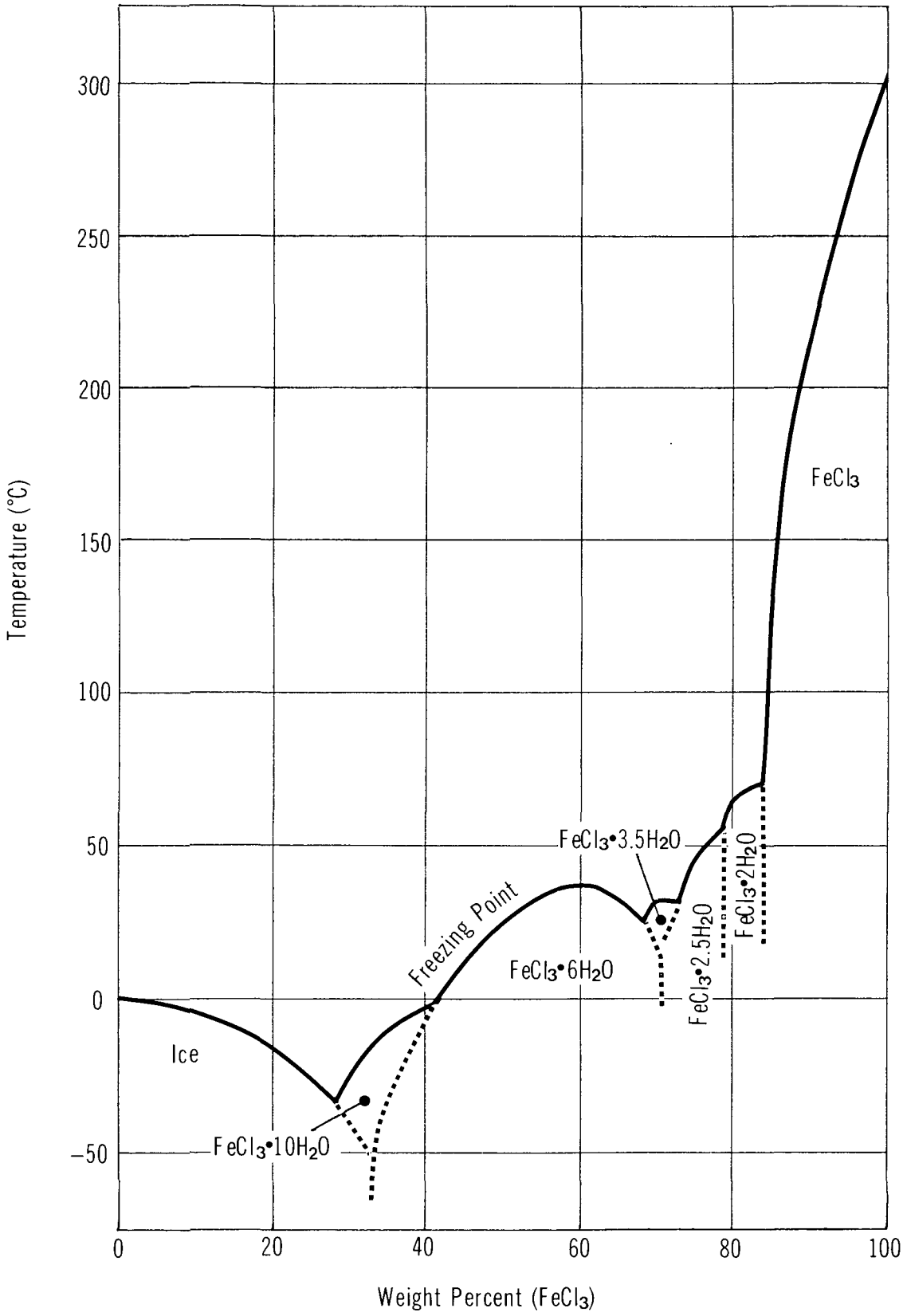


FIGURE 2

FERRIC CHLORIDE (HEXAHYDRATE)

DENSITY OF SOLUTIONS

Reference: CRC 1982

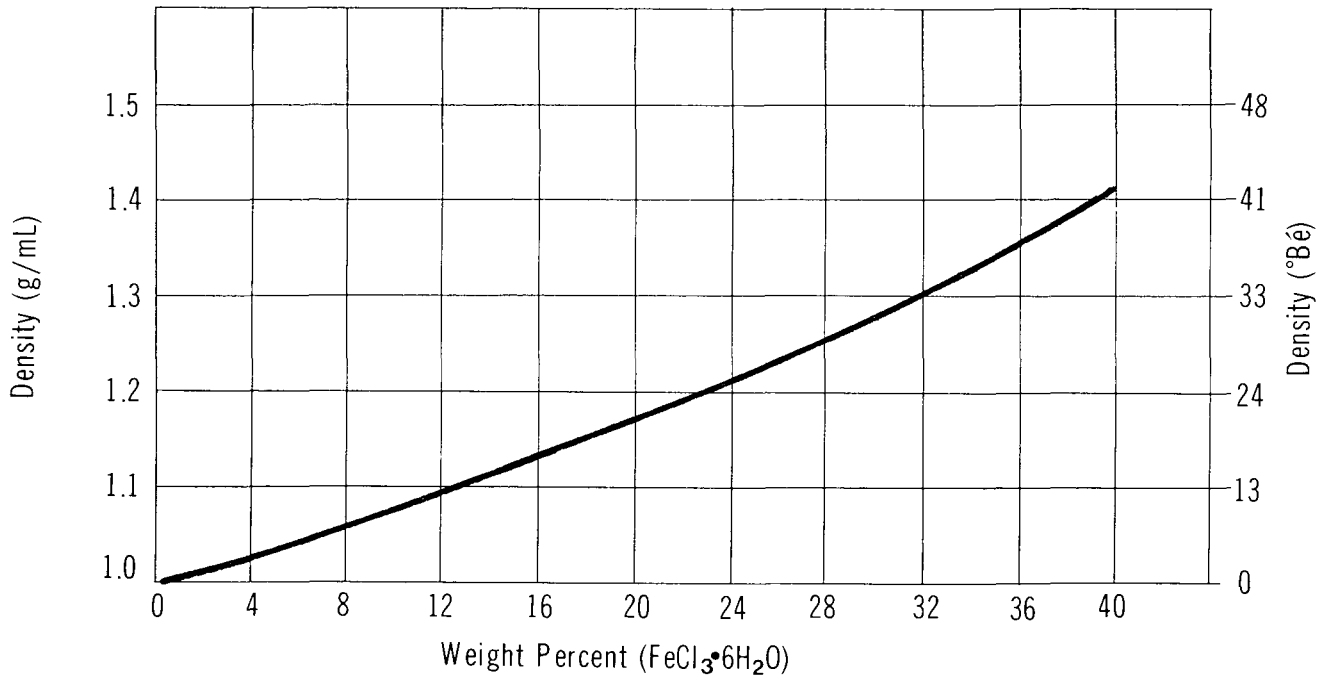


FIGURE 3

FERRIC CHLORIDE

VAPOUR PRESSURE VS TEMPERATURE

Reference: CRC 1982

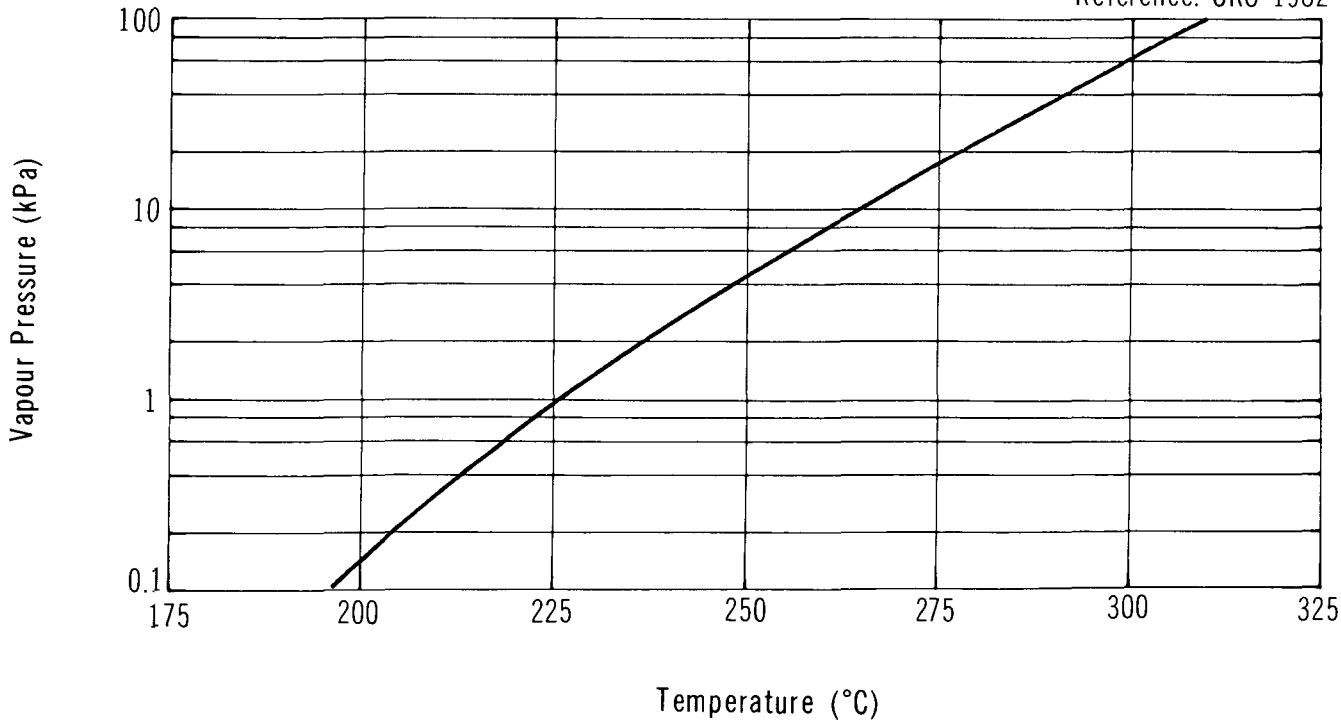
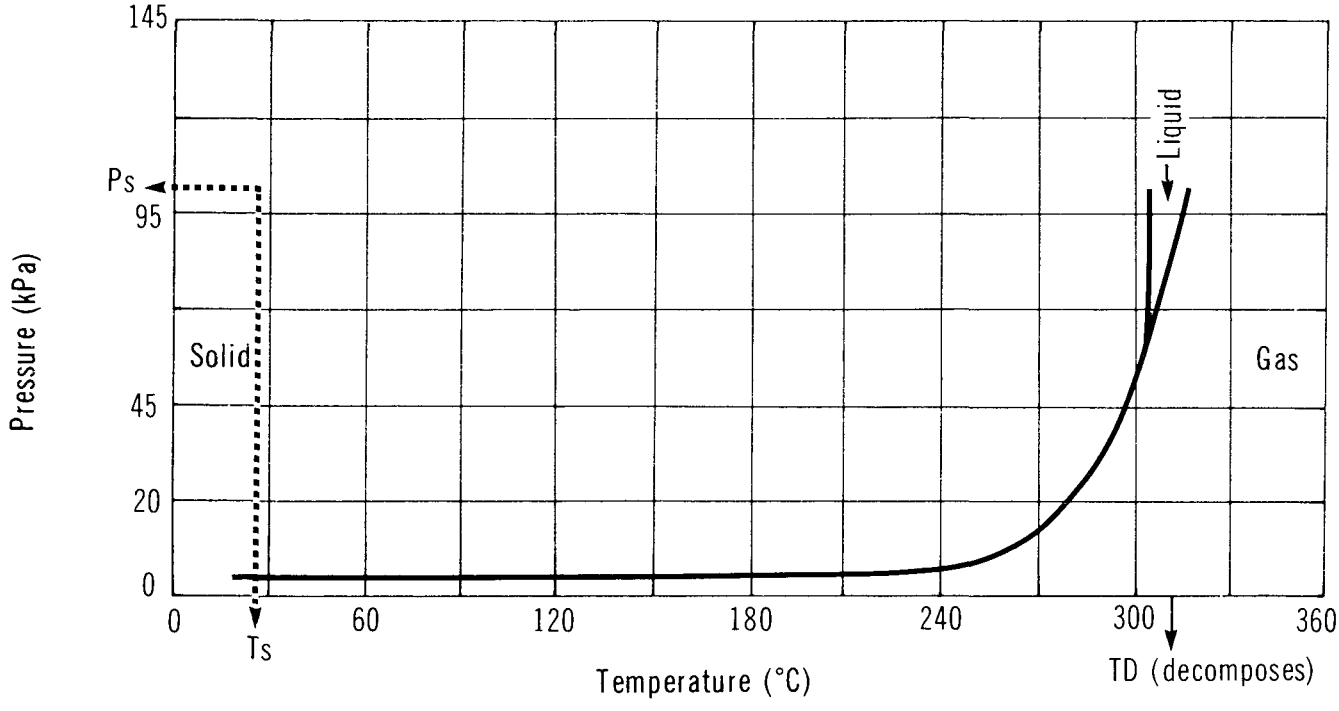


FIGURE 4

FERRIC CHLORIDE

PHASE DIAGRAM



3 COMMERCE AND PRODUCTION

3.1 Grades and Concentrations (PC 1981; Ullmann 1975)

Ferric chloride is available as a solid (96 to 99 percent $FeCl_3$, or as the hexahydrate, $FeCl_3 \cdot 6H_2O$, with a purity of 60 percent, taken as $FeCl_3$ equivalent) and in solution form (sometimes referred to as coagulation or flocculation grades, with concentrations from 29 percent to 45 percent).

3.2 Domestic Manufacturer (CCR 1978; Scott 1979)

Diversey Corporation of Canada
2645 Royal Windsor Drive
Mississauga, Ontario
L5J 1L1
(416) 291-1137

3.3 Other Supplier (CBG 1980)

Pennwalt of Canada Ltd.
700 Third Line
Oakville, Ontario
L6J 5A3
(416) 827-9841

3.4 Major Transportation Routes

Since ferric chloride is used extensively in sewage treatment plants, it is shipped across Canada.

3.5 Production Levels (PC 1981)

Company, Plant Location	Capacity kilotonnes/yr (1981)
Diversey Wyandotte, Mississauga, Ont. (solution)	23

3.6 Manufacture of Ferric Chloride (PC 1981)

3.6.1 General. Ferric chloride solution is made by reacting hydrochloric acid and ferric oxide, or by chlorination of spent steel pickling liquor. (Pickling liquor is an acid, usually hydrochloric, sulphuric or phosphoric, used to remove scale, oxides, or other impurities from steel surfaces.)

3.6.2 Raw Materials. Materials used in the manufacture of ferric chloride include hydrochloric acid, high-grade ferric oxide from steel processing, spent steel pickling liquor, and chlorine.

3.6.3 Manufacturing Process. Two methods are used to produce ferric chloride solution (Campbell 1982):

a) Ferric oxide dry cake (magnetite) from the steel industry, an iron-rich dust from the process furnaces, is slurried and fed to a reactor system. Anhydrous hydrogen chloride (usually a by-product material from the manufacture of chlorinated hydrocarbons) is then added. Since the resulting ferric chloride solution contains impurities, it is next fed to a purification system for processing and then to storage from which it is shipped to the users. The overall reaction is represented by the following equation:

$$Fe_2O_3 + 6HCl \rightarrow 2FeCl_3 + 3H_2O$$

Due to a shortage of magnetite coupled with increasing costs of the material, a second process was developed and is currently in use in Canada.

b) Waste pickling liquor (ferrous chloride) from the steel industry is treated with chlorine to produce usable ferric chloride:

$$2FeCl_2 + Cl_2 \rightarrow 2FeCl_3$$

Again, the chlorine may be considered a by-product material of the caustic soda industry. Since waste pickling liquor may contain ferrous sulphate instead of the chloride, this material may also be treated with chlorine. This results in the presence of sulphate as well as chloride ions in the resulting solution containing the ferric ion, but this is acceptable as the latter is the active ingredient for water treatment purposes. Purification to remove undesirable contaminants is carried out and the material made ready for shipment to the user.

3.7 Major Uses in Canada (PC 1981; Campbell 1982)

Ferric chloride solution is used for coagulation and flocculation in sewage treatment plants, as a general chemical reagent, and for etching of electronic printed circuit boards. It is also used for water purification and is particularly effective in removal of phosphate from municipal sewage treatment plant effluents.

3.8 Major Buyers in Canada

Sewage treatment and water purification plants are the major buyers of the product.

4 MATERIAL HANDLING AND COMPATIBILITY

4.1 Containers and Transportation Vessels

4.1.1 General. Ferric chloride solutions are shipped bulk in specially designed railway tank cars and tank motor vehicles.

Anhydrous or dry ferric chloride is shipped in drums and bags and other small containers. The hexahydrate ($FeCl_3 \cdot 6H_2O$) is more corrosive to metals than the anhydrous, and thus is shipped in nonmetallic or lined containers.

4.1.1.1 Railway tank cars. Railway tank cars used in the transportation of ferric chloride solutions are listed in Table 2. Cars are equipped for unloading from the top by compressed air. Ferric chloride is withdrawn through an eduction pipe which extends from the bottom of the tank to the top operating platform where it terminates with a 63 mm (2 1/2 in.) blind flange. Air pressure of 207 kPa (30 psi) maximum is applied through a 63 mm (2 1/2 in.) flange connection (FC).

TABLE 2 RAILWAY TANK CAR SPECIFICATIONS (RTDCR 1974; TCM 1979)

CTC/DOT* Specification Number	Description
103BW	Steel fusion-welded tank with dome. Uninsulated or insulated. Rubber-lined tank. Top unloading arrangement required. Bottom outlet or washout prohibited. Test pressure 414 kPa (60 psi).
111A60W5	Steel fusion-welded tank without dome. Uninsulated or insulated. Rubber-lined tank. 2% minimum outage. Gauging device. Top unloading arrangement required. Bottom outlet or washout prohibited. Test pressure 414 kPa (60 psi).
111A100W5	Steel fusion-welded tank without dome. Uninsulated or insulated. Rubber-lined tank. 2% minimum outage. Gauging device. Top unloading arrangement required. Bottom outlet or washout prohibited. Test pressure 690 kPa (100 psi).

* Canadian Transport Commission and Department of Transportation (U.S.)

4.1.1.2 Tank motor vehicles. Ferric chloride solutions are frequently shipped by tank motor vehicle. The capacities of the tank trucks are dependent upon the applicable provincial truck weight regulations (FC). A double-axle type vehicle is the most common carrier of ferric chloride solutions, ranging in capacity from 12 700 L to 19 000 L (2800-4200 gal.) or 18 200 kg to 27 300 kg (40 000-60 000 lb.) net mass, respectively (PC 1972). Similar to railway tank cars, these trailers are unloaded by compressed air not exceeding 207 kPa (30 psi) (HFC 1965).

Tank motor vehicles must conform to Transport Canada specification TC307 as outlined in Table 3 (TDGC 1980). As in the case of railway tank cars, the materials of construction and thickness of material are similar. Highway tankers must be pressure tested at 276 kPa (40 psi) minimum to allow for the compressed air pressure of 207 kPa (30 psi) used during top unloading. The maximum working pressure of the tanker is in effect 207 kPa (30 psi).

TABLE 3 TANK MOTOR VEHICLE SPECIFICATIONS

Transport Canada Specification Number	Description
TC307 (Incorporating MC304 and MC307)	Steel butt-welded tank. Design and construct in accordance with ASME Code when working pressures in excess of 345 kPa (50 psi). Minimum design pressure 172 kPa (25 psi). Gauging device required. Minimum one pressure relief device per compartment. One minimum 380 mm (15 in.) diameter manhole per compartment. Bottom washout optional. Corrosion allowance required. Minimum test pressure 276 kPa (40 psi).

4.1.2 Packaging. Anhydrous ferric chloride is also transported in drums and bags. Bags must have a minimum 3-ply moisture-resistant paper construction. Maximum net mass is 50 kg (110 lb.) (TDGC 1980).

Drums fabricated from a variety of construction materials are permitted. Table 4 lists drum types and descriptions (TDGC 1980).

TABLE 4 DRUMS

Type of Drum	Designation	Description	Figure No.
Steel	1A1	Nonremovable head, reusable	
	1A1A	1A1 with reinforced chime	
	1A1B	1A1 with welded closure flange	
	1A1C	1A1 with lead coating	5
	1A1D	1A1 with coating (other than lead)	
	1A2	Removable head, reusable	
	1A2A	1A2 with reinforced chime	
	1A2B	1A2 with lead coating	
	1A3	Nonremovable head, single use only	
	1A4	Removable head, single use only	
Aluminum	1B1	Nonremovable head	5
	1B2	Removable head	
Plywood	1D2		
Fibreboard	1G1	Convolutely wound plies. Maximum capacity 250 L (55 gal.) Maximum net mass 400 kg (882 lb.)	
Plastic	1H1	Nonremovable head. Maximum capacity 250 L (55 gal.) Maximum net mass 400 kg (882 lb.)	
	1H2	1H1 except head removable	
Steel Drums with inner plastic receptacles	6HA1	Outer steel sheet in the shape of drum. Inner plastic receptacle. Maximum capacity 225 L (49 gal.)	
Plywood Drums with inner plastic receptacles	6HD1	Outer plywood in shape of drum. Inner plastic receptacle. Maximum capacity 225 L (49 gal.)	
Fibreboard Drums with inner plastic receptacles	6HG1	Outer containers of convolutely wound plies of fibreboard. Inner plastic receptacle in shape of drum. Maximum capacity 225 L (49 gal.)	

4.2 Off-loading

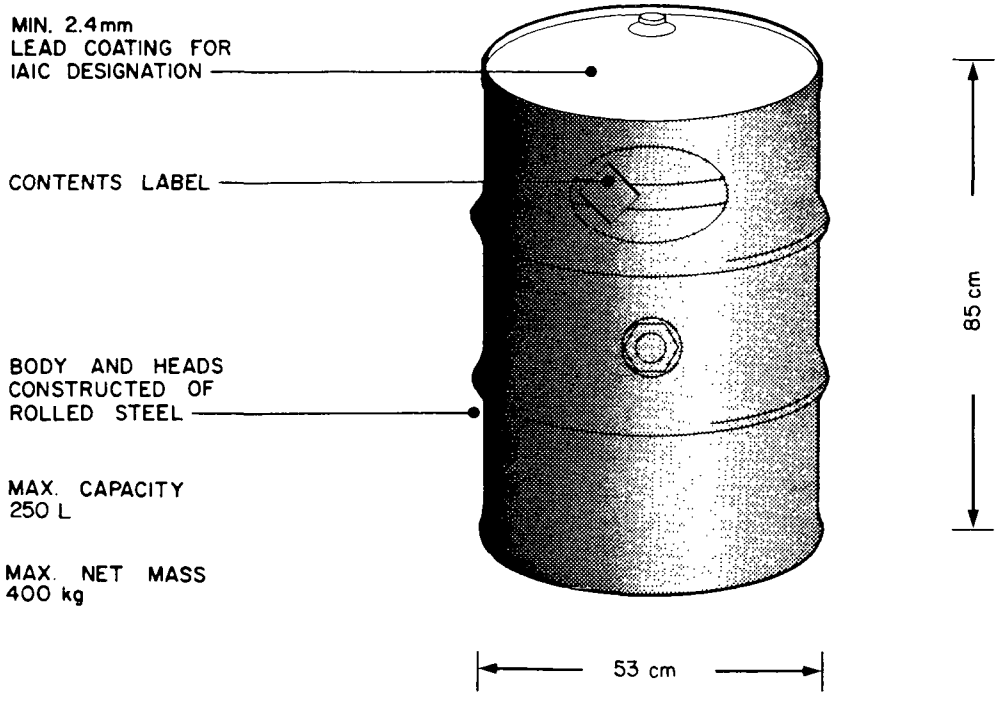
4.2.1 Off-loading Equipment and Procedures for Railway Tank Cars. Prior to off-loading, certain precautions must be taken (HFC 1965):

FIGURE 5

FERRIC CHLORIDE

TYPICAL DRUM CONTAINER

Typical steel drum (lined for ferric chloride service)



- The storage tank must be checked to make sure that it will hold the contents of the car.
- Personnel must not enter the car under any circumstances.
- Brakes must be set, wheels chocked and a derail employed.
- A safe operating platform must be provided at the unloading point.

Proceed with off-loading as follows (HFC 1965):

- After removing the safety vent cap and assembly from the air inlet flange, and the blind flange from the liquid unloading connection, connect the 51 mm (2 in.) unloading line and then the 25 mm (1 in.) air line. Air pressure must be reduced to 207 kPa (30 psi) for unloading. A safety relief valve must be installed in the air line to release at 242 kPa (35 psi).
- Apply air pressure to unload the car.
- Once the car is empty, the air supply valve must be closed and the vent valve in the air line opened to allow the line pressure to equalize to atmospheric pressure.
- Reverse the above procedure to close up the car.

4.2.2 Off-loading Equipment and Procedures for Tank Motor Vehicles. The general procedures for railway tank car unloading also apply to tank trailers (HFC 1965).

4.2.3 Specifications and Materials for Off-loading Equipment. The materials of construction for off-loading system components discussed in this section along with specifications refer to those generally used in ferric chloride solution service. It is recognized that other materials may be used for particular applications as indicated in Table 5. The components of a typical off-loading system that will be discussed include pipes and fittings, flexible connections, valves, gaskets, pumps and storage tanks.

The corrosion of carbon steel in contact with aqueous solutions depends on many factors, e.g., solution pH, dissolved oxygen in the water, temperature, dissolved salts, and solution velocity. It is readily known that many inorganic salts are highly corrosive to steel (corrosion rate of greater than 1.27 mm per year), with acidic or neutral salts tending to increase corrosion rates and alkaline salts tending to lower corrosion rates. Also important is whether the added salt yields soluble or insoluble reaction products. Ferric chloride solutions fall into the highly corrosive category, primarily due to chloride content, oxidizing character, and acidity. They are extremely corrosive to carbon steel; their use with nickel and nickel-based alloys is limited. For example, acidic solutions containing the oxidizing ferric ion accelerate the acidic corrosion of Alloy B (62-70 percent Ni, 28 percent Mo). The ferric ions provide rapid and strong cathodic reactions to support the anodic dissolution of nickel and molybdenum. The ferric ion is reduced to the ferrous ion; if the solution contains a sufficient amount of dissolved oxygen to regenerate the ferric ions, the corrosion can be maintained. Alloy C (59 percent Ni, 16 percent Mo and 16 percent Cr), on the other hand, can be used under 65°C and limited concentrations, while Alloy 400 (65 percent Ni, 32 percent Cu) is severely corroded. Types 304 and 316 stainless steels also suffer severe corrosion and chloride pitting when exposed to ferric chloride solutions. These steels are also subject to stress corrosion cracking. Materials listed as giving good service for the full range of solution concentration up to boiling or to the temperature limit of the nonmetallic material are tantalum, glass/silicates, carbon impregnated with furan or phenolic, Teflon (FEP/TFE), furan resin, and epoxy resin (CE 1980b).

Schedule 40 seamless ASTM A106 carbon steel pipes and fittings lined or coated with rubber or polyvinylidene fluoride are recommended (HFC 1965). Flanged joints and welded connections should be used. The first portion of the unloading line should be heavy-duty rubber hose.

The unloading line should be 51 mm (2 in.) pipe to suit the standard 63 x 50 mm (2 1/2 x 2 in.) rubberized reducing elbow (FC) on ferric chloride solution tank cars. Pipe under 25 mm (1 in.) is not recommended. Outdoor lines must be self-draining (HFC 1965).

Cast iron or cast steel diaphragm valves lined with polyvinylidene chloride resin will give best service (DPLV 1972). Viton gaskets can be used.

A flanged, sealless, magnetic drive centrifugal pump with wet end parts of 316 stainless steel is recommended for pumping. However, a rubber-lined centrifugal pump with rotating and stationary Teflon seals may also be used (HFC 1965).

Welded steel storage tanks with rubber linings are commonly used. Exposed surfaces are painted with a ferric chloride-resistant paint (FC). Storage tanks should be sized to contain at least one and a half times the usual shipment (HFC 1965).

4.3 Compatibility with Materials of Construction.

The compatibility of ferric chloride with materials of construction is indicated in Table 5. The unbracketed abbreviations are described in Table 6. The rating system for this report is briefly described below.

Recommended: This material will perform satisfactorily in the given application.

Conditional: Material will show deterioration in the given application; however, it may be suitable for intermittent or short-term service.

Not Recommended: Material will be severely affected in this application and should not be used.

TABLE 5 COMPATIBILITY WITH MATERIALS OF CONSTRUCTION

	Chemical		Material of Construction		
Application	Conc.	Temp. (°C)	Recommended	Conditional	Not Recommended
1. Pipes and Fittings	>5%	49	PE (DPPED 1967)		
		52	PVDC (DCRG 1978)		
		60	PVC I PVC II (DPPED 1967)		
		71	ABS (DPPED 1967)		

TABLE 5 COMPATIBILITY WITH MATERIALS OF CONSTRUCTION (Cont'd)

Application	Chemical		Material of Construction		
	Conc.	Temp. (°C)	Recommended	Conditional	Not Recommended
1. Pipes and Fittings (Cont'd)		107	PP (DCRG 1978)		
		121	Chlorinated Polyether (DCRG 1978)		
		135	PVDF (DCRG 1978)		
		To operating limit of material	PVC I ABS PE (MWPP 1978)		
			Rubber- or PVCD-lined CS, Hard Rubber, "Pyrex", PVC Piping (HFC 1965)		
2. Valves	>5%	21			SS 316 (JSSV 1979)
3. Pumps	>5%	93	GRP with FPM "0" Ring		
		Cold	High Silicon Cast Iron (HIS 1969)		
4. Storage	>5% to saturated		CS, Lined with Semi-hard Rubber, Glass, Phenolic Resins, Epoxy Resins, PVC (HFC 1965)		
	Up to 10%	40	μPVC, PE, PP POM, NR, NBR, IIR, EPDM, CR, FPM, CSM (GF)		
5. Others	1%, still	21	SS 304, 304L, 321, 347, 316, 316L, Ni (SFC 1982)	Monel, Inconel (SFC 1982)	

TABLE 5 COMPATIBILITY WITH MATERIALS OF CONSTRUCTION (Cont'd)

Application	Chemical		Material of Construction		
	Conc.	Temp. (°C)	Recommended	Conditional	Not Recommended
5. Others (Cont'd)		boiling		SS 316, 316L (SFC 1982)	SS 304, 304L, 321, 347, Ni, Monel, Inconel (SFC 1982)
	5%, still	21		SS 316, 316L, Inconel (SFC 1982)	SS 304, 304L, 321, 347, Ni, Monel (SFC 1982)
	5%, agitated	21	Nb, Hastelloy C-276 (CE 1983)	SS 304, 304L, 321, 347, 316*, 316L, Inconel (SFC 1982)	Ni, Monel (SFC 1982) Alloy 20, Hastelloy B-2 (CE 1983)
	5%, aerated	21		SS 304, 304L, 321, 347, 316, 316L, Inconel (SFC 1982)	Ni, Monel (SFC 1982)
	5%	RT	Ti, Ta (AMC)		Zr (AMC)
		100	Ti, Ti-Pd, Ta (AMC)		Zr (AMC)
	10%	20	Ti, Ta (AMC)	SS 316 (ASS)	SS 302, 304, 410, 430, (ASS) Zr (AMC)
		65.5	FGRP (red) FGRP (silver) (AOS 1982)		
		93	FGRP (Poly-thread) (AOS 1982)		
		96	FGRP (green) FGRP (Chemline) (AOS 1982)		
		100	Ti, Ti-Pd, Ta (AMC)		Zr (AMC)
	10-100%	24-100	Glass (CDS 1967)		
		24-52	Wood (CDS 1967)		

TABLE 5 COMPATIBILITY WITH MATERIALS OF CONSTRUCTION (Cont'd)

Application	Chemical		Material of Construction		
	Conc.	Temp. (°C)	Recommended	Conditional	Not Recommended
5. Others (Cont'd)		84			Concrete (CDS 1967)
		100			Wood (CDS 1967)
	20%	20	Ti, Ta (AMC)		Zr (AMC)
		100	Ti, Ti-Pd, Ta (AMC)		Zr (AMC)
	30%	20	Ti, Ta (AMC)		Zr (AMC)
		100	Ti, Ti-Pd, Ta (AMC)		Zr (AMC)
	40%	65.5	FGRP (red)		
			FGRP (silver) (AOS 1982)		
		93	FGRP (Poly-thread (AOS 1982)		
		96	FGRP (green) FGRP (Chemline) (AOS 1982)		
	Satu-rated	21	PVDF (TPS 1978)		
		60	PVC (TPS 1978) "Soft", Natural Rubbers and Plastics (HFC 1965)		
		65	Alloy C, Phenolic Resin		
		66	PP (TPS 1978)		
		71	PE, Polystyrene, Polyvinyl (HFC 1965)		
		80	PP, NBR, IIR, EPDM, CR, FPM, CSM (GF)	PE, POM (GF)	uPVC, NR (GF)

TABLE 5 COMPATIBILITY WITH MATERIALS OF CONSTRUCTION (Cont'd)

Application	Chemical		Material of Construction		
	Conc.	Temp. (°C)	Recommended	Conditional	Not Recommended
5. Others (Cont'd)		82	"Hard" Linings of Rubbers and Plastics (HFC 1965)	PP (TPS 1978)	
		85	CPVC (TPS 1978) Glass, Chemical Porcelain, Chemical Stoneware, Acid Brick (HFC 1965)		
	Saturated	100	NBR, IIR, EPDM, CR, FPM, CSM (GF)	PP (GF)	uPVC, PE, POM, NR (GF)
	not stated	not stated	Glass/Silicates, Carbon Impregnated with Furan, Carbon Impregnated with Phenolic, TFE, FEP, Furan Resin, Epoxy Resin (CE 1980b)		
	100%	21	IIR, Gatron, (CE 1980a)	CSM, Polyester elastomer, FPM (CE 1980a)	CS, SS 304, 316 Al, Brass (CE 1980a)

* This material has been given a lower rating in a similar application by another reference.

TABLE 6 MATERIALS OF CONSTRUCTION

Abbreviation	Material of Construction
ABS	Acrylonitrile Butadiene Styrene
	Alloy 20
	Alloy C
	Aluminum
	Cast Iron, High Silicon

TABLE 6 MATERIALS OF CONSTRUCTION (Cont'd)

Abbreviation	Material of Construction
	Chlorinated Polyether
CPVC	Chlorinated Polyvinyl Chloride
CR	Polychloroprene (Neoprene)
CS	Carbon Steel
CSM	Chlorosulphonated Polyethylene (Hypalon)
	Concrete
	Epoxy Resin
EPDM	Ethylene Propylene Rubber
	Fibreglass Reinforced Plastic
FPM	Fluorine Rubber (Viton)
	Furan Resin
	Glass
GRP	Glass Reinforced Vinyl Ester
	Hastelloy B-2
	Inconel
IIR	Isobutylene/Isoprene (Butyl) Rubber
	Monel
NBR	Acrylonitrile/Butadiene Rubber (Nitrile, Buna N)
NR	Natural Rubber
	Nickel
	Phenolic
PE	Polyethylene
POM	Polyoxymethylene
PP	Polypropylene
	Porcelain
PTFE	Polytetrafluoroethylene (Teflon)
PVC (followed by grade, if any)	Polyvinyl Chloride
PVDC	Polyvinylidene Chloride
PVDF	Polyvinyledene Fluoride
	Polystyrene
SBR	Styrene/Butadiene (GR-5, Buna S) Rubber

TABLE 6 MATERIALS OF CONSTRUCTION (Cont'd)

Abbreviation	Material of Construction
SS (followed by grade)	Stainless Steel
Ta	Tantalum
Ti	Titanium
Ti-Pd	Titanium-paladium
uPVC	Unplasticized Polyvinyl Chloride
	Wood (Plywood, Fir)
Zr	Zirconium

5 CONTAMINANT TRANSPORT

5.1 General Summary

Ferric chloride is sometimes transported as a solid, or as aqueous solutions of 29 to 46 percent concentration in railway tank cars or in tank trucks. When spilled in water, all forms will dissolve rapidly. When spilled on soil, the liquid forms will spread on the surface and penetrate into the soil at a rate dependent on the soil type and its water content. Movement of ferric chloride toward the water table may be an environmental problem. Because ferric chloride is essentially nonvolatile, dispersion in air is not a problem.

Factors considered for the transport of a ferric chloride spill in water and soil include the following:

- Contaminant Transport
  - Leak or spill
    - Rate of discharge
    - Percent remaining
  - Water — Diffusion and downstream concentration
  - Soil — Depth and time of penetration

It is important to note that, because of the approximate nature of the contaminant transport calculations, the approach adopted throughout has been to use conservative estimates of critical parameters so that predictions are approaching worst case scenarios for each medium. This may require that the assumptions made for each medium be quite different and to some extent inconsistent. As well as producing worst case scenarios, this approach allows comparison of the behaviours of different chemicals under consistent assumptions.

5.2 Leak Nomograms

5.2.1 Introduction. In solution form, ferric chloride would be transported in largest quantities by railway tank car. While the capacities of the tank cars vary widely, one tank car, with a carrying capacity of about 80 000 L, has been chosen throughout the EnviroTIPS series for development of the leak nomograms.

If the tank car loaded with dilute aqueous solution is punctured on the bottom, all of the contents will drain out by gravity. The aim of the nomograms is to provide a

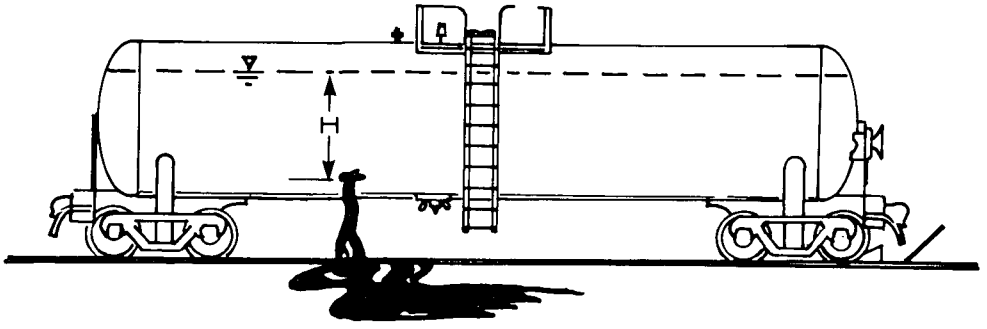


FIGURE 6 TANK CAR WITH PUNCTURE HOLE IN BOTTOM

simple means to obtain the time history of the conditions in the tank car and the leak rate of the liquid.

Because of the low volatility of ferric chloride solution and the fact that tank cars are not pressurized, no leak nomograms have been prepared for vapour release from a puncture in the top of the tank.

5.2.2 Nomograms.

5.2.2.1 Figure 7: Percent remaining versus time. Figure 7 provides a means of estimating the percent of liquid remaining in the standard tank car after the time of puncture for a number of different hole diameters. The hole diameter is actually an equivalent diameter and can be applied to a noncircular puncture.

The standard tank car (2.75 m Ø x 13.4 m long) is assumed to be initially full (at t=O) with a volume of about 80 000 L of ferric chloride solution. The amount remaining at any time (t) is not only a function of the discharge rate over time, but also of the size and shape of the tank car.

5.2.2.2 Figure 8: Discharge rate versus time. Figure 8 provides a means of estimating the instantaneous discharge rate (L/s) at any time (t) after the time of puncture for a number of equivalent hole diameters. The nomogram is only applicable to the standard tank car size with an initial volume of 80 000 L.

5.2.3 Sample Calculations.

i) Problem A

The standard tank car (80 000 L capacity) filled with a 45 percent aqueous solution of ferric chloride has been punctured on the bottom. The equivalent diameter of the hole is 150 mm. What percent of the initial 80 000 L remains after 10 minutes?

FIGURE 7

FERRIC CHLORIDE

PERCENT REMAINING VS TIME

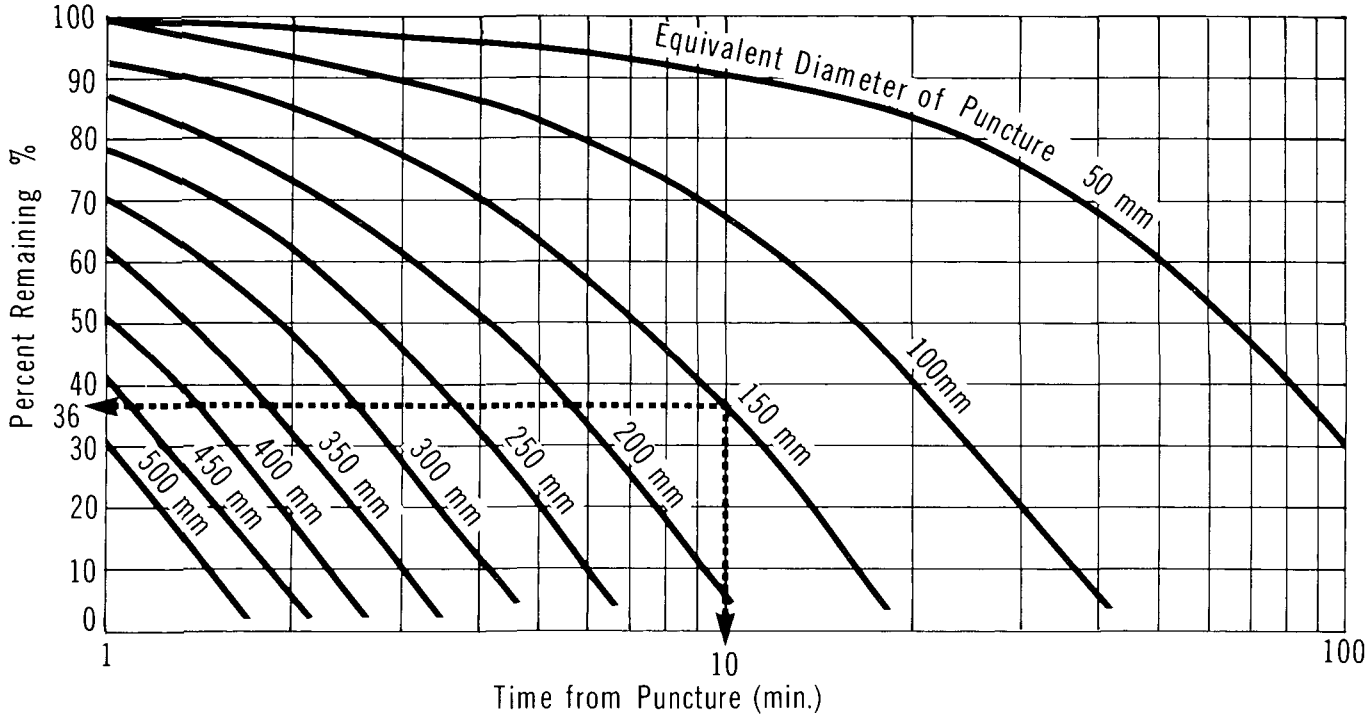
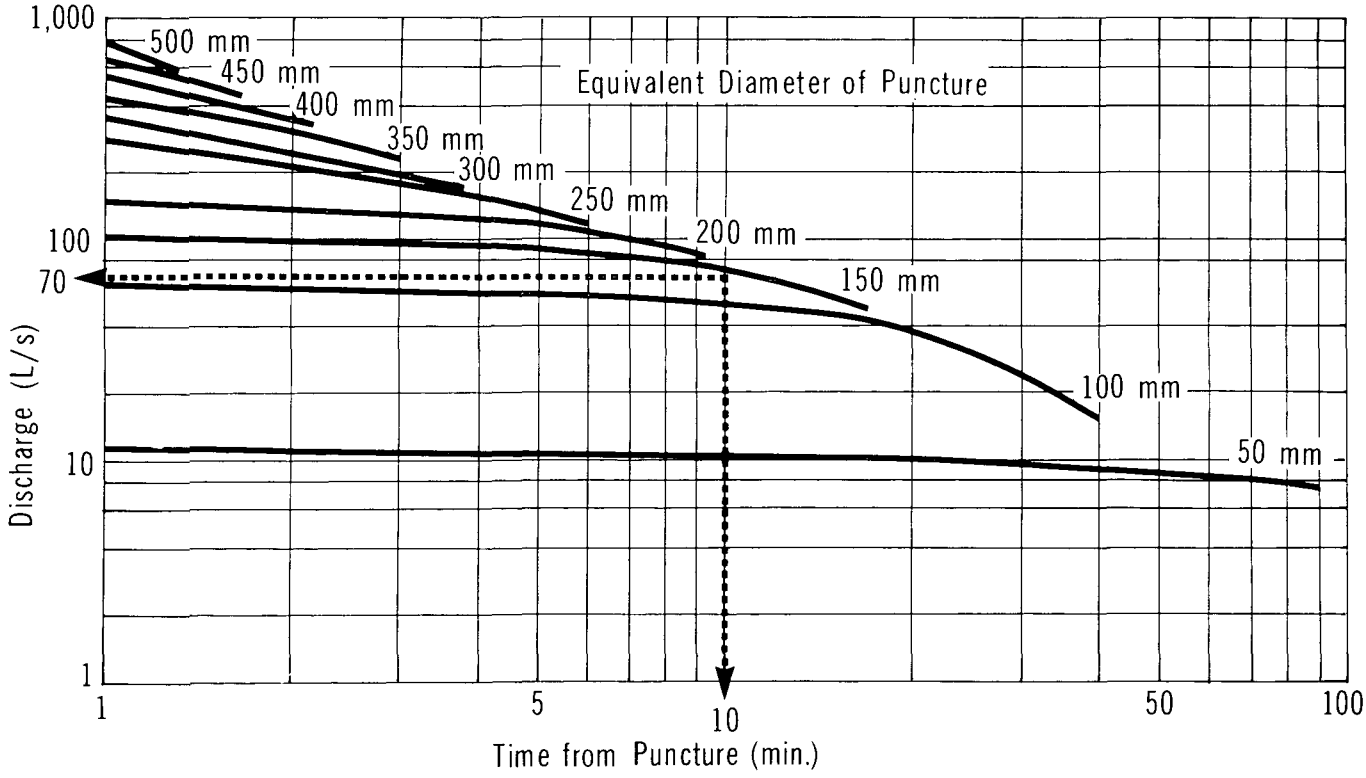


FIGURE 8

FERRIC CHLORIDE

DISCHARGE RATE VS TIME



Solution to Problem A

- Use Figure 7
- With t=10 min and d=150 mm, the amount remaining is about 36 percent, or 28 800 L

ii) Problem B

With the same conditions as Problem A, what is the instantaneous discharge rate from the tank 10 minutes after the accident?

Solution to Problem B

- Use Figure 8
- With t=10 min and d=150 mm, the instantaneous discharge rate (q) = 70 L/s

5.3 Dispersion in the Air

Because ferric chloride is nonvolatile in foreseeable spill circumstances, there is no significant potential for dispersion in the air.

5.4 Behaviour in Water

5.4.1 Introduction. When spilled on a water surface, ferric chloride solid or solution will dissolve or mix rapidly. This mixing can generally be described by classical diffusion equations with one or more diffusion coefficients. In rivers, the principal mixing agent is stream turbulence, while in calm water mixing takes place by molecular diffusion.

As mentioned in Section 2 of this manual, the ferric ion in aqueous solution will tend to hydrolyze in all except the most acid water. Hydroxyl ions often have a stronger affinity for ferric ion than do organic or inorganic bases. Ferric ions also have a relatively strong tendency to form complexes in aqueous solution. It will, for example, react with orthophosphate to form the soluble complex $FeHPO_4^+$, which under acid conditions will precipitate as $FePO_4$. In neutral or slightly basic water, the precipitate is a metastable compound probably containing both PO_4^{-3} and OH^- ions in variable proportions depending on the pH. The hydrolysis of ferric ion forms the mononuclear species $(Fe(H_2O)_5OH)^{+2}$ and $(Fe(H_2O)_4(OH)_2)^{+1}$. Other species that have been identified are $(Fe(H_2O)_4(OH)_2Fe(H_2O)_4)^{+4}$, $(Fe_3(H_2O)_4(OH)_4)^{+5}$ and $(Fe(H_2O)_2(OH)_4)^-$. In the pH range 5 to 8, ferric ion cannot remain in solution, but only in suspension or as complexes. Generally, the hydrolysis of ferric ion will lead to the formation of hydrous ferric oxide, FeO(OH), as a suspension. Once spilled into a water system, the form of the ion will depend upon the water conditions and will almost certainly lower the pH of the immediate

surrounding water (Cotton 1972; Hesse 1972; Water Analysis 1982). The remainder of Section 5.4 in no way attempts to take into account the ultimate form of the spilled ferric ion or the final condition of the water, e.g., pH, resulting from interactions of ferric ion with water.

To estimate the pollutant concentration in a river downstream from a spill, the turbulent diffusion has been modelled. The model employed is strictly applicable to neutrally buoyant liquids and solids that dissolve in water. As ferric chloride is denser than water, initially the maximum concentration would be expected near the bottom. As mixing takes place, the concentration becomes uniform.

The one-dimensional model uses an idealized rectangular channel section and assumes a uniform concentration of the pollutant throughout the section. Obviously, this applies only to points sufficiently far downstream of the spill where mixing and dilution have distributed the pollutant across the entire river channel. The model is applicable to rivers where the ratio of width to depth is less than 100 ($W/d < 100$) and assumes a Manning's roughness coefficient of 0.03. Details of the model are outlined in the Introduction Manual.

No modelling has been carried out for molecular diffusion in still water. Rather, nomograms have been prepared to define the hazard zone and the average concentration within the hazard zone as a function of spill size, but independent of time.

5.4.2 Nomograms. The following nomograms are presented to calculate pollutant concentrations in non-tidal rivers and in lakes (still water).

Non-tidal Rivers

Figure 10:	distance versus time for a range of average stream velocities
Figure 11:	hydraulic radius versus channel width for a range of stream depths
Figure 12:	diffusion coefficient versus hydraulic radius for a range of average stream velocities
Figure 13:	alpha* versus diffusion coefficient for various time intervals
Figure 14:	alpha versus delta* for a range of spill sizes
Figure 15:	maximum concentration versus delta for a range of river cross-sectional areas

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* Alpha and delta are conversion factors only and are of no significance other than to facilitate calculation of downstream concentration.

Lakes or Still Water Bodies

Figure 16: volume versus radius for the hazard zone for a range of lake depths

Figure 17: average concentration versus volume for the hazard zone for a range of spill sizes

The flow chart in Figure 9 outlines the steps required to estimate downstream concentration after a spill and identifies the nomograms to be used. These nomograms (Figures 10 through 17) are described in the following sub-sections.

5.4.2.1 Nomograms for non-tidal rivers.

Figure 10: Distance versus time. Figure 10 presents a simple relationship between average stream velocity, time, and distance. Using an estimate of average stream velocity (U), the time (t) to reach any point of interest, at some distance (X) downstream of the spill, can be readily obtained from Figure 10.

Figure 11: Hydraulic radius versus channel width. The model used to estimate downstream pollutant concentration is based on an idealized rectangular channel of width (W) and depth (d). The hydraulic radius (r) for the channel is required in order to estimate the turbulent diffusion coefficient (E). The hydraulic radius (r) is defined as the stream cross-sectional area (A) divided by the wetted perimeter (P). Figure 11 is a nomogram for computation of the hydraulic radius (r) using the width and depth of the idealized river cross-section.

Figure 12: Diffusion coefficient versus hydraulic radius. Figure 12 permits calculation of the longitudinal diffusion coefficient (E), knowing the hydraulic radius (r) from Figure 11 and the average stream velocity (U).

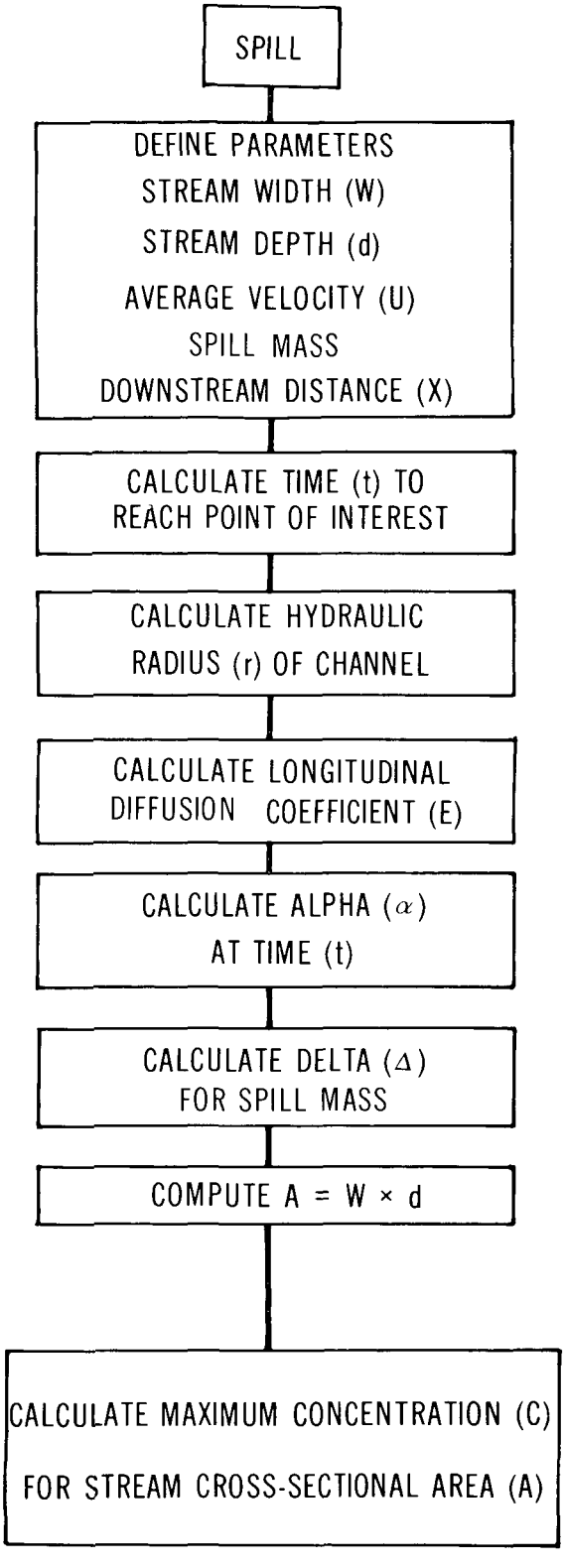
Figure 13: Alpha versus diffusion coefficient. Figure 13 is used to estimate a conversion factor alpha (α), which is a function of the diffusion coefficient (E) and the time (t) to reach the point of interest downstream of the spill.

Figure 14: Alpha versus delta. A second conversion factor, delta (Δ), must be estimated from Figure 14 to allow determination of the pollutant concentration at the point of interest. Delta (Δ) is a function of alpha (α) and the spill size.

Figure 15: Maximum concentration versus delta. Figure 15 represents the final step for calculation of the maximum downstream pollutant concentration (C) at the point of interest. Using the factor delta (Δ) and knowing the stream cross-sectional area (A), the concentration (C) is readily obtained from the nomogram. The value obtained

FERRIC CHLORIDE

FLOW CHART TO DETERMINE POLLUTANT CONCENTRATION IN NON-TIDAL RIVERS



Step 1: Observed or Estimated

W = _____ m

d = _____ m

U = _____ m/s

MASS = _____ tonnes

X = _____ m

Step 2: Use Figure 10

t = _____ minutes

Step 3: Use Figure 11

r = _____ m

Step 4: Use Figure 12

E = _____ m^2/s

Step 5: Use Figure 13

α = _____

Step 6: Use Figure 14

Δ = _____

Step 7: Compute stream cross-sectional Area (A)

A = W × d _____ m^2

Step 8: Use Figure 15

C = _____ ppm

FIGURE 10

FERRIC CHLORIDE

TIME vs DISTANCE

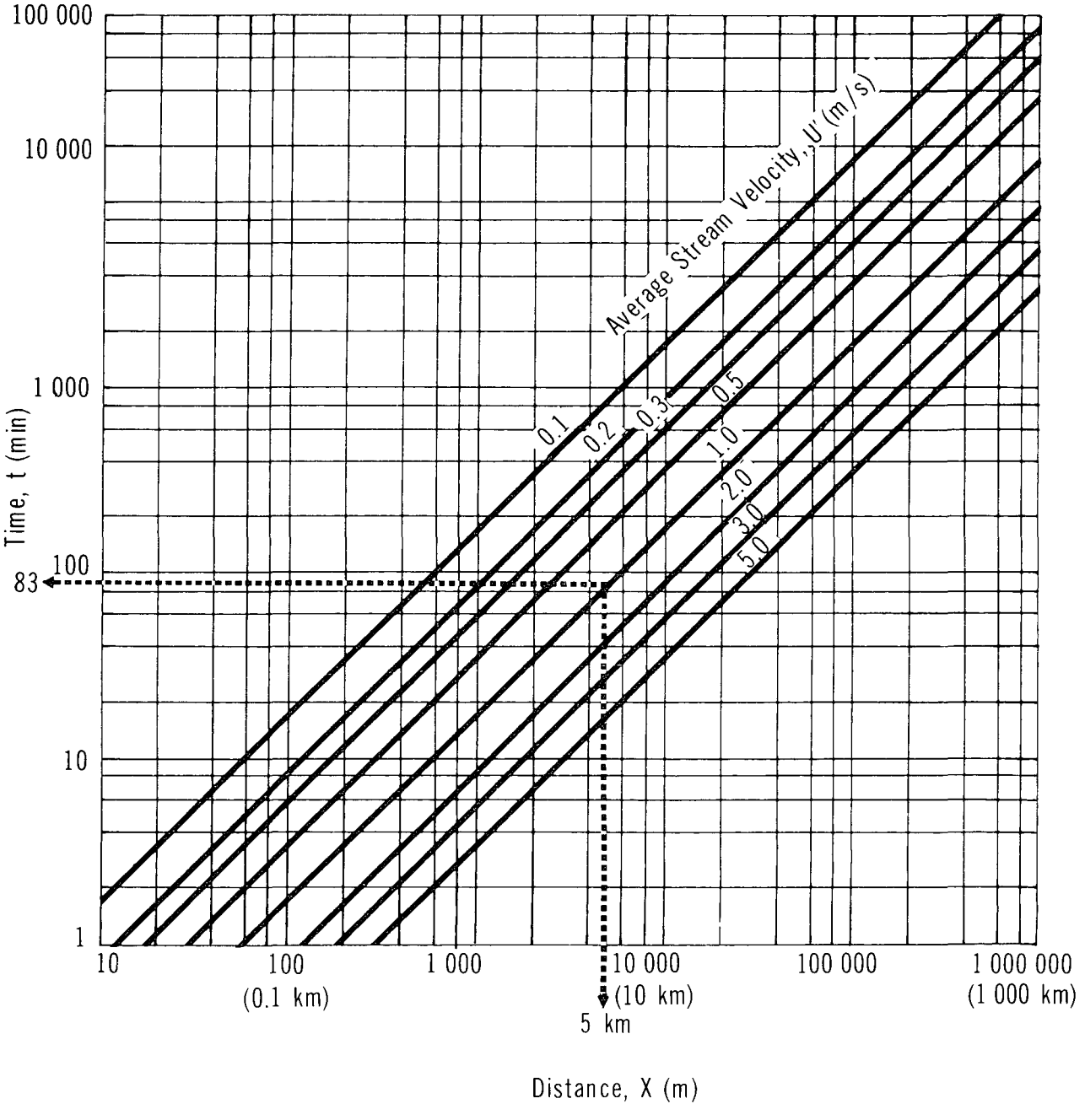


FIGURE 11

FERRIC CHLORIDE

HYDRAULIC RADIUS VS CHANNEL WIDTH

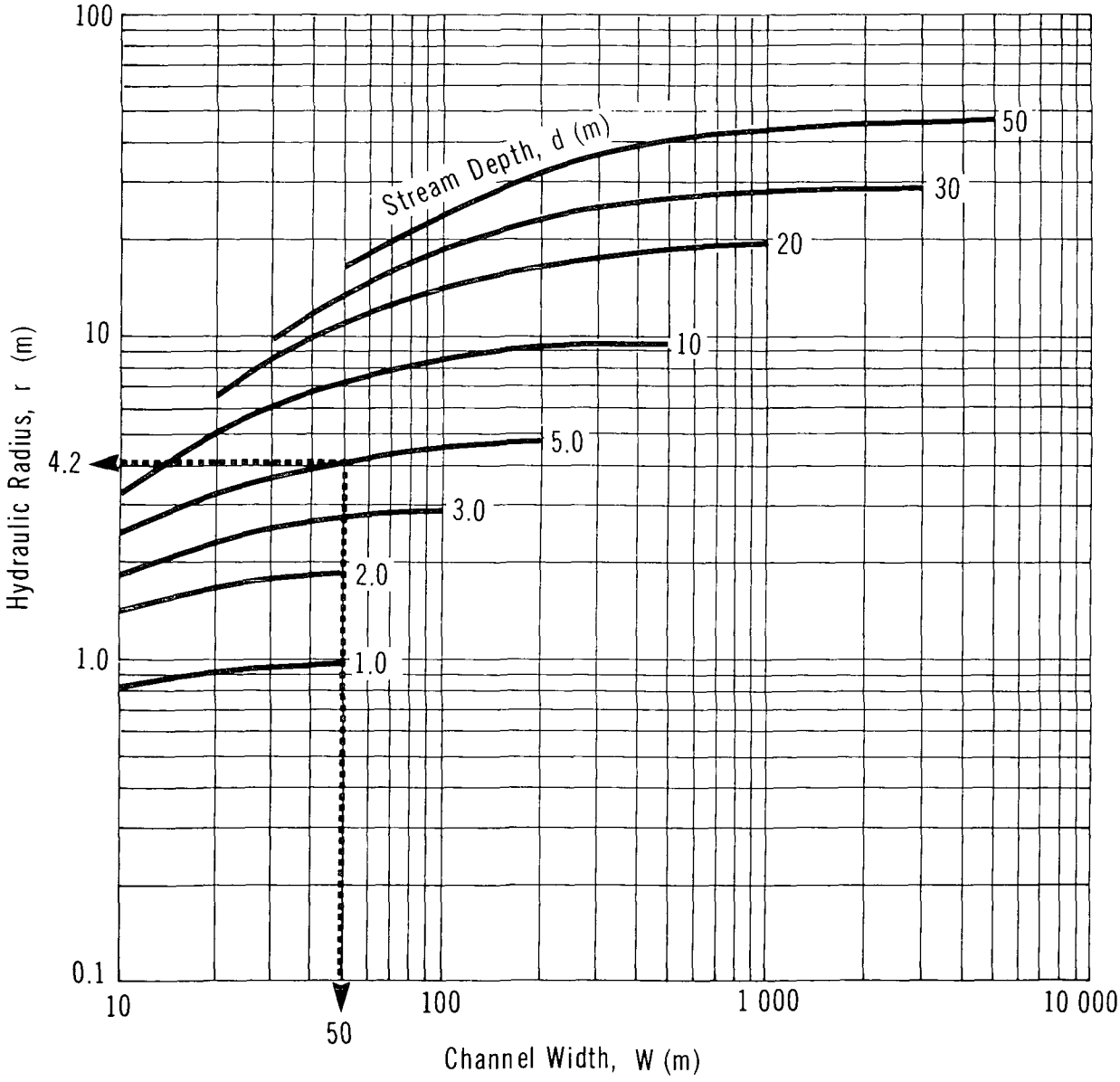


FIGURE 12

FERRIC CHLORIDE

DIFFUSION COEFFICIENT VS HYDRAULIC RADIUS

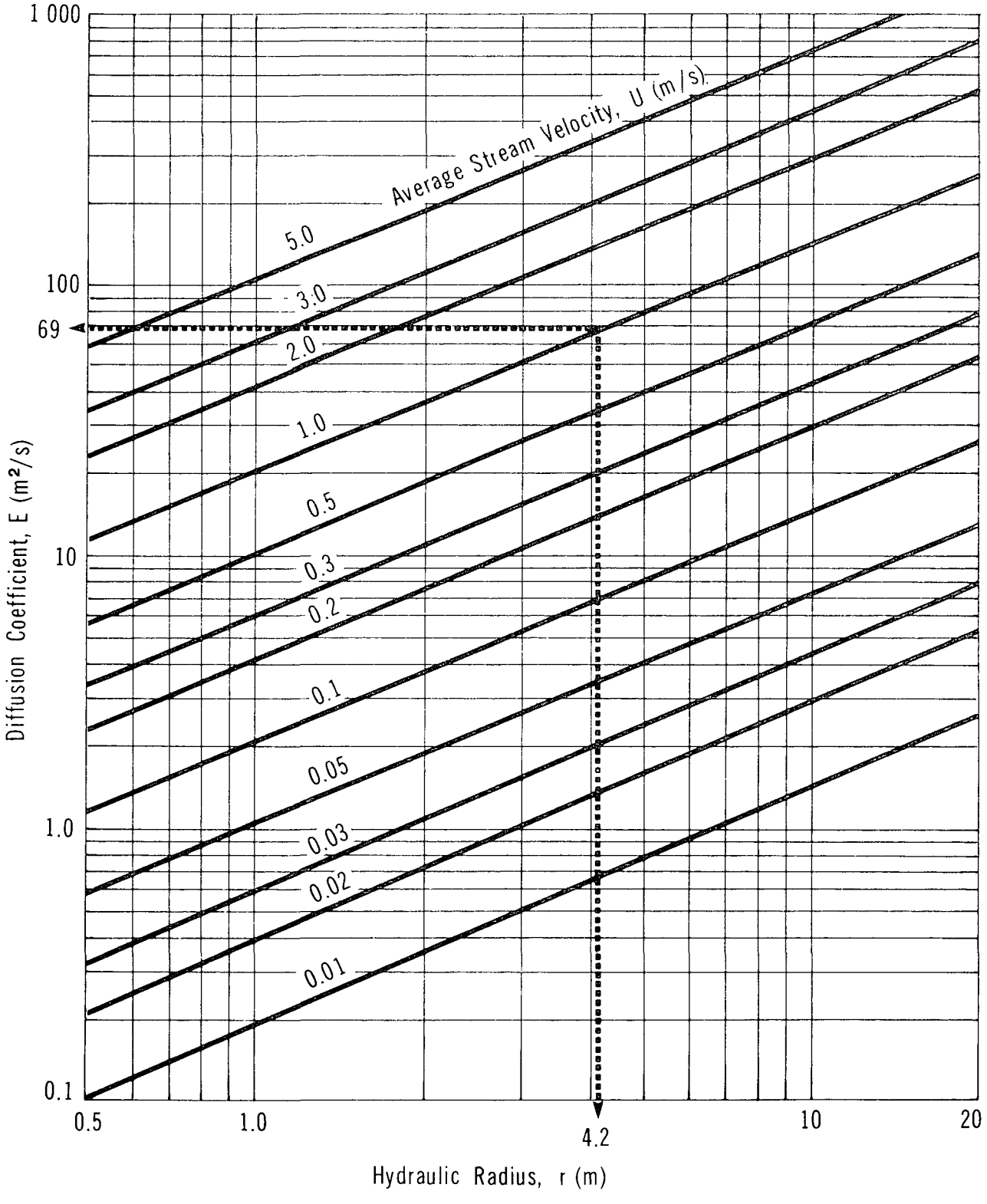


FIGURE 13

FERRIC CHLORIDE

ALPHA vs DIFFUSION COEFFICIENT

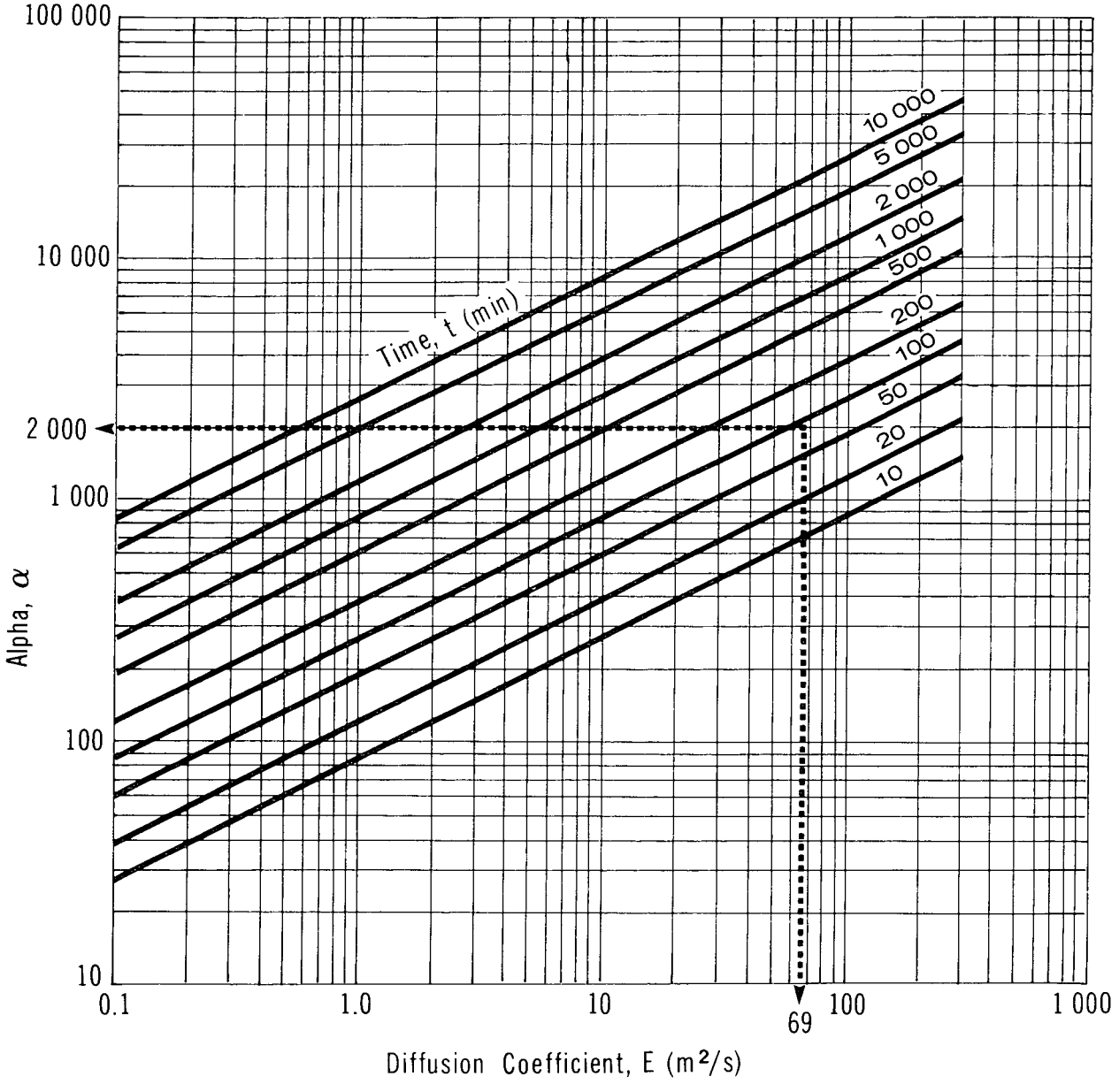


FIGURE 14

FERRIC CHLORIDE

ALPHA vs DELTA

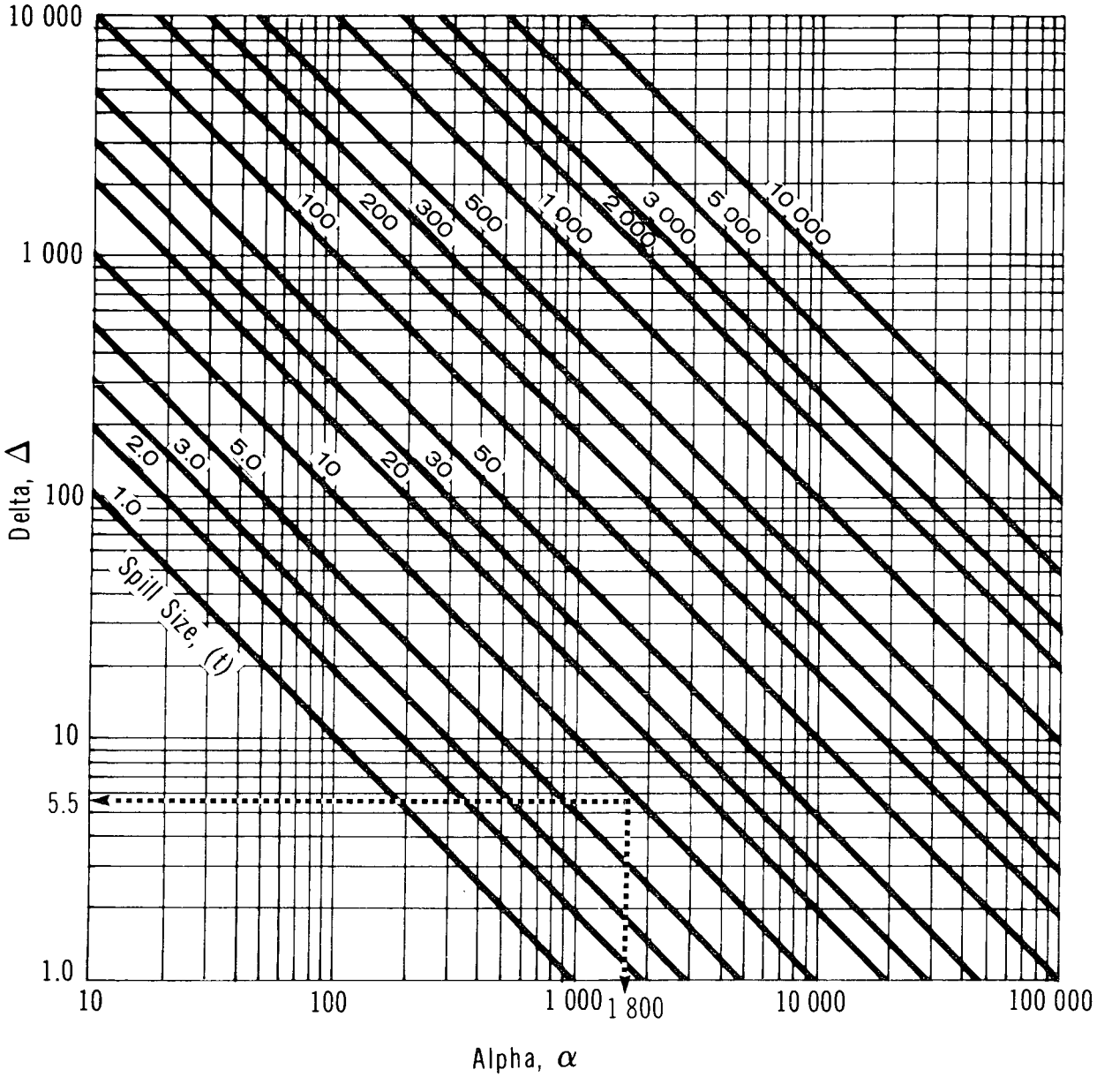
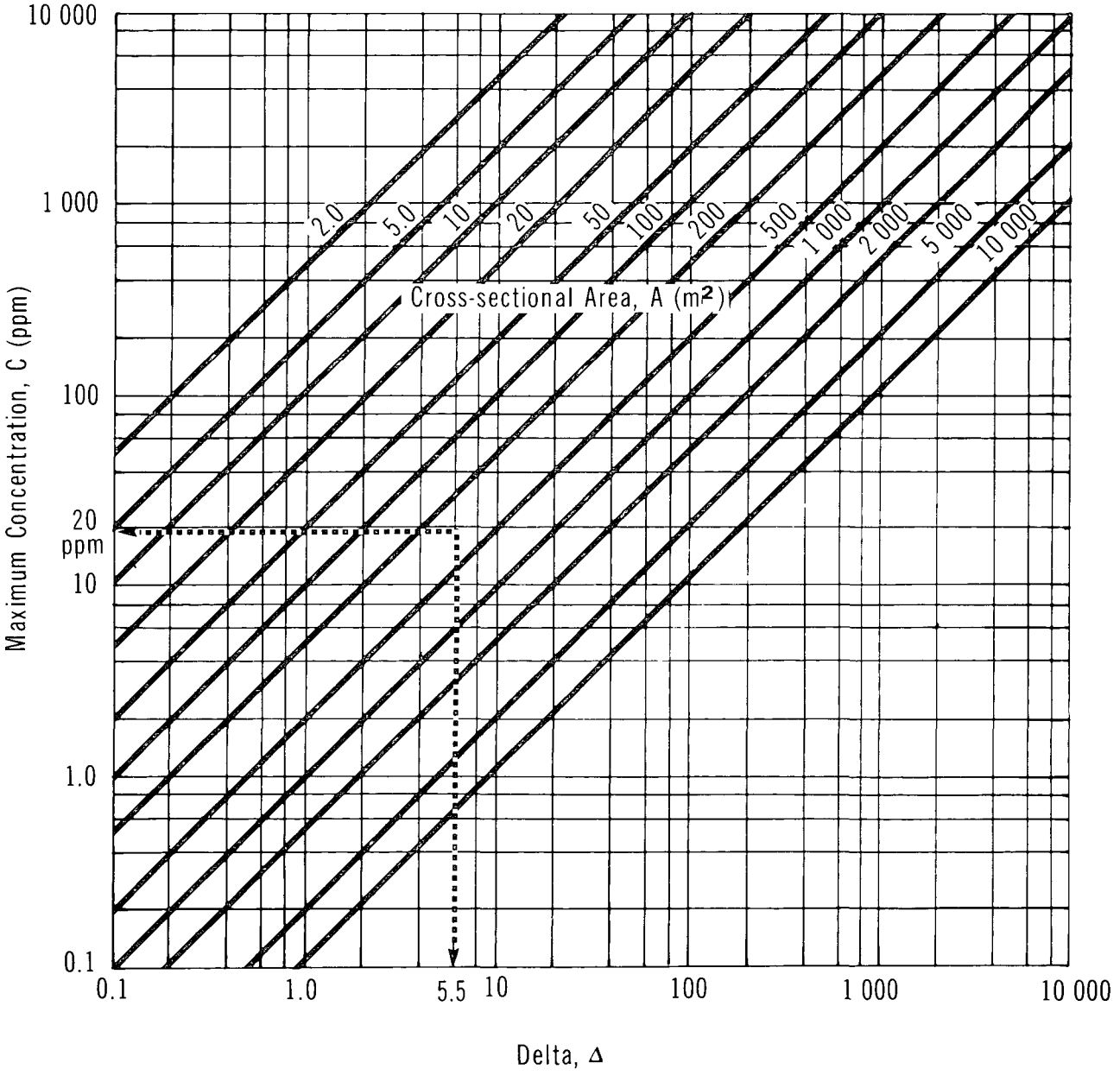


FIGURE 15

FERRIC CHLORIDE

MAXIMUM CONCENTRATION vs DELTA



from Figure 15 applies to neutrally buoyant liquids or solids and will vary somewhat for other pollutants which are heavier or lighter than water.

5.4.2.2 Nomograms for lakes or still water bodies.

Figure 16: Volume versus radius. The spill of a neutrally buoyant liquid in a lake in the absence of wind and current has been idealized as a cylinder of radius (r) and length (d), equivalent to the depth of the lake at the point of spill. The volume of water in the cylinder can be obtained from Figure 16. The radius (r) represents the distance from the spill to the point of interest.

Figure 17: Average concentration versus volume. For a known volume of water (within the idealized cylinder of radius (r) and length (d)), the average concentration of pollutant (C) can be obtained from Figure 17 for a known mass of spill. This assumes the pollutant is spread evenly throughout the cylinder. For pollutants that are more or less dense than water, the actual concentration at the bottom would be higher or lower, respectively.

5.4.3 Sample Calculations.

5.4.3.1 Pollutant concentration in non-tidal rivers. A 20 tonne spill of 45 percent ferric chloride solution has occurred in a river. The stream width is 50 m and the stream depth is 5 m. The average stream velocity is estimated at 1 m/s. What is the maximum concentration expected at a water intake located 5 km downstream?

Solution

Step 1: Define parameters

- W = 50 m
- d = 5 m
- X = 5000 m
- U = 1 m/s
- spill size = 20 tonnes of 45 percent solution, contains the equivalent of 9 tonnes of ferric chloride

Step 2: Calculate time to reach point of interest

- Use Figure 10
- With X = 5000 m and U = 1 m/s, t = 83 min

FIGURE 16

FERRIC CHLORIDE

VOLUME vs RADIUS

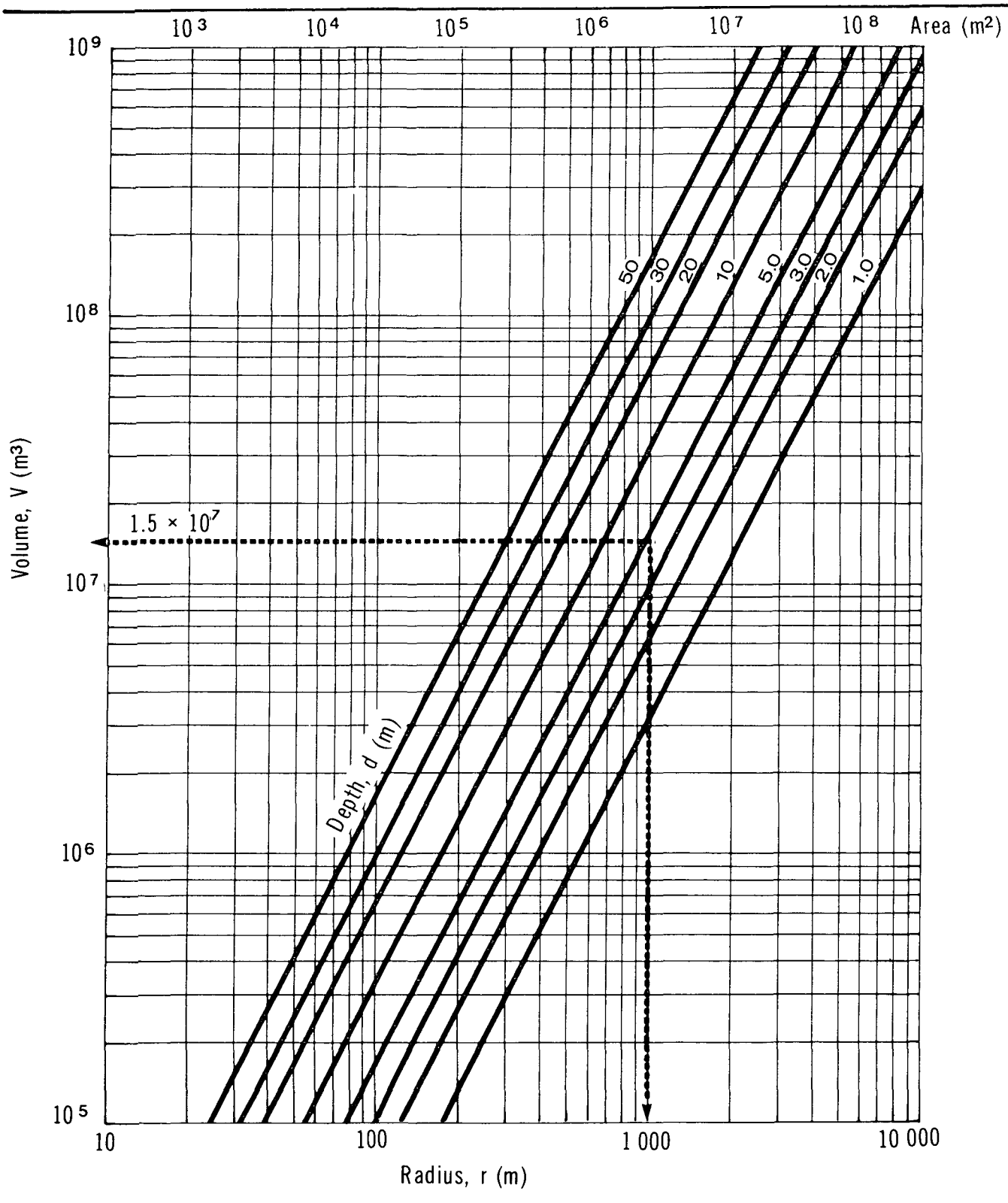
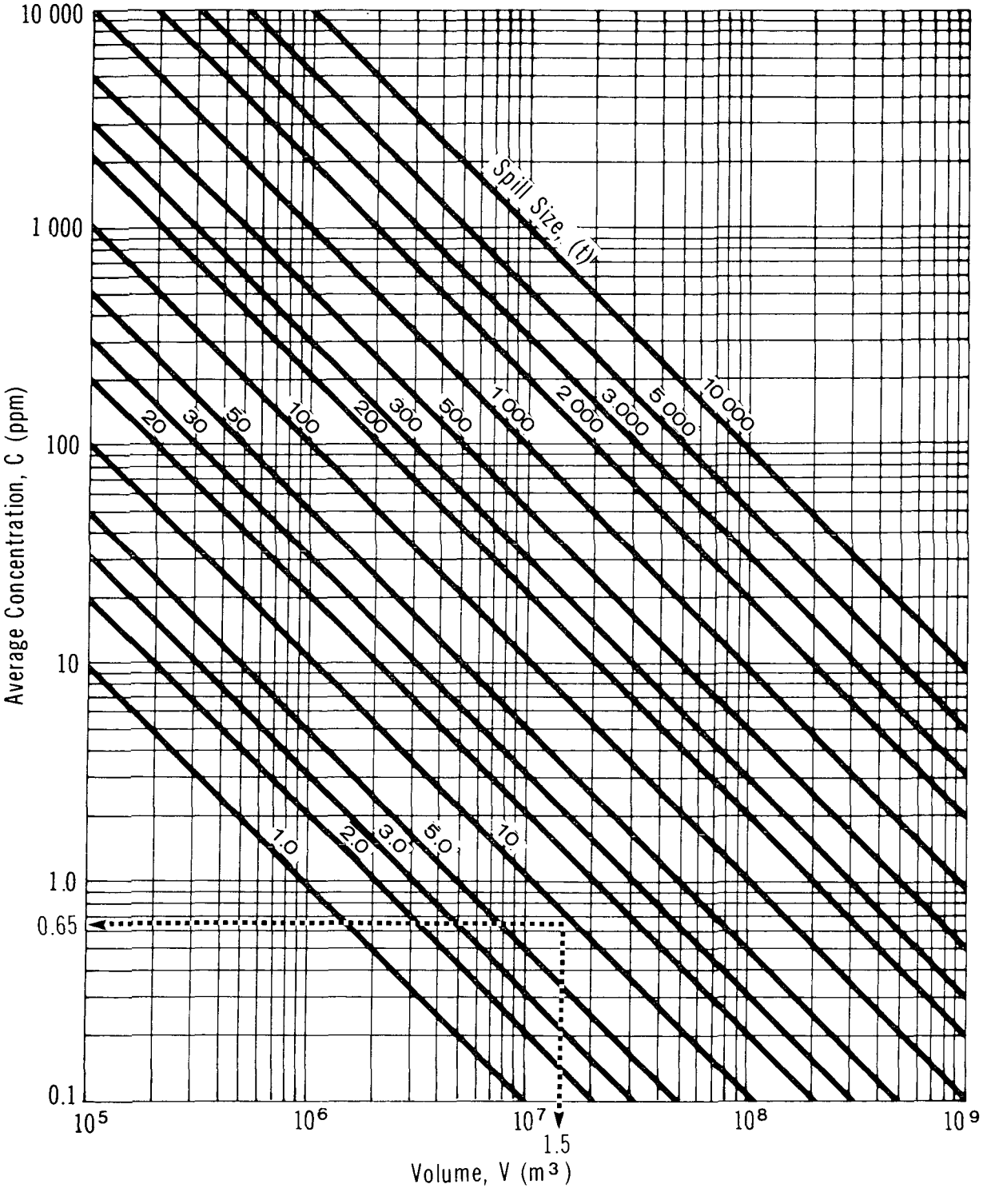


FIGURE 17

FERRIC CHLORIDE

AVERAGE CONCENTRATION vs VOLUME



Step 3: Calculate hydraulic radius (r)

- Use Figure 11
- With W = 50 m and d = 5 m, r = 4.2 m

Step 4: Calculate longitudinal diffusion coefficient (E)

- Use Figure 12
- With r = 4.2 m and U = 1 m/s, E = 69 m^2/s

Step 5: Calculate alpha (α)

- Use Figure 13
- With E = 69 m^2/s and t = 83 min, α = 1800

Step 6: Calculate delta (Δ)

- Use Figure 14
- With alpha (α) = 1800 and spill size = 9 tonnes, delta (Δ) = 5.5

Step 7: Compute stream cross-sectional area (A)

- A = W x d = 50 x 5 = 250 m^2

Step 8: Calculate maximum concentration (C) at point of interest

- Use Figure 15
- With Δ = 5.5 and A = 250 m^2, C = 20 ppm

5.4.3.2 Average pollutant concentration in lakes or still water bodies. A 20 tonne spill of 45 percent ferric chloride solution has occurred in a lake. The point of interest is located on the shore approximately 1000 m from the spill. The average depth between the spill site and the point of interest is 5 m. What is the average concentration which could be expected?

Solution

Step 1: Define parameters

- d = 5 m
- r = 1000 m
- spill size = 9 tonnes (equivalent)
- X = 1000 m

Step 2: Determine the volume of water available for dilution or

- Use Figure 16
- With r = 1000 m, d = 5 m, the volume is approximately 1.5×10^7 m^3

Step 3: Determine the average concentration

- Use Figure 17
- With V = 1.5×10^7 m^3 and spill size = 9 tonnes, the average concentration is 0.65 ppm

5.5 Subsurface Behaviour: Penetration into Soil

5.5.1 Mechanisms. The principles of contaminant transport in soil and their application to this work are presented in the Introduction Manual. Special considerations related to the spill of ferric chloride onto soil and its transport downward through the soil are presented here.

Ferric chloride is shipped as a solid or in solution up to 46 percent by weight. The solutions are miscible with water and, when spilled onto soil, will infiltrate. However, 46 percent by weight ferric chloride is highly viscous and will only infiltrate slowly. If moisture is present at the time of the spill, either as precipitation or as water used to flush the site, dilution of the chemical will occur and infiltration will be enhanced. If, on the other hand, the soil surface is saturated with moisture at the time of the spill, as might be the case after a rainfall, the spilled chemical will runoff or remain ponded.

If solid ferric chloride is spilled, groundwater and soil contamination should not be a problem provided that cleanup occurs prior to precipitation or some other form of water addition.

For this work, the soils have been assumed to be at field capacity. This situation provides very little interstitial water to dilute the chemical during transport or to impede its downward movement and thus represents "worst case" analysis.

During transport through the soil, ferric ion can interact with the soil material and, due to hydrolysis, can contribute to the acidity of a soil. With respect to the latter, for example, it was shown that some of the replaced acidity in soils was due to the presence of ferric ion. Experiments to investigate hydrolysis of ferric ion in soils were carried out using neutral, normal chlorides. The replaced acidity was titrated potentiometrically. It was demonstrated that exchangeable ferric ion is unstable, tending to hydrolyze; the protons produced dissolve the lattice cations. It was also shown that normally the hydrolysis does not proceed to the stage of hydrous ferric oxide formation.

Soluble iron leached through a soil may be precipitated by adsorption on clay materials or possibly by an increase in pH (possibly at lower depths) which will reduce the positive charge acquired by the colloidal particles. In the pH range of 5 to 8, ferric ion cannot remain in solution, but only in suspension or as complexes. For example, the forms of iron found in natural groundwaters in one investigation were $(Fe(H_2O)_6)^{+3}$, $(Fe(H_2O)_5OH)^{+2}$, $(Fe(H_2O)_5(OH))^{+}$, $(Fe(H_2O)_4(OH)_2)^{+}$ and $(Fe(H_2O)_6)^{+2}$. Hydrous ferric oxide may be present in waters of pH greater than 7.

The solubilities of the oxides of iron in the +3 state are low and thus movement is not as common as in the +2 state. Reduction of ferric ion can be afforded by the products from the decomposition of organic materials. A more important factor in the mobilization of iron, however, is the formation of organic complexes or chelates. For example, leachates of leaves and forest litter are active in the mobilization of iron. Iron complexes by fulvic acids is mobile whereas iron complexed by humic acids is insoluble. (Hesse 1972). The movement of ferric ion from a ferric chloride spill may be a complex phenomenon; no attempt to model this potential process is afforded in this section. It is expected that some iron will remain in a mobile form for transport downward to the groundwater table. Upon reaching the groundwater table, the contaminant will continue to move, now in the direction of groundwater flow. A contaminated plume will be produced, with dilution and dispersion serving to reduce the concentrations. This is shown schematically in Figure 18.

5.5.2 Equations Describing Ferric Chloride Movement into Soil. The equations and assumptions used to describe contaminant movement downward through the unsaturated soil zone toward the groundwater table have been described in the Introduction Manual. Transport velocities have been based on Darcy's Law assuming saturated piston flow.

5.5.3 Saturated Hydraulic Conductivity of Ferric Chloride in Soil. The saturated hydraulic conductivity (K_o), in m/s, is given by:

$$K_o = \frac{(\rho g)k}{\mu}$$

where: k = intrinsic permeability of the soil (m^2)

ρ = mass density of the fluid (kg/m3)

μ = absolute viscosity of the fluid (Pa•s)

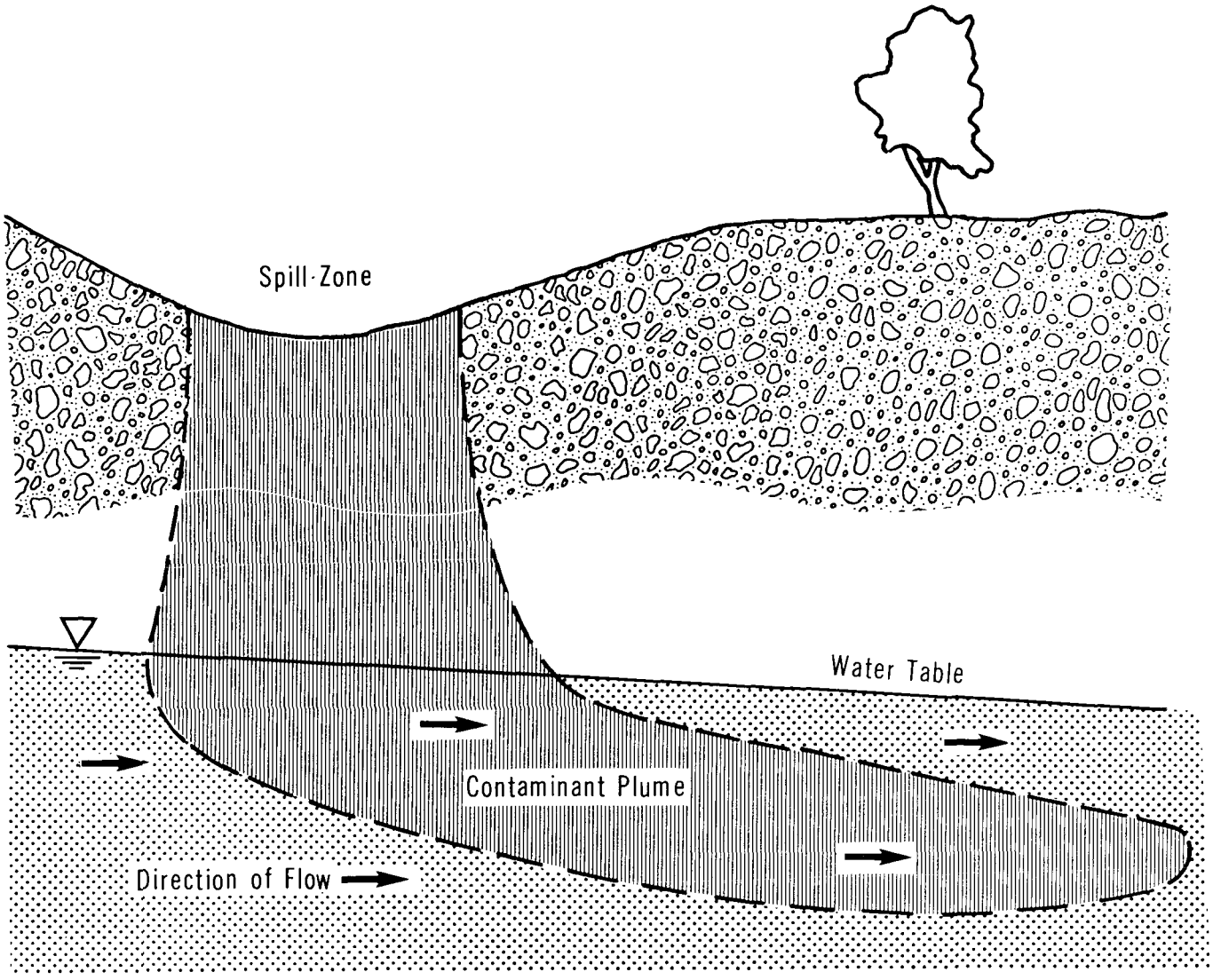
g = acceleration due to gravity = 9.81 m/s^2

The fluids involved are 46 and 14.5 percent by weight ferric chloride, and water. The water calculations represent the extreme as ferric chloride is diluted.

FIGURE 18

FERRIC CHLORIDE

SCHEMATIC SOIL TRANSPORT



Soil: Coarse Sand

–Porosity (n) = 0.35

–Intrinsic Permeability (k) = 10^{-9} m^2

–Field Capacity (θ_{fc}) = 0.075

Property	Ferric Chloride 46 percent by Wt. 20°C	Ferric Chloride 46 percent by Wt. 4°C	Ferric Chloride 14.5 percent by Wt. 20°C	Ferric Chloride 14.5 percent by Wt. 4°C	Water 20°C
Mass density (ρ), kg/m^3	1485	1500	1130	1140	998
Absolute viscosity (μ), Pa•s	15.4×10^{-3}	10.6×10^{-3}	1.93×10^{-3}	1.88×10^{-3}	1.0×10^{-3}
Saturated hydraulic conductivity (Ko), m/s	$(0.1 \times 10^7)k$	$(0.14 \times 10^7)k$	$(0.57 \times 10^7)k$	$(0.6 \times 10^7)k$	$(0.98 \times 10^7)k$

5.5.4 Soils. The Introduction Manual describes the three soils selected for this work. Their relevant properties are:

Property	Soil Type: Coarse Sand	Silty Sand	Clay Till
Porosity (n), m^3/m^3	0.35	0.45	0.55
Intrinsic permeability (k), m^2	10^{-9}	10^{-12}	10^{-15}
Field capacity (Θ_{fc}), m^3/m^3	0.075	0.3	0.45

5.5.5 Penetration Nomograms. Nomograms for the penetration of ferric chloride into the unsaturated zone above the groundwater table were prepared for each soil. They present penetration time (t_p) plotted against depth of penetration (B). Because of the methods and assumptions used, the penetration depth should be considered as a maximum depth in time t_p. A flowchart for the use of the nomogram is presented in Figure 19. The nomograms are presented as Figures 20, 21 and 22.

The water line on the nomograms represents the maximum penetration of water at 20°C in time t_p. It is a limiting condition as ferric chloride becomes diluted with water.

5.5.6 Sample Calculation. A 20 tonne spill of 46 percent by weight ferric chloride solution has occurred on coarse sand. The temperature is 20°C; the spill radius is 8.6 m. Calculate the depth of penetration 80 minutes after the spill.

FIGURE 19

FERRIC CHLORIDE

FLOWCHART FOR NOMOGRAM USE

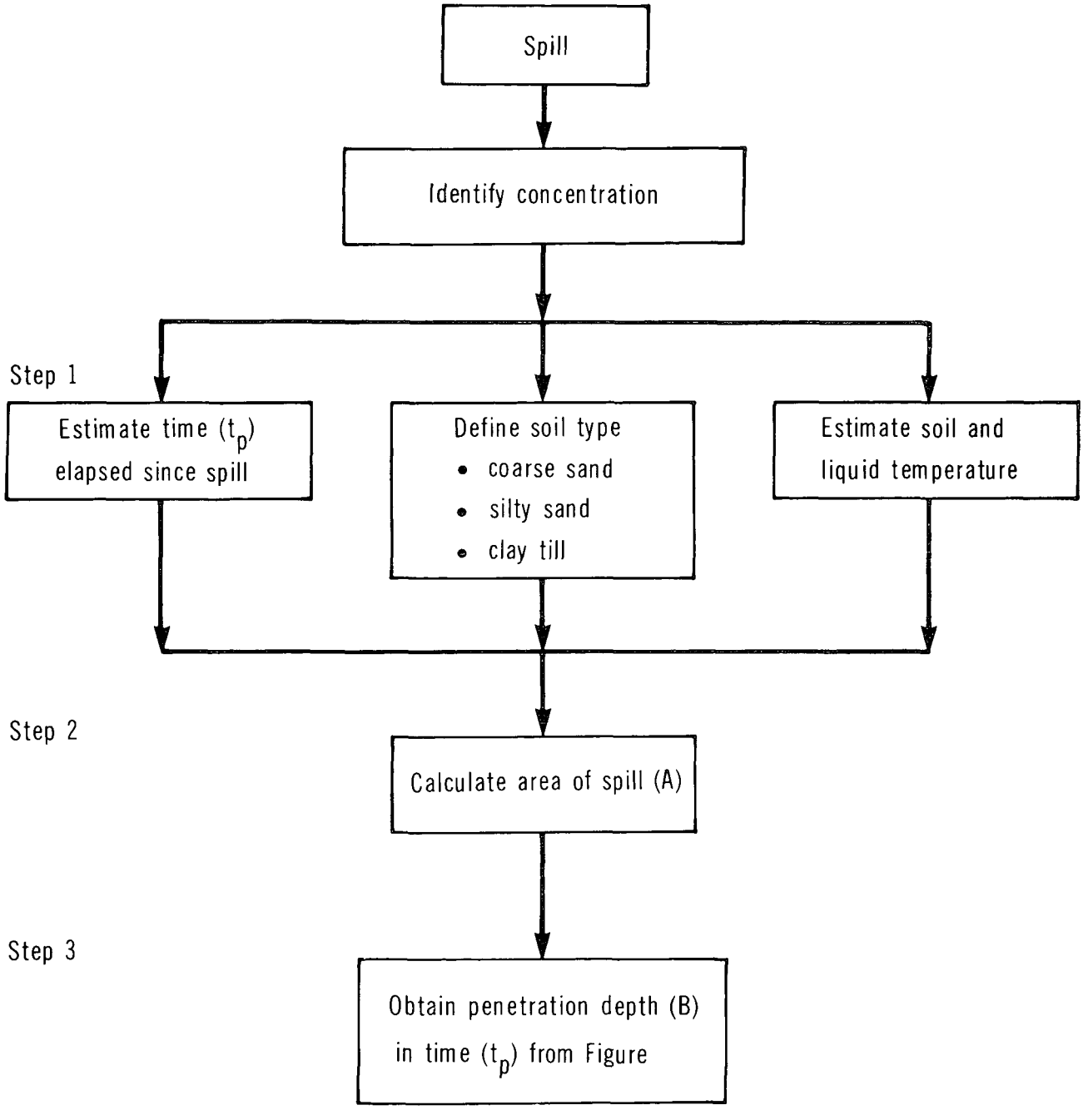


FIGURE 20

FERRIC CHLORIDE

PENETRATION IN COARSE SAND

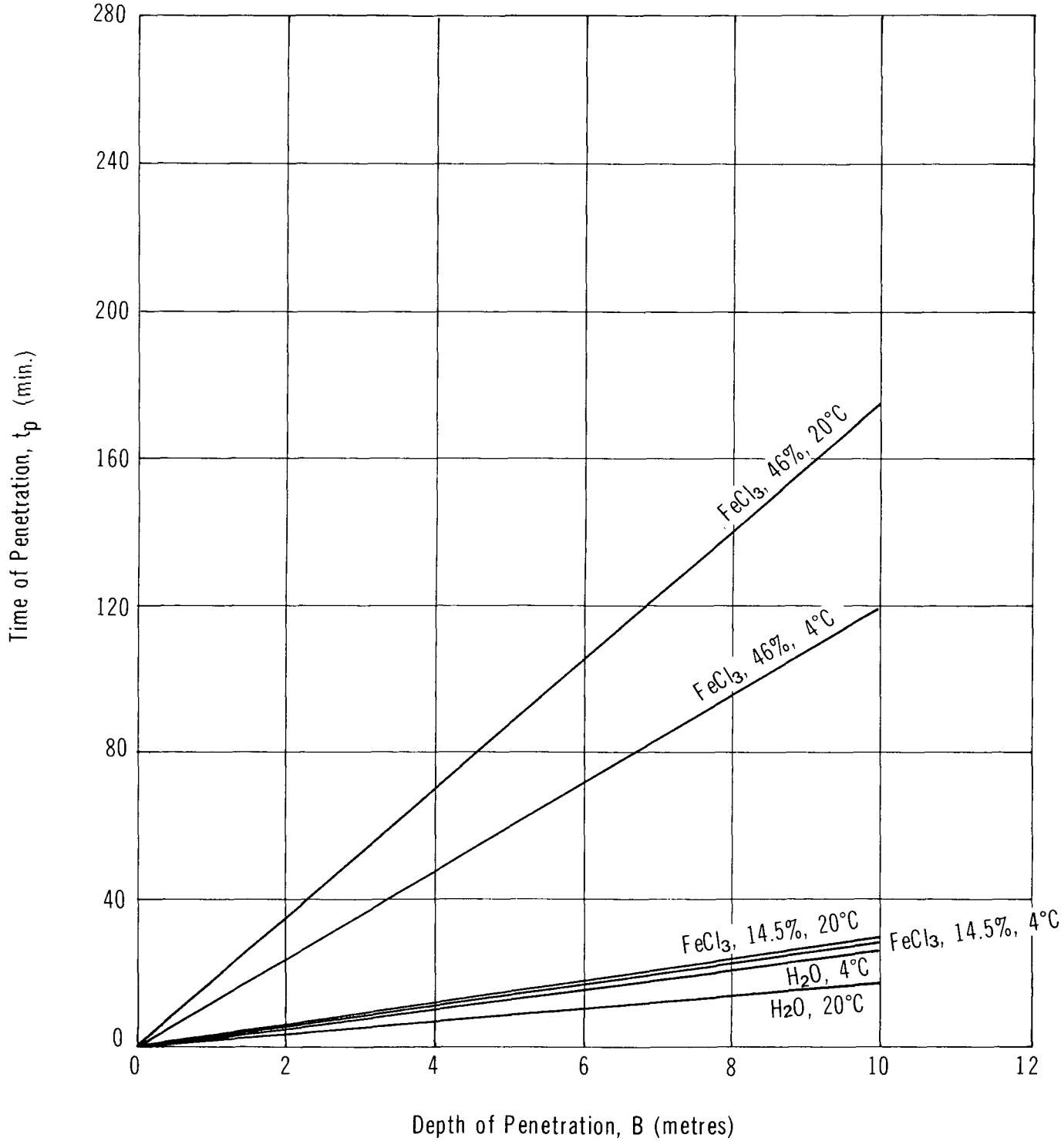


FIGURE 21

FERRIC CHLORIDE

PENETRATION IN SILTY SAND

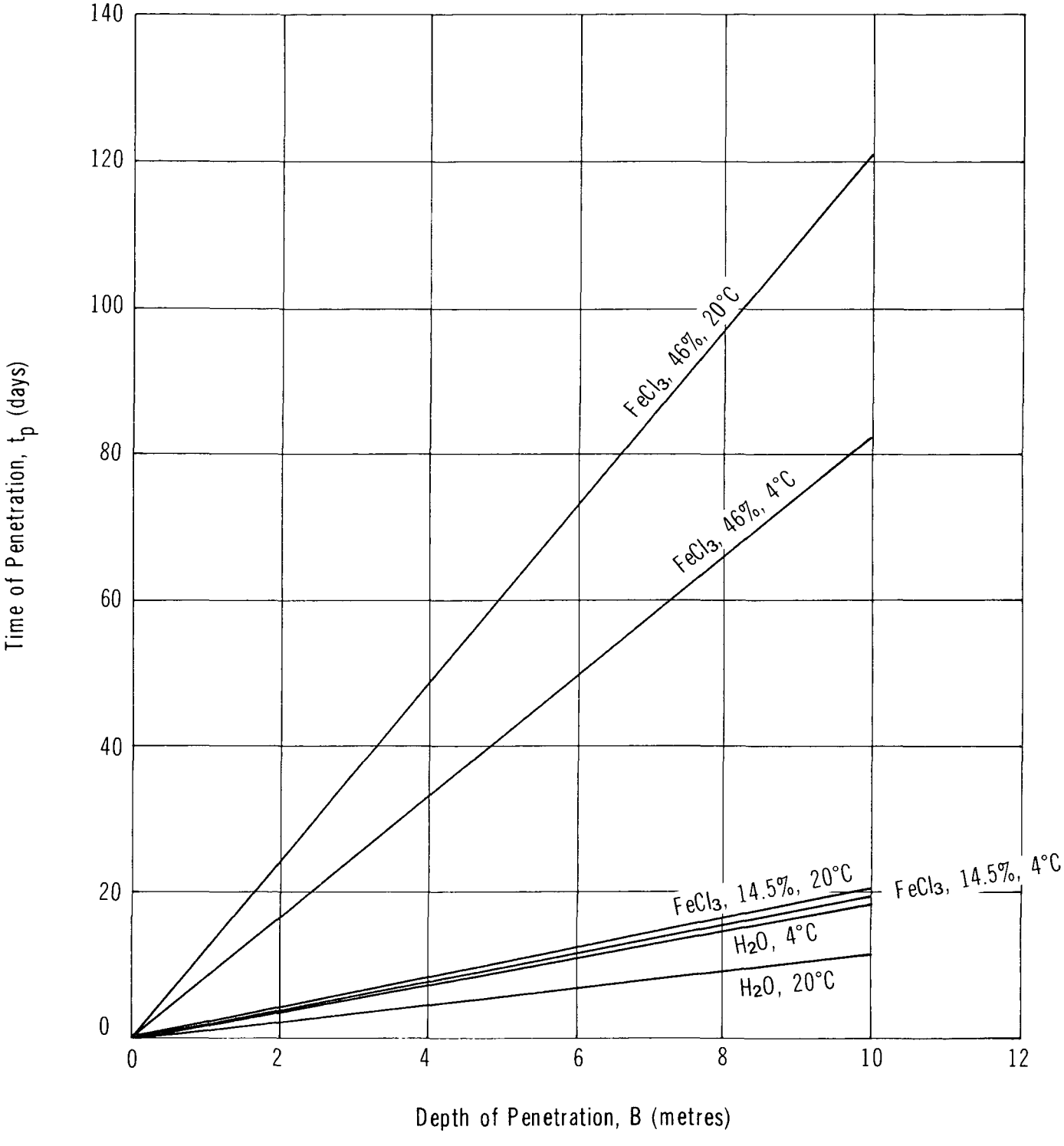
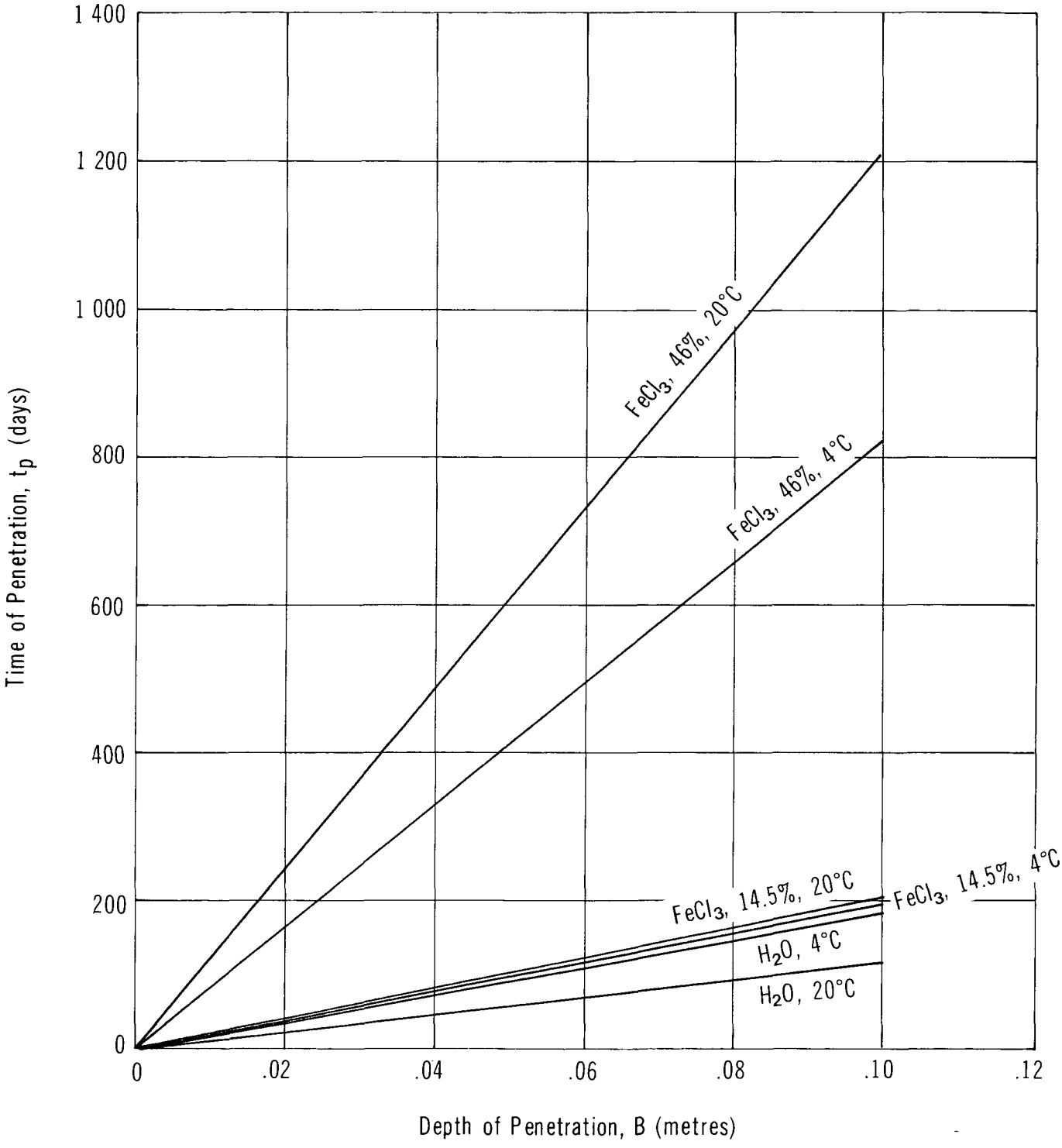


FIGURE 22

FERRIC CHLORIDE

PENETRATION IN CLAY TILL



Solution

Step 1: Define parameters

- Mass spilled = 20 000 kg (20 tonnes)
- T = 20°C
- r = 8.6 m
- Soil = coarse sand
- Groundwater table depth (d) = 13 m
- Time since spill (t_p) = 80 min

Step 2: Calculate area of spill

- $A = \pi r^2 = 232\ m^2$

Step 3: Estimate depth of penetration (B) at time (t_p)

- For coarse sand, B = 4.6 m at t_p = 80 min
- Groundwater table has not been reached in this time

6 ENVIRONMENTAL DATA

6.1 Suggested or Regulated Limits

6.1.1 Water. In Canada, the maximum acceptable concentration of iron in water is 0.3 mg/L. The objective is less than 0.05 mg/L, based on esthetic considerations (Guidelines/Canadian/Water 1978; Water Management Goals 1978).

In the United States, both drinking water and livestock watering limits of 0.3 ppm iron have been proposed, with chloride limits much higher (350 ppm for irrigation and 1500 ppm for livestock) (WQC 1971).

6.1.2 Air. Although no specific limit for ferric chloride is given, Ontario limits ferric oxide concentration to 75 $\mu g/m^3$ of air (Ontario E.P. Act 1971).

6.2 Aquatic Toxicity

6.2.1 U.S. Toxicity Rating. No TL_m96 has been assigned to ferric chloride (RTECS 1979).

6.2.2 Measured Toxicities.

6.2.2.1 Freshwater toxicity.

Conc. (mg/L)	Time (hours)	Species	Result	Water Conditions	Reference
Fish Kill Data					
0.6	not stated	Goldfish	harmful	tap	OHM-TADS 1981
5 to 10	96	Goldfish	lethal	laboratory bioassay	WQCDB-5 1973
4.35	72	Goldfish	harmful	tap	WQC 1963; OHM-TADS 1981
9	20	Goldfish	harmful	distilled	OHM-TADS 1981
20	144	Goldfish	harmful	tap	WQC 1963; OHM-TADS 1981
100 ($FeCl_3$)	96	Goldfish	safe	hard, pH 5.5	WQC 1963; OHM-TADS 1981; Doudoroff 1953
100 ($FeCl_3$)	80 min	Goldfish	fatal(*)	soft, pH 3.4	Doudoroff 1953
100	1.5	Goldfish	TL_m	soft	Doudoroff 1953

Conc. (mg/L)	Time (hours)	Species	Result	Water Conditions	Reference
10 ($FeCl_3$)	96	Goldfish	safe	very soft, pH 5.0	Doudoroff 1953
0.0000166 N	1200 min	Crucian carp	survived	-	WQC 1971
0.284 N	29 min	Crucian carp	survived	-	WQC 1971
0.9 (as Fe)	not stated	Carp	toxic	pH 5.5	QCW 1976
1 to 2 (as Fe)	not stated	Pike, trout	lethal	-	QCW 1976
0.16 N (as $FeCl_3$)	47 min (59)**	Minnow (*Leucisus phoxinus*)	survived	-	Carpenter 1927
0.125 N (as $FeCl_3$)	50 min (63)	Minnow (*Leucisus phoxinus*)	survived	-	Carpenter 1927
0.10 N (as $FeCl_3$)	54 min (68)	Minnow (*Leucisus phoxinus*)	survived	-	Carpenter 1927
0.08 N (as $FeCl_3$)	67 min (79.5)	Minnow (*Leucisus phoxinus*)	survived	-	Carpenter 1927
0.05 N (as $FeCl_3$)	68.5 min (103.5)	Minnow (*Leucisus phoxinus*)	survived	-	Carpenter 1927
0.033 N (as $FeCl_3$)	77 min (111.5)	Minnow (*Leucisus phoxinus*)	survived	-	Carpenter 1927
0.01 N (as $FeCl_3$)	86 min (144)	Minnow (*Leucisus phoxinus*)	survived	-	Carpenter 1927
0.005 N (as $FeCl_3$)	89.5 min (187)	Minnow (*Leucisus phoxinus*)	survived	-	Carpenter 1927
$3x10^{-5}$ M	>50	*Orizias latipes*	highest conc. tolerated	-	Doudoroff 1953
1 ppm $FeCl_3$ (as Fe)	10 d	Sticklebacks (*Gasterosteus aculeatus*)	survived	pH 5.0	Doudoroff 1953
1.5 ppm $FeCl_3$ (as Fe)	3 d	Sticklebacks (*Gasterosteus aculeatus*)	survived	pH 4.6	Doudoroff 1953
2.0 ppm $FeCl_3$ (as Fe)	2 d	Sticklebacks (*Gasterosteus aculeatus*)	survived	pH 4.4	Doudoroff 1953

Conc. (mg/L)	Time (hours)	Species	Result	Water Conditions	Reference
3 ppm $FeCl_3$ (as Fe)	1 d	Sticklebacks (*Gasterosteus aculeatus*)	survived	pH 4.2	Doudoroff 1953
7 ppm $FeCl_3$ (as Fe)	6	Sticklebacks (*Gasterosteus aculeatus*)	survived	pH 3.8	Doudoroff 1953
20 ppm $FeCl_3$ (as Fe)	3	Sticklebacks (*Gasterosteus aculeatus*)	survived	pH 3.4	Doudoroff 1953
5 to 10	96	Goldfish	lethal	lab	WQCDB-5 1973
0.6	not stated	Goldfish	harmful	bioassay tap	OHM-TADS 1981
0.9 (as Fe)	not stated	Carp	toxic	pH 5.5	QCW 1976
1 to 2 (as Fe)	not stated	Pike and trout	lethal	-	QCW 1976
<u>Fish Toxicity Tests</u>					
1	240	Stickleback	safe	tap	OHM-TADS 1981
1.2	144	Stickleback	harmful	tap	CHRIS 1978
6 (as Fe)	24 to 96	Striped bass fingerlings	LC_{50}	static	Hughes 1973
8 (as Fe)	24 to 96	Striped bass fingerlings	LC_{100}	static	OHM-TADS 1981
4 (as Fe)	24 to 96	Striped bass larvae	LC_{50}	static	Hughes 1973
6 (as Fe)	48 to 96	Striped bass larvae	LC_{100}	static	OHM-TADS 1981
8 (as Fe)	24	Striped bass larvae	LC_{100}	static	OHM-TADS 1981
74	96	Mosquitofish	TL_m	turbid	WQC 1963
270	1.5	Minnow	safe	-	OHM-TADS 1981
540	1.5	Minnow	TL_m	-	WQC 1963
2700	1	Minnow	TL_m	-	OHM-TADS 1981
5400	1	Minnow	TL_m	soft	WQC 1963

Conc. (mg/L)	Time (hours)	Species	Result	Water Conditions	Reference
3 (FeO(OH))	not stated	Coho salmon	no effect on hatchability, embryonic development or survivability	static	EPA 600/J-79-073
Microorganisms					
36 ($FeCl_3$)	25	*Daphnia magna*	TL_m	standard reference water and lake	Dowden 1965
21 ($FeCl_3$)	50	*Daphnia magna*	TL_m	standard reference water and lake	Dowden 1965
15 ($FeCl_3$)	100	*Daphnia magna* (young)	TL_m	standard reference water and lake	Dowden 1965
116	not stated	*Cyclops vernalis*	threshold concentration	Lake Erie, 20°C	WQC 1963
<18	64	*Daphnia magna*	threshold limit for immobilization		WQC 1963
130	theoretically infinite	*Daphnia magna*	threshold limit for immobilization	centrifuged Lake Erie water	WQC 1971
Invertebrates					
200	24	Vector snails (both *Biomorpholaria alexandrina* and *Bulinus truncatus*	killed	26°C	Gohar 1961

* Hydrochloric acid in hard water at pH 3.5 is almost as rapidly fatal.
** Values in brackets are for $FeSO_4$ under same conditions.

6.2.3 Aquatic Studies. Iron salts were found to be most harmful to goldfish immediately after dissolving in water. Gill blockage (reversible), absorption in the digestive system, and iron precipitation in the epithelium and renal tubules occur (WQCDB-5 1973). Toxicity appears to be a function of several other parameters: hardness, pH, temperature and buffering, but principally acidity (EPA 440/9-75-009).

Ferric chloride is toxic to threespine stickleback in a manner similar to that of hydrochloric acid solutions of the same pH. This suggests that trivalent iron has a very low toxicity. The divalent species, on the other hand, can cause problems as it acts as a reducing agent; when oxidized, it forms hydrous ferric oxide which precipitates as a red-brown slime. It was also shown that the toxicity of ferric ion to eels and the fish *Orizias latipes* is similar to that of the metals tin, aluminum and nickel, is greater than that of ferrous ion, and is less than that of the metals mercury, copper, zinc and cadmium.

6.3 Toxicity to Other Biota

6.3.1 Livestock. An acute toxicity ranking of 500 to 999 mg/kg has been assigned to ferric chloride by determination of the LD_{50} in mammals (WQCDB-2 1971). The chronic animal toxicity limit is 1500 ppm (chloride threshold) (OHM-TADS 1981).

6.3.2 Plants. Iron available from plants exists as the ferrous ion (Fe^{+2}) and as the complex ion $((Fe(OH)_n)^{(3-n)+}$. The availability of iron, which exists in more than one valence state, is affected by the state of oxidation of the soil. Iron toxicity has been known to occur when soils are flooded, a condition which might place more iron in a condition that is available for plant uptake (Hesse 1972).

Iron in irrigation waters, however, is not likely to cause plant toxicity problems because it is not sufficiently soluble in aerated soils at pH levels where plants grow well (WQC 1972). Chloride is the limiting factor. The chronic plant toxicity limit is 1000 ppm (as chloride) (OHM-TADS 1981).

6.3.3 Insects.

Conc. (mg/L)	Time (days)	Species	Results	Water Conditions	Reference
16.0 ($FeCl_3$)	7	Caddisfly	50% survived	pH 8.1, hardness 50*	Warnick 1969
0.32 ($FeCl_3$)	96 h	Mayfly	TL_m	pH 8.2, hardness 48*	Warnick 1969

Conc. (mg/L)	Time (days)	Species	Results	Water Conditions	Reference
16.0 ($FeCl_3$)	9	Stonefly	50% survived	pH 7.7, hardness 48*	Warnick 1969

* DO = 8.0 mg/L in all cases

6.4 Degradation

6.4.1 Chemical Degradation. Ferric chloride does not biodegrade. In water treatment plants, it will contribute to sludge volume accumulation. Inhibition of waste water sludge digestion has occurred at 500 mg/L, with total inhibition above 1000 mg/L (Ghosh 1974). Greater than 100 mg/L ferric ion (Fe^{+3}) caused significant inhibition of oxygen uptake (degraded ability of bacteria to utilize oxygen) (PB 276724).

Gasification in a sewage treatment process was not affected by $FeCl_3$ at <5 ppm but was increasingly retarded by increasing the concentration of $FeCl_3$ up to 20 ppm. This was attributed partly to the acidity caused by hydrolysis of the ferric ion. It was determined that the numbers of bacteria in fresh solids treated with 10 ppm $FeCl_3$ were greatly reduced. On the other hand, it was noted that the rate and quantity of oxygen absorbed by fresh solids was independent of the quantity of iron compounds present. It was also shown that treating fresh solids with $FeCl_3$ increased the rate of digestion, but lowered the volume of gas produced (Rudolfs 1950).

6.5 Long-term Fate and Effects

Ferric chloride forms an acid solution, ultimately precipitating the red-brown, gelatinous hydrous oxide (Fe(OH)) on dilution (Cotton 1972; Bailar 1973). Phosphates are also effectively removed as insoluble iron salts (Campbell 1982). An iron-rich sludge on the bottom may result in continuous release of small amounts of iron, capable of producing toxic effects with chronic exposure (EPA 440/9-75-009). Marine plants concentrate iron by a factor of 50 000; marine invertebrates by 20 000; marine fish by 3000; freshwater plants by 5000, invertebrates by 3200; and fish by 300 (Chapman 1968).

7 HUMAN HEALTH

There is a limited amount of information in the published literature concerning the toxicological effects of test animal and human exposures to ferric chloride. Much of the published information pertaining to the health effects of this chemical deals with its irritant effects on the eyes and skin, and the consequences of ingestion. No information was encountered in the literature on the effects of ferric chloride on reproduction, nor its mutagenicity or carcinogenicity. Little information is available on the effects of chronic exposures to this chemical.

Ferric chloride was reported in the EPA TSCA inventory in July 1979. The data summarized here are representative of information found in the literature.

7.1 Recommended Exposure Limits

The exposure standards for ferric chloride are based upon its irritant properties. Canadian provincial guidelines generally are similar to those of USA-ACGIH unless indicated otherwise.

Guideline (Time)	Origin	Recommended Level	Reference
Time-weighted Averages (TWA)			
TLV® (8 h)	USA-ACGIH	1 mg/m^3 (iron salts, soluble, as Fe)	TLV 1983
Short-term Exposure Limits (STEL)			
STEL (15 min)	USA-ACGIH	2 mg/m^3 (iron salts, soluble, as Fe)	TLV 1983

7.2 Irritation Data

7.2.1 Skin Contact.

Exposure Level (and Duration)	Effects	Reference
SPECIES: Human		
-	Solid will burn skin	CHRIS 1978

Exposure Level (and Duration)	Effects	Reference
Concentration equivalent to 42° Bé	Prolonged contact with ferric chloride solution will cause skin staining and may cause dermatitis similar to that caused by weak acids	GE 1979
Direct contact	Anhydrous form is an irritant to skin and tissue	Hawley 1977; Merck 1983

7.2.2 Eye Contact.

Exposure Level (and Duration)	Effects	Reference
SPECIES: Human		
-	Dust is irritating to eyes. Solid will burn eyes	CHRIS 1978

7.3 Threshold Perception Properties

7.3.1 Odour. Odour Characteristics: Solution in water has slight acid odour (specifically of hydrochloric acid) (GE 1979).

7.3.2 Taste. Taste Characteristics: Iron taste (AAR 1981).

Parameter	Media	Concentration	Reference
Median Taste Threshold	distilled water	3.4 ppm	QCW 1976
Readily Detectable	spring water	1.8 mg/L	QCW 1976

7.4 Long-term Studies

7.4.1 Inhalation.

Very few data were found with regard to the inhalation of iron compounds, in general, and none regarding ferric chloride, specifically. It was stated that only ferric oxide, Fe_2O_3, has any industrial risk with very long term chronic inhalation leading to

siderosis (a pneumoconiosis due to the inhalation of iron or other metallic particles) (Patty 1981; Kirk-Othmer 1981). It is not probable that a spill situation involving anhydrous ferric chloride could set up a situation leading to chronic exposure to airborne particles. The hygroscopic nature of the material would lead to the hydrate formation and clumping. Acute exposure to airborne ferric chloride, anhydrous or hydrated, would more than likely produce symptoms similar to those expected from hydrochloric acid aerosol due to hydrolysis while in solution. Since only limited data were found regarding the toxicity of ferric chloride, data are also included in this section for other compounds of ferric ion (Fe^{+3}) as well as for the ferrous ion (Fe^{+2}) and reduced iron powder. It is felt, in most cases, that the cation is of primary importance and the anions of lesser importance. Ferrous compounds are included for comparative purposes.

Iron salts in both the +2 and +3 oxidation states are not exceptionally toxic when ingested. Humans appear to suffer no harmful effects from drinking natural waters containing iron. It is known that iron in the +2 oxidation state (ferrous) is generally absorbed from the gastrointestinal tract more readily than iron in the +3 state (ferric), presumably because of its greater solubility. A number of factors, including acidity of the gastric juice, composition of the diet (high phosphate and phytic acid reduce absorption), vitamin B_6, calcium, copper, and others, appear to influence absorption of iron (Patty 1981; Water Analysis 1982).

When iron salts are introduced directly into the bloodstream, they are highly and instantaneously toxic, particularly the ferric salts. This can be verified by reviewing the intraperitoneal and intravenous data shown below, and comparing them to ingestion data (Patty 1981).

The immediate cause of death from inorganic iron compounds in animals is respiratory failure. Preceding death, the clinical signs are anorexia, oligodipsia, oliguria, alkalosis, diarrhea, loss of body weight, hypothermia, and alternating irritability and depression. Autopsy reveals loss of weight in most organs, accompanied by dehydration when death occurred early and by edema when death was delayed. The dominant histopathological sign is vascular congestion of the gastrointestinal tract, liver, kidneys, heart, lungs, brain, spleen, adrenals and hymus gland. Toxic signs begin to disappear in survivors towards the end of 1 week and completely disappear after 2-4 weeks (Patty 1981).

7.4.2 Ingestion.

Exposure Level (and Duration)	Effects	Reference
SPECIES: Human		
60 mg/kg ($FeSO_4$), adult, female	TD_{LO} (affects primarily the CNS)	Patty 1981
200-250 mg/kg (as Fe)	Lethal dose	Kirk-Othmer 1981
600 mg/kg ($FeSO_4$), infant	TD_{LO} (affects gastrointestinal tract)	Patty 1981
30 g	Estimated fatal dose	TDB (on-line) 1981
390 mg/kg ($FeSO_4$), child	LD_{LO}	Kirk-Othmer 1981
200 mg/kg ($FeSO_4$), woman	LD_{LO}	Kirk-Othmer 1981
SPECIES: Rabbit		
890 mg/kg ($FeCl_2 \cdot 4H_2O$)	LD_{LO}	Kirk-Othmer 1981
7 mg/kg	LD_{LO}	TDB (on-line) 1981
SPECIES: Rat		
98.6 g/kg (reduced Fe powder)	LD_{50}	Patty 1981
5 to 0.5 g/kg	LD_{50}	CHRIS 1978
3250 mg/kg ($Fe(NO_3)_3 \cdot 9H_2O$)	LD_{50}	Patty 1981
1480 mg/kg ($FeSO_4$)	LD_{50}	Patty 1981
900 mg/kg (anhydrous $FeCl_3$)	LD_{50} (from older literature)	Patty 1981
900 mg/kg ($FeCl_3 \cdot 6H_2O$)	LD_{LO}	OHM-TADS 1981
984 mg/kg ($FeCl_2 \cdot 4H_2O$)	LD_{50}	Kirk-Othmer 1981
319 mg/kg ($FeSO_4$)	LD_{50}	Kirk-Othmer 1981
1989 mg/kg ($FeSO_4 \cdot 7H_2O$)	LD_{LO}	Kirk-Othmer 1981
SPECIES: Dog		
200 to 800 mg (anhydrous $FeCl_2$)	no effect	Patty 1981
1550 mg/kg (ferrocene, $(C_5H_5)_2Fe$)	LD_{50}	Patty 1981
1520 mg/kg ($FeSO_4 \cdot 7H_2O$)	LD_{50}	Kirk-Othmer 1981

Exposure Level (and Duration)	Effects	Reference
1278 mg/kg (only $FeCl_3$)	LD_{50}	RTECS 1979
979 mg/kg ($FeSO_4$)	LD_{50}	Kirk-Othmer 1981
540 mg/kg	LD_{50}	TDB (on-line) 1981
440 mg/kg (anhydrous $FeCl_3$)	LD_{50} (from older literature)	Patty 1981
400 mg/kg (anhydrous $FeCl_3$)	LD_{50}	Patty 1981
SPECIES: Mouse		

7.4.3 Intraperitoneal.

Exposure Level (and Duration)	Effects	Reference
SPECIES: Mouse		
335 mg/kg (ferrocene, $(C_2H_5)_2Fe$)	LD_{50}	Patty 1981
260 mg/kg ($FeCl_3 \cdot 6H_2O$)	LD_{50}	Patty 1981
100 mg/kg ($FeSO_4$)	LD_{50}	Patty 1981
68 mg/kg (anhydrous $FeCl_3$)	LD_{50}	Patty 1981

7.4.4 Intravenous.

Exposure Level (and Duration)	Effects	Reference
SPECIES: Rabbit		
7.2 mg/kg	LD_{50}	AAR 1981; OHM-TADS 1981
7 mg/kg (anhydrous $FeCl_3$)	LD_{LO}	RTECS 1979
7 mg/kg ($FeCl_3$)	LD_{LO} (from older literature)	Patty 1981

Exposure Level (and Duration)	Effects	Reference
SPECIES: Mouse		
142 mg/kg (anhydrous $FeCl_3$)	LD_{50}	RTECS 1979
49 mg/kg ($FeCl_3$)	LD_{50}	AAR 1981; OHM-TADS 1981

7.4.5 Subcutaneous.

Exposure Level (and Duration)	Effects	Reference
SPECIES: Mouse		
104 000 mg/kg, 13 wk (Fe-dextran)	TD_{LO}	Patty 1981
4200 mg/kg, 21 wk (ferrous lactate)	TD_{LO}	Patty 1981
2000 mg/kg, 13 wk (ferrous gluconate)	TD_{LO}	Patty 1981
1600 mg/kg, 16 wk ($FeSO_4$)	TD_{LO}	Patty 1981

7.4.6 Carcinogenicity, Teratogenicity, Mutagenicity (Patty 1981). Iron and its inorganic compounds were screened by subcutaneous injection for tumorigenicity. With the exception of one fibroma from ferrous sulphate, neither iron nor any of its inorganic compounds, including hematite ($FeO(OH)\cdot xH_2O$), produced any tumours. Ferric oxide was given by inhalation or by intratracheal route to mice, hamsters and guinea pigs and was not found to be tumorigenic. Among the organic derivatives, ferrocene ($Fe(C_5H_5)_2$), ferritin (iron storage protein), ferric citrate ($FeC_6H_5O_7\cdot 3H_2O$), ferric salicylate ($Fe(C_6H_4(OH)COO)_3$) and ferric ascorbate ($Fe(C_6H_7O_6)_3$) did not produce tumours. Fe-dextran, a complex with a molecular weight of 180 000, resulted in several types of tumours at the injection site (massive doses). It was later determined that the risk of malignancy in man was low.

The condition called siderosis in man is now considered to be a benign pneumoconiosis phenomenon because it does not lead to fibrous proliferation, is of low order of severity, and usually requires 6 to 10 years of exposure before diagnosable roentgenographic changes occur. Even though iron ore miners suffer from a higher percentage of lung cancer than the norm, the carcinogenic effect could not be definitely blamed on iron oxide. No data were found pertaining to ferric chloride.

7.5 Symptoms of Exposure

General symptoms of exposure found in most information sources have not been specifically referenced. Only those of a more specific or unusual nature have their sources indicated. References from ITII (1981) pertain to effects of anhydrous ferric chloride. References from GE (1979) pertain to 42°Bé ferric chloride. References from AAR (1981) pertain to ferric chloride solution (over 50 percent).

7.5.1 Inhalation of Dust or Solution Vapour.

1. Irritation of nose and throat.
2. Tingling and burning sensation in respiratory tract
3. Coughing.
4. Difficult breathing (AAR 1981).

7.5.2 Ingestion.

1. Salivation.
2. Burning sensation in mouth and throat.
3. Burning sensation in stomach.
4. Stomach cramps.
5. Nausea and vomiting (GE 1979).
6. Diarrhea.
7. Albuminuria (ITII 1981).
8. Hematuria (ITII 1981).
9. Possible convulsion (Lefèvre 1980).
10. Gastric hemorrhage (ITII 1981).
11. Albuminuria and hematuria (ITII 1981).
12. Death.

7.5.3 Skin Contact.

1. Itching.

2. Staining of skin (GE 1979).
3. Irritation and burning sensation.
4. Dermatitis with prolonged exposure (GE 1979).
5. Ulceration and possibly profound necrosis.

7.5.4 Eye Contact.

1. Stinging and burning sensation.
2. Watering of eyes.
3. Inflammation of conjunctiva.
4. Risk of serious lesions.

7.6 Human Toxicity to Decay or Combustion Products

Under fire/extreme heat conditions, anhydrous ferric chloride may dissociate to ferrous chloride and chlorine gas:

$$2FeCl_3 \rightarrow 2FeCl_2 + Cl_2\uparrow.$$

Similar conditions may hydrolyze ferric chloride solutions to the hydrous ferric oxide, with the simultaneous evolution of hydrogen chloride fumes (Cotton 1972):

$$(Fe(H_2O)_6)^{+3} + 3Cl^- \rightarrow (Fe(H_2O)_5(OH))^{+2} + 2Cl^- + HCl\uparrow$$

Solid hydrated species may also evolve hydrogen chloride fumes upon heating (Merck 1983; Hawley 1977).

Ferrous chloride thus formed may oxidize and hydrolyze when heated in moist air, as would be the case in a fire condition. The products would be ferric oxide (Fe_2O_3) and hydrogen chloride (HCl) (Merck 1983).

7.6.1 Hydrogen Chloride, Ferrous Chloride and Chlorine. Hydrogen chloride is a colourless gas with an irritating, pungent odour. It may cause skin burns, bleeding of nose and gums, and ulceration of the nasal and oral mucosa (Patty 1963). Severe exposures result in pulmonary edema and laryngeal spasm (Sax 1968). Uninterrupted work in dangerous concentrations of the gas is unlikely because of its irritating nature. The TLV® for hydrogen chloride is 5 ppm (7 mg/m^3) (8 h-TWA) (TLV 1983).

Ferrous chloride at room temperature is a white solid, possibly with a greenish tint due to the presence of hydrates; it is freely soluble in water. It is a mild irritant. There is no TLV® specific for ferrous chloride. The TLV® for iron salts, soluble, as Fe, is 1 mg/m^3 (8 h-TWA) and 2 mg/m^3 (STEL) (TLV 1983).

Chlorine is a greenish-yellow diatomic gas with a suffocating odour. This gas is a powerful irritant and can cause fatal pulmonary edema if inhaled. Other symptoms include respiratory complaints, corrosion of the teeth, inflammation of the mucous membranes of the nose, and increased susceptibility to tuberculosis in workers chronically exposed. The TLV® for chlorine is 1 ppm (3 mg/m^3) (8 h-TWA) and the STEL is 3 ppm (9 mg/m^3) (TLV 1983).

8 CHEMICAL COMPATIBILITY

8.1 Compatibility of Ferric Chloride with Other Chemicals and Chemical Groups

SPECIFIC CHEMICAL OR CHEMICAL GROUP	HEAT GENERATION	FIRE	EXPLOSION	FORMATION OF FLAMMABLE GASES	VIOLENT POLYMERIZATION	DECOMPOSITION	FORMATION OF TOXIC FUMES	FORMATION OF SUBSTANCES OF GREATER TOXICITY	PRESSURIZATION IN CLOSED VESSELS	SOLUBILIZATION	VIOLENT REACTION	NON-HAZARDOUS REACTION	SPECIFICS	REFERENCE
GENERAL														
Heat (hydrated species or solutions)						•							When heated to decomposition forms hydrochloric acid	Sax 1979; Merck 1983
Heat (anhydrous)						•							Will form Cl_2 gas and ultimately HCl	Cotton 1972
Water						•								Sax 1979
SPECIFIC CHEMICALS														
Allyl Chloride				•										NFPA 1978
Ethylene Oxide			•	•										Bretherick 1979
Potassium			•							•			Very violent explosion on impact	NFPA 1978
Sodium			•							•			Very violent explosion on impact	NFPA 1978
Sodium Hypochlorite					•			•					Forms chlorine gas	HMN 1983

8 CHEMICAL COMPATIBILITY

8.1 Compatibility of Ferric Chloride with Other Chemicals and Chemical Groups (Cont'd)

SPECIFIC CHEMICAL OR CHEMICAL GROUP	HEAT GENERATION	FIRE	EXPLOSION	FORMATION OF FLAMMABLE GASES	VIOLENT POLYMERIZATION	DECOMPOSITION	FORMATION OF TOXIC FUMES	FORMATION OF SUBSTANCES OF GREATER TOXICITY	PRESSURIZATION IN CLOSED VESSELS	SOLUBILIZATION	VIOLENT REACTION	NON-HAZARDOUS REACTION	SPECIFICS	REFERENCE
CHEMICAL GROUPS														
Metals			•								•		Primarily K and Na	Bretherick 1979
Metals (for solutions)				•									Electropositive metals will react in acidic solution to liberate hydrogen	Bailar 1973

9 COUNTERMEASURES

9.1 Recommended Handling Procedures

The following procedures have been derived from a literature review. To avoid any deviation from the intended meaning, the wording of the original source has been presented essentially unchanged - in so doing, it is recognized that there may be some discrepancies between different sources of information. It is recognized that countermeasures are dependent on the situation, and thus what may appear to be conflicting information may be in fact correct for different situations. The following procedures should not be considered as Environment Canada's recommendations.

9.1.1 Fire Concerns. Ferric chloride is a noncombustible material. Under fire conditions, however, the anhydrous material may dissociate to give ferrous chloride and chlorine. The former, when heated in moist air, may oxidize and hydrolyze, giving ferric oxide and hydrogen chloride. Solid hydrated species may also evolve hydrogen chloride fumes when heated. Ferric chloride aqueous solution, when heated, may hydrolyze to hydrous ferric oxide with simultaneous evolution of hydrogen chloride fumes (Cotton 1972; Merck 1983; Hawley 1977; GE 1977; HMIS 1981).

9.1.2 Fire Extinguishing Agents. The fire extinguishing agents generally recommended for fires where ferric chloride, anhydrous or solution, is in the immediate fire area are (AAR 1977; DOT 1980; HMIS 1981; TC 1979):

Small fires: dry chemical, CO_2, water spray or foam
Large fires: water spray, fog or foam

9.1.3 Spill Actions.

9.1.3.1 General. Do not come into contact with spilled material. Stop or reduce discharge of material if this can be done without risk.

9.1.3.2 Spills on land. When spilled in a solid form, shovel into containers for recovery or disposal (after appropriate treatment). When spilled in a solution form, contain if possible and adsorb on sand or other adsorption materials. Neutralization of the acid solution with concurrent precipitation of hydrous ferric oxide is recommended. Precipitating agents include 6 M aqueous ammonia (GE 1977) and/or sodium bicarbonate ($NaHCO_3$) (OHM-TADS 1981).

9.1.3.3 Spills in water. A spill of ferric chloride solid or solution into water can be detrimental to aquatic life by lowering the pH of the water body or by creating a floc of hydrous ferric oxide due to natural hydrolysis or addition of a neutralizing/precipitating agent as a result of a cleanup procedure. A general recommended spill action procedure is to contain the spill if possible. Since removal of soluble iron from a water body may best be afforded by precipitation of a solid, it is recommended that the water body be treated with a base to neutralize the acid and convert the iron to the relatively insoluble hydrous ferric oxide. This can be accomplished by many basic agents, but due to its buffering action, sodium bicarbonate ($NaHCO_3$) is recommended (OHM-TADS 1981). It is a buffering agent that will not readily lead to a drastic increase in pH above 7.0 if excess is added, and it is only moderately toxic to aquatic life (DOT 1976). It is imperative to note that further treatment may be required after addition of a neutralizing agent.

9.1.4 Cleanup and Treatment.

9.1.4.1 General. Recovered ferric chloride, solid or liquid, can be treated for disposal by sifting or pouring the material onto a layer of sodium bicarbonate contained in an impervious holding pond-like area or a large vessel. The solid or slurry should be mixed thoroughly and then sprayed or covered as completely as possible with 6 M aqueous ammonia. The latter should be applied until the cloud of ammonium chloride has subsided. If possible, the mixture should be cooled with iced water or crushed ice during the addition of aqueous ammonia. It is important to keep added water to a minimum to facilitate the ultimate disposal process. Any water to be returned to the environment must be at a pH level acceptable for aquatic life, usually in the 6.0-9.0 range (OHM-TADS 1981; DOT 1976).

Contaminated water, after treatment with a basic material (sodium bicarbonate, lime (CaO) or other readily accessible/acceptable material), should filtered or allowed to stand to facilitate gravity separation of the oxide floc. The pH of the water should be brought within the acceptable range before the water is released to the environment. The sludge/bottom sediment should be removed for disposal in a proper waste disposal facility (EPA 600/2-77-227).

9.1.5 Disposal. Waste ferric chloride must never be discharged directly into sewers or surface waters. Following treatment, either at the spill site or at a waste management facility, the resultant sludge can be disposed of in a secure landfill. See Sections 9.1.3.3 and 9.1.4.1 for treatment alternatives.

9.1.6 Protective Measures. For entry into a situation where the spilled material and its characteristics are unknown, self-contained breathing apparatus and a totally encapsulated chemical suit should be worn.

If the spilled material is known to be ferric chloride:

- Safety goggles and acid resistant clothing should be worn (OHM-TADS 1981; GE 1977).
- Rubber is recommended for gloves (GE 1977).
- The following chemical suit materials are recommended for protection against ferric chloride (EE-20): cloropel, PVC and neoprene (excellent resistance).
- Eye wash stations and chemical safety showers should be readily available in areas of use and spill situations (GE 1977).

9.1.7 Storage Precautions. Ferric chloride solutions are extremely corrosive to normal construction materials and will attack most metals with the exception of titanium and tantalum. Piping, containers, etc., for ferric chloride solutions are generally of glass, rubber or plastic. Floors, walls and equipment that are subject to being splashed with this solution should be protected with corrosion-resistant paint (GE 1977).

9.2 Specialized Countermeasures Equipment, Materials or Systems

The following items are taken from a previous study (Dillon 1982) and should not be considered to be the only suitable specialized countermeasures equipment, materials or systems available. More details on the specifications, performance and availability of these items can be found in the referenced study.

Treating Agents	Hazorb (Sorbent)
Neutralizing/Precipitating Agents	Sodium Bicarbonate ($NaHCO_3$)
	Lime (CaO, $CaCO_3$)
	Soda Ash (Na_2CO_3)
	Calcium Carbonate ($CaCO_3$)

10 PREVIOUS SPILL EXPERIENCE

This section contains information on previous spill experience which will be useful to readers in understanding spill response and countermeasures. Only those which meet the criteria are included, and thus, the number of experiences is not an indication of the problems or frequency of spillage. As technology in spill control advances, this section will be updated in future manual revisions to include the most useful information.

10.1 Tank Spill (ECSER 1976)

During routine yard inspection at a chemical facility, a tank containing approximately 318 000 L of ferric chloride solution began leaking its contents through a 2.5 cm hole. About 14 000 L of the solution spilled onto the ground before the chemical itself plugged the hole, 2 hours later.

The spilled material collected in three holding areas located in the yard. Response crews arrived at the site and began pumping the contained material into a specially made rubber tank car. Soda ash (Na_2CO_3) was then applied to neutralize the remaining ferric chloride and contaminated soil. The neutralized soil was disposed of at a nearby landfill site.

The incident shows that concentrated solutions of ferric chloride may solidify (through evaporation of water) and thus plug a leak. The incident also illustrates some cleanup methods.

11 ANALYTICAL METHODS

The general approach adopted for each of the Priority Chemicals was as follows.

Methods have been documented here for the analysis of samples from air, water and soil in a normally equipped chemical laboratory remote from the spill site. Customary sources of standard or recommended analytical methods were consulted, and outlines are presented for each chemical. These sources included publications of the U.S. National Institute for Occupational Safety and Health (NIOSH), the U.S. Environmental Protection Agency (EPA), the American Water Works Association (AWWA), the American Society for Testing and Materials (ASTM), and the American National Standards Institute (ANSI).

If the standard or recommended methods were judged to be reliable and specific enough for the analysis of environmental and material samples from spill sites and if they do not require highly specialized laboratory equipment, no additional methods were sought. If especially simple, reliable tests (e.g., commonly used industrial methods) were found, they have been presented as well.

11.1 Quantitative Method for the Detection of Ferric Chloride in Air

11.1.1 Atomic Absorption (NIOSH 1979). The range of concentrations for iron which may be determined is 21 to 210 $\mu g/m^3$ when a 180 L air sample is taken (1.45 to 14.5 ppm ferric chloride).

A measured volume of air, approximately 180 mL, is drawn through a cassette filter holder containing a cellulose-ester membrane filter with a 0.8 μm pore size and 37 mm diameter, at a flow rate of 1.5 L/min. The sample and a blank are transferred to clean 125 mL beakers and 6.0 mL of 10 percent (1.67 M) nitric acid are added. Each beaker is covered with a watchglass and heated on a hotplate (165°C) in a fume hood until the sample dissolves and a slightly yellow solution is produced. This will take approximately 4 hours for most air samples. However, subsequent additions of nitric acid may be needed to completely ash and destroy high concentrations of organic material and, under these conditions, longer ashing times will be needed. Once the ashing is complete, as indicated by a clear solution in the beaker, the watchglass is removed and rinsed with 10 percent nitric acid. The rinsing is placed in the sample beaker. The beakers are then placed on a 100°C steambath and allowed to go to dryness. Once the sample is dry, the walls of the beaker are rinsed with 3-5 mL 10 percent nitric acid and reheated for

5 minutes at 100°C to solubilize the residue. The sample is then quantitatively transferred to a 10 mL volumetric flask and diluted to the mark with 10 percent nitric acid. The 10 mL solution may be analyzed directly for elements of low concentrations or may be diluted accordingly for higher concentrations.

Standard solutions should match the sample matrix as closely as possible and should be run in duplicate. Working standards should be prepared fresh daily. An oxidizing flame is used with an air-acetylene gas mixture setting the wavelength at 248.3 nm. Blank filters must be analyzed as well, following the same procedure as for the samples. This procedure is a simple method using a standard Atomic Absorption instrument and is a method that may be used for many metals. Interferences of importance include high nickel concentrations and silicon.

11.2 Qualitative Method for the Detection of Ferric Chloride in Air

The sample is collected and extracted as described in Section 11.1.1.

The presence of the ferric ion may be indicated on the addition of 2-3 drops of 6 M hydrochloric acid, followed by about 1 mL of 1 M potassium thiocyanate (KCNS) solution to a 5 drop sample of solution. The formation of a deep red colour indicates the presence of ferric ion (Welcher 1955). A light pink colour may be due to a small amount of iron that has entered the test solution as an impurity from the reagents or glassware (Welcher 1955; Hogness 1954).

11.3 Quantitative Method for the Detection of Ferric Chloride in Water

11.3.1 Atomic Absorption (ASTM 1979; Water Analysis 1982). Concentrations of 0.15 to 14.5 ppm ferric chloride in water may be determined using this atomic absorption method.

The dissolved iron may be determined by aspirating the filtered sample directly into the atomic absorption spectrophotometer. A blank and at least four standard solutions which bracket the expected iron concentration in the sample are prepared. The spectrophotometer should be set at 248.3 nm and an air-acetylene flame should be used. The detection limit is 5 μg/L.

11.4 Qualitative Method for the Detection of Ferric Chloride in Water

The presence of ferric ion in the water sample may be indicated as follows. To about 5 drops of sample solution, add 2-3 drops of 6 M hydrochloric acid followed by about 1 mL of 1 M potassium thiocyanate (KCNS) solution. The presence of ferric ion is confirmed by the formation of a deep red colour. A light pink colour may be due to a

small amount of iron that has entered the test solution as an impurity from the reagents or improper cleaning of the glassware (Hogness 1954).

The presence of chloride can be confirmed by treating a representative portion (5 mL) of the collected water sample with 2-3 drops of concentrated nitric acid followed by about 5 drops of 0.1 M silver nitrate ($AgNO_3$) solution. The formation of a white precipitate indicates the presence of chloride ion. This can be confirmed by making the solution just basic with 6 M aqueous ammonia solution with stirring. The precipitate should dissolve. Acidifying with nitric acid again should cause the white solid to form again (Hogness 1954).

11.5 Quantitative Method for the Detection of Ferric Chloride in Soil

11.5.1 Spectrophotometric Method (Hesse 1972). This method is suitable for approximately 0.05 to 0.25 mg total iron in the aliquot of extract taken for analysis.

A sample containing 0.1 g of oven-dry, 0.15 mm soil, accurately weighed, is placed into a platinum crucible and moistened with a drop or two of water. Five millilitres of hydrofluoric acid and 0.5 mL 50 percent perchloric acid are added and the crucible nearly covered with its lid. The crucible is then heated on a sand-tray at 200°C until the liquid has evaporated. Any unoxidized organic matter deposited on the upper part of the crucible or its lid can be oxidized with the flame of a Meker burner.

After cooling, 5 mL of 6 M hydrochloric acid are added and the crucible filled halfway with water. The crucible is then placed on a hotplate and the contents gently boiled for 5 minutes. If this treatment does not completely dissolve the solids, the solution is evaporated to dryness and the acid digestion repeated. The solution is then transferred to a 100 mL volumetric flask and diluted to volume.

The slightly acidic iron solution is then treated with 1-2 drops of 3 percent hydrogen peroxide to oxidize to the iron III state. A 10 mL aliquot of a buffer solution is placed in a 50 mL volumetric flask and diluted to about 30 mL. The buffer solution is prepared by mixing equal volumes of 1 M acetic acid and 1 M sodium acetate and adjusting the pH to 4.7 with acetic acid or sodium hydroxide. Five millilitres of 4 percent w/v aqueous (freshly prepared) Tiron reagent (1,2-dihydroxybenzene-2,5-disulphonic acid disodium salt) and an aliquot of test solution containing about 0.05-0.25 mg of iron is added to the buffer solution and diluted to 50 mL. The absorbance is read at 565 nm after 5 minutes.

The standard curve should cover the range 0-10 μg/mL iron.

11.6 Qualitative Method for the Detection of Ferric Chloride in Soil

A small portion of freshly sampled soil is placed in a cavity of white tile and moistened with dilute (1 M) hydrochloric acid. One drop of 10 percent w/v aqueous potassium thiocyanate (KCNS) solution is added. The presence of iron (III) is indicated by a red colour (Hesse 1972).

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EnviroTIPS

Common Abbreviations

BOD	biological oxygen demand
b.p.	boiling point
CC	closed cup
cm	centimetre
CMD	count median diameter
COD	chemical oxygen demand
conc.	concentration
c.t.	critical temperature
eV	electron volt
g	gram
ha	hectare
Hg	mercury
IDLH	immediately dangerous to life and health
Imp. gal.	Imperial gallon
J	Joule
kg	kilogram
kJ	kilojoule
km	kilometre
kPa	kilopascal
kt	kilotonne
L	litre
lb.	pound
LC_{50}	lethal concentration fifty
LC_{LO}	lethal concentration low
LD_{50}	lethal dose fifty
LD_{LO}	lethal dose low
LEL	lower explosive limit
LFL	lower flammability limit
m	metre or meta
M	molar
MAC	maximum acceptable concentration
max	maximum
mg	milligram
MIC	maximum immission concentration
min	minute or minimum
mm	millimetre
μg	microgram
μm	micrometre
°Be	degrees Baumé (density)
MMAD	mass median aerodynamic diameter
MMD	mass median diameter
m.p.	melting point
MW	molecular weight
N	newton
NAS	National Academy of Sciences
NFPA	National Fire Protection Association
NIOSH	National Institute for Occupational Safety and Health
nm	nanometre
o	ortho
OC	open cup
p	para
P_c	critical pressure
PEL	permissible exposure level
pH	measure of acidity/alkalinity
ppb	parts per billion
ppm	parts per million
P_s	standard pressure
psi	pounds per square inch
s	second
STEL	short-term exposure limit
STIL	short-term inhalation limit
T_c	critical temperature
TC_{LO}	toxic concentration low
T_d	decomposition temperature
TD_{LO}	toxic dose low
TL_m	median tolerance limit
TLV	Threshold Limit Value
T_s	standard temperature
TWA	time weighted average
UEL	upper explosive limit
UFL	upper flammability limit
VMD	volume mean diameter
v/v	volume per volume
w/w	weight per weight