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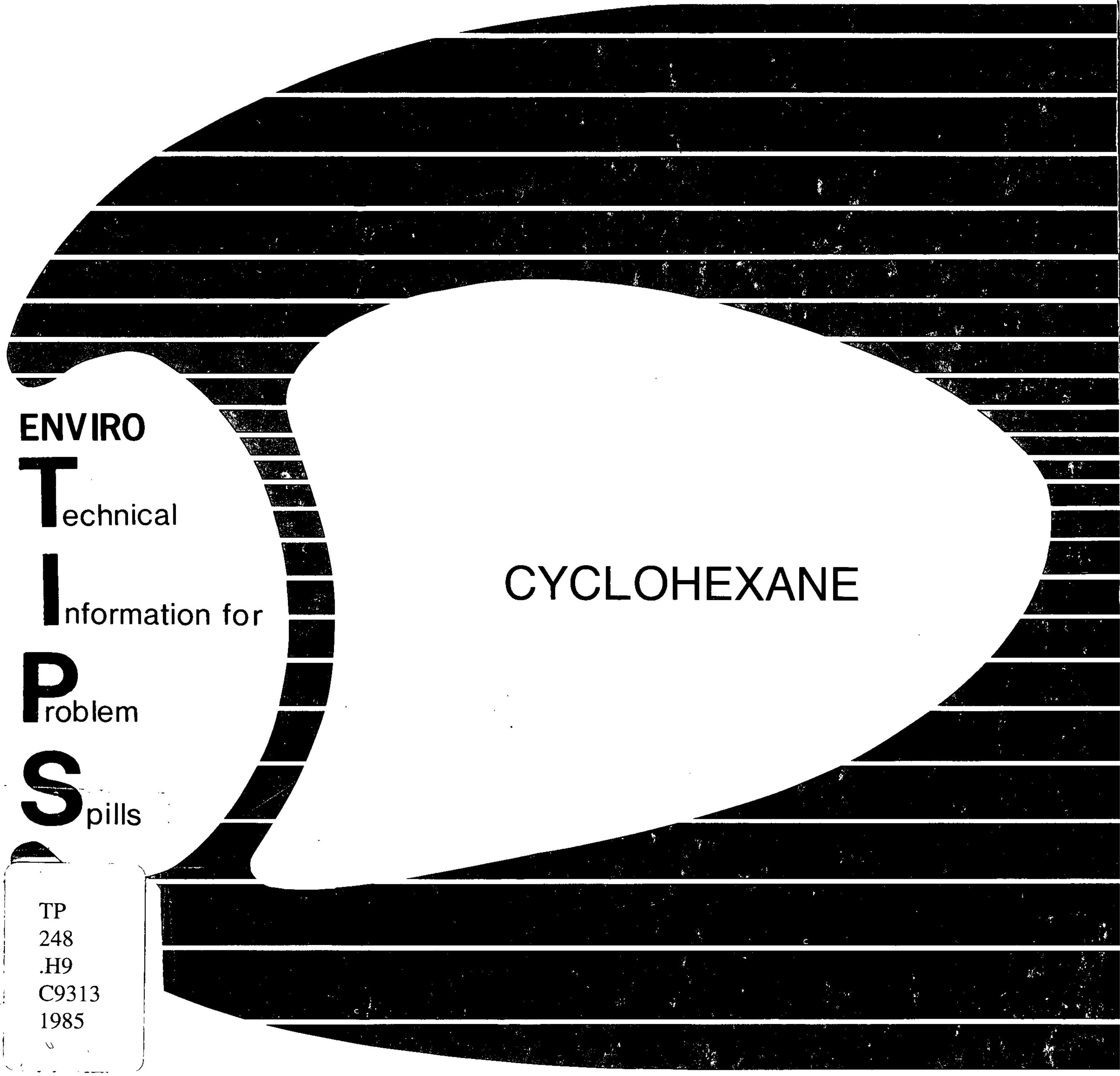

**ENVIRO**

**T**echnical
**I**nformation for
**P**roblem
**S**pills

# CYCLOHEXANE

June 1985

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## ENVIRONMENTAL AND TECHNICAL INFORMATION FOR PROBLEM SPILLS MANUALS

The 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 report has been reviewed by the Technical Services Branch, Environmental Protection Service, and approved for publication. Approval does not 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.

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Ottawa, Ontario
CANADA
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# CYCLOHEXANE

# ENVIRONMENTAL AND TECHNICAL INFORMATION FOR PROBLEM SPILLS

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## FOREWORD

The Environmental and Technical Information for Problem Spills (EnviroTIPS) 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 of and input to this manual. 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

## CYCLOHEXANE ($C_6H_{12}$)

Clear, colourless liquid with an aromatic odour.

### SYNONYMS

Hexahydrobenzene, Hexamethylene, Hexanaphthene.

### IDENTIFICATION NUMBERS

UN No. 1145; CAS No. 110-82-7; OHM-TADS No. 7216659; STCC No. 4908132

### GRADES & PURITIES

Commercial: 99.9 percent
Solvent grade: 85 percent

### IMMEDIATE CONCERNS

Fire: Highly flammable. Flashback along vapour trail may occur.

Human Health: Low toxicity by inhalation and ingestion.

Environment: Harmful to aquatic life in low concentrations and to animals in high concentrations.

### PHYSICAL PROPERTY DATA

State: (15°C, 1 atm): liquid
Boiling Point: 80.7°C
Melting Point: 6.6°C
Flammability: flammable
Flash Point: -20°C (CC)
Vapour Pressure: 16.2 kPa (30°C)

Density: 0.7785 g/mL (20°C)
Solubility (in water): insoluble
Behaviour (in water): floats with no reaction
Behaviour (in air): vapours may form explosive mixtures in air
Odour Threshold Range: 25 to 370 ppm

### ENVIRONMENTAL CONCERNS

Cyclohexane is toxic to fish in low concentrations, generally below 50 mg/L. Acute toxicity ranking for animals is over 2500 mg/kg body weight. Waterfowl are threatened by cyclohexane slicks. Cyclohexane does not have potential for bioaccumulation or food chain concentration.

### HUMAN HEALTH

TLV®: 300 ppm (1050 mg/m$^3$)

IDLH: 10 000 ppm

Exposure Effects

Inhalation: Irritating to mucous membranes, headache, nausea, vomiting, dizziness, unconsciousness, convulsions, respiratory failure and possibly death.

Contact: Irritating to skin and eyes. Dermatitis may occur following prolonged contact.

Ingestion: Causes dizziness, nausea, vomiting and possible loss of consciousness.

## IMMEDIATE ACTION

### Spill Control

Restrict access to spill site. Issue warning: "FLAMMABLE". Call fire department and notify manufacturer. Eliminate sources of ignition including traffic and equipment. Stop the flow and contain spill, if safe to do so. Avoid contact with liquid and vapour; stay upwind of release. Keep contaminated water from entering sewers or watercourses.

### Fire Control

Use alcohol foam, dry chemical or carbon dioxide to extinguish. Water may be ineffective as it may spread material. Cool fire-exposed containers with water. Stay clear of tank ends. Containers may explode in heat of fire.

## COUNTERMEASURES

### Emergency Control Procedures in/on

Soil: Construct barriers to contain spill or divert to impermeable holding area. Remove material with pumps or vacuum equipment. Adsorb small amounts of spill with natural or synthetic sorbents and shovel into containers with covers.

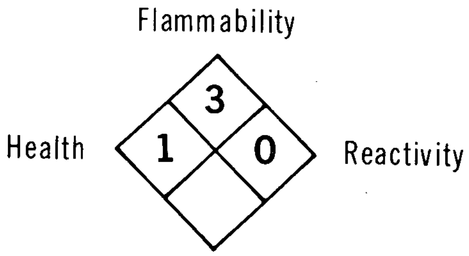
Water: Contain with booms, weirs or natural barriers. Use (oil) skimming equipment or suction hoses to remove slick. Sorbents may be used to adsorb the spilled material.

Air: Use water spray to control and disperse flammable vapour. Control runoff for later treatment and/or disposal.

## NAS HAZARD RATING

| Category | Rating |
|---|---|
| Fire | 3 |
| Health | |
| Vapour Irritant | 1 |
| Liquid or Solid Irritant | 1 |
| Poison | 2 |
| Water Pollution | |
| Human Toxicity | 1 |
| Aquatic Toxicity | 2 |
| Aesthetic Effect | 2 |
| Reactivity | |
| Other Chemicals | 0 |
| Water | 0 |
| Self-reaction | 0 |

NFPA
HAZARD
CLASSIFICATION



## 2 PHYSICAL AND CHEMICAL DATA

### Physical State Properties

| | |
|---|---|
| Appearance | Colourless liquid (Verschueren 1984) |
| Usual shipping state | Liquid |
| Physical state at 15°C, 1 atm | Liquid |
| Melting point | 6.55°C (CRC 1980) |
| Boiling point | 80.74°C (CRC 1980) |
| Vapour pressure | 16.2 kPa (30°C) (Kirk-Othmer 1980) |

### Densities

| | |
|---|---|
| Density | 0.7785 g/mL (20°C) (PPH 1984) |
| Specific gravity | 0.77855 (20°/4°C) (CRC 1980) |
| Vapour density | 2.90 (Verschueren 1984) |

### Fire Properties

| | |
|---|---|
| Flammability | Extremely flammable liquid (DuPont 1984) |
| Flash point | |
| CC | -20°C (NFPA 1978)<br>-17°C (Kirk-Othmer 1980) |
| Autoignition temperature | 245°C (NFPA 1978) |
| Burning rate | 6.9 mm/min (CHRIS 1978) |
| Upper flammability limit | 8 percent (v/v) (NFPA 1978)<br>8.4 percent (v/v) (Kirk-Othmer 1980) |
| Lower flammability limit | 1.3 percent (v/v) (NFPA 1978) |
| Heat of combustion | 3919 kJ/mole (25°C) (CRC 1980) |
| Combustion products | Carbon dioxide and water (CRC 1980) |
| Flashback potential | Flashback may occur along vapour trail (AAR 1981) |
| Electrical ignition hazard | May be ignited by static discharge |

### Other Properties

| | |
|---|---|
| Molecular weight of pure substance | 84.16 (CRC 1980) |
| Constituent components of typical commercial grade | 85 to 99.86 percent cyclohexane<br>85 percent solvent grade may contain hexanes, pentanes, benzene and toluene (ISH 1977) |

| | |
|---|---|
| Refractive index | 1.42662 (20°C) (CRC 1980) |
| Viscosity | 0.980 mPa•s (20°C) (Kirk-Othmer 1980)<br>0.083 mPa•s (vapour at 20°C) (PPH 1984) |
| Liquid interfacial tension with air | 25.5 mN/m (20°C) (CRC 1980) |
| Liquid interfacial tension with water | 50 mN/m (25°C) (CHRIS 1978) |
| Latent heat of fusion | 2.6 kJ/mole (at melting point) (CRC 1980) |
| Latent heat of sublimation | 33.0 kJ/mole (25°C) (Lange's Handbook 1979) |
| Latent heat of vaporization | 32.8 kJ/mole (at boiling point) (CRC 1980) |
| Heat of formation | -156.3 kJ/mole (25°C) (Sussex 1977) |
| Ionization potential | 9.88 eV (Lossing 1976) |
| Heat capacity | |
| constant pressure (Cp) | 152 J/(mole•°C) (25°C) (CRC 1980)<br>116 J/(mole•°C) (vapour at 25°C) (PPH 1984) |
| constant volume (Cv) | 140 J/(mole•°C) (25°C) (CRC 1980; CHRIS 1978) |
| Critical pressure | 4050 kPa (CRC 1980) |
| Critical temperature | 280.4°C (CRC 1980) |
| Saturation concentration | 357 g/$m^3$ (20°C)<br>532 g/$m^3$ (30°C) (Verschueren 1984) |
| Diffusivity (in air) | 0.086 $cm^2$/s (45°C) (Perry 1973) |
| Log$_{10}$ octanol/water partition coefficient | 3.44 (Hansch and Leo 1979) |
| Dielectric constant | 2.023 (20°C) (Kirk-Othmer 1980) |
| Entropy | 298.435 J/(mole•K) (25°C) (Ullmann 1975) |
| Evaporation rate | 2.6 g/($m^2$s) (20°C) (this work) |
| Thermal conductivity | 1.3 x $10^{-3}$ W/(cm•K) (liquid at 20°C)<br>1.26 x $10^{-4}$ W/(cm•K) (gas at 25°C) (PPH 1984) |

**Solubility**

| | |
|---|---|
| In water | 0.0055 g/100 mL (55 ppm) (20°C),<br>0.0045 g/100 mL (15°C) (Verschueren 1984) |
| In other common materials | Miscible in ethanol, diethyl ether, acetone, benzene and carbon tetrachloride (CRC 1980) |

Azeotropes

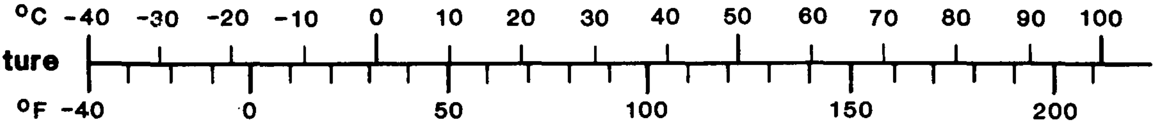
| Second Component | Cyclohexane (Weight %) | Boiling Point (°C) |
|---|---|---|
| Water | 91.6 | 69.0 |
| Benzene | 48.7 | 77.6 |
| Methanol | 39 | 54.2 |
| Ethanol | 70 | 64.9 |
| Acetone | 32.5 | 53.0 |
| Isopropyl Alcohol | 67 | 68.6 |

(Ullmann 1975; Kirk Othmer 1980)

**Vapour Weight to Volume Conversion Factor** 1 ppm = 3.492 mg/m$^3$ (20°C) (Verschueren 1984)

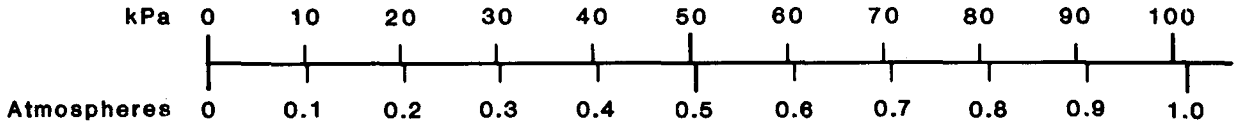
# CONVERSION NOMOGRAMS

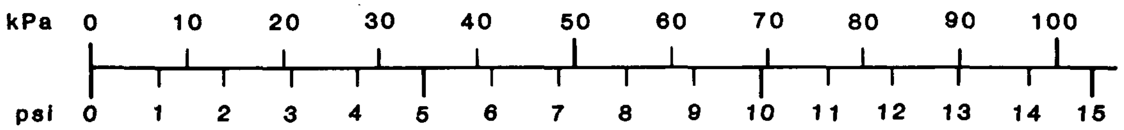
## Temperature

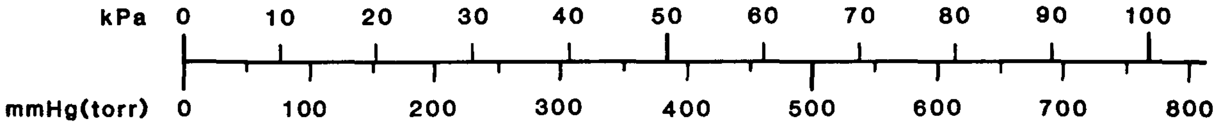


## Pressure

1 kPa = 1 000 Pa







## Viscosity

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

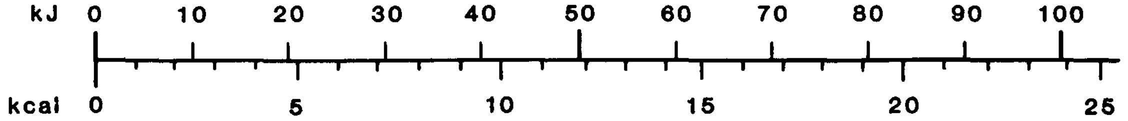
**Kinematic** 1 $m^2/s$ = 1 000 000 centistokes (cSt)

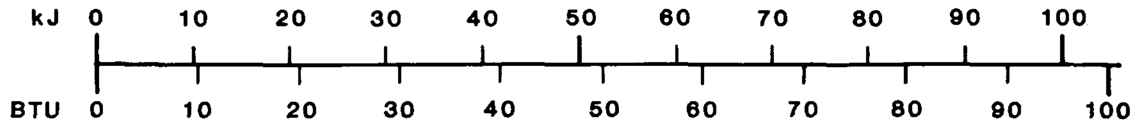
## Concentration (in water)

1 ppm ≅ 1 mg/L

## Energy (heat)

1 kJ = 1 000 J





## Density

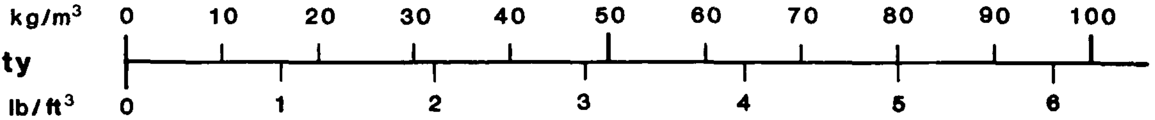


Figure 1

CYCLOHEXANE

## VAPOUR PRESSURE VS TEMPERATURE

Reference: WILHOIT 1971

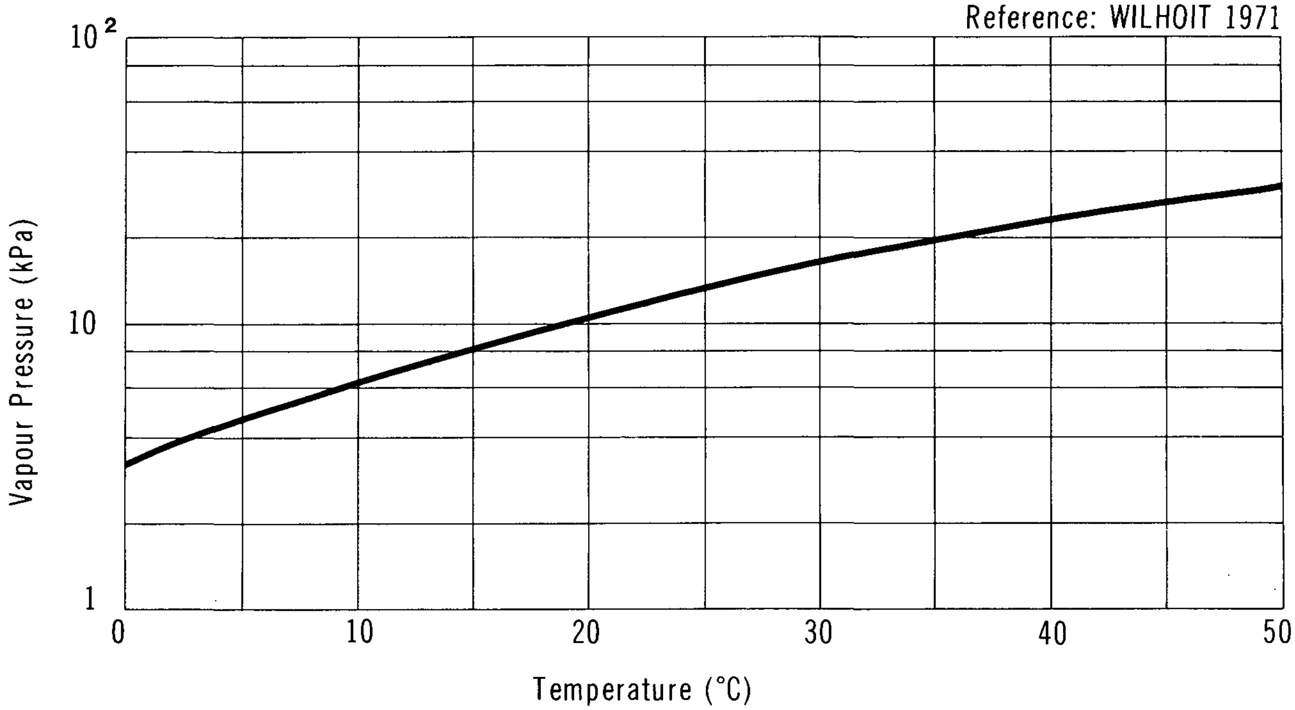


FIGURE 2

CYCLOHEXANE

## LIQUID INTERFACIAL TENSION WITH AIR

Reference: PPH 1984

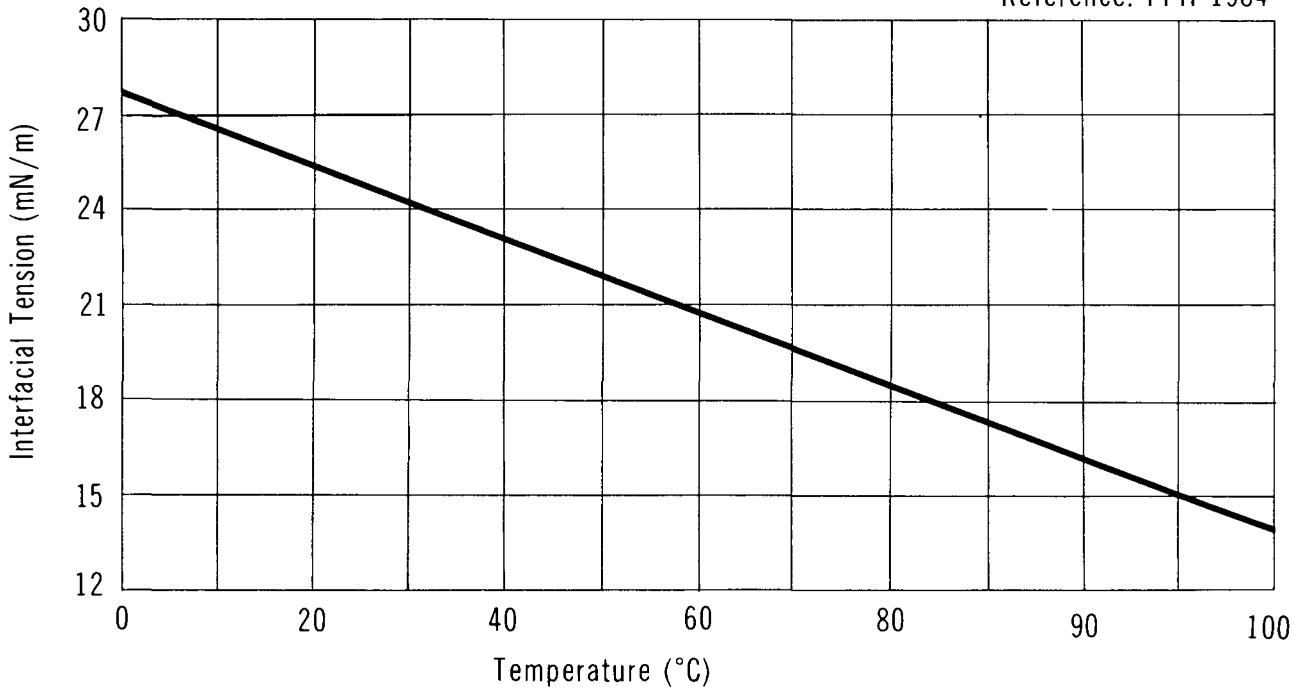


FIGURE 3

CYCLOHEXANE

## LIQUID DENSITY VS TEMPERATURE

Reference: CHRIS 1978

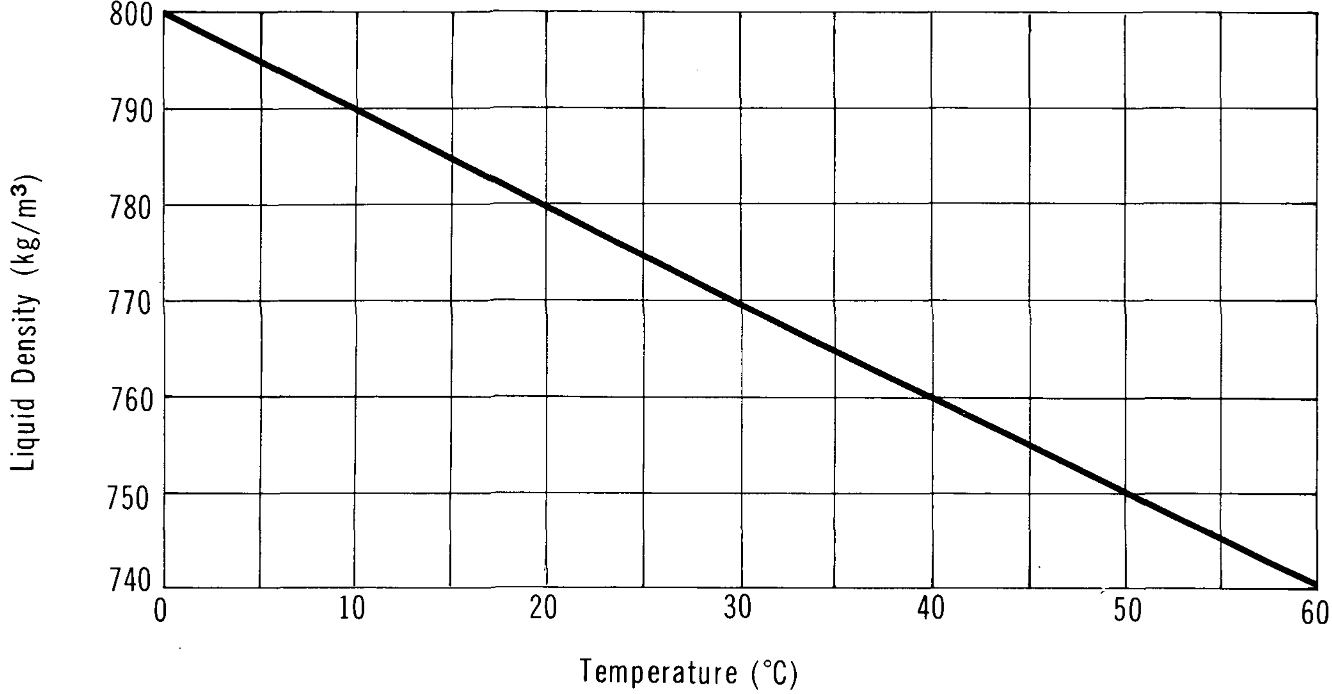


FIGURE 4

CYCLOHEXANE

## LIQUID VISCOSITY VS TEMPERATURE

PPH 1984

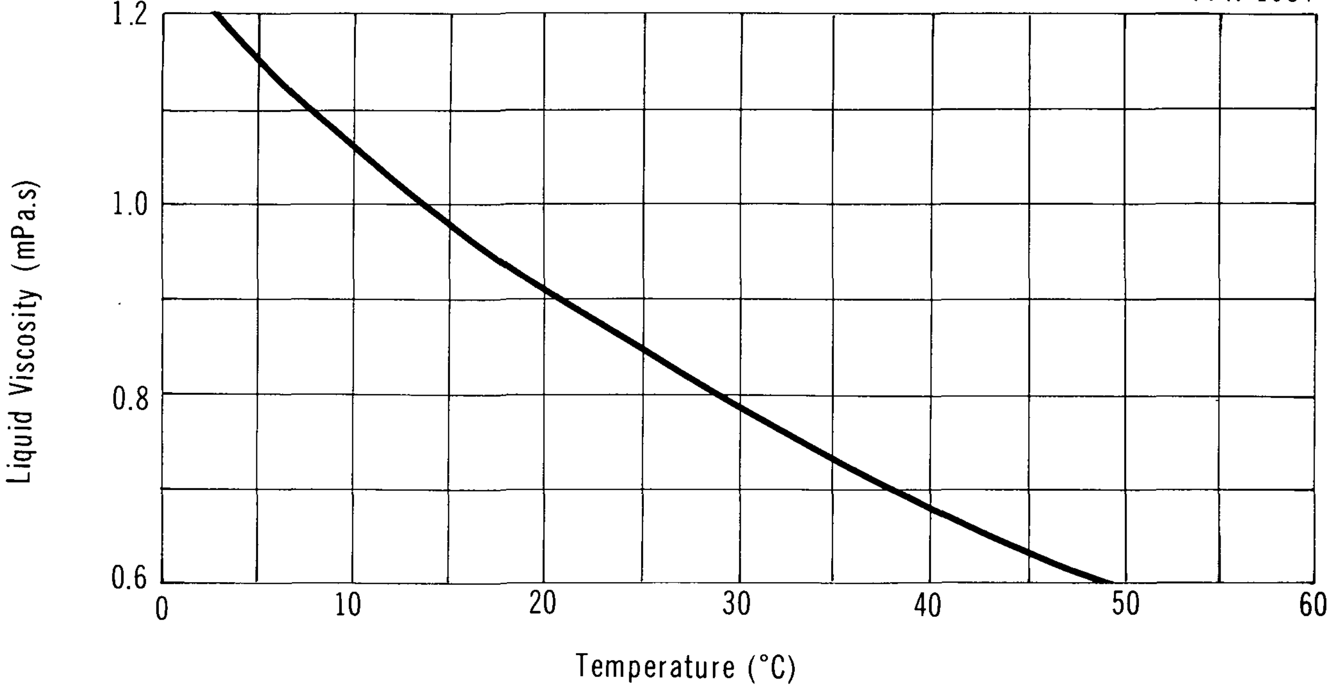
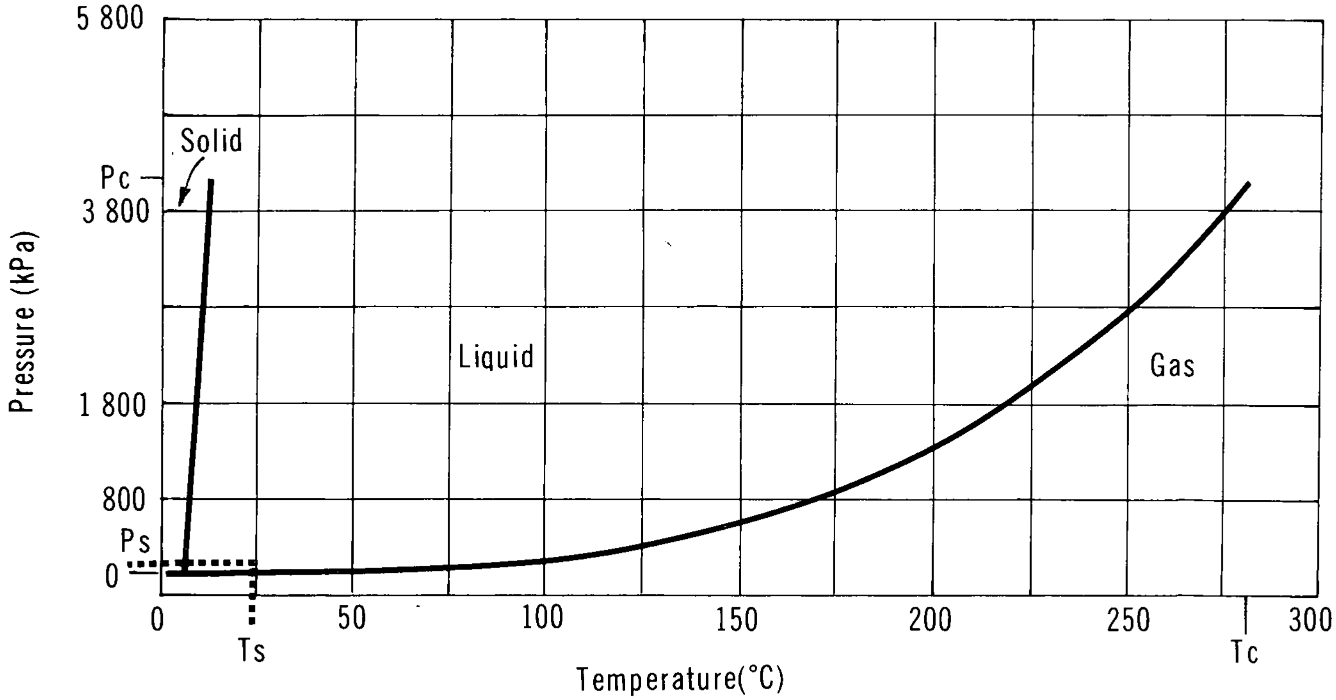


Figure 5

CYCLOHEXANE

# PHASE DIAGRAM



## 3 COMMERCE AND PRODUCTION

### 3.1 Grades, Purities (FKC 1975; Kirk-Othmer 1980)

Cyclohexane is produced as a commercial grade of 99.9 percent and a solvent grade of 85 percent purity. The latter contains hexanes, pentanes, benzene and toluene.

### 3.2 Domestic Manufacturer (Corpus 1984; CBG 1980)

This is a corporate headquarters' address and is not intended as a spill contact:

Gulf Canada Products Co.
Division of Gulf Canada Ltd.
800 Bay Street
Toronto, Ontario
M5S 1Y8
(416) 924-4141

### 3.3 Other Suppliers (CBG 1980)

Ashland Chemical/Solvents Division
Valvoline Oil and Chemicals
150 Bronoco Avenue
Toronto, Ontario
M6E 4Y1
(416) 651-2822

General Intermediates of Canada
10817 138 Street
Edmonton, Alberta
T5M 1P6
(403) 454-5780

Kingsley and Keith (Canada) Ltd.
310 Victoria Avenue
Montreal, Quebec
H3Z 2M8
(514) 487-1550

Montedison Canada Ltd.
2045 Stanley Street
Montreal, Quebec
H3A 2V4
(514) 842-5084
(514) 842-4355

### 3.4 Major Transportation Routes

Current Canadian production of cyclohexane is in Montreal, Quebec. The product is primarily shipped to a major buyer located in Maitland, Ontario.

### 3.5 Production Levels (Corpus 1984)

| Company, Plant Location | | Nameplate Capacity kilotonnes/yr (1983) |
|---|---|---|
| Gulf Canada, Montreal, Quebec | | 104 |
| | TOTAL | 104 |
| Domestic Production (1983) | | 93.5 |
| Imports (1983) | | 0.03 |
| | TOTAL SUPPLY | 93.53 |

### 3.6 **Manufacture of Cyclohexane** (FKC 1975; Kirk-Othmer 1980)

**3.6.1 General.** Cyclohexane is made by the catalytic hydrogenation of benzene at elevated temperatures and pressures, or by the fractionation of petroleum naphthas from natural gas. The former method is used by the Canadian plant.

### 3.6.2 Manufacturing Processes.

**3.6.2.1 Catalytic hydrogenation of benzene.** Benzene and hydrogen, mixed with cyclohexane, are passed through catalyst beds (nickel or platinum based) at 210°C and 2.4-3.5 kPa, usually via several reactors in series. The cyclohexane diluent is present to absorb the heat of reaction which, if uncontrolled, would lead to isomerization and ring opening:

$$C_6H_6 + 3H_2 \xrightarrow{\text{Ni, Pt}} C_6H_{12}\ (99\%+)$$

The process stream is degassed in a small "reboiler" tower. The resulting cyclohexane is about 99.9 percent pure.

**3.6.2.2 Fractionation of naphtha.** A solvent grade cyclohexane, about 85 percent pure, may be produced by fractionation of naphtha. Greater purification is impractical because of the similar boiling points of the naphtha constituents. To produce higher purity cyclohexane, the naphtha must be fractionated to remove methyl cyclopentane, leaving cyclohexane and dimethyl pentanes. It is then dehydrogenated in a catalytic reformer to produce benzene and methylcylopentane:

$$C_6H_{12} \rightarrow C_6H_6 + CH_3C_5H_9$$

This is fractionated to remove low boilers (pentanes, etc.) and yield a mixture of methyl cyclopentane (90 percent) and benzene (10 percent). This, plus the methyl cyclopentane cut from the first fractionation, is hydrogenated and the benzene converted to cyclohexane:

$$C_6H_6 \rightarrow C_6H_{12}$$

The final process step is to isomerize the methyl cyclohexane in the process stream to cyclohexane by passing it over aluminum chloride:

$$CH_3C_5H_9 \rightarrow C_6H_{12}$$

### 3.7 Major Uses in Canada (Corpus 1984)

Cyclohexane is used for the production of adipic acid (for nylon intermediates), cyclohexanol, cyclohexanone, and as a solvent. Almost all Canadian production is used for adipic acid production.

### 3.8 Major Buyer in Canada (Corpus 1984)

Du Pont Canada, Maitland, Ont.

# 4 MATERIAL HANDLING AND COMPATIBILITY

## 4.1 Containers and Transportation Vessels

**4.1.1 Bulk Shipment.** Transportation vessels and containers under this category have been grouped under the classifications of railway tank cars and highway tank vehicles.

**4.1.1.1 Railway tank cars.** Railway tank cars used to transport cyclohexane fall under a variety of specifications (Table 2) (TCM 1979; RTDCR 1974). However, the class 111A60W1 is the most commonly used (Figure 6). Railway tank car details associated with Figure 6 are shown in Table 3 (TCM 1979). Cars are equipped for unloading by pump from the top. Cyclohexane is withdrawn through an eduction pipe which extends from the bottom of the tank to the top operating platform where it terminates with an unloading connection valve. Air pressure is never used for unloading cyclohexane tankers (MCA 1957).

A safety relief valve set at 241 kPa (35 psi) and a safety vent set at 414 kPa (100 psi) are required on top of the rail car (TCM 1979). A gauging device, either the rod type or the tape type, is required. The top unloading connection must be protected by a housing.

**4.1.1.2 Tank motor vehicles.** Cyclohexane is transported by tank motor vehicles with tanks classed as nonpressure vessels (MCA 1957). Design pressure for such tanks does not exceed 21 kPa (3 psi). Motor vehicle tanks carrying cyclohexane are similar to the railway tanks previously described. These highway tankers are usually unloaded by pump from the top unloading connection valve.

The off-loading equipment and procedures for tank motor vehicles are similar to those for railway tank cars.

**4.1.2 Packaging.** Cyclohexane, in addition to railway bulk shipments, is also transported in drums. Drums fabricated from a variety of construction materials are permitted (Table 4) (TDGC 1980).

## 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 (MCA 1957):

- The vented storage tank must be checked to make sure that it will hold the contents of the car.

TABLE 2 RAILWAY TANK CAR SPECIFICATIONS

| CTC/DOT* Specification Number | Description |
|---|---|
| 111A60W1 | Steel fusion-welded tank without dome.<br>Uninsulated or insulated.<br>2% minimum outage. Gauging device.<br>Test pressure 414 kPa (60 psi). |
| 111A60F1 | Steel forge-welded tank without dome.<br>Uninsulated or insulated.<br>2% minimum outage. Gauging device.<br>Test pressure 414 kPa (60 psi). |
| 111A100W1 | Steel fusion-welded tank without dome.<br>Uninsulated or insulated.<br>2% minimum outage. Gauging device.<br>Bottom outlet or washout optional.<br>Test pressure 690 kPa (100 psi). |
| 103W | Steel fusion-welded tank with dome.<br>Uninsulated or insulated.<br>2% dome. Safety valve 242 kPa (35 psi) or safety vent 414 kPa (60 psi).<br>Bottom outlet or washout optional. |
| 103ALW | Aluminum fusion-welded tank with dome.<br>Uninsulated or insulated.<br>2% dome. Safety valve 242 kPa (35 psi) or safety vent 414 kPa (60 psi).<br>Bottom outlet or washout optional. |
| 104W | Same as 103W except insulated. |

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

- For night-time unloading, lights must have an explosion-proof rating.
- Personnel must not enter the car under any circumstances.
- Brakes must be set, wheels chocked, derails placed and caution signs displayed.
- A safe operating platform must be provided at the unloading point.
- Tools used during unloading must be spark-resistant.
- Effectively ground the tank car.

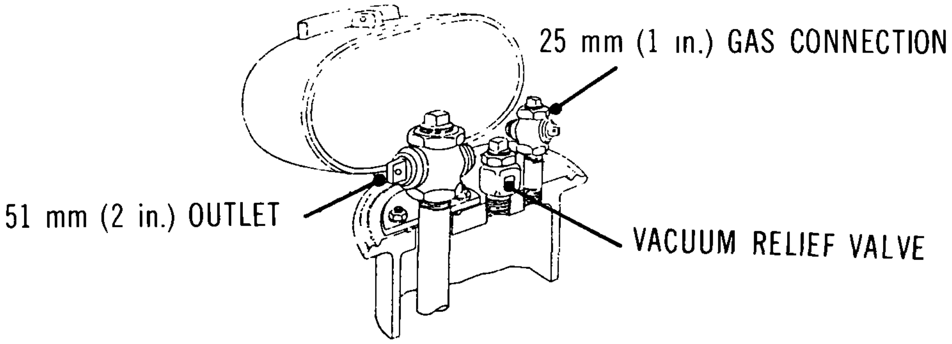
Proceed with top off-loading as follows (MCA 1957):

- Relieve the tank of internal vapour pressure by cooling the tank with water or venting at short intervals.

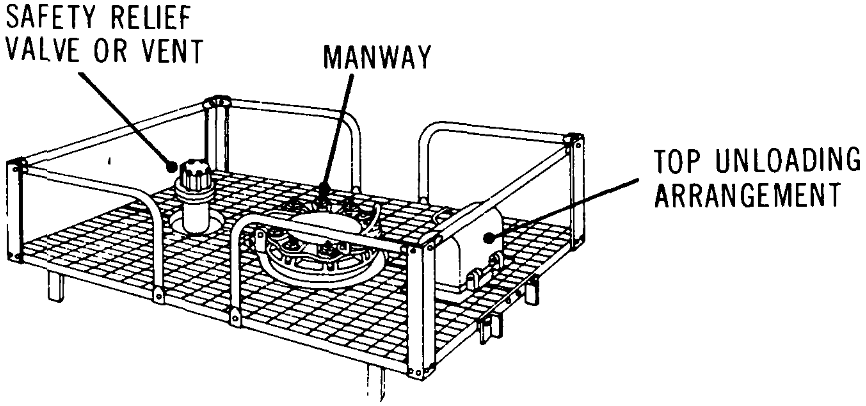
FIGURE 6

## RAILWAY TANK CAR - CLASS 111A60W1

(Reference - TCM 1979; RTDCR 1974)



**Detail of top unloading arrangement**



**Detail of loading platform**

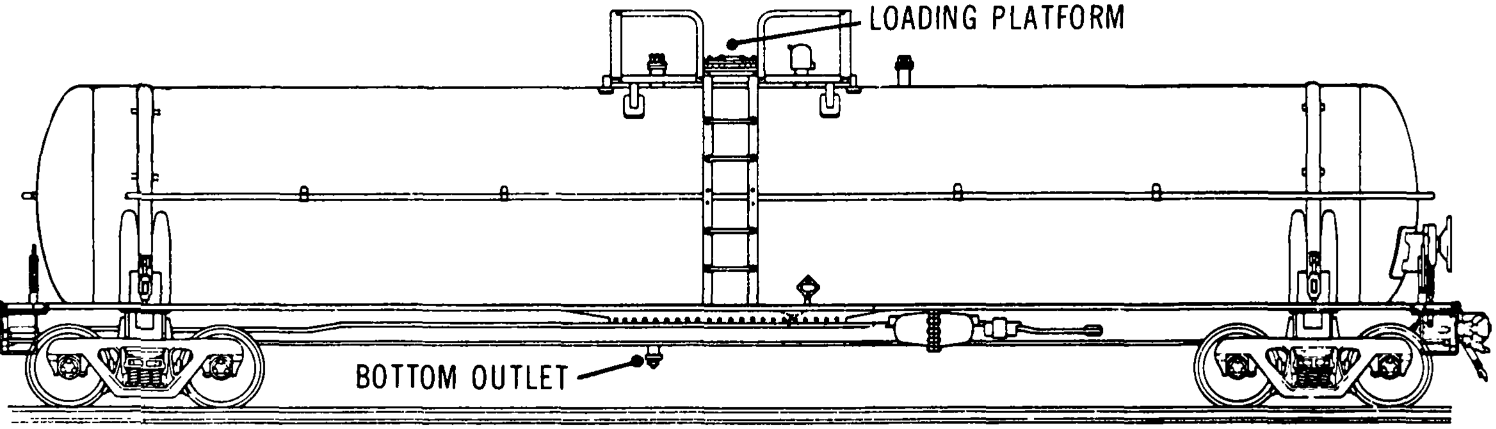


**Illustration of tank car layout**

TABLE 3 TYPICAL RAILWAY TANK CAR SPECIFICATIONS - CLASS 111A60W1 (TCM 1979; RTDCR 1974)

| | Tank Car Size (Imp. Gal.) | | | | | |
|---|---|---|---|---|---|---|
| Description | 16 700 | | 17 200 | | 20 000 | |
| <u>Overall</u> | | | | | | |
| Nominal capacity | 75 700 L | (16 700 gal.) | 78 000 L | (17 200 gal.) | 90 900 L | (20 000 gal.) |
| Car weight - empty | 33 900 kg | (74 700 lb.) | 33 900 kg | (74 700 lb.) | 38 900 kg | (85 800 lb.) |
| Car weight - max. | 119 000 kg | (263 000 lb.) | 83 500 kg | (184 000 lb.) | 119 000 kg | (263 000 lb.) |
| <u>Tank</u> | | | | | | |
| Material | Steel | | Steel | | Steel | |
| Thickness | 11.1 mm | (7/16 in.) | 11.1 mm | (7/16 in.) | 11.1 mm | (7/16 in.) |
| Inside diameter | 2.60 m | (102 in.) | 2.62 m | (103 in.) | 2.74 m | (108 in.) |
| Test pressure | 414 kPa | (60 psi) | 414 kPa | (60 psi) | 414 kPa | (60 psi) |
| Burst pressure | 1640 kPa | (240 psi) | 1640 kPa | (240 psi) | 1640 kPa | (240 psi) |
| <u>Approximate Dimensions</u> | | | | | | |
| Coupled length | 17 m | (57 ft.) | 17 m | (57 ft.) | 18 m | (60 ft.) |
| Length over strikers | 16 m | (53 ft.) | 16 m | (53 ft.) | 17 m | (57 ft.) |
| Length of truck centres | 13 m | (42 ft.) | 13 m | (42 ft.) | 14 m | (45 ft.) |
| Height to top of grating | 4 m | (12 ft.) | 4 m | (12 ft.) | 4 m | (12 ft.) |
| Overall height | 5 m | (15 ft.) | 5 m | (15 ft.) | 5 m | (15 ft.) |
| Overall width (over grabs) | 3.2 m | (127 in.) | 3.2 m | (127 in.) | 3.2 m | (127 in.) |
| Length of grating | 2 to 3 m | (8 to 10 ft.) | 2 to 3 m | (8 to 10 ft.) | 2 to 3 m | (8 to 10 ft.) |
| Width of grating | 1.5 to 2 m | (5 to 6 ft.) | 1.5 to 2 m | (5 to 6 ft.) | 1.5 to 2 m | (5 to 6 ft.) |
| <u>Loading/Unloading Fixtures</u> | | | | | | |
| <u>Top Unloading</u> | | | | | | |
| Unloading connection | 51 mm | (2 in.) | 51 mm | (2 in.) | 51 mm | (2 in.) |
| Manway/fill hole | 203 to 356 mm | (8 to 14 in.) | 203 to 356 mm | (8 to 14 in.) | 203 to 356 mm | (8 to 14 in.) |
| Air connection | 25 to 51 mm | (1 to 2 in.) | 25 to 51 mm | (1 to 2 in.) | 25 to 51 mm | (1 to 2 in.) |
| <u>Bottom Unloading</u> | | | | | | |
| Bottom outlet | 102 to 152 mm | (4 to 6 in.) | 102 to 152 mm | (4 to 6 in.) | 102 to 152 mm | (4 to 6 in.) |
| <u>Safety Devices</u> | Safety vent or valve | | | | | |
| <u>Dome</u> | None | | | | | |
| <u>Insulation</u> | Optional | | | | | |

TABLE 4 DRUMS

| Type of Drum | Designation | Description | Figure No. (if any) |
|---|---|---|---|
| Steel | 1A1 | Nonremovable head, reusable | 7 |
| | 1A1A | 1A1 with reinforced chime | 7 |
| | 1A1B | 1A1 with welded closure flange | 7 |
| | 1A1D | 1A1 with coating (other than lead) | 7 |
| | 1A2 | Removable head, reusable | 7 |
| | 1A3 | Nonremovable head, single use only | 7 |
| Monel* | TC5M | | 7 |
| Aluminum | 1B1 | Nonremovable head | 7 |
| | 1B2 | Removable head | 7 |
| Steel Drums with inner plastic receptacles | 6HA1 | Outer steel sheet in the shape of drum. Inner plastic receptacle. Maximum capacity of 225 L (49 gal.) | |
| Fibreboard Drums with inner plastic receptacles | 6HG1 | Outer containers of convolutely wound plies of fibreboard. Inner plastic in shape of drum. Maximum capacity of 225 L (49 gal.) | |

* See Section 4.3 of this report.

- After removing the protective housing from the discharge line at the top of the car, connect the 51 mm (2 in.) unloading line.
- Off-load the tanker by pump only.

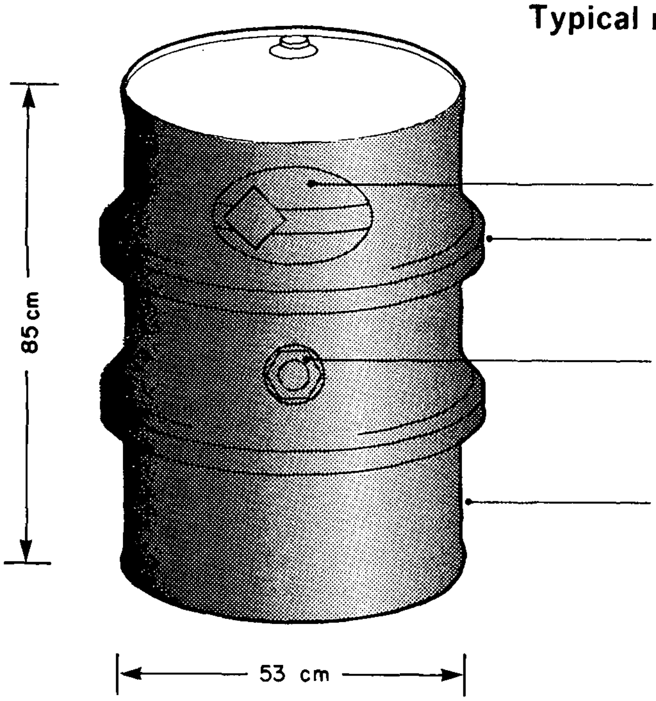
**4.2.2 Specifications and Materials for Off-loading Equipment.** The materials of construction for off-loading system components discussed in this section along with specifications refer to those generally used in cyclohexane service. Other materials may be used for particular applications, as indicated in Table 5. The components of a typical off-loading system that will be discussed include pipes and fittings, flexible connections, valves and pumps.

Schedule 40 seamless ASTM A106 carbon steel pipes and fittings lined with chlorinated polyether resins are recommended for cyclohexane lines (DCRG 1978). Flanged joints should be used and these should be welded, because threaded pipes and

FIGURE 7

# TYPICAL DRUM CONTAINERS

### Typical monel drum



CONTENTS LABEL

ROLLING HOOPS OF PLIABLE SOLID NATURAL RUBBER

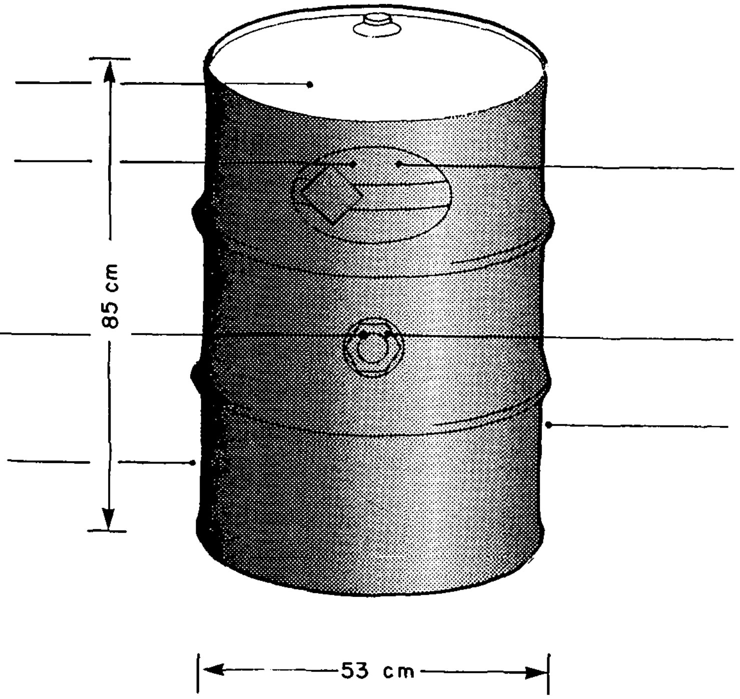
MAX. DIAMETER OPENING 59 mm

BODY AND HEADS CONSTRUCTED OF 10 mm MONEL

MAX. CAPACITY 220 L

### Typical steel drum

### Typical aluminum drum



MIN. 2.4 mm LEAD COATING FOR IAIC DESIGNATION

CONTENTS LABEL

MAX. DIAMETER OPENING 70 mm

BODY AND HEADS CONSTRUCTED OF ROLLED STEEL

MAX. CAPACITY 250 L

MAX. NET MASS 400 kg

CONTENTS LABEL

MAX. DIAMETER OPENING 70 mm

BODY AND HEADS CONSTRUCTED OF MIN. 99% PURE ALUMINUM

MAX. CAPACITY 250 L

MAX. NET MASS 400 kg

fittings tend to leak after a very short time. Stress relief at the weld will also lengthen the serviceability of the pipe. The pipeline should be tested with air at pressures from 345 to 518 kPa (50-75 psi) and all leaks carefully stopped.

The unloading line should be 51 mm (2 in.) pipe because this is the standard fitting on tank cars, but process pipe may be almost any size. Pipe under 25 mm (1 in.), however, is not recommended. Outdoor lines should be self-draining.

Flexible bellows-type expansion joints should be used for the flexible sections of the unloading line. They are manufactured with ASA ductile iron flanges with expansion members molded from tetrafluoroethylene resin (Dow PPS 1972). Some installations of flexible line are made with polypropylene resin-lined hoses.

Cast iron or cast steel diaphragm valves lined with chlorinated polyether or polyvinylidene fluoride resin will serve adequately (Dow PPS 1972).

A single-suction centrifugal pump with "wet end" material of 316 stainless steel gives good results. Provision must be made for draining the pump so that repairs can be made safely (MCA 1957). The pump should be equipped with flanges at both suction and discharge openings; screw connections are more subject to leakage and should be avoided.

### 4.3 Compatibility with Materials of Construction

The compatibility of cyclohexane 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

| Application | Chemical | | Material of Construction | | |
|---|---|---|---|---|---|
| | Conc. | Temp. (°C) | Recommended | Conditional | Not Recommended |
| 1. Pipes and Fittings | | | | | PE, ABS<br>PVC I<br>(MWPP 1978) |
| | | 135 | PVDF<br>(DCRG 1978) | | |
| | 100% | 21 | CS | | |
| | | 107 | Chlorinated Polyether<br>(DCRG 1978) | | |
| | | 52 | PVDC<br>(DCRG 1978) | | |
| | | 24 | PP<br>(DCRG 1978) | | |
| 2. Valves | | 21 | SS 316<br>(JSSV 1979) | | |
| 3. Storage | 100% | 21 | CS | | |
| 4. Others | | | | | PVC I<br>PE, ABS<br>(MWPP 1978) |

TABLE 6 MATERIALS OF CONSTRUCTION

| Abbreviation | Material of Construction |
|---|---|
| ABS | Acrylonitrile Butadiene Styrene |
| CS | Carbon Steel |
| | Chlorinated Polyether |
| | Glass |
| GRP | Glass Reinforced Vinyl Ester |
| NR | Natural Rubber |
| | Nickel-Copper Alloy (Monel) |
| PE | Polyethylene |
| PP | Polypropylene |
| PVC (followed by grade) | Polyvinyl Chloride |
| PVDC | Polyvinylidene Chloride |
| PVDF | Polyvinylidene Fluoride |
| SS (followed by grade) | Stainless Steel |

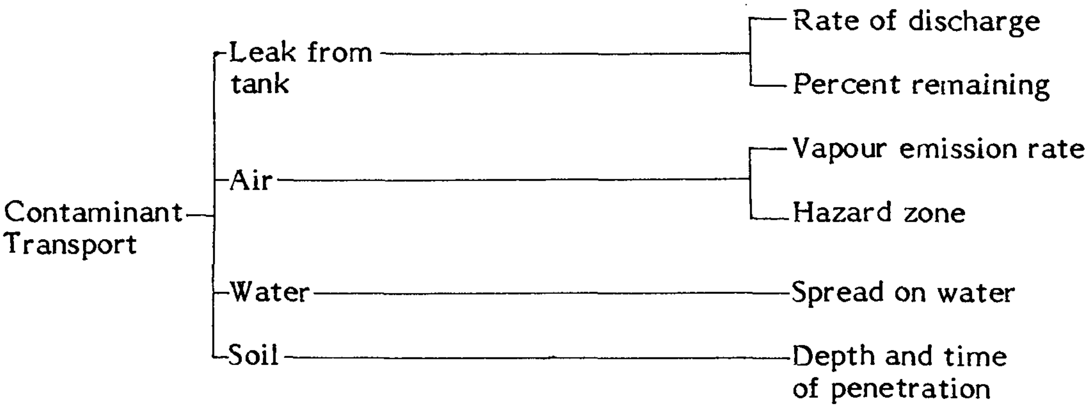
# 5 CONTAMINANT TRANSPORT

## 5.1 General Summary

Cyclohexane is a low viscosity liquid that is nearly insoluble in water. When spilled in the environment, it will form a liquid pool, spreading on the surface of a water body or on the ground. Vapour will be released continuously to the atmosphere.

Cyclohexane when spilled on the ground will partly vaporize and partly adsorb at a rate dependent on the soil type and its degree of saturation with water. At the same time, the downward transport of the liquid toward the groundwater table may cause environmental problems.

The following factors are considered for the transport of a cyclohexane spill in the air, water and soil media:



It is important to note that, because of the approximate nature of the contaminant transport calculations, the approach adopted throughout the EnviroTIPS series has been to use conservative estimates of critical parameters so that predictions are approaching worst case scenarios for each medium.

## 5.2 Leak Nomograms

**5.2.1 Introduction.** Cyclohexane is commonly transported in railway tank cars as a nonpressurized liquid. While the capacities of the tank cars vary widely, one tank car size has been chosen throughout the EnviroTIPS series for development of the leak nomograms. It is approximately 2.75 m in diameter and 13.4 m long, with a carrying capacity of about 80 000 L.

If a tank car loaded with cyclohexane is punctured on the bottom, all of the contents will drain out by gravity. The aim of the nomograms is to provide a simple means to obtain the time history of the conditions in the tank car and the venting rate of the liquid. Because of the moderate volatility of cyclohexane and the fact that the tank cars are not pressurized, no leak nomograms have been prepared for vapour release from a puncture in the top of the tank.

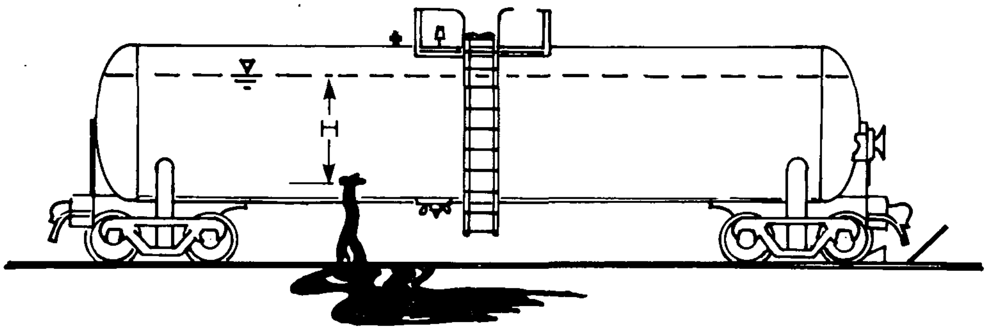


FIGURE 8 TANK CAR WITH PUNCTURE HOLE IN BOTTOM

### 5.2.2 Nomograms.

**5.2.2.1 Figure 9: Percent remaining versus time.** Figure 9 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 is assumed to be initially full (at $t=0$) with a volume of about 80 000 L of cyclohexane. 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 10: Discharge rate versus time.** Figure 10 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 filled with cyclohexane 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 9

CYCLOHEXANE

## PERCENT REMAINING VS TIME

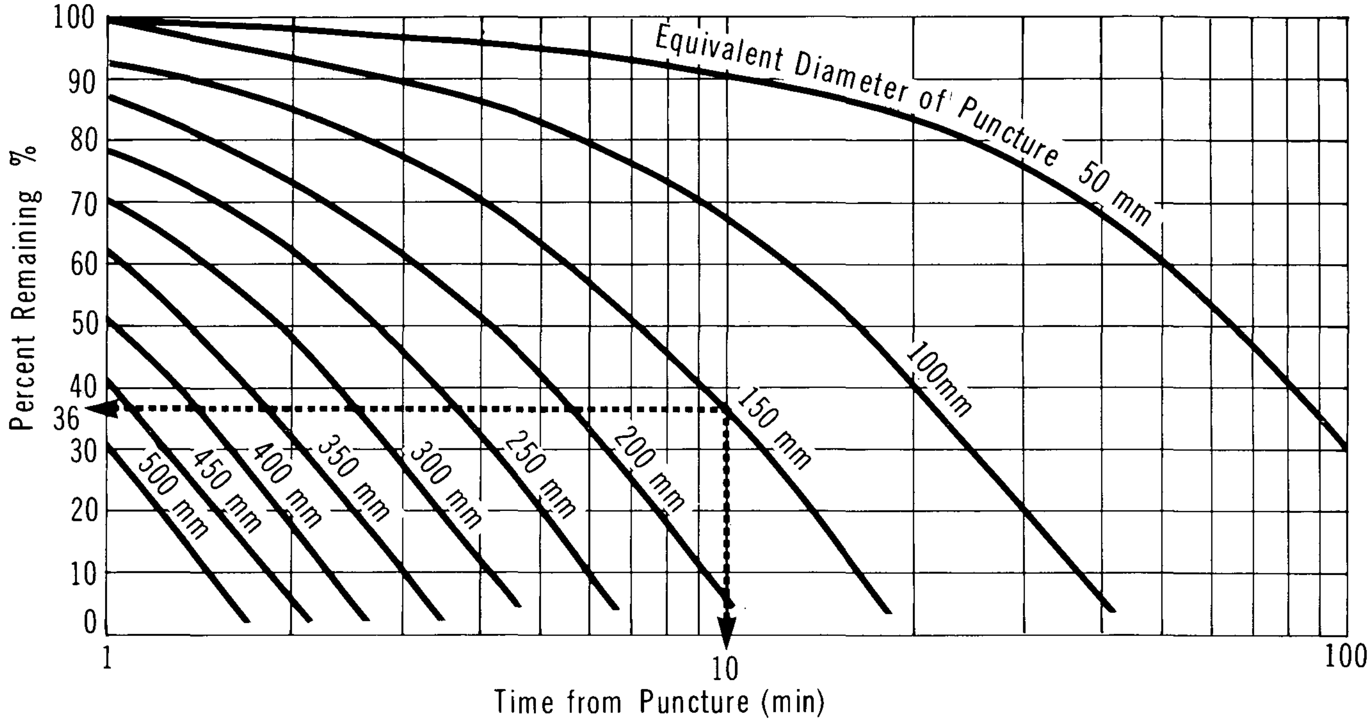
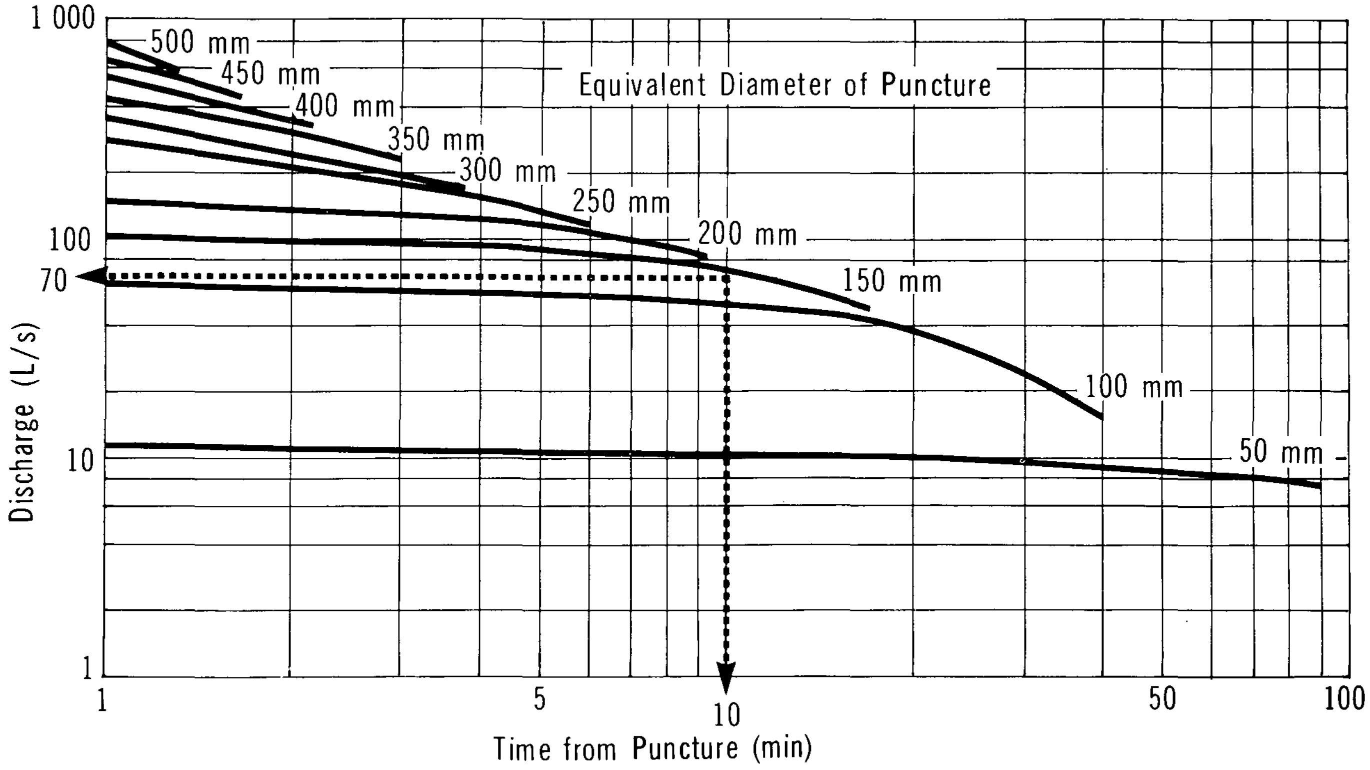


FIGURE 10

CYCLOHEXANE

## DISCHARGE RATE VS TIME



Solution to Problem A

- Use Figure 9
- 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 10
- With t=10 min and d=150 mm, the instantaneous discharge rate (q) = 70 L/s

### 5.3 Dispersion in the Air

**5.3.1 Introduction.** Since cyclohexane is a moderately volatile liquid, direct venting of the vapour to the atmosphere from a hole in a punctured vessel does not constitute a significant hazard downwind. Only vapour released from a liquid pool spilled on a ground or water surface is treated here.

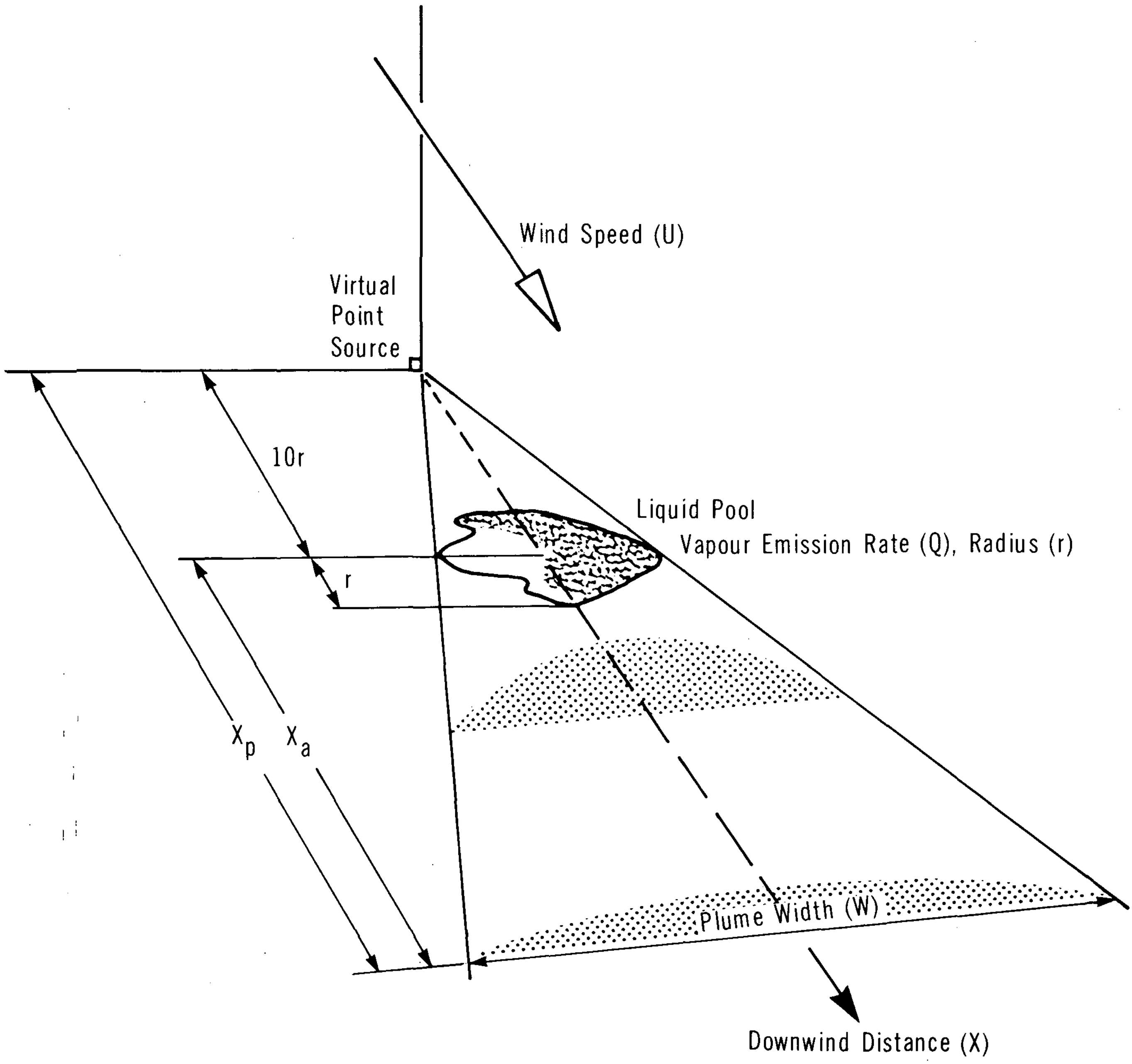
To estimate the vapour concentration downwind of the accident site for the determination of the flammability or toxicity hazard zone, the atmospheric transport and dispersion of the contaminant vapour must be modelled. The models used here are based on Gaussian formulations and are the ones most widely used in practice for contaminant concentration predictions. The model details are discussed in the Introduction Manual.

Figure 11 schematically depicts the contaminant plume configuration from a continuous surface release. The dispersion model represents the liquid pool area source as a virtual point source (with the same vapour emission rate, Q) located 10 equivalent pool radii upwind.

**5.3.2 Vapour Dispersion Nomograms and Tables.** The aim of the air dispersion nomograms is to define the hazard zone due to toxicity or flammability of a vapour cloud. The following nomograms and data tables are contained in this section (to be used in the order given):

| | |
|---|---|
| Figure 13: | vapour emission rate from a liquid pool as a function of maximum pool radius |
| Table 7: | weather conditions |
| Figure 14: | normalized vapour concentration as a function of downwind distance and weather conditions |

# SCHEMATIC OF CONTAMINANT PLUME



| | |
|---|---|
| Table 8: | maximum plume hazard half-widths |
| Figure 17: | vapour plume travel distance as a function of time elapsed since the spill and wind speed |

The flowchart given in Figure 12 outlines the steps necessary to make vapour dispersion calculations and identifies the nomograms or tables to be used. This section deals only with the portion contained within the dashed box. Data on "total liquid discharged" and "equivalent pool radius" are contained in Sections 5.2 and 5.4, respectively. A description of each vapour dispersion nomogram and its use follows.

**5.3.2.1 Figure 13: Vapour emission rate versus liquid pool radius for various temperatures.** An evaporation rate for cyclohexane has been calculated using the evaporation rate equations found in the Introduction Manual. The computed evaporation rate for cyclohexane at 20°C and a wind speed of 4.5 m/s (16.1 km/h) is 2.6 g/($m^2$s). Evaporation rates at other temperatures have been calculated using the evaporation rate equation which, at a given speed, is dependent on ambient temperature and the vapour pressure of cyclohexane at that temperature (Perry 1973). Evaporation rates of 0.90 g/($m^2$s) at 0°C and 3.74 g/($m^2$s) at 30°C for example, were calculated for a wind speed of 4.5 m/s.

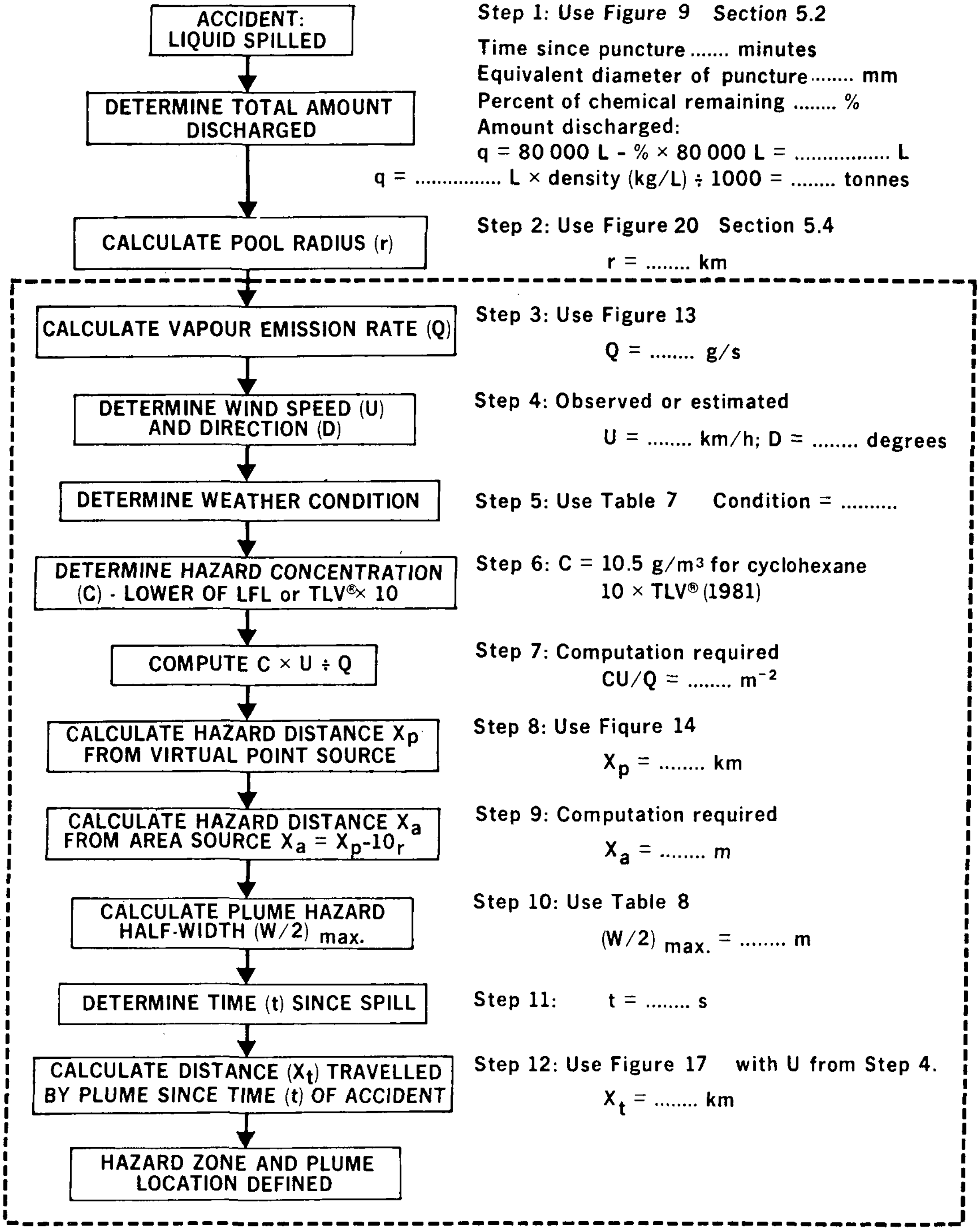
Using Figure 20, Section 5.4, the maximum spill radius corresponding to various spill amounts of cyclohexane may be determined. The resultant spill areas and the cyclohexane evaporation rates provide the basis for preparation of the vapour release rate versus spill radius nomogram in Figure 13.

Use: For a pool of cyclohexane of known radius, the rate (Q) at which cyclohexane vapour is released to the atmosphere at a given temperature can then be estimated from Figure 13. The solid portions of the curves represent spills of 0.05 to 62 tonnes, the latter representing about one standard 80 000 L rail car load of cyclohexane. It should be noted that Figure 13 is valid for a wind speed of 4.5 m/s (16.1 km/h) and therefore can only be used to provide an approximation of cyclohexane vapour emission rates at other wind speeds. The Introduction Manual contains the appropriate equation to convert the evaporation rate at 4.5 m/s to an evaporation rate at another wind speed should it be desired.

It should also be noted that the determination of the emission rate is based on the spill radius on calm water (Table VI, CHRIS 1974). Since calm water represents a flat, unbounded surface compared to the type of ground surface that would normally be encountered in a spill situation (namely, irregular and porous), the spill radius on calm

FIGURE 12

## FLOW CHART TO DETERMINE VAPOUR HAZARD ZONE



Step 1: Use Figure 9 Section 5.2

Time since puncture ....... minutes
Equivalent diameter of puncture ........ mm
Percent of chemical remaining ........ %
Amount discharged:
q = 80 000 L - % × 80 000 L = ................. L
q = ................ L × density (kg/L) ÷ 1000 = ........ tonnes

Step 2: Use Figure 20 Section 5.4

r = ........ km

Step 3: Use Figure 13

Q = ........ g/s

Step 4: Observed or estimated

U = ........ km/h; D = ........ degrees

Step 5: Use Table 7 Condition = ..........

Step 6: C = 10.5 g/m³ for cyclohexane
10 × TLV® (1981)

Step 7: Computation required

CU/Q = ........ $m^{-2}$

Step 8: Use Fiqure 14

$X_p$ = ........ km

Step 9: Computation required

$X_a$ = ........ m

Step 10: Use Table 8

$(W/2)_{max.}$ = ........ m

Step 11: t = ........ s

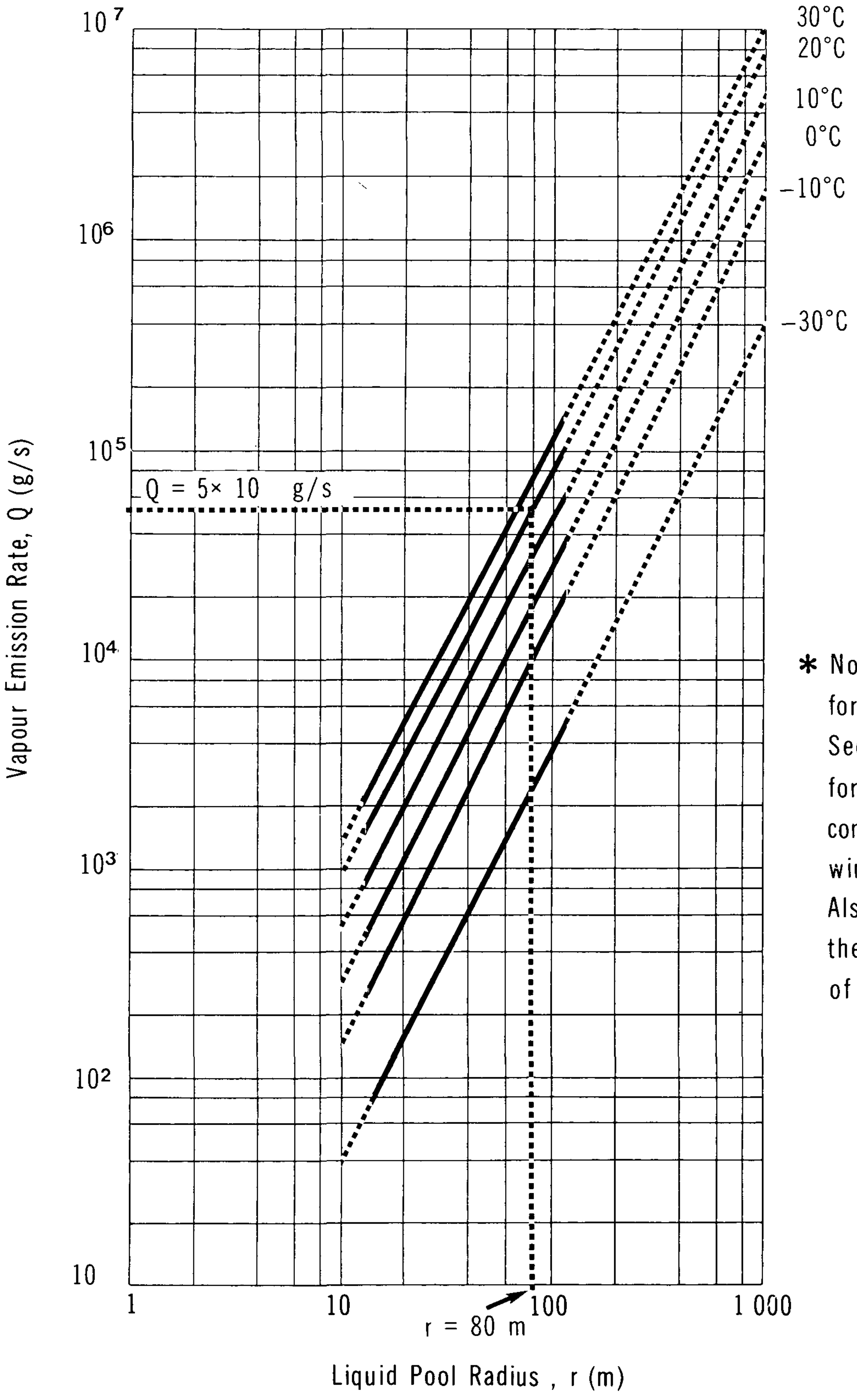
Step 12: Use Figure 17 with U from Step 4.

$X_t$ = ........ km

FIGURE 13

CYCLOHEXANE

## VAPOUR EMISSION RATE VS LIQUID POOL RADIUS FOR VARIOUS TEMPERATURES



* Note: Nomogram applies for wind speed of 4.5 m/s. See Introduction Manual for relationships to compute values for other wind speeds, if necessary. Also, the solid portions of the curves represent spills of 0.05 to 62 tonnes.

water is considered to provide the maximum value. Therefore, when spills on land are assessed by using the water algorithm, the spill radius would be overestimated and worst case values provided.

**5.3.2.2 Figure 14: Normalized vapour concentration versus downwind distance.** Figure 14 shows the relationship between the vapour concentration and the downwind distance for weather conditions D and F. The nomograms were developed using the dispersion models described in the Introduction Manual. The vapour concentration is represented by the normalized, ground-level concentration (CU/Q) at the centreline of the contaminant plume. Weather condition F is the poorest for dispersing a vapour cloud and condition D is the most common in most parts of Canada. Before using Figure 14, the weather condition must be determined from Table 7.

TABLE 7 WEATHER CONDITIONS

| Weather Condition F | Weather Condition D |
|---|---|
| Wind speed <11 km/h<br>(≃ 3 m/s) and one of the following:<br>- overcast day<br>- night time<br>- severe temperature inversion | Most other weather conditions |

<u>Use</u>: The maximum hazard distance, $X_p$, downwind of the spill can be calculated from Figure 14 knowing:

- Q, the vapour emission rate (g/s)
- U, the wind speed (m/s)
- the weather condition
- the hazard concentration limit, C which is the lower value of 10 times the Threshold Limit Value (TLV®, in g/m$^3$), or the Lower Flammability Limit (LFL, in g/m$^3$). Note: To convert the TLV® (in ppm) and the LFL (in % by volume) to concentrations in g/m$^3$, use Figures 15 and 16.

A hazard concentration limit of 10 times the TLV® has been arbitrarily chosen as it represents a more realistic level at which there would be concern for human health on the short-term (i.e., on the order of 30 minutes). The TLV® is the workplace standard

FIGURE 14

CYCLOHEXANE

## NORMALIZED VAPOUR CONCENTRATION VS DOWNWIND DISTANCE

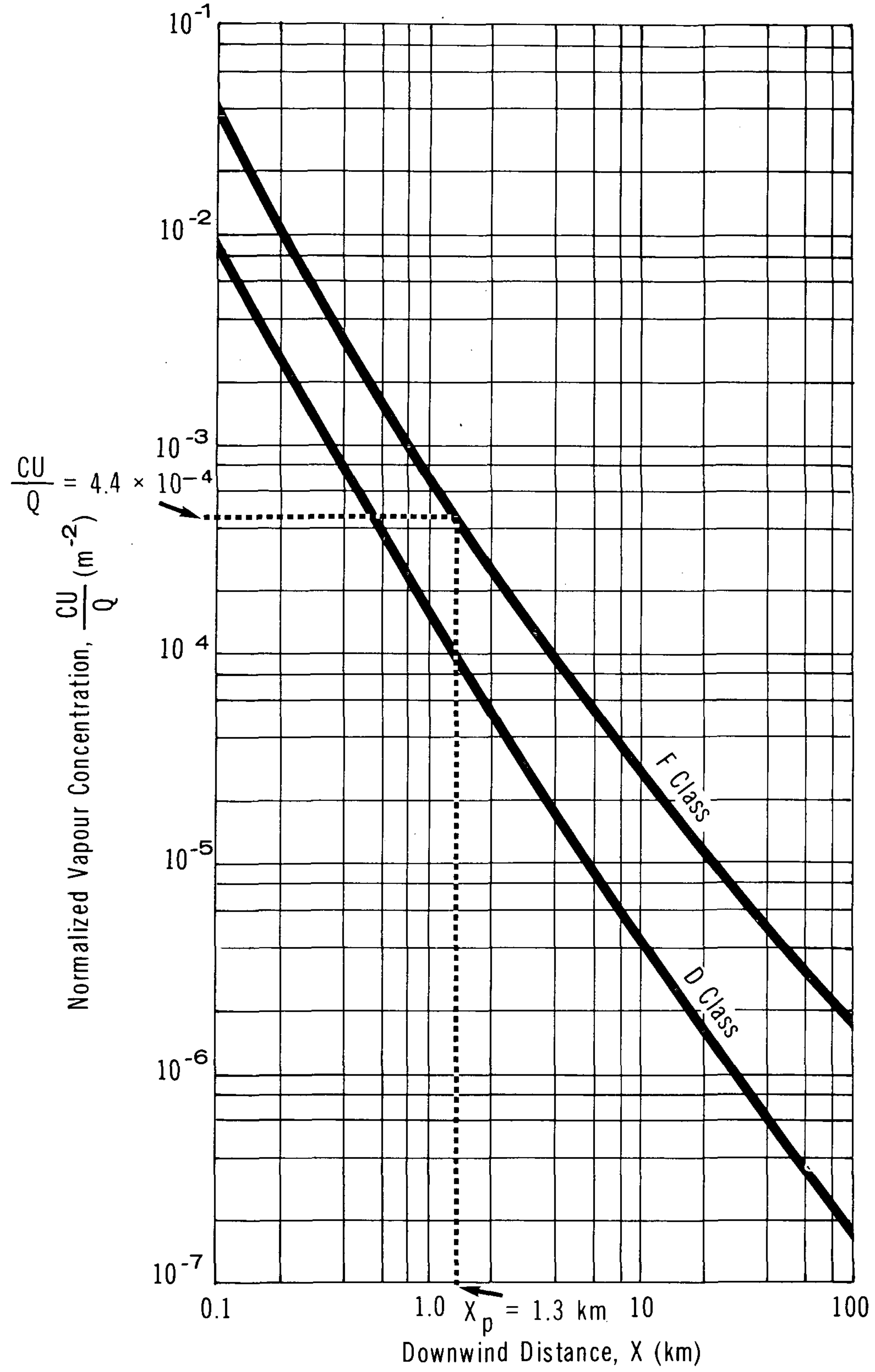
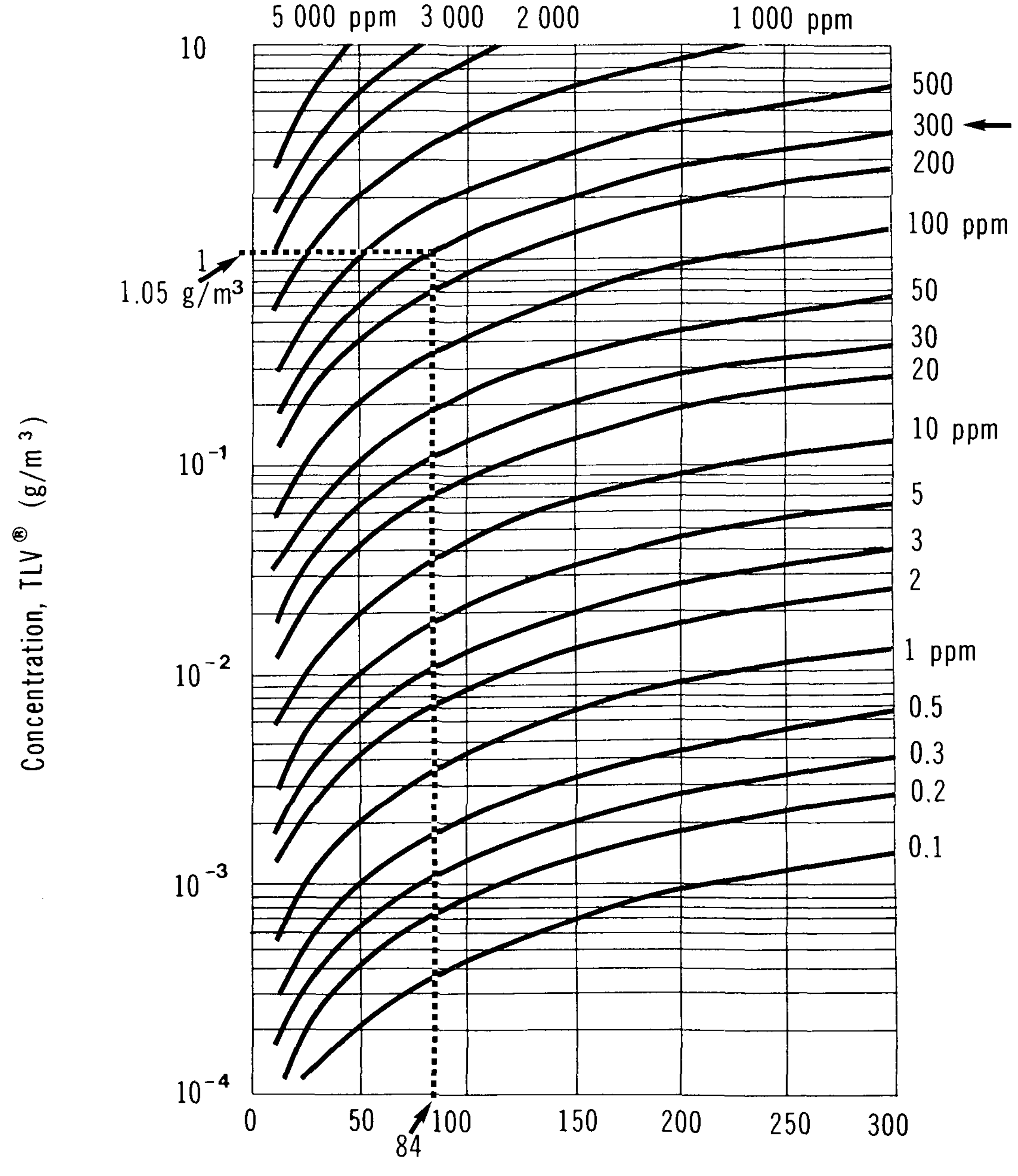


FIGURE 15

## CONVERSION OF THRESHOLD LIMIT VALUE (TLV®) UNITS (ppm to g/m³)



Molecular Weight

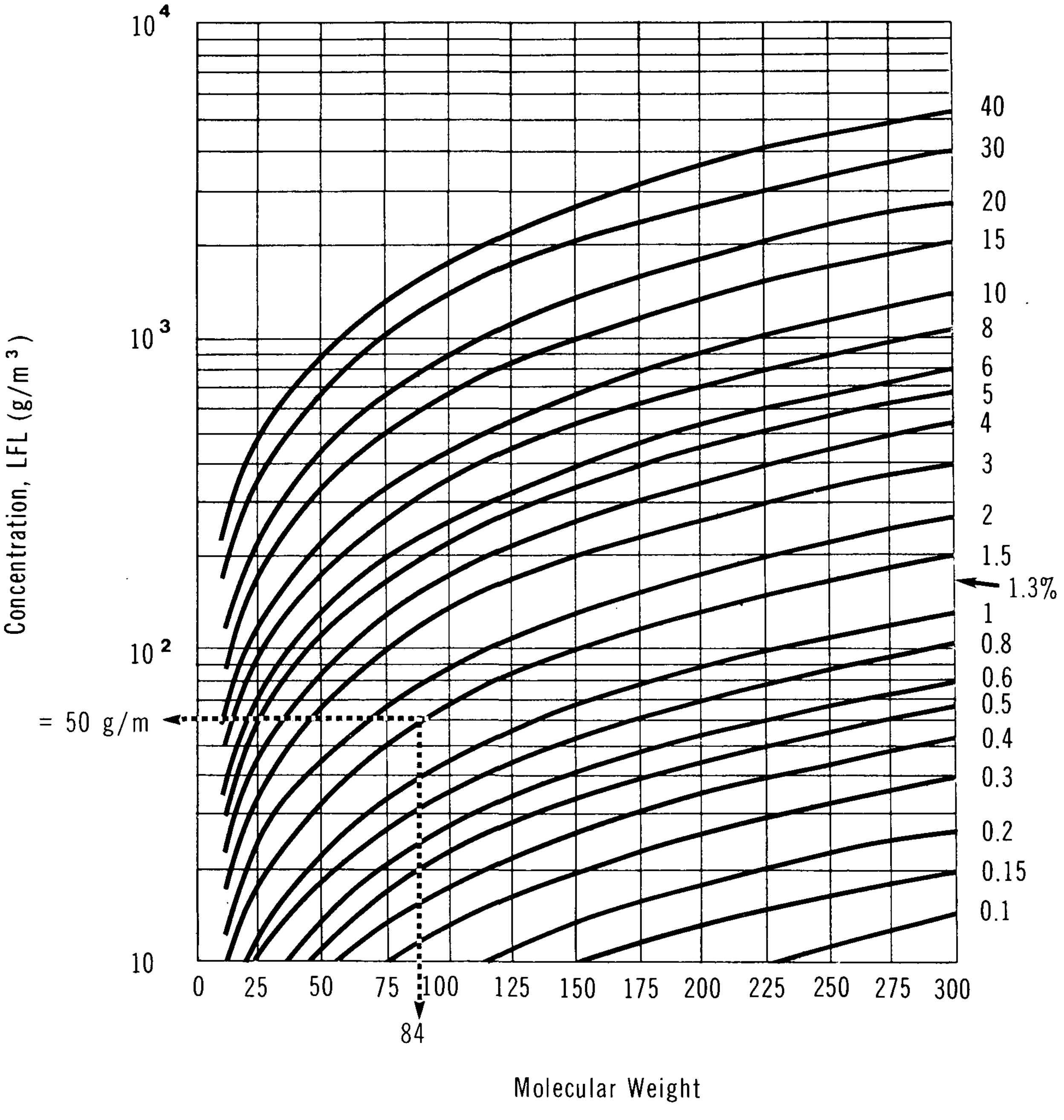
Example: Cyclohexane, MW = 84, TLV® = 300ppm, then TLV® in g/m³= 1.05

Note: data applicable at 25°C and 760 mm Hg pressure

FIGURE 16

CYCLOHEXANE

## CONVERSION OF LOWER FLAMMABILITY LIMIT (LFL) UNITS (volume % to g/m³)



Example: Cyclohexane, MW = 84, LFL = 1.3%,
then LFL in g/m³ = 50

Note: data applicable at 25°C and 760 mm Hg pressure for long-term exposure; use of this value as the hazard limit would result in unrealistically large hazard zones.

**5.3.2.3 Table 8: Maximum plume hazard half-widths.** This table presents data on the maximum plume hazard half-width, $(W/2)_{max}$, for a range of Q/U values under weather conditions D and F. These data were computed using the dispersion modelling techniques given in the Introduction Manual for a value of 10 times the cyclohexane Threshold Limit Value (TLV®) of 1.05 $g/m^3$ (10.5 $g/m^3$). The maximum plume hazard half-width represents the maximum half-width of the cyclohexane vapour cloud, downwind of the spill site, corresponding to a hazard concentration limit of 10 x TLV®. Table 8 is therefore only applicable for a cyclohexane hazard concentration limit of 10 x TLV® or 10.5 $g/m^3$. Also, data are provided up to a maximum hazard distance downwind of 100 km.

Under weather condition D, the wind speed (U) range applicable is 3 to 30 m/s. The range of vapour emission rates (Q) used was 75 000 to 65 000 000 g/s, corresponding to cyclohexane spills in the range of about 40 to greater than 10 000 tonnes, respectively. If the entire contents of an 80 000 L (17 600 Imp. gal.) tank car spills, the mass spilled would be 62 300 kg, or approximately 62 tonnes. Therefore, under class D of Table 8, data are provided for up to 160 times this amount.

Under weather condition F, the wind speed (U) range applicable is 1 to 3 m/s. The range of vapour emission rates (Q) used was 7500 to 6 500 000 g/s, corresponding to cyclohexane spills in the range of about 1 to 10 000 tonnes, respectively. Therefore, under class F of Table 8, data are provided for up to 160 times a standard rail car load.

Use: Knowing the weather condition, Q and U, compute Q/U. Choose the closest Q/U value in the table and the corresponding $(W/2)_{max}$, the maximum plume hazard half-width, in metres. (For an intermediate value, interpolate Q/U and $(W/2)_{max}$ values.) Also refer to the example at the bottom of Table 8.

**5.3.2.4 Figure 17: Plume travel time versus travel distance.** Figure 17 presents plots of plume travel time (t) versus plume travel distance ($X_t$) as a function of different wind speeds (U). This is simply the graphical presentation of the relationship $X_t = Ut$ for a range of typical wind speeds.

Use: Knowing the time (t) since the spill occurred and the wind speed (U), the distance ($X_t$) can be determined which indicates how far downwind the plume has travelled.

**5.3.3 Sample Calculation.** The following sample calculation is intended to outline the steps required to estimate the downwind hazard zone which could result from a spill

TABLE 8 MAXIMUM PLUME HAZARD HALF-WIDTHS (FOR CYCLOHEXANE AT 20°C)

| Weather Condition D | | | Weather Condition F | | |
|---|---|---|---|---|---|
| Q/U (g/m) | $(W/2)_{max}$ (m) | | Q/U (g/m) | $(W/2)_{max}$ (m) | |
| 65 000 000 | 3 180 | (99.5 km)* | 6 500 000 | 1 465 | (99.5 km)* |
| 60 000 000 | 3 025 | | 5 000 000 | 1 205 | |
| 50 000 000 | 2 700 | | 3 750 000 | 970 | |
| 25 000 000 | 1 760 | | 2 500 000 | 720 | |
| 20 000 000 | 1 530 | | 2 000 000 | 610 | |
| 15 000 000 | 1 280 | | 1 500 000 | 490 | |
| 10 000 000 | 1 000 | | 1 000 000 | 360 | |
| 5 000 000 | 650 | | 750 000 | 300 | |
| 3 750 000 | 550 | | 500 000 | 230 | |
| 2 500 000 | 435 | | 250 000 | 150 | |
| 2 000 000 | 385 | | 200 000 | 130 | |
| 1 500 000 | 325 | | 150 000 | 110 | |
| 1 000 000 | 255 | | 100 000 | 85 | |
| 750 000 | 215 | | 75 000 | 70 | |
| 500 000 | 170 | | 50 000 | 55 | |
| 250 000 | 115 | Q/U = 23 800 → | 25 000 | 35 | → $(W/2)_{max}$ = 35 m |
| 100 000 | 65 | | 10 000 | 25 | |
| 75 000 | 60 | | 5 000 | 15 | |
| 50 000 | 45 | | 2 500 | 10 | |
| 25 000 | 30 | | | | |
| 10 000 | 20 | | | | |
| 5 000 | 15 | | | | |
| 2 500 | 10 | | | | |

*Data are provided up to a maximum downwind hazard distance of 100 km.

Example: A spill releasing cyclohexane vapour at the rate of $Q = 5 \times 10^4$ g/s under weather condition F and a wind speed U = 2.1 m/s means Q/U = 23 800 g/m, which results in a maximum plume hazard half-width $(W/2)_{max}$ = 35 m.

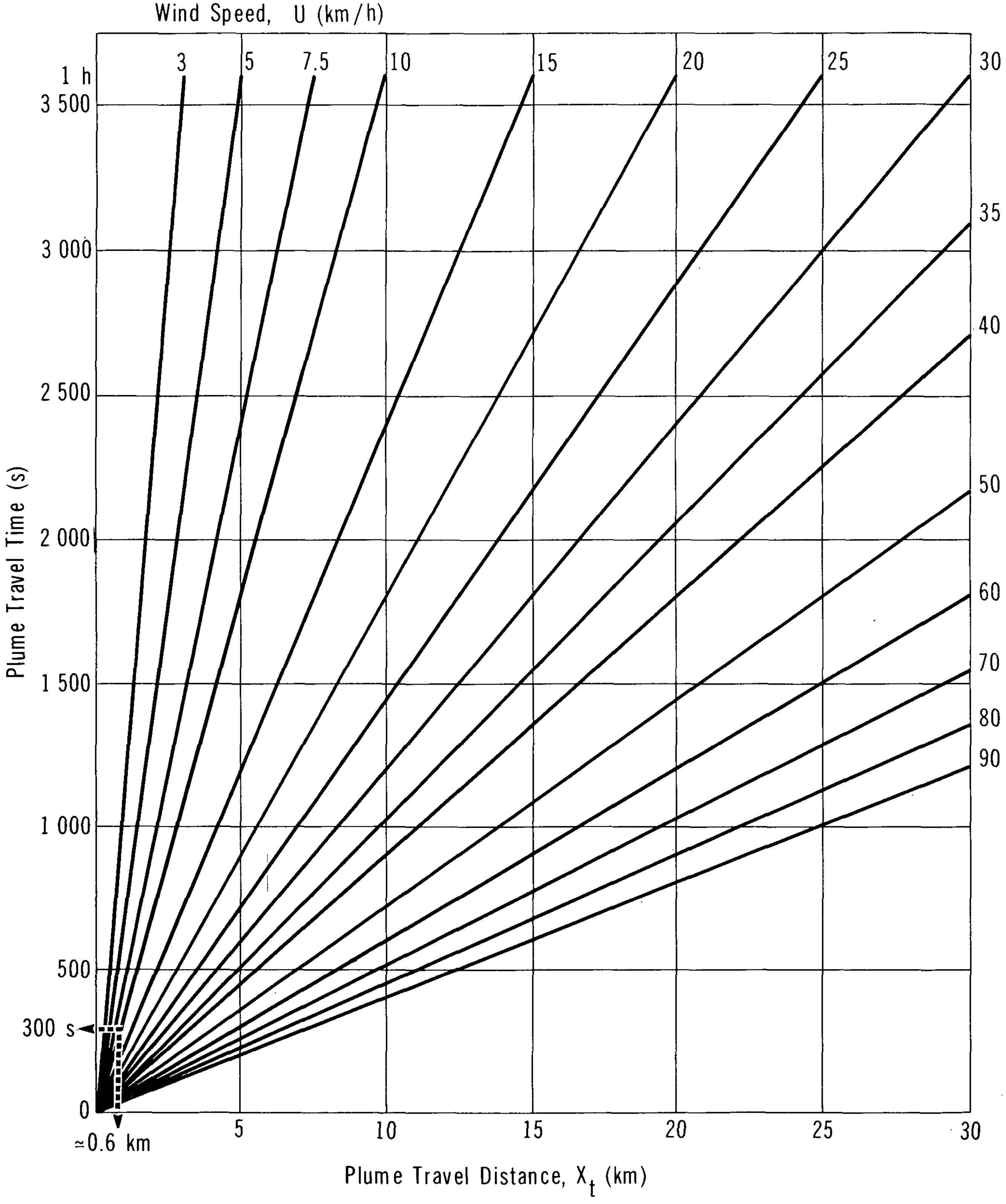
Note: Above table is valid only for a cyclohexane concentration of 10 x TLV®, or 10.5 $g/m^3$.

of liquid cyclohexane. The user is cautioned to take note of the limitations in the calculation procedures described herein and in the Introduction Manual. The estimates provided here apply only for conditions given. It is recommended that the user employ known or observational estimates (i.e., of the spill radius) in a particular spill situation if possible.

Problem:

During the night, at about 2:00 a.m., 20 tonnes of cyclohexane were spilled on

## PLUME TRAVEL TIME VS TRAVEL DISTANCE



a flat ground surface. It is now 2:05 a.m. The temperature is 20°C and the wind is from the NW at 7.5 km/h. Determine the extent of the vapour hazard zone.

Solution:

Step 1: Quantity spilled is given, $q = 20$ tonnes

Step 2: Determine the pool radius (r) for a spill of 20 tonnes

- Use observed (measured) pool radius if possible. If not, use the maximum radius from Figure 20, Section 5.4. Note that use of this data, which applies specifically to spills on water, will result in an exaggerated pool radius on land
- Radius $(r) = 80 \text{ m} \div 1000 \text{ m/km} = 0.08 \text{ km}$

Step 3: Calculate the vapour emission rate (Q) at $T = 20°C$

- From Figure 13, for $r = 80$ m and $T = 20°C$, $Q = 5.0 \times 10^4$ g/s

Step 4: Determine the wind speed (U) and direction (D)

- Use available weather information, preferably on-site observations
- Given:
  $U = 7.5$ km/h, then $U = 7.5 \div 3.6 = 2.1$ m/s
  $D$ = NW or 315° (D = Direction from which wind is blowing)

Step 5: Determine the weather condition

- From Table 7, weather condition = F since U is less than 11 km/h and it is night

Step 6: Determine the hazard concentration limit (C)

- This is the lower of 10 times the TLV®, or the LFL, so for cyclohexane
  $C = 10.5 \text{ g/m}^3$ (TLV® = $1.05 \text{ g/m}^3$; LFL = $50 \text{ g/m}^3$)

Step 7: Compute CU/Q

- $$CU/Q = \frac{10.5 \times 2.1}{5.0 \times 10^4} = 4.4 \times 10^{-4} \text{ m}^{-2}$$

Step 8: Calculate the downwind distance ($X_p$) from the virtual point source

- From Figure 14, with $CU/Q = 4.4 \times 10^{-4} \text{ m}^{-2}$ and weather condition F, $X_p \simeq 1.3$ km

Step 9: Calculate the hazard distance ($X_a$) downwind of the area source

- With $X_p = 1.3$ km and $r = 0.08$ km, then
  $X_a = X_p - 10\,r = 1.3 \text{ km} - 10\,(0.08 \text{ km}) = 0.5 \text{ km}$

Step 10: Calculate the plume hazard half-width $(W/2)_{max}$

- Use Table 8

- With $Q = 5.0 \times 10^4$ g/s and $U = 2.1$ m/s

  then $Q/U = \dfrac{5.0 \times 10^4}{2.1} = 23\ 800$ g/m
- Then for weather condition F, the closest Q/U value is 25 000 g/m, which gives $(W/2)_{max} \simeq 35$ m

Step 11: Determine the time since the spill

- t = 5 min x 60 s/min = 300 s

Step 12: Calculate the distance travelled ($X_t$) by the vapour plume since the time of the accident

- Using Figure 17, with t = 300 s and U = 7.5 km/h, then $X_t = 0.6$ km (more accurately from Ut = 2.1 m/s x 300 s = 630 m = 0.63 km)

Step 13: Map the hazard zone

- This is done by drawing a rectangular area with dimensions of twice the maximum plume hazard half-width (35 m) by the maximum hazard distance downwind of the area source (0.5 km) along the direction of the wind, as shown in Figure 18
- If the wind is reported to be fluctuating by 20° about 315° (or from 315 ± 10°), the hazard zone is defined as shown in Figure 19
- Note that for a wind speed of 7.5 km/h, the cyclohexane vapour plume will have travelled 0.63 km in the 5 minutes since the spill occurred and that the downwind hazard distance is only 0.5 km

## 5.4 Behaviour in Water

**5.4.1 Introduction.** When spilled on water, cyclohexane will both vaporize and spread on the surface. Because it is relatively insoluble in water, only a very small amount will be dissolved, the rest evaporating until it is removed.

For the purpose of nomogram preparation, the extent of spread on the surface of the water has been estimated, assuming that none of the cyclohexane is dissolved in the water. However, the loss due to vaporization has been taken into account.

The rate of spreading on water is based on the balance of forces tending to spread the liquid (gravity and surface tension) and those tending to resist spreading (inertial and viscous forces). The maximum size of the spill pool depends to a large extent on the rate of vaporization.

The equations representing the spreading of the spill on water are presented in the Introduction Manual. For the purposes of the nomograms presented, the water

FIGURE 18

CYCLOHEXANE

## HAZARD AREA FOR STEADY WINDS, EXAMPLE PROBLEM

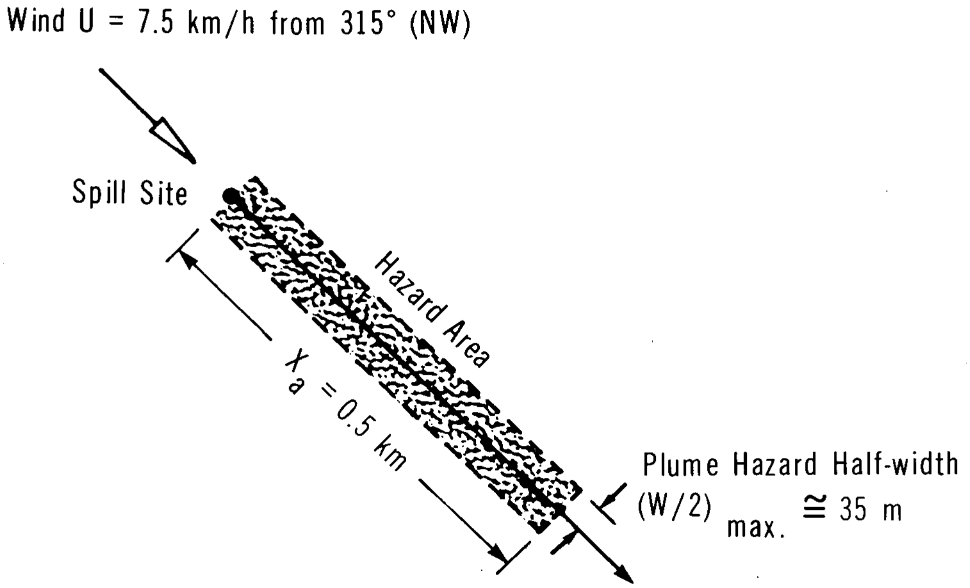
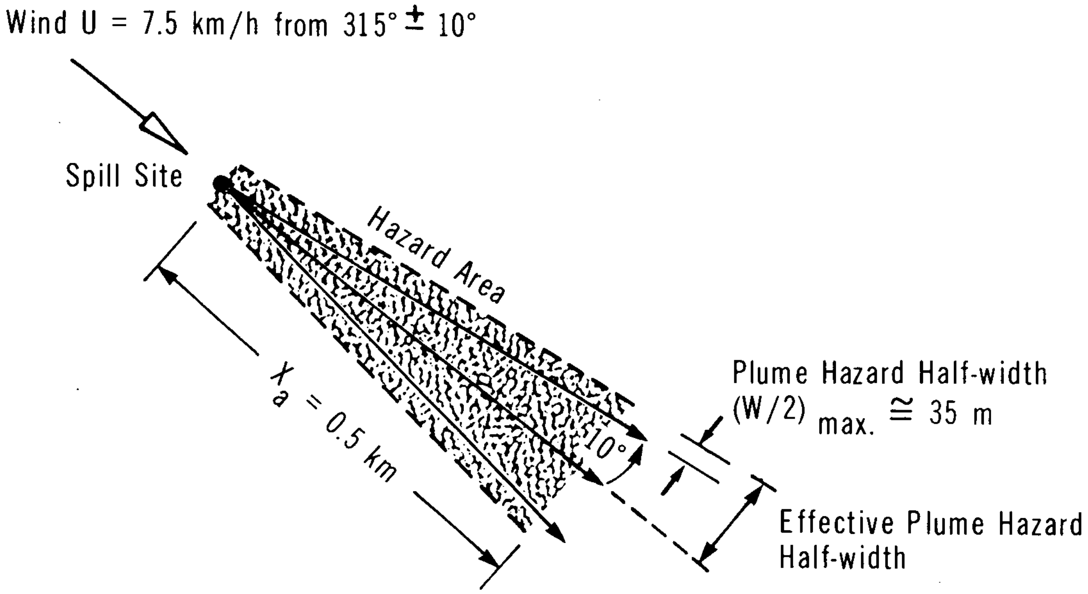


FIGURE 19

CYCLOHEXANE

## HAZARD AREA FOR UNSTEADY WINDS, EXAMPLE PROBLEM



$$= X_a \times 1000 \times \tan 10° + (W/2)_{max}$$

$$= 0.5 \times 1000 \times \tan 10° + 35 \text{ m}$$

$$= 125 \text{ m}$$

temperature has been taken as 20°C, representing a reasonable maximum for surface water bodies. This condition maximizes the spill size.

### 5.4.2 Nomogram for Spreading on Still Water.

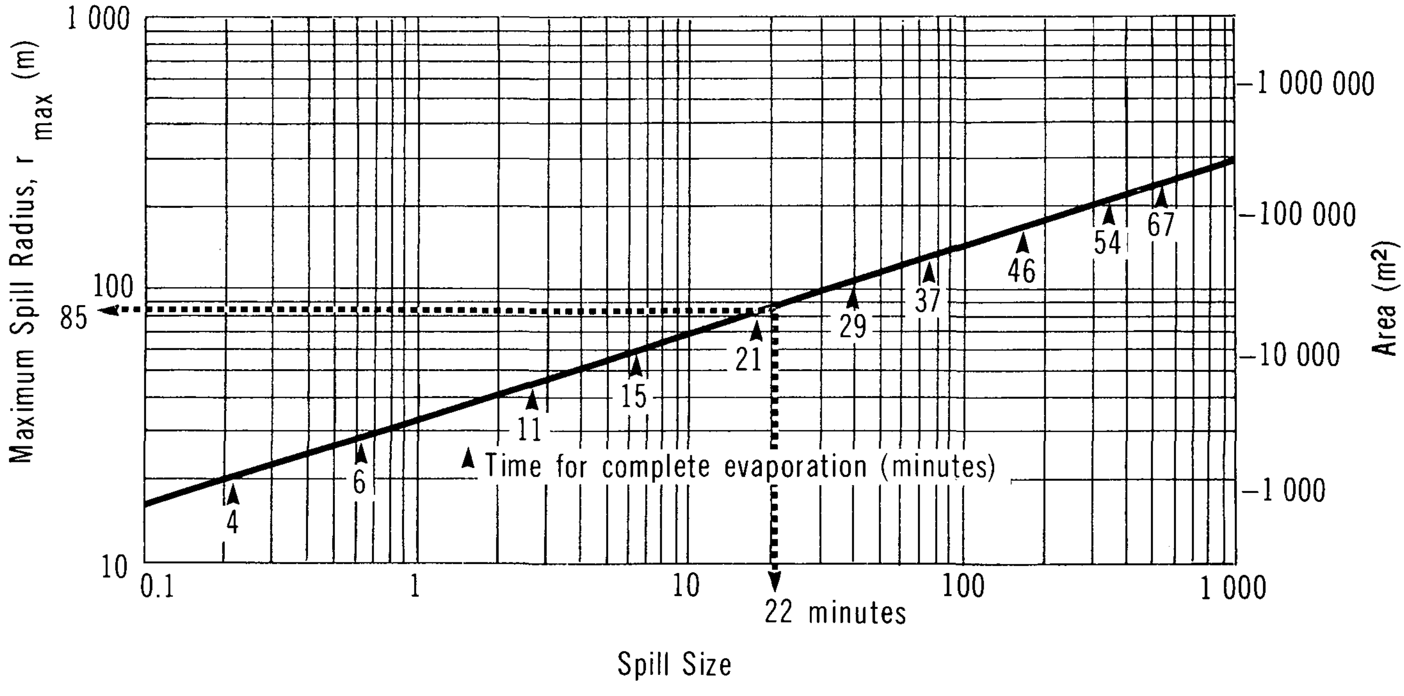
Figure 20 is presented to simplify the calculation of spreading on still water (without dissolution) and to estimate the time for complete evaporation for a range of spill sizes.

Assuming no dissolution in water, Figure 20 provides a simple means of estimating the maximum spill radius for cyclohexane, if the spill size is known. The nomogram is based on data presented in the Hazard Assessment Handbook (CHRIS 1974) and a computer model for simultaneous spreading and evaporation of a liquid spilled on water (Raj 1974). The arrow points on the nomogram provide an estimate of time for complete evaporation of the spill. Because of the short times involved, the complete time history of the spread of the spill has not been considered. Similarly, the translation distance of the spill by wind or surface current is not considered.

FIGURE 20

CYCLOHEXANE

**MAXIMUM SPILL RADIUS vs SPILL SIZE**



### 5.4.3 Sample Calculation.

A 20 tonne spill of cyclohexane has occurred on a large lake. What is the maximum size of the spill (assuming no dissolution) and approximate time for complete evaporation?

Solution

- Use Figure 20
- With spill mass = 20 tonnes, $r_{(max)}$ = 85 m
- Time for complete evaporation is about 22 min

## 5.5 Subsurface Behaviour: Penetration into Soil

**5.5.1 Introduction.** Cyclohexane has a very low solubility in water. Consequently, when spilled onto soil, infiltration and transport downward through the soil involve multiphase phenomena. The phases of concern are liquid cyclohexane, water, soil and gas or vapours.

Unfortunately, sufficient data do not exist to permit a detailed assessment of contaminant transport in a specific circumstance. A few extensive field investigations have been carried out, especially involving spills of oil, gasoline and PCBs. However, very limited information exists for cyclohexane. Consequently, it is necessary to simplify the soil and groundwater conditions and to express contaminant behaviour through analogy to other more extensively studied materials. A pattern for the downward movement of insoluble fluids such as cyclohexane in soil has been prepared by comparison to oil spilled onto soil surfaces (Blokker 1971; Freeze and Cherry 1979).

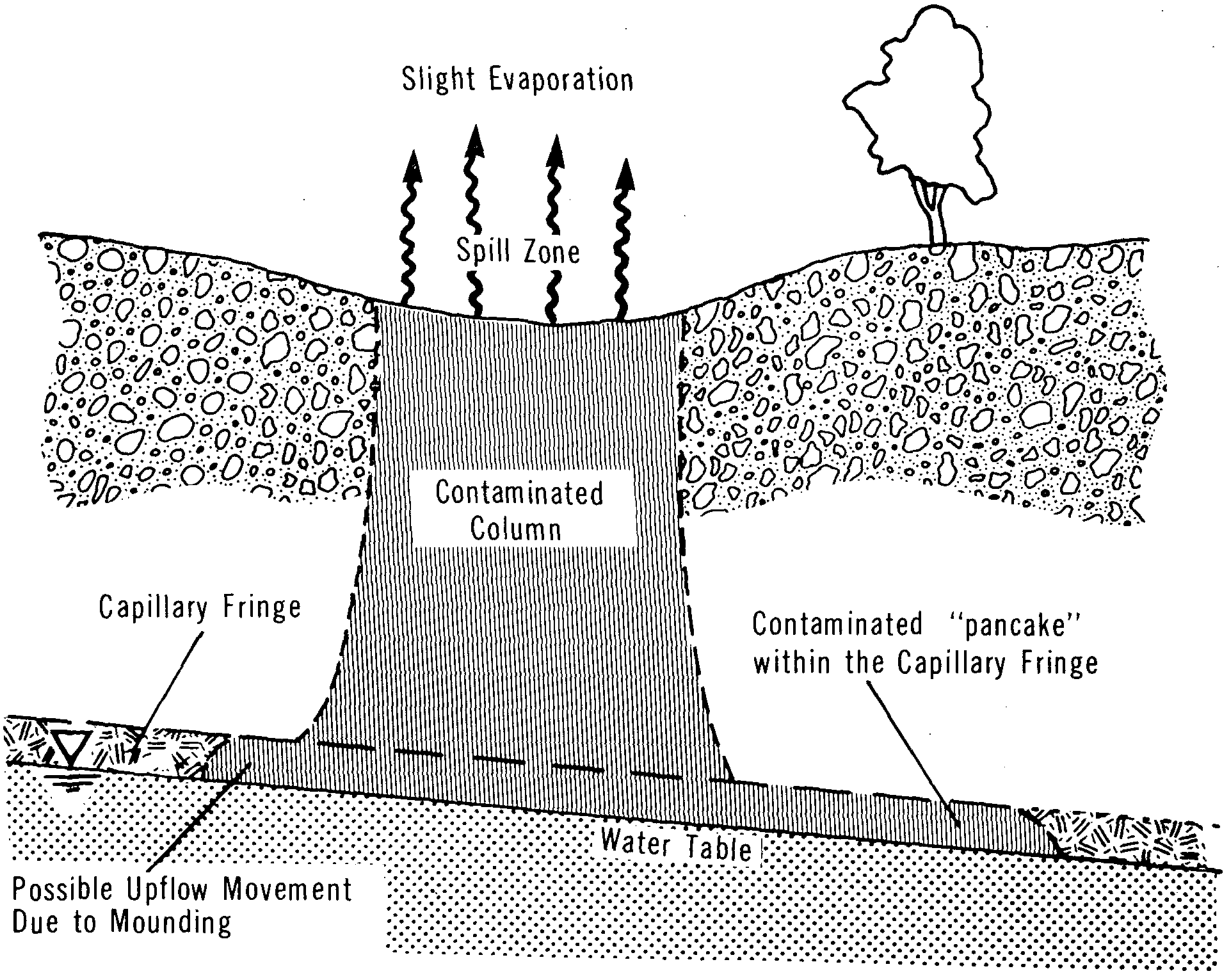
It is assumed that when the spill occurs, the soil contains water only up to its field capacity (the maximum amount of water the soil can hold after the excess is drained off) and that this condition prevails down to the groundwater table. The spilled cyclohexane fills the pores at the soil surface and begins to penetrate downward. It is assumed that the liquid moves downward through the soil as a saturated slug, but leaving behind a constant residual amount ($S_o$) within the soil pores. Downward transport will continue until the volume of cyclohexane spilled per area ($B_o$) equals the amount retained in the soil as $S_o$. Some lateral spreading may occur due to capillary action. If $B_o$ is greater than the volume that can be retained as $S_o$ above the groundwater table, the excess cyclohexane will spread as a pancake within the saturated groundwater capillary fringe. The resultant contaminated zone consists of a "vertical column" and "horizontal" pancakes of soil containing the residual amount of cyclohexane, $S_o$. This is shown schematically in Figure 21.

**5.5.2 Equations Describing Cyclohexane 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.

FIGURE 21

CYCLOHEXANE

## SCHEMATIC SOIL TRANSPORT



Soil: Coarse Sand

–Porosity (n) = 0.35

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

–Field Capacity ($\theta_{fc}$) = 0.075

**5.5.3 Saturated Hydraulic Conductivity of Cyclohexane 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 ($m^2$)

$\rho$ = mass density of the fluid ($kg/m^3$)

$\mu$ = absolute viscosity of the fluid (Pa•s)

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

The appropriate properties of cyclohexane are given in the chart below:

| Property | Cyclohexane 20°C |
|---|---|
| Mass density ($\rho$), $kg/m^3$ | 779 |
| Absolute viscosity ($\mu$), Pa•s | $0.94 \times 10^{-3}$ |
| Saturated hydraulic conductivity ($K_o$), m/s | $(8.1 \times 10^6)k$ |

**5.5.4 Soils.** Three soils were selected for this work. Their relevant properties are:

| | Soil Type | | |
|---|---|---|---|
| Property | 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 ($\Theta_{fc}$), $m^3/m^3$ | 0.075 | 0.3 | 0.45 |
| Residual fraction ($S_o$), $m^3/m^3$ | 0.05 | 0.1 | 0.2 |

**5.5.5 Penetration Nomograms.** Nomograms for the penetration of cyclohexane into the unsaturated zone above the groundwater table were prepared for each soil. The nomograms show the total depth of cyclohexane penetration (B) versus penetration time ($t_p$) for various volumes spilled per unit area of soil ($B_o$). A temperature of 20°C was used. Calculations were based on the equations developed in the Introduction Manual. A flowchart for use of the nomograms is shown in Figure 22. The nomograms are presented in Figures 23, 24 and 25.

**5.5.6 Sample Calculation.** A 20 tonne spill of cyclohexane has occurred on coarse sandy soil. The temperature is 20°C; the spill radius is approximately 8.6 m. Calculate the depth of penetration 12 minutes after the accident.

Solution

Step 1: Define parameters

- Mass spilled = 20 000 kg (20 tonnes)
- $T = 20°C$
- Mass density ($\rho$) = 779 kg/m$^3$
- $t = 12$ min

Step 2: Estimate the depth of penetration (B)

- For coarse sand, use Figure 23
- $B = 5.8$ m

FIGURE 22

# FLOWCHART FOR NOMOGRAM USE

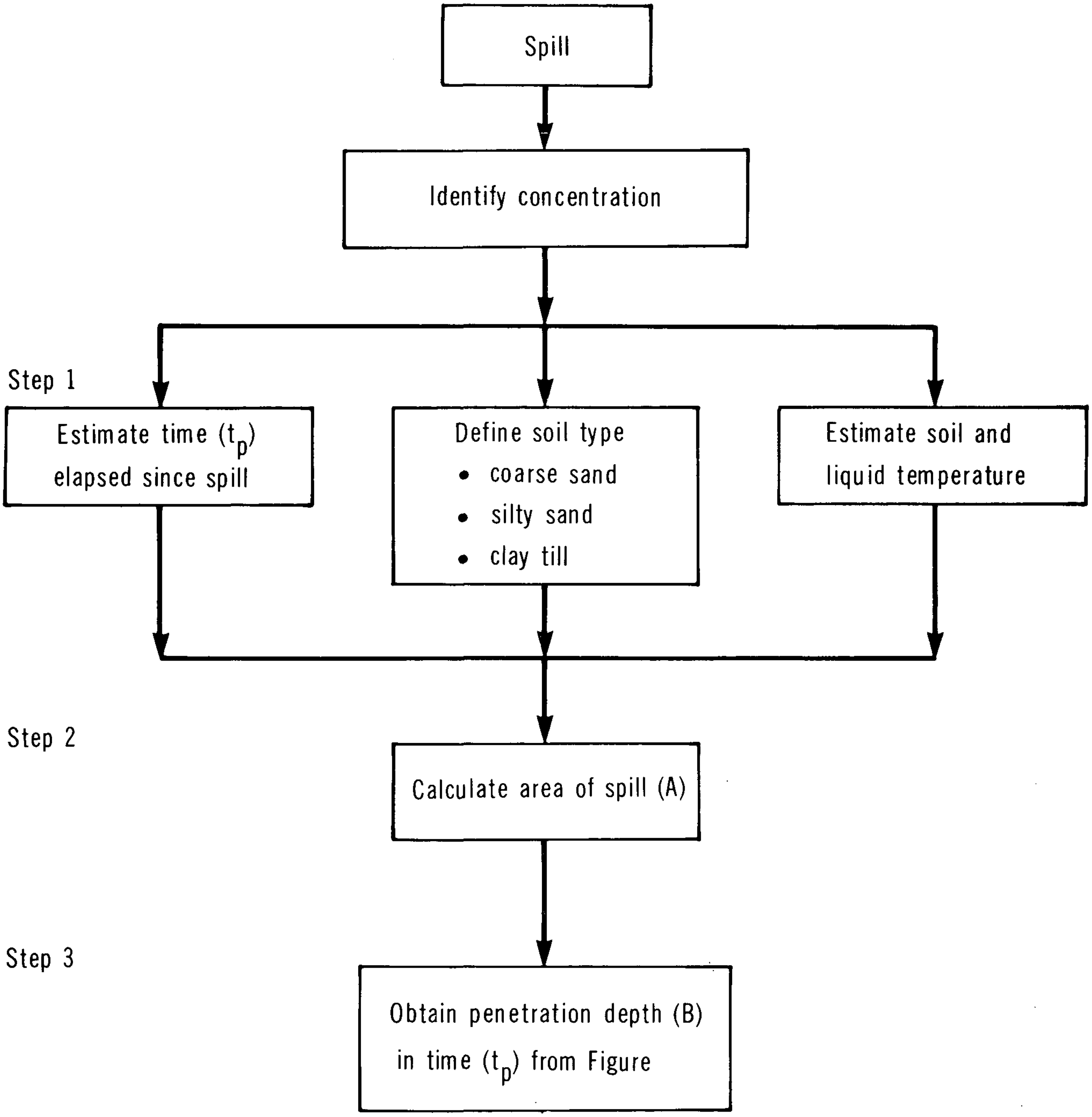
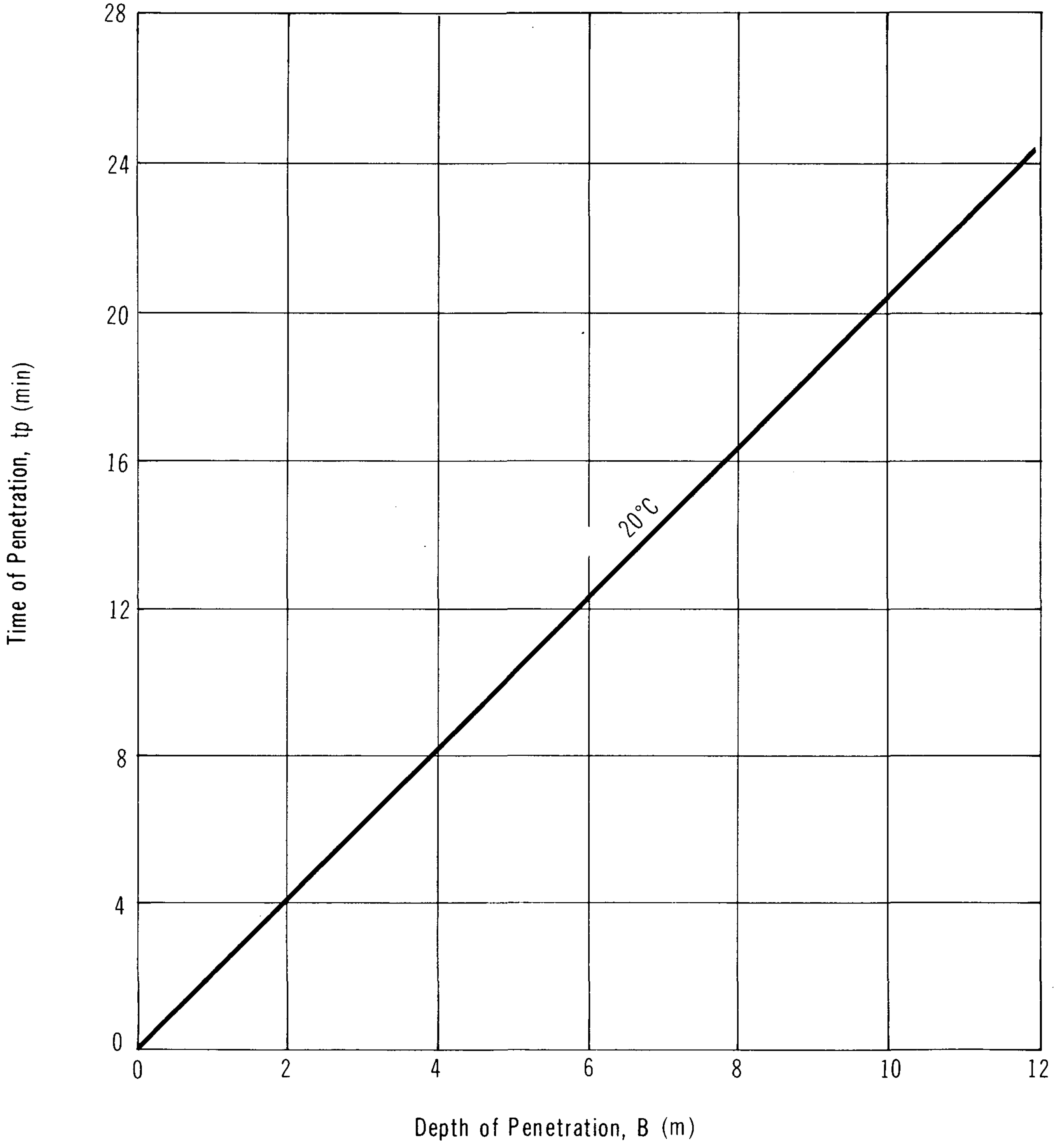


FIGURE 23

CYCLOHEXANE

## PENETRATION IN COARSE SAND



## PENETRATION IN SILTY SAND

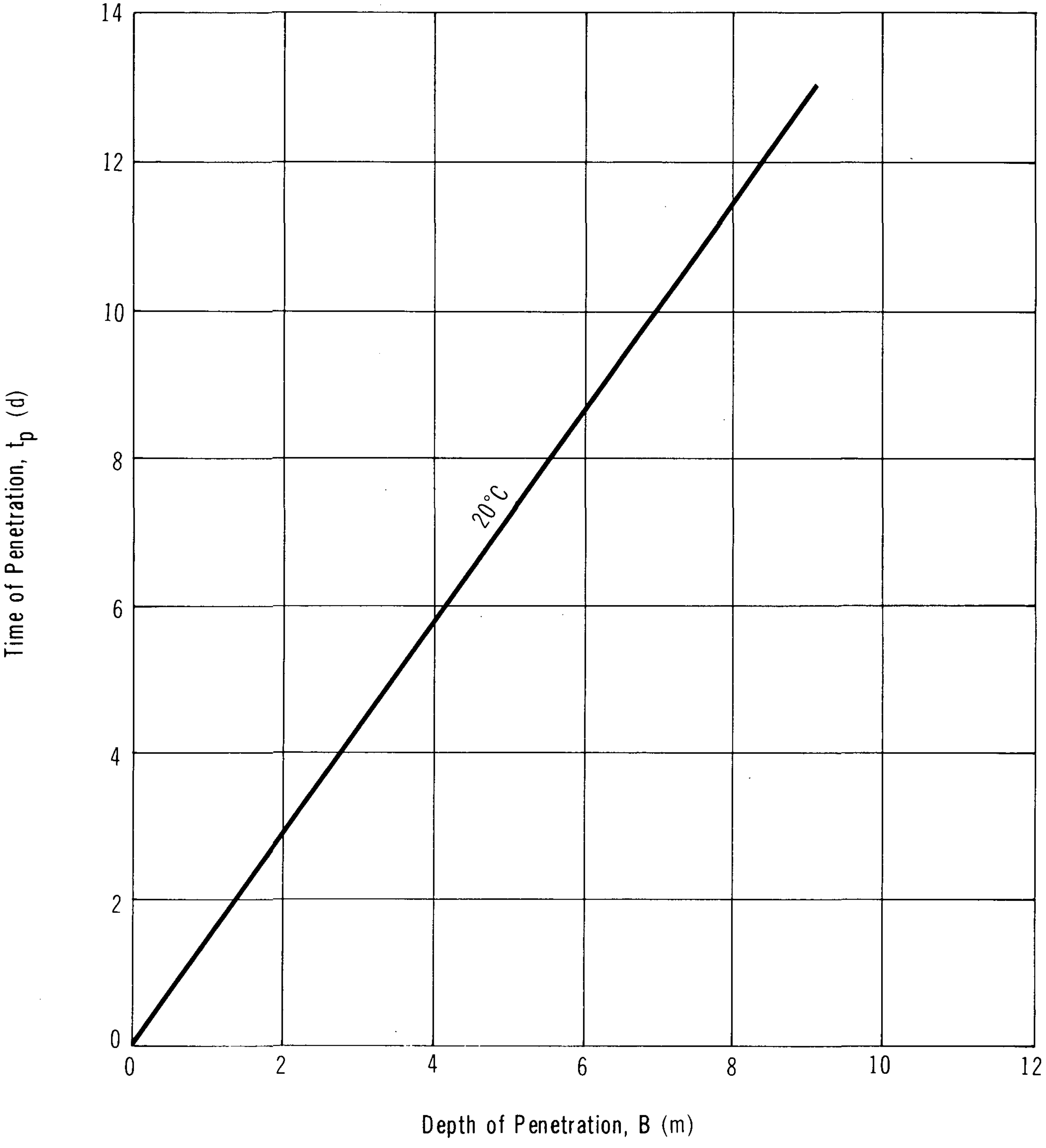
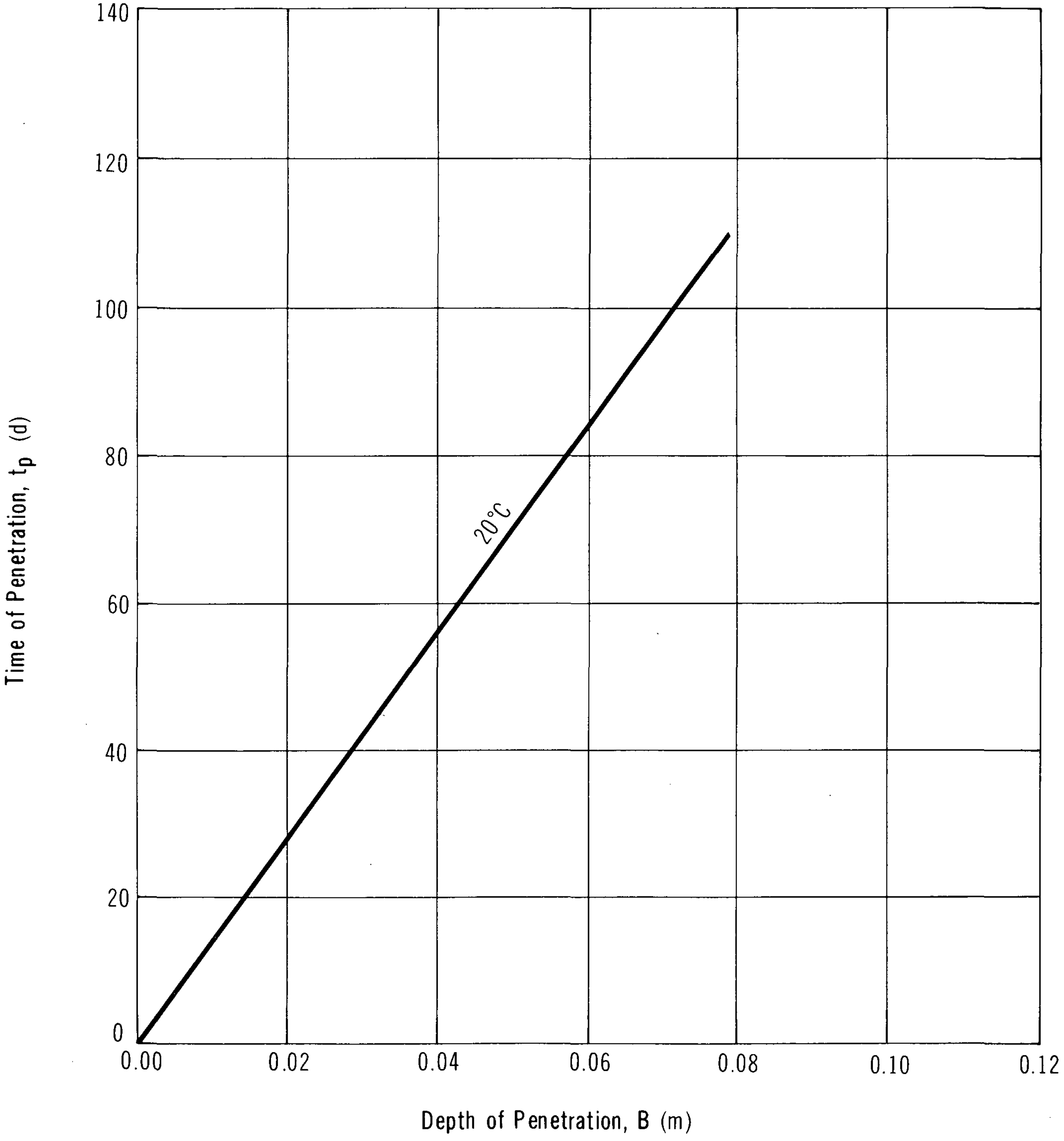


FIGURE 25

CYCLOHEXANE

## PENETRATION IN CLAY TILL



# 6 ENVIRONMENTAL DATA

## 6.1 Suggested or Regulated Limits

**6.1.1 Water.** No specific recommendations have been made in Canada; the toxicological limit in the USSR is 0.1 mg/L (Verschueren 1984).

**6.1.2 Air.** Although workplace limits have been set (the 8-hour TLV® is 1050 mg/m$^3$), no environmental recommendations have been specifically made.

## 6.2 Aquatic Toxicity

**6.2.1 U.S. Toxicity Rating.** Cyclohexane has been assigned a $TL_m96$ of 10 to 100 ppm (RTECS 1979).

**6.2.2 Measured Toxicities.**

| Conc. mg/L | Time (hours) | Species | Result | Water Conditions | Reference |
|---|---|---|---|---|---|
| Freshwater Fish | | | | | |
| 42.3 | 24 | Bluegill | $TL_m$ | 25°C | Middlebrooks 1973 |
| 31 | 96 | Bluegill | $LC_{50}$ | - | WQCDB 1971 |
| 34.7 | 96 | Bluegill | $TL_m$ | 25°C | Middlebrooks 1973 |
| 95 | 24 | Fathead minnow | $LC_{50}$ | static, 18-22°C, Lake Superior water | Mattson 1976 |
| 126 | 24 | Fathead minnow | $LC_{50}$ | static, 18-22°C, reconstituted water | Mattson 1976 |
| 93 | 48, 72, 96 | Fathead minnow | $LC_{50}$ | static, 18-22°C, Lake Superior water | Mattson 1976 |
| 117 | 48, 72, 96 | Fathead minnow | $LC_{50}$ | static, 18-22°C, reconstituted water | Mattson 1976 |
| 32 to 43 | 24-96 | Fathead minnow | $LC_{50}$ | - | Verschueren 1984 |
| 30 | 96 | Fathead minnow | $LC_{50}$ | - | Pickering 1966 |

| Conc. mg/L | Time (hours) | Species | Result | Water Conditions | Reference |
|---|---|---|---|---|---|
| 42.3 | 24-96 | Goldfish | $TL_m$ | 25°C, soft | Middlebrooks 1973 |
| 33 | 96 | Goldfish | $LC_{50}$ | - | WQCDB 1971 |
| 57.7 | 24-96 | Guppy | $TL_m$ | 25°C, soft | Middlebrooks 1973 |
| 48 | 96 | Guppy | $LC_{50}$ | - | WQCDB 1971 |
| >84 | 168 | Guppy | $LC_{50}$ | - | Verschueren 1984 |
| Microorganisms | | | | | |
| 50 | tns | Protozoa (*Uronema parduczi*) | threshold inhibition of cell multiplication | - | Verschueren 1984 |
| Saltwater Species | | | | | |
| 1 to 100 | tns | Mussel larvae (*Mytilus edulis*) | 10 to 20 percent increase in growth | - | Verschueren 1984 |
| 100 | 96 | Young coho salmon | no significant mortalities | 8°C | Verschueren 1984 |

## 6.3 Mammalian Toxicity

The $LC_{50}$ (1 h) for mice is 18 000 ppm. The "no effect" level for rabbits is 786 ppm over 10 weeks (Verschueren 1984).

The "acute toxicity ranking", based on $LD_{50}$ for oral administration to mammals, is 2500-5000 mg/kg body weight; 400 mg/kg administered to rats resulted in reduced catalase and cholinesterase activities (WQCDB-1 1970).

## 6.4 Other Land and Air Toxicity

Waterfowl may be adversely affected by cyclohexane slicks (OHM-TADS 1981).

## 6.5 Degradation

Cyclohexane is not subject to rapid biodegradation but will quickly volatilize from water (OHM-TADS 1981). The BOD in 25-35 days is 2.39 in seawater, and the theoretical oxygen demand is 3.42 (both in weight to weight ratios). In one study, the biodegradation in groundwater was found to be 45 percent after 192 hours with an initial concentration of 0.12 μL/L (Verschueren 1984). Cyclohexane degrades to cyclohexanol, then to cyclohexanone, subsequently to oxy-heptanes and hexanes, and then to adipic acid (Verschueren 1984).

## 6.6 Long-term Fate and Effects

Cyclohexane may be persistent in the environment, but no bioconcentration or food chain concentration effects have been cited. The half-life in the atmosphere from an initial concentration of 10 ppm is about 7 hours (Jorgensen 1979).

# 7 HUMAN HEALTH

Cyclohexane is considered to be a compound of low toxicity (MCA 1957; Doc. TLV 1981). Its principle toxic effects are local in nature although it may produce a narcotic effect in humans (Patty 1981) and act as general central nervous system depressant. The compound is not considered to have good warning properties. Benzene is a common impurity in solvent grade cyclohexane (MCA 1957).

No recently published reports pertaining to the toxicity of cyclohexane were found in the literature. No monographs or toxicity studies were found concerning to the compound's potential as a carcinogen or teratogen. Cyclohexane is reported in the EPA TSCA Inventory. The toxicological data summarized here have been extracted from reliable standard reference sources and are representative of information in the literature.

## 7.1 Recommended Exposure Limits

The exposure standards for cyclohexane are based upon its central nervous system and narcotic effects; however, the standards are borderline in the prevention of irritation (Doc. TLV 1981). Canadian provincial guidelines generally are similar to those of the USA-ACGIH, unless indicated otherwise.

| Guideline (Time) | Origin | Recommended Level | Reference |
|---|---|---|---|
| Time-weighted Averages (TWA) | | | |
| TLV® (8 h) | USA-ACGIH | 300 ppm (1050 mg/m$^3$) | TLV 1983 |
| PEL (8 h) | USA-OSHA | 300 ppm (1050 mg/m$^3$) | NIOSH/OSHA 1981 |
| Short-term Exposure Limits (STEL) | | | |
| STEL (15 min) | USA-ACGIH | 375 ppm (1300 mg/m$^3$) | TLV 1983 |
| Other Human Toxicities | | | |
| IDLH | USA-NIOSH/ OSHA | 10 000 ppm | NIOSH Guide 1978 |

### Inhalation Toxicity Index

The Inhalation Toxicity Index (ITI) is a measure of the potential of a substance to cause injury by inhalation. It is calculated as follows:

ITI = 1315.12 (Vapour Pressure, in mm Hg/TLV ®, in ppm)

At 25°C, ITI = 1315.12 (99.8 mm Hg/300 ppm)

At 25°C, ITI = $4.37 \times 10^2$

## 7.2 Irritation Data

### 7.2.1 Skin Contact.

| Exposure Level (and Duration) | Effects | Reference |
|---|---|---|
| SPECIES: Human | | |
| Unspecified | Prolonged or repeated exposures can cause skin rashes. | NIOSH/OSHA 1981 |
| 300 ppm | Irritation to skin and eyes. | Verschueren 1984 |
| SPECIES: Rabbit | | |
| 1 548 mg (2 d, intermittently) | Irritation. | RTECS 1979 |

### 7.2.2 Eye Contact.

| Exposure Level (and Duration) | Effects | Reference |
|---|---|---|
| SPECIES: Human | | |
| 5 ppm | Irritation. | RTECS 1979 |

## 7.3 Threshold Perception Properties

### 7.3.1 Odour.

| | |
|---|---|
| Odour Characteristic: | Pungent (Doc. TLV 1981). Mild, sweet (resembling chloroform) (MCA 1957). |
| Odour Index: | 203 000 (Verschueren 1984) |

| Parameter | Media | Concentration | Reference |
|---|---|---|---|
| Detection Threshold | in air | 0.32 to 370 ppm | Verschueren 1984 |
| Recognition Threshold | in air | 32 ppm | Verschueren 1984 |
| Threshold | in air | 25 ppm | Amoore 1983 |
| Threshold | in water | 0.011 ppm | Amoore 1983 |

### 7.3.2 **Taste.** No data.

## 7.4 Toxicity Studies

### 7.4.1 Inhalation.

| Exposure Level (and Duration) | Effects | Reference |
|---|---|---|
| Acute Exposures | | |
| SPECIES: Human | | |
| 500 000 ppm | May rapidly produce unconsciousness. Death might occur from respiratory failure. | MCA 1957 |
| 13 000 ppm | Dizziness with nausea and vomiting. In the exceptional case, a mild stimulation, such as that from surgical anaesthetics, has been known to occur. | MCA 1957 |
| 300 ppm | Detectable by odour, and somewhat irritating to the eyes and mucous membranes. | Doc. TLV 1981 |
| SPECIES: Rabbit | | |
| 26 600 ppm (1 h) | Lethal | Doc. TLV 1981 |
| 18 500 ppm (8 h) | Survival | Doc. TLV 1981 |
| 12 600 ppm | Lethargy, narcosis, increased respiration rate and convulsions. | Doc. TLV 1981 |
| 3300 ppm | No effect. | Doc. TLV 1981 |
| SPECIES: Mouse | | |
| 18 000 ppm (1 h) | $LC_{50}$ | AAR 1981 |

### 7.4.2 Ingestion.

| Exposure Level (and Duration) | Effects | Reference |
|---|---|---|
| Acute Exposures | | |
| SPECIES: Human | | |
| 19.5 mg/kg | Suggested maximum oral dose. | AAR 1981 |
| SPECIES: Mammals | | |
| 2500 to 5000 mg/kg | $LD_{50}$ | RTECS 1979 |
| SPECIES: Rabbit | | |
| 5500 mg/kg | $LD_{LO}$ | RTECS 1979 |
| SPECIES: Rat | | |
| 29 820 mg/kg | $LD_{50}$ | RTECS 1979 |
| 5600 mg/kg | $LD_{50}$ (young rats) | AAR 1981 |
| 1297 mg/kg | $LD_{50}$ | RTECS 1979 |
| 813 mg/kg | $LD_{LO}$ | AAR 1981 |
| 83 mg/kg | $LD_{LO}$ | AAR 1981 |

### 7.4.3 Intravenous.

| Exposure Level (and Duration) | Effects | Reference |
|---|---|---|
| SPECIES: Rabbit | | |
| 77 mg/kg | $LD_{LO}$ | RTECS 1979 |

**7.4.4 Mutagenicity, Teratogenicity and Carcinogenicity.** No mutagenicity was shown in a test with salmonella; revertants were less than 0.006 per nanomole (Verschueren 1984).

## 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.

### 7.5.1 Inhalation.

1. Irritation to eyes and mucous membranes (Doc. TLV 1981).
2. Dizziness.
3. Nausea.
4. Vomiting.
5. Central nervous system depression (MCA 1957).
6. Unconsciousness (CHRIS 1978).
7. Respiratory failure.
8. Death (MCA 1957).

### 7.5.2 Ingestion.

1. Dizziness (AAR 1981).
2. Nausea.
3. Vomiting.
4. Loss of consciousness (AAR 1981).

### 7.5.1 Skin Contact.

1. Irritation
2. Severe dermatitis following prolonged contact (AAR 1981).

### 7.5.4 Eye Contact.

1. Irritation (AAR 1981).

## 8 CHEMICAL COMPATIBILITY

### 8.1 Compatibility of Cyclohexane 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 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| <u>GENERAL</u> | | | | | | | | | | | | | | |
| Fire | | ● | ● | | | | | | | | | | Flammable liquid, moderate explosion hazard | Sax 1979 |
| Heat | | ● | | | | | | | | | | | | Sax 1979 |
| <u>SPECIFIC CHEMICALS</u> | | | | | | | | | | | | | | |
| Dinitrogen Tetraoxide | | | ● | | | | | | | | | | With hot cyclohexane | Bretherick 1979 |
| Nitrogen Dioxide | | | ● | | | | | | | | | | With liquid nitrogen dioxide | NFPA 1978 |
| <u>CHEMICAL GROUPS</u> | | | | | | | | | | | | | | |
| Oxidizing Agents | ● | ● | | | | | | | | | | | | EPA 600/2-80-076 |

# 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 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 in fact be correct for different situations. The following procedures should not be considered as Environment Canada's recommendations.

**9.1.1 Fire Concerns.** Cyclohexane is a highly flammable liquid. Its vapours can readily form explosive mixtures with air and may flow along surfaces to distant sources of ignition and flash back. Burning products may include toxic carbon monoxide (GE 1978). Container may explode in heat of fire (ERG 1980).

**9.1.2 Fire Extinguishing Agents.** Use water spray to cool containers involved in a fire to prevent rupture or explosion (ERG 1980). Water spray may be ineffective in putting out fire due to the spreading it causes (NFPA 1978).

| | |
|---|---|
| Small fires: | Dry chemical, $CO_2$, water spray or alcohol foam. |
| Large fires: | Water spray, fog or alcohol foam. |

Move containers from fire area if this can be done without risk. Stay away from tank ends. For massive fires, use unmanned hose holder or monitor nozzles (ERG 1980).

### 9.1.3 Spill Actions.

**9.1.3.1 General.** Stop or reduce discharge of material if this can be done without risk. Eliminate all sources of ignition. Avoid skin contact and inhalation (GE 1978). A fluorocarbon water foam can also be applied to diminish vapour and fire hazard (EPA 670/2-75-042). Cellosize and Hycar, absorbent materials, have shown possible applicability for vapour suppression and/or containment of cyclohexane spills (ICI 1982).

**9.1.3.2 Spills on land.** For small spills, adsorb spilled material on vermiculite, dry sand or other sorbents and pick up for disposal (GE 1978). For large spills, contain if possible by forming mechanical barriers to prevent spreading. Remove with pumps or vacuum equipment. Application of fly ash, cement powder, or other sorbents to absorb the liquid bulk is recommended for final cleanup (EPA 670/2-75-042).

**9.1.3.3 Spills on water.** Contain if possible by using natural barriers or oil spill control booms to limit spreading. Remove trapped material with pumps, suction equipment, or oil skimmers (EPA 670/2-75-042). Sorbent materials such as activated carbon, polyurethane foam and polypropylene fibres are recommended to take up any residual spilled material (CG-D-38-76).

### 9.1.4 Cleanup and Treatment.

**9.1.4.1 Spills on water.** After containment, explosion-proof oil skimming or vacuum equipment may be used to remove the slick (EPA 670/2-75-042).

**9.1.4.2 General.** For treatment of contaminated water, gravity separation followed by dual media filtration and carbon adsorption is recommended. Recommended carbon ratio: 1.0 to 10 kg soluble materials. The wastewater from backwash of the filtration and adsorption units is returned to the gravity separator. Skimming of cyclohexane off the surface may be sufficient (EPA 600/2-77-277).

**9.1.5 Disposal.** Waste cyclohexane must never be discharged directly into sewers or surface waters. Waste cyclohexane can be disposed of by incineration.

**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 cyclohexane:

- Protective clothing, apron, face shield and impervious gloves should be worn to prevent contact with liquid (GE 1978).
- Splash-proof safety goggles are recommended for eye protection (GE 1978).
- Neoprene is recommended for gloves and clothing (Du Pont 1984).
- The following clothing materials showed breakthrough times greater than 1 hour: nitrile, polyvinyl alcohol, and Viton. The following material showed breakthrough times of 1 hour: chlorinated polyethylene. The following material had breakthrough times of less than 1 hour: butyl rubber, natural rubber, polyvinyl chloride and neoprene-coated rubber (Little 1983).
- Eye wash stations and chemical safety showers should be readily available in areas of use and spill situations (GE 1978).
- Approved respirators with full-face protection should be used for exposure above the TLV® under emergency or nonroutine conditions. Organic vapour cartridge respira-

tors can be used below 1000 ppm. Gas masks with canister are used at 1000 to 10 000 ppm. Self-contained or air-supplied respirators are needed for unknown or higher concentrations (GE 1978).

**9.1.7 Storage Precautions.** Store cyclohexane in tightly closed containers in a cool, well-ventilated area away from sources of heat and ignition, and from strong oxidizing agents. Transfers of liquid require electrical bonding and grounding of containers to prevent static sparks. Use safety cans for small amounts of cyclohexane (GE 1978).

## 10 PREVIOUS SPILL EXPERIENCE

This section contains information on previous 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.

# 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 materials 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 Cyclohexane in Air

**11.1.1 Gas Chromatography** (NIOSH 1977). A range of 510 to 2010 mg/m$^3$ (148 to 584 ppm) of cyclohexane in air may be determined by gas chromatography.

A known volume of air is drawn through a 7 x 0.6 cm O.D. charcoal tube containing 2 sections of 20/40 mesh activated charcoal separated by a 2 mm portion of urethane foam. The first section contains 100 mg charcoal whereas the second section contains 50 mg. A silylating glass wool plug is placed before the front absorbing section. A sample size of 2.5 L at a flow of 200 mL/min is recommended.

The charcoal tube sample is scored before the first section of charcoal and broken. The larger section of charcoal is transferred to a 1 mL stoppered sample container containing 0.5 mL of carbon disulphide. The same operation is performed with the backup section. The sample should be allowed to drain for 30 min.

A 5 µL aliquot of sample is injected into a gas chromatograph equipped with a flame ionization detector. The sample concentration is determined using a suitable electronic integrator which measures peak area in conjunction with a calibration curve.

Typical gas chromatograph operating conditions are: nitrogen carrier gas flow at 50 mL/min, hydrogen gas flow at 65 mL/min, air flow at 400 mL/min, injector temperature at 185°C, detector temperature at 255°C, column temperature at 210°C, and a 4 ft. by 1/4 in. stainless steel column packed with 50/80 mesh Porapak, Type Q.

### 11.2 Qualitative Method for the Detection of Cyclohexane in Air

A range of 100 to 1500 ppm of cyclohexane in air may be determined using a Drager gas detector tube for cyclohexane. A known volume of air is drawn through a Drager gas detector tube pump. A colour change of the orange indicating layer to greenish brown indicates cyclohexane. The colour change is based on the reaction between cyclohexane and chromic acid (Drager 1979).

### 11.3 Quantitative Method for the Detection of Cyclohexane in Water

**11.3.1 Partition Infrared** (AWWA 1981). A range of 40 to 400 ppm cyclohexane in water may be determined using partition infrared spectrophotometry. This limit can be reduced accordingly with concentration of the sample.

A minimum volume of 1 L of representative sample is collected in an appropriate container. The sample is acidified to pH 2 or lower with dilute hydrochloric acid. A 5 mL volume should be sufficient. The sample is transferred to a separatory funnel and a 30 mL volume of Freon® 113 (1,1,2-trichloro-1,2,2-trifluoroethane) is added after it is used to rinse the sample container. The solvent layer is drained into a 100 mL volumetric flask. Two more 30 mL Freon® extracts are carried out and the extracts combined in the 100 mL volumetric flask. The volume is adjusted to 100 mL with Freon®.

The sample is scanned on a suitable infrared spectrophotometer from 3200 to 2700 cm$^{-1}$ using matched 1 cm infrared silica cells. The sample concentration is determined from a calibration curve.

### 11.4 Qualitative Method for the Detection of Cyclohexane in Water

The sample is collected and extracted as in Section 11.3.1. The sample is scanned on a suitable infrared spectrophotometer from 3200 to 2700 cm$^{-1}$ using matched 1 cm infrared silica cells. The presence of cyclohexane is indicated by characteristic I.R. absorption bands.

### 11.5 Quantitative Method for the Detection of Cyclohexane in Soil

**11.5.1 Partition Infrared** (AWWA 1981). A range of 40 to 400 ppm of cyclohexane in soil may be determined using partition infrared spectrophotometry.

Approximately 20 g of soil, accurately weighed, are collected in a glass jar and dried by the addition of magnesium sulphate. Freon®113 (1,1,2-trichloro-1,2,2-trifluoroethane) is used to extract the cyclohexane. Three extractions using 30 mL of Freon® each time are carried out. The extracts are combined in a 100 mL volumetric flask. The volume is adjusted to 100 mL with Freon®. The sample is scanned on a suitable infrared spectrophotometer from 3200 to 2700 $cm^{-1}$ using matched 1 cm near infrared cells. The sample concentration is determined using a calibration curve.

### 11.6 Qualitative Method for the Detection of Cyclohexane in Soil

The sample is collected and extracted as in Section 11.5.1. The sample is scanned on a suitable infrared spectrophotometer from 3200 to 2700 $cm^{-1}$ using matched 1 cm near infrared cells. The presence of characteristic absorption bands indicates cyclohexane (AWWA 1981).

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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 |
| in. | inch |
| 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 |
| *m* | 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 |
| Td | decomposition temperature |
| $TD_{LO}$ | toxic dose low |
| $TL_m$ | median tolerance limit |
| TLV | Threshold Limit Value |
| Ts | 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 |