Kirk-Othmer Encyclopedia of Chemical Technology.

DIMETHYLFORMAMIDE

N,N-Dimethylformamide [68-12-2], DMF, is a clear, colorless, hygroscopic liquid with a slight amine odor. The odor is due to trace amounts of dimethylamine, which, along with formic acid, is formed as a by-product from hydrolysis by trace amounts of water. The rate of hydrolysis in the absence of catalysts and at ambient temperature is quite slow, but the odor of dimethylamine is detectable at less than 1 mm (1). DMF was first synthesized in 1893 by heating a mixture of sodium formate and dimethylammonium chloride, and later prepared by direct reaction of dimethylamine with formic acid (2). Although it does not occur widely in nature, traces of DMF have been detected in sausage, cooked mushrooms, grapes, and wine. The pure liquid is kinetically stable to its boiling point, but its decomposition is catalyzed by strong bases and some transition metals. It is also susceptible to photochemical degradation. The solvent properties of DMF are particularly attractive. Because of its high dielectric constant, aprotic nature, wide liquid range, and low volatility, it is frequently used for chemical reactions and other applications requiring high dissolving power. It is often referred to as “the universal solvent.”

1. Physical and Chemical Properties

Tables 1 and 2 list some physical properties and thermodynamic information for DMF (3, 4).

2. Manufacture and Shipment

There are two processes used commercially for DMF manufacture. A two-step process involves carbonylation of methanol [67-56-1] to methyl formate [107-31-3], and reaction of the formate with dimethylamine.

$$CH_3OH + CO \longrightarrow HCOOCH_3 \qquad (1)$$

$$HCOOCH_3 + (CH_3)_2NH \longrightarrow HCON(CH_3)_2 + CH_3OH \qquad (2)$$

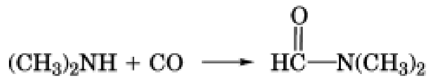
The methanol carbonylation is performed in the presence of a basic catalyst such as sodium methoxide and the product isolated by distillation. In one continuous commercial process (6) the methyl formate and dimethylamine react at 350 kPa (3.46 atm) and from 110 to 120°C to effect a conversion of about 90%. The reaction mixture is then fed to a reactor–stripper operating at about 275 kPa (2.7 atm), where the reaction is completed and DMF and methanol are separated from the lighter by-products. The crude material is then purified in a separate distillation column operating at atmospheric pressure.

Table 1. Physical Properties of DMF, $HCON(CH_3)_2$

Property	Value
molecular formula	C_3H_7NO
molecular weight	73.09
boiling point, °C	153.0
freezing point, °C	−60.4
density, g/cm^3	
20°C	0.949
40°C	0.931
vapor pressure, kPa^a	
20°C	0.38
40°C	1.3
60°C	3.9
vapor density	2.5
critical values	
temperature, °C	374
pressure, MPa^b	4.42
volume, cm^3/g	3.65
refractive index, $n^{25}{}_D$	1.4269
dielectric constant, ϵ, 10 kHz, 25°C	36.7
surface tension, 25°C, mN/m(=dyn/cm)	36.42
dipole moment, $C{\cdot}m$,[c] 20°C	1.27×10^{-29}
viscosity, 25°C, mPa·s(=cP)	0.802
solubility parameter, δ, $MPa^{1/2d}$	24.8
donor number[e]	26.6
hydrogen-bonding index[f]	6.4

[a] To convert kPa to mm Hg, multiply by 7.5.
[b] To convert MPa to psi, multiply by 145.
[c] To convert C·m to debye, divide by 3.336×10^{-30}.
[d] To convert $MPa^{1/2}$ to $(cal/cm^3)^{1/2}$, divide by 2.05.
[e] Ref. 5.
[f] On a Du Pont scale.

A second process is the direct carbonylation of dimethylamine [124-40-3] in the presence of a basic catalyst or a transition metal. This carbonylation is often run in the presence of methanol in order to help solubilize the catalyst (7), and presumably proceeds through methyl formate as an intermediate.



$$(CH_3)_2NH + CO \longrightarrow HC(=O)-N(CH_3)_2 \qquad (3)$$

Again, the basic catalyst is typically sodium methoxide, although other bases such as phenoxides (8) and basic anion-exchange resins (9) have also been used. The reaction using sodium methoxide is performed at 4.9 MPa (48 atm) and 120°C (10).

A modification of the direct process has recently been reported using a circulating reactor of the Buss Loop design (11). In addition to employing lower temperatures, this process is claimed to have lower steam and electricity utility requirements than a more traditional reactor (12) for the direct carbonylation, although cooling water requirements are higher. The reaction can also be performed in the presence of an amidine catalyst (13). Related processes have been reported that utilize a mixture of methylamines as the feed, but require transition-metal catalysts (14).

Table 2. Thermodynamic Properties of DMF[a]

Property	Value
flash point, °C	
TCC	58
TOC	67
autoignition temperature, °C	445
flammability limits in air, vol %	
lower (100°C)	2.2
upper (100°C)	15.2
free energy of formation, $\Delta G^0{}_g$, 25°C, kJ/mol	−88.4
enthalpy of formation, 25°C, kJ/mol	
$\Delta H^0{}_g$	−191.7
$\Delta H^0{}_l$,	−239.3
heat of combustion, liquid, 25°C, kJ/mol	1921
heat of vaporization, kJ/mol	
25°C	47.6
140°C	38.3
heat capacity, kJ/(kg·K)	
liquid at 25°C	2.03
gas at 127°C, 101.3 kPa[b]	1.61
entropy, S, gas at 25°C, 101.3 kPa,[b] kJ/(mol·K)	0.33

[a] To convert kJ to kcal, divide by 4.184.

[b] 101.3 kPa = 1 atm·

Another method of preparation involving methyl formate has been reported wherein the formate reacts with ammonia and methanol in the presence of ammonium chloride at 255°C and 2.9 MPa (28.6 atm) (15). In this case, monomethylformamide is present in considerable quantities as a by-product.

In another DMF process, hydrocyanic acid reacts with methanol in the presence of water and a titanium catalyst (16), or in the presence of dimethylamine and a catalyst (17).

DMF can also be manufactured from carbon dioxide, hydrogen, and dimethylamine in the presence of halogen-containing transition-metal compounds (18). The reaction has also been performed with metal oxides and salts of alkali metals as promoters (19).

The direct, one-step production of DMF from carbon monoxide, hydrogen, and ammonia has also been reported. A ruthenium carbonyl catalyst is used, either in a polar organic solvent (20) or in a phosphonium molten salt medium (21).

Formamide has been alkylated with methanol in the presence of a metal catalyst to give DMF (22). The alkylation reaction can also be catalyzed by tetralkylammonium salts (23).

Oxidative processes can also be used to prepare DMF. For example, it can be produced from tetraoxane (a source of formaldehyde (qv)), oxygen, and dimethylamine over Pd–Al_2O_3 (24) or from trimethylamine and oxygen in the presence of a metal halide catalyst (25).

DMF can be purchased in steel drums (DOT 17E, UN1A1, 410 lbs net = 186 kg), tank trucks, and railcars. On Oct. 1, 1993, new regulations in the United States were established for DMF under HM-181; the official shipping name is *N,N*-dimethylformamide (shipping designation UN 2265, Packing Group III, Flammable Liquid). Formerly, it was classified as a Combustible Liquid in bulk quantities, but as “Not Regulated” in drums (49 CFR). International overseas shipments have an IMCO classification of 3.3.

Table 3. DMF Manufacturers and Capacities

Manufacturer	Location	Capacity, t	World capacity, %
Air Products and Chemicals	Pensacola, Fla., United States	7,000	2.6
BASF Aktiengesellschaft	Ludwigshafen, Germany	60,000	22.5
BASF Quimica da Bahia SA	Camaçari, Brazil	6,000	2.2
Celanese Mexicana	Cosoleacaque, Veracruz, Mexico	6,000	2.2
Chinook	Toronto, Canada	10,000	3.8
E. I. du Pont de Nemours & Co., Inc.	Belle, W. Va., United States	41,000	15.4
Ertisa	Huelva, Spain	5,000	1.9
ICI-Petrochemicals Division	Billingham, U.K.	15,000	5.6
Korea Fertilizer Co.	Ulsan, Republic of Korea	8,000	3.0
Lee Chang Yung Chemical Industry	Hsinchu City, Taiwan	10,000	3.8
Leuna Werke	Leuna, Germany	19,000	7.1
Mitsubishi Gas Chemicals	Matsuhama, Niigata, Japan	20,000	7.5
Nitto Chemical Industries	Tsurami, Yokohama, Japan	35,000	13.1
PT Langgeng Kimindo Pratama	Karawang, West Java, Indonesia	40,000[a]	
Rashtriya, Chemicals and Fertilizer	Thane, India	2,500[a]	
UCB	Gent, Belgium	25,000	9.4
Total		*267,000*	

[a] Planned; not included in total.

3. Economic Aspects

World capacity of DMF is about 267,000 t, with plans for an additional 50,000 t announced for India and Indonesia. Table 3 lists capacities of principal producers. World production is considerably less than capacity and is probably around 125,000 t. In the United States, 1991 production was about 25,000 t, and a significant portion of that was exported, mostly to Taiwan. DMF production and export are closely tied to economic conditions and exchange rates, as demonstrated by the dramatic turnaround from 1983 when a substantial portion of U.S. demand was met by imports (26). Demand in the United States has slowed since the early 1980s because of decreased acrylic fiber production (see Fibers, acrylic). As with all organic solvents, growing concerns about emissions have caused many users to make more efficient use of DMF. Increasingly, it is being recovered and purified for reuse. Because many applications require pure DMF, this recovery is not trivial. However, this trend toward recycling will certainly continue and impact future demand for this solvent. The 1992 selling price for DMF was $1.39/kg.

4. Specifications, Analytical Methods, and Storage and Handling

Table 4 lists the specifications set by Du Pont, the largest U.S. producer of DMF (4). Water in DMF is determined either by Karl Fischer titration or by gas chromatography. The chromatographic method is more reliable at lower levels of water (<500 ppm) (4). DMF purity is determined by gc. For specialized laboratory applications, conductivity measurements have been used as an indication of purity (27). DMF in water can be measured by refractive index, hydrolysis to DMA followed by titration of the liberated amine, or, most conveniently, by infrared analysis. A band at $1087\ \text{cm}^{-1}$ is used for the ir analysis.

A number of methods are available for determination of DMF in air. Air can be drawn through an absorbent to concentrate the DMF vapors. The absorbent is then heated and the released DMF is analyzed

Table 4. Specifications for DMF[a]

Quality	Value
water, %	0.05
basicity (as dimethylamine, ppm)	15
formic acid, ppm	20
iron, ppm	0.05
color (APHA)	15
pH at 25°C, 20% aqueous solution	6.5–9.0

[a] Ref. 4.

by gc. Infrared analysis of an air sample can also be used, although enhanced sensitivity can be attained by scrubbing an air sample with perchloroethylene and then analyzing the resultant solution by ir. The most convenient procedure is to use indicating absorbent tubes that change color when air contaminated with DMF is passed through them. In the United States, these sampling tubes are available from Lab Safety Supply, Inc. (Janesville, Wisconsin) or Mine Safety Appliances (Pittsburgh, Pennsylvania).

Pure DMF is essentially noncorrosive to metals; however, copper, tin, and their alloys should be avoided. Ideal materials for its handling and storage are nonalloy (carbon) steels, stainless steels, and aluminum. Seals and other soft materials should be made of polytetrafluoroethylene, polyethylene, or high molecular weight polypropylene. Ethylene–propylene rubber O-rings and diaphragms can also be used with DMF. Oils and grease are not suitable as lubricants; however, graphite can be used to lubricate moving parts in contact with DMF. Since DMF is hygroscopic, it should be kept under a blanket of dry nitrogen. High purity DMF required for acrylic fiber production is best stored in aluminum tanks.

5. Uses

The two largest uses for DMF in the United States had been in pharmaceutical processing and acrylic fiber production. Until the late 1980s, acrylic fiber spinning was the largest single use for DMF and accounted for around 25% of total U.S. consumption. However, in 1990, Du Pont ceased U.S. production of acrylic fibers (28). The remaining U.S. producers use dimethylacetamide [127-19-5], DMAC, in their fiber-spinning processes and acrylic fiber production no longer consumes significant quantities of DMF in the United States.

Worldwide demand for DMF in acrylic fiber production has held up better than in the United States. The high solubility of polyacrylonitrile in DMF, coupled with DMF's high water miscibility, makes it an attractive solvent for this application. Its principal competition in this area comes from DMAC.

The combined pharmaceutical applications account for an estimated 25% of DMF consumption. In the pharmaceutical industry, DMF is used in many processes as a reaction and crystallizing solvent because of its remarkable solvent properties. For example, hydrocortisone acetate [50-03-3], dihydrostreptomycin sulfate [5490-27-7], and amphotericin A [1405-32-9] are pharmaceutical products whose crystallization is facilitated by the use of DMF. It is also a good solvent for the fungicide griseofulvin [126-07-8] and is used in its production.

Another significant application for DMF is as a solvent for depositing polyurethane coatings on leather and artificial leather fabrics. This use, too, has fallen somewhat since the late 1970s, as changing fashions have decreased the demand for "wet look" fabrics, and it now accounts for about 5% of U.S. demand for DMF. Again, this use is more significant in the rest of the world than it is in the U.S. Other polymers that dissolve in DMF include poly(vinyl chloride), vinyl chloride–vinyl acetate copolymers, and some polyamides. DMF is also used as a solvent in epoxy formulations. Dicyandiamide [461-58-5], an important epoxy curing agent, is soluble in DMF, and solutions are used in preparing formulations for use in laminated printed circuit boards.

Another use is in various extraction and absorption processes for the purification of acetylene or butadiene and for separation of aliphatic hydrocarbons, which have limited solubility in DMF, from aromatic hydrocarbons. DMF has also been used to recover CO_2 from flue gases. Because of the high solubility of SO_2 in DMF, this method can even be used for exhaust streams from processes using high sulfur fuels. The CO_2 is not contaminated with sulfur-containing impurities, which are recovered from the DMF in a separate step (29).

Because of its ability to dissolve both inorganic- and organic-based residual fluxes, DMF is used as a quench and cleaner combination for hot-dip tinned parts. The high solubility of inorganic salts in DMF has led to the use of such solutions in high voltage capacitors. DMF is also used in some industrial paint stripping applications. However, toxicity concerns limit its use in consumer products of this type. The high solvent strength of DMF also contributes to its use as a carrier for inks and dyes in various printing and fiber-dyeing applications.

DMF is used extensively as a solvent, reagent, and catalyst in synthetic organic chemistry. Several comprehensive reviews describe its uses in this area (30–32).

6. Health and Safety Factors

The acute toxicity of DMF is relatively low. The LD_{50} by oral ingestion in rats is 2800 mg/kg and the LC_{50} for mice is 9400 mg/m^3 (2 h). Skin absorption is also an important route by which DMF can be introduced into the body, and an LD_{50} of 4720 mg/kg has been observed in rabbit-skin exposure studies.

The chemical has been linked to alcohol intolerance among workers, which is manifested by a reddening of the skin upon ingesting alcoholic beverages shortly after exposure to DMF. The skin reddening is temporary and is usually limited to the face and neck. Chronic exposure to high levels of DMF causes liver damage. In some studies, blood tests on exposed workers showed evidence of liver damage even when no symptoms were reported by the workers (33).

Earlier reports of a link between testicular cancer and DMF exposure have not been corroborated in a study of 4000 Du Pont employees (34). Very recently, inhalation studies in mice and rats have shown no oncogenic effect from DMF (35). The International Agency for Research on Cancer (IARC) has concluded that evidence associating DMF with cancer in animals is “inadequate,” but has classified DMF as “possibly carcinogenic to humans” (Group 2B) (36).

Although DMF has led to increased embryo mortality in pregnant animals at doses close to the lethal level for the pregnant animal, DMF exposures below the OSHA limits should not represent a hazard to pregnant workers as long as prudent work practices are followed (4).

The American Conference of Governmental Industrial Hygienists (ACGIH) has recommended that time weighted-average exposures for DMF not exceed 10 ppm or 30 mg/m^3 (skin designation, 1989 standard) for an eight-hour work day. In the United States, OSHA has accepted the ACGIH limits in setting regulations for worker exposures. As with other industrial chemicals, regulations and expert opinion evolve over time, and DMF exposure limits may be tightened in the future. A Biological Exposure Index (BEI) of 40 mg DMF metabolites/g of creatinine in urine has also been adopted by ACGIH and applies in cases where there is significant potential for absorption of DMF is liquid or vapor through the skin.

Although it is a versatile and generally stable solvent, DMF must be used with care in some applications. It can react violently with very strong reducing and oxidizing agents. Runaway reactions have been reported under certain conditions with nitric acid and its salts, permanganates, dichromates, bromine, chlorine gas, sodium hydride, sodium borohydride, and alkyl aluminum compounds (37). DMF is widely used as a solvent for many of these reagents, and in some instances, the conditions leading to the violent reactions are not well understood. Incidents have occurred after several successful experiments (38). Any scale-up involving DMF should proceed with caution and workers should consult the literature carefully. DMF can also react violently with some organic halides, particularly in the presence of iron salts and other metal contaminants; violent reactions have been reported with ethylene dibromide (39), carbon tetrachloride, and hexachlorobenzene. Additional caution is called for when using inorganic halide reagents such as thionyl chloride and phosphorus oxychloride, since dimethylcarbamoyl chloride, a suspect carcinogen, can be formed from DMF and these reagents (40). The reaction with thionyl chloride can also be accelerated by iron or zinc contaminants, resulting in a sudden pressure rise (41).

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Formic acid; Fibers, acrylic