

[®]
CYGLINK

NMA Monomer N-Methylol Acrylamide



CYTEC

CYLINK[®] NMA Monomer

N-Methylol Acrylamide
(48% Aqueous Solution)
[N-(Hydroxymethyl) acrylamide]

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Introduction

NMA is a unique monomer. It is truly bifunctional, possessing both vinyl and hydroxymethyl groups. Thermoplastic polymers can be formed through the copolymerization of NMA with a variety of vinyl monomers via emulsion, solution, or suspension techniques. The resulting products, having pendant hydroxymethyl groups, are self-crosslinkable under moderate conditions. This mechanism permits the conversion of thermoplastic backbone polymers to thermoset materials at the point of use without the need for an external crosslinker. Conversely, the hydroxymethyl group can first be reacted with a substrate like cellulose and subsequently cross-linked by free radical polymerization.

Applications of NMA range from adhesives and binders in papermaking, textiles, and non-wovens to a variety of surface coatings and resins for varnishes, films and sizing agents. The Cytec brochure entitled, "Applications — Processes — Products — References — N-methylolacrylamide" contains the references for commercial and potential applications of NMA.

NMA is offered as a 48% aqueous solution shipped in 55 gallon fiber drums which contain a one piece polyethylene lining with no joints, seams or seals. The cover is injection-molded polypropylene having a flowed-in rubber gasket and is fully removable. This construction eliminates the possibility of NMA solution exposure to metals, particularly iron, copper, brass or bronze which will cause slow polymerization in the area of contact. Shipments can also be made in tank trucks and tank cars of the proper materials of construction such as SS type 304 or 306, fiberglass reinforced, corrosion resistant polyester, phenol-formaldehyde resin and polyvinyl chloride coatings. Details are described on page 9 of this booklet.

Typical Properties

Appearance	Clear to slightly hazy, liquid yellow to pink in color.
Color, APHA	40
Molecular Weight	101
KCN Assay, % by Weight	40-44
Acrylamide, % by Weight	<5.0
Total Solids, % by Weight by Bromination	46-50
Free Formaldehyde, % by Weight	<2.0
Copper in Solution, ppm (as is)	<2.0
MEHQ Inhibitor, ppm (as is)	30 ± 5
pH (as is)	6.0 — 7.0*
Specific Gravity at 25°C (77°F)	1.08
Heat of Polymerization	20k Cal./Mole.
Crystallization Point	-10°C (14°F)
Homopolymer Tg	34°C

If the methods of analysis used to obtain this data are of interest to you, a Cytex technical representative can discuss them with you.

Solubility (as 100% dry)	g/100 ml. Solvent	Temperature	
		°C	°F
Water	122	10	50
Water	188	20	68
Water	354	40	104
Water	755	60	140
Methanol	149	30	86
Ethanol, Absolute	77	30	86
Ethanol — 90%	116	30	86
Isopropanol	53	30	86
n-Butanol	42	30	86
Acrylonitrile	12	30	86
Vinyl Acetate	2	30	86
Methyl Methacrylate	3	30	86
Ethyl Acrylate	3	30	86

Small amounts of alcohols, glycol or water plus a surfactant will improve the solubility of NMA in the four monomers listed at the bottom of the table.

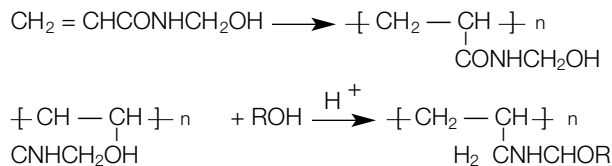
Stability

Aqueous solutions of NMA are highly reactive; thus, it is essential that precautions be taken to maintain stability during shipping and storage. The stability of NMA solution is dependent mainly upon oxygen level, contaminants, storage temperature, and pH. The details are discussed on page 11 under the heading "Bulk Storage and Handling".

Chemical Properties and Reactions

The outstanding property of NMA is that it possesses both vinyl and hydroxymethyl functionality and reactions may be carried out at either group independently. This property allows consecutive reactions to be carried out in either of two ways:

1. The vinyl addition product, polymer or copolymer may be formed first and then modified by crosslinking or other reaction mechanisms at the hydroxymethyl group, for example:



This allows for polymerization with itself or other vinyl monomers leaving the hydroxymethyl group available for later cure by heat, acid or free radical catalysis; crosslinking with themselves (other co-monomers containing amino, hydroxyl or carboxyl functionality) or a reactive substrate.

N-methylolacrylamide can be copolymerized with vinyl acetate, ethyl acrylate and itaconic acid to form latices which are free from coagulum and have low viscosity and excellent shelf stability. The latex and film properties of two such copolymers are described on the following pages. Recipes and procedures for the preparation of these latexes are described on pages 3-4.

* At time of manufacture; pH may decrease in storage to 5.5.

**Tetrapolymer of Vinyl Acetate/
Ethyl Acrylate/N-methylol-
acrylamide/ Itaconic Acid
(VA/EA/NMA/IA)
(Parts by Weight)**

	Recipe 1 (70/25/4/1)	Recipe 2 (66/25/8/1)
Solids (%)	45.8	45.9
Coagulum (% based on total latex)	0	Less than 0.1
Viscosity (cps) at 25°C. Brookfield Model RVF, No. 3 Spindle, 100 rpm.	100	150
Surface Tension (dynes/cm) at 25°C. DuNouy Method.	33	33
Particle Size (A°)	1,500 to 1,850	1,200 to 1,600

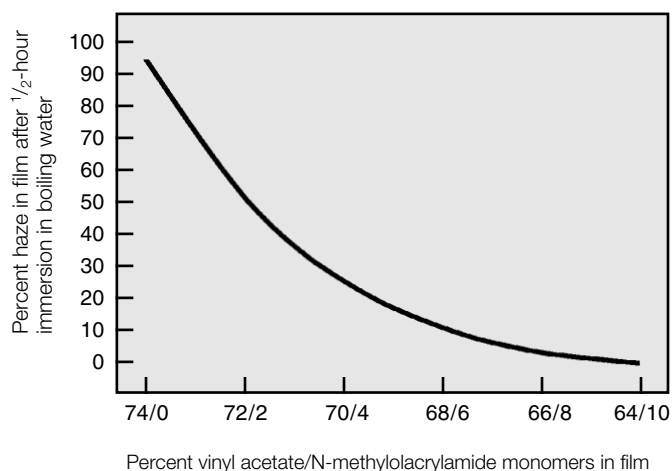
Typical Film Properties

Latices prepared from Recipes 1 and 2 were coated* on cellophane. The resulting films possess excellent clarity and continuity and have excellent water resistance, as illustrated by the small amount of haze developed after a half-hour immersion in boiling water. Figure 1 shows the effect of increasing the amount of N-methylolacrylamide and decreasing the amount of vinyl acetate in the tetrapolymer on haze development in the film. Initial haze of all films was 2%. Haze was measured on the integrating sphere hazemeter described in ASTM D 1003.

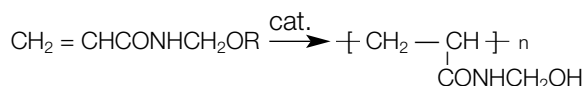
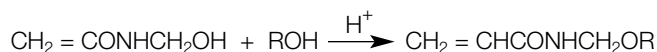
* Latex was applied to cellophane using a #3 wire-wound applicator rod. The films were cured at 100°C for 3 minutes using 0.5% tartaric acid and 0.5% MgCl₂ in the latex.

Figure 1.

Haze developed in latex polymer films containing various amounts of N-methylolacrylamide. (Vinyl acetate/ethyl acrylate/N-methylolacrylamide/itaconic acid tetrapolymer, ethyl acrylate = 25%, itaconic acid = 1%.)



- The hydroxymethyl group may be reacted first, followed by addition at the vinyl group or by polymerization or copolymerization.



Therefore NMA monomer can be chemically attached to a substrate such as cellulose and then crosslinked through the vinyl group. This type of reaction introduces a broad range of applications.

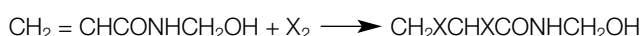
Most of the chemical reactions, crosslinking catalysts and potential applications of N-methylolacrylamide presented in the following sections are based on information available in the literature. For details, it is recommended that the reader consult the original literature citations. The data presented on emulsion polymerization are based primarily on Cytec Industries' laboratory work (See Reference 1).

* Latex was applied to cellophane using a #3 wire-wound applicator rod. The films were cured at 100°C for 3 minutes using 0.5% tartaric acid and 0.5% MgCl₂ in the latex.

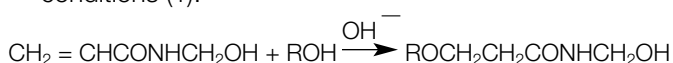
Reactions of the Vinyl Group

The reactions which occur at the vinyl group of N-methylolacrylamide are typical of an acrylic double bond.

1. Addition of halogens (2, 15) or chloroalkanol (59).

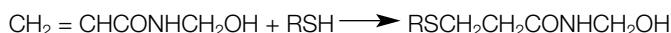


2. Michael addition of alcohols under alkaline conditions (1).



Polyhydric alcohols, such as glycerine, glucose, starch, cellulose and polyvinyl alcohol also undergo this reaction.

3. Addition of thiols in the presence of sodium ethylate (10).



Polymerization and Copolymerization

N-methylolacrylamide can be homopolymerized (4, 8, 16, 17, 18, 19, 20) or copolymerized with monomers such as acrylamides (20), N-alkylacrylamides (21), acrylonitrile (22), acrylic acids, vinyl ethers and esters (8, 13, 23) and vinyl acetate by emulsion or solution polymerization methods. Copolymers of NMA with these and other monomers are given in the Cytec brochure entitled, "NMA — Applications — Processes — Products — References". (PRT-708-B)

Preparation of Latex

The preparation of latex recipes 1 and 2 (see properties on page 2) is described below. Other recipes are available on request.

A. Reactor Charge	VA/EA/NMA/IA Tetrapolymer (Parts by Weight)	
	Recipe 1 (70/25/4/1)	Recipe 2 (66/25/8/1)
Boiled, Deionized Water	36.0	36.0
Potassium Persulfate (5% solution)	10.0	10.0
Sodium Bicarbonate	0.4	0.4
AEROSOL® A-102 Surfactant ⁽¹⁾ Solids Basis	3.1	3.1
Tert-Dodecyl Mercaptan	0.1	0.1
B. Delayed-Addition Solution, Part I		
Diammonium Phosphate	0.2	0.2
Sodium Metabisulfite (2% solution)	5.0	5.0
C. Pre-Emulsified Monomers		
AEROSOL® OT 75% Surfactant ⁽¹⁾ Solids Basis	0.67	1.3
Boiled, Deionized Water	47.2	47.2
Methanol	5.0	5.0
Vinyl Acetate	70.0	66.0
Ethyl Acrylate	25.0	25.0
Itaconic Acid	1.0	1.0
Sodium Metabisulfite (2% solution)	10.0	10.0

⁽¹⁾ Trademark Cytec Industries Inc.

D. Delayed-Addition Solution, Part II	VA/EA/NMA/IA Tetrapolymer (Parts by Weight)	
	Recipe 1 (70/25/4/1)	Recipe 2 (66/25/8/1)
Surfonic [®] N-95 surface active agent ⁽²⁾	1.0	1.0
Boiled, Deionized Water	10.0	10.0
N-Methylol-acrylamide (48% solution)	8.4	16.6

A. Reactor Charge

Charge the water, potassium persulfate solution, sodium bicarbonate. Add AEROSOL A-102 surfactant and mercaptan to the polymerization vessel and stir until dissolved. Slowly bubble nitrogen through the solution to expel oxygen. Heat to 65°C, while stirring and maintaining the nitrogen flow. Meanwhile, prepare the delayed-addition solution, Part I and the pre-emulsified monomers.

B. Delayed-Addition Solution, Part I

Dissolve the diammonium phosphate in the 2% sodium metabisulfite solution.

C. Pre-emulsified Monomers

Dissolve the AEROSOL OT-75% surfactant in water and methanol. Add the vinyl acetate, ethyl acrylate, itaconic acid, and sodium metabisulfite solution. Slowly bubble nitrogen through the emulsion for 15 minutes. Transfer to a suitable addition vessel equipped with an agitator. Maintain agitation throughout addition to the polymerization vessel.

When the contents of the polymerization vessel reach 65°C, minimize the nitrogen flow and add the delayed-addition solution, Part I. Then begin addition

of the pre-emulsified monomers at the rate of approximately one part per minute. Meanwhile, prepare the delayed-addition solution, Part II.

D. Delayed-Addition Solution, Part II

Dissolve the Surfonic N-95 and N-methylolacrylamide in water.

During the addition of the first 15% of the pre-emulsified monomers, initiation of polymerization occurs as indicated by a slight exotherm or a change in the translucency of the emulsion in the polymerization vessel. At this point introduce the delayed-addition solution, Part II, to the addition vessel containing the remaining 85% of pre-emulsified monomers and, without interruption, continue addition of the total monomers to the polymerization vessel at the same rate (one part per minute) over a period of about three hours. Hold the resulting latex at 65°C for one hour. Cool, filter, and transfer to a suitable container.

Solution Polymerization

N-methylolacrylamide can be polymerized in aqueous solutions using water-soluble catalysts (16, 17). A typical polymerization procedure is as follows: Place 200 g of deionized water in a one-liter, three-necked flask fitted with a stirrer and a water condenser. Heat to reflux, then cool to 40-45°C. Slowly bubble nitrogen through the water to sweep the flask and its contents free of oxygen. Add 52.5 g of 48% aqueous N-methylolacrylamide and stir. Buffer to pH 5.75 with potassium acetate and acetic acid. Add 1.5 g of isopropanol. Add a solution of 0.5 g of ammonium persulfate in 8 g of deionized water. Slowly warm the solution to 60°C and stir at that temperature for two hours while bubbling nitrogen through the solution. Cool the resulting polymer solution to room temperature and adjust to pH 7.5 with sodium hydroxide.

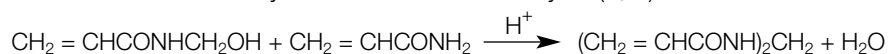
A polymer solution prepared in this way had a viscosity of 2,650 cps (Brookfield Model RVF) and a molecular weight of 60,000 ± 500, as determined by osmometry.

⁽²⁾ Product of Huntsman Chemical Company.

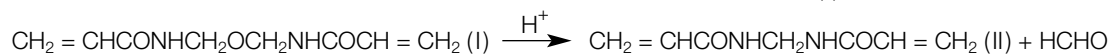
Reactions of the Hydroxymethyl Group

The hydroxymethyl group of N-methylolacrylamide undergoes a variety of reactions, several of which are summarized below.

1. Condensation with acrylamide under acid catalysis (2, 3).

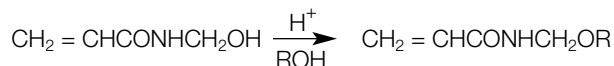


N-methylolacrylamide alone can be condensed similarly by acids or heat or both to form bis-(acrylamidomethyl) ether (I) or N,N'-methylenebisacrylamide (II), depending on the severity of the conditions (4, 5, 6) (57).



These reactions are comparable to the crosslinking reactions of N-methylolacrylamide copolymers described below.

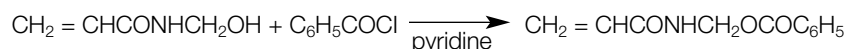
2. Condensation with alcohols, phenols, resorcinol or hydroquinone, under acid catalysis, also takes place readily (7, 8, 60).



N-methylolacrylamide reacts with cellulose in the same way to yield $\text{CH}_2 = \text{CHCONHCH}_2\text{-O-cellulose}$ (9). This condensation product can be polymerized by means of beta irradiation (39).

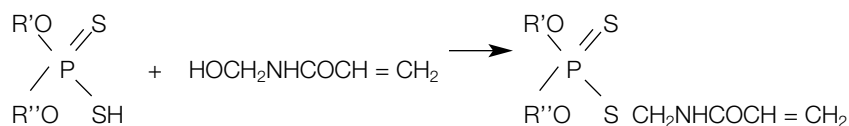
3. Esterification with acid chlorides (2).

The reaction with acid chlorides is exemplified by treatment of N-methylolacrylamide with benzoyl chloride in pyridine (2).



4. Condensation with O,O'-diesters of phosphorodithioic acid (10).

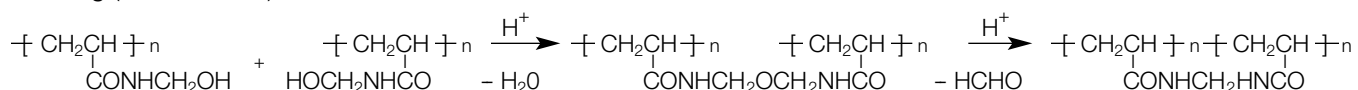
(S-Acrylamidomethyl) phosphorodithioic triesters can be prepared from N-methylolacrylamide and O,O'-diesters of phosphorodithioic acid (10).



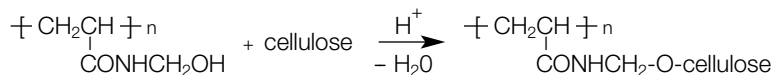
The triesters are used as antioxidants for hydrocarbon oils.

5. Crosslinking of N-methylolacrylamide copolymers.

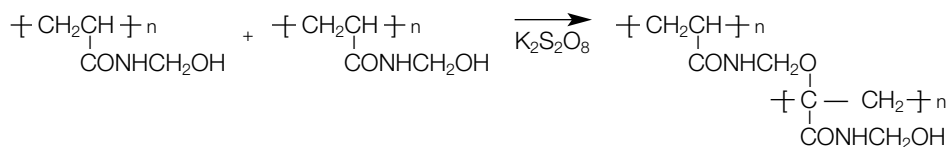
Crosslinking of copolymer units through the hydroxymethyl groups can be effected by acid catalysis or simply by heating (8, 11, 12, 54).



Similarly, a copolymer unit can be anchored to a reactive substrate such as cellulose (13).



Crosslinking through a free radical mechanism involving a hydroxymethyl group of one unit and a hydrogen alpha to a carbonyl group of another unit can be effected by use of potassium persulfate (11).



The crosslinking densities of N-methylolacrylamide/ethyl acrylate emulsion copolymers have been determined (14). Cyclization of N-methylolacrylamide to tetrahydro-4H-1,3-oxazin-4-one in the presence of alkali catalysts has been reported (58).

Latex and solution polymers containing N-methylolacrylamide can be crosslinked readily by heat alone (50) or, more effectively, by heat in the presence of acid-generating catalysts (24-27) and metal salts plus organic acids [Lewis Acids] (25).

The following catalysts have been found effective in promoting crosslinking:

- Aluminum chloride
- Aluminum zirconium acetate
- Ammonium chloride
- Ammonium phosphates
- Magnesium chloride
- Organic acids such as citric, fumaric and lactic acids
- Organic or inorganic acids, plus magnesium, zinc and aluminum salts*
- Sodium chlorate and magnesium or zinc salts
- Sodium fluoroborate and magnesium chloride
- Sodium perchlorate and magnesium chloride
- Sodium perchlorate and magnesium or zinc salts
- p-Toluenesulfonic acid
- Zinc nitrate
- Zinc nitrate and magnesium chloride
- Zinc perchlorate
- Zinc sulfoxalate and zinc nitrate

The above catalysts are used in the concentration range of 0.1% to 3.5% on the total weight of polymers depending on temperature and the rate of "cure"

(crosslinking) desired. The amount of catalyst used must be determined experimentally for any particular temperature to which the crosslinkable polymer or treated article is to be heated (25, 50). Thus, if a relatively high temperature (300-450°F) is to be used, then "cure" (crosslinking) will take place either by heat alone (50) or heat in the presence of a "mild" catalyst such as diammonium phosphate (53). If crosslinking is desired at temperatures below 300°F, then increasingly "stronger" catalyst systems are required (25, 50). With catalyst systems such as the Lewis Acids, crosslinking will take place simply by drying the films or the coated or treated articles under ambient conditions (53). The degree of "cure" or crosslinking will increase with the age of the film or coating.

The pH is also an important factor in degree and rate of crosslinking (53, 24, 25, 50).

The presence of small amounts (1-5%) of carboxyl, hydroxyl, amide or amino groups in the polymer containing N-methylolacrylamide will further accelerate the rate of "cure".

The following test methods used for measuring the effects of crosslinking have been discussed in detail in various publications (4, 5, 6, 13, 14, 20, 24, 25, 50-56): swelling index in organic solvents and water, resistance to acid and alkali, clarity of film on immersion in water or solvents, tensile strength and elongation, flexibility, recovery, crease resistance, resistance to laundering and dry cleaning, and infra-red and NMR examination.

*Aluminum chloride, magnesium chloride or zinc nitrate are often used in combination with organic acids or sodium fluoroborate to give Lewis Acid catalyst systems (25). Polymer composition may be a factor in obtaining suitable crosslinking (26, 27, 53).

FDA Status (21 CFR)

175.105 **Adhesives, Homopolymers and Copolymers with Other Listed Monomers Limitations:**

The adhesive is either separated from the food by a functional barrier or used subject to the following additional limitations.

- a. In dry foods. The quantity of adhesive that comes into contact with packaged dry foods shall not exceed the limits of good manufacturing practice.
- b. In fatty and aqueous foods. The quantity of adhesive that comes into contact with packaged fatty and aqueous foods shall not exceed the trace amount at seams and at the edge exposure between packaging laminates that may occur within the limits of good manufacturing practice.

177.1010 **Acrylic and Modified Acrylic Plastics, Semirigid and Rigid**

Copolymers with other listed monomers.

Limitations:

Finished copolymers shall contain no more than 5 weight percent of total polymer units derived from N-methylolacrylamide.

177.2260 **Filters, Resin-Bonded**

Copolymers with ethyl acrylate alone or with other listed monomers.

Limitations:

- a. The quantity employed shall not exceed the amount reasonably required to accomplish the intended physical or technical effect.
- b. The finished copolymer shall contain no more than 5 weight percent of total polymer units derived from N-methylolacrylamide.

176.170 **Vinyl Acetate Polymer with Ethylene and N-(Hydroxymethyl) Acrylamide**

Limitations:

1. Polymer does not contain more than 6 weight percent of total polymer units derived from N-(hydroxymethyl) acrylamide.
2. For use only in coatings for paper and paper-board intended for use in contact with foods as specified in 21 CFR 176.170.

Health & Safety Information

The acute oral (rat) and dermal (rabbit) LD₅₀ values are greater than 500 mg/kg and 1800 mg/kg, respectively. Direct contact with this material may cause moderate skin and eye irritation. Rats, mice and guinea pigs exposed for six hours to air saturated with the vapor of N-methylolacrylamide exhibited no evidence of toxic effect. The acute inhalation (4 hour rat) is greater than 5 mg/L.

The acute oral (mouse), acute oral (rat) and acute dermal (rabbit) LD₅₀ value for N-methylolacrylamide are 0.4 g/kg, 0.25 g/kg and 16.0 g/kg, respectively. Moderate skin irritation was produced during testing in rabbits at dermal doses of 2-16 g/kg. Mild eye irritation was produced during primary eye irritation testing in rabbits. Neurotoxicity can result after a single ingestion of N-methylolacrylamide but is more likely to occur after ingestion of small amounts over a period of several days or weeks. Signs and symptoms include increasing sweating of the hands and feet, numbness, tingling and weakness in the extremities, unsteady gait and decreased reflexes. N-methylolacrylamide is readily absorbed through the unbroken skin. Prolonged or repeated dermal exposure may cause signs and symptoms of neurotoxicity as described above but is preceded by peeling and redness of the skin of the hands and feet, the usual areas of exposure. Airborne N-methylolacrylamide is absorbed through the lungs and upon overexposure causes neurotoxicity. The National Toxicology Program (NTP) has completed lifetime carcinogenicity studies of N-methylolacrylamide in rats and mice. Doses of up to 12 mg/kg/day in rats produced no evidence of carcinogenic activity. Doses of up to 50 mg/kg/day in mice produced increased incidences of tumors of the lung, liver, ovary, and the hardy gland, an accessory gland of the eye.

In addition to the neurotoxic effect of acrylamide, there is evidence that acrylamide and formaldehyde cause cancer in laboratory animals. Acrylamide and formaldehyde are used as raw materials in the manufacture of N-methylolacrylamide, and therefore the product contains residues of these two materials. Acrylamide and formaldehyde are regulated by OSHA, and workplace practices should be used which assure compliance with these regulations. Refer to the Cytec Material Safety Data Sheet (MSDS) before handling.

Engineering Controls and Personal Protective Equipment (PPE)

Utilize a closed system process where feasible. Where a closed system is not used, good enclosure and local exhaust ventilation should be provided to minimize exposure. After N-methylolacrylamide is in solution, exposure to liquid and mist must be controlled. Food, beverages and tobacco products should not be carried, stored or consumed where this chemical is in use. Before eating, drinking or smoking, wash face and hands with soap and water. Shower after completion of workshift. Launder work clothing at end of workshift prior to reuse. Store street clothing separately from work clothing and protective equipment. Work clothing and shoes must not be taken home. Where adequate engineering controls are in effect, and measurements confirm airborne concentrations are below the Permissible Exposure Level, no respiratory protection is required. NIOSH does not approve a cartridge respirator for use with N-methylolacrylamide. However, tests conducted show that organic vapor cartridges provide protection from airborne levels up to 2.5 mg/M³. **The cartridges must be changed at the beginning of each shift.** Full facepiece, positive pressure, supplied air respirators or self-contained breathing apparatus must be used for higher or unknown concentrations. Full facepiece respirators provide additional eye protection where handling makes it desirable. Note that N-methylolacrylamide exhibits no warning properties at concentrations at or below the Permissible Exposure Level. Wear the following to prevent skin contact: impervious rubber or plastic gloves, rubber shoes and long sleeved coveralls, which are provided clean daily. For operations where eye and face contact with N-methylolacrylamide solution can occur, wear chemical splash proof goggles, a faceshield and head covering. **Wash gloves thoroughly before removing and discard gloves that are contaminated on the inside.** When solutions are used, provide eyewash fountain and safety shower in close proximity to points of potential exposure.

Steps To Be Taken in Case Material is Released or Spilled

Where exposure level is not known, wear NIOSH approved, positive pressure, self-contained respirator. Where exposure level is known, wear NIOSH approved respirator suitable for level of exposure. In addition to the protective clothing/equipment in the above Section

(Engineering Controls and Personal Protective Equipment), wear impervious boots. Cover spills with some inert absorbent material; sweep up and place in a waste disposal container. Flush area with water.

Handling and Storage

Do not get in eyes, on skin, on clothing. Buildup of heat and pressure in closed containers may result if polymerization occurs. Avoid contamination with iron, copper, aluminum, brass, bronze, acids, bases, oxidizing, reducing and chelating agents, precipitant for copper and known initiators for vinyl polymerizations. Wash thoroughly after handling. Keep container closed. Wear clean work clothing daily.

This product is stabilized by dissolved oxygen, cupric ion (Cu+2) and MEHQ (monomethyl ether of hydroquinone). Maintain air sparge at 0.2-0.4 cfm per 1000 gal. Maintain temperature between 32-85°F. Maintain pH between 5.5 and 7.0. To prevent loss of dissolved oxygen: do not heat, do not use an inert blanket, and do not sparge with an inert gas. Avoid temperatures above 85°F, initiators such as bisulfites, peroxides, reducing agents, oxidizing and redox systems.

Drums that have contained the material should be stored separately and not used for any other purpose. These drums should be incinerated. It is important that surfaces subject to acrylamide contamination be thoroughly cleaned on a routine basis. This is to prevent inadvertent skin contact and reduce airborne limits since acrylamide sublimates (goes directly from solid to vapor). NOTE: Due to sublimation of the dried material, a concentration of vapor up to 120 mg/M³ can be reached in an enclosed area at 40°C.

Inhibition

Oxygen is the best inhibitor for NMA. As shipped, NMA is saturated with oxygen. The air in the head space of the container should be sufficient to maintain an adequate oxygen level for 90-days storage under normal conditions. NMA also contains 30± 5 ppm MEHQ.

All conditions leading to the displacement of oxygen from the solution should be avoided.

Storage Temperatures

N-methylolacrylamide should be stored in a cool place where the temperature range is between 32°F (0°C) and 85°F (29°C). Storage at temperatures above 85°F is not recommended. Drums placed near heat sources or in warm warehouses or vehicles have burst on occasion as a result of polymerization and the contents have spilled over the immediate area. The material is easily swept up, but, of course, is no longer usable.

At temperatures below 14°F (-10°C), N-methylolacrylamide will crystallize from a 48% solution. If this happens, slowly warm the contents to about 32°F (0°C) while occasionally mixing or rolling the drum to facilitate redissolving the crystals. This will in no way affect the quality of the material. If cold solutions are to be warmed, the use of tempered water up to 120°F (49°C) is recommended. **Steam or other direct heat must not be used to warm NMA solutions.**

Length of Storage

Drums containing N-methylolacrylamide should not be stored for more than three months, even under ideal conditions. Thus, it is important to observe a FIFO system of stock rotation so that the oldest material is used first.

Partially Emptied Containers

Once a drum of N-methylolacrylamide has been opened, the entire contents should be used if possible. If this cannot be done, replace the lid immediately after withdrawing the required amount while making certain that the polyethylene disc liner on the lid is in place so that the N-methylolacrylamide solution will not come into contact with the metal lid. The container should not be opened when there is a possibility of contamination from foreign matter.

Effect of pH

48% N-methylolacrylamide is shipped in the pH range of 5.5-6.5. The pH of 48% NMA tends to drift downward with time. This tendency is minimized if the temperature is maintained between 32-85°F (0-28°C). At a pH below 5.0 both vinyl and crosslinking reactions can take place simultaneously if accidentally initiated. Thus, it is important to monitor the pH of NMA. Make adjustment with 3% sodium hydroxide solution if pH drifts below 5.0 and back adjust if required with 5% formic or sulfuric acid.

Processing Equipment; Materials of Construction

Storage Tanks — Pipes, fittings, valves, flanges and pumps.

Acceptable Materials

- Stainless steel type 304 or 316.
- Fiber glass-reinforced, corrosion resistant polyester*.
Example: Laminac polyester resin 4173 (U.S. Steel Chemicals), Atlac polyester resin 382 (ICI America), Hetron polyester resin 197 (Ashland).
- Phenol-formaldehyde resin coating*.
Example: Lithcote LC-24 (Lithcote Company).
- Poly (vinyl chloride) coating*.
Example: Lithcote LC-100 (Lithcote Company).
- Polypropylene or Teflon tetrafluoroethylene fluorocarbon polymer** lining in stainless steel sheath.

Unacceptable Materials

Carbon steel, iron, copper, brass, bronze and aluminum **must not be used.**

Foreign matter, such as dirt, fibers, pigments, heavy metal salts, crystals and polymers can be very harmful to N-methylolacrylamide because this monomer is subject to crosslinking. The presence of impurities such as these would act as sites to promote crosslinking during polymerization, particularly emulsion polymerization. In many instances, filters are installed in feed lines to prevent dirt particles and crystals from entering the kettle or the batch.

Equipment Design and Metering Devices

Good equipment design is an important factor in preventing pre-polymerization during pumping operations. N-methylolacrylamide may tend to holopolymerize at points of strong turbulence during both stirring and transporting through feed lines. Sharp bends in pipes, scratched or partially corroded equipment, and agitators constructed of aluminum, iron, copper or brass may

* These materials were tested and found to be acceptable. Customers contemplating use of these materials should conduct their own tests.

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promote formation of homopolymer. In addition to those mentioned above Monel^{®***} metal, glass and plastic are also satisfactory construction materials.

Piping must be designed so that all lines containing NMA solution are self-draining and contain no stagnant areas.

All process equipment should be cold water flushed and air dried following use.

N-methylolacrylamide can be pumped and metered with the usual equipment used for low-viscosity liquids, provided that the materials of construction meet the conditions previously specified.

Effect of Light

Sunlight or strong light from other sources should be avoided. Light acts as a catalyst in the production of free radicals which will promote homopolymerization, especially in the presence of metallic impurities.

Oxidizers

Any conditions giving rise to peroxides promotes polymerization of N-methylolacrylamide. Adequate control has been taken to prevent premature polymerization by the addition of a small amount of MEHQ to the 48% solution at the manufacturing plant. The small amount of inhibitor will not hinder subsequent copolymerizations.

Solvents as Stabilizers

Small amounts of solvents such as methanol, ethanol, isopropanol, ethyl acetate, butyl ethylene glycol monobutyl ether and methyl ethyl ketone may often be added to N-methylolacrylamide by the user to impart stability for storing, pumping and metering the feed solutions.

Determination of Prepolymer

N-methylolacrylamide is soluble in n-butanol; the polymer is not. To determine the possible presence of polymer in N-methylolacrylamide as supplied, add 25 grams of the 48% solution to 25 grams of n-butanol. Allow it to stand for 30 minutes. If polymer is present, a precipitate will form. Filter, dry and weigh the precipitate to determine the polymer content. This method may also be used for dilute N-methylolacrylamide solutions.

Bulk Storage and Handling

Caution:

N-methylolacrylamide solution (48% in water) is a potentially hazardous material. In the interest of safety and health this material must be handled and stored properly.

- N-methylolacrylamide should be handled as a toxic monomer.
- Storage tanks must not be filled beyond 75% of capacity.
- If initiated, chemical reaction of 48% N-methylolacrylamide solution can generate sufficient heat and pressure to burst closed vessels or vessels with restricted openings.

Details concerning the toxicity of N-methylolacrylamide and factors affecting its storage stability are discussed in this brochure.

Handling precautions and procedures must be made clear to all personnel involved in storing and handling this product, and they should be carefully supervised to assure compliance.

Precautions should be taken to maximize the stability of N-methylolacrylamide solution in bulk storage.

1. Provide for air sparge 0.2-0.4 cfm / 1000 gal.
2. Avoid contamination of N-methylolacrylamide solution with copper, aluminum, brass, bronze, iron, acids, bases, oxidizing agents, reducing agents, vinyl polymerization initiators, and certain organic compounds identified elsewhere in this brochure.
3. Recommended bulk storage temperature is 32-85°F (0.29°C).
4. Recommended pH is 5.5-7.0.
5. Do not store longer than 3 months at 32-85°F (0-29°C). If stored above this temperature range, the length of storage will be considerably reduced.
6. Storage tanks should be emptied, cleaned and inspected every 3 months.

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Handle N-methylolacrylamide as a toxic monomer:

- Do not allow N-methylolacrylamide solution, spray, vapor, or dust to contact skin, eyes or clothing.
- Do not breathe vapors, spray or dust.
- Keep all empty containers in a protected location.
- Clean up spills and dispose of N-methylolacrylamide solution and rinse waters properly.
- Safety showers should be available.

Details of the above mentioned precautions are discussed in this brochure.

Cytec Industries Inc. is prepared to discuss handling and storage methods concerning N-methylolacrylamide solution and to offer suggestions relating to the design of handling systems for N-methylolacrylamide.

Unloading Station

All fittings, pumps, in-line instruments and piping should be constructed of stainless steel type 304 or 316 or other suitable materials. N-methylolacrylamide solution should be transferred through pipes or into tanks at a temperature between 32-85°F (0-29°C). During winter months care must be taken to avoid temperatures below 32°F (0°C) to prevent crystallization. Therefore, all exterior unloading facilities should be insulated. Draining and flushing of transfer lines are absolutely necessary — **stagnant material left in lines will gel, causing plugging of lines.** Tank trucks must be rinsed with water after contents are removed.

Handling and Metering

N-methylolacrylamide solution has a low viscosity. Thus, it can be transferred through meters by gravity flow and by pumps. Explosion-proof electrical equipment is not necessary for N-methylolacrylamide, but many of the monomers with which N-methylolacrylamide can be copolymerized do require explosion-proof electrical equipment. Any type pump that does not generate excessive heat in the liquid may be used, such as the centrifugal pumps.

Aeration

Bulk storage tanks for N-methylolacrylamide solution must be provided with continuous air sparging. Clean air must be used (i.e., instrument air). The air rate should be 0.2-0.4 cfm per 1,000 gallons of storage capacity. For a 24,000 gallon (75% full) tank, the sparging rate may be set at about 5.4 cfm of air. The rate of sparging must be sufficient to maintain oxygen in the solution and to

agitate the solution mildly, but not to cause evaporation of the water. Air flow must be monitored to assure constant aeration.

Pipe Lines

Piping must be designed so that all lines containing N-methylolacrylamide solution are self-draining and contain no stagnant areas. Where necessary, three-way plug valves with dead leg draining should be provided. Pipes should be insulated to maintain required temperatures. Pipes from storage tanks to polymerization kettles must be designed to prevent back-up of polymerization solution into storage tanks.

Insulation and Temperature Control

Storage tanks and all lines containing N-methylolacrylamide solution should be adequately insulated. Bulk storage of N-methylolacrylamide solution should be maintained between 32-85°F (0.29°C). One method of cooling is by recirculation of the tank contents through heat exchangers. A temperature sensor and alarm system are recommended.

Storage Tank Venting

Storage tank venting must be provided as a precaution in case polymerization of the N-methylolacrylamide solution is initiated accidentally. The vent sizes are derived by orifice flow calculations.* This calculation requires knowledge of the pressure rating of the storage tank, the amount of liquid in storage and the heat release rate expected during spontaneous polymerization. The last variable is difficult to determine because the amount of an initiator accidentally introduced cannot be predicted. As a practical matter, the vent should be made as large as possible. A 6" vent plus a 20"-24" Protectoseal** manhole vent or equal is believed adequate to protect a 10,000-gallon tank in the event of polymerization caused by contact with metal or other contaminants, loss of air sparge or external heat.

Water Quench

Storage tanks must not be filled beyond 75% of capacity to allow space for solution contact with air and to allow adequate room for dilution with water should polymerization begin. Means for rapid addition of cold quench water must be provided in case the tank contents begin to polymerize, as indicated by a rapid rise in temperature. Mixing can be accomplished by a recirculation loop.

*Perry, John H. *Chemical Engineering Handbook*, second edition. Equation 13 (page 403) and Equation 14 (page 404).

**Protectoseal, Engineering and Manufacturing Company, 1920 South Western Avenue, Chicago, Illinois 60608.

Diking

The storage tank should be surrounded by a dike adequate to contain the total contents of the tank in case of tank failure.

Monitoring

The temperature, pH and air sparge should be monitored as an aid in preventing premature polymerization of the N-methylolacrylamide solution in the storage tank. A polymer check should be made daily.

Air Sparge

An air flow meter should be set up to monitor the air at the specified rate. It is recommended that an alarm be provided to signal discontinuation of air flow. **Air sparge is by far the most important factor in maintaining NMA stability.**

Temperature

Temperature of stored solutions should be checked frequently (every eight hours is suggested). A temperature sensor connected to an alarm is recommended. Set the sensor to sound an alarm if the temperature of the N-methylolacrylamide solution rises 9°F (5°C) above normal storage temperature. If the cause of a temperature rise is found to be a mechanical failure, take appropriate corrective measures. If corrective measures cannot be instituted immediately, and the temperature continues to rise, then activate the quenching procedure.

pH

The pH should be checked daily when stored at recommended temperatures. At higher storage temperatures more frequent pH checks are necessary. Adjustments in pH should be made with 3% sodium hydroxide solution and 5% formic or sulfuric acid.

Length of Storage

NMA should not be stored in bulk for more than 3 months. If stored above the recommended temperature the length of storage in bulk will be considerably reduced.

Cleaning Storage Tanks Prior to N-methylolacrylamide Service

All tanks, drums and containers to be used for storing N-methylolacrylamide solution, temporarily or for a prolonged period of time, must undergo a rigid inspection and a thorough cleaning and rinsing before they can be put into service. The following procedures are mandatory for assuring safe storage of N-methylolacrylamide solution:

1. Check previous history of tank or container. If it has a history of leaks, inadequate piping, poor temperature control, or was used for storing strong oxidizers or reducers, such as nitric acid, hydrogen peroxide, persulfates, metabisulfites, hyposulfites, sulfites, or metallic powders or salts, such as those of iron, aluminum, copper, brass, tin, zinc, or contains any materials of construction other than those recommended, then it cannot be used for storing N-methylolacrylamide solution without major overhaul.
2. Inspect tank or container thoroughly for cleanliness (i.e., film on walls, inside of covers or piping; and debris on interior surfaces). Manual scouring or abrasive cleaning is usually essential for tanks or containers previously used for storage of chemicals which leave residues that cannot be removed by use of detergents and water rinses. All surfaces and pipes should be visibly clean and should not leave dirt or stains on a wiping cloth.
3. Fill the tank or container with water. Add detergent as required. Circulate water or detergent solution through tank and all piping. Inspect for leaks, empty and rinse well with fresh water until the washings are clear, colorless and are about neutral in pH. Continue cleaning and rinsing until such is the case.
4. After the washings meet the specified requirements, a final rinse with deionized water is recommended.

If all the other items of safety have been approved, the resultant tank or container may be used for storing N-methylolacrylamide solution.

Once the storage system is in use for storing N-methylolacrylamide solution, periodic inspection for leaks, cleanliness and absence of contamination is advised. If the storage system is to be emptied and is to be idled, it should be thoroughly rinsed with water to remove residual N-methylolacrylamide solution.

Potential Applications

Many applications of N-methylolacrylamide compounds are known and many detailed references describing the most recent applications are listed and summarized in the Cytec booklet entitled "NMA — Applications — Processes — Products — References". (PRT-708-B)

Listed below are major potential applications along with the co-reactants used to produce the desired product.

Application	Monomers Reacted With N-methylolacrylamide And Reference
Surface coating and molding resins	Aliphatic and monomeric amides (28). Vinyl ethers and esters, phenols, or cresols (23).
Varnishes, films, and adhesives	Acrylamide or other monomers (29). Methacrylic acid, methyl methacrylate, and methacrylic anhydride (30). Vinyl ethers and esters, phenols, or cresols (23).
Bonding agents for paper and paper-pigment binders	Vinyl propionate, <i>tert</i> -butyl acrylate and acrylic acid (31).
Sizing Agents	Alcohols (32). Vinyl ethers and esters, phenols or cresols (23). Vinyl monomers or sulfonated reaction products of N-methylolacrylamide (33).
Wet-strength and dry-strength agents for paper	Vinyl acetate and an internal plasticizing monomer such as vinyl stearate (34).
Binders for flocking fibrous materials	Butyl acrylate, ethyl acrylate, acrylonitrile and acrylic acid (35).
Textile finishing agents	Acrylonitrile or ethyl acrylate (37). Compounds that release water upon condensation, such as urea (38). Alcohols (32).
Irradiation polymerization in textile finishing	N-methylolacrylamide (39).

Application	Monomers Reacted With N-methylolacrylamide And Reference
Anti-static agents	Poly (alkyleneoxy) ester (40). Vinyl monomers or sulfonated reaction products of N-methylolacrylamide (33).
Dispersing agents	Vinyl monomers or sulfonated reaction products of N-methylolacrylamide (33).
Polymer emulsifying agents	Acrylic esters (41). Alkyl acrylates, dialkyl maleates or dialkyl fumarates (21).
Crosslinking agents	Aliphatic and aromatic epoxy resins (42). Ethyl acrylate (43). Polyvinyl alcohol (44, 45).
Emulsion polymers	Vinyl monomers (literature available upon request).
Automotive product adhesives for brake bodies, clutch discs and abrasive discs	Vinyl ethers and esters, phenols or cresols (23).
Additives in oil well drilling fluids and drilling muds	Vinyl monomers or sulfonated reaction products of N-methylolacrylamide (33).
Soil conditioners and soil stabilization	N,N'-methylenebis-acrylamide (46, 47). Monomer containing maleic groups or homopolymer (16). Monomeric triazine (48). N-methylolacrylamide (49). Vinyl monomers or sulfonated reaction products of N-methylolacrylamide (33).

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