

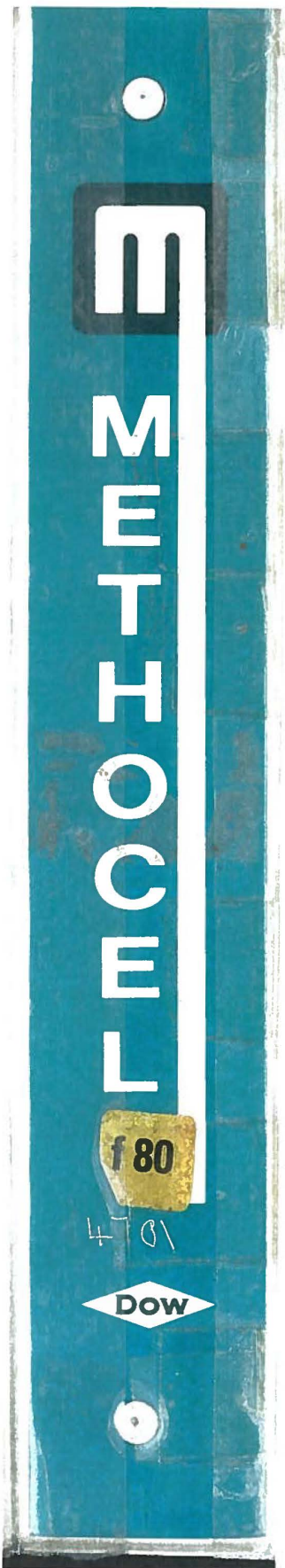
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METHOCEL

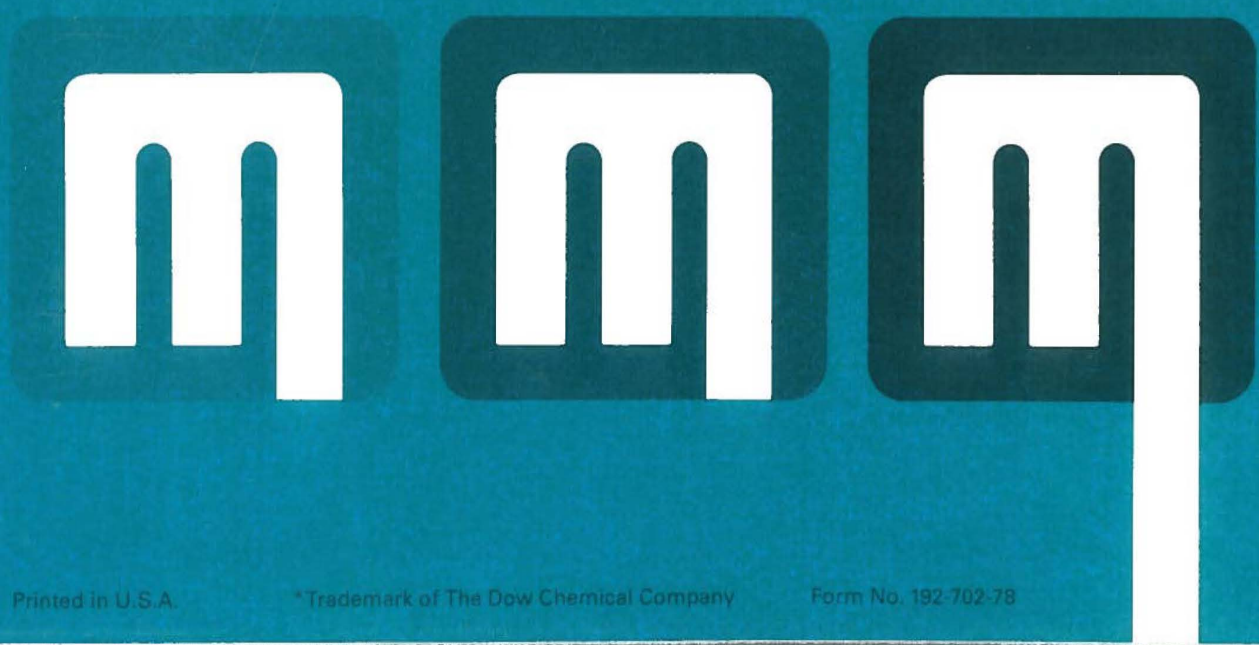
Cellulose Ethers





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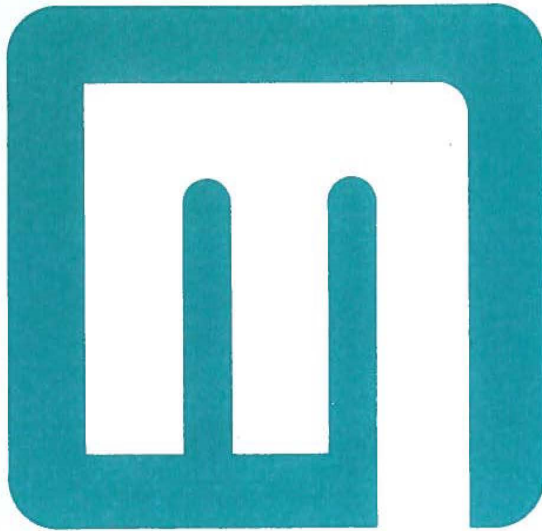
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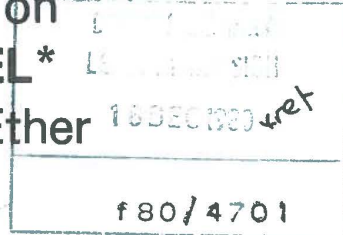
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Handbook on
METHOCEL*
Cellulose Ether
Products



Because you need quality, reliability and performance predictability in a thickener, protective colloid, film-former, emulsifier... that's why Dow designs, manufactures, and continues to improve METHOCEL water-soluble cellulose ether products.



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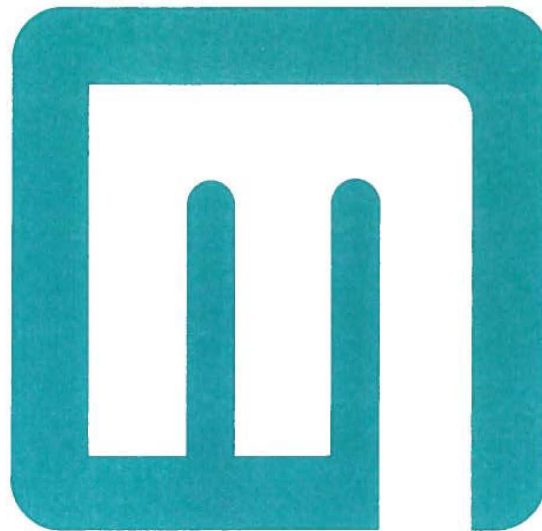
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Handbook on **METHOCEL*** Cellulose Ether Products



Because you need quality, reliability and performance predictability in a thickener, protective colloid, film-former, emulsifier... that's why Dow designs, manufactures, and continues to improve METHOCEL water-soluble cellulose ether products.



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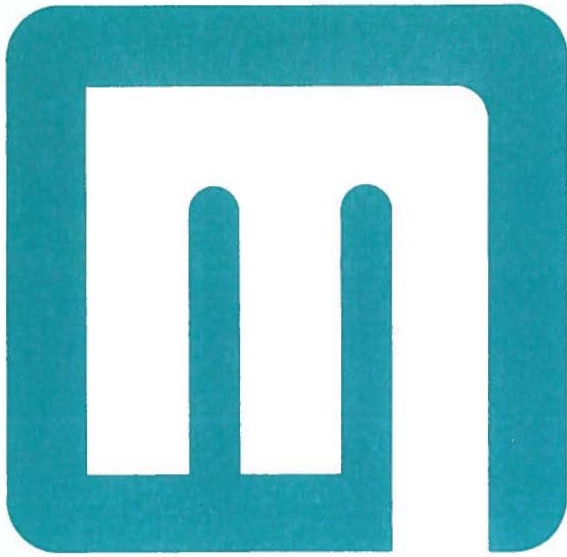


HANDBOOK ON METHOCEL* CELLULOSE ETHER PRODUCTS

SECTION 1	PRODUCT BASICS	
	TYPES	1•1
	GRADES	1•1
	VISCOSITIES	1•1
	PHYSICAL FORMS	1•1
	PRODUCTS / FORMS / VISCOSITIES / GRADES	1•2
SECTION 2	PROPERTIES / APPLICATIONS / BY PRODUCTS	
	PROPERTIES	2•1
	APPLICATION LITERATURE	2•2
	APPLICATIONS	2•3
	APPLICATIONS BY PRODUCTS	2•5
SECTION 3	STRUCTURES / D.S.	
	METHYLCELLULOSE	3•1
	HYDROXYPROPYL METHYLCELLULOSE	3•1
	HYDROXYBUTYL METHYLCELLULOSE	3•2
	D.S. — DEGREE OF SUBSTITUTION	3•2
	D.S. AND WT. % FOR VARIOUS PRODUCTS	3•3
SECTION 4	SOLUTION PREPARATION	
	PREPARATION	4•1
	TREATED POWDERS	4•1
	UNTREATED POWDERS	4•3
SECTION 5	PROPERTIES: POWDER	
	MISCELLANEOUS PROPERTIES	
	Resistance to Microorganisms	5•1
	Moisture Sorption	5•1
	Equilibrium Moisture Content, 25°C	5•2
	SOLUBILITY IN NON-AQUEOUS SOLVENTS	
	Solvent Solubility at Ambient Temp.	5•3
	Solvent Solubility at Elevated Temp.	5•3
	Representative Solvents at Elev. Temp.	5•4
SECTION 6	PROPERTIES: SOLUTION	
	FUNDAMENTAL PROPERTIES OF SOLUTIONS	
	General Aqueous Solution Properties	6•1
	Viscosity, MW / Viscosity	6•3
	Effect of Concentration on Viscosity	6•5
	Effect of pH on Viscosity	6•7
	Effect of Additives on Viscosity	6•7

—continued

	Rheology of Solutions	6•9
	Thermal Gelation, Aqueous Media	6•11
	Gel Strength, Texture	6•12
	Effect of Additives on Thermal Gel	6•13
	Gel Point Raising Additives	6•14
	FACTORS AFFECTING USE OF SOLUTIONS	
	Recommended Concentrations for Coatings	6•15
	Surface Activity, Aqueous Solutions	6•17
	Defoamers, Aqueous Solutions	6•17
	Effect of Freezing, Solutions	6•19
	Preservatives, Aqueous Solutions	6•21
	Compatibility, Aqueous Solutions	6•21
SECTION 7	PROPERTIES: FILM	
	GENERAL FILM PROPERTIES	7•1
	EFFECT OF ADDITIVES	7•2
	RESISTANCE TO SOLVENTS	7•2
	THERMOPLASTIC FORMING	7•2
SECTION 8	REGULATED USES	
	FOODS	8•1
	“A” Brand Products	8•1
	“E”, “F”, “K”, Brand Products	8•1
	PESTICIDE USE	8•1
	PHARMACEUTICALS, PARENTERAL INJECTIONS	8•2
	COSMETICS	8•2
SECTION 9	ANALYSIS	
	MEASUREMENT OF VISCOSITY	9•1
	Ubbelohde Tube Measurement	9•2
	SUMMARY, OTHER METHODS	9•3
	References	9•3
SECTION 10	HANDLING CONSIDERATIONS	
	HEALTH	10•1
	STORAGE	10•1
	SPILLS, HOUSEKEEPING	10•1
	DISPOSAL	10•1
	FACSIMILE MATERIAL SAFETY DATA SHEETS	
	Methylcellulose	10•3
	Hydroxypropyl Methylcellulose	10•5
	Hydroxybutyl Methylcellulose	10•7



SECTION 1 PRODUCT BASICS

Because you need quality, reliability and performance predictability in a thickener, protective colloid, film-former, emulsifier ... that's why Dow designs, manufactures, and continues to improve METHOCEL water-soluble cellulose ether products.

1
PRODUCT BASICS

TYPES	1•1
GRADES	1•1
VISCOSITIES	1•1
PHYSICAL FORMS	1•1
PRODUCTS/FORMS/VISCOSITIES/GRADES ..	1•2

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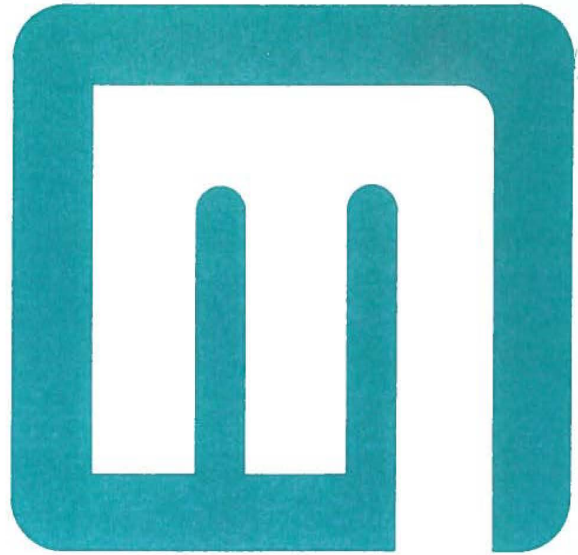
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TYPES	1•1
GRADES	1•1
VISCOSITIES	1•1
PHYSICAL FORMS	1•1
PRODUCTS/FORMS/VISCOSITIES/GRADES ..	1•2

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METHOCEL*

Product Information

PRODUCT BASICS

TYPES

METHOCEL cellulose ether products are made by reacting cellulose with appropriate chemical reagents in the presence of caustic soda. The products are manufactured in essentially three different chemical types:

- Methylcellulose...made using methyl chloride...these are the METHOCEL A brand products.
- Hydroxypropyl methylcellulose...made using propylene oxide and methyl chloride...these are the METHOCEL E, F, J, and K brand products.
- Hydroxybutyl methylcellulose...made using butylene oxide and methyl chloride...these are the METHOCEL HB brand products.

Brands	Chemical Types
METHOCEL A METHOCEL E, F, J, K METHOCEL HB	Methylcellulose Hydroxypropyl methylcellulose Hydroxybutyl methylcellulose

GRADES

METHOCEL cellulose ether products are supplied in two grades: *premium* and *standard*.

ALL *premium*-grade METHOCEL methylcellulose and hydroxypropyl methylcellulose products:

- are accepted as additives for a variety of food and drug uses
- meet the requirements of the Food Chemicals Codex and the international Codex Alimentarius

METHOCEL A brands of *premium*-grade cellulose ether products:

- meet *U.S. Pharmacopeia* (U.S.P.) requirements for methylcellulose

METHOCEL E, METHOCEL F, and METHOCEL K brands of *premium*-grade cellulose ether products:

- meet *U.S. Pharmacopeia* (U.S.P.) requirements for hydroxypropyl methylcellulose

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VISCOSITIES

METHOCEL cellulose ether products are available in various viscosity types, ranging from 5 cps to 75,000 cps. All solution viscosities are measured with Ubbelohde capillary tubes at a 2 percent concentration at 20°C (68°F). Viscosities available for each product designation are summarized below: Labelling will use the abbreviation "C" for 100 and "M" for 1000 units.

Products	Viscosities Available
METHOCEL A	15; 400; 1,500; 4,000
METHOCEL E	5; 15; 50; 4,000
METHOCEL F	50; 4,000; 40,000
METHOCEL J	5,000; 12,000; 20,000; 40,000; 75,000
METHOCEL K	35; 100; 4,000; 15,000
METHOCEL HB	12,000
METHOCEL 228	4,000
METHOCEL 240	40,000
METHOCEL 856	75,000

PHYSICAL FORMS

METHOCEL cellulose ether products are available in two physical forms: **Powder** (*and Surface-Treated Powder*) and **Granular**. Solutions of untreated powder must be prepared by the hot-cold technique or alternate methods (described in Section 4 of the handbook). Surface-treated powder forms are dispersible in cold water. The dissolution of surface-treated powder products can be controlled by a shift in pH. The following table shows the individual products available in each physical form. Surface treatment is designated by the letter "S".

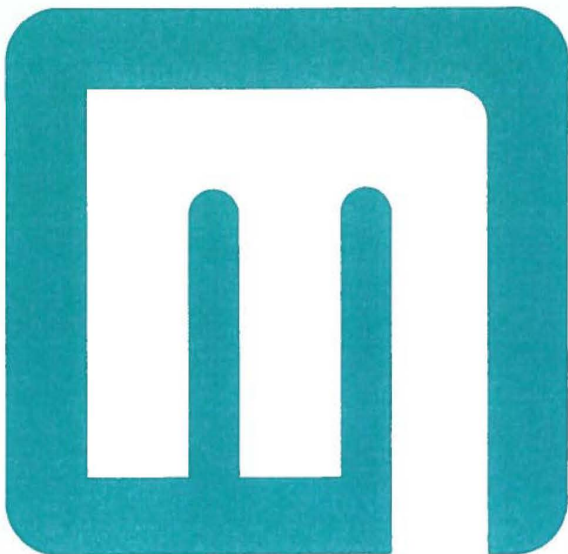
Physical Form	Product Availability
Powder	Most types, grades, and viscosities are available in powder form.
Surface-Treated Powder	METHOCEL 228, METHOCEL 240S METHOCEL J5MS, METHOCEL J12MS, METHOCEL J20MS METHOCEL J40MS METHOCEL J75MS, METHOCEL 856S METHOCEL K15MS
Granular	METHOCEL K4MDGS METHOCEL K15MDGS

Product information on Types, Grades, Viscosities, and Forms of METHOCEL cellulose ether product is summarized on p 1•2.



Products, Forms, Viscosities, Grades for METHOCEL Cellulose Ether Products

VISCOSITY*	5	15	35	50	100	4C	15C	4M	5M	12M	15M	20M	40M	75M
RANGE	4-6	13-19		40-60	80-120	350-550	1200-1800	3500-5600			12,000-18,000		30,000-50,000	
METHOCEL A Methylcellulose		P st pr				P st pr	P st	P st pr						
METHOCEL E Hydroxypropyl Methylcellulose	P P	P pr		P pr				P pr						
METHOCEL F Hydroxypropyl Methylcellulose				P st pr				P st pr						
METHOCEL J Hydroxypropyl Methylcellulose									SP st	SP st		SP st	SP st	SP st
METHOCEL K Hydroxypropyl Methylcellulose			P st		P st pr			SG st P st pr			st pr P st pr SP st			
METHOCEL HB Hydroxybutyl Methylcellulose										P st				
METHOCEL 228								SP st						
METHOCEL 240													P st SP st	
METHOCEL 856														P st SP st
<p>Grades P — Powder, untreated SP — Powder, surface-treated SG — Granular, surface-treated</p> <p>Forms st — standard pr — premium</p>														
* in 2 percent aqueous solutions at 20°C, viscosity in centipoise														



SECTION 2 PROPERTIES/ APPLICATIONS/ BY PRODUCTS

2

PROPERTIES/APPLICATIONS/BY PRODUCTS

Because you need quality, reliability and performance predictability in a thickener, protective colloid, film-former, emulsifier... that's why Dow designs, manufactures, and continues to improve METHOCEL water-soluble cellulose ether products.

PROPERTIES	2•1
APPLICATION LITERATURE	2•2
APPLICATIONS	2•3
APPLICATIONS BY PRODUCTS	2•5

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METHOCEL*

Product Information

PROPERTIES AND APPLICATIONS

PROPERTIES

METHOCEL cellulose ether products have an exceptional and valuable combination of *physical* and *chemical* properties. These include:

Water Solubility

They dissolve in water in all proportions; the maximum concentration is limited only by viscosity.

Organic Solubility

Certain types and grades are soluble in a number of binary organic and organic-water solvent systems; these provide a unique combination of organic and water solubility.

No Ionic Charge

They have no ionic charge (are not polyelectrolytes); therefore, they will not complex with metallic salts and ionic organics to form insoluble precipitates.

Thermal Gelation

Their aqueous solutions gel when heated beyond a definite temperature, providing controllable quick-set properties. The gels formed go back into solution upon cooling, unlike gels formed by protein thickeners.

Surface Activity

They provide surfactancy in solutions where emulsification, protective colloid action, and phase stabilization are required. Their surface tensions range from 42 to 56 dynes per cm (water is 72 dynes per cm); a typical surfactant has a surface tension of 30 dynes per cm.

Metabolic Inertness

Used as food and drug additives, they are not metabolized and therefore do not add calories in the diet.

Enzyme Resistance

They are comparatively enzyme-resistant, providing excellent viscosity stability during long-term storage.

Low Ash

The non-ionic structures leave low residual ash, a benefit in refractory uses.

Low Taste and Odor

They are therefore excellent in use in food and pharmaceutical applications.

pH Stability

They have a useful and wide pH range of 3.0-11.0.

Water Retention

They retain water in the presence of water-absorbing porous surfaces such as wallboard, cement blocks, bricks, etc.

METHOCEL cellulose ether products also have a number of noteworthy *performance* properties making them useful as:

Thickeners

They thicken both aqueous and non-aqueous systems. The degree of thickening is related to specific product viscosity and chemical type.

Film Formers

They form clear, tough, flexible films that are excellent barriers to oils and greases.

Binders

They have excellent functionality as binders for pigments, tobacco products, paper... in both coating and adhesive end-uses.

Lubricants

They reduce friction in rubber, asbestos cement, and ceramic extrusions and improve pumpability of concrete slurries.

Suspending Aids

They prevent solid particles from settling and thus inhibit the formation of sediments.

Protective Colloids

They prevent droplets and particles from coalescing or agglomerating.

Emulsifiers

They stabilize emulsions both by reducing surface and interfacial tension, and by thickening the water phase.

2 • 1

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2 • 2 Application and formulation information is available in the following reports. Please request from your Dow salesman those in which you are interested.

Application Literature

Title or Description	Form No.
METHOCEL In Bakery Goods	192-347
METHOCEL In Food Dressings	192-348
METHOCEL In Breaded Foods	192-349
METHOCEL In Low Cal Salad Dressing	192-350
Why/How To Specify METHOCEL	192-400
METHOCEL In Fried Foods	192-405
METHOCEL Thickeners in Latex Paint	192-406
METHOCEL In Paint Removers	192-407
METHOCEL In PVC Processing	192-408
METHOCEL In Dietetic Foods	192-409
METHOCEL, Aqueous Systems for Tablet Coating	192-622-77
METHOCEL In Ceramic Mixes	192-626-78
METHOCEL In Detergent Additives	192-639-78
METHOCEL In Food Products	192-642-78
METHOCEL In Cosmetics and Toiletries	192-671-78
METHOCEL In Drywall	192-672-78

Application literature is regularly updated and new pieces are regularly produced. Please have your Dow salesman keep your literature on METHOCEL current.



METHOCEL*

Product Information

PROPERTIES AND APPLICATIONS

APPLICATIONS

Increasing numbers of manufacturers use the favorable properties of METHOCEL cellulose ether products to improve many commercial products. Special bulletins on many of these uses are available.

METHOCEL cellulose ether products are used in:

Adhesives

For their innate adhesive properties and to provide thickening to adhesive formulations.

Agricultural Chemicals

As spray adherents, fungicide stickers, protective films, and dispersing agents for wettable powders.

Ceramics

To provide green strength, water retention, and lubricity in refractory mortars and cements, and in glaze slips. They leave minimal ash when fired.

Chemical Specialties

As thickeners, suspending agents, binders, film formers, and emulsion stabilizers in chemical-manufacturing and processing operations.

Construction Products

To provide water retention and to add workability to cement and gypsum formulations such as dry-wall joint cements, cement plasters and stucco, tile mortars and grouts, and portland cement mortars.

Cosmetics

To provide viscosity control, emulsification and stabilization, lubricity and feel, clarity, foam stability, and surfactant compatibility in lotions, hand and face creams, hair dressings, deodorants, depilatory creams, shampoos, and toothpastes.

Foods

As thickeners, binders, emulsifiers, stabilizers, and colloidal suspending agents in a variety of products, including salad dressings, fruit pie fillings, baked goods, dietetic foods, breading batters,

fried foods, milk shake drinks, and convenience snacks. The thermal gelation property can be used to advantage in many of these applications.

Leather

The thermal gelation property is utilized in pasting adhesives during the drying operation, and the thickening property is an advantage in leather finishing.

Paint

As protective colloids, thickeners, and pigment-suspension aids for latex paints. The products offer viscosity stability, wet-edge retention, ease and flexibility of incorporation, and also contribute to paint film integrity.

Paint Removers

Where the unique combination of organic and water solubility allows use as a thickener for scrape-off and flush-off paint removers.

Paper Products

As film formers useful for greaseproof coatings, adhesives, surface sizings, and release coatings.

Pharmaceuticals

As binders, granulating agents, and film coatings for tablets; as bulk laxatives; as film formers for burn therapy; as stabilizers for ointments and creams; as thickeners for lotions and jellies; and as troche bases.

Polyvinyl Chloride

As protective colloids in suspension polymerizations to provide improved resin porosity, higher plasticizer absorption rates, and control of resin particle-size distribution.

Plywood

To control viscosity in glues used for lamination.

Printing Inks

As thickeners and suspending agents for water-base inks.

Resins

As mold-release agents for fiber-reinforced plastics and as thickeners and stabilizers for water-based coating emulsions.

2•3

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PROPERTIES AND APPLICATIONS

Rubber

As mold-release agents and as stabilizers and thickeners in rubber latexes.

Textiles

As binders in textile printing pastes, in latex coatings, and as sizing agents. As thickeners for carpet backsizing formulations, and for providing faster drying speeds, wider effective coating viscosity range, more uniform coating, higher holdout, and quick grab.

Tobacco

As binders and film formers for reconstituted tobacco sheets, and as adhesives in cigar and cigarette fabrication.

Choice of the specific METHOCEL cellulose ether product will vary in each industry according to the application. The range of METHOCEL cellulose ether products is deliberately designed to accommodate the widest possible range of formulating and processing equipment, and of application environments.



PROPERTIES AND APPLICATIONS

APPLICATIONS BY PRODUCTS

The types of METHOCEL cellulose ether products used commercially in different applications are listed in the following table. For specific formulation assistance or for further information on making a choice of product best suited for your specific need, contact the Designed Products group in your local DOW Sales Office.

ucts are intended to simplify customers' understanding of their purchases. The prefix letter designates chemical type. The numeral denotes the centipoise viscosity, with a C and an M indicating the viscosity in hundreds and thousands, respectively. Suffix designation of S denotes surface-treated powder. If a product has no such suffix designation, it is available only in non surface-treated powder form.

The designations for METHOCEL cellulose ether prod-

APPLICATION	METHOCEL A	METHOCEL E	METHOCEL F	METHOCEL J	METHOCEL K
ADHESIVES					
Leather Pasting	A4M				
Temporary Binding Agent for Glass Fibers	A15				
Thickener in Phenol-Formaldehyde Adhesives	A4M				
Stationery Adhesives	A4C		F4M 228		K15M
Wallpaper	A4M				K4M K4M DGS
General Adhesives	A4M		F4M		
AGRICULTURE					
Dispersing Agent For Wetting Powders	A15	E50	F50 F4M		
Dust Stickers					
Seed Stickers	A15				
Spray Drift Control	A15			J75MS	
Spray Stickers			F4M		
Weed Killers	A15C				
Suspending Aids				J5MS-J75M	K15M

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PROPERTIES AND APPLICATIONS

APPLICATION	METHOCEL A	METHOCEL E	METHOCEL F	METHOCEL J	METHOCEL K
ASPHALT Asphalt Emulsion Release Coating	A15			J12MS J75MS	K4M K35
CAULKING COMPOUNDS		E50	F4M	J5MS J12MS	K4M K15M
CERAMICS Refractory Mortars Glaze Slips Hi-temp Glaze Slips Porcelain Enamels Cements Tile Mortars Plastic Mixes	A4M A15 A4M A15 A4M A4M A4M		F4M F4M F4M F4M F4M	J5MS J5MS	K4M K4M
CHEMICAL SPECIALTIES Aerosols Cleaning and Polishing Compounds Insecticides Sanitizers	A15 A4M A15 A4M	E50	F50 F4M	J5MS	K4M
CONSTRUCTION PRODUCTS Dry Wall Joint Cements Masonry Mortars Pumpability Aids Release Coatings Stuccos Tile Grouts and Adhesives	A15		F4M F4M F4M F4M	J5MS-J75MS J5MS-J12MS J75MS J5MS-J12MS J5MS J12MS 856	K4M-K15M K4M-K15M K15M K4M-K15M K4M K15M

—Continued

PROPERTIES AND APPLICATIONS

APPLICATION	METHOCEL A	METHOCEL E	METHOCEL F	METHOCEL J	METHOCEL K
COSMETICS					
Creams and Lotions		E4M		1	K4M K15M
Deodorants				1	
Hair Dressings		E4M		1	
Shampoos		E50, E4M			
Tooth Pastes			F4M	1	K4M, K15M
FOODS					
Baked Goods	A4M		F4M		K4M
Breading Batters	A15		F50		K100
Dietetic Foods	1	1	1		1
Milk Shake Drinks	A15	E50	F50		
Pie Fillings	A4M		F4M		K4M
Salad Dressings	A4M				K4M
Snack Foods	1	1	1		1
Whipped Toppings		E15	F50		K100
LATEXES					
Creaming of Natural Rubbers	A4M		F4M	J5MS	K4M K15M K100
Protective Colloids	A15	E50	F50		
Thickeners			228	J75MS	
LEATHER					
Finishings	A4M			J5MS J12MS	
Pasting Adhesives	A4M		F4M		
PAINTS					
Acrylics, Polyvinyl Acetate, Styrene-Butadiene				J5MS J12MS J20MS J40MS J75MS	K4MS K15MS

¹Viscosities dependent on application. Consult Dow.

—Continued

PROPERTIES AND APPLICATIONS

APPLICATION	METHOCEL A	METHOCEL E	METHOCEL F	METHOCEL J	METHOCEL K	METHOCEL HB
PAINTS—cont. Cement Paints Multicolor Lacquers Texture Paints	A4M		F4M F50-F4M F4M 228	J5MS J12MS J20MS J40MS J75MS	K4MS K15MS K4MS K15M	
PAINT REMOVERS		E4M	F4M PRG ¹		K15M	HB
PAPER Adhesives Barrier Coatings Dielectric Papers Release Coatings	A15-A4C A15-A4C A15-A4C A15-A4C		F50 F50 F50		K100 K100 K100	
PENCILS AND CRAYONS	A15-A4M		F50-F4M			
PHARMACEUTICALS Bulk Laxatives Creams and Ointments Ophthalmic Preparations Suspensions Tablet Binders Tablet Film Coats	A4M A4M A15	E4M E4M E50 E5-E15-E50	F4M F4M F4M F50		K15M K4M K4M K4M K100	

¹ Paint remover grade

—Continued

PROPERTIES AND APPLICATIONS

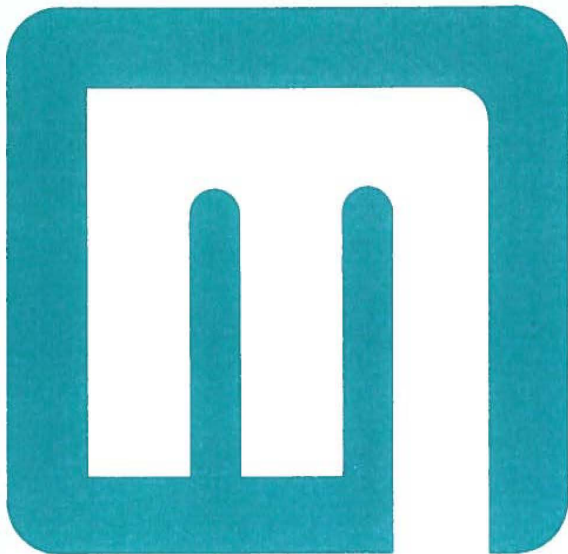
APPLICATION	METHOCEL A	METHOCEL E	METHOCEL F	METHOCEL J	METHOCEL K
PLYWOOD Control of Glue Viscosity	A4M		F4M		K15M
PRINTING INKS (Water-Base Inks)	A15-A4M	E15-E4M	F50-F4M	J5MS J12MS	K100-K4M
POLYVINYL CHLORIDE	A15	E5-E15-E50	F50		K35-K100
RESINS Emulsion Coatings Mold-Release Agents	A15	E4M	F4M F50	J5MS J12MS	K4M K15M K35-K100
RUBBER Latex Stabilizers and Thickeners Mold Release	A4M A15		F4M F50	J5MS J12MS	K4M K35-K100

—Continued

PROPERTIES AND APPLICATIONS

APPLICATION	METHOCEL A	METHOCEL E	METHOCEL F	METHOCEL J	METHOCEL K
TEXTILES					
Adhesives Carpet Backsizing	A4C		F50	J20MS	K35-K100 K15MS
Dye Thickening	A4M		F4M	J75MS	K4M K15M
Flocking Adhesives			F4M	J5MS J12MS	K4M K15M
Latex Coatings	A15 A4M		F4M	J12MS	K15M
Printing Pastes	A4M		F4M	J5MS J75MS	K15MS
Warp Sizes	A15		F50		K100
TOBACCO					
Reconstituted Sheet	A4C A4M		F4M		
Viscosity Control					K15M

Additional information on METHOCEL products and their applications is available from The Dow Chemical Company, Designed Products Department, 2040 Dow Center, Midland, Michigan, 48640.



SECTION 3 STRUCTURES/D.S.

3
STRUCTURES/D.S.

Because you need quality, reliability and performance predictability in a thickener, protective colloid, film-former, emulsifier ... that's why Dow designs, manufactures, and continues to improve METHOCEL water-soluble cellulose ether products.

METHYLCELLULOSE	3•1
HYDROXYPROPYL METHYLCELLULOSE	3•1
HYDROXYBUTYL METHYLCELLULOSE	3•2
D.S.—DEGREE OF SUBSTITUTION	3•2
D.S. AND WT. % FOR VARIOUS PRODUCTS .	3•3

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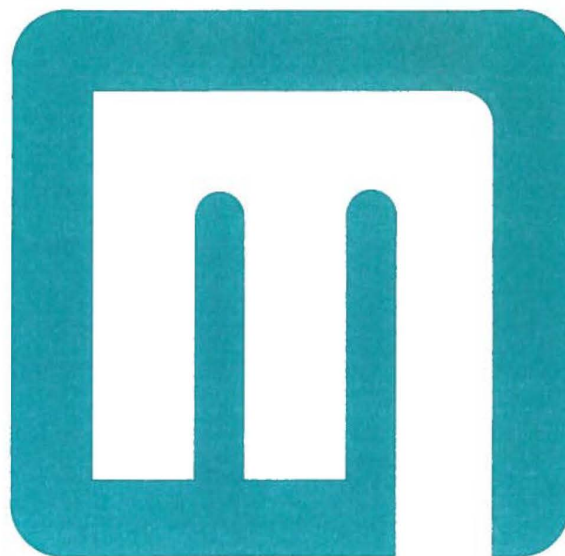
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SECTION 3 STRUCTURES/D.S.



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METHOCEL*

Product Information

STRUCTURES AND D.S.

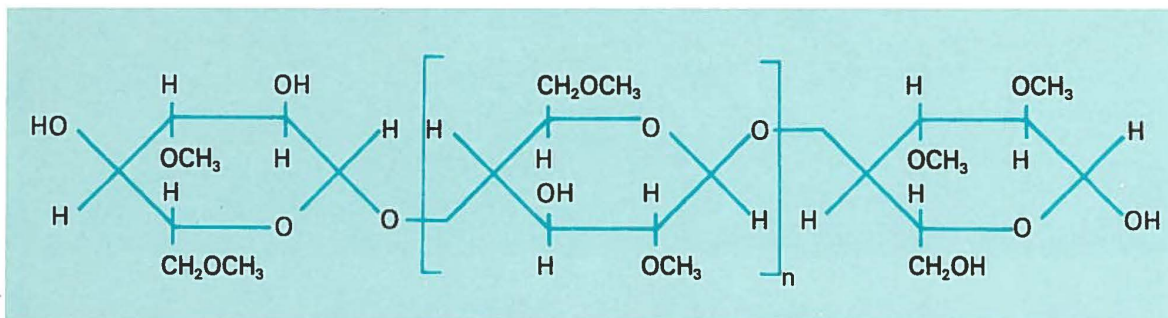
3•1

STRUCTURES

METHOCEL cellulose ether products are derived from and have the polymeric backbone of cellulose, a natural carbohydrate that contains a basic repeating structure of anhydroglucose units.

Methylcellulose ... METHOCEL A brand products—
Cellulose fibers, obtained from cotton linters or wood

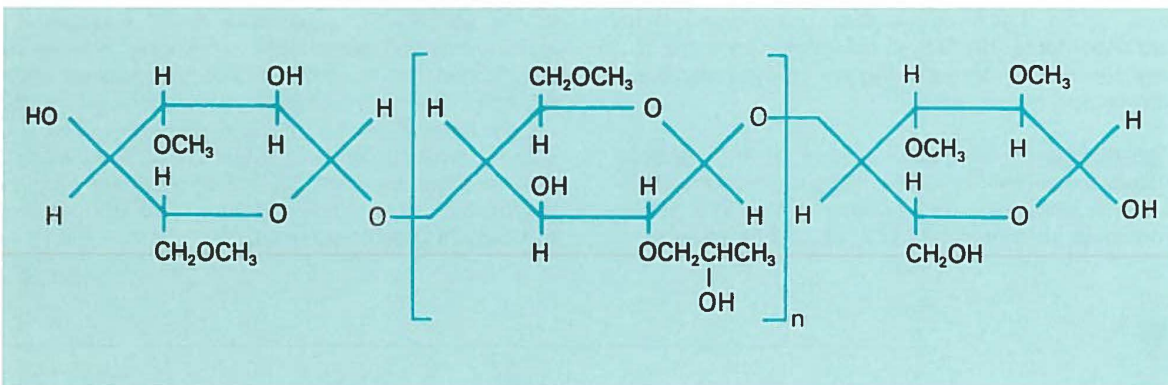
pulp, are treated with caustic solution to produce alkali cellulose which in turn is treated with methyl chloride, yielding the methyl ether of cellulose. The fibrous reaction product is purified and ground to a fine, uniform powder or granule. The products obtained have the chemical structure shown below.



Hydroxypropyl Methylcellulose ... METHOCEL E, METHOCEL F, METHOCEL J, AND METHOCEL K brand products—

In the manufacture of these products, propylene oxide is used in addition to methyl chloride to obtain hydroxypropoxyl substitution on the anhydroglucose units. This substituent group (-OCH₂CH(OH)CH₃) contains a secondary hydroxyl on the number two carbon,

and may also be considered to form a propylene glycol ether of cellulose. The products possess varying ratios of such propylene glycol ether substitution to methoxyl substitution, a factor which influences organic solubility and thermal-gel point temperature of aqueous solutions. The chemical structure of these products is:



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—Continued

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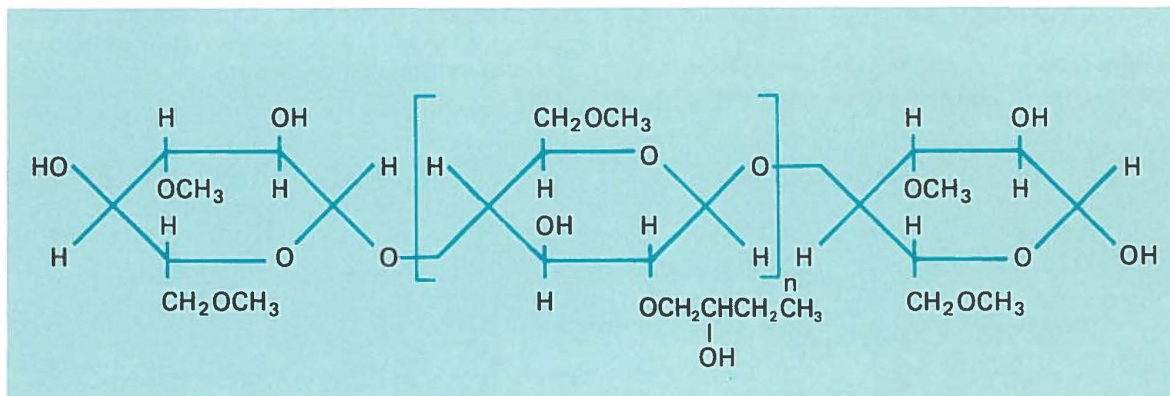


STRUCTURES AND D.S.

Hydroxybutyl Methylcellulose...METHOCEL HB brand products—

These products differ from hydroxypropyl meth-

ylcellulose in that 1,2-butylene oxide, rather than propylene oxide, has been used to obtain hydroxybutoxyl substitution on the anhydroglucose units. this substituent group, as the following formula shows, contains a secondary hydroxyl on the number two carbon.

**D.S.****Degree and Significance of Substitution**

The number of substituent groups on the anhydroglucose units of cellulose can be designated by weight percent or by the average number of substituent groups attached to the ring hydroxyls, a concept known to cellulose chemists as "degree of substitution," (D.S.). If all three available positions on each unit are substituted, the D.S. is designated as three; if an average of two on each ring are reacted, the D.S. is designated as two, etc.

The number of substituent groups on the ring determines the properties of the various products. METHOCEL A cellulose ether contains 27.5 to 31.5 percent methoxyl, or a methoxyl D.S. of 1.60 to 1.9, a range

that yields maximum water solubility. A lower degree of substitution gives products having lower water solubility; higher degrees of substitution produce methylcellulose products that are soluble only in organic solvents. In the METHOCEL E, METHOCEL F, and METHOCEL K cellulose ether products, the methoxyl substitution is still the major constituent, comprising 80 to 95 percent of the groups attached to the ring.

In the METHOCEL J cellulose ether products, the hydroxypropoxyl substitution is roughly half of the substitution. In the METHOCEL HB cellulose ether products, the hydroxybutoxyl group comprises 2-5 weight percent of the cellulose derivative. The substitution is similar to that of the METHOCEL A cellulose ether product, but the modification resulting from the hydroxybutoxyl groups is sufficient to impart improved solubility in organic solvents.

—Continued

STRUCTURES AND D.S.

3•3

The preceding "substitution" information on METHOCEL cellulose ether products is summarized in the following table.

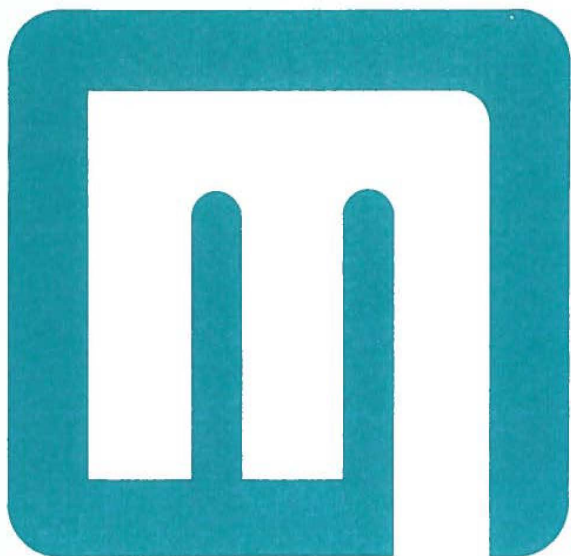
Degree of Substitution and Typical Weight Percent

Products	Methoxyl Degree of Substitution	Methoxyl Percent ⁽¹⁾	Hydroxy-propoxyl MS ⁽²⁾	Hydroxy-propoxyl Percent ⁽¹⁾	Hydroxy-butoxyl Ether MS ⁽²⁾	Hydroxy-butoxyl Ether Percent
METHOCEL A Prem³ & Std⁴	1.6-1.9	27.5-31.5	—	—	—	—
METHOCEL E Prem	1.8-2.0	28-30	0.20-0.31	7-12	—	—
METHOCEL F Prem & Std	1.7-1.9	27-30	0.10-0.20	4.0-7.5	—	—
METHOCEL J Std	1.1-1.6	16.5-20	0.7-1.0	23-32	—	—
METHOCEL K Prem & Std	1.1-1.6	19-25	0.10-0.3	4-12	—	—
METHOCEL HB Std	1.9 min	31.0 min	—	—	0.04 min	2 min

ASTM D 1347 is the method for testing methoxyl in METHOCEL A products.

¹ ASTM D 2363 is method for testing for methoxyl and hydroxypropoxyl content

² Molar substitution ³Premium Grade ⁴Standard Grade



SECTION 4 SOLUTION PREPARATION

Because you need quality, reliability and performance predictability in a thickener, protective colloid, film-former, emulsifier... that's why Dow designs, manufactures, and continues to improve METHOCEL water-soluble cellulose ether products.

PREPARATION	4-1
TREATED POWDERS	4-1
UNTREATED POWDERS.....	4-3

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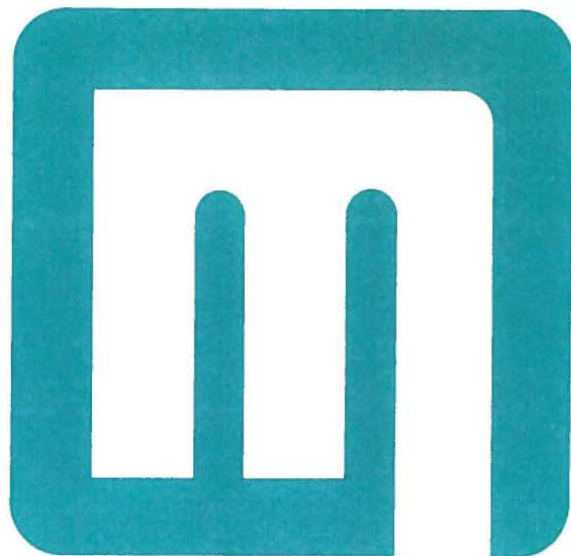
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SECTION 4 SOLUTION PREPARATION



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METHOCEL*

Product Information

SOLUTION PREPARATION

PREPARATION OF METHOCEL SOLUTIONS

METHOCEL cellulose ether products are carbohydrate polymers which dissolve both in water and in some instances in certain organic solvents... by swelling and successive hydration of their structural layers. Because of the swelling mechanism, there is no sharp solubility limit such as occurs in the dissolution of ionizing salts. The concentration of METHOCEL cellulose ether products in solution is usually limited by the viscosity that a manufacturer is equipped to handle. This also would depend on the viscosity and type of the product being used. The low-viscosity products can be made at 10-15 percent concentration; the high-viscosity products find a normal limit at 2-3 percent concentration.

The physical form of METHOCEL cellulose ether product chosen (powder or surface-treated powder) influences the preparation techniques used in making solutions. Regardless, there are three basic steps in making good gel-free solutions of METHOCEL:

1. **Disperse**—allow each particle of METHOCEL cellulose ether to be wet out.
2. **Agitate**—keep dispersed.
3. **Dissolve**—hydrate (build viscosity).

It is important to *disperse* the particles before attempting to dissolve them. Good dispersion prevents lumping caused by the formation of gels.

Each of the different physical forms requires different techniques for most effective solution preparation.

TREATED POWDERS

The surface-treated powders of METHOCEL cellulose ether products are added directly to water systems. Such powder products will *disperse* readily with mild agitation and are dissolved (build viscosity) when made alkaline.

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METHOCEL cellulose ether surface-treated powders provide exceptional handling flexibility and control of solubilization rate. The hydration of these products has been inhibited in a manner that permits the use of pH to control the point at which solubilization will occur. They are readily wet out by cold water and will disperse uniformly without lumping, and with a minimum of agitation.

Aqueous slurries, up to concentrations as high as 10 percent, can be prepared readily from surface-treated powders with minimum agitation and using ordinary tap water. Above this concentration, the holding time decreases and the material quickly becomes too viscous to pour or pump readily.

Aqueous slurries can be held for 45 minutes and still remain usable. However, complete solubilization of the slurried powder is achieved rapidly and at a desired point and time by making the slurry alkaline. Suitable methods include:

1. Addition of sufficient ammonium hydroxide to the aqueous dispersions (after the proper concentration of all products has been obtained by diluting with water) to obtain a pH of 8.5-9.0 results in rapid viscosity development and a smooth and gel-free solution*. **CAUTION:** Attempts to adjust the pH of high concentration slurries may lead to excessively high viscosities so that the slurry cannot be pumped or poured. *The pH adjustment should be made only after the slurry is diluted to the concentration at which it will be used.*

NOTE: Once the pH has been shifted to the alkaline side (pH 8.5-9.0) to permit full and rapid solubilization of the surface-treated product, solutions are stable over the pH range of 3-11.

2. The addition of a slurry to an alkaline pigment grind or filler dispersion (or vice-versa) or the addition of slurry to a basic pigment-latex formulation provides rapid solubilization and uniform viscosity development.
3. The addition of dry alkaline pigments or fillers to a slurry on high or low speed mixing equipment also results in rapid solubilization and viscosity development.

*U.S. Patent 3,297,583 covers dissolution by pH adjustment immediately after formation of the slurry.

— Continued

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4 • 2

SOLUTION PREPARATION

The surface-treated METHOCEL cellulose ether products can be added into latex paint at various points in the manufacturing process, either by direct powder addition, a glycol slurry, or through the use of a stock solution. They can be added, for example:

1. As dispersible powders, directly into the pigment grind, prior to the pigments. NOTE: These thickeners

should not be added at the end of formulation preparation with high solids, or to formulations that will remain neutral or acid in pH without a shift in pH to alkaline values.

2. As stock solutions, glycol slurries, or water slurries into the finished pigment grind.
3. As stock solutions, glycol slurries, or water slurries into the paint letdown after incorporating the latex.



METHOCEL*

Product Information

SOLUTION PREPARATION

UNTREATED POWDERS

Three different techniques are commonly used to disperse untreated powders of METHOCEL cellulose ether products:

1. Dispersion in Hot Water

Untreated METHOCEL cellulose ether powders have the unique and valuable property of hot-water insolubility coupled with solubility in cold water

Untreated METHOCEL cellulose ether powders can be dispersed by mixing thoroughly with 1/5 to 1/3 of the required total volume of water as hot water (80-90°C; 176-194°F). Mixing (agitation) should continue until all particles are thoroughly wetted.

For complete solubilization, the remainder of the water should then be added as cold water or even as ice to obtain the proper temperature. Agitation should continue until the mixture is smooth.

NOTE: For maximum clarity and reproducible control of viscosities, solutions of METHOCEL A cellulose ether (methylcellulose) products should be cooled to 0-5°C (32-41°F) for 20-40 minutes.

In general, solutions of METHOCEL E, METHOCEL F, METHOCEL J, and METHOCEL K cellulose ether (hydroxypropyl methylcellulose) products require cooling to a temperature of 20-25°C (68-77°F) or below.

Because METHOCEL cellulose ether products disperse well in water above 80°C (176°F), the preliminary use of hot water assures better wetting of the powder particles prior to addition of cold water. If cold water is mixed directly with the powder, it creates a gelatinous membrane on the outside of the particles which causes lumping and slow diffusion of water into the interior of the particles. *It is very important, however, to have*

adequate cooling after wetting with hot water to insure complete dissolution of METHOCEL. Where low temperature water is not available, the use of METHOCEL E, METHOCEL F, METHOCEL J, and METHOCEL K cellulose ether products, which do not require as much cooling as METHOCEL A or METHOCEL HB grades, is suggested.

The figure indicates that slurries of METHOCEL K cellulose ether require much less cooling than those of METHOCEL A product for the material to be put in solution. Slurries of the other hydroxypropyl methylcellulose products (those with "E," "F," "J" in the product designation) also require less cooling than the "A" brand products.

2. Dispersion in Non-Aqueous Solvents

METHOCEL cellulose ether products in untreated powder form may be dispersed into a water-miscible organic solvent such as alcohol or glycol at a ratio of 5-8 parts of solvent to 1 part METHOCEL cellulose ether powder. The dispersion of METHOCEL cellulose ether product and organic solvent may then be added to cold water or the cold water to it.

NOTE: When adding the water, if there are not 5-8 parts of organic solvent available from the formulation, the mixture may be too thick and pasty to give good results.

3. Dispersion by Dry Blending

METHOCEL cellulose ether products in untreated powder form can be used in formulations containing other dry powder ingredients by dry blending thoroughly prior to the addition of water. The use of other dry powder products keeps the particles of METHOCEL dispersed.

The figure on page 4 • 4 illustrates the effects of cooling hot slurries of METHOCEL K15C and METHOCEL A15C cellulose ether products, showing the degree of cooling required for maximum viscosity development.

4 • 3

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— Continued

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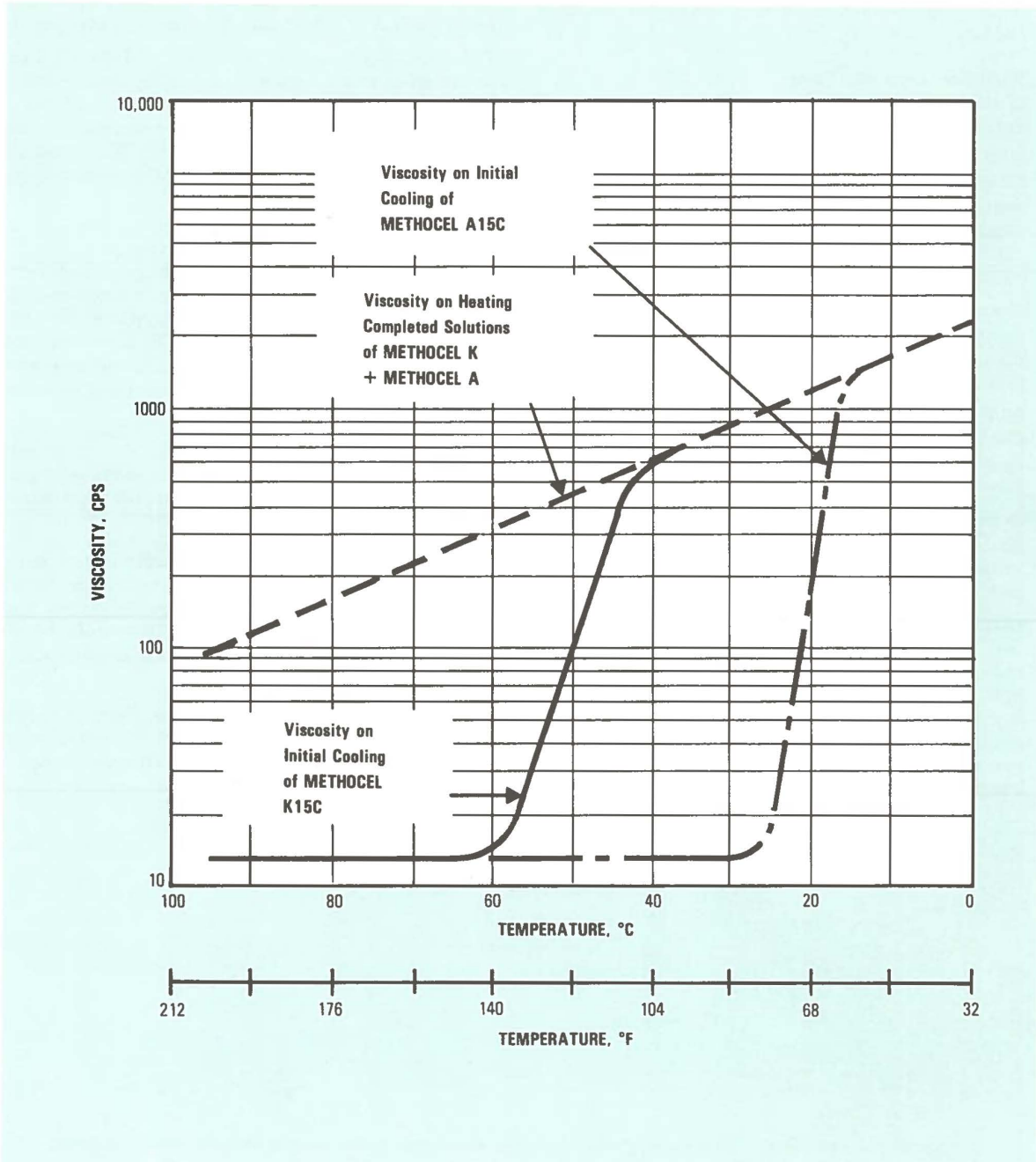
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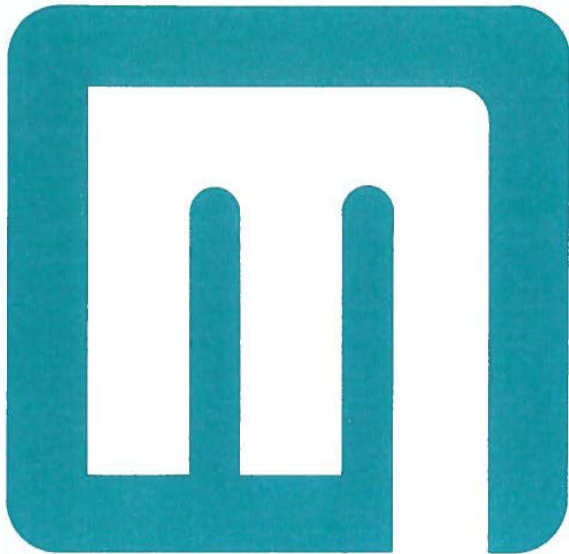
Mylan v. Qualicaps, IPR2017-00203
QUALICAPS EX. 2017 - 33/86

4 • 4

SOLUTION PREPARATION

*Viscosity Development of METHOCEL K15C and METHOCEL A15C
Brand Products Slurried at 2 Percent in Hot Water*





SECTION 5 PROPERTIES: POWDER

Because you need quality, reliability and performance predictability in a thickener, protective colloid, film-former, emulsifier... that's why Dow designs, manufactures, and continues to improve METHOCEL water-soluble cellulose ether products.

MISCELLANEOUS PROPERTIES	5•1
Resistance to Microorganisms	5•1
Moisture Sorption	5•1
Equilibrium Moisture Content, 25°C	5•2
SOLUBILITY IN NON-AQUEOUS	
SOLVENTS	5•3
Solvent Solubility at Ambient Temp.	5•3
Solvent Solubility at Elevated Temp.	5•3
Representative Solvents at Elev. Temp.	5•4

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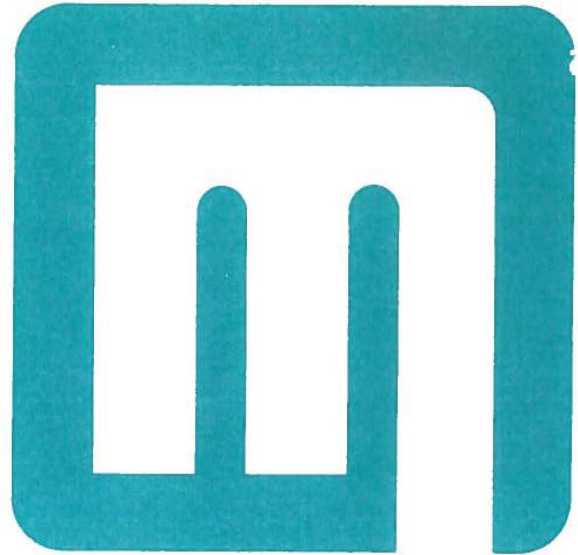
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SECTION 5 PROPERTIES: POWDER



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SOLVENTS	5•3
Solvent Solubility at Ambient Temp.	5•3
Solvent Solubility at Elevated Temp.	5•3
Representative Solvents at Elev. Temp.	5•4

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METHOCEL*

Product Information

POWDER

5 • 1

PROPERTIES: POWDER

Miscellaneous Powder Properties of METHOCEL Products

Physical Appearance	white to slightly off-white, essentially odorless and tasteless powders
Apparent Density g/cc	0.25-0.70
Browning Temperature	190-200°C (374-392°F)
Charring Temperature	225-230°C (437-446°F)
Relative Flammability in a Furnace at 700°C (1292°F)	90+
Specific Gravity	1.39
Weight/ Gallon, Pounds	11.6

Resistance to Microorganisms

An important property of METHOCEL cellulose ether products is their high resistance to attack by microorganisms. The higher substitutions of the METHOCEL J brand products are especially resistant to enzymes. Maintenance of shelf life in paints and other latex-based coatings, and stability of a range of solutions and other products containing METHOCEL cellulose ether are greatly increased by this resistance to microorganisms.

The fact that both METHOCEL methylcellulose and METHOCEL hydroxypropyl methylcellulose can be completely recovered from the intestinal tract after ingestion is for all practical purposes proof that they are stable to a wide range of bio-chemical and enzyme systems.

As the cellulose is modified by substitution with various radicals, such as alkyl and hydroxyalkyl groups, resistance to microbial attack increases. Reese, Siu, and Levinson¹ found that the degree of substitution (D.S.) of water-soluble cellulose derivatives was a primary factor, with a threshold D.S. value of 1.0 required for protection. Because the METHOCEL cel-

lulose ether products have excellent uniformity of substitution, higher than a D.S. of 1.0, they do possess excellent resistance to microbial attack.

Moisture Sorption

METHOCEL cellulose ether products sealed in their original shipping containers absorb little to no atmospheric moisture. Once a container is opened however, there is pick-up of moisture from the air. When "exposed" METHOCEL cellulose ether is weighed, a portion of the total weight therefore may be water. Such weight must be corrected for moisture content to assure using the proper weight of METHOCEL cellulose ether to give the viscosity desired. To minimize moisture pick-up, opened bags should be tightly resealed. The moisture sorption rate of METHOCEL K brand products is somewhat greater than that for METHOCEL A and METHOCEL HB brand cellulose ether products; the moisture sorption rates are however about the same within a single type. Typical moisture sorption is shown on the following graph, page 5•2.

¹ H. S. Levinson and E. T. Reese, *J. Gen. Physiol.* 33, No. 601 (1950); E. T. Reese, R. G. H. Siv, and H. G. Levinson, *J. Bacteriology*, 59, No. 485 (1950); E. T. Reese; *Ind. Eng. Chem.* 49, No. 104 (1957).

—Continued

NOTE: Blending chart for METHOCEL powders is located in Section 6, page 6•6.

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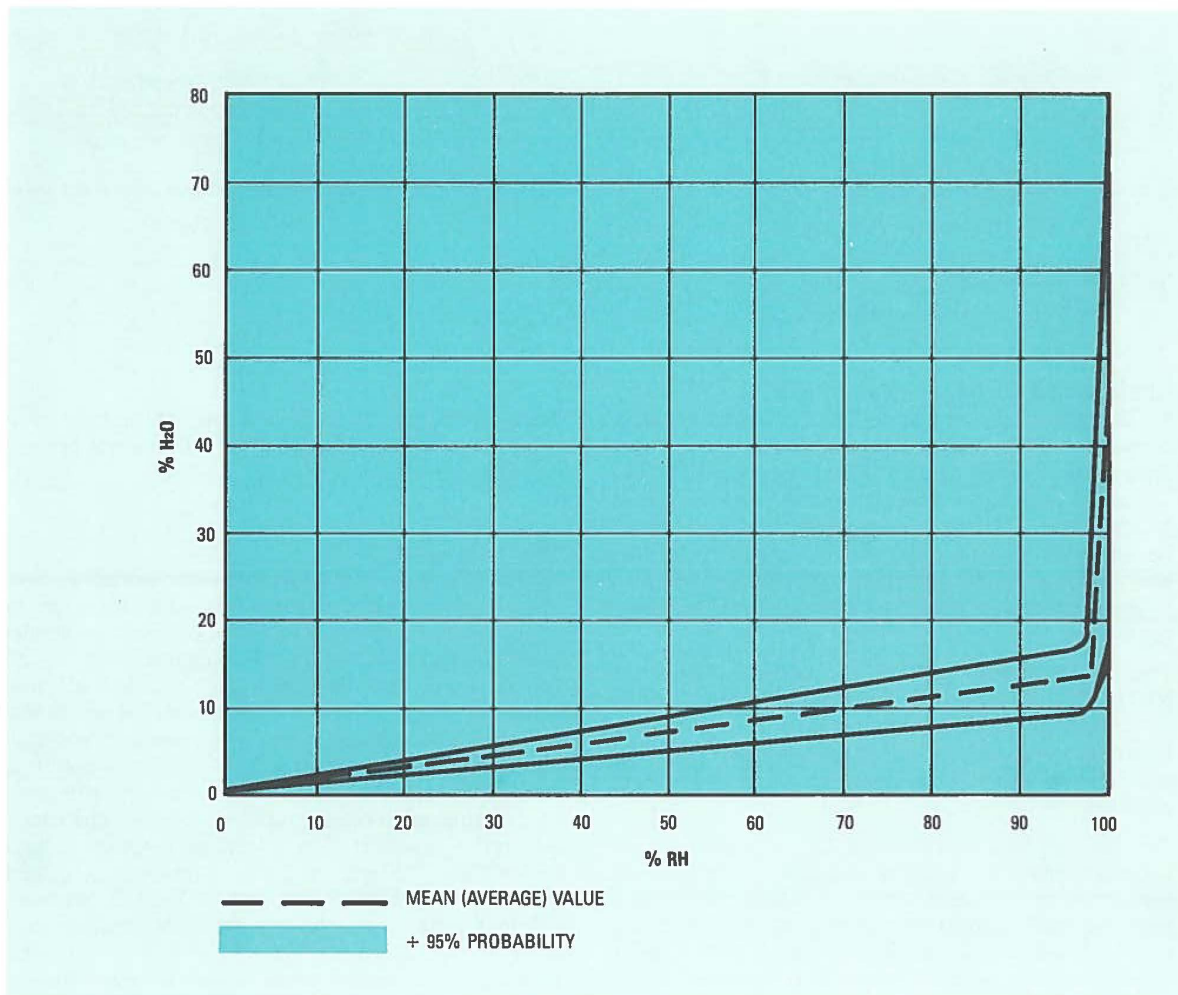
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5 • 2 POWDER

Equilibrium Moisture Content 25°C vs Percent Relative Humidity for METHOCEL Products





METHOCEL*

Product Information

POWDER

SOLUBILITY IN NON-AQUEOUS SOLVENTS

The solubility of METHOCEL cellulose ether products in non-aqueous media will vary according to the nature and quantity of the substituent groups on the anhydroglucose chain. Hydroxypropyl methylcellulose types, such as METHOCEL E and METHOCEL J brands, and hydroxybutyl methylcellulose, METHOCEL HB brand, are the products of choice for end-users where solubility in, or compatibility with, non-aqueous media is desired. *Since the surface-treated products are designed to control dispersibility and solubility rate in aqueous media*, those forms should not be used in non-aqueous media.

Because METHOCEL cellulose ether products are soluble in water, their solubility in non-aqueous solvents offers unique dual solubility. The use of non-aqueous coating systems to apply water-soluble films to pharmaceutical tablets has resulted in substantial economies and improved efficiency in pharmaceutical tablet manufacture. This capability of METHOCEL cellulose ether product is under investigation in other application areas.

Solvent Solubility at Ambient Temperatures

In general, binary solvent systems function more effectively than single solvents. Where alcohols comprise part of the binary solvent, solvent power improves as the

molecular weight of the alcohol decreases; methanol therefor performs very well.

5 • 3

The following compounds are typical of the types of solvent that can be used with certain METHOCEL cellulose ether products:

- | | |
|---|---------------------|
| Furfuryl alcohol | Propylene carbonate |
| Dimethyl formamide | Formic acid |
| Dimethyl sulfoxide | Glacial acetic acid |
| Methyl salicylate | Pyridine |
| Mixtures of methylene chloride and ethyl, methyl, or isopropyl alcohols | |
| Mixtures of chloroform and methanol or ethanol. | |

Solvent Solubility at Elevated Temperatures

METHOCEL "E", METHOCEL "J", and METHOCEL "HB" cellulose ether products possess structures that provide unusual solubility properties. They are soluble in certain non-aqueous media at elevated temperatures, permitting the formation of mixes which can be fabricated by techniques of extrusion, hot-melt casting, injection and compression molding. Examples of suitable "hot solvents" are found in the table on page 5 • 4.

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— Continued

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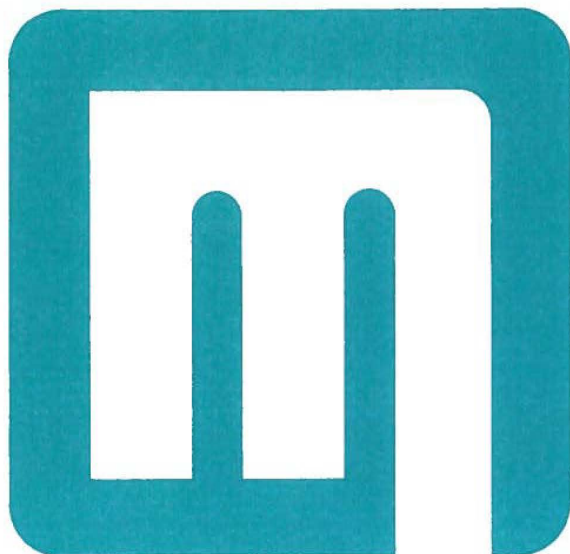
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5 • 4 POWDER

Representative Solvents for METHOCEL E and METHOCEL J and METHOCEL HB Brands at Elevated Temperatures

Compound	Boiling Point, °C	Solubility Point, °C	Degree of Solubility ¹
GLYCOLS			
Ethylene glycol	197.3	158	C
Diethylene glycol	244.8	135	C
Propylene glycol	188.2	140	C
1,3-Propanediol	214	120	C
Glycerine	290	260	P
DOWANOL* EE	134.7	120	C
ethylene glycol ethyl ether			
DOWANOL TPM	242.4	160	P
tripropylene glycol methyl ether			
ESTERS			
Ethyl glycolate	160	110	C
Glyceryl monoacetate (Acetin)	127/3 mm	100	C
Glyceryl diacetate (Diacetin)	123-133/4 mm	100	C
AMINES			
Monoethanolamine	170-172	120	C
Diethanolamine	268-269	180	C
* Trademark of The Dow Chemical Company ¹ C: completely soluble P: partially soluble			



SECTION 6 PROPERTIES: SOLUTION

Because you need quality, reliability and performance predictability in a thickener, protective colloid, film-former, emulsifier... that's why Dow designs, manufactures, and continues to improve METHOCEL water-soluble cellulose ether products.

FUNDAMENTAL PROPERTIES OF SOLUTIONS

General Aqueous Solution Properties	6•1
Viscosity, MW/Viscosity	6•3
Effect of Concentration on Viscosity	6•5
Effect of pH on Viscosity	6•7
Effect of Additives on Viscosity	6•7
Rheology of Solutions	6•9
Thermal Gelation, Aqueous Media	6•11
Gel Strength, Texture	6•12
Effect of Additives on Thermal Gel	6•13
Gel Point Raising Additives	6•14

FACTORS AFFECTING USE OF SOLUTIONS

Recommended Concentrations	
for Coatings	6•15
Surface Activity, Aqueous Solutions	6•17
Defoamers, Aqueous Solutions	6•17
Effect of Freezing, Solutions	6•19
Preservatives, Aqueous Solutions	6•21
Compatibility, Aqueous Solutions	6•21

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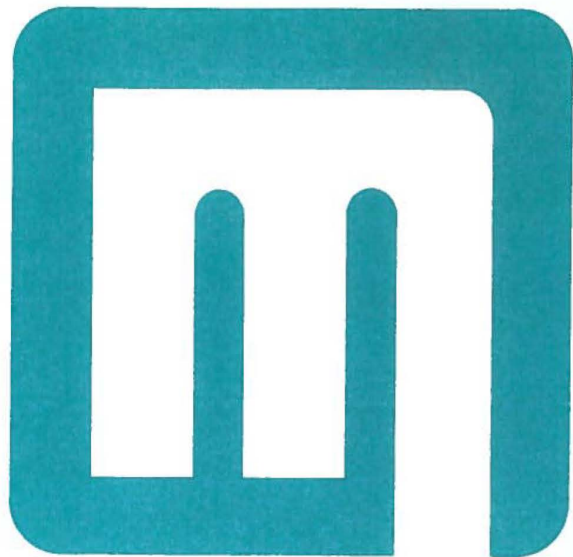
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Gel Strength, Texture	6•12
Effect of Additives on Thermal Gel	6•13
Gel Point Raising Additives	6•14

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Recommended Concentrations	
for Coatings	6•15
Surface Activity, Aqueous Solutions	6•17
Defoamers, Aqueous Solutions	6•17
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METHOCEL*

Product Information

SOLUTION

6 • 1

Fundamental Properties of Solutions

General Properties of Aqueous Solutions of METHOCEL

Specific Gravity, 20°/4°C (all types)			Freezing Point	°C
1%	1.0012		All types 2%	0.0
5%	1.0117			
10%	1.0245			
Refractive Index (2%) n _D ^{20°} (all types)			Surface Tension, ¹ 25°C	
1.336			Brand METHOCEL A. 47-53 dynes/cm METHOCEL E. 44-50 dynes/cm METHOCEL F. 44-50 dynes/cm METHOCEL J. 48-52 dynes/cm METHOCEL K. 50-56 dynes/cm METHOCEL HB. 49-55 dynes/cm	
Partial Specific Volume			Interfacial Tension ¹ (Paraffin Oil), 25°C	
Brand			Brand METHOCEL A. 19-23 dynes/cm METHOCEL E. 18-19 dynes/cm METHOCEL F. 19-23 dynes/cm METHOCEL J. 26-30 dynes/cm METHOCEL K. 26-28 dynes/cm METHOCEL HB. 20-22 dynes/cm	
METHOCEL A	4,000 cps	0.725 cc/g (0.087 gal/lb)	Specific Heat 10% METHOCEL A10M } 0.93 M 0.05 cal/gm/°C 25% METHOCEL A10 } from 20°C to 90°C	
METHOCEL E	4,000 cps	0.767 cc/g (0.092 gal/lb)		
METHOCEL F	4,000 cps	0.734 cc/g (0.088 gal/lb)		
METHOCEL J		0.725 cc/g (0.087 gal/lb)		
METHOCEL K	4,000 cps	0.717 cc/g (0.086 gal/lb)		
METHOCEL K	15,000 cps	0.724 cc/g (0.087 gal/lb)		
METHOCEL HB		0.774 cc/g (0.093 gal/lb)		

¹For solutions below 500 cps. In many cases, concentrations of 0.001% will give the same value as 1% solutions.

NOTE: Data on thermal gelation in aqueous media are located on pages 6•9—6•12.

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