

# Revealed to the second second

Physical and Chemical Properties



# Natrosol® HEC and Its Applications



# NATROSOL® Hydroxyethylcellulose

A Nonionic Water-Soluble Polymer

#### CONTENTS

PAGE

NATROSOL HYDROXYETHYLCELLULOSE — A NONIONIC WATER-SOLUBLE POLYMER 2 A Guide to Where and Why Natrosol	
Hydroxyethylcellulose Is Used 2	
CHEMISTRY	
Substitution	
Viscosity	
R-Grades	
B-Grades	
Particle Size 4	
Selected Types 4	
PROPERTIES 5	
Solubility in Water 5	
Moisture Absorption 5	
Dermatology and Toxicology 5	
FDA and EPA Status 6	
Microbiological Information 6	
Product Safety	
DISSOLVING NATROSOL HEC IN WATER 7	
Natrosol R-Grade	
Solubility in Organic Solvents	
OF NATROSOL HEC	
Viscosity	
Effect of Concentration	
Effect of Blending	
Effect of Shear Rate	
Effect of Mixed Solvents	
Effect of Temperature	
Effect of pH	
Viscosity Stability	
Preservatives11	
Use of Defoamers 12	
Tolerance for Inorganic Salts	

COMPATIBILITY WITH OTHER MATERIALS13	5
Latex Emulsions	;
Cellulosic Water-Soluble Polymers	;
Water-Soluble Resins 13	5
Natural Gums 13	
Preservatives or Biocides	;
Surfactants and Detergents	5
Defoamers	
Plasticizers	3
Organic Solvents13	
PROPERTIES OF FILMS OF NATROSOL 16	;
Unplasticized Films 16	;
Plasticizers 16	;
Heat-Seal Characteristics	;
Solubility in Water 17	,
Insolubility in Organic Solvents	,
Resistance to Grease and Oil	,
Photosensitivity 17	,
Adhesion	,
Insolubilizing Films of Natrosol	,
APPENDIX—METHODS OF ANALYSIS	
FOR NATROSOL 18	3
Viscosity Determination	5
Moisture Determination	3
Solution Preparation	3
Viscosity Measurement	)
Ash Content 19	)
Procedure	)
Calculation 19	)
QUALITY CONTROL, PACKAGING,	
AND SHIPPING 20	)
SOURCE AND DESCRIPTION OF	
TRADEMARKED PRODUCTS 20	)

# NATROSOL® HYDROXYETHYLCELLULOSE A NONIONIC WATER-SOLUBLE POLYMER

Natrosol hydroxyethylcellulose (HEC) is a nonionic watersoluble polymer derived from cellulose. Like Aqualon<sup>®</sup> cellulose gum (sodium carboxymethylcellulose), it is a cellulose ether, but it differs in that it is nonionic and its solutions are unaffected by cations.

Natrosol dissolves readily in cold or hot water. Its solutions have somewhat different flow properties from those obtained with other water-soluble polymers. It is used to produce solutions having a wide range of viscosity. Such solutions are pseudoplastic—that is, they vary in viscosity depending on the amount of shear stress applied.

This booklet describes the properties of Natrosol, Natrosol solutions, and Natrosol films. It suggests the use of Natrosol as a thickener, protective colloid, binder, stabilizer, and suspending agent in a variety of industrial applications, including pharmaceuticals, textiles, paper, adhesives, decorative and protective coatings, emulsion polymerization, ceramics, and many miscellaneous uses. For a guide to these uses, see Table 1.

#### Table I — A Guide to Where and Why Natrosol Hydroxyethylcellulose is Used

Types of Uses	Specific Applications	Properties Utilized
Adhesives	Wallpaper adhesives	Thickening and lubricity
	Latex adhesives	Thickening and water-binding
	Plywood adhesives	Thickening and solids holdout
Binders	Welding rods	Water-binding and extrusion aid
	Ceramic glaze	Water-binding and green strength
	Foundry cores	Water-binding
Coatings	Latex paint	Thickening and protective colloid
	Texture paint	Water-binding
Cosmetics	Hair conditioners	Thickening
	Toothpaste	Thickening
	Liquid soaps and bubble bath	Stabilizing
	Hand creams and lotions	Thickening and stabilizing
Laundry Aids	Fabric finishes	Film-former
	Aerosol starches	Film-former
	Liquid cleaners	Thickening and stabilizing
Miscellaneous	Joint cements	Thickening
	Hydraulic cements	Water-binding and set retarder
	Plaster	Water-binding
	Caulking compound and putty	Thickening
	Printing inks	Thickening and rheology control
	Asphalt emulsions	Thickening and stabilizing
Paper	Coating colors	Water-binding and rheology control
·	Size press solutions	Water-binding and solids holdout
Pharmaceuticals	Lotions and emulsions	Thickening and stabilizing
	Jellies and ointments	Thickening and water-binding
Polymerization	PVAC and acrylic latices	Protective colloid and surface activity
	PVC suspension	Protective colloid and surface activity
Textiles	Latex-back sizes	Thickening
	Glass-fiber size	Film-former
	Printing pastes	Thickening and water-binding

## CHEMISTRY

The Natrosol® hydroxyethylcellulose polymer is a hydroxyethyl ether of cellulose. The structure of the cellulose molecule, showing its chain composed of anhydroglucose units, is depicted in Figure 1. Each anhydroglucose unit contains three hydroxyls capable of reaction. By treating cellulose with sodium hydroxide and reacting with ethylene oxide, hydroxyethyl groups are introduced to yield a hydroxyethyl ether. The reaction product is purified and ground to a fine white powder.

#### SUBSTITUTION

Each anhydroglucose unit in the cellulose molecule has three reactive hydroxyl groups, shown in blue in Figure 1. The number that is substituted in any reaction is known as the "degree of substitution," or DS. Theoretically, all three hydroxyls can be substituted. The product from such a reaction would have a DS of three.

Hydroxyethyl groups can be introduced into the cellulose molecule in two ways. First, ethylene oxide reacts at the hydroxyls in the cellulose chain. Second, ethylene oxide, reacting at previously substituted hydroxyls, can polymerize to form a side chain.

The average number of moles of ethylene oxide that become attached to each anhydroglucose unit in cellulose, in the two ways described, are called moles of substituent combined, or MS.

In reacting ethylene oxide with cellulose to form the hydroxyethyl ether of cellulose, solubility in water is achieved as the degree of substitution is increased. By selecting appropriate reaction conditions and moles of substituent, complete and quick solubility in water is obtained. Natrosol 250, which has optimum solubility in water, has an MS of 2.5. An idealized structure of Natrosol 250 is shown in Figure 2. This example has an MS of 2.5 (5 ethylene oxide groups/2 units) and a DS of 1.5 (3 hydroxyls substituted/2 units). Figure 1 Structure of Cellulose

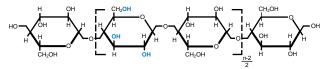
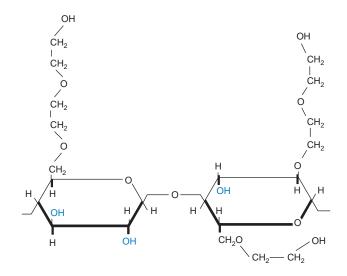


Figure 2 Idealized Structure of Natrosol 250



# GRADES AND VISCOSITY TYPES

#### VISCOSITY

 $Natrosol^{\ensuremath{\circledast}}$  250 HEC is produced in the 10 viscosity types listed in Table II.

#### **R-GRADES**

All grades and types of Natrosol can be treated to provide a powder that displays fast dispersion without lumping when added to water. The grades so treated are designated by the letter R and are discussed further on page 7. This treatment does not alter the solution viscosity. Some grades and viscosity types are available without this treatment if desired.

#### **B-GRADES**

Certain medium- and high-viscosity types of Natrosol 250 are available in a grade that has superior biostability in solution. These grades are designated by the letter B (e.g., Natrosol 250HBR) and are fully described in publication 250-19.

#### PARTICLE SIZE

Natrosol can generally be supplied in any one of the three particle sizes listed in Table III. All types and grades are available in the regular grind, and most are available in the X and W grinds also.

#### SELECTED TYPES

Aqualon has achieved its position as a prime supplier of cellulose derivatives through its ability to provide products to meet specific requirements in the marketplace. The viscosity, hydroxyethyl substitution, particle size, and hydration delay of Natrosol can be varied to meet particular needs. Users are encouraged to discuss their needs with an Aqualon technical sales representative in order to obtain the type and grade best suited to their application.

#### Table II — Typical Viscosity Ranges<sup>(a)</sup> and Typical Molecular Weights for Natrosol HEC

		kfield Viscosity <sup>(b)</sup> at 2 at Varying Concentra			
Viscosity Type	1%	2%	5%	Degree of Polymerization	Molecular Weight <sup>(c)</sup>
250HHR	3,400-5,000	_	_	4,800	$1.3 imes10^{ m 6}$
250H4R	2,600-3,300	_	_	4,000	$1.1 imes10^{\circ}$
250HR	1,500-2,500	_	_	3,700	$1.0 imes10^{ m 6}$
250MHR	800-1,500	—	_	_	—
250MR	_	4,500-6,500	_	2,600	7.2 × 10⁵
250KR	_	1,500-2,500	_	_	—
250GR	_	150-400	_	1,100	$3.0 imes10^{\circ}$
250ER	_	25-105	_	_	—
250JR	_	—	150-400	_	—
250LR	_	_	75-150	300	$9.0 imes10^4$

<sup>(a)</sup>Ranges given are not necessarily the viscosity specification of the type.

<sup>(b)</sup>The method for measuring viscosity of solutions of Natrosol is described in the Appendix.

(c)Estimated (or calculated) from intrinsic viscosity measurements.

## PROPERTIES

Natrosol<sup>®</sup> HEC is a white to light tan, free-flowing powder. It dissolves readily in water to give clear, smooth, viscous solutions that are nonionic. The typical properties of the polymer, its solutions, and its films are given in Table III and in later sections of this booklet.

#### SOLUBILITY IN WATER

Natrosol dissolves quickly in cold or hot water to form clear, smooth, uniform solutions. Solutions of Natrosol do not gel or precipitate, even when heated to the boiling point of water. An idea of the clarity and high viscosity of these solutions can be seen in the photographs in Figure 3, page 7. Procedures for rapid dissolving are also described on page 7.

#### MOISTURE ABSORPTION

Natrosol can absorb moisture from the atmosphere, as do other hygroscopic or finely divided materials. The amount of moisture absorbed depends on the initial moisture content of Natrosol and on the relative humidity of the surrounding air. Opened bags not totally used may experience moisture uptake.

The moisture content of Natrosol, when packed by Aqualon, does not exceed 5% by weight. During prolonged storage, the moisture content of Natrosol tends to reach an equilibrium level that varies with the humidity of the surrounding atmosphere, particularly after the bag is opened.

Equilibrium moisture content of Natrosol 250 at 73°F:

At 50% relative humidity										6%	o o
At 84% relative humidity										29%	ó

To maintain its original moisture content, Natrosol should be stored in tightly closed containers in a dry atmosphere.

#### DERMATOLOGY AND TOXICOLOGY

Animal toxicology and human dermatology studies have been conducted on Natrosol 250 HEC in independent laboratories. Acute oral toxicity tests showed no adverse effects on rats at doses up to 7 g/kg body weight. Subchronic oral tests resulted in no gross signs of toxicity in rats fed up to 5% in the diet. In addition, gross and microscopic examinations of organs and tissues did not reveal any abnormalities that could be attributed to the feeding of Natrosol 250. Moderate eye irritation, which cleared in 24 hours, was observed in albino rabbits after instilling 3 mg of dry Natrosol 250 into the eyes. Instillation of aqueous solutions (0.2 to 2.0%) caused very mild or mild transient irritation.

#### Table III — Typical Properties of Natrosol HEC

#### Polymer

Folymer
Powder, white to light tan
Moisture content (as packed), %, max 5.0
Ash content (calculated as Na <sub>2</sub> SO <sub>4</sub> ), %
Effect of heat
Softening range, °C 135-140
Browning range, °C 205-210
Bulk density, g/ml0.6
Bulking value, lbs/gal (gal/lb) 11.5 (0.087)
Particle size
Regular grind—on U.S. 40 mesh, %, max
X grind—on U.S. 60 mesh, %, max
W grind—on U.S. 80 mesh, %, max
Biological oxygen demand (BOD), <sup>(a)</sup> ppm
Type H (250)
Type L (250)
.,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,
Solutions
Specific gravity, 2% solution, g/ml
Refractive index, 2% solution
pH
Surface tension, dynes/cm (N/m)
Natrosol 250LR at 0.1%
at 0.001% 67.3 (0.0673)
Interfacial tension vs. Fractol, dynes/cm (N/m)
Natrosol 250LR at 0.001%
Bulking value in solution, gal/lb (L/kg)
Films
Refractive index
Specific gravity at 50% relative humidity, g/ml 1.34
Moisture content (equilibrium), %
At 73°F, 50% relative humidity
At 73°F, 84% relative humidity
(a) After F development in a charter of the sector and the sector

<sup>(a)</sup>After 5 days' incubation. Under these test conditions, cornstarch has a BOD of over 800,000 ppm.

Natrosol 250 was found to produce no more skin irritation in the Schwartz prophetic patch test in humans than did commercial wheat starch, which caused mild erythema in some subjects, and showed no evidence of causing sensitization. Human repeat-insult patch tests indicated that Natrosol 250 is nonirritating and nonsensitizing.

As a result of these tests, Natrosol 250 has found use in many pharmaceuticals and cosmetic preparations. It is available in both R-treated and nontreated grades. Aqualon does not recommend the use of R-grade material in pharmaceutical products intended for oral use or in those that will contact mucous membranes. R-grade Natrosol is suitable for use only in topical applications. Natrosol is not recommended for use in preparations for parenteral injections. Additional data are reported in Aqualon Bulletin T-101, Natrosol 250, Summary of Toxicological Investigations.

#### FDA AND EPA STATUS

Natrosol<sup>®</sup> hydroxyethylcellulose is included in the list of materials that are in compliance with requirements of the U.S. Food and Drug Administration for use in adhesives and in resinous and polymeric coatings employed on the food-contact surfaces of metal, paper, or paperboard articles, and other suitable substrates intended for use in food packaging as specified in the U.S. Code of Federal Regulations, Title 21, subject to the limitations and requirements of each regulation under the following Sections. Natrosol R-grades, described on page 7, are in compliance with the requirements specified in 21 CFR, Sections 175.105, 176.170, and 176.180.

#### **Regulated or Functional Uses**

#### FDA Regulation (21 CFR)

175.105	Components of food-packaging adhesives
175.300	Components of resinous and polymeric coatings
176.170	Components of paper and paperboard in
	contact with aqueous and fatty foods
176.180	Components of paper and paperboard in
	contact with dry foods
177.1210	Closures with sealing gaskets for food containers
182.99	Exempt from the requirement of a residue tol-
	erance when used in accordance with 40 CFR
	180.1001(c) as an adjuvant for pesticides in
	dilutions by a grower or applicator prior to
	application to the raw agricultural commodity

Natrosol is not the subject of a direct food additive regulation.

#### EPA Regulation (40 CFR)

180.1001(c) Exempt from the requirement of a residue tolerance when used in accordance with good agricultural practice as an inert ingredient in pesticide formulations applied to growing crops or to raw agricultural commodities after harvest

Natrosol hydroxyethylcellulose is listed on the EPA/TSCA chemical substance inventory.

#### MICROBIOLOGICAL INFORMATION

Natrosol hydroxyethylcellulose (HEC) is routinely sampled and subjected to microbiological testing by an independent laboratory to provide evidence of good manufacturing practice. This testing is not done on a lot-by-lot basis, and microbiological specifications have not been formalized. Typical results obtained using our standard protocol are shown below.

Aerobic plate count, cfu/g<100
Mold, cfu/g<100
Yeast, cfu/g<100
Coliforms, MPN/g <30
<i>E. coli</i> Negative
Staphylococcus aureus Negative
Salmonella Negative
Pseudomonas Negative

Aqualon utilizes officially approved methods to determine the above microbial parameters but recommends that users of HEC assure themselves of compliance with any microbiological specification by testing each lot.

#### PRODUCT SAFETY

As with most organic materials, Natrosol HEC is a flammable dust when finely divided and suspended in air. If the suspended dust is ignited, it can cause an explosion. Proper design and operation of facilities and good housekeeping practices can minimize this hazard.

Surfaces subject to spills or dusting with Natrosol will become extremely slippery, particularly when wetted with water. Follow good housekeeping practices and clean up spills promptly.

Read and understand the Material Safety Data Sheet (MSDS) for Natrosol HEC before using this product.

# DISSOLVING NATROSOL® HEC IN WATER

Natrosol is readily soluble in either hot or cold water. However, as with most water-soluble thickeners, the particles have a tendency to agglomerate, or lump, when first wetted with water. Thus, the time required to achieve complete solution of Natrosol is usually governed by the degree of lumping that is allowed to develop during the solution process. In general, the low-viscosity types are more easily dissolved than are the high-viscosity types. Solutions of the H-type, for instance, should not be prepared at greater than 2% solids concentration.

Aqualon has solved the problem of lumping and slow dissolving by producing a surface-treated grade of Natrosol, known as the R-grade. This grade disperses without lumping when added to water. After an inhibition period, the dispersed particles begin to dissolve and smooth, lump-free solutions are readily achieved.

All viscosity types are available in the R-grade.

Additional information on the dissolution characteristics of the R-grade is given in the section entitled Natrosol R-Grade.

If the R-grade is not used, the following procedures are suggested for the easiest and most efficient preparation of solutions of Natrosol.

#### Method 1

Natrosol should be sifted slowly into the vortex of vigorously agitated water. The rate of addition should be slow enough for the particles to separate in water without lump formation, but not so slow that the solution thickens appreciably before all the solid is added. Agitation should be continued until all of the swollen or gelatinized particles are dissolved to yield a smooth solution.

#### Method 2

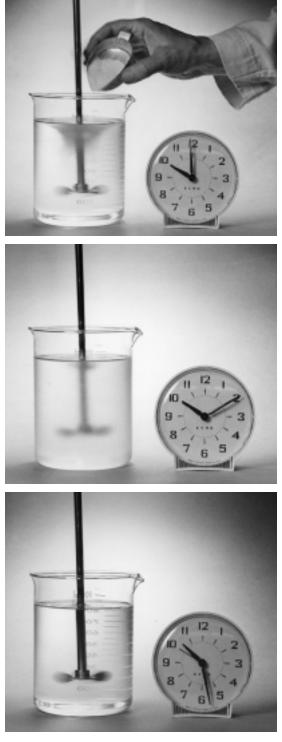
Excellent solution rates can be obtained by prewetting Natrosol with a water-miscible organic liquid before the powder is added to water. Anhydrous ethyl alcohol, higher alcohols, and ketones are suitable for this use, as are other organic liquids such as diethylene glycol monoethyl ether, diethylene glycol monoethyl ether acetate, ethylene glycol monobutyl ether acetate, or hexylene glycol. Ethylene glycol, however, is not recommended, as it swells the particles slightly so that they tend to stick together instead of separating as the prewetting liquid is dissolved away.

#### Method 3

Improved solution rates can be obtained by dry-blending Natrosol with other dry materials (preferably insoluble) that are to be added to the solution. This technique effectively separates particles of Natrosol so that no lumping is experienced when the dry mix is added to water.

#### NATROSOL R-GRADE

The special R-grade is designed to be added to water without lumping, and thus greatly facilitates solution preparation. The R-grade is widely used when higher viscosity types are involved, or when the mixing equipment is inadequate to handle the regular grades.



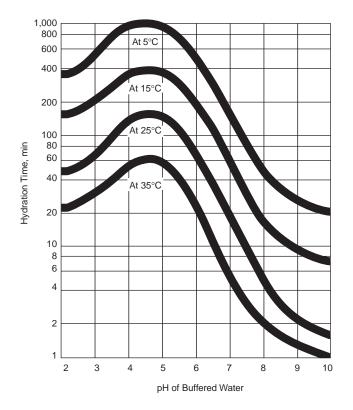
#### Figure 3

Natrosol R-grade disperses readily in water without lumping. Within minutes the R-treatment dissolves and viscosity begins. Some 28 minutes later, the viscous solution is clear. A 1% solution of Natrosol 250 HR was used in water with a pH of 7.0. Hydration of the Natrosol® R HEC particles has been inhibited. When the particles are added to water, they disperse without lumping and, following a predetermined delay, begin to dissolve. This process permits the preparation of clear, smooth, viscous solutions in a short period of time by simply adding the R-grade to water and stirring to prevent settling of the particles, until the polymer is completely dissolved. The use of R-grade simplifies solution preparation using conventional equipment, and avoids the need for more elaborate procedures described in the foregoing section for the non-R-grade product.

The inhibition period, from the initial wetting to the start of dissolution, is referred to as the hydration time of the Natrosol. This hydration time can be varied to meet specific needs. The typical range is 4 to 25 minutes. The hydration time is markedly affected by two factors: the pH and temperature of the water. Figure 4 illustrates the magnitude of these effects and shows that both a higher temperature and a higher pH decrease the hydration time. After the initial hydration time has been satisfied, the polymer begins to dissolve, usually evidenced by an increase in viscosity of the system. Time is then required for complete dissolution of the particles. This dissolution time, also known as the solution time, is also significantly affected by pH and temperature; the longer the hydration time, the longer the solution time. This relationship is illustrated in Figure 5, where the rate of viscosity increase during dissolving at pH 8.0 is greater than the rate of increase at pH 6.0. The magnitude of the effect of a pH change from 8.0 to 6.0 on the hydration time is also illustrated.

Generally, it is recommended that the R-grade be added to room-temperature water at neutral or slightly acidic pH. If higher pH or higher temperature is used, the hydration time may be sufficiently decreased so that lumping occurs as the dry product is added to the aqueous system.

#### Figure 4 Effect of pH and Temperature on the Hydration Time of Natrosol R-Grade



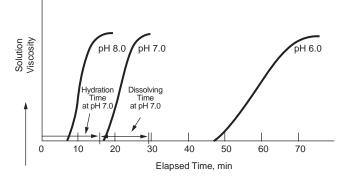
#### SOLUBILITY IN ORGANIC SOLVENTS

Natrosol water-soluble polymer is essentially insoluble in organic solvents. It is, however, either swollen or partly soluble in some solvents. Usually those that are miscible with water, or that contain polar groups, show some effect, Table IV reports solubility behavior with typical organic solvents. Data were obtained by adding 1 g of Natrosol 250 to 100 g of the respective solvent at 25°C (cold) and 55 to 60°C (hot).

#### Table IV — Solubility Behavior in Organic Solvents

	Temperature		
Solvent	Cold 25°C	Hot 55-60°C	
Alcohols Methanol (anhydrous) Ethanol (anhydrous) Ethanol:water (70:30 by wt) (60:40 by wt) (30:70 by wt)	I I PS PS S	I I PS PS S	
Esters Ethyl lactate Methyl salicylate	I I	I I	
Ethers Methyl Cellosolve Cellosolve	I		
<b>Glycols</b> Ethylene glycol Glycerin Propylene glycol	SW SW SW	– PS PS	
Acids Formic (90%) Glacial acetic	S I	_	
Miscellaneous Aniline Dimethyl formamide Dimethyl sulfoxide Ethylene chlorohydrin N-acetylethanolamine Mineral spirits Toluene Hexane	 PS       	       	
Key: I = Insoluble SW = Swollen PS = Partially soluble S = Soluble			

#### Figure 5 Typical Effect of pH on the Hydration Time and Dissolution Time for a Natrosol R at 25°C



# **PROPERTIES OF SOLUTIONS OF NATROSOL® HEC**

Natrosol hydroxyethylcellulose differs from methylcellulose, methylhydroxypropylcellulose, hydroxypropylcellulose, and ethylcellulose in that Natrosol is soluble in both cold and hot water. Thus, Natrosol does not display a "cloud" or "precipitation" point and does not precipitate from aqueous solution at elevated temperature.

Natrosol differs from carboxymethylcellulose in that it is nonionic. For this reason, Natrosol solutions are less affected by pH change and are more tolerant of the presence of anions and organic co-solvents.

#### VISCOSITY

Solutions of Natrosol are non-Newtonian in flow, since they change in viscosity with rate of shear. Because Natrosol is extensively used to modify the viscosity of solutions, dispersions, emulsions, and the like, the effect of a number of factors on solution viscosity will be discussed in detail. The viscosity measurement method is given in detail in the Appendix.

#### **Effect of Concentration**

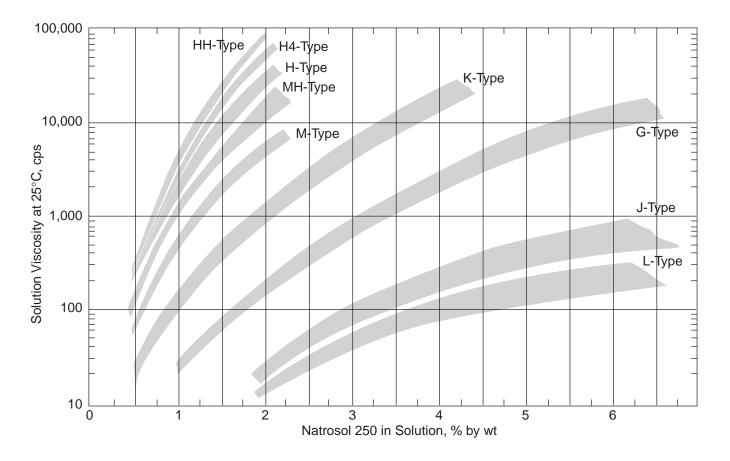
When Natrosol is dissolved in water, the viscosity of the aqueous solution increases rapidly with concentration, as shown in Figure 6. The viscosity-concentration relationship is nearly linear when viscosity is plotted vs. concentration on a semilog basis.

#### Effect of Blending

Two viscosity types of Natrosol can be blended to obtain an intermediate viscosity. Because viscosity is an exponential function of concentration and degree of polymerization, the viscosity resulting from blending is not an arithmetic mean.

A blending chart (VC-440), which is available from Aqualon, can be used to determine the result of blending various amounts of two viscosity types of Natrosol. Or, it can be used to determine the amount of Natrosol required to achieve a desired viscosity when blending two types of known viscosity.

#### Figure 6 Effect of Concentration on Viscosity of Aqueous Solutions of Natrosol



As an example, it is desired to obtain a solution of Natrosol<sup>®</sup> having a viscosity of 150 cps at 5% concentration, and the available materials consist of Natrosol 250 J (200 cps at 5% concentration) and Natrosol 250 L (100 cps at 5% concentration). A line is drawn connecting these two viscosities on left and right, respectively. (This is shown by the solid line on the chart.) The point at which this line intersects the desired viscosity line (dotted line), 150 cps, is then determined, and the percentage it represents is read from the bottom of the chart. In this example, 42% of Natrosol 250 L and 58% of Natrosol 250 J types will give a viscosity of 150 cps.

#### Effect of Shear Rate

A solution of Natrosol in water will appear to have a wide range of viscosities when different conditions of physical force are imposed on the solution.

These conditions of physical force may be referred to as high, intermediate, or low stress. For example, rolling or spreading a liquid as if it were an ointment or lotion would be high stress. On the other hand, after the liquid has been applied, gravity and surface tension control flow. These forces are conditions of low stress. Intermediate stress can be typified by pouring a liquid out of a bottle. Thus, if a solution of high-viscosity Natrosol HEC appears to be a viscous syrup as it is poured from a bottle, it will behave as a runny liquid when applied as a lotion, and yet when high stress is removed, it will instantly revert to its original high viscosity. This type of flow behavior is referred to as pseudoplastic, or non-Newtonian. This behavior differs from the time-dependent viscosity change that is usually called thixotropy. A different molecular weight or viscosity grade of Natrosol will behave in a similar fashion, but to a different degree. The lower the molecular weight, the less change in viscosity will occur as stress conditions are varied. These differences in viscosity-stress properties are shown in Figure 8.

#### **Effect of Mixed Solvents**

Natrosol HEC will also impart varied thickening properties to solvent systems other than plain water. Behavior of Natrosol in these mixed solvent systems is similar to its behavior in water. However, the viscosity of the solvent must be taken into account. For example, if sucrose-water is the solvent, and if this solvent is 50 times thicker than water, the resulting solution of Natrosol in the mixed solvent will be 50 times thicker than it will be in water. This behavior, shown in Figure 9, is predictable for many solvent-water mixtures.

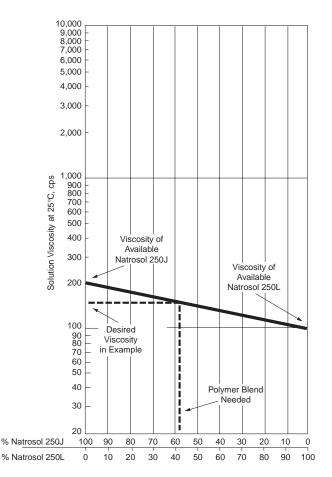
#### **Effect of Temperature**

The viscosity of solutions of Natrosol changes with temperature, increasing when cooled, decreasing when warmed. Figure 10 presents a convenient nomograph from which, knowing the viscosity of a solution of Natrosol at one temperature, one can conveniently estimate its viscosity at a different temperature.

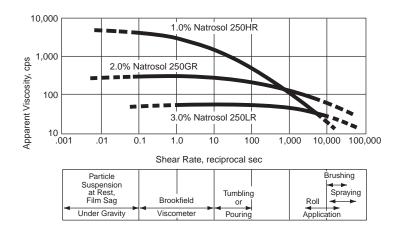
Example using nomograph (Figure 10):

The viscosity of the solution is 100 cps at 25°C. The unknown is the viscosity when the temperature is raised 20°C. By placing a straightedge at 20 in the right column and at 100 in the center column, one can read the answer in the left column, 52 cps.

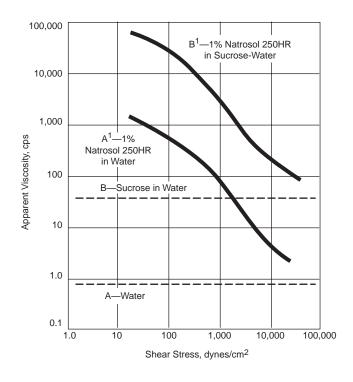
#### Figure 7 Blending Chart for Natrosol



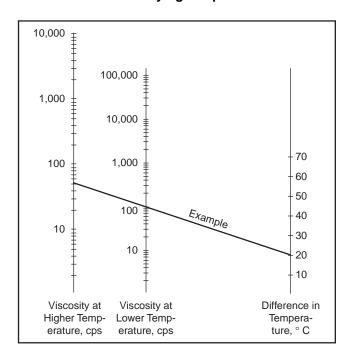
#### Figure 8 Effect of Shear Rate on Viscosity of Solutions of Natrosol



#### Figure 9 Effect of Mixed Solvents on Viscosity of Solutions of Natrosol®



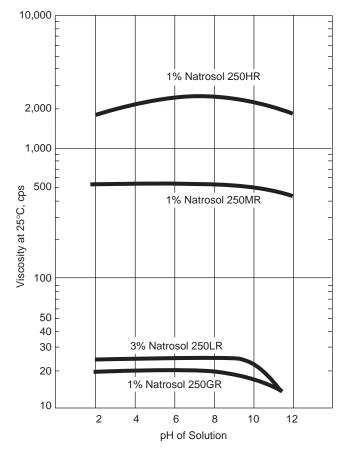
#### Figure 10 Nomograph for Estimating the Viscosity of Solutions of Natrosol HEC at Varying Temperatures



#### Effect of pH

Solutions of Natrosol undergo little viscosity change over the pH range of 2 to 12. However, solutions possess the greatest viscosity stability in the pH range of 6.5 to 8.0. Below pH 3, solutions may show some drop in viscosity resulting from acid hydrolysis. This is common to all water-soluble polysac-charide polymers, and is accelerated by high temperature and high acidity. Under highly alkaline conditions, some oxidative degradation may occur, accelerated by heat and light, that will lower the viscosity. See Figure 11.

Figure 11 Effect of pH on Viscosity of Solutions of Natrosol



#### VISCOSITY STABILITY

Natrosol HEC does not degrade when properly stored in closed containers. Nine lots of Natrosol, viscosity grades M, H, H4, and HH, were monitored over a period of a year for viscosity stability. Average viscosity retention was 93%.

If Natrosol is kept in inventory for extended periods, Aqualon recommends checking the viscosity at least once per year.

#### PRESERVATIVES

Natrosol HEC, particularly the B grades, is more resistant to biological attack than are some other water-soluble gums and polymeric materials. This better resistance is an important factor in its use in latex paints.

Since solutions of Natrosol are not immune to biological attack, it is recommended that a preservative be added when the solutions are to be stored. As a convenience, some preservatives that have been found effective in laboratory tests are listed in Table V, along with the names of their manufacturers, in the case of proprietary products.

It is suggested that advice be obtained from the producers of the preservatives as to the kind and amount of preservative to be used in specific compositions for various end products.

For use in cosmetics and pharmaceuticals, particular care should be exercised, both in the selection of an acceptable preservative and in the maximum quantity used. The same is true where Natrosol<sup>®</sup> is used in adhesives or coatings employed on the food-contact surfaces of metal or paper articles intended for use in the packaging of food.

#### Table V — Preservatives Effective for Solutions of Natrosol

Preservatives	Manufacturers of Trademarked Products
Dowicil 75	The Dow Chemical Co.
Merbac 35	Merck/Calgon Corp.
Vancide TH	R. T. Vanderbilt Co., Inc.
Proxel CRL	ICI Americas Inc.
Sodium benzoate	—
Sorbic acid	_
Methyl-propylparaben	
combinations	_

As an alternative to adding preservatives, some consideration might be given to heat sterilization. Many microorganisms are destroyed by heating solutions to 80°C for 30 minutes, or to 100°C for 1 minute, and such heat cycles are not severe enough to materially affect the properties of the solutions of Natrosol. This suggestion should be followed only when the solution is to be stored under sterile conditions, otherwise the solution will become recontaminated with microorganisms. Enzymes, however, can persevere even when microorganisms are killed, depending on the time and temperature of such heat cycles.

#### USE OF DEFOAMERS

Solutions of Natrosol HEC are mildly surface-active. They are lower in surface tension than water, as shown in Table III. If any difficulty is encountered with air entrainment in solutions, a defoamer should be utilized. Water-dispersible defoamers such as Advantage<sup>®</sup> 357 defoamer, Foamaster NDW, Colloid 581-B, and Dow Antifoam AF are effective, and can be added to the water prior to solution preparation.

#### TOLERANCE FOR INORGANIC SALTS

Because of its solubility and nonionic character, Natrosol can be dissolved in many salt solutions that will not dissolve other water-soluble polymers. Data in Table VI show that Natrosol is soluble in most 10% salt solutions and many of the 50% (saturated) salt solutions.

This study indicated that solubility was not affected by the viscosity type of Natrosol used. In obtaining these data, 1 ml of a solution of Natrosol was added to 15 g of each of the salt solutions. The ratio of Natrosol to salt solids ranged from 1:150 to 1:750.

#### Table VI — Compatibility of Solutions of Natrosol With Inorganic Salt Solutions

	Natrosol®	250 <sup>(a)</sup> HEC
Salt	10% Salt Solution	50% Salt (Saturated)
Aluminum nitrate	С	С
Aluminum sulfate	С	Р
Ammonium chloride	С	С
Ammonium nitrate	С	С
Ammonium sulfate	С	Р
Barium nitrate	С	С
Borax	С	С
Calcium chloride	С	С
Calcium nitrate	С	С
Calcium sulfate	С	С
Chromic nitrate	С	С
Chromic sulfate	С	С
Diammonium phosphate	С	Р
Disodium phosphate	Р	Р
Ferric chloride	С	Р
Ferric sulfate	С	С
Magnesium chloride	С	С
Magnesium sulfate	С	Р
Potassium ferricyanide	С	С
Potassium ferrocyanide	С	Р
Potassium metasulfite	С	С
Silver nitrate	С	С
Sodium acetate	С	С
Sodium carbonate	Р	Р
Sodium chloride	С	С
Sodium dichromate	С	С
Sodium metaborate	С	Р
Sodium nitrate	С	Р
Sodium perborate	С	С
Sodium sulfate	Р	Р
Sodium sulfite	С	Р
Stannous chloride	С	С
Trisodium phosphate	С	Р
Zinc chloride	С	С
Zinc sulfate	С	Р

(a) The H (high) viscosity types were used in this compatibility study.

Key: C = Compatible

P = Precipitate

# COMPATIBILITY WITH OTHER MATERIALS

Natrosol<sup>®</sup> HEC, being a nonionic polymer, is compatible with a broad range of water-soluble materials, including other water-soluble polymers and natural gums.

Table VII lists the viscosity and appearance of solutions of Natrosol and some of the materials with which it can be formulated. The data were obtained by adding a high-viscosity Natrosol 250 to the additive, or solution of the additive, with moderate stirring to completely dissolve the Natrosol.

Summary comments on the compatibility of Natrosol with the product groups shown in Table VII are given on this page. The chemical identities and manufacturers of trademarked products in this table are given in the Appendix.

#### LATEX EMULSIONS

Natrosol is compatible with a broad range of latex emulsions, hence its general use as a thickener and protective colloid for these systems.

#### CELLULOSIC WATER-SOLUBLE POLYMERS

Natrosol is generally compatible with other cellulosic watersoluble polymers to give clear, homogeneous solutions. However, when it is mixed with an anionic polymer such as carboxymethylcellulose, an unusually high viscosity can be obtained owing to a tendency for interaction between anionic and nonionic polymers. This is illustrated in Table VII by the viscosity obtained with Aqualon<sup>®</sup> sodium carboxymethylcellulose (CMC) type 7H.

#### WATER-SOLUBLE RESINS

The nonionic Natrosol has a wide tolerance for water-soluble resins. As shown in the table, solutions with Elvanol may

show synergistic viscosity increase, as evidenced with anionic polymers such as CMC.

#### NATURAL GUMS

Natrosol HEC has excellent compatibility with natural gums.

#### PRESERVATIVE OR BIOCIDES

A variety of preservatives can be used with Natrosol. No unusual effects were observed with use of those shown in Table VII.

#### SURFACTANTS AND DETERGENTS

Natrosol has excellent tolerance for anionic, nonionic, amphoteric, and cationic surfactants. No unusual effects were observed. Representative materials from each of these groups are shown in the table.

#### DEFOAMERS

Silicone-in-oil dispersions, silicone emulsions, and organic defoamers can all be used with Natrosol with no unusual effects.

#### PLASTICIZERS

Water-soluble plasticizers generally do not adversely affect the quality of solutions of Natrosol.

#### ORGANIC SOLVENTS

Although Natrosol is insoluble in most common organic solvents (see page 17), it will tolerate relatively high concentrations of many water-miscible polar solvents. Examples of a number of compatible systems combining Natrosol, water, and organic solvents are given in Table VII.

	Concentration, %		Viscosity, cps	Appearance
Material	Additive	Natrosol	After 48 Hours	After 48 Hours
Latex Emulsions Polyco 2161	99.5	0.5	8,300	Opaque
Rhoplex AC-22	99.5	0.5	7,720	Opaque
Rhoplex AC-25	99.5	0.5	8,250	Opaque
Ucar 130	99.5	0.5	83,000	Opaque
Cellulosic Water-Soluble Polymers				
Aqualon <sup>®</sup> CMC-7H	1.0	1.0	36,000	Clear
	0.5	1.0	22,500	Clear
Benecel <sup>®</sup> MP 843 HPMC	0.5	1.0	5,200	Clear
Klucel <sup>®</sup> HF HPC	1.0	1.0	22,000	Hazy
	0.5	1.0	13,000	Hazy
Water-Soluble Resins				
Elvanol 72-60	1.0	1.0	9,500	White
	5.0	1.0	22,750	White
Kymene 709	50.0	1.0	5,750	Clear
	5.0	1.0	2,800	Clear
Natural Gums				
Galactasol <sup>®</sup> 20H5F guar gum	0.5	1.0	7,900	Opaque
Kelzan xanthan gum	0.5	1.0	6,700	Opaque
Preservatives or Biocides				
Dowicil 75	0.1	1.0	1,880	Clear
Formaldehyde	0.1	1.0	1,800	Clear
	1.0	1.0	2,550	Clear
Merbac 35	0.1	1.0	1,780	Opaque
Methyl Parasept PMA-18	0.2 0.1	1.0	2,100	Clear
PIMA-18	0.1	1.0 1.0	1,720 1,560	Hazy Opaque
Sodium benzoate	0.5	1.0	2,500	Clear
	0.0	1.0	2,000	olcal
Surfactants and Detergents Anionic				
Aerosol OT	1.0	1.0	1,840	Hazy
Ammonium lauryl sulfate	1.0	1.0	920	Clear
Bio Terge AS-40	1.0	1.0	1,560	Clear
Igepon CN-42	1.0 1.0	1.0	2,100	Hazy Clear
Sodium lauryl sulfate	1.0	1.0	580	Clear

# Table VII — Effect of Formulation Additives on Properties of Solutions of Natrosol® 250 HEC and Water

	Concentration, %		Viscosity, cps	Appearance	
Material	Additive	Natrosol	After 48 Hours	After 48 Hours	
Nonionic					
Igepal CO-990	1.0	1.0	2,300	Clear	
Tergitol NP-10	1.0	1.0	2,600	Clear	
Triton X-100	1.0	1.0	1,520	Clear	
Tween 60	1.0	1.0	1,850	SI. Hazy	
Tween 80	1.0	1.0	2,500	Clear	
Amphoteric					
N-Methyltaurine	1.0	1.0	1,650	Clear	
Miranol C2MSF Concentrate	1.0	1.0	1,560	Clear	
Lexaine C	1.0	1.0	1,360	Clear	
Cationic (quaternary cpds)					
Arguad C-50	1.0	1.0	1,580	Clear	
Benzyl trimethylammonium			,		
bromide	1.0	1.0	1,520	Clear	
Cetyl trimethylammonium					
bromide	1.0	1.0	1,640	Clear	
Hyamine 1622	0.5	1.0	2,600	Clear	
-	10.0	0.5	4,500	Clear	
Defoamers					
Hercules 1052 defoamer	1.0	1.0	1,570	Opaque	
Dow Antifoam A	0.05	1.0	3,000	Hazy	
Plasticizers					
Glycerin	1.0	1.0	2,980	_	
Triethanolamine	1.0	1.0	2,950	Clear	
Triethylene glycol	1.0	1.0	2,950	Clear	
Organic Solvents					
Ăcetone	10.0	1.0	2,100	Clear	
	20.0	1.0	3,950	Clear	
Ethanol	10.0	1.0	3,550	Clear	
	20.0	1.0	3,700	Clear	
Ethyl acetate	10.0	1.0	3,900	Clear	
,	20.0	1.0	4,350	Clear	
Methanol	10.0	1.0	3,000	2-Phase	
			•	Separation	
	20.0	1.0	2,950	Hazy	

# Table VII — Effect of Formulation Additives on Properties of Solutions of Natrosol® 250 HEC and Water (Contd.)

# PROPERTIES OF FILMS OF NATROSOL® HEC

Clear films of moderate strength can be cast from solutions of Natrosol. Such films are odorless, tasteless, and insensitive to light, and have excellent flexibility. They are resistant to oils and greases, and are insoluble in most organic solvents. Film properties can be tailored through use of suitable plasticizers. Films of Natrosol cast from aqueous solutions will redissolve in water. Such films can be made waterinsoluble by reacting with a resin and a catalyst.

#### UNPLASTICIZED FILMS

Typical properties of films of unplasticized Natrosol 250 are shown by the examples given in Table VIII.

#### PLASTICIZERS

In applications where greater flexibility and elongation are desired, various plasticizers can be used with Natrosol 250.

With the addition of 10 to 30% plasticizer (solid basis), elongation and folding endurance can be substantially increased. Representative types of plasticizer that have been found effective are:

Glycerin Ethanolamines Lower glycols Sorbitol Sulfonated castor oil

The effect of plasticizer on impact resistance is shown in Table IX.

#### HEAT-SEAL CHARACTERISTICS

Unplasticized films of Natrosol are not truly thermoplastic. When plasticized, low-viscosity types form films that flow when heated, as shown in Table X.

#### Table VIII — Properties of Unplasticized Films<sup>(a)</sup> of Natrosol 250 HEC

Film	Tensile Strength, psi	Maximum Elongation, %	MIT Folds	Ability to Heat-Seal	Tendency to Block at 90% Relative Humidity
Natrosol 250LR	4,000	30	>10,000	Yes	Yes
Natrosol 250GR	3,700	41	>10,000	Weak	Moderate
Natrosol 250MR	3,900	35	>10,000	Weak	Moderate
Natrosol 250HR	3,900	14	—	No	No
(a)2-mil film cast from solut	tion. Properties measured at	50% RH, 25°C.			

#### Table IX—Effect of Plasticizer on Tensile Impact Resistance of Films of Natrosol 250

Film	At 10% RH, <sup>(a)</sup> 10°C, ft-lbs/in. <sup>2</sup>	At 50% RH, <sup>(a)</sup> –15°C, ft-lbs/in. <sup>2</sup>	At 50% RH, <sup>(a)</sup> 5°C, ft-lbs/in. <sup>2</sup>
Natrosol 250LR	6	0	13
Natrosol 250LR + 20% plasticizer	23	7	46
(a)Relative humidity.			

#### Table X — Effect of Plasticizer on Flow and Heat-Seal Characteristics of Natrosol 250 HEC Films

Composition	Amount of Plasticizer <sup>(a)</sup>	Tinius Olson Flow, in. in 2 min	Pressure, psi	Temperature, °C	Condition of Weld
Natrosol 250LR	None	1.47	500	150	Fair
Natrosol 250LR	20 Propylene glycol	0.62	500	110	Good
Natrosol 250LR	20 Propylene glycol	1.30	500	120	Good
Natrosol 250LR	20 Propylene glycol	2.80	500	150	Good
	ed in a <i>sigma</i> -bladed mixer.	2.00			

#### SOLUBILITY IN WATER

Films of Natrosol<sup>®</sup> HEC will dissolve in water. For example, a 2-ml film of Natrosol 250LR placed in water at 122°F will redissolve in 3 minutes.

#### INSOLUBILITY IN ORGANIC SOLVENTS

Films of Natrosol 250 HEC are insoluble in hydrocarbons. They are slightly soluble in some hydroxyl- and nitrogencontaining compounds; up to 1% in ethylene glycol, propylene glycol, glycerin, sorbitol, and the ethanolamines; and up to 5% in N-acetylethanolamine.

#### **RESISTANCE TO GREASE AND OIL**

Films of Natrosol 250 are insoluble in, and form highly resistant barriers to, animal and vegetable oils, greases, and petroleum hydrocarbons, and prevent their penetration.

#### PHOTOSENSITIVITY

Films of Natrosol 250 can be rendered photosensitive by chrome and by azo dyes, and can then be rendered insoluble in water by exposure to ultraviolet light.

#### ADHESION

For improved adhesion to glass, metal, fiber, and other surfaces, one can add small amounts of protein glues, lowmolecular-weight polysaccharides, or water-dispersible synthetic resins to the solution of Natrosol 250 prior to coating.

#### INSOLUBILIZING FILMS OF NATROSOL

Films of Natrosol 250 can be made insoluble by crosslinking the hydroxyl groups with a polyfunctional compound. By controlling the amount of reactant added, almost any degree of water insolubility can be obtained. A number of substances are capable of performing this crosslinking reaction. A list of some reactants and their insolubilizing effect is given in Table XI. In this work, films containing resins were cast from a 5% solution of Natrosol. When a 100-mil-wetthickness casting knife was used, dry-film thickness averaged 4 mils. Films were cured by heating for 5 minutes at 150°C. For short curing times at high temperature, a highintensity infrared oven is needed to ensure rapid and uniform heating to the required high temperature. Ordinary convection and air-draft ovens are satisfactory only for long curing times and (preferably) lower temperatures (e.g., 1 to 3 hours at 100 to 140°C). In general, any heating apparatus is suitable if the heat-up time to the required temperature is only a small percentage of the total curing time.

#### Table XI—Effect of Resins<sup>(a)</sup> and Catalysts on Insolubilizing Films of Natrosol 250G

Film Resin, No.	Concentration <sup>(b)</sup>	Catalyst, Concentration <sup>(c)</sup>	рH	Water- Insoluble, % <sup>(d)</sup>	Moisture Absorbency, % <sup>(e)</sup>	Tensile, Dry <sup>(f)</sup>	Lbs/in., <sup>2</sup> Wet <sup>(g)</sup>
1	None	None	6.7	0	81 <u>+</u> 2	3,900	0
2	DMU, 10%	NH₄CI, 10%	7.2	98	61 <u>+</u> 1	1,700	200
3	Aerotex M-3, 10%	NH₄CI, 10%	7.1	97	58	2,600	150
4	DMU, 10%						
	Casein, 5%	NH₄CI, 10%	6.8	96	74	2,770	200
5	DMU, 10%						
	Casein, 10%	NH₄CI, 10%	7.4	97	76 <u>+</u> 2	2,840	600
6	Kymene 917, 10%						
	Casein, 5%	NH₄CI, 10%	6.4	97	77 <u>+</u> 2	1,530	200
7	Kymene 917, 10%						
	Casein, 10%	NH₄CI, 10%	6.6	96	78 <u>+</u> 2	3,400	400
8	Glyoxal, 30%	Formic acid in					
		glyoxal, 3%	2.9	81	81 <u>+</u> 2	1,300	0
9	Glyoxal, 30%	None	4.7	89	_	2,000	0

(a)See table on page 20 for the manufacturers of products listed by trademark.

(b)Resin concentration based on Natrosol 250GR.

(c)Catalyst concentration based on resin.

<sup>(d)</sup>Water-insoluble test: Cured films dried 2 hrs at 80°C. Tumbled 20 hrs in water buffered to pH 7.0. Loss in film weight noted.

(e)Water-absorbency test: Films conditioned 12 days at 73°F and 100% relative humidity. Moisture pickup noted.

(f) Tensile strength, dry: Films conditioned 2 days at 73°F and 50% relative humidity. Tensile determined on Amthor tensile tester.

(9) Tensile strength, wet: Films soaked 5 min and tensile determined on Amthor tester with the Finch attachment.

# APPENDIX — METHODS OF ANALYSIS<sup>(1)</sup> FOR NATROSOL<sup>®</sup> HEC

#### VISCOSITY DETERMINATION

There are frequent occasions when, in addition to chemical analysis, an accurate determination of the viscosity of a solution of Natrosol is needed. As explained on page 9, the apparent viscosity of a solution of Natrosol depends on a number of factors, and if reproducible results are to be obtained, a closely standardized method of solution preparation and viscosity determination must be followed.

The standardized Aqualon method for determining viscosity of Natrosol solutions specifies the Brookfield viscometer.<sup>(2)</sup> The spindle guard supplied with this instrument should be used for all determinations.

Solution volumes specified should not be less or they may not cover the appropriate Brookfield spindle.

The preparation of the solution is critical, in that the Natrosol must be completely dissolved in order to obtain a significant measurement. To determine the proper amount of polymer, a moisture correction must be made. This correction compensates for the moisture in Natrosol and bases the viscosity measurement on dry Natrosol. The viscosity measurement test must be rigidly standardized because the viscosity reading obtained is dependent on rate of shear, temperature, amount of agitation prior to measurement, and elapsed time between agitation and measurement. The method used in Aqualon laboratories is therefore given here in detail.

#### **Moisture Determination**

- 1. Weigh duplicate samples of 5 g to the nearest 0.001 g into previously dried and weighed moisture cans with covers.
- 2. Place the samples in a gravity convection oven maintained at 105  $\pm$  0.5°C and heat for 3 hours. Cool in a desiccator and weigh.
- 3. Return the samples to the oven for 45 minutes; cool and weigh as before. If the second dried weight is not within 0.005 g of the first, repeat the 45-minute oven periods until two subsequent weighings are in agreement. Then, using the lowest dried weight obtained, calculate % moisture as follows:

 $\frac{\text{Original sample wt} - \text{dry sample wt}}{\text{Original sample wt}} \times 100 = \% \text{ moisture}$ 

#### **Solution Preparation**

Immediately after portions of the sample of Natrosol are taken for moisture determination, portions of the same undried Natrosol should be taken for the viscosity solution preparation. The moisture sample and solution sample weighings should be carried out practically together to ensure that the moisture content of the respective portions is the same at the time of weighings.

#### Table XII – Approximate Weights for Solutions

Natrosol, Type		Sample Weight, g	Solution Concentration, %	
	L, J	13.0	5	
	E, G, K, M	5.2	2	
	MH, H, H4, HH	2.3	1	

- 1. Weigh the required amount of Natrosol (see Table XII), to the nearest 0.005 g, into a clean weighing bottle. Minimize exposure of the sample to the air during weighing and transfer to avoid moisture pickup.
- 2. For medium- and high-viscosity types, carefully (to avoid any sample loss from dusting) transfer the bulk of the sample to a 12-oz, screw-cap, widemouthed bottle. Add 10.0 ml of 80% *tert*-butanol and allow to stand for 5 to 10 minutes. Make sure the entire sample is thoroughly wetted. Low-viscosity-type samples readily dissolve without the addition of *tert*-butanol. Medium- and high-viscosity-type samples are more readily dissolved with the inclusion of *tert*-butanol. This is particularly important for samples that are not R-treated, because lumping can occur.

Calculate the amount of water required to obtain the desired solution concentration, using Equations a and b for 1% and 2% solutions, respectively. The weight of water required is reduced by the weight of *tert*-butanol added (8.31 g). From the determined % moisture, calculate the water to be added for the respective viscosity solutions as follows:

- a. For 1% viscosity solution: Wt of undried Natrosol × (99 – % moisture) – 8.3 = Wt of water
- b. For 2% viscosity solution: (Wt of undried Natrosol  $\times \frac{(98 - \% \text{ moisture})}{2}) - 8.3 = \frac{\text{Wt of water}}{\text{water}}$
- 3. **For low-viscosity types**, calculate the amount of water required to obtain a 5% solution using Equation c.

c. (Wt of undried Natrosol 
$$\times \frac{(95 - \% \text{ moisture})}{5}$$
) = Wt of water

<sup>&</sup>lt;sup>(1)</sup>Several analytical procedures are contained in ASTM D2364, "Standard Test Methods for Hydroxyethylcellulose." Copies are available directly from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

<sup>&</sup>lt;sup>(2)</sup>Brookfield LVT, Brookfield Engineering Laboratories, Middleboro, Massachusetts.

Place the calculated amount of water into a 12-oz, screw-cap, widemouthed bottle. While stirring in a constanttemperature bath with mechanical agitation, using an anchor-type stirrer, sprinkle the sample into the side of the vortex. When the sample is completely dissolved, stir at high speed for an additional 10 to 15 minutes.

#### **Viscosity Measurement**

Select from Table XIII the Brookfield spindle-speed combination corresponding to the viscosity type of Natrosol® HEC being tested.

Attach the selected spindle to the instrument, which is then set for the corresponding speed.

If the solution has been stirred in a constant-temperature bath, determine the viscosity of the Natrosol immediately after removing it from the stirrer. If it has not been stirred, in a constant-temperature bath, cap it tightly and place it in a 25°C constant-temperature bath for 30 minutes. Remove it from the bath and shake it vigorously for 10 seconds.

Being careful not to trap air bubbles, insert the Brookfield viscometer spindle into the solution. After allowing the spindle to rotate for 3 minutes, stop the instrument and read the dial. To determine the solution viscosity in centipoises, multiply the dial reading by the factor in Table XIII that corresponds to the speed and spindle used.

#### ASH CONTENT

The sample is charred over a low Bunsen flame to remove the bulk of the volatile matter, cooled, and moistened with sulfuric acid. The excess acid is evaporated, and the ashing is completed in the normal way. The sulfate ash is calculated to percentage.

#### Procedure

Weigh a 2- to 3-g sample to the nearest 0.001 g into a previously ignited and tared platinum or porcelain dish of about 75-ml capacity.

#### Table XIII – Brookfield Spindle and Speeds for Viscosity Determinations

Type of Natrosol	Spindle No.	Spindle Speed, rpm	Factor	Maximum Reading, cps
E, L	1	30	2	200
J or G	2	60	5	500
K	3	30	40	4,000
М	4	60	100	10,000
MH	3	30	40	4,000
H or H4	3	30	40	4,000
HH	4	30	200	20,000
High concen-	4	12	500	50,000
tration of the				
above types	4	6	1,000	100,000

Ignite it over a low Bunsen flame and allow it to burn, with only occasional application of heat, until most of the carbonaceous material has burned off.

Cool and moisten the entire residue with 1:1 sulfuric acid. Evaporate the excess acid over a low Bunsen flame slowly, so as to avoid spattering, and complete the ignition by placing the sample in a muffle at 800 to 850°C for 30 minutes or until the ash is free of carbon. Cool, desiccate, and weigh.

#### Calculation

 $\frac{\text{g of residue} \times 100}{\text{g of sample}} = \text{Ash as Na}_2\text{SO}_4$ 

Duplicate results should agree within 0.05%.

# QUALITY CONTROL, PACKAGING, AND SHIPPING

To ensure quality control, plant production is both completely automated and visually inspected. In addition, tests are conducted throughout the production cycle to keep the important properties of each lot within specification limits.

Natrosol<sup>®</sup> HEC is packaged in foil-lined, multiwall 50-lb bags, stretch-wrapped, capped, and palletized for handling convenience. It is also available in 100-lb Leverpak drums. Shipments are made from our plants in Hopewell, Virginia,

and Parlin, New Jersey, and from warehouses located throughout the country.

#### ISO INFORMATION

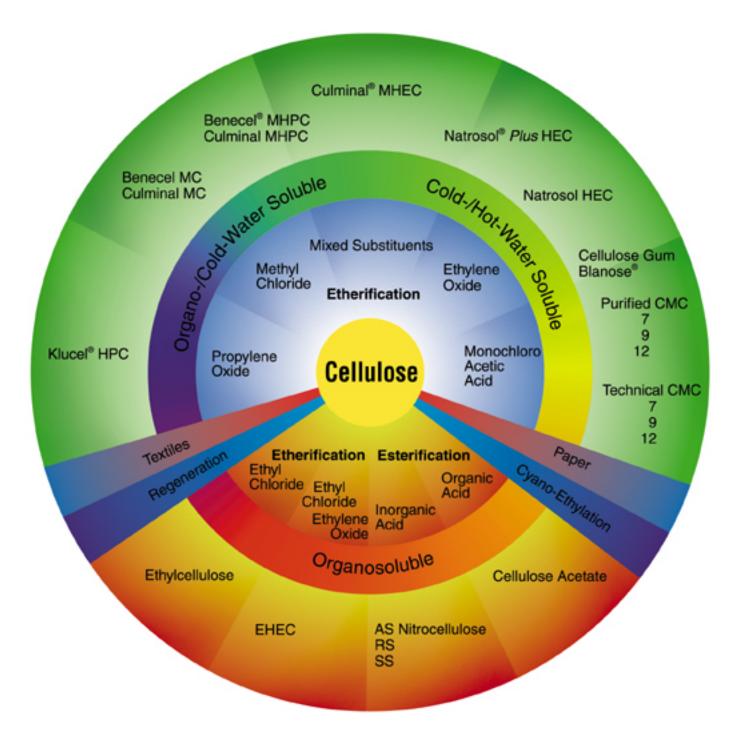
The quality systems of the Aqualon Division of Hercules Incorporated plants located at Hopewell, VA and Parlin, NJ have been registered by UL to the ISO 9002 standards.

# SOURCE AND DESCRIPTION OF TRADEMARKED PRODUCTS

This listing gives the chemical identities and manufacturers of the specific materials referred to by trademark in text and in Tables IV, V, VII, and XI. It is not our intention to imply that these products are the only ones that can or should be used with Natrosol.

Product	Chemical Identity or Function	Manufacturer		
Aerosol OT	Dioctyl sodium solfosuccinate	Cytec Industries, Inc.		
Aerotex M-3	Melamine-formaldehyde resin	Cytec Industries, Inc.		
Aqualon <sup>®</sup> CMC-7H	Sodium carboxymethylcellulose	Aqualon		
Arquad C-50	n-Alkyltrimethylammonium chloride	AKZO Chemie America		
Benecel <sup>®</sup> MP 843	Hydroxypropylmethylcellulose	Aqualon		
Bio Terge AS-40	al pha-olefin sulfonate	Stepan Chemical Co.		
Cellosolve	Coalescing aid	Union Carbide Corp.		
Colloid 581-B	Hydrocarbon-based material containing surfactants, waxes, and metallic soaps	Rhodia		
DMU	Dimethylolurea	Dexter Chemical Corp.		
Dow Antifoam A, AF	Silicone-in-oil dispersion	Dow Corning Corp.		
Dowicil 75	1-(3-chloroallyl)-3,5,7-triaza-1-azoniaadamantane chloride	The Dow Chemical Co.		
Elvanol 72-60	Polyvinyl alcohol	E. I. du Pont de Nemours & Co.		
Foamaster NDW	Defoamer	Henkel		
Fractol	Mineral oil	Exxon Company, U.S.A.		
Galactasol <sup>®</sup> 20H5F	Guar gum	Aqualon		
Hercules <sup>®</sup> 357 Defoamer	Silica-in-oil dispersion	Hercules Incorporated		
Hyamine 1622	Diisobutyl phenoxyethoxy ethyl dimethylbenzyl NH₄Cl	Rohm and Haas Co.		
Igepal CO-990	Nonyl phenoxy polyethoxy ethanol	Rhodia		
Igepon CN-42	Sodium N-cyclohexyl-N-palmitoyl taurate	Rhodia		
Kelzan	Xanthan gum	Kelco Corp.		
Klucel <sup>®</sup> HF	Hydroxypropylcellulose	Aqualon		
Kymene <sup>®</sup> 709	Polyamide-epichlorohydrin resin	Hercules Incorporated		
Kymene 917	Urea-formaldehyde resin	Hercules Incorporated		
Lexaine C	Cocoamidopropyl betaine	Inolex Co.		
Merbac 35	Benzyl bromoacetate	Merck/Calgon Corp.		
Methyl Cellosolve	Ethylene glycol monomethyl ether	Union Carbide Corp.		
Methyl Parasept	Methyl para-hydroxybenzoate	Creanova		
N-Methyltaurine	Surfactant	Rhodia		
Miranol C2MSF Concentrate	Surfactant	The Miranol Chemical Co., Inc.		
Polyco 2161	Vinyl acetate-acrylic latex	Rohm and Haas Co.		
Proxel CRL	1,2-benzisothiazolin-3-one	ICI Americas Inc.		
Rhoplex AC-22, -25	Acrylic latex	Rohm and Haas Co.		
Tergitol NP-10	Nonylphenol ethoxylate	Union Carbide Corp.		
Triton X-100	Octyl phenol polyethoxylate	Union Carbide Corp.		
Tween 60	Polyoxyethylene (20) sorbitan monostearate	ICI Americas Inc.		
Tween 80	Polyoxyethylene sorbitan monooleate	ICI Americas Inc.		
Ucar 130	Vinyl acetate latex	Union Carbide Corp.		
Vancide TH	Hexahydro-2,3,5-triethyl-s-triazine	R. T. Vanderbilt Co., Inc.		

## Cellulose and Its Derivatives



#### **HERCULES INCORPORATED**

Aqualon Division Hercules Plaza 1313 North Market Street Wilmington, DE 19894-0001 (302) 594-5000 www.herc.com

ORDER PLACEMENT INFORMATION (800) 334-8426

PRODUCT AND TECHNICAL INFORMATION (800) 345-0447



The products and related information provided by Hercules are for **manufacturing use only.** Hercules makes no express, implied, or other representation, warranty, or guarantee concerning (i) the handling, use, or application of such products, whether alone, in combination with other products, or otherwise, (ii) the completeness, definitiveness, or adequacy of such information for user's or other purposes, (iii) the quality of such products, except that such products are of Hercules' standard quality. Users are advised to make their own tests to determine the safety and suitability of each product or product combination for their own purposes. Read and understand the Material Safety Data Sheets (MSDSs) before using these products.