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INITIAL SUBMISSION: LETTER FROM MONSANTO CO TO USEPA REGARDING ENCLOSED INFORMATION ON DEQUEST 2000 WITH ATTACHMENTS, DATED 01/10/84		
<b>Chemical Category</b>		
DEQUEST 2000		



INIT 07/14/94



84948888168

Monsanto

*Lou Boggs*  
**74I-0794-001679**

**#012484(13)**

MONSANTO INDUSTRIAL CHEMICALS CO.  
800 N. Lindbergh Boulevard  
St. Louis, Missouri 63167  
Phone: (314) 684-1000

**Contains No CBI**

January 10, 1984

*12-441*

*Nitrilo tris (methylphosphonic) acid*

Martin Greif, Executive Secretary  
TSCA Interagency Testing Committee  
Environmental Protection Agency (TS-792)  
401 M Street SW  
Washington, D.C. 20460

94 JUL 14 AM 9:28  
RECEIVED  
OPPT CBIC

Dear Sir:

Federal Register/Vol. 48 No. 218/ p 51519 requested information on a series of chemicals as part of ITC program. Monsanto manufactures one of the chemicals, CAS No. 6419-19-3 (Dequest® 2000) and herein submits the following information in response to this request:

1. Technical Bulletin No. IC/WT-101
2. Material Safety Data Sheet "Dequest® 2000 Deflocculant and Sequestrant".

Monsanto is updating this MSDS. We expect to have the revised version available by about the end of the first quarter of 1984. If you would like a copy of the revised MSDS please let me know.

3. Monsanto's annual production rate (1982) was between 1,000,000-10,000,000 lbs./yr. This rate has remained relatively consistent for the last several years but should increase modestly in the near term.
4. Environmental Data Sheet Dequest® 2000.

If I can be of any other assistance, please let me know.

Very truly yours,

*Gilbert J. McEwan*  
Gilbert J. McEwan  
Manger, Product Acceptability

GJM:jk  
enclosures

cc: J. R. Condray, Monsanto

## DEQUEST DATA SHEET - DEQUEST 2000

### Current Environmental Safety Assessment

No major environmental concerns exist for Dequest phosphonates. Use and physical properties of the products indicate that the aquatic environment will be the environmental compartment containing the majority of the released Dequest products. The products are practically non-toxic to and non-accumulative in aquatic species. The products undergo slow photodegradation and biodegradation under aerobic conditions. Flocculation and chlorination readily remove Dequest products during wastewater treatment and drinking water purification processes. Production volumes are comparatively low and the material reaching the environment is likely to partition to sediment. Environmental exposure concentration (both water and sediment) are expected to be several orders of magnitude less than toxic effect levels. It is highly unlikely that anticipated environmental concentrations of Dequest products would cause any significant metal mobilization from sediments.

### Environmental Properties

#### Environmental Mobility

Molecular Weight	299
Aqueous Solubility, mg/L	Inf
Molar Water Solubility (AQS/MWT x 10 <sup>3</sup> )	Inf
Vapor Pressure, Torr	1.0x10 <sup>-7</sup>
Henry's Constant (VPR/MWS)	-
log Octanol/Water Partition Coefficient (K <sub>ow</sub> )	-3.53
Calculated Bioconcentration Factor	0.016
Measured Bioconcentration Factor (Carp)	<1-4
Sediment/Water Partition Coefficient	170-1300
Metal Stability Constants (log K)	
Ca <sup>++</sup> , Mg <sup>++</sup>	7.6, 6.7
Cu <sup>++</sup> , Ni <sup>++</sup>	17.0, 15.5
Cd <sup>++</sup> , Pb <sup>++</sup>	12.7, 16.4
Co <sup>++</sup> , Zn <sup>++</sup>	18.4, 14.1
Hg <sup>++</sup>	21.7

#### Environmental Persistence

Semi-continuous activated sludge, % primary biodeg.	0.5-1.2
BOD Estimated	0
COD Measured, mg O <sub>2</sub> /mg Dequest	0.23
TOD Calculated, mg O <sub>2</sub> /mg Dequest	0.24
River Water, % primary degradation (138 d)	55
River Water, % <sup>14</sup> C <sub>2</sub> evolution (60 d)	12.3
Lake Water, % <sup>14</sup> C <sub>2</sub> evolution (38 d)	5-12
Soil Biodegradation, % CO <sub>2</sub> evolution (119-148 d)	0.6-14.6
Photolysis, % transformation (17 d)	0.2
+ Ferric nitrate	38-45
+ Zinc chromic nitrate	0-3
+ Cupric nitrate	2-3

Predicted Environmental Concentration and Distribution

Compartment

Water, ppm	2.0x10 <sup>-9</sup>
Air	-
Soil	-
Sediment, ppm	2.5x10 <sup>-6</sup>
Biota	-

Aquatic Toxicity

Acute Toxicity (mg/L)

Fish

Bluegill, 96 hr LC50	> 330
Rainbow Trout, 96 hr LC50	> 330
Rainbow Trout, 96 hr LC50	160
Rainbow Trout, 14d LC50	150
Channel Catfish, 96 hr LC50	1212
Sheepshead Minnow, 96 hr LC50	8132
Killifish, 96 hr LC50	700

Invertebrates

<u>Daphnia magna</u> , 48 hr EC50	297
Midge larvae, 48 hr LC50	11400
Grass Shrimp, 96 hr LC50	4574
Oyster, 96 hr LC50	201

Algae

<u>Spirodela</u> , Inhib. conc.	> 20
<u>Selenastrum</u> , Inhib. conc.	100
<u>Anabaena</u> , Inhib. conc.	10
<u>Chlorella</u> , Inhib. conc.	100
<u>Nostoc</u> , Inhib. conc.	> 30

Chronic Toxicity (mg/L)

Fish

Mosquito Fish, NOEL 60 d	-
Golden Shiner, NOEL 60 d	-
Green Sunfish, NOEL 60 d	-
Guppy, NOEL 5 months	-
Swordtail, NOEL 5 months	-
Blue Moon Pleety, NOEL 5 months	-
Rainbow Trout, MATC	> 23<47

Invertebrates

<u>Daphnia magna</u> , MATC	<25>20
-----------------------------	--------

Algae

<u>Selenastrum</u> , 96 hr, 336 hr, NOEL	20,20,7
--	---------

# MATERIAL SAFETY DATA

NO REPRESENTATION IS MADE AS TO THE ACCURACY OF THE INFORMATION AND RECOMMENDATIONS HEREIN. SEE PAGE 4 FOR CONDITIONS UNDER WHICH DATA ARE FURNISHED.

## MONSANTO PRODUCT NAME

Dequest<sup>®</sup> 2000 Deflocculant and Sequestrant

MONSANTO COMPANY  
800 N. LINDBERGH BLVD.  
ST. LOUIS, MO. 63166  
EMERGENCY PHONE NO.  
(CALL COLLECT)

314-694-1000

## PRODUCT IDENTIFICATION

SYNONYMS - Aminotri(methylenephosphonic acid)  
Nitrilo tri(methylenephosphonic acid)

CHEMICAL FORMULA -  $N(CH_2PO_3H_2)_3$

CAS NO. - 006419198

\*DOT PROPER SHIPPING NAME - Corrosive Liquid, nos

\*DOT HAZARD CLASS - Corrosive Material

\*DOT LABEL(S) - Corrosive Label

\*Applies only to material in containers other than portable tanks, tank cars or tank trucks.

## WARNING STATEMENTS

### CAUTION:

Causes eye irritation.

## PRECAUTIONARY MEASURES

Avoid contact with eyes.

Corrosive to mild steel.

## EMERGENCY AND FIRST AID PROCEDURES

In case of skin contact - Flush with water.

In case of eye contact - Flush with water for at least 15 minutes  
and get medical attention.

MONSANTO MATERIAL SAFETY DATA

**OCCUPATIONAL CONTROL PROCEDURE**

**Protective Equipment -**

Eyes - Goggles

Skin - Impervious gloves appropriate for this material should be used. Protective long-sleeved clothing should be worn and laundered before reuse.

**Permissible Air Concentrations:**

Product: amino tri(methylenephosphonic acid) wt %  $\approx$  38  
water wt % = 50  
other phosphonates wt %  $\approx$  12

TLV/TWA - Not established

**FIRE PROTECTION INFORMATION**

IGNITION TEMPERATURE  $^{\circ}$ F\* \_\_\_\_\_  
FLAMMABLE LIMITS \* \_\_\_\_\_

FLASH POINT  $^{\circ}$ F\* \_\_\_\_\_

Extinguishing Media - \*

\*Not applicable, aqueous solution.

Although this material does not meet the parameters for flammability, it will burn in the presence of a strong ignition source after the water has been removed.

**PHYSIOLOGICAL EFFECTS SUMMARY:**

50% Water Solution  
ORAL LD<sub>50</sub> (Rats) 2900 mg/kg  
"slightly toxic"  
DERMAL LD<sub>50</sub> (Rabbits) >6,300 mg/kg  
"practically non-toxic"

Undiluted  
SKIN IRRITATION (Rabbit) Moderately irritating  
FSHA 4.6  
EYE IRRITATION (Rabbit) Moderately irritating  
FSHA 53.6

MATERIAL SAFETY DATA Monsanto

**PHYSICAL DATA**

Appearance and odor: clear pale yellow solution with a slightly aromatic odor

Boiling Point: 107 °C

Freezing Point: 14.5 °C

Specific Gravity: 1.323 @ 20 °F

Viscosity: 36.5 cp @ 20 °C

**REACTIVITY DATA**

**HAZARDOUS DECOMPOSITION PRODUCTS** - Phosphines may form after all of the water has been removed. CO, CO<sub>2</sub>, and oxides of nitrogen may also be formed.

**HAZARDOUS POLYMERIZATION** - Does not occur.

**MATERIALS TO AVOID** - Avoid contact with concentrated caustic, will result in the evolution of heat.

**SPILL, LEAK & DISPOSAL INFORMATION**

**WASTE DISPOSAL** Dispose of by flushing into the sewer with large amounts of water.

**SPILL OR LEAKAGE PROCEDURES** Flush with water or neutralize with lime or soda ash and flush to sewer.

**ADDITIONAL COMMENTS**

Refer to Monsanto Technical Bulletin IC/SCS-320 for additional information on toxicity and environmental acceptability.

DATE January 1982

REVISED

x

SUPERCEDES

December 1977

FOR ADDITIONAL NON-EMERGENCY INFORMATION, CONTACT:

M. E. Klaus, Coordinator  
Product Acceptability and  
Environmental Affairs  
Specialty Chemicals Division  
Monsanto Industrial Chemicals Co.  
(314) 694-4437  
(A Unit of Monsanto Co.)

Monsanto MATERIAL SAFETY DATA

**NOTE:**

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NO WARRANTY, EITHER EXPRESS OR IMPLIED, OF MERCHANTABILITY OR FITNESS OF ANY OTHER NATURE WITH RESPECT TO THE PRODUCT OR TO THE INFORMATION AND RECOMMENDATIONS HEREIN IS MADE HEREUNDER.

# DEQUEST® 2000 and 2006 Phosphonates

For scale and corrosion control  
chelation, dispersion

2000

2006

2000

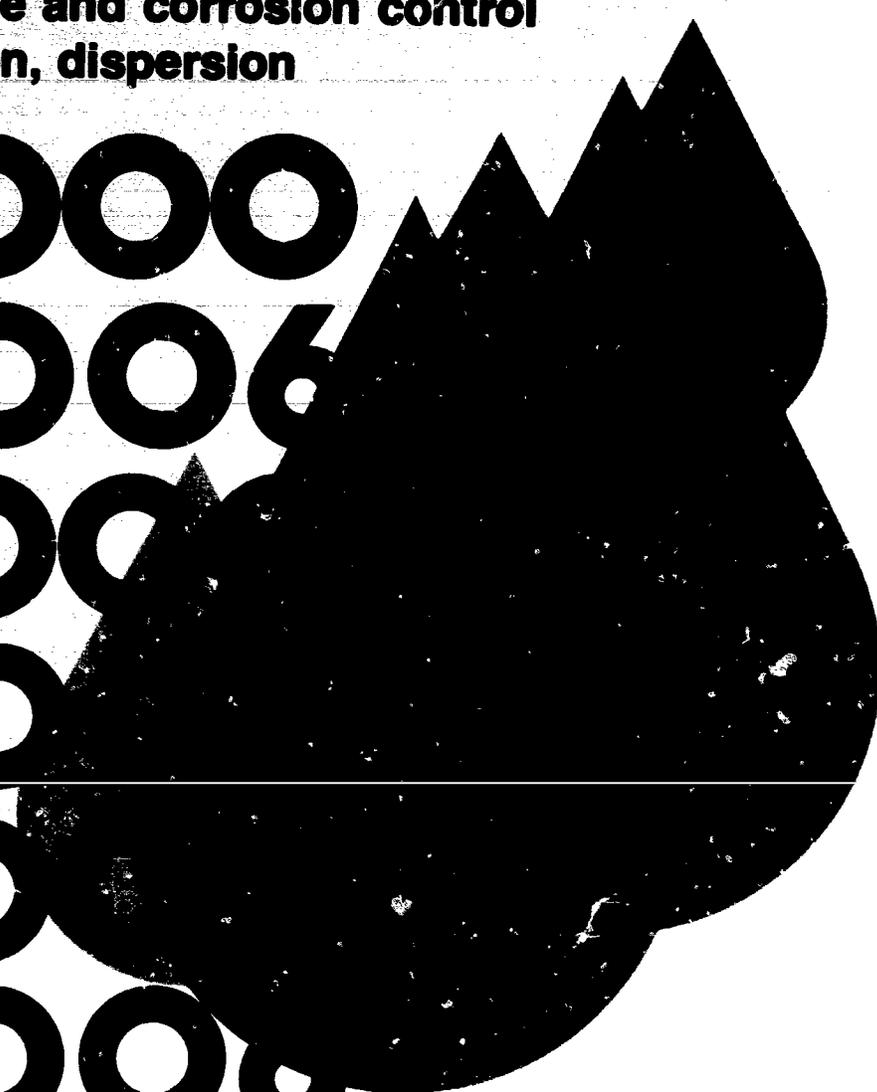
2006

2000

2006

2000

2006



Monsanto

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# DEQUEST® 2000 and 2006 Phosphonates

## Versatile Metal Ion Control Agents

### Introduction

DEQUEST® 2000 and 2006 phosphonates have led a revolution in the area of water treatment. In the past, chromate and acid formulations were used almost exclusively for control of scale and corrosion in cooling systems. With the advent of environmental regulations on chromate discharges, however, a new generation of alkaline treatments has been developed. Many of these formulations are built around DEQUEST 2000 and 2006 phosphonates because of their proven cost/performance effectiveness.

Other revolutionary developments are on the horizon. The wide range of phosphonate functionalities offer unique opportunities to develop novel products for many other application areas such as boilers, textiles, bottiewashing, oil fields, wet processing and a host of other industrial operations.

DEQUEST 2000 and 2006 phosphonates should be evaluated in any application requiring a hydrolytically stable, water-soluble product for:

- Threshold scale inhibition
- Chelation of calcium, magnesium and many other metal ions
- Deflocculation/dispersion of solids
- Crystal growth modification
- Sludge modification
- Corrosion inhibition

Already, this unique combination of properties has made these phosphonates key components for a variety of water treating applications. For many needs in water treating and aqueous processing, significant cost savings are possible using a DEQUEST phosphonate to replace part or all of another product to give:

- Multifunctional performance from a single raw material
- Superior cost/performance in scale inhibition
- Replacement of sequestration levels of other chelants with threshold levels of phosphonate
- Synergistic performance from formulated mixtures.

## Specific Applications

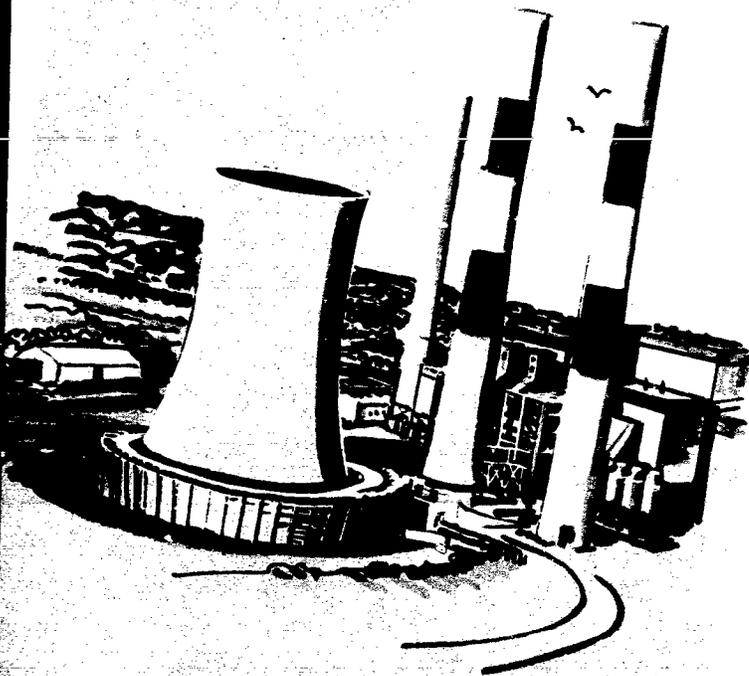
The advantages of DEQUEST 2000 and 2006 phosphonates are summarized. Sections following the summary contain technical details.

### Cooling Systems—DEQUEST 2000 and 2006 phosphonates

- Inhibit typical scales.
- Inhibit corrosion in formulations.
- Resist degradation in water.
- Minimize or eliminate acid feed.
- Offer acid or salt solutions.
- Are compatible with other treatment chemicals.
- Have field analytical methods.
- Offer better environmental acceptability than chromate.

## Boilers

- Resist degrading to orthophosphate.
- Chelate calcium, magnesium, iron and copper.
- Modify crystal growth.
- Are compatible with other treatment chemicals.



### **Textiles**

In peroxide bleaching, DEQUEST 2000 and 2005 phosphonates:

- Stabilize peroxide against oxygen loss.
- Stabilize peroxide baths against silicate deposits.
- Control water hardness.
- Control heavy metals.
- Improve bleaching performance.
- Improve fabric quality.
- Reduce bleaching cost.

A specially formulated product, DEQUEST XK, is available for scouring textiles. This product:

- Controls iron and removes iron stains.
- Controls water hardness.
- Improves soil removal and prevents soil redeposition.
- Reduces scouring costs.

### **Bottlewashing**

- Prevent scale formation.
- Prevent spotting and filming.
- Decrease costly downtime for descaling.

## Oilfield

In *continuous injection treatments*, DEQUEST 2000 and 2005 phosphonates:

- Inhibit typical scales.
- Assist in corrosion inhibition.
- Resist degradation in water.
- Are compatible with other treatment chemicals.
- Control iron fouling.
- Offer acid or salt solutions.
- Are stable to acid or base.
- Disperse existing solids.
- Are soluble in most brines.
- Are insoluble in crude oil.

in *squeeze treatments*, DEQUEST 2000 and 2006 also:

- Offer selective adsorption or precipitation.
- Have field analytical methods.

## Additional Applications

Based on the properties of DEQUEST 2000 and 2006 phosphonates, they should be evaluated for use in other industrial operations such as:

Aluminum Etching

Dairy Cleaners  
Deflocculation of Slurries

Electrochemical Machining  
Electroplating

Industrial Cleaners

Ion Exchange Resin  
Regeneration/Cleaning

Metal Finishing  
Metal Ion Control

Peroxide Stabilization  
Pigment Dispersion

Scale Removal

Toilet Bowl Cleaners  
Trace Metal Carrier in Fertilizer

Monsanto supplies several other DEQUEST phosphonates for use in specialized formulations. These products are available in various forms—as acids or as salts—as solutions or as dry solids—for the optimization of particular properties as well as for formulating convenience. Since the phosphonate structure is very stable in water, the DEQUEST phosphonates will not degrade under extended time and elevated temperature storage conditions.

Commercially available products are based on the following phosphonic acids:

DEQUEST 2000—

Aminotri (methylenephosphonic acid)

DEQUEST 2010—

1-Hydroxyethylidene-1, 1-diphosphonic acid

DEQUEST 2041—

Ethylenediaminetetra  
(methylenephosphonic acid)

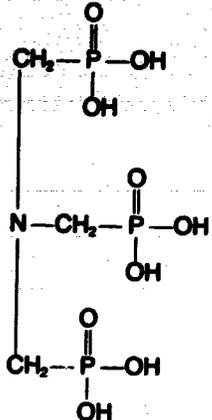
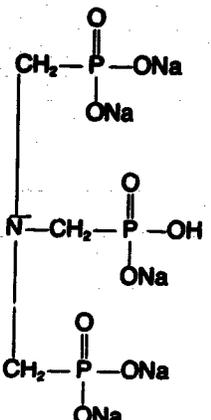
DEQUEST 2051—

Hexamethylenediaminetetra  
(methylenephosphonic acid)

DEQUEST 2060—

Diethylenetriaminepenta  
(methylenephosphonic acid)

# Chemical and Physical Properties

Structure	DEQUEST® 2000 Phosphonate (Acid)	DEQUEST 2006 Phosphonate (Na salt)
		
Molecular Weight	299	409
Chemical Name	Aminotri (methylene-phosphonic acid)	Aminotri (methylene-phosphonic acid), pentasodium salt
Abbreviation	ATMP	Na <sub>5</sub> ATMP
Chemical Abstracts Name	Phosphonic acid, nitrilotris (methylene) tri-	
Commercial Form	Aqueous Solution	Aqueous Solution
Typical Analysis*	50% (as acid)	(30% as acid) 40% (as Na <sub>5</sub> Salt)
Active Content		
Color	Pale Yellow	Yellow
Specific Gravity* 20°/15°	1.3	1.4
pH 1% Solids Solution @ 25°C.	< 2	10 - 11
Iron as Fe Chloride (Cl)	< 35ppm < 1%	< 35ppm < 1%
Viscosity (cps)* @ 20°C.	36.5	204
@ 40°C.	15.0	21
Viscosity (cts)* @ 20°C.	11.08	57.51
@ 40°C.	6.10	17.37
@ 60°C.	3.85	7.66

\*These data are based on samples tested in the laboratory and are not guaranteed for all samples. Please write for our current specifications.

### Solubility\*

DEQUEST 2000 and 2006 phosphonates are generally soluble in aqueous acids or bases and are insoluble in most organic solvents. Both products are miscible in all proportions with water, 40% NaOH, 98% H<sub>2</sub>SO<sub>4</sub>, and ethylene glycol.

The following table gives solubilities for the solid acid, ATMP, isolated from DEQUEST 2000. These data may be useful for determining the solubility of DEQUEST 2000 in various solvents.

Solubility of the Solid Acid Isolated from DEQUEST® 2000 Phosphonate at 25° C †

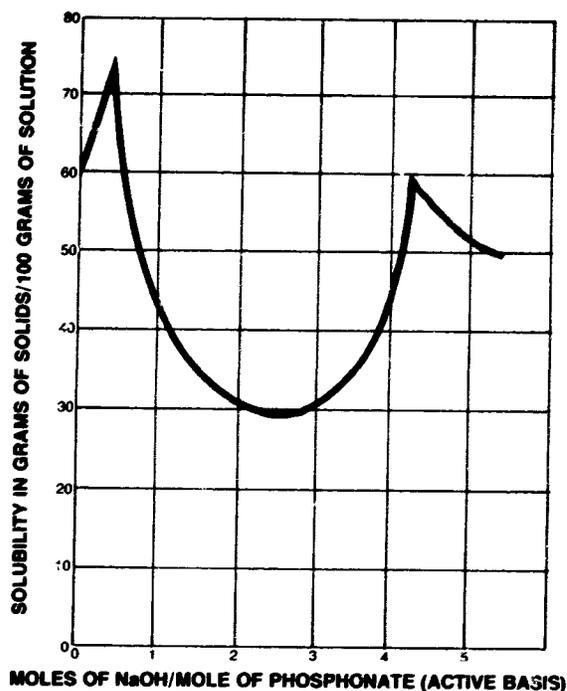
Solvent & Percent Concentration	ATMP Solubility, % by weight
HCl	60%
10%	28%
37%	16%
H <sub>3</sub> PO <sub>4</sub>	56%
10%	6%
85%	
H <sub>2</sub> SO <sub>4</sub>	49%
10%	49%
100%	
CH <sub>3</sub> OH	4%

\*These data are based upon samples tested in the laboratory and are not guaranteed for all samples.

†Does not constitute an express warranty. See NOTICE at the front of this bulletin.

Increasing the concentration of HCl or H<sub>3</sub>PO<sub>4</sub> causes decreased solubility of the active acid. In H<sub>2</sub>SO<sub>4</sub>, however, there is no difference in solubility between 10% and 100% acid solutions.

Figure 1  
Solubility of DEQUEST® 2000 Phosphonate in NaOH SOLUTIONS @ 25° C †



## Acidity

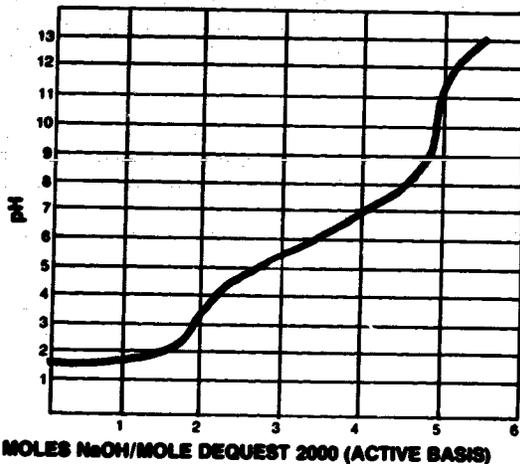
A pH titration curve of active ATMP acid isolated from DEQUEST 2000 phosphonate is shown below. The molecule titrates as a pentabasic acid with inflections at two and five moles of base per mole of acid. The sixth proton is so weakly acidic that it does not cause a break in the titration curve.

The acidity constants, determined in 1.0 molar  $\text{KNO}_3$  at 25° C, are<sup>†</sup>:

$\text{pK}_1$	<2
$\text{pK}_2$	<2
$\text{pK}_3$	4.30
$\text{pK}_4$	5.46
$\text{pK}_5$	6.66
$\text{pK}_6$	12.3

Figure 2

Titration of 0.1 N ATMP  
(the isolated active acid from DEQUEST® 2000  
Phosphonate) with 1.0 N  
 $\text{NaOH}$  †



<sup>†</sup>These data are based upon samples tested in the laboratory and are not guaranteed for all samples.

†Does not constitute an express warranty.  
See NOTICE at the front of this bulletin.

## Hydrolytic Stability

A well-known disadvantage of the inorganic polyphosphates in water treatment is their relatively rapid hydrolysis, or reversion, to orthophosphate. This reversion destroys scale inhibition and creates the additional problem of calcium orthophosphate scale formation. DEQUEST 2000 and DEQUEST 2006 phosphonates, however, are hydrolytically stable over a wide range of pH values and operating temperatures. This superior resistance to decomposition in aqueous solutions, even at elevated temperatures and extremes in pH, is a major advantage of the DEQUEST phosphonates when compared to polyphosphates and phosphate esters.

A screening study of the stability of DEQUEST phosphonates under simulated boiler conditions showed the excellent stability of these compounds toward degradation to orthophosphate. The study (Special Report 8035) used a simulated boiler water containing the phosphonates which was heated in an autoclave at 260° C (600 psig) for periods of two to seven days. Analyses for organically-bound phosphorus and for orthophosphate indicate that DEQUEST 2000 phosphonate has sufficient stability to merit further evaluation as a boiler water additive in either chelation or sludge-modification formulations. (See page 19, Boiler Water Treatment, for details.) The phosphonates' sequestration, crystal growth modification and scale inhibition properties may provide a beneficial synergism in boiler water treating compounds or other high temperature applications.

# Functional Properties of DEQUEST® 2000 and 2006 Phosphonates

## "Threshold Effect" Scale Inhibition

Inorganic polyphosphates have been used for scale inhibition since the 1930's when it was discovered that 1-10 ppm of sodium hexametaphosphate would retard or inhibit precipitation of  $\text{CaCO}_3$  from supersaturated solutions. This "threshold effect" is the prevention of precipitation from supersaturated solutions of scalants by ppm levels of inhibitor. However, one of the problems encountered with the polyphosphates is their instability which often leads to orthophosphate scale resulting from decomposition.

In the 1960's it was discovered that DEQUEST 2000 phosphonate also exhibited a threshold effect. The DEQUEST phosphonates show a decided advantage over the polyphosphates for threshold scale inhibition since they are hydrolytically stable and function over a wide pH range. A demonstration of the threshold effect is given in the following:

If separate solutions of calcium chloride and sodium sulfate are mixed in the presence of two ppm DEQUEST 2000 (active acid), precipitation of 6500 ppm  $\text{CaSO}_4$  is completely inhibited for more than seven days. The normal solubility of  $\text{CaSO}_4$  at this temperature is about 2100 ppm. Consequently, the amount of  $\text{CaSO}_4$  held in solution in excess of its normal solubility is 4400 ppm. This excess corresponds to a molar ratio of  $\text{Ca}^{++}$  to DEQUEST 2000 of the order of 10,000:1—whereas the normal molar ratio expected for  $\text{Ca}^{++}$  sequestration would be about 2:1. Obviously a mechanism other than sequestration is operative.

The threshold inhibition effectiveness of DEQUEST phosphonates varies with the products themselves and with the scalant compounds. The phosphonates are very effective in a wide variety of precipitating systems which include calcium carbonate, calcium sulfate, calcium phosphate, strontium sulfate, barium sulfate, ferric hydroxide, aluminum hydroxide, copper hydroxide, cobalt hydroxide and others.

## Calcium Carbonate Inhibition

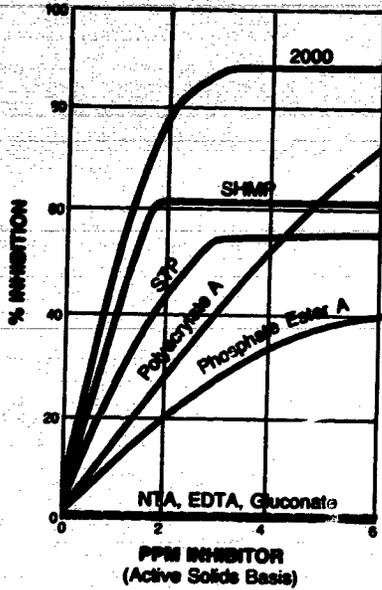
Monsanto tests have shown that DEQUEST 2000 is one of the best  $\text{CaCO}_3$  scale inhibitors of all the phosphonates and is far superior to polyphosphate, phosphate esters and polyacrylates.

Typical sequestering agents such as EDTA, NTA and gluconic acid are effective in holding stoichiometric amounts of calcium salts in solution. However, they are totally ineffective at the usual threshold levels where DEQUEST 2000 phosphonate gives its best performance. Therefore, threshold levels of phosphonate should be evaluated as replacements for stoichiometric amounts of chelants for control of precipitation. Significant cost savings are possible with such low concentrations of DEQUEST phosphonate.

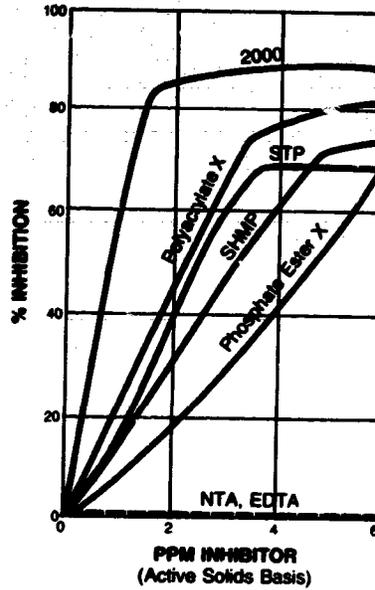
In judging threshold inhibitors, many factors have to be considered in the use system: pH, temperature, contaminating ions, inhibitor stability, scalant concentration and the amount of inhibition desired. The inhibitor must also be evaluated on a "cost/performance" basis—rather than on a simple "cost per pound" or "amount required" basis. This type of comparison is discussed in detail on page 12.

The following graphs show the inhibiting effect of DEQUEST 2000 on several scalants at various temperatures and pH levels. Furthermore, the efficacy over extended time periods is shown by the seven-day scale inhibition results. Several competitive threshold inhibitors are included to show that DEQUEST 2000 phosphonate gives markedly greater scale inhibition at lower dosage levels. These competitors are the best among the large number of commercially-available products tested.

**Average Seven-Day Percent Scale Inhibition\* †**  
400 ppm CaCO<sub>3</sub>, pH 10, 25°C



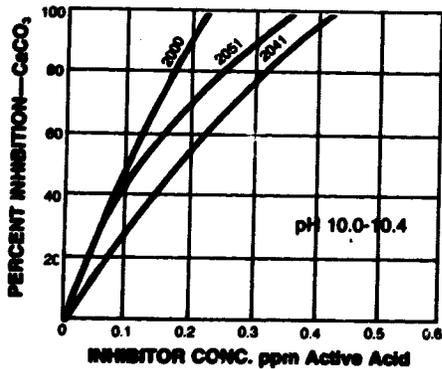
**Average Seven-Day Percent Scale Inhibition\* †**  
1600 ppm CaCO<sub>3</sub>, pH 8.5, 25°C



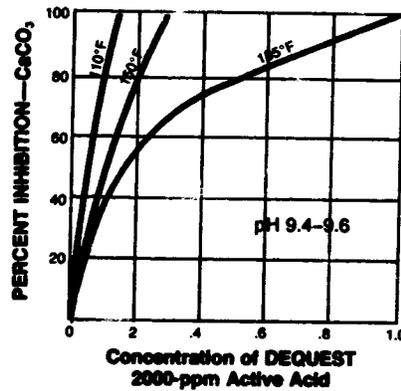
The following graphical presentation<sup>2</sup> of CaCO<sub>3</sub> scale inhibition at elevated temperatures demonstrates the superior performance, and also cost/performance, of DEQUEST 2000 phosphonate over 2041 and 2051.

The second graph<sup>3</sup> shows that increasing the temperature of a system increases the amount of DEQUEST 2000 required to control a fixed amount of CaCO<sub>3</sub>. Likewise, an increase in scalant concentration will require more phosphonate to maintain complete inhibition.

**Inhibition of Precipitation of 96 ppm Calcium Carbonate<sup>2</sup>**  
180°F, 24 Hours



**Effect of Temperature on Inhibition of 96 ppm Calcium Carbonate Precipitation after 24 Hour with 3 ppm DEQUEST<sup>®</sup> 2000 Phosphonate<sup>3</sup>**



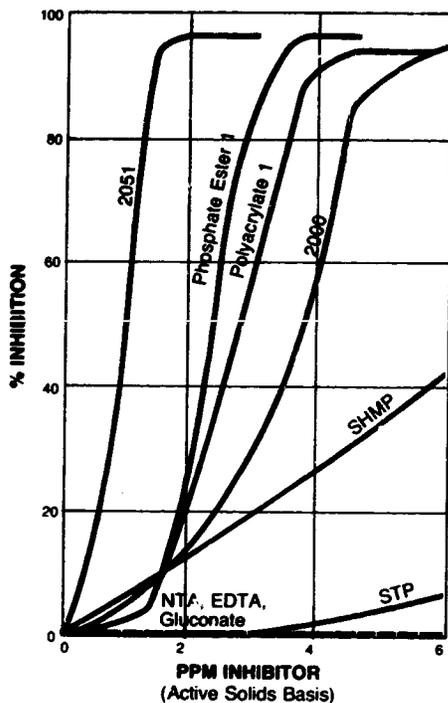
\*These data are based upon samples tested in the laboratory and are not guaranteed for all samples.

†Does not constitute an express warranty. See NOTICE at the front of this bulletin.

## Calcium Sulfate Inhibition

Calcium sulfate is another troublesome scalant encountered in desalination and other water treating applications. While DEQUEST 2000 is effective as an inhibitor for this scalant, particularly at lower  $\text{CaSO}_4$  concentrations, the most effective threshold inhibition is obtained with DEQUEST 2051 phosphonate. As with  $\text{CaCO}_3$ , typical sequestering agents such as NTA, EDTA and gluconic acid do not inhibit precipitation at threshold levels.

Average Ten-Day Percent Scale Inhibition\* †  
10,000 ppm  $\text{CaSO}_4$ , pH 7, 25°C



\*These data are based upon samples tested in the laboratory and are not guaranteed for all samples.

†Does not constitute an express warranty. See NOTICE at the front of this bulletin.

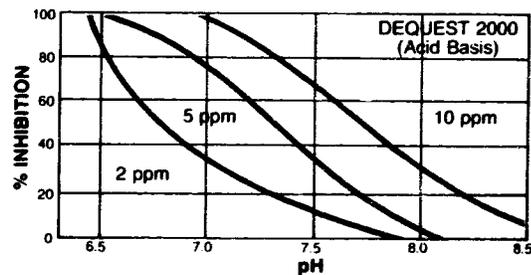
## Calcium Phosphate Inhibition

The problem of calcium orthophosphate precipitation in industrial cooling water systems is becoming increasingly important. Higher orthophosphate levels are occurring in cooling waters due to higher cycles of concentration, use of lower quality make-up water and use of polyphosphate corrosion inhibitors which can be degraded to orthophosphate in the system.

The most commonly identified calcium orthophosphate deposit in cooling water systems is hydroxyapatite,  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ . However, it is not hydroxyapatite which is initially formed, but instead a precursor which is widely identified as amorphous calcium phosphate.

Studies<sup>4</sup> have shown DEQUEST 2000 and 2010 to be the two most effective inhibitors among the DEQUEST phosphonates. The graph (Fig. 3) shows the percent inhibition by DEQUEST 2000 at various pH levels in concentrations from 2–10 ppm at 65.5°C in the presence of 50 ppm calcium and 120 ppm of orthophosphate.

Figure 3  
Percent Inhibition of Calcium Orthophosphate Scale by DEQUEST® 2000 Phosphonate at Various pH Levels and Concentrations\* †



### Barium Sulfate Inhibition

Barium sulfate is an especially difficult problem in secondary and tertiary oil recovery situations. Conclusions from laboratory data on inhibition of barium sulfate precipitation vary considerably depending upon the test conditions. Under certain conditions<sup>†</sup>, DEQUEST 2000 phosphonate is an effective inhibitor. However, Monsanto experiments (detailed in Special Report 8299) indicate the 2010, 2041 and 2060 families may be the most effective products on a cost/performance basis.

### Metal Hydroxide Inhibition

The ability of DEQUEST 2000 phosphonate to inhibit precipitation is not limited to alkaline earth systems. Hydroxides of iron, cobalt, copper, and aluminum and others can also be controlled with substoichiometric amounts of 2000.

Titrations of diluted solutions of DEQUEST 2000 with metal ions at pH 9 show that molar ratios of metal to phosphonate between 5 and 20 to one are held in solution. Metal ions tested were aluminum (III), cobalt (II), iron (III), nickel (II), manganese (II) and copper (II).

DEQUEST 2000 is also an effective product for control of iron fouling. The following table demonstrates this ability to maintain iron (III) in solution at neutral or alkaline pH with phosphonate levels well below the one to one molar ratio required by typical chelants. DEQUEST 2010 phosphonate, also known as a very effective iron control agent, is included for comparison. In the tests at pH 7, 2000 gave better results than 2010.

Inhibition of Fe (OH)<sub>3</sub> Precipitation at 25° C †  
Inhibition at pH = 7

DEQUEST® Phosphonate Additive	Additive Concentration ppm Active Acid	Fe (III) ppm	Fe (III) to Phosphonate Molar Ratio	Observations
2000	2	8	21:1	Clear 96 hrs " " "
	2	10	27:1	
	7	52	40:1	
	7	60	46:1	
2010	9	79	32:1	Heavy precipitate, 0.5 hours
Inhibition at pH = 9				
2000	50	50	5.4:1	Red-brown solution after 4 days " " "
	25	50	11:1	
	10	20	11:1	
	5	20	21:1	

\*These data are based upon samples tested in the laboratory and are not guaranteed for all samples.

†Does not constitute an express warranty.  
See NOTICE at the front of this bulletin.

## Scalant Mixture Inhibition

Frequently, less DEQUEST 2000 phosphonate is needed to inhibit precipitation from a mixture of scalants than might be expected from data on the individual scales. The concentration of 2000 required is not necessarily the sum of the concentrations used for each scalant.

Therefore, laboratory tests simulating such mixtures should be run to determine the use levels for the best cost/performance.

## Cost/Performance Evaluation of Scale Inhibitors

The preceding data show the excellent scale inhibiting performance of DEQUEST 2000 phosphonate under a variety of conditions on a weight (ppm) basis. However, in water treating and wet processing operations cost is a major parameter for evaluation. Scale inhibitors cannot be best evaluated on their chemical efficacy or on their cost per pound alone. A comparison of cost/performance is needed: how much of a given compound at its normal sales price is needed to do the job. This represents the true cost of treating a system based upon the monetary value of chemical necessary rather than upon the "pounds" or "ppm". In many instances, the higher cost inhibitor is actually the most economical product due to its greater efficiency.

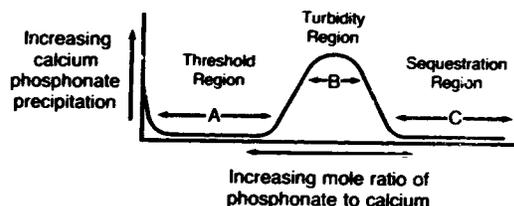
On a weight basis, DEQUEST 2000 phosphonate is one of the most effective inhibitors of  $\text{CaCO}_3$  commercially available. Evaluated on a cost/performance basis, its economic value is equally impressive.

In many comparison tests with polyacrylates, polyacrylamides, acrylate-silicate esters, and with phosphate ester formulations, DEQUEST 2000 has given a higher percent inhibition of  $\text{CaCO}_3$  at pH 8.5 or 10 for half to three-fourths the price. For  $\text{CaSO}_4$  inhibition, DEQUEST 2051 phosphonate exhibits a superior cost/performance over the same types of competitive materials and over DEQUEST 2000 as well. Moreover, these cost/performance comparisons reflect *only* the scale inhibition property of the products. The phosphonates offer further benefits of hydrolytic stability, control of iron fouling and relative indifference to pH.

Monsanto has carried out cost/performance studies with many of the widely used polyphosphates, polymers and phosphate ester formulations in comparison with DEQUEST phosphonates. Further information is available in Product Bulletin IC/SCS-319.

## Use Level Considerations

Like many other scale inhibitors, DEQUEST 2000 and 2006 phosphonates give best cost/performance at low concentrations around 1 to 20 ppm active acid. Higher levels can cause calcium phosphonate precipitation, a phenomenon termed the "turbidity region". This is shown below, where A indicates the range for optimum cost/performance, B shows the region of increasing calcium phosphonate precipitation and C gives the levels at which sequestration of calcium occurs:



The concentration of phosphonate at which turbidity begins differs for every combination of pH, calcium and phosphonate. Higher pH and increasing calcium levels shift the onset of precipitation toward lower concentrations of phosphonate. The order of susceptibility of the DEQUEST phosphonates to turbidity formation is:

2051 < 2000, 2041, 2060 < 2010

In actual field use in cooling water systems, turbidity region precipitates are generally not encountered at 1 to 20 ppm active levels or with phosphonates other than DEQUEST 2010. Special Report 8433 gives details and representative graphs determined under laboratory conditions.

### Crystal Growth Modification

DEQUEST 2000 phosphonate functions as a crystal growth modifier as well as providing threshold scale inhibition<sup>2</sup>. Crystals of  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  grown from supersaturated solutions are small, thin, sharp and elongated. With the addition of 1 ppm DEQUEST 2000 (active acid basis), precipitation is reduced and the crystals formed are larger and less needle-like. Increasing the concentration of DEQUEST 2000 further reduces precipitation and yields thicker, larger and more rounded crystals.

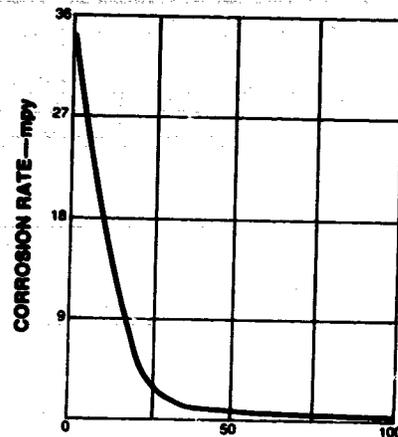
Crystal growth modification may be one of the dominant mechanisms of action by sludge conditioning agents when used in phosphate and carbonate cycle boilers. Additives are used to produce a fluid, non-adherent sludge which can more easily be removed during boiler blowdown. The thermal and hydrolytic stability of DEQUEST 2000 phosphonate and its capability to modify crystal habit suggest it as a candidate for evaluation as a boiler water additive.

### Corrosion Inhibition

Metals such as steel, copper and brass which are used in construction of water-carrying systems must be protected from various forms of corrosion. Formulations of DEQUEST 2000 or the other DEQUEST phosphonates with corrosion inhibitors such as zinc, triazoles or others will give both scale inhibition and synergistic corrosion protection.

DEQUEST 2000, alone, acts as a corrosion inhibitor for mild steel if relatively high concentrations are used<sup>3</sup>:

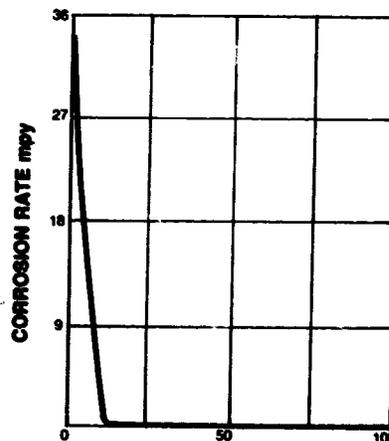
Corrosion Inhibition of Mild Steel with DEQUEST<sup>®</sup> 2000 Phosphonate alone<sup>3, 4</sup>



Conc. of DEQUEST 2000 Phosphonate—ppm active acid

However, synergistic formulations of DEQUEST 2000 phosphonate with other corrosion inhibitors are more effective on a cost/performance basis. For example, the following graph shows the same experiments as before, but now using a 1.4 to 1 molar ratio of zinc ion to DEQUEST 2000 (active acid). With this formulation, the corrosion rate at 12–15 ppm 2000 is approximately the same as that given by 50 ppm of 2000 alone<sup>5</sup>.

Corrosion Inhibition of mild steel with a combination of zinc and DEQUEST<sup>®</sup> 2000 Phosphonate (1.4:1 molar ratio)<sup>5, 6</sup>



Conc. of DEQUEST 2000—mg/l

<sup>3</sup>These data are based upon samples tested in the laboratory and are not guaranteed for all samples.

<sup>4</sup>Does not constitute an express warranty. See NOTICE at the front of this bulletin.

The molar ratio of zinc to phosphonate significantly affects the performance of a formulation. Approximately a 1 : 1 molar ratio of zinc to DEQUEST 2000 phosphonate (active acid basis) gives the best corrosion inhibition on mild steel in laboratory evaluations. Bulletin IC/SCS-314 provides formulating details for acid or neutralized solutions of zinc with several DEQUEST phosphonates.

Combinations of DEQUEST 2000 phosphonate with benzotriazole and tolyltriazole give good corrosion inhibition on both brass and steel as shown in the table below<sup>§</sup>. DEQUEST 2000 used alone accelerates the corrosion of brass<sup>§</sup> and copper<sup>§</sup>. Formulations, though, function as corrosion inhibitors for copper and its alloys.

Combinations of DEQUEST phosphonates with phosphates have been found to be effective corrosion inhibitors for steel. Details are provided in Special Reports available from Monsanto.

Most other typical corrosion inhibiting compounds are compatible with DEQUEST 2000 and 2006 phosphonates and in combination may show synergistic effect. In addition to zinc and triazoles, the following other types of inhibitors should be evaluated in formulations with phosphonates:

- Phosphates & polyphosphates
- Polymers
- Silicates
- Diethyldithiocarbamate
- Nitrites
- Oxazoles
- Imidazoles
- Lignins
- Lignosulfonates
- Tannins
- Phosphoric acid esters
- Boric acid esters
- Molybdates
- Chromates

**Weight Loss and Percent Inhibition Efficiency for Triazoles and DEQUEST® 2000 Phosphonate in Simulated Cooling Water (110 ppm Hardness)<sup>§</sup> †**

Inhibitor Conc. (ppm)		Brass		Steel	
Triazole	DEQUEST 2000	Wt. Loss (mg)	% I.E.	Wt. Loss (mg)	% I.E.
None	None	1.83	—	94.73	—
10 benzotriazole	—	0.01	100	90.88	4
10 tolyltriazole	—	0.01	100	90.48	4
10 benzotriazole	10	0.05	97	65.12	31
10 benzotriazole	20	0.28	85	20.94	72
10 tolyltriazole	10	0.05	97	39.58	58
10 tolyltriazole	20	0.41	78	17.35	82
—	10	2.09	0	39.40	58
—	20	4.23	0	26.22	72

<sup>§</sup>These data are based upon samples tested in the laboratory and are not guaranteed for all samples.

<sup>†</sup>Does not constitute an express warranty.

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## Sequestration

A common manner of comparing the effectiveness of sequestering agents is the stability constant of the complex formed. This value is a measure of the extent to which a complex will form from free metal ion and sequestrant.

For formation of a given complex,  $M^{+n} + L^{-m} \rightleftharpoons ML^{(n-m)}$  the equilibrium constant would be:

$$K_s = \frac{[ML^{(n-m)}]}{[M^{+n}][L^{-m}]}$$

where  $M^{+n}$  is the metal ion,  $L^{-m}$  the sequestering agent, and  $ML^{(n-m)}$  the complex formed. The stability constant is then expressed as the logarithm of the equilibrium constant,

$$\text{Stability Constant} = \text{Log } K_s$$

A comparison of the stability constants thus gives an indication of the amount of

unsequestered metal ion in solution. The larger the value, the smaller the amount of free ion.

The following are the stability constants of the active acid component of DEQUEST 2000 and 2006 phosphonates indicating their sequestering effect with various metals.

In application areas such as textiles and bottiewashing, sequestering agents can be evaluated in laboratory tests on a practical weight basis. This is done by determining the weight of calcium held in solution per gram of sequestrant in the presence of a precipitating moiety such as carbonate or oxalate. The results are affected by the precipitating agent, method of measurement and solution pH. Consequently, comparisons should be made only within a specific experiment. Such data, however, will separate superior sequestrants from other products.

Stability Constants for 1:1 Complexes of ATMP with Metal Ions  
Stability Constant

Metal Ion	Complex	log $K_s$	Conditions	Reference
Ca <sup>+2</sup>	ML	6.68	25°, $\mu = 1.0$	1
	ML	7.5	25°, $\mu = 0.1$	7
Mg <sup>+2</sup>	ML	6.49	25°, $\mu = 1.0$	1
	ML	7.2	25°, $\mu = 0.1$	7
Sr <sup>+2</sup>	ML	6.5	25°, $\mu = 0.1$	7
Ba <sup>+2</sup>	ML	6.5	25°, $\mu = 0.1$	7
Mn <sup>+2</sup>	ML	10.20	25°, $\mu = 0.1$	8
Fe <sup>+3</sup>	ML	14.6	20°, $\mu = 0.1$	9
Co <sup>+2</sup>	ML	14.37	25°, $\mu = 0.1$	8
Ni <sup>+2</sup>	ML	11.06	25°, $\mu = 0.1$	8
Cu <sup>+2</sup>	ML	17.40	25°, $\mu = 0.1$	8
Zn <sup>+2</sup>	ML	16.37	25°, $\mu = 0.1$	8
Th <sup>+4</sup>	ML	12.6	20°, $\mu = 0.1$	9
Cd <sup>+2</sup>	ML	12.7	25°, $\mu = 0.1$	10

On both a weight and a molar basis, DEQUEST 2000 phosphonate is much more effective than typical sequestrants in either the carbonate or the oxalate titration method. As shown in the following table, chelation values, expressed as milligrams of CaCO<sub>3</sub> per gram of "as is" product, are higher for DEQUEST 2000 by both methods than for any other product. Furthermore, on a molar basis in the carbonate test, DEQUEST 2000 and 2006 hold more than two moles of CaCO<sub>3</sub> in solution per mole of active acid phosphonate.

The next table shows the effect of pH on calcium sequestration by DEQUEST 2000 and 2006 phosphonates. These products give better calcium sequestration as the pH of the test solution increases from 9 to 12.

Sequestration of Calcium by DEQUEST® 2000 and 2006 Phosphonates at Various pH Values\* †  
(Carbonate Method, 25° C)

Product	Mg. CaCO <sub>3</sub> Per Gram of "As Is" Product			
	pH 9	pH 10	pH 11	pH 12
2000	350	390	400	425
2006	258	285	290	310

A Comparison of DEQUEST® 2000 and 2006 Phosphonates with Other Calcium Sequestrants\* †

Product	Form	Mg. CaCO <sub>3</sub> Per Gram of As Is Product		Moles CaCO <sub>3</sub> Per Mole of Active Acid Sequestrant	
		Carbonate, pH 11	Oxalate, pH 11-12 <sup>1</sup>	Carbonate	Oxalate
2000	50% Acid Solution	400	232	2.54	1.38
2006	40% Sodium Salt Solution	290	121	2.99	1.24
Na <sub>3</sub> NTA·H <sub>2</sub> O	93% Solid Sodium Salt	375	Not Applicable	1.04	—
Na <sub>4</sub> EDTA	40% Sodium Salt Solution	140	107	1.33	1.02
Na <sub>5</sub> DETPA	40% Sodium Salt Solution	124	84	1.55	1.05
Na <sub>5</sub> HEDTA	40% Sodium Salt Solution	153	123	1.27	1.02

NTA — Nitriotriacetic Acid.  
EDTA — Ethylenediaminetetraacetic Acid.  
DETPA — Diethylenetriaminepentaacetic Acid.  
HEDTA — N-Hydroxyethylthylenediaminetriacetic Acid.

<sup>1</sup>American Association of Textile Chemists and Colorists oxalate method.

\*These data are based upon samples tested in the laboratory and are not guaranteed for all samples.

†Does not constitute an express warranty.

See NOTICE at the front of this bulletin.

In any application requiring control of calcium, the DEQUEST phosphonates should be evaluated against other sequestrants, both on a performance and a cost/performance basis. The other functionalities of the phosphonates—hydrolytic stability, threshold scale inhibition, iron control and solids dispersion—may also increase the effectiveness of formulations in use. This combination of properties should be tested in plant and field trials following laboratory evaluations.

### Carbonate Test Method

Weigh accurately 2.00 grams of as is DEQUEST 2000 or 2006 phosphonate and dissolve in approximately 50 ml distilled water. Similarly, weigh out two grams (as is basis) of any other complexing agent whose activity is to be compared and dissolve in a like amount of distilled water. Adjust the pH to about 8 with NaOH. Add 10 ml of 2% Na<sub>2</sub>CO<sub>3</sub> solution. Adjust pH to 11.0 using 0.1N NaOH and a pH meter and make up the volume to 150 ml.

Titrate the samples with a 44.1 gram/liter calcium acetate monohydrate solution to a distinct, permanent turbidity by adding increments of 0.1–0.4 ml without stirring. Between increments, stir thoroughly. During titration maintain the pH constant at 11.0 by adding 0.1N NaOH.

Since the calcium acetate solution is equivalent to 25 mg CaCO<sub>3</sub> per ml, at the turbidity endpoint:

Milligrams CaCO<sub>3</sub> sequestered per gram of as is product =  $25 \frac{(\text{ml calcium acetate})}{(\text{Sample weight in grams})}$  of as is product.

### Deflocculation

DEQUEST 2000 and 2006 phosphonates are effective deflocculating agents for numerous slurries such as kaolin clay, barium carbonate, barium sulfate and drilling muds. In tests with kaolin clay, DEQUEST 2000 is comparable on a weight basis at room temperature to sodium tripolyphosphate. Furthermore, at elevated temperatures, DEQUEST 2000 remains effective while polyphosphates chemically degrade and fail. This superior hydrolytic stability makes DEQUEST 2000 a valuable additive for slurries exposed to high temperatures or long storage times.

Stability of Deflocculated Refined Georgia Kaolin\* †  
(50% Solids, 40° C)

Deflocculant	Wt. % Active Salt	Viscosity in Centipoise (Time in Days)				
		0	15	45	60	90
DEQUEST® 2006 Phosphonate	0.19	7	—	30	160	200
Tetrasodium Pyrophosphate	0.15	8	—	5,500	11,000	20,000
Sodium Tripolyphosphate	0.15	7	—	4,300	16,500	25,000
Sodium Hexametaphosphate	0.14	7	1,000	25,000	40,000	50,000

\*These data are based upon samples tested in the laboratory and are not guaranteed for all samples.

†Does not constitute an express warranty.  
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## Specific Applications for DEQUEST® 2000 and 2006 Phosphonates

The introductory section listed general advantages of DEQUEST phosphonates in a variety of application areas. In "Chemical and Physical Properties", the technical data giving the basis for these numerous applications were presented. The following sections give further details of specific application areas.

### Cooling Water Treatment

#### Scale Inhibition

DEQUEST 2000 phosphonate is a highly effective inhibitor for calcium carbonate and calcium sulfate, the two most prevalent scalants in cooling water systems. The data given previously have shown its excellent control of these and other scale-forming minerals at dosages of one to five ppm.

#### Corrosion Inhibition

DEQUEST 2000 phosphonate exhibits beneficial synergism when formulated with typical corrosion inhibitors such as chromate, zinc and polyphosphates. Monsanto Bulletin IC/SCS-314 gives detailed information for preparing acid or neutralized formulations of zinc with DEQUEST 2000 and with the other DEQUEST phosphonates. Laboratory data in this bulletin show that about a one-to-one molar ratio of zinc ion to DEQUEST 2000 (active acid) gives the best performance.

#### Chlorine Stability

In actual use in cooling water systems, DEQUEST 2000 phosphonate shows sufficient chlorine stability for practical threshold inhibition. Field experience by many companies has shown that this phosphonate can be used effectively in slug-fed chlorinated systems either with or without zinc. However, if greater inherent molecular stability to chlorine is desired, DEQUEST 2010 (which is a non-nitrogen phosphonate) should be evaluated.

The stability of DEQUEST 2000 in chlorinated systems can be increased by separating inhibitor and chlorine feed lines, by using slug, rather than continuous chlorination, and by formulating with zinc. Furthermore, other factors such as the presence of scalant ions and of easily oxidized microorganisms may also increase the stability of the phosphonate.

Formulations of DEQUEST 2000 phosphonate with a 1:1 molar ratio of zinc ion show substantially greater stability to chlorine than the phosphonate alone. The following table displays this effectiveness of zinc in preventing degradation of 5 ppm of phosphonate (25° C, pH 7) as indicated by the gradual loss of free chlorine with time.

**Stabilization of DEQUEST® 2000 Phosphonate to 2.4 ppm Free Chlorine by a 1:1 Molar Ratio of Zinc\* †**

Time Elapsed (Hours)	Percent of Original Free Chlorine Remaining
1	94%
17	77%
63	66%

\*These data are based upon samples tested in the laboratory and are not guaranteed for all samples.  
Special Report 8432 contains further data.

†Does not constitute an express warranty.  
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### Boiler Water Treatment

DEQUEST 2000 phosphonate is resistant to degradation to orthophosphate under high pressure and elevated temperatures. Since this product functions as a crystal growth modifier and as a chelating agent, it should be evaluated as a synergistic additive in both sludge modification and chelation formulations.

Tests to determine the thermal stability of DEQUEST 2000 phosphonate have been run under simulated boiler operating conditions of 280°C and 600 psi over periods from two to seven days. In the study, simulated boiler water was treated with 61 ppm and 42 ppm of DEQUEST 2000 phosphonate. Analyses were made to determine the amount of degradation of the phosphonate to orthophosphate. Under these temperature and pressure conditions, the half-life for degradation of DEQUEST 2000 to orthophosphate is approximately nine days. For practical purposes, the calculated decomposition to orthophosphate under these conditions is 1.3% after six hours; 2.6% after twelve hours; 5.3% after 24 hours.

Crystal growth modification may be the primary functionality necessary in a sludge modification agent to produce a fluid, non-adherent sludge. DEQUEST 2000 causes altered crystal growth which may give a synergistic effect in formulations with other agents such as polymers.

Evaluation of the phosphonates in combination with other chemicals should be done on both phosphate and carbonate cycle treatments. These tests should include both clean and dirty boiler conditions to determine the ability of the phosphonates to provide synergistic removal of existing calcium, magnesium and iron deposits.

The DEQUEST phosphonates are also effective chelating agents for calcium, magnesium, iron and copper. Chelation treatments using formulations of phosphonates and chelants such as NTA and EDTA may give complimentary performance with the phosphonate providing functionality where the usual chelants are weak. Synergism between phosphonates and NTA or EDTA may produce uniquely useful scale control formulations.

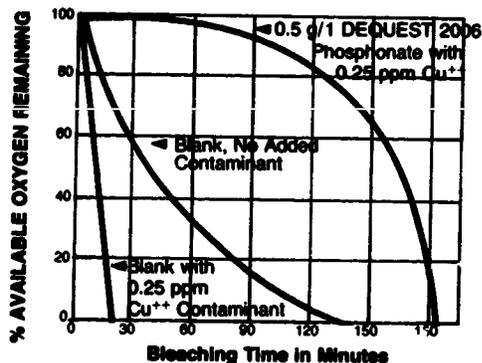
DEQUEST 2000 phosphonate is compatible with most typically used chelants, sludge modification agents, corrosion inhibitors and oxygen scavengers.

### Textile Applications

In the absence of trace metal contamination, peroxide solutions used in fabric bleaching show only gradual loss of available oxygen. However, as little as 0.25 ppm of copper or iron causes rapid loss of oxygen resulting in costly degradation of peroxide and consequent poor bleaching performance. As shown in the following graph, the presence of a small amount of DEQUEST 2006 phosphonate will stabilize the bleach solution at 210°F. This stabilization decreases peroxide loss, reduces peroxide costs and allows more efficient bleaching.

#### Stabilization of Peroxide Bleaching Solutions by DEQUEST® 2006 Phosphonate\* †

11 g/l H<sub>2</sub>O<sub>2</sub>, 35%, 11 g/l Sodium Silicate, pH 10.5 at 210°F in Soft Water Without Fabric



\*These data are based upon samples tested in the laboratory and are not guaranteed for all samples.

†Does not constitute an express warranty. See NOTICE at the front of this bulletin.

A typical improvement in bleaching performance due to the phosphonate sequestrant is shown by the following data. (Fabric having a whiteness number only two units higher than another is noticeably whiter to the eye).

**Bleaching Results With Three Fabrics\* †**

(Bleaching Solution:  
11 g/l 35% H<sub>2</sub>O<sub>2</sub>, 11 g/l sodium silicate,  
0.25 ppm Cu<sup>++</sup>, pH 10.5  
Procedure:  
1 hr. bleach @ 210° F, two 30-min. rinses with soft water).

**Whiteness Number<sup>1</sup>**

Fabric	No Sequestrant	DEQUEST® 2000 Phosphonate, 1.0 g/l
100% Cotton	75	84
50% Cotton/50% Polyester	78	84
35% Cotton/65% Polyester	100	109

<sup>1</sup>Whiteness Number = L - 3b, where L = 10 Rd determined on the Gardner Color Difference Meter.

\*These data are based upon samples tested in the laboratory and are not guaranteed for all samples.

†Does not constitute an express warranty. See NOTICE at the front of this bulletin.

Monsanto has also developed DEQUEST XK, a special phosphonate for textile scouring. This chelating agent has a high iron sequestering power that eliminates fabric spotting and improves cleanliness. The product is supplied in a neutralized form that is soluble in water in all proportions and in caustic up to 35% NaOH concentration.

Laboratory and production performance data are available from Monsanto. For further information on textile applications, including compatibility with caustic silicate scouring solutions, scouring performance and control of iron and calcium during scouring, contact your Monsanto representative.

**Bottlewashing**

DEQUEST 2000 phosphonate is recommended as a trifunctional additive in conjunction with gluconic acid for caustic bottlewashing formulations. The ideal sequestrant for caustic bottlewashing formulations is one that sequesters metal ions, disperses solids in the soaker stage, and prevents scale formation in the rinse stage. Most conventional chelating agents' activity is limited to complexing metal ions—with no effect on dispersion or clean rinsing. Inorganic polyphosphates possess these three desirable properties, but their poor hydrolytic stability at high pH and elevated temperatures seriously reduces their effectiveness.

DEQUEST 2000 phosphonate offers all three of the desirable functionalities in addition to hydrolytic stability. Possible benefits from its use in bottlewashing formulations include improved overall quality of washing effectiveness and gradual descaling of equipment down to a hard crust.

In the rinsing operation, the exceptional threshold scale inhibiting power of DEQUEST 2000 phosphonate may decrease spotting from mineral deposits on the glass surfaces. In the normal course of washing, the glass surfaces carry a "drag out" of NaOH and sequestrant from the wash solution. Scale from hard rinse water, soil removed from bottles and "drag out" solids all accumulate as unsequestered scalants. The caustic present raises the pH of the rinsing water film and the carbonate ion concentration increases. Any ordinary chelating agent has already spent its effect in the wash cycle, so little or no chelation occurs in the rinse and scale deposits on the glass surface. With DEQUEST 2000, however, such deposition may be inhibited by the threshold amounts of the phosphonate present in the drag out.

Laboratory and production plant evaluations indicate that formulations containing weight ratios in the range of 2:1 to 1:1.75 of DEQUEST 2000 (as is) to gluconic acid will give the most efficient bottle wash. Blends of DEQUEST 2000 phosphonate with glucoheptonate appear to be somewhat less

effective. Since finely divided microcrystalline precipitates form when DEQUEST 2000 is mixed with 50% NaOH, prior dilution of the caustic to about 45% may be necessary to provide a stable formulation.

### **Oil Field Water Treatment**

Secondary oil recovery flood water can cause scale deposition within subterranean formations and well casings. The water can also corrode pumps and flow lines. DEQUEST 2000 phosphonate is unique in the combination of properties it offers oil field formulators. No other class of compounds gives such a wide range of secondary properties along with threshold scale inhibition. Multi-purpose treatments to inhibit scale, to control corrosion, to disperse solids and to control iron fouling can be formulated with this phosphonate.

In the continuous treatment of injection water, threshold levels of DEQUEST 2000 phosphonate are effective in controlling scales such as  $\text{CaCO}_3$  and  $\text{CaSO}_4$ . Formulations with corrosion inhibitors such as zinc give products which may provide both corrosion and scale inhibition in a single package.

DEQUEST 2000 phosphonate is also particularly well suited to "squeeze treatment" needs. These treatments have given effective scale control for as long as 24 months. To be most effective, a scale inhibitor must control  $\text{CaCO}_3$  and  $\text{CaSO}_4$ , usually in combination with silica, iron and others. For long-term effectiveness, the inhibitor must be thermally and hydrolytically stable. It should have value in corrosion control. The performance of DEQUEST 2000 and 2006 on these counts has been covered in detail in preceding sections. In addition to these properties, DEQUEST phosphonates offer still others.

Selective solubility is a valuable secondary property for holding adequate treatment in the formation. Once the product solution has penetrated the formation, it is often desirable to precipitate the inhibitor to ensure continuous slow dissolution. Typically, phosphonates can be precipitated as the calcium phosphonate from alkaline solutions high in calcium.

DEQUEST phosphonates are effective dispersants. When silt or prior precipitate is present, dispersion of these solids will help minimize plugging of well bores and capillary conduits.

The ability to stabilize dissolved iron is another ancillary property. Phosphonate treatment inhibits formation of the voluminous floc of ferric hydroxide which plugs water injection surfaces and collects in well bore capillaries. Thus the scale inhibitor also serves to inhibit iron fouling.

Since resqueezing is done based on the amount of inhibitor returned, simple, accurate, field analysis methods are necessary. The amount of phosphonate in the produced water can be determined by either a thorium chelometric titration or by a colorimetric orthophosphate analysis after digestion.

Finally, phosphonates are compatible with most other common oil field treatment chemicals such as polymers, corrosion inhibitors, demulsifiers, surfactants and non-oxidizing biocides. These phosphonates are stable in acids and are thus compatible with acid treatments. They are also generally soluble in concentrated brines but are insoluble in crude oil.

For squeeze treatments where calcium carbonate is the predominant scalant, DEQUEST 2000 is recommended as the phosphonate that will yield the best cost/performance. Where calcium sulfate is the predominant scalant, one of the DEQUEST 2051 or 2041 phosphonates may show superior cost/performance. All these compounds possess the property of hydrolytic stability at elevated temperature necessary for effective squeeze treatment chemicals.

Further information on the use of DEQUEST phosphonates in oil field squeeze treatments is available in Special Report 8305.

# Conversion of DEQUEST® 2000 to DEQUEST 2006

## General:

Monsanto has used glass-lined and 316 stainless steel jacketed vessels for the neutralization reaction. Although corrosion rates for both materials have been acceptable, the use of 316 SS by customers must be at their own risk because of the 0.5% chloride content in DEQUEST 2000 and possible resulting stress

DEQUEST 2000 is a 50% aqueous solution of amino tri(methylene phosphonic acid) or "ATMP". DEQUEST 2006 is a 40% aqueous solution of the pentasodium salt of ATMP. The conversion procedure consists of reacting DEQUEST 2000 with 50% NaOH solution to a final pH of 10–11. The reaction is typical of a strong acid-strong base reaction and is therefore exothermic. Jacket cooling and agitation are required to prevent the solution from boiling. An operating temperature range of 60–90°C is suggested. Using a 1000 gallon jacketed vessel and ambient temperature cooling water, NaOH addition time for a 11,500 lb. batch is about 3 hours. Total cycle time including drumming is 8 hours. There are trace levels of ammonium ion in the DEQUEST product, so an ammoniacal odor during neutralization is typical.

If glass-lined equipment is used the DEQUEST 2000 charge is added first to the vessel followed by slow addition of the 50% NaOH solution. This procedure protects the glass from the very high alkalinity of the caustic solution. If 316 SS is used the procedure is reversed. In this case the full charge of 50% NaOH is added to the vessel and DEQUEST 2000 added slowly to the stirred caustic solution. This procedure protects the stainless from attack by hot acidic DEQUEST 2000 solution.

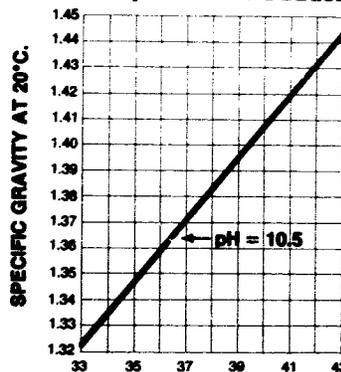
## Procedure: (100 lbs. of DEQUEST 2006; glass-lined reactor)

- Charge into the reactor 58.5 lbs. of DEQUEST 2000 and turn on the agitator.
- Begin addition of the 50% NaOH solution, allowing the temperature to reach 60°C before turning on the jacket cooling water. Adjust the caustic addition rate (with jacket water flowing) to hold temperature between 60° and 90°C. Once NaOH addition is begun, the entire amount should be added without intermediate stops and cooling. This

can lead to precipitation as intermediate di- and tri-sodium salts of ATMP have limited solubility (See Fig. 1).

- A total of 40.5 lbs. of 50% NaOH solution is required to obtain a pH of 10–11 (1% active solution).
- After caustic addition, withdraw a sample and measure pH of a 1% solution (2.5 g. of DEQUEST 2006 solution per 98 cc of distilled water). If the pH is not within specification, add DEQUEST 2000 or caustic as necessary to make adjustments.
- The specific gravity of the solution is then determined using a hydrometer. The measurement is made at 20°C. Using the density curve (See table below) and the specific gravity value obtained, the % active is found. This should be between 38 and 42%. If a concentration greater than 42% is obtained, water is added. If the concentration is below 38%, excess water is boiled off. Assuming no evaporation 1.0 lb. of dilution water should be required.
- When the correct pH and specific gravity are obtained, the DEQUEST 2006 is cooled to below 50°C and drummed. Viscosity of this solution at 20°C is about 250 cps. and at 60°C about 25 cps. Typical color is 175 APHA.
- For a sparkling clear solution, (optional) the product can be filtered through an in-line sparkler filter (polypropylene, fiber glass cartridge). As the solution is more viscous at ambient temperatures filtration should be made at about 60°C.

Specific Gravity of Neutralized DEQUEST® 2000



% "ACTIVE" AS DEQUEST 2006 EQUIVALENT

## Materials of Construction

## Analytical Methods

DEQUEST 2000 phosphonate is a strong acid similar to 75% phosphoric and should be handled accordingly. DEQUEST 2006 is a nominally alkaline solution. Corrosion studies of their effect on common materials of construction at 120° F maximum indicate the following:

Material	DEQUEST® 2000 Phosphonate	DEQUEST 2006 Phosphonate
316 SS	Not recommended*	Not recommended*
Steel	Unsatisfactory	Unsatisfactory
Natural Rubber	May yield black specks	OK
Butyl Rubber	OK	May turn product cloudy
Baked phenolic	May yield white flakes	OK
Polyethylene linings	Satisfactory	Satisfactory
Glass Fiber Reinforced Polyester	Satisfactory	Satisfactory

\*Subject to stress corrosion cracking due to the presence of about 0.5% chloride in DEQUEST 2000 and 2006.

A number of procedures are available for determining quantitatively the amount of phosphonate present in a variety of media. In addition to the methods listed below, two methods have been developed more recently:

Hach Chemical Company offers a method based on oxidation of the phosphonate to phosphate by ultra-violet light catalyztion.

A method based on oxidation of the phosphonate to phosphate by the Cerium ion at ambient temperature was developed by Monsanto and presented to the CTI as paper number TP-240A.

Detailed analytical methods will be supplied by Monsanto on request.

Further information is available in Bulletin IC/SCS-317.

Report #	Type of Analysis	Where Used	Quantity Range* (ppm)	Accuracy	Elapsed Time (min.)	Interferences
7158	Chelometric Thorium Titration	Laboratory Field	0-20 0-20 0-400	± 1 ppm ± 2 ppm ± 20 ppm	30 10	Transition metal ions, F <sup>-</sup> , PO <sub>4</sub> <sup>-3</sup> Other chelating agents
7256	Colorimetric Total Phosphorus	Laboratory	0-20	± 1 ppm	90	Si, other P forms
7666	Chelometric Copper Titration	Laboratory	0-50	± 2 ppm	60	Transition metal ions Other chelating agents
7823	Colorimetric Total Phosphorus (Hach Test Kit Model PO-24)	Field Laboratory	0-50	± 2 ppm	60	Si, other P forms
7917	Colorimetric Chelating Agent (LaMotte Polyphosphate Test Kit Model PPK-R)	Field Laboratory	0-10	± 2 ppm	10	Iron Other chelating agents
8097	Chelometric Thorium Titration (Taylor Test Kit Model 1583)	Field Laboratory	0-30	± 2 ppm	10	Transition metal ions, F <sup>-</sup> , PO <sub>4</sub> <sup>-3</sup> Other chelating agents

# Patent Bibliography

## Monsanto Patenting Licensing Policy on DEQUEST Phosphonates

The following U.S. Patents related to DEQUEST phosphonates are owned or licensed to Monsanto, and are available for license from Monsanto. Monsanto also owns a number of other patents relating to DEQUEST phosphonates. Inquiries concerning patent licenses should be addressed to:

Monsanto Company  
800 North Lindbergh Blvd.  
St. Louis, Missouri 63166  
Attention: Patent Department, MICC

**U.S. Patent 3,122,417**—Claims a process for stabilizing peroxy compounds by the addition of from 0.5 to 3% by weight of a defined organophosphorus compound. Covers DEQUEST 2010, 2015 DN.

Terms: Invoice or formal license at 3% of LTL selling price.

**U.S. Patent 3,148,151**—Claims the per-compound obtained by reacting an alkali metal salt of DEQUEST 2010 with hydrogen peroxide.

Terms: Formal license at 5% of LTL selling price.

**U.S. Patent 3,214,454**—Claims the process of adding certain organophosphorus compounds in threshold or sequestration amounts to aqueous systems to form metal ion complexes. This is a basic use patent which applies to scale inhibition, corrosion inhibition, swimming pool treatment and practically all other water treatment applications. Covers DEQUEST 2010, 2015 DN.

Terms: Invoice or formal license at 3% of LTL selling price.

**U.S. Patent 3,234,124**—Claims a method for inhibiting scale by adding sequestration amounts of amino tri (lower alkylene phosphonic acid) compounds. Covers DEQUEST 2000 and water soluble salts thereof.

Terms: Royalty free.

**U.S. Patent 3,234,140**—Claims a method for stabilizing peroxy solutions by the addition of at least 0.001 weight percent of an amino tri (lower alkylene phosphonic acid). Covers DEQUEST 2000 and salts thereof.

Terms: Formal license at 5% of LTL selling price.

**U.S. Patent 3,317,340**—Claims a process of treating iron and steel surfaces with certain defined organophosphorus compound for the purpose of removing iron salts prior to enameling. Covers DEQUEST 2010, 2015 DN.

Terms: Invoice or formal license at 3% of LTL selling price.

**U.S. Patent 3,336,221**—Claims a method for inhibiting scale by adding threshold amount of an aminomethylene phosphonic acid compound. This is the basic use patent in threshold scale inhibition. Covers DEQUEST 2000, DEQUEST 2041 and salts thereof.

Terms: Royalty free.

**U.S. Patent 3,380,924**—Claims a surface active composition consisting of a sodium potassium or ammonium soap stabilized against rancidity with from 0.05 to 2.5 weight percent hydroxyethane diphosphonic acid or salt. Covers DEQUEST 2010, 2015 DN.

Terms: Invoice or formal license at 3% of LTL selling price.

**U.S. Patent 3,475,293**—Claims a process for the electrodeposition of a divalent metal, e.g., copper, iron, nickel, zinc, cadmium, etc., in a neutral or alkaline aqueous dispersion containing an organophosphonate. Covers DEQUEST 2000, 2010, 2015 DN.

Terms: Formal license at 3% of LTL selling price.

**U.S. Patent 3,713,634**—Claims a process for the electrodeposition of a metal, e.g., gold, copper, nickel, zinc, cadmium, etc., from an aqueous solution at pH 6.0-13.0 containing an organophosphonate. Covers DEQUEST 2000, 2010, 2015 DN, 2041.

Terms: Formal license at 3% of LTL selling price.

**U.S. Patent 3,706,635**—Claims a process for the electrodeposition of a metal, e.g., gold, copper, nickel, zinc, cadmium, etc., from an aqueous solution containing a mixture of organophosphonates. Covers DEQUEST 2010, 2041.

Terms: Formal license at 3% of LTL selling price.

**U.S. Patent 3,928,147**—Claims a method of treating zinc die castings with an organophosphonate prior to immersion in an electroplating bath to inhibit immersion plating of copper from cyanide-free copper plating, electrolytes. Covers DEQUEST 2000, 2010, 2041, 2054, etc.

Terms: Formal license at 3% of LTL selling price.

Purchasers of DEQUEST phosphonates may request an invoice license wherein the amount of royalty will be added to the purchase price of the product and the purchaser licensed to use the invoiced amount in the patented method. A formal license unrestricted as to source of supply is also available from Monsanto.

The above licensing terms, including royalties, for each U.S. Patent is subject to change without notice. No representation is made respecting any other patents owned or licensed to Monsanto. Nothing contained herein is to be construed as a recommendation to use any product or process in conflict with any other patents.

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IC/SCS-320 DEQUEST 2000 and 2006 Aminotrimethylene phosphonates for water treatment.  
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8405 The DEQUEST Phosphonates in Boilers, Compatibility with Catalyzed Sodium Sulfite.

### Oil Field

- 8299 Inhibition of barium sulfate precipitation by the DEQUEST phosphonates at threshold levels.  
8305 Application of the DEQUEST phosphonates to Oil Field squeeze treatments—a literature survey.

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The use of DEQUEST sequestering agents in hydrogen peroxide bleaching and scouring of textiles.  
8295 Selected DEQUEST Phosphonates for Special Problems in Textiles.

### Bottlewashing

- 7243 Use of DEQUEST Organophosphorus Products in Bottlewashing.

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8433 Sensitivity of the DEQUEST Phosphonates to Elevated Levels of Calcium.  
MSL-0084 Use of DEQUEST Phosphonates for Corrosion Inhibition.  
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## Safety and Handling

Material Safety Data Sheet and Sales Specifications may be obtained from Product Acceptability and Environmental Affairs Group, Specialty Chemical Division, Monsanto Industrial Chemicals Co.

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*1114 Avenue of the Americas  
Tel. (212) 764-5000*

### **ST. LOUIS, MISSOURI 63167**

*800 N. Lindbergh Blvd.  
Tel. (314) 694-1000*

### **SAN FRANCISCO BAY AREA**

*2710 Lafayette  
Santa Clara, California 95052  
Tel. (408) 243-0414*

### **WILMINGTON, DELAWARE 19810**

*Suite 204, Bancroft Bldg.  
3411 Silverside Road  
Tel. (302) 478-4600*

### **Monsanto Industrial Chemicals Co.**

*a unit of Monsanto Company*

**800 N. Lindbergh Blvd., St. Louis, Mo. 63167**

0 0 2 5

**Monsanto Industrial Chemicals Co.**  
*a unit of Monsanto Company*  
**800 N. Lindbergh Blvd., St. Louis, Mo. 63167**

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