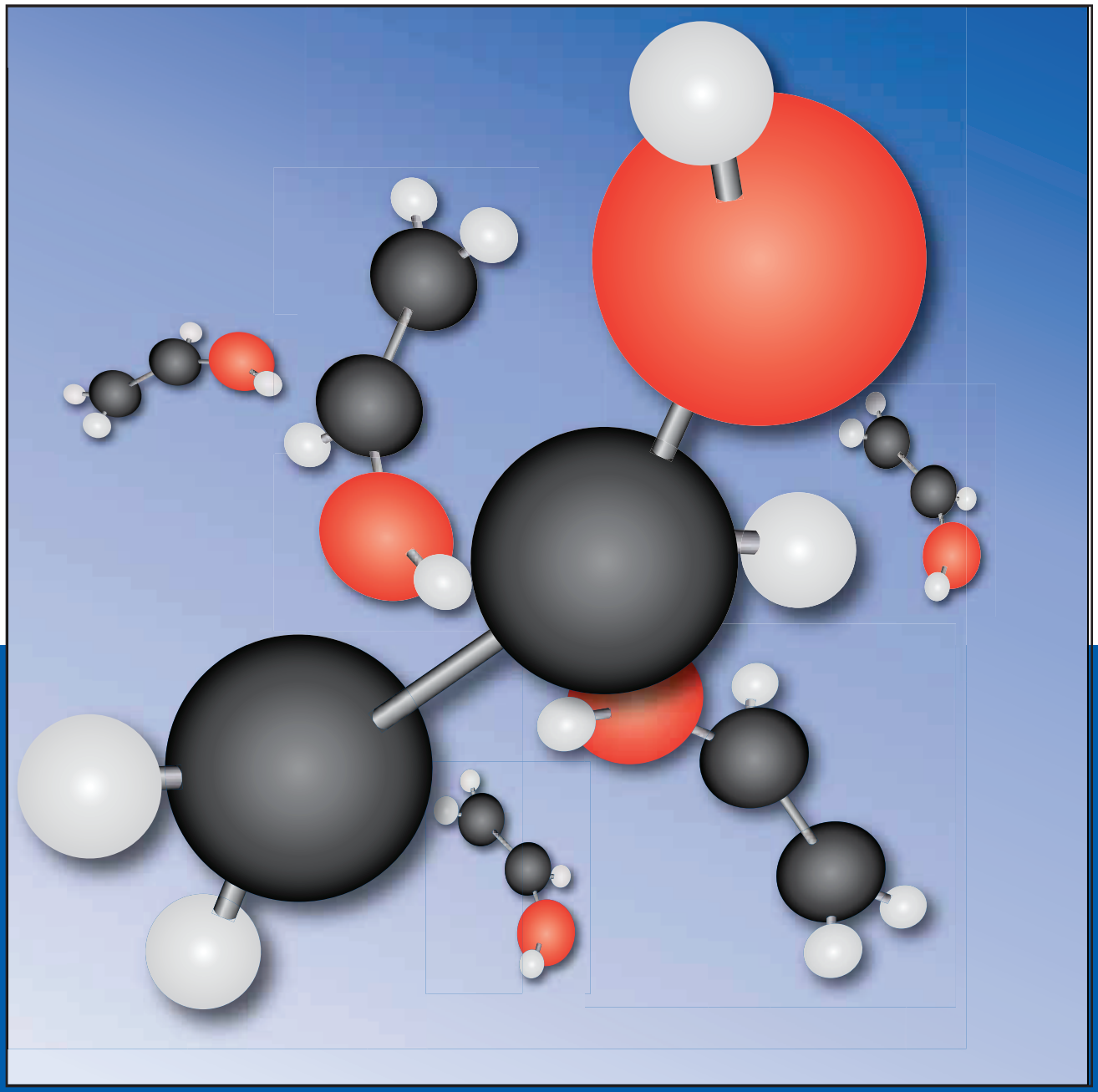
SEKISUI

SELVOL™
POLYVINYL ALCOHOL

# Selvol™ Polyvinyl Alcohol…
# Solution Preparation Guidelines



# *Solution Preparation Guidelines*



## About the Company

Sekisui America Corporation was formed in 1983. The corporation has grown to major player status in a number of business categories. Our activities cover import, export, manufacturing, sales and R & D. Products include housing materials, piping, wall coverings, films and tapes, chemicals and medical reagents and equipment. Our enterprise has recently expanded into the fields of civil engineering and advanced pharmaceutical development.

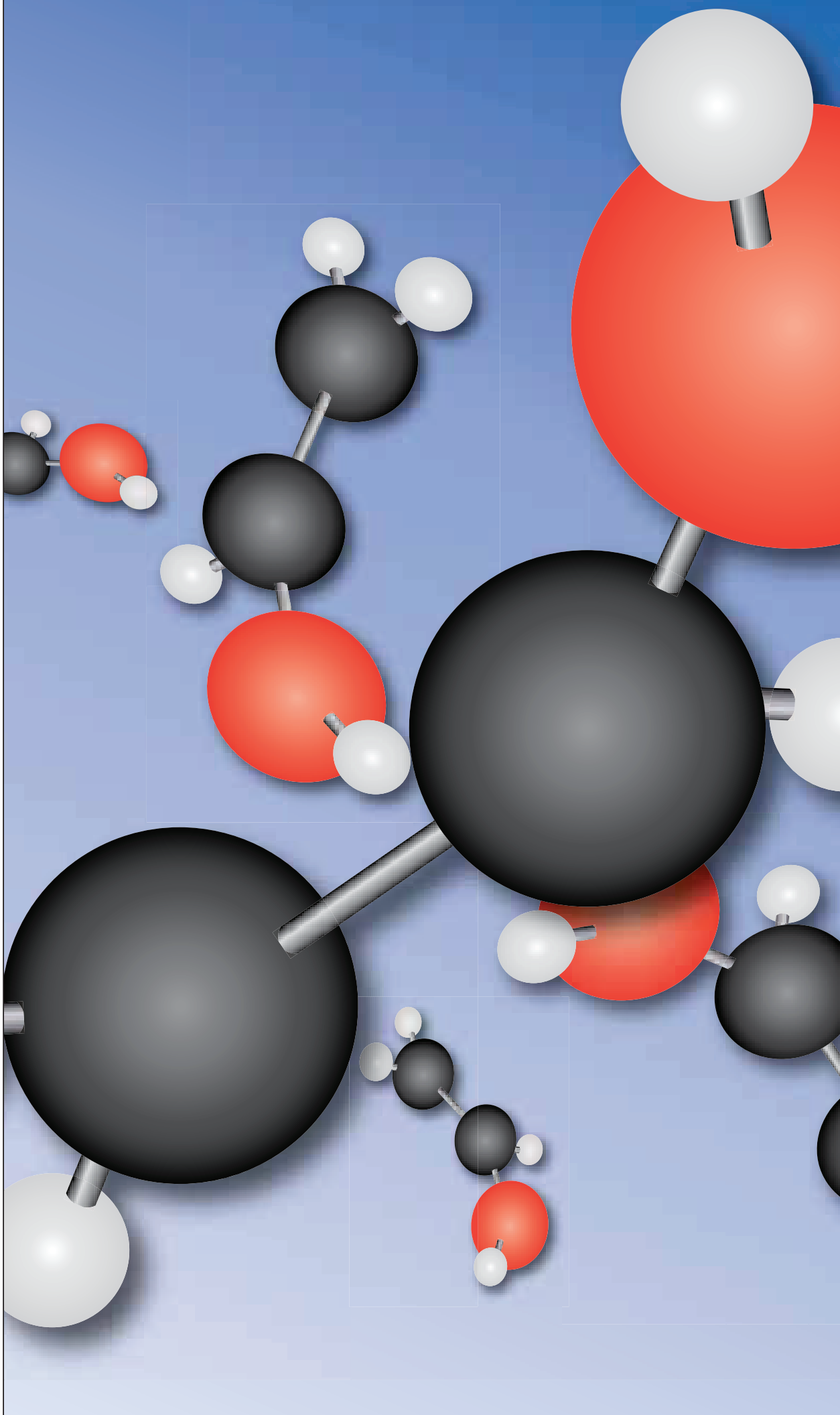
We have one overriding corporate goal: to use advanced technology to improve the quality of life.

## Polyvinyl Alcohol Business

Sekisui produces a full line of polyvinyl alcohol resins. Important end use markets for these high-quality polymer products include textiles, paper, adhesives, building products, and specialty applications.

Since the introduction of Selvol polyvinyl alcohol in 1956, Sekisui has become the recognized leader for new product and applications development of PVOH. The combined capacity of our three plants in Calvert City, Kentucky; Pasadena, Texas; and Tarragona, Spain makes Sekisui a leading global merchant supplier of PVOH.

**Selvol™ Polyvinyl Alcohol**



# Solution Preparation Guidelines

## SOLUBILITY

All Selvol polyvinyl alcohol grades are readily soluble in water. Other solvents include dimethyl sulfoxide, acetamide, glycols and dimethylformamide. Conditions for the dissolution are governed primarily by degree of hydrolysis, but they are influenced by other factors such as molecular weight, particle size distribution and particle crystallinity.

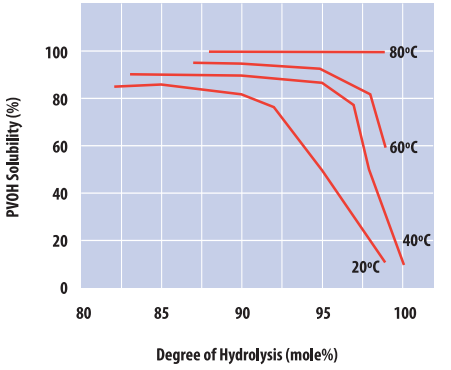
Optimum Solubility occurs at 87-89% hydrolysis. Grades in this range exhibit a high degree of cold water solubility. For complete dissolution, however, these grades require temperatures of 185°F (85°C), with a hold time of 30 minutes.

Higher hydrolysis grades, including the intermediate, fully and super grades, require progressively more energy to dissolve because of their greater degree of crystallinity. For dissolution, these grades require temperatures in the range of 200-205°F (93-95°C), with a hold time of 30 minutes (see Figure 1).

Once in solution, only partially hydrolyzed grades are viscosity stable with time. Super and fully hydrolyzed Selvol polyvinyl alcohol grades will tend to thicken somewhat through hydrogen bonding.

Viscosity relationships are portrayed as a function of concentration at various temperatures in Figure 2. These viscosities are for general guidelines only. Actual values will vary due to differences in the testing method.

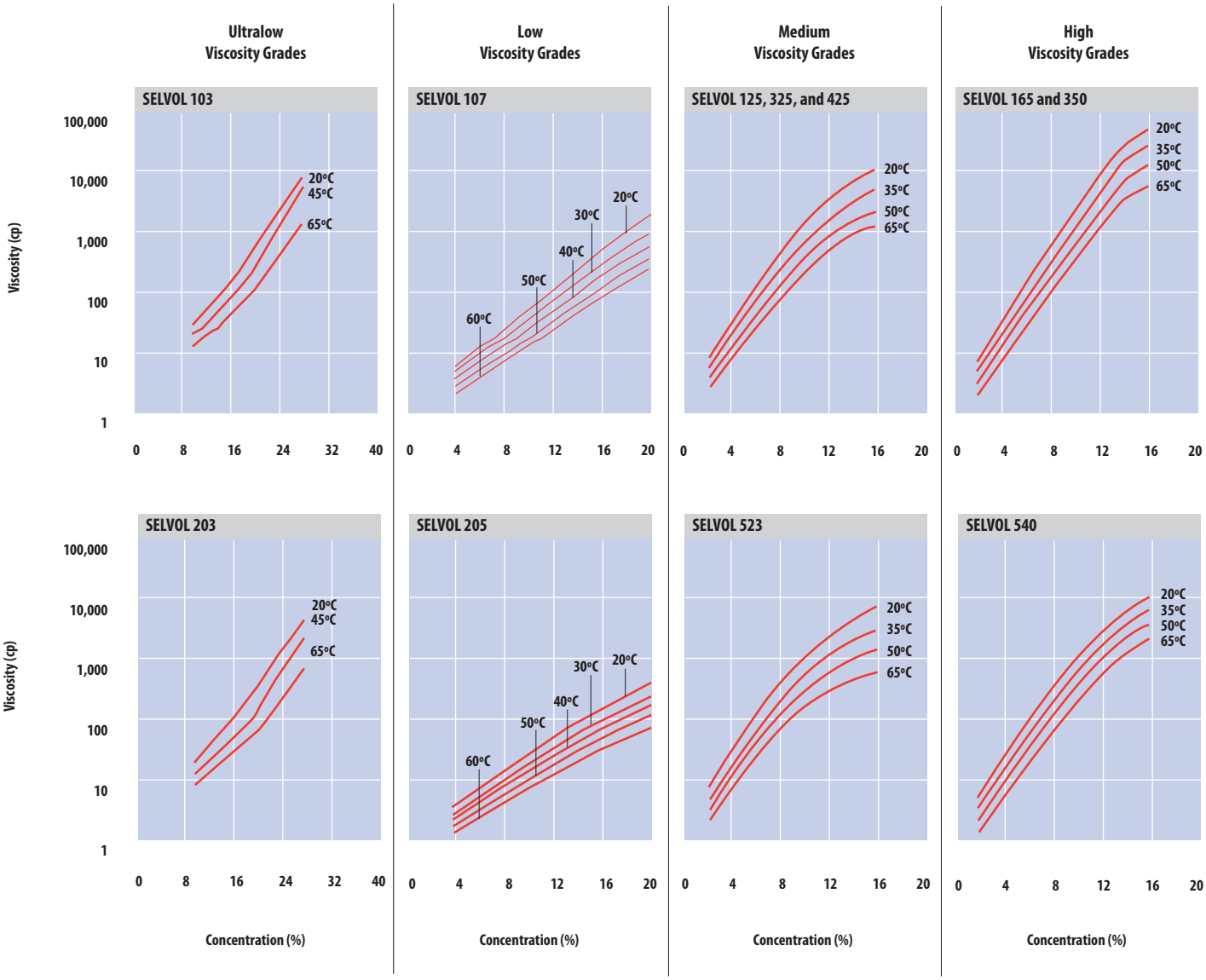
**Figure 1**




Higher hydrolysis grades require a lot of heat to dissolve, but even low hydrolysis grades require some heat for complete dissolution.

**Selvol™ Polyvinyl Alcohol**

**Figure 2** How the Concentration of Selvol Polyvinyl Alcohol Affects Solution Viscosity*




* Brookfield Viscometer, Model LVF at 60 rpm, 25˚C

# Solution Preparation Guidelines

## GENERAL GUIDELINES

The most critical step in effectively dissolving polyvinyl alcohol is to completely disperse the particle in water. Since the surface of the particles will swell very quickly an then clump together, it is very important to add the granule slowly, using good agitation, to cool water (<100°F/38°C).

Note that good agitation does not mean high shear. It means sufficient agitation to disperse the particles, without whipping air into the solution.

Cool water is important, particularly for the partially hydrolyzed grades, to allow good dispersion before particle swelling. If polyvinyl alcohol is added to hot water, the particles swell rapidly and clump together before complete dissolution can be achieved. Once the granules are well dispersed in cool water, the suspension can be heated. Partially hydrolyzed grades should be heated to at least 185°F (85°C); fully and super hydrolyzed grades should be heated to at least 205°F (95°C). The cook solution should be held at these temperatures for at least half an hour. See pages 7 and 8 for step-by-step solution preparation instructions.

### UNDISSOLVED PARTICLES

Undissolved polyvinyl alcohol particles will appear as transparent gels when incompletely cooked. They are not easily visible just by peering into the cook vessel, nor are they easily filtered out. If polyvinyl alcohol is not fully solubilized, it will not achieve its optimum performance. Complete solubility, however, is easy to achieve if you follow a consistent cooking procedure based on the recommendations in this brochure.

### FOAM

The tendency for polyvinyl alcohol to generate foam is highly dependent upon the degree of hydrolysis, and to a lesser extent, on the mechanical dynamics unique to each preparation and end-use process. Generally, the higher the hydrolysis, the less tendency to foam. Fully and super hydrolyzed grades may be used without defoamers, whereas intermediate and partially hydrolyzed grades nearly always require a defoamer. See Table 1 for recommended defoamers.

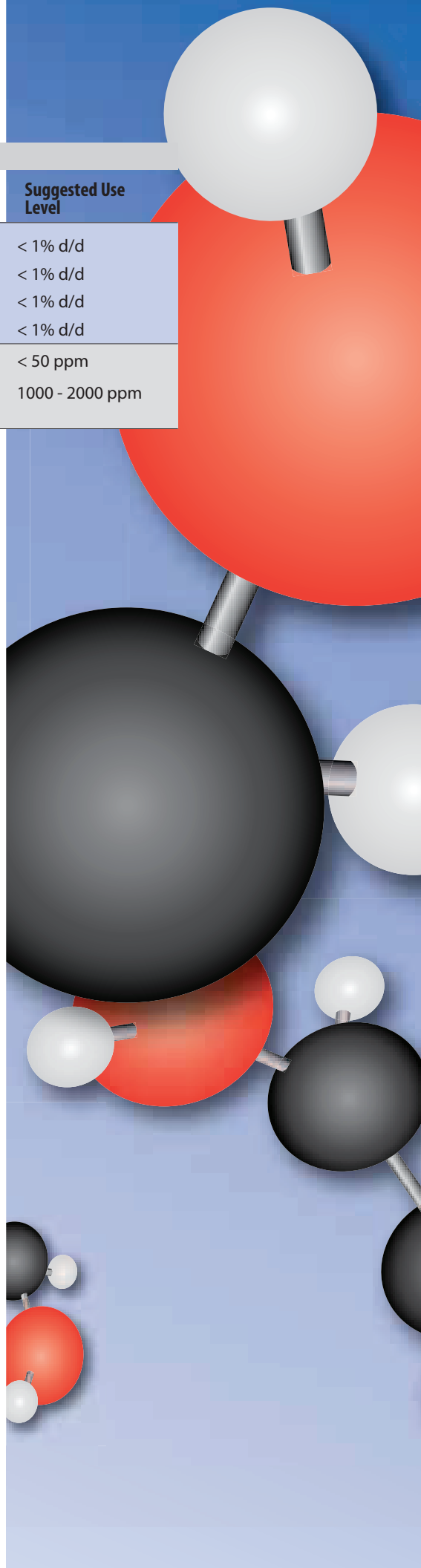
### BIOCIDES

If polyvinyl alcohol solutions are held for more than 24 hours, a biocide addition is recommended. See Table 1 for recommended biocides.

**Table 1** Recommended Additives for Selvol Polyvinyl Alcohol*

| Type | Brand or Generic Name | Manufacturer | Suggested Use Level |
|---|---|---|---|
| Defoamers | Foamaster VF | Henkel | < 1% d/d |
| | Foamaster KB | Henkel | < 1% d/d |
| | Antifoam 116 FG | Harcros | < 1% d/d |
| | Industrol DF 132 | BASF | < 1% d/d |
| Biocides | Kathon LX | Dow Chemical | < 50 ppm |
| | Dowicil 75 | Dow Chemical | 1000 - 2000 ppm |

* The FDA compliance status fo the recommended additives should be verified with the respective manufacturer.



**Determination of Polyvinyl Alcohol Addition for Desired Solution Solids Content**

$$\text{Polyvinyl Alcohol Addition (dry wt.)} = \frac{X * Y}{100\% - \%\ \text{Total Volatiles}^1}$$

X = Desired solution solids content[2]
Y = Net weight of final solution

**EXAMPLE**

To make 100 grams of a 20% solution of Selvol 205, add the following amount of polyvinyl alcohol. The % Total Volatiles for your current lot of Selvol 205 is 4.5%.

X = 20%
Y = Net weight of final solution

$$\text{Polyvinyl Alcohol Addition (dry wt.)} = \frac{\left(\frac{20}{100}\right) * 100\,\text{grams}}{\left(\frac{100\% - 4.5\%}{100}\right)} = 20.94\ \text{grams}$$

[1] The specification for % *Total Volatiles* is 5.0% maximum. Refer to the C of A for actual value.
[2] For best results do not exceed maximum solids guidelines (See Table 2).

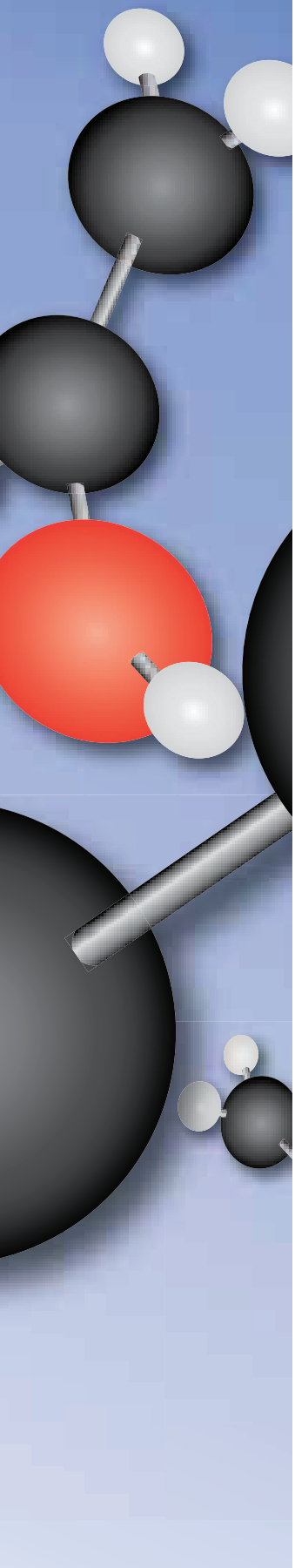
# Solution Preparation Guidelines

## SOLUTION PREPARATION BY LIVE STEAM, JACKETED VESSELS OR IMMERSED COILS

1. Fill tank with ***unheated water***. All lines should be free of borax and other con tainments.
2. Turn on mixer. Surface of water should move vigorously. Top blade should be submerged to half the height of the water.
3. If using a defoamer, add it at this point, prior to any polyvinyl alcohol addition.
4. Add polyvinyl alcohol to unheated water with agitation. Recommended rates of addition depend on the grade of polyvinyl alcohol. See Table 2.
5. For best results, do not exceed maximum solids guidelines. See Table 2.
6. Elevate solution temperature to 185-205°F (85-95°C). See Table 2 below. If using live steam, allow 15-20% condensation from the steam injection.
7. Upon reaching cook temperature, hold at temperature for 30 minutes.
8. Selvol polyvinyl alcohol is now in solution. It can be used at any temperature.
9. If the Selvol solution is stored for more than 24 hours, a biocide addition is recommended.

**Table 2**

| Selvol Grade | Addition Rate | Maximum Recommended Solids | Minimum Cook-Out Temperature |
|---|---|---|---|
| 103 | 10 sec/bag | 30% | 200˚F (93˚C) |
| 107 | 10 sec/bag | 20% | 200˚F (93˚C) |
| 310 | 10 sec/bag | 15% | 200˚F (93˚C) |
| 125 | 10 sec/bag | 10% | 205˚F (96˚C) |
| 325 | 10 sec/bag | 10% | 200˚F (93˚C) |
| 165 | 10 sec/bag | 7% | 205˚F (96˚C) |
| 350 | 10 sec/bag | 7% | 200˚F (93˚C) |
| WS-724 | 1 min/bag | 15% | 195˚F (91˚C) |
| WS-53NF | 1 min/bag | 10% | 185˚F (85˚C) |
| 418 | 1 min/bag | 12% | 195˚F (91˚C) |
| 425 | 2 min/bag | 10% | 195˚F (91˚C) |
| 203 | 2 min/bag | 30% | 185˚F (85˚C) |
| 502 | 2 min/bag | 30% | 185˚F (85˚C) |
| 205 | 2 min/bag | 20% | 185˚F (85˚C) |
| 805 | 2 min/bag | 20% | 185˚F (85˚C) |
| 513 | 2 min/bag | 15% | 185˚F (85˚C) |
| 523 | 2 min/bag | 10% | 185˚F (85˚C) |
| 823 | 2 min/bag | 10% | 185˚F (85˚C) |
| 540 | 2 min/bag | 7% | 185˚F (85˚C) |
| 840 | 2 min/bag | 7% | 185˚F (85˚C) |



## SOLUTION PREPARATION BY JET COOKER

1. Fill slurry tank with unheated water. All lines should be free of borax and other containments.
2. Turn on mixer. Surface of water should move vigorously. Top blade should be submerged to half the height of the water.
3. If using a defoamer, add it at this point, prior to any polyvinyl alcohol addition.
4. Add polyvinyl alcohol to unheated water with agitation. Recommended rates of addition depend on the grade of polyvinyl alcohol. See Table 3 below. To compensate for steam dilution, the intial concentration of Selvol polyvinyl alcohol should be higher than the required concentration.
5. For best results, do not exceed maximum solids guidelines. See Table 3.
6. Make sure all filters between the tank and jet, and the jet orifice and retention coil have a minimum hole diameter of 0.0185 in (4.75mm, U.S. Series Equivalent 4).
7. Pump slurry through the jet.
8. Jet temperature of 200-350°F (93-177°C) should be used. Steam pressure should be maintained above 25 psig.
9. Residence coil of 5-10 minutes is strongly recommended.
10. Allow for a 2% solids drop coming out of jet.
11. Unlike starch, polyvinyl alcohol does not instantaneously solubilize and must be kept in the hold vessel for 15 minutes prior to use. Solution temperatures in hold vessel should be maintained at 205°F for super hydrolyzed grades, 200°F for fully and intermediate hydrolyzed grades, and 185°F for partially hydrolyzed grades.
12. Water dilutions should take place after the 15 minute hold time.
13. If the Selvol solution is stored for more than 24 hours, a biocide addition is recommended.

**Table 3**

| Selvol Grade | Addition Rate | Maximum Recommended Solids | Minimum Cook-Out Temperature |
|---|---|---|---|
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# *Solution Preparation Guidelines*

EQUIPMENT

**Selvol™ Polyvinyl Alcohol**

### VESSEL

A vessel for dissolving Selvol polyvinyl alcohol should be constructed of 304 stainless steel or other corrosion-resistant materials to ensure contamination-free solutions. A height-to-diameter ratio of 1.0 is common. Baffles, which help to reduce particle descent and prevent severe vortex formation, should be positioned 90 degrees apart and extend the entire height of the vessel. It is recommended that the baffle width be 1/12 x the vessel diameter and that the offset from the wall be 1/72 x the diameter. The bottom of the vessel must be equipped with a flush valve to prevent particles from settling into and plugging the discharge line. Alternatively, a recycle loop may be employed off the bottom discharge line below the surface to minimize foam. See Figure 3 for recommended vessel design.

### AGITATION

Adequate agitation is required for both dispersing particles and for proper heat transfer. The agitation system generally consists of either one or two pitched turbine impellers. These impellers are most effective when they extend to 50-70% of the vessel diameter and are adjusted to a speed of 60-80 rpm. A variable speed agitator is useful in finding the optimum rpm level, and it can better accommodate viscosity changes during polymer dissolution. See Figure 3 for recommended guidelines on agitation.

### HEAT

Heat can be transferred to the solution via coils, jacket or the most widely preferred, direct steam injection (live steam injection or steam jet cooker).

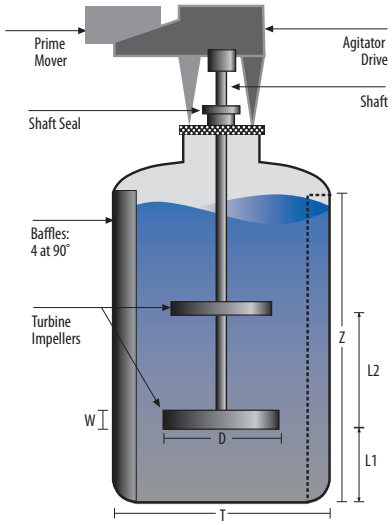
### LIVE STEAM INJECTION

Direct steam injection allows shorter heating times than indirectly heated jacketed or immersion coiled vessels. However, solution concentration is more difficult to control. Since 15-20% of the water content of the batch may come from condensed steam, care must be taken to allow for the dilution effect on the final concentration of the solution. Boiler treatment chemicals or other impurities that might be present in steam could have an adverse effect on the solution. Steam coils should enter through the side or bottom of the vessel, and steam pressure should be 15-25 psig to avoid localized water evaporation and baking of the resin at the steam inlet.

### JACKETED VESSELS

Jacketed vessels allow good control of the solution concentration, since no steam condensate dilutes the final solution. The heating jacket should cover only the bottom half to bottom third of the vessel. To prevent heat transfer loss, reduce concentration and cleaning problems, the temperature differential from the jacket to the vessel should be kept low to avoid baking the Selvol polyvinyl alcohol onto the hot vessel wall. Low pressure steam at 15-25 psig is recommended. Good agitation reduces the possibility of fouling.

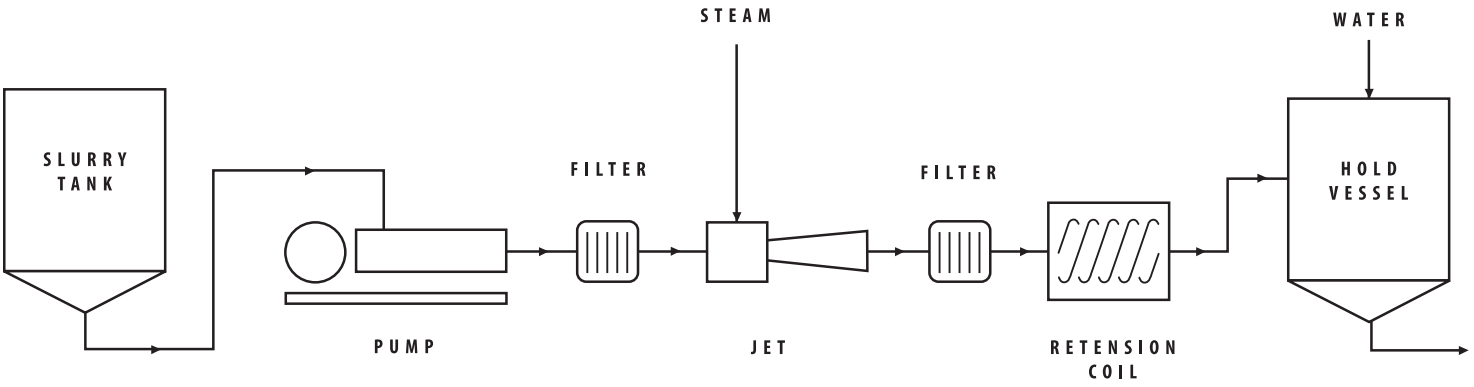
**Figure 3** General Design Considerations for Dissolving Systems




**Process Vessel and Major Components of a Double Impeller Turbine Agitator**

| | | Single Impeller | Double Impeller |
|---|---|---|---|
| N | Agitator Speed (rpm) | $1300/(V)^{0.4}$ | $1640/(V)^{0.4}$ |
| T | Tank Diameter (ft) | $\sqrt[3]{0.17\,V}$ | $\sqrt[3]{0.085\,V}$ |
| D | Impeller Diameter (ft) | T/2 | T/2 |
| W | Impeller Width (ft) | D/8 | D/8 |
| P | Agitator Power (horsepower) | $2.45 \times 10^{-8}\ N^3\ D^5$ | $5.0 \times 10^{-8}\ N^3\ D^5$ |
| L1 | Height of Bottom Impelleer from Tank Bottom (ft) | Z/4 | T/4 |
| L2 | Height of Top Impeller frm Tank Bottom (ft) | | (⅔)Z |
| V | Vessel Volume (gallons) | | |
| Z | Liquid Height in Tank (ft) | | |

**Figure 4** Typical Design for Steam Jet Cooking Systems



**IMMERSION COILS**

Immersion coils allow good control of the solution concentration. If immersion coils are used, the temperature of the coils should be kept below 240°F. Excessive heat may cause localized water evaporation, leading to polymer film formation on the heated surface. This film may flake off and cause subsequent problems. Immersion coils, if fouled, are not as easily cleaned as are jacketed vessels.

**STEAM JET COOKER**

High pressure steam jet cookers are designed and manufactured by starch suppliers for use in dissolving starch. While most Selvol polyvinyl alcohol customers batch cook, a small number of customers prefer to jet cook. When jet cooking Selvol polyvinyl alcohol, it is important to remember two critical differences between polyvinyl alcohol and starch:

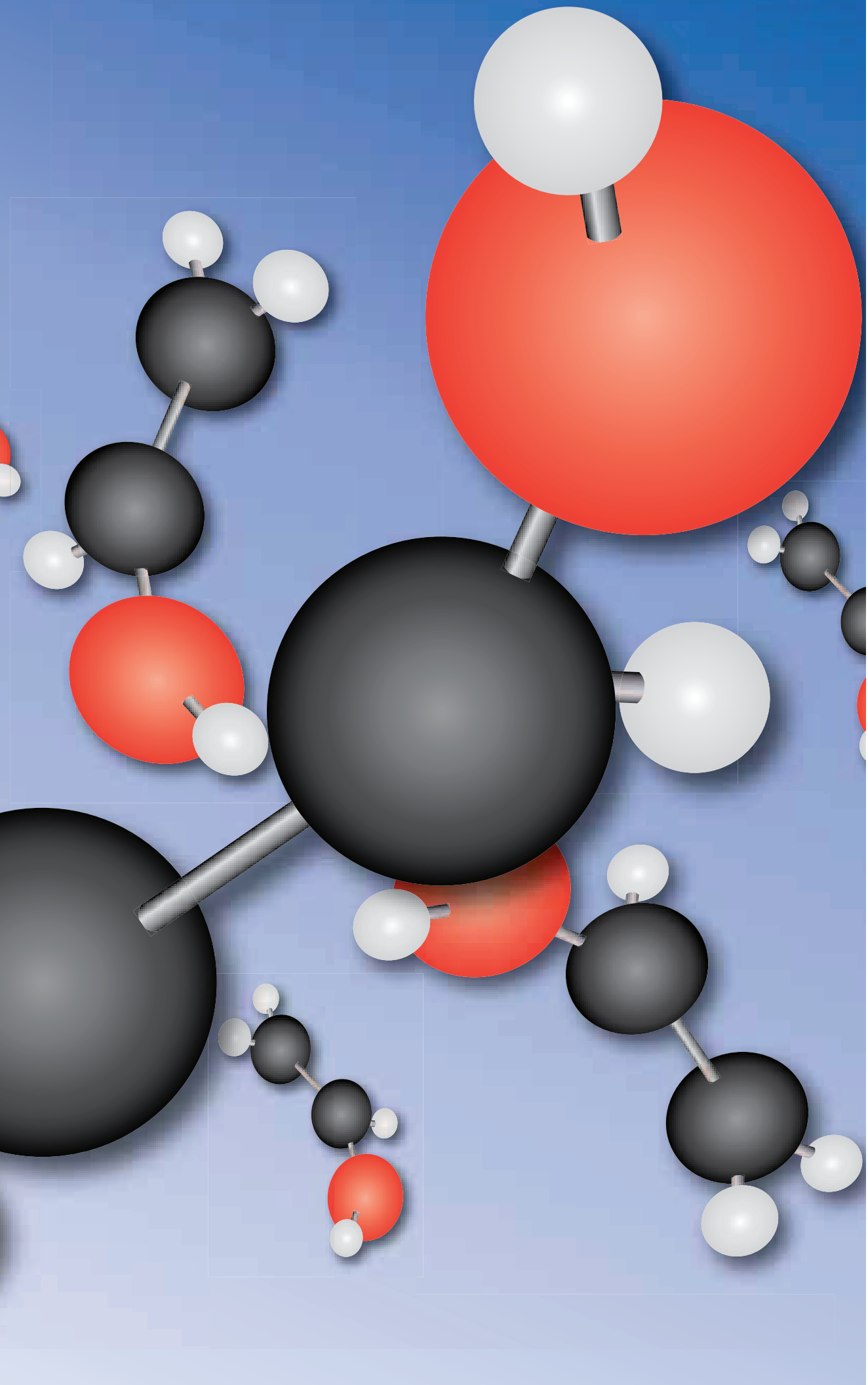
1. Selvol polyvinyl alcohol has a much larger particle size than starch.
2. Unlike starch, Selvol polyvinyl alcohol does not instantaneously dissolve when impinged with high pressure/ temperature steam. Therefore, additional heat and time are required to solubilize polyvinyl alcohol after the jet.

NOTE: Prior to purchasing your cooking system, please contact your Sekisui representative. Many of the design considerations will be based on the grade being dissolved and on your specific requirements.

Typically, Selvol polyvinyl alcohol is slurried in an agitated vessel. To cook the solution, the slurry is pumped from the slurry tank through a mixing head into which steam is injected (see figure 4). Steam pressure should be maintained above 25 psig. As pressure builds up in the mixing head, it acts like a pressure cooker, resulting in a substantial reduction in time required for dissolution as compared with conventional methods.

To ensure complete dissolution of all polyvinyl alcohol particles, a residence coil (5 to 10 minutes) and hold vessel are strongly recommended. The holding vessel should be insulated and equipped with a heat source to maintain solution temperatures of 205°F for super hydrolyzed grades, 200°F for fully and intermediate hydrolyzed grades, and 185°F for partially hydrolyzed grades.

All in-line filter screens, before and after the jet, must be adequately sized to allow all the swollen resin particle to pass through. The filters should have a minimum hole diameter of 0.0185 in (4.75mm, U.S. Series Equivalent 4).



International Offices

**North America:**
Sekisui Specialty Chemicals America
1603 West LBJ Freeway
Dallas, TX 75234-6034
Tel +1-972-277-2901
Fax +1-972-277-2907
www.sekisui-sc.com

**Europe:**
Sekisui Specialty Chemicals Europe S.L.
Ctra. N-340 Km. 1157  Apdo. 1388
43080 Tarragona
Spain
Tel +34 977549899
Fax +34 977544982



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