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MEDICAL DEPARTMENT



84948880254

Contains No CBI

January 4, 1984

74I-0794-001189

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

IR-406A

IR-453

RECEIVED
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94 JUL 14 AM 9:25

Dear Mr. Greif:

Thank you for the opportunity to provide information to the Interagency Testing Committee on dicumyl peroxide (CAS #80-43-3) and cationic acrylamide polymer (CAS #26006-22-4). Confidential information will be sent separately.

Cumyl peroxide [80-43-3]

This substance is sold as Di-Cup dicumyl peroxide. Enclosed are the following technical bulletins:

- ORC-201G Di-Cup® Dicumyl Peroxide Vulcanizing Agent and Polymerization Catalyst
- ORC-202C Di-Cup® Dicumyl Peroxide Safety Aspects of Storing and Handling 55-Gallon Shipping Containers
- ORC-203D Di-Cup® Dicumyl Peroxide Summary of Hazard Tests
- ORC-204E Di-Cup® Dicumyl Peroxide Toxicology
- ORC-208 Di-Cup® Peroxide Semibulk Container Melting and Handling System
- OR-614 Di-Cup Material Safety Data Sheet.

Copy sent to Perry
with attachments
1/10/84.

Mr. Martin Greif - EPA

- 2 -

January 4, 1984

Ethanaminium, N,N,N-trimethyl-2-[2-methyl-1-oxo-2-propenyl)oxy]-,
methyl sulfate, polymer with 2-propenamide [26006-22-4)

This product has been sold under a variety of tradenames. Currently, it is sold as Reten® 210, Reten 220, and Reten 240 cationic acrylamide polymers. The products differ in cationic functionality. Enclosed you will find the following technical bulletins:

T-112C	Cationic Reten® Products Summary of Toxicological Investigations ✓
420-7	Reten® 210 and 220 Cationic Water-Soluble Polymers
VC-471B	Preparation of Solutions of Reten® Polymer
WS 033	Reten® 210 Material Safety Data Sheet.

I trust this information will be helpful in your evaluation.

Sincerely,



Judith A. DeFranco
Regulatory Specialist

JAD:clw

Enclosures

0355i

0004



Hercules Incorporated
 610 Market Street
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Technical Information

BULLETIN VC-471B
 (Supersedes VC-471A)

PREPARATION OF SOLUTIONS OF RETEN® POLYMER

This bulletin discusses the preparation of solutions of **RETEN®** polymer for use in the plant and laboratory. Suitable types of equipment and preferred procedures are emphasized.

Reten polymer products are a series of high molecular weight, water-soluble, acrylamide-based homopolymers and copolymers in powder form. When solutions are required, proper makeup and metering procedures are important to ensure successful use of all the properties of the polymer.

These products are available as anionic, nonionic, and cationic polymers for use in aqueous systems as thickeners, flocculants, friction reducers, film-formers, retention aids, and adhesives. Product information on this series of polymers is available from the sales offices listed on the back page of this bulletin.

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GENERAL CONSIDERATIONS IN SOLUTION PREPARATION

As with most water-soluble thickeners, Reten polymer particles have a tendency to agglomerate, or lump, when first wet with water. Thus, the time required to achieve complete solution of all the polymer particles is usually governed by the degree of dispersion achieved as the dry polymer is initially wetted. Proper equipment and care in product handling ensures good initial dispersion and satisfactory solution preparation.

The chemical composition of the Reten polymer, together with the fact that the polymer may be anionic, nonionic, or cationic, imposes a number of conditions that must be met if good-quality solutions are to be prepared in the shortest possible time. Consequently, the rate of solubility of these polymers is generally dependent on the following:

- Good initial dispersion to obtain uniform wetting of the particles without lump formation.
- Moderate agitation during solution preparation to ensure complete hydration of the particles and thus avoid leaving residual gels in the system. (High-shear agitation should be avoided, as this can result in polymer degradation and some reduction in solution viscosity.)
- Use of clean water, free of contaminants that would be ionically incompatible with the particular Reten polymer being dissolved.
- Proper consideration of solids concentration, water temperature, and pH of the solution.

Dispersing the Polymer

A number of procedures can be used to obtain good dispersion of the dry polymer and avoid formation of swollen lumps, which are slow to dissolve. *Do not add water to the dry powder or dump the powder into water*, as this results in undesirable lumping.

To prepare large volumes of solution, disperse the dry polymer with a suction-lift eductor. Several types of these eductors are described in this bulletin.

If an eductor is not available, other procedures can be employed to achieve good initial dispersion of the dry powder. Some of these procedures are:

- Carefully sift the Reten polymer into the vortex of well-agitated water. The rate of addition must be slow enough to permit the particles to separate in the water without lumping. Complete the powder addition before any appreciable viscosity buildup is obtained in the solution. The rate of agitation can then be reduced, but it should be continued until a smooth, gelfree solution is obtained. Avoid agitation involving high rates of shear, as this can result in polymer degradation and reduced viscosity and flocculation efficiency.
- Dry-blend the Reten powder with any inert, or nonpolymeric soluble material such as sugar or salt that will be used in the formulation. Blending aids separation of the particles as they are first wetted, and reduces the tendency to lump. For best results, Reten polymer should comprise less than 20% of the total dry blend. This blend is then handled as described in the paragraph above.
- Slurry the Reten powder in a liquid that is a nonsolvent for the Reten, but miscible with water. In this manner, the particles of Reten can be rapidly dispersed in water and lumping will not occur. Suitable nonsolvents include acetone, alcohols, and propylene glycol.

Water Quality

Use clean, high-quality makeup water for preparation of Reten polymer solutions. The water should be free from suspended matter, dissolved salts, organic material, and chlorine. For best results, the hardness should be less than 100 ppm and the residual chlorine less than 1 ppm. Contaminants in the water can interact with the polymer to form insoluble flocs, reduce viscosity, and impair polymer efficiency.

Extremes of pH can adversely affect the viscosity and storage stability of Reten polymer solutions. The optimum pH range for each Reten polymer is given in separate product bulletins. Generally, a pH below 5 and above 10 should be avoided.

A water temperature of 50 to 90°F (10 to 32°C) is recommended. Temperatures above 100°F (38°C) should be avoided because they promote polymer degradation.

Solution Storage

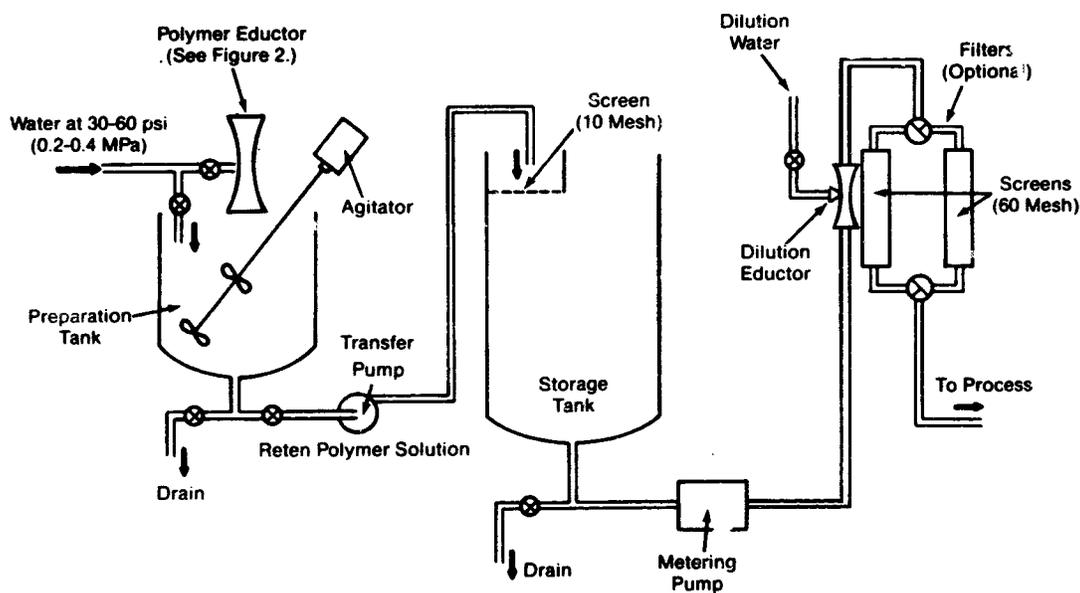
Reten polymer solutions of greater than 0.5% solids concentration can usually be stored for several days at room temperature without reducing their effectiveness. Avoid storing polymer solutions at temperatures above 100°F. Diluted solutions (0.1% solids) are less stable and must be used within a few hours.

The Reten polymer itself does not support the growth of microorganisms, but some waters contain enough organic matter to support bacterial growth. If microbial activity is evident, use a suitable bactericide. Your Hercules representative can suggest one that will be compatible with your process.

SOLUTION PREPARATION IN THE PLANT

A typical system for preparing large volumes of dilute solutions of Reten polymer for plant operations is shown schematically in Figure 1. It includes a preparation tank, storage tank, propeller or turbine agitator, transfer and metering pumps, Reten polymer eductor, and water dilution eductor. If necessary, suitable filters can be used to ensure solutions free of gels or particulate matter.

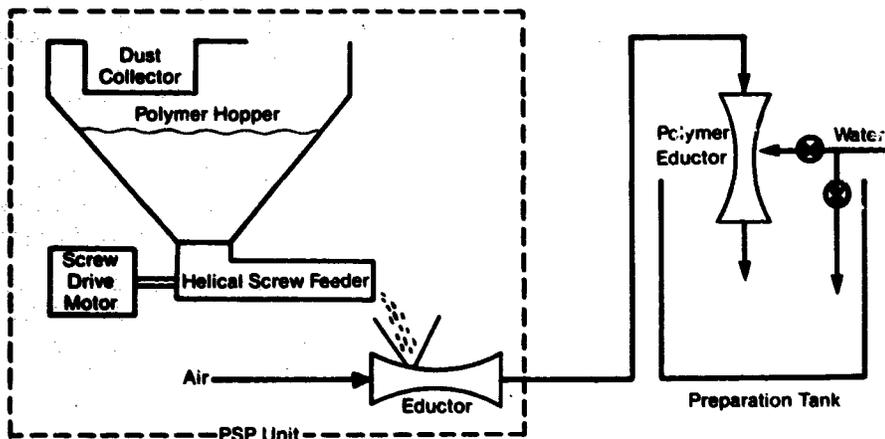
Figure 1
Typical Reten Polymer Solution Preparation System



PSP® Feed Unit

For users wishing the convenience of an automatic system, Hercules offers the PSP (polymer solution preparation) system. The PSP unit adds a Reten polymer feeder with hopper, feed screw, and air eductor, plus tank level indicators and an electrical control panel, to the basic polymer makeup system shown in Figure 1. The PSP unit requires initial calibration to set cycle times and tank liquid levels. It will then operate automatically. Operator attention is needed only to fill the hopper with polymer. A schematic of the PSP unit and its operation is shown in Figure 2.

Figure 2
Polymer Feeding and Wetting Technique



Hercules brochure 750-16 describes the PSP system in more detail. Contact your Hercules representative for additional information.

Polymer Eductors

Several mixing devices are available that allow polymer granules to wet properly and thus avoid formation of difficult-to-dissolve agglomerates. The eductors shown in Figure 3 are recommended for use with Reten polymer and are available from your Hercules representative.

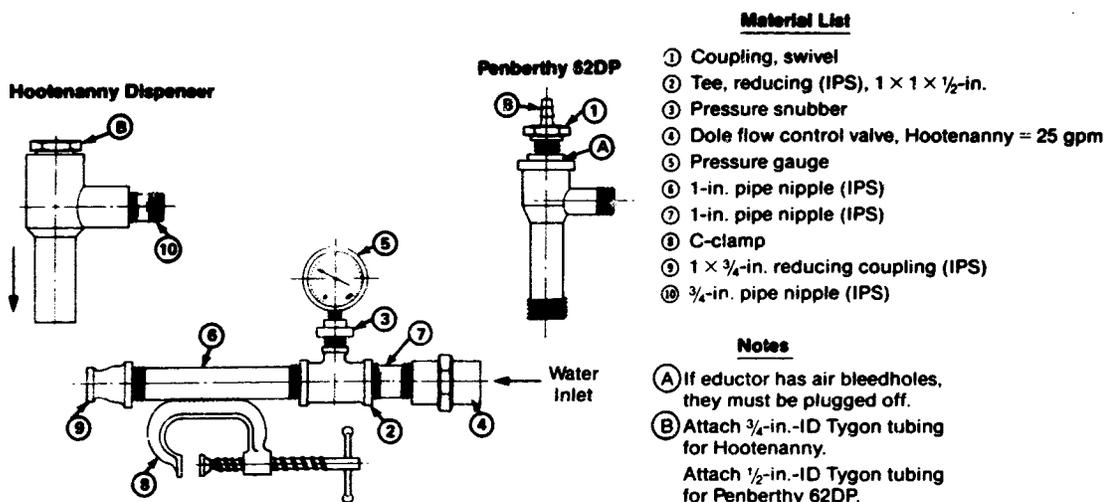
The most common and trouble-free mixing devices are the Hootenanny⁽¹⁾ and the Penberthy 62DP.⁽²⁾ Either of these units is connected to an adapter consisting of a C-clamp, flow control valve, pressure gauge, and suitable pipe reducers. A length of vacuum hose connected to the top of the eductor can be placed directly in a bag to remove the polymer by suction. This eliminates the dust that is sometimes caused when polymer is poured from one container to another.

Polymer mixing devices that employ a funnel to feed the powder to the water eductor are considered less suitable for handling Reten than the suction-feed types described above. However, if a funnel-feed unit is desired, it can be obtained from your Hercules representative. The Penberthy and Hootenanny eductors can also be fitted with a funnel instead of a suction hose.

(1) C. E. Hooten Company, Miami, FL.

(2) Penberthy Division of Houdaille Industries, Inc., Prophetstown, IL.

Figure 3
Hootenanny and Penberthy 62DP Polymer Mixing Devices



Material List

- ① Coupling, swivel
- ② Tee, reducing (IPS), 1 × 1 × 1/2-in.
- ③ Pressure snubber
- ④ Dole flow control valve, Hootenanny = 25 gpm
- ⑤ Pressure gauge
- ⑥ 1-in. pipe nipple (IPS)
- ⑦ 1-in. pipe nipple (IPS)
- ⑧ C-clamp
- ⑨ 1 × 3/4-in. reducing coupling (IPS)
- ⑩ 3/4-in. pipe nipple (IPS)

Notes

- (A) If eductor has air bleedholes, they must be plugged off.
- (B) Attach 3/4-in.-ID Tygon tubing for Hootenanny.
Attach 1/2-in.-ID Tygon tubing for Penberthy 62DP.

Makeup Procedure

Calculate the quantity of Reten polymer to be used and weigh it, or adjust the scale to re-balance when the proper weight of polymer has been removed from the bag. An initial solution concentration of 0.4 to 0.5% solids by weight is recommended; 0.3 to 0.6% solids is considered acceptable, however. If tank size permits, it is better to use a full bag for each batch to eliminate moisture absorption and contamination of the material held over in an already-opened bag.

Check the preparation tank and rinse it free of dirt. Add enough clean water to cover the lower agitator blade. Turn on the agitator and start the water through the eductor.

Water pressure through the Penberthy eductor should be regulated at 20 to 40 psi. This will give a waterflow rate of approximately 16 gallons per minute and a Reten polymer feed rate of about 6 pounds per minute. The Hootenanny is a slightly larger unit and can be operated at a flow rate of up to 20 gallons per minute to give a somewhat high feed rate for the Reten. The feed rate of Reten is influenced by the length of the hose and the amount of lift required to get the powder to the eductor. Excessive water pressure should be avoided, as this can result in splashback and subsequent plugging of the eductor by wet powder.

Begin to feed Reten polymer through the suction hose and watch that dry polymer flow is not interrupted before the full amount is added. Polymer addition will be complete before significant solution viscosity develops.

Turn off the waterflow through the eductor when polymer addition is complete. Continue adding water from the direct line until the full batch volume is reached. Stir the solution for 30 to 60 minutes until all particles dissolve and a smooth, clear solution is obtained.

Shut off the agitator and transfer the solution from the preparation tank to a storage tank when space is available. After solution preparation is complete, no further agitation is required.

Final Dilution

If the stock solution is to be diluted further, use an in-line static mixer, a mixing eductor, or a series of street elbows in the piping to achieve good mixing. To ensure proper mixing, there must be at least 100 pipe diameters of distance between the dilution water addition and the point of use.

Equipment

Reten polymer solutions are no more corrosive than the water used in their preparation, so standard materials of construction can be used. Suitable materials for tanks include fiberglass-reinforced polyester (FRP), epoxy resin, polyethylene resin, rigid PVC resin, or stainless steel. Mild steel is acceptable if it has been coated with a suitable rust-resistant paint. Suitable piping components can be selected from FRP, rigid PVC, ABS, or polybutylene. In areas where rusting is not likely to occur, black or galvanized iron piping is acceptable.

Size the preparation tank to supply at least 2 hours' needs. A storage tank should have at least 150% of the preparation tank's capacity. If a system is designed to both mix and meter from the same tank within a group of tanks, choose a tank with a capacity that allows enough time for preparation and mixing of each batch before it is again put on-line for use.

A tank large enough to use a full 50-pound bag of Reten polymer for each batch reduces product spillage, contamination, and moisture pickup in the partially used bag. A working capacity of 1,200 gallons is needed to prepare a 0.5% solution from a 50-pound bag. To allow sufficient freeboard for mixing, a tank's working capacity is usually 5 to 10% less than the actual tank capacity.

Follow the recommendations of your Hercules representative in selecting the correct sizes of agitator and pumps for your system. The agitator must be large enough to draw a good vortex in a half-filled tank, yet not overload as solution viscosity increases during makeup.

Use a low-shear centrifugal pump or gravity flow to transfer solution from the preparation tank to the storage tank. Use a positive-displacement, variable-speed gear or piston pump to meter solution from the storage tank to the process. To reduce degradation in the polymer, avoid excessive shear in the agitator and pumps.

Install a 10-mesh screen at the inlet to the storage tank to collect any foreign material or undissolved polymer. If completely gelfree solutions are needed, use in-line 60-mesh cartridge filters after the polymer has been diluted with water.

A platform scale, hook scale, or similar weighing device will be needed to weigh the polymer before and during batch preparation. A scale is not necessary if full bags are used for each batch.

SOLUTION PREPARATION IN THE LABORATORY

Small quantities of Reten polymer solution can be prepared by any of the procedures described on page 2 for dispersing the polymer without an eductor.

Solutions to be stored should contain at least 0.5% Reten solids, and the storage time is extended if they are stored cold. If solutions below 0.2% solids are to be used, they should be prepared fresh or from stock solution on a daily basis. The precautions already described regarding water purity, pH, temperature, preservatives, and avoidance of high-shear agitation should be observed.

When the dry polymer is to be added directly to stirred water, lumping can be avoided by slowly flowing the powder into the shoulder of the vortex. Continue stirring at moderate speed until a smooth, gelfree solution is obtained. If the solution is not complete in 30 to 60 minutes, allow it to stand for an hour, then stir it again. Lumping can be avoided if the Reten powder can be slurried in a nonsolvent such as alcohol before it is added to the stirred water.

In some cases, it may be possible to dry-blend the Reten with inert carrier materials before solution preparation. This assists particle dispersion, particularly if the Reten is less than 20% of the weight of the dry blend. Reten powders are hygroscopic and should be kept in closed containers to maintain their good dry-flow properties.

PRODUCT HANDLING AND DISPOSAL

Solutions prepared from Reten polymer are very slippery. Care must be taken to prevent spills or leaks around the solution preparation equipment.

If dry Reten powder is spilled, promptly sweep it up and dispose of it. If the powder becomes wet, it will become very slippery. Use an absorbent and sweep up the residue for disposal.

PRODUCT SAFETY

Reten polymers have a low order of toxicity and are not primary skin irritants or sensitizers. They can be handled with the normal precautions used for most industrial chemicals. A Material Safety Data Sheet should be obtained before using any Reten polymer product. Contact the appropriate sales office listed at the end of this bulletin.

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Water-Soluble Polymers
Worldwide Business Center
910 Market Street
Wilmington, DE 19899

Technical Information

BULLETIN ORC-201G
(Supersedes ORC-201F)

DI-CUP® Dicumyl Peroxide Vulcanizing Agent and Polymerization Catalyst

DI-CUP® dicumyl peroxide is used as a high-temperature catalyst in the rubber and plastics industries. Compounds containing Di-Cup are normally processed at temperatures up to 250°F (121°C) and can be cured at temperatures above 300°F (149°C).

Grades

Di-Cup is available in several commercial forms to suit its varying uses. Table I lists these grades and gives their purity, composition, and specific gravity. The molecular weight of dicumyl peroxide is 270; its structural formula is shown below.

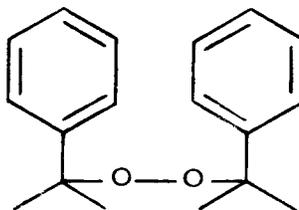


Table I
Commercially Available Grades of Di-Cup

Grade	Purity and Composition	Specific Gravity at 25/25° C
Di-Cup T	94-97% active dicumyl peroxide	1.02
Di-Cup R	98-100% active dicumyl peroxide	1.02
Di-Cup 40C	39.5-41.5% active dicumyl peroxide supported on precipitated calcium carbonate	1.57 (calculated)
Di-Cup 40KE	39.5-41.5% active dicumyl peroxide supported on Burgess KE clay	1.55 (calculated)

We cannot anticipate all conditions under which this information and our products, or the products of other manufacturers in combination with our products may be used. We accept no responsibility for results obtained by the application of this information or the safety and suitability of our products, either alone or in combination with other products. Users are advised to make their own tests to determine the safety and suitability of each such product or product combination for their own purposes. Unless otherwise agreed in writing, we sell the products without warranty, and buyers and users assume all responsibility and liability for loss or damage arising from the handling and use of our products, whether used alone or in combination with other products.

Di-Cup T, a pale yellow, fused, semicrystalline solid, slowly melts at temperatures around 85°F (30°C). Di-Cup R, a pale yellow to white granular solid, slowly melts at 100°F (38°C). Di-Cup 40C and Di-Cup 40KE are free-flowing off-white powders under normal storage conditions. Tests have shown that these materials do not lump or cake below 100°F (38°C).

Solubility

Di-Cup at practical use concentrations is soluble in a variety of organic compounds, as shown in Table II. In addition, Di-Cup is soluble or disperses readily in natural and synthetic rubber compounds, silicone gums, and polyester resins. It is soluble in vegetable oils and insoluble in water.

Table II
Solvents for Di-Cup T and Di-Cup R

Solvent	Wt % of Di-Cup in a Saturated Solution	
	At Room Temperature, 70-80° F (21-27° C)	At Freezing Temperature, 25-35° F (-4 to 2° C)
Cyclohexane	80	70
Xylene	70	50
<i>alpha</i> -Methylstyrene	70	45
<i>tert</i> -Butylbenzene	65	<60
Isopropyl ether	80	<75
Dioxane	75	<70
Amyl alcohol	<60	<60
Diacetone alcohol	<70	<60
Cumene hydroperoxide	60	<50
Carbon tetrachloride	<75	<75
Trichloroethylene	<75	<75
Diocetyl phthalate	55-60	<10

Use of Di-Cup

Di-Cup can be used to cross-link a wide variety of polymers. In developing a new formulation using Di-Cup, the following basic compounds can be used as a starting point:

	<u>Parts by Weight</u>
Polymer	100
Filler	Variable
Zinc oxide	5.0
Antioxidant	0.5
Di-Cup 40KE	(a)

(a) EPDM, EPM, CSM, phr	6.0-8.0
VMQ, BR, phr	1.25-2.5
All other materials, phr	2.5-5.0

Typical results using this basic formulation are shown in Table II. Equivalent level (active ingredient basis) of the various forms of Di-Cup are listed below:

- 1.0 part Di-Cup R = 1.08 parts Di-Cup T
- 1.0 part Di-Cup R = 2.4 parts Di-Cup 40C and 40KE
- 1.0 part Di-Cup T = 0.92 part Di-Cup R
- 1.0 part Di-Cup T = 2.26 parts Di-Cup 40C and 40KE
- 1.0 part Di-Cup 40C and 40KE = 0.41 part Di-Cup R
- 1.0 part Di-Cup 40C and 40KE = 0.44 part Di-Cup T

Table III
Basic Formulations and Properties
for Typical Rubbers Cured With Di-Cup

	EPM ^(a)	EPDM ^(b)	NR ^(c)	CR ^(d)	NBR ^(e)	SBR ^(f)	EU ^(g)
Polymer	100.0	100.0	100.0	100.0	100.0	100.0	100.0
HAF Black	50.0	50.0	50.0	30.0	30.0	50.0	30.0
Agerite Resin D ^(h)	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Zinc oxide	5.0	5.0	5.0	5.0	5.0	5.0	5.0
Di-Cup 40KE	7.2	6.8	4.5	3.5	4.5	3.5	4.5
Cure time, min	30	30	20	10	25	20	15
Cure temperature, °F (°C)	← 330 (166) →						
Tensile strength, psi (MPa)	1,200 (8.2)	2,430 (16.7)	2,700 (18.6)	2,785 (19.2)	3,100 (21.3)	2,810 (19.3)	3,240 (22.3)
100% modulus, psi (MPa)	150 (1.0)	380 (2.6)	460 (3.1)	380 (2.6)	335 (2.3)	1,050 (7.2)	860 (5.9)
200% modulus, psi (MPa)	320 (2.2)	1,430 (9.8)	1,525 (10.5)	—	1,120 (7.7)	—	2,675 (18.4)
Elongation, %	500	280	275	190	345	185	230
Shore A hardness, points	53	68	68	71	62	73	70

(a) Vistalon 404 (Exxon Chemical).
 (b) Nordel 1040 (Du Pont).
 (c) No. 1 smoked sheet.
 (d) Neoprene W.
 (e) Hycar 1032 (B. F. Goodrich).
 (f) SBR 1500.
 (g) Adiprene C (Du Pont).
 (h) R. T. Vanderbilt Company, Inc.

Cure Time and Temperature

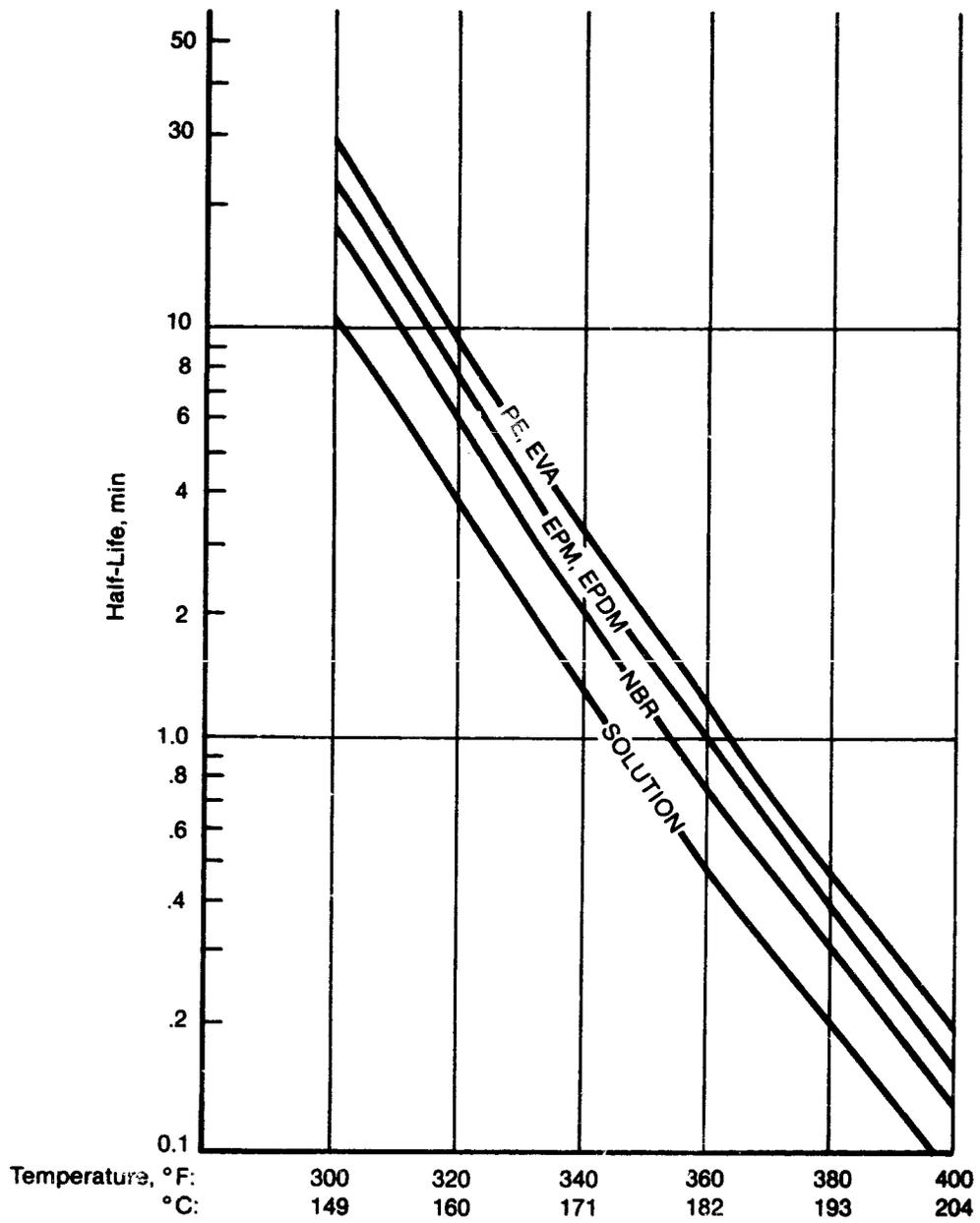
Di-Cup decomposes thermally when heated to form peroxy radicals that, in turn, abstract hydrogen from the polymer backbone, forming polymer radicals. A combination of two of these polymer radicals results in a cross-link. In general, the cure rate (or rate of cross-linking) is equivalent to the rate of Di-Cup peroxide thermal decomposition. The rate of Di-Cup cure, therefore, is primarily dependent on cure temperature and is predictable for each polymer system. Care should be exercised to differentiate between rate of cure and state of cure. In a given polymer, rate of cure with Di-Cup is affected primarily by temperature, while state of cure is influenced by the level of Di-Cup and many other factors.

Simplistically stated, the only factor affecting the rate of peroxide thermal decomposition and, therefore, cure rate, is temperature; however, the polymer or medium in which the peroxide decomposes has a pronounced effect on rate of peroxide decomposition. Di-Cup, although much less sensitive to its environment than many other peroxides, will require a different cure time at a preset temperature in each different polymer system. Selection of the proper cure time for a vulcanizate based on Di-Cup depends on performance requirements of that vulcanizate.

Figure 1 is a plot of the cross-linking half-life of Di-Cup in various polymer compounds. In addition to the polymers shown, *cis*-polybutadiene (BR) has a half-life curve between those of nitrile rubber (NBR) and ethylene-propylene terpolymer (EPDM); polyisoprene (IR), natural rubber (NR), and styrene-butadiene rubber (SBR) have approximately identical half-life curves, and this common curve lies between the NBR curve and the solution curve.

Under commercial curing conditions, the stock temperature and peroxide decomposition rate are influenced by mold heat-up time, vulcanizate thickness and shape, and other practical factors. Therefore, optimum factory cure conditions require experimentation. This is best accomplished by test-curing the compounds in production equipment for the cure times calculated from the half-lives shown in Figure 1. Resulting vulcanizates are then tested for either modulus, compression set, or unreacted peroxide. Plotting any one of these against cure time will result in a curve from which cure time to reach the desired state can be read. Cure conditions developed in this manner will assure optimum performance with the peroxide-cured vulcanizate. A laboratory evaluation will optimize laboratory procedure, but will serve only as a guide to production practice.

Figure 1
Cross-Linking Half-Life of DI-Cup vs Temperature



Odor of Cured Stocks

Nearly all compounds that have been freshly cured with Di-Cup have an odor, caused by acetophenone, a normal byproduct of dicumyl peroxide decomposition. The odor is not unpleasant, but is different from the odor associated with other methods of vulcanization. There is no known way to prevent formation of acetophenone. If odor-free stocks are required, either heat treatment or masking can be employed. A mixture of equal parts of Alamask 137B and 520A (Florasynth Corporation) substantially reduces acetophenone odor in vulcanizates cured with Di-Cup, replacing it with a mild, perfumelike odor. Addition levels from 0.05 to 0.5 phr should be investigated for each system where odor improvement is sought. An equal-parts mixture, however, need not necessarily be used.

Product Safety

This product may be considered hazardous under the U.S. Occupational Safety and Health Act of 1970. A Material Safety Data Sheet should be obtained prior to use of this product, and user's employees informed of its contents. User should also obtain and review Hercules Di-Cup Bulletins ORC-202 — Safety Aspects of Storing and Handling 55-Gallon Shipping Containers; ORC-203 — Summary of Hazard Tests; and ORC-204 — Summary of Toxicological Investigations.

Procedure for Emulsifying Di-Cup

For applications involving aqueous systems (e.g., curing latexes), the following procedure is satisfactory for emulsifying Di-Cup in the laboratory.

<u>Component</u>	<u>Parts by Weight</u>
Di-Cup T or R	100
Toluene	35
Oleic acid	5.6
Water	101
30% KOH solution	2.62

Melt the Di-Cup in a suitable container in a warm-water bath (40 to 50°C). Add the toluene, mix, then add the oleic acid. Pour this solution into a glass Waring blender containing the water (warmed to 40 to 50°C) and the KOH solution. Mix for 2 min at high speed.

When cooled to room temperature, an emulsion of Di-Cup prepared in this manner is stable for several weeks. Slight shaking will redisperse any settling. If toluene is objectionable, a less stable emulsion can be prepared by omitting it. However, if toluene is omitted, the emulsion should be used directly following preparation and should not be allowed to cool below 45°C.



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Technical Information

BULLETIN ORC-202C
(Supersedes ORC-202B)

DI-CUP® Dicumyl Peroxide **Safety Aspects of Storing and Handling** **55-Gallon Shipping Containers**

DI-CUP® dicumyl peroxide is used as a high-temperature catalyst in the rubber and plastics industry. It is available in several commercial grades to suit its varying uses. The use of Di-Cup stems from its ability to decompose and form free radicals, which, in turn, promote cross-linking. Because this decomposition occurs at an ever-increasing rate as the temperature increases, certain precautions are necessary to ensure the safe storage and handling of Di-Cup. This bulletin summarizes Hercules' recommendations for the safe storage and handling of 55-gallon shipping containers.

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Handling Di-Cup R

As manufactured and shipped, DI-CUP R is a white to pale yellow, granular, crystalline material at normal temperatures. Over long periods of storage, Di-Cup R tends to agglomerate, or cake, and must be removed from the open-head shipping containers in lumps. These lumps pose no mixing problems because they are readily reduced to smaller particles during the compounding operations.

Di-Cup R is a low-melting solid that is readily reduced to an oily liquid state by applying a small amount of heat. In this form it can be conveniently handled. Safe equipment designed especially for melting Di-Cup R, operation of that equipment, and suggestions for control are detailed on page 8.

Handling Di-Cup T

As manufactured and shipped, DI-CUP T is a pale yellow, oily, semicrystalline solid. Prolonged standing of Di-Cup T causes the oil phase to drain gradually from between the crystals and settle to the bottom of the container. While the oil phase is still predominantly dicumyl peroxide, it contains less peroxide than the crystals. The product should be thoroughly remixed, therefore, before using. Simply warm the Di-Cup T in its container and stir until the oil phase and melted crystals are a uniform liquid product. Details on a simple and safe method for accomplishing this are given on page 3. When the Di-Cup T is a uniformly mixed liquid, it can be metered directly into the process, or standard charges can be withdrawn into smaller containers and stored until needed.

Physical Properties

Physical properties of Di-Cup T and R that should be considered in devising safe and effective means of handling these products are given below.

<u>Physical Properties</u>	<u>Di-Cup T</u>	<u>Di-Cup R</u>
Melting point, °C	30-38	38
Heat of fusion, cal/g	18 ± 2	18 ± 2
Specific heat, solid	0.35	0.35
Liquid	0.42	0.42
Specific gravity at 50/50°C	1.007	1.014
Viscosity, cps, ^(a) at 40°C	12.5	19.1
At 50°C	10.3	10.6
Flashpoint, SETA-CC, °F (°C)	170 (77)	260 (127)

^(a)Brookfield viscometer, Model LVF, Spindle No. 1, at 60 rpm.

Melting Di-Cup

Di-Cup T or R can be safely handled with a minimum of precaution at ordinary temperatures and within a limited temperature range in which it is a liquid. The basis for its commercial utilization, however, requires that it decompose at elevated temperatures and at an ever-increasing rate as the temperature increases. Because of this inherent characteristic, certain precautions regarding temperature control should be observed in melting Di-Cup.

- SAFE:** At 86 to 131°F (30 to 55°C), Di-Cup T or R can be held for several days without significant decomposition.
- DANGEROUS:** The handling temperature should *never* exceed 131°F (55°C). Above this, the rate of thermal decomposition and heat evolution increases rapidly. At about 244°F (118°C), rapid exothermic decomposition takes place, as evidenced by flammable white smoke.
- EMERGENCY ACTION:** Should the temperature reach 176°F (80°C), the drums of Di-Cup should be deluged with water. This will stop the high rate of decomposition and cool the peroxide. Cooling should continue until the temperature of the Di-Cup is 131°F (55°C) or lower.

The only method Hercules recommends for melting Di-Cup is a hot water system with a temperature that is automatically kept at 122 to 131°F (50 to 55°C). When properly designed, such a system minimizes the chance of overheating. A simple, economical system often used for melting drums of Di-Cup involves placing the drums in a water bath. Details of a typical water bath system are given below. *Electric drum heaters, direct steam, and hotplates should not be used for melting Di-Cup*, since localized heating may cause hot spots and eventual decomposition of the peroxide. Ovens and hot rooms are other sources of heat that are not considered safe.

Melt Bath System

Figure 1 (at the end of this bulletin) is a schematic of a typical melt bath system, including necessary instrumentation and auxiliary equipment. Figures 2 and 3 show details of a water bath and drum hoist used by Hercules in melting Di-Cup. The bath is constructed of ¼-in. (6.35-mm) boiler plate, and is divided into three compartments, with space underneath the divisions to allow water circulation (accomplished by an agitator). The water is heated by injection of steam through an automatic control valve and sparge pipe.

Three to 6 hours at 122 to 131°F (50 to 55°C) is required to completely melt a drum of Di-Cup T; 6 to 12 hours is required for Di-Cup R. After these periods of time, convection currents within the drum will normally have caused the contents to become uniformly mixed.

Following are important safety precautions for the melt bath system:

- The melt bath for Di-Cup should be located away from metal catalysts, reducing agents, and strong mineral acids to prevent contamination.
- Drums of Di-Cup should be vented before the Di-Cup is melted. This can be done by removing or loosening the drum cover or opening the bung.
- The melt bath and pumping facility for Di-Cup (if provided) should be located in an area protected by an automatic sprinkler system.
- A manually activated water spray deluge system should be provided directly above the melt bath. Should the bath temperature reach 176°F (80°C), this system should be activated. The activation valve should be located in an area remote from the melt bath.

- A temperature controller alarm should be provided to maintain the bath water temperature at 122 to 131°F (50 to 55°C). The controller should be set to alarm at 133°F (56°C) and 176°F (80°C). An audible alarm should be provided and located where area personnel can hear it and take corrective action.
- A fusible plug with a melt temperature of 136°F (58°C) should be provided in the drainpipe serving the melt bath. Should the bath temperature exceed 136°F, the alloy in the plug will melt, allowing water to drain from the bath. These plugs are available from Hercules on request.
- Water in the melt bath should be agitated to aid in heat transfer. A small, portable air-driven agitator will serve this purpose.
- Any electrical motors, switches, wiring, etc., located at the melt bath should be rated for use in a Class I, Group D, Division 2 electrical classification as defined in the National Electrical Code, Articles 500 to 503.

Pumping Molten Di-Cup

If a pump is used to transfer warm, liquid Di-Cup from a drum to the process, Hercules recommends the following precautions:

- For cleanliness, drums of Di-Cup should be lifted out of the melter prior to being emptied. During emptying, care must be taken to keep foreign materials out of the open drums. If Di-Cup is removed from the drum while it is still in the water bath, restraining bars should be attached to the drum to counteract its buoyancy as the contents decrease. This will also minimize spillage of Di-Cup and contamination of the hot water.
- The pump should have a mechanical seal rather than a stuffing box. Duraseal mechanical seal, manufactured by Durametall Corporation, Kalamazoo, Michigan, is recommended for its performance and flexibility of maintenance, possible because of interchangeability of its parts. The mechanical seal should not contain copper, brass, or natural or synthetic rubber. Copper and brass promote decomposition of Di-Cup and natural and synthetic rubber become brittle through extended contact with Di-Cup and will not last long. The preferred material is 316 stainless steel. Teflon O-rings and gasketing material are also recommended.
- Pumps used for Di-Cup should be self-priming, and either rotary gear or centrifugal type. Wetted parts should be of 316 stainless steel or Teflon; O-rings and gasketing should be of Teflon. Pumps can be either pneumatically or electrically driven.

Hercules uses two different pumps made by Eco Engineering Company, Newark, New Jersey, for handling molten Di-Cup. *Model PP-2M*, 316 stainless steel with Teflon propellers, is a self-priming rotary pump with Gast air-motor drive and a capacity of about 3 gallons (11.4 liters) per minute. *Model PTA-8*, of 316 stainless steel, is a pedestal-mounted Centri-Chem centrifugal pump with Gast air-motor drive and a capacity of about 3 gallons (11.4 liters) per minute. A high-temperature cutout on the pump is recommended, set to open at 140°F (60°C).

If electrically driven pumps are used, the motor switches, wiring, etc., located at the pumping site should have a Class I, Group D, Division 2 electrical classification.

- All rigid piping handling molten Di-Cup should be of 304 stainless steel construction with Teflon gasketing or pipe dope.
- Provisions should be made for blowing out all lines carrying Di-Cup with nitrogen, carbon dioxide, or other inert gas.
- The pump suction hose should be of flexible stainless steel.

- The pump and piping should be hot-water-traced (not steam-traced) and insulated to maintain a temperature of 104 to 113°F (40 to 45°C) to prevent crystallization of the Di-Cup. A special temperature-limited electrical tracing system known as Chemelex Auto-Trace is an acceptable alternative to hot-water tracing. This system is manufactured by Raychem Corporation, 837 Second Avenue, Redwood City, California 94063. It must be specified as factory-mutual-approved for use in a Class I, Group D, Division 2 electrical classified area. In addition, the Chemelex system must be engineered so that the temperature of the Di-Cup will not exceed 122°F (50°C).
- A nonwicking type of insulation such as foam-glass is recommended for use in all equipment and piping handling molten Di-Cup.

Storage Requirements

When stored at room temperatures of 75 to 79°F (24 to 26°C), Di-Cup T and R dicumyl peroxide are very stable chemically and can be held for several years without any appreciable loss of peroxide activity. Uncontrollable thermal decomposition of uncontaminated Di-Cup T or R should not occur below 200°F (93°C). Decomposition to any significant degree would not be expected at temperatures below 131°F (55°C); however, since temperatures of about 131°F (55°C) are not uncommon in unventilated storage areas in the summer, or under direct sunlight, Di-Cup T and R should be stored in ventilated areas, in the dark or under cover, and at as low a temperature as practical. Isolation from other chemicals is strongly recommended.

Safety Precautions for Di-Cup Dicumyl Peroxide Storage Area

The following safety precautions should be followed regarding storage of Di-Cup dicumyl peroxide:

- Storage of Di-Cup should be separated from process areas, important buildings, and public neighboring localities, so that in case of fire these other areas will not be jeopardized.
- The storage area should be under cover and protected from direct sunlight. A shed with walls on the sides exposed to the sun would provide this protection.
- The storage area must be sprinkler-protected.
- At least one fire hydrant with sufficient lengths of hose should be quickly available, located preferably not closer than 50 feet nor more than 150 feet from the storage area.
- No acids or other chemicals (such as Friedel-Crafts catalysts, reducing agents, and oxidation catalysts) may be stored in an area used for Di-Cup.
- The storage area should be isolated from the storage of flammables such as solvents.
- The storage area should be protected against access by unauthorized personnel.
- No smoking should be permitted in the storage area.
- Housekeeping in and around the storage area should prevent accumulation of easily combustible materials such as dry grass or paper.

Explosive Characteristics

1. Impact

No sign of detonation or burning occurred when a 2-kilogram weight was dropped 100 centimeters onto a 25-milligram sample of Di-Cup R placed between two pieces of flat, hardened steel; and no evidence of ignition or explosion was observed when a 4-kilogram weight was dropped 10 times from a height of 50 centimeters onto Di-Cup R placed on an anvil inclined 4°.

2. Compression

No sign of detonation was in evidence after a 10-gram sample of Di-Cup R was placed in a steel bomb and initiated with a No. 8 blasting cap.

3. Heat

No visible reaction occurred when a 25-milligram sample of Di-Cup R was inserted in a copper shell and heated to 175°C. At 325°C, the sample foamed, but did not ignite or detonate. When larger (50-gram) samples were heated at the rate of 1°C per minute, they began to undergo exothermic decomposition at 120°C. This exothermic decomposition reached a peak temperature of 180°C in 1 minute.

A sample of Di-Cup T was examined by quantitative differential thermal analysis (DTA) at a heating rate of 10°C per minute. Exothermic decomposition peaks at 178°C (352°F). Isothermal runs indicate that extensive decomposition takes place as low as 90°C (194°F).

A 50-gram sample of a 50% solution of Di-Cup R in an aromatic hydrocarbon was maintained at 50°C in loosely stoppered bottles. Chemical analysis revealed no decomposition after 50 days.

Burning Characteristics

The following report on burning properties of dicumyl peroxide was obtained from the Bureau of Explosives Laboratory in South Amboy, New Jersey.

"Di-Cup R or T can be ignited by contact with external flame. When so treated, it melts and burns quietly at first, then with the characteristic acceleration of organic peroxides. While this ignition or burning test was run on a comparatively small amount, the burning rate accelerated as the combustion progressed, and indicated the possibility of vigorous or perhaps violent combustion if a large quantity should become involved in fire."

A larger scale burning test was conducted by Hercules: A 5-gallon (18.9-liter), rubber-sealed, galvanized drum containing 40 pounds (18.2 kilograms) of Di-Cup T was exposed to heat and flames of a kindling fire. There was no violent ignition, nor did the Di-Cup T show any noticeable acceleration in the rate of burning. It burned at about the same rate as a similar volume of turpentine, and much less actively than a gasoline-type solvent or an aromatic hydrocarbon under similar conditions.

Major Hazards

Three major hazards are encountered in storing and handling Di-Cup T or R:

- Chemical decomposition caused by acid contamination
- Chemical decomposition catalyzed by contact with copper and copper alloys
- Thermal decomposition induced by excessive heat exposure (>131°F, 55°C)

Other chemical materials that promote rapid decomposition of Di-Cup are catalysts of the Friedel-Crafts type, reducing agents, and oxidation catalysts. Once this reaction has been initiated, it is highly exothermic and self-sustaining. Temperatures could conceivably reach a level at which the contents of a container would ignite.

When small samples of dicumyl peroxide were heated, thermal decomposition began at about 118°C. Taking into consideration the effect of volume on the relationship between heat and decomposition characteristics (see Burning Characteristics, page 6), thermal decomposition may start in larger amounts at a lower temperature. This thermal decomposition is also exothermic and could result in a fire.

As with any other chemical product of comparable flammability, fire-extinguishing facilities should be provided in any area where sizable quantities of Di-Cup are being handled or stored. Fires have been readily controlled with water as well as CO₂, dry chemical, and foam extinguishers.

It is strongly recommended that Di-Cup be isolated from all other chemicals during storage and use.

Waste Disposal

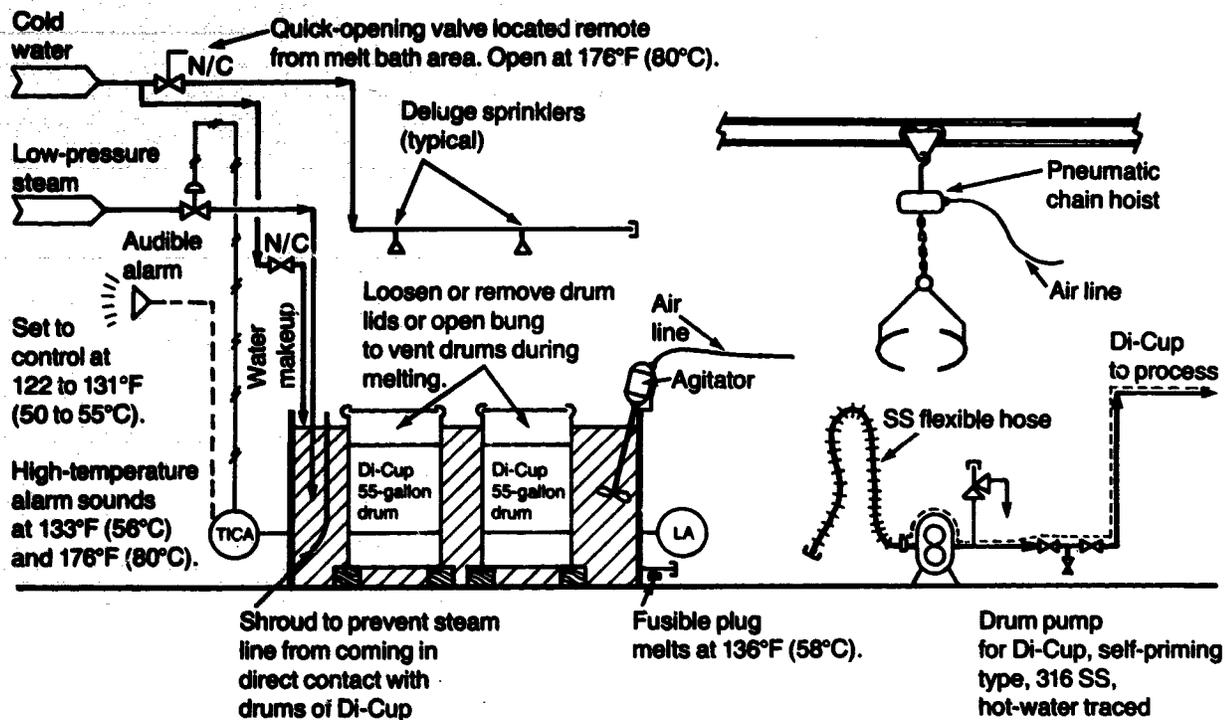
All waste Di-Cup, or materials containing appreciable quantities of it, should be disposed of in accordance with local, state, and Federal regulations.

CAUTION: Do not attempt to burn Di-Cup in containers. Do not mix concentrations or solutions of Di-Cup with other chemical wastes. Do not put solutions containing Di-Cup into sewer systems.

Product Safety

This product may be considered hazardous under the U.S. Occupational Safety and Health Act of 1970. A Material Safety Data Sheet should be obtained prior to use of this product. Toxicological data are available in Hercules Technical Data Bulletin ORC-204.

Figure 1
Melt Bath Schematic for Di-Cup



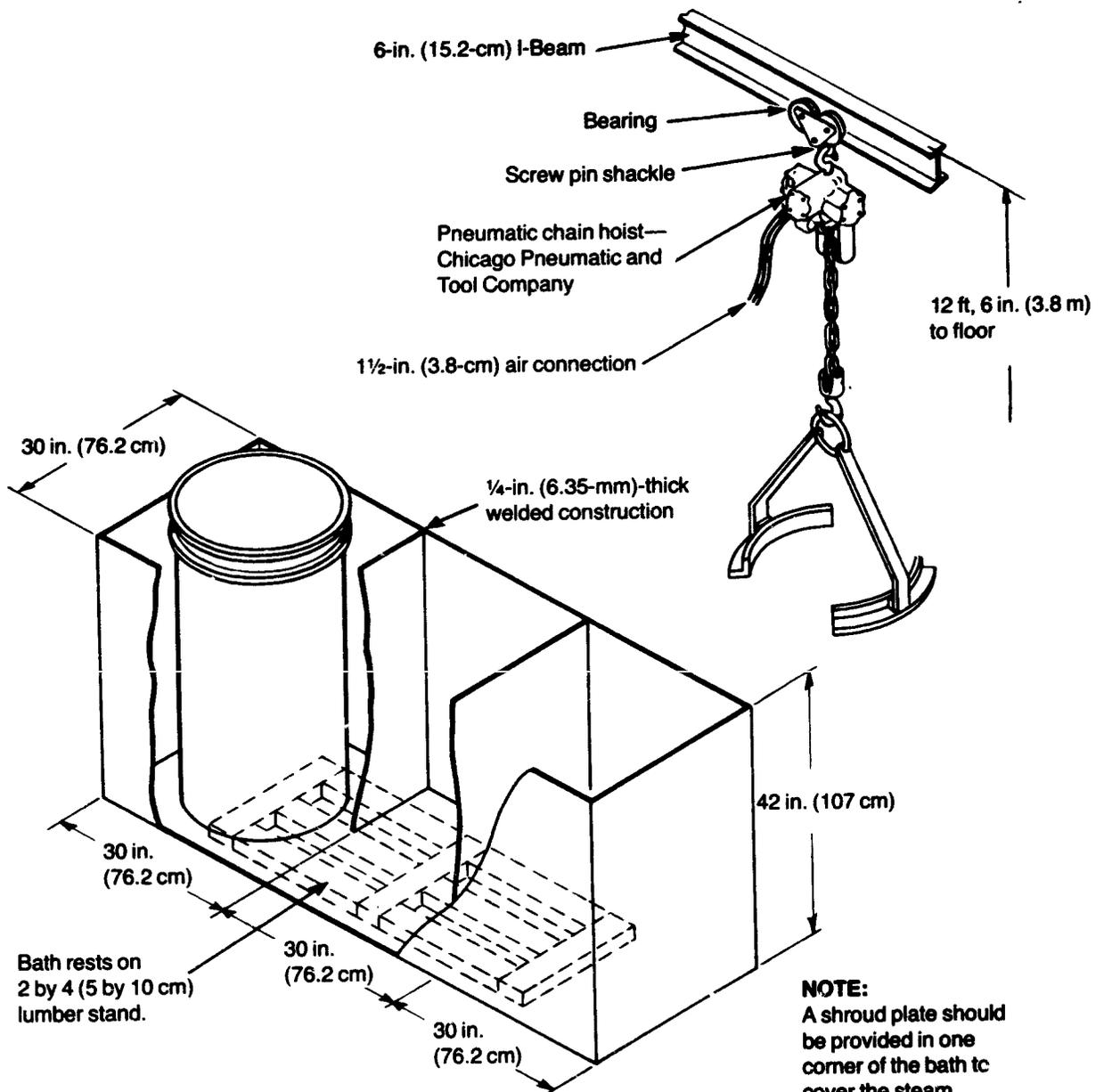
LEGEND

- TICA — Temperature-Indicating control alarm
- LA — Low-level alarm
- #—#— Pneumatic tubing
- — — — — Electrical wiring
- — — — — Heat-traced equipment (hot water or Chemetex temperature, limiting electrical tracing)
- N/C — Normally closed

NOTE

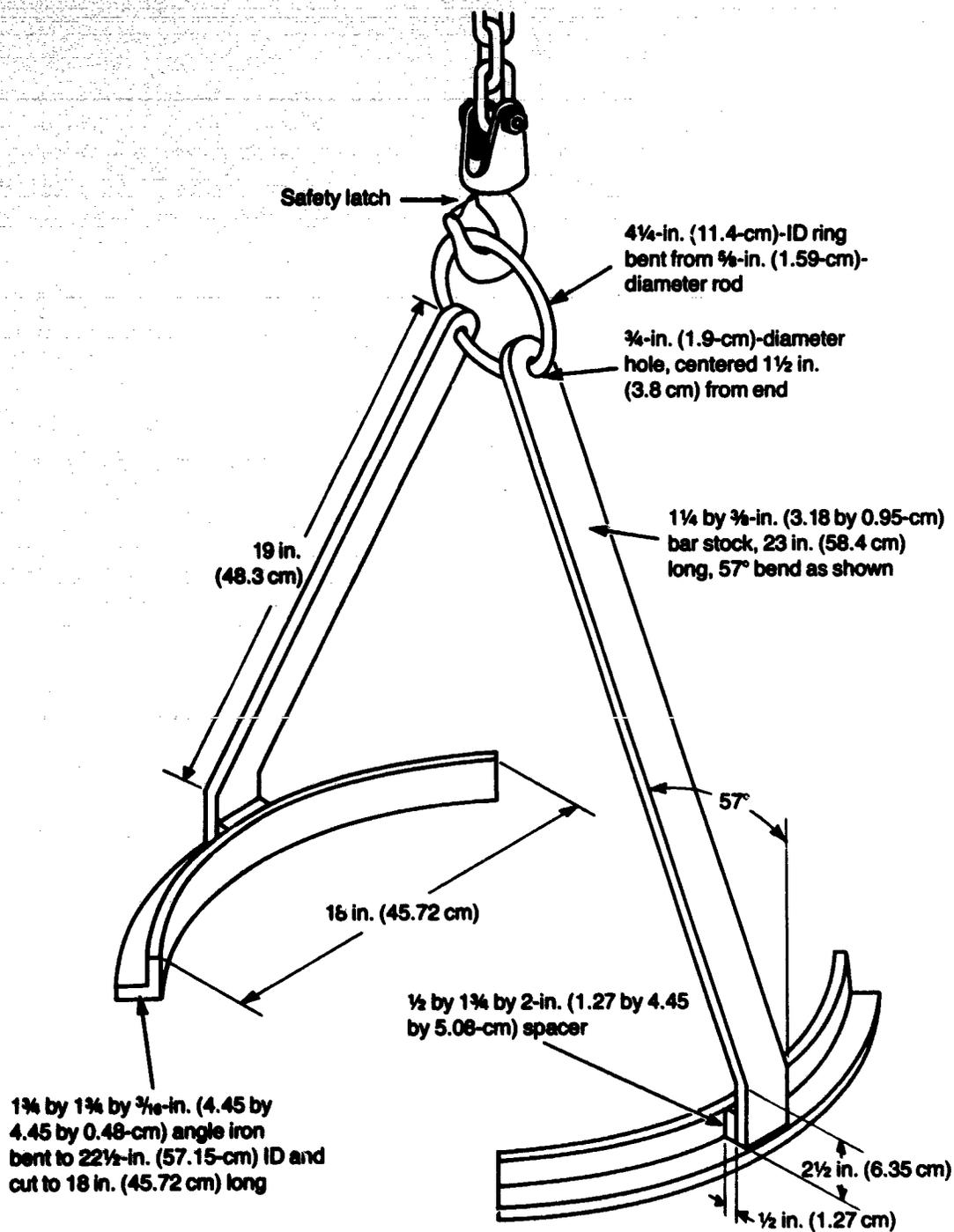
If Di-Cup is pumped from the drum while still in the melt bath, it is recommended that restraining bars be attached to the drum to counteract the buoyancy that will occur as the drum contents decrease. This will prevent spillage of Di-Cup and contamination of the hot water.

Figure 2
Drum Melting Bath



NOTE:
A shroud plate should
be provided in one
corner of the bath to
cover the steam
sparge line and prevent
the steam line from
making direct contact
with drums of Di-Cup.

Figure 3
Drum Holst Detail





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Technical Information

BULLETIN ORC-203D
(Supersedes ORC-203C)

DI-CUP® Dicumyl Peroxide **Summary of Hazard Tests**

DI-CUP® dicumyl peroxide, like other organic peroxides, should be clearly characterized in terms of potential hazard from:

- Chemical contamination
- Physical shock
- Heat and flame

This summary describes briefly the extent of Hercules' knowledge in each of these areas, and offers conclusions and recommendations on the safe handling of Di-Cup T (technical-grade) and Di-Cup R (recrystallized-grade) peroxide.

CHEMICAL CONTAMINATION

Properly handled, Di-Cup can be safely mixed with many organic and inorganic chemicals. It is readily soluble in a broad spectrum of organic solvents and has about the same stability to heat in solution as it does in its pure form. There are some classes of chemicals, however, that must not be mixed with Di-Cup, either accidentally or purposely.

Di-Cup decomposes vigorously when in contact with mineral acids (or acid-forming salts), oxidation catalysts, reducing agents, and catalysts of the Friedel-Crafts type. This reaction, once initiated, is highly exothermic and may become self-sustaining. Temperatures can reach a level at which violent expansion or even autoignition may occur.

Contamination of Di-Cup with such products in storage, during use, or in waste disposal must be avoided. Isolated storage is strongly recommended, and Di-Cup should be handled in clean containers only. Lines through which liquid Di-Cup is being pumped should have no connections to lines from containers of other chemicals. Spillage or leakage should not be allowed to flow into common drains. In short, care must be exercised that Di-Cup is not mixed with chemicals that can induce ionic cleavage of peroxides.

PHYSICAL SHOCK

Unlike the great majority of organic peroxides, Di-Cup is not sensitive to physical shock. Attempts to induce detonation of dicumyl peroxide have been completely unsuccessful under even the most severe conditions.

We cannot anticipate all conditions under which this information and our products, or the products of other manufacturers in combination with our products, may be used. We accept no responsibility for results obtained by the application of this information or the safety and suitability of our products, either alone or in combination with other products. Users are advised to make their own tests to determine the safety and suitability of each such product or product combination for their own purposes. Unless otherwise agreed in writing, we sell the products without warranty, and buyers and users assume all responsibility and liability for loss or damage arising from the handling and use of our products, whether used alone or in combination with other products.

Impact Tests

A 25-mg portion of Di-Cup R (98 to 100% crystalline dicumyl peroxide) was placed between plates of flat, hardened steel and struck by a 2-kg weight falling 100 cm. There was no sign of detonation or burning.

A 4-kg weight was dropped from a height of 50 cm onto a portion of Di-Cup R placed on an anvil inclined at an angle of 4°. This test was repeated 10 times. There was no evidence of ignition or explosion.

Compression Tests

A 10-g sample of Di-Cup R was placed in a steel bomb and initiated with a No. 8 cap. There was no evidence of any detonation.

To resolve the question of the possibility of shock detonation of Di-Cup, two experiments were performed under as severe conditions as could be developed. These are briefly described as follows:

A 5-in. Schedule 40 steel pipe, 28 in. (71.1 cm) long, was filled with approximately 18 lbs (8.2 kg) of Di-Cup T. A Titan™ 500 booster consisting of 475 g of a 50:50 mixture of TNT:PETN and a No. 8 electric blasting cap were inserted in the pipe in intimate contact with the Di-Cup. Both ends of this pipe were capped, the pipe was lowered into 4 to 6 ft (1.2 to 1.8 m) of water at 65°F (18.3°C), and the booster was detonated.

The result was only a small boil in the water, followed by a black, oily film rising to the surface. The pressure generated and the rate recorded [4,350 ft/sec (1,326 m/sec)] were about as would be expected from the booster alone.

In order to observe remnants of such a test, a smaller charge was detonated under sand. A 3-in. Schedule 40 steel pipe, 28 in. (71.1 cm) long, was similarly loaded with about 5.5 lbs (2.5 kg) of Di-Cup T, a Titan 500 booster, and a No. 8 cap. Both ends of the pipe were capped, the assembly was buried in about 18 in. (45.7 cm) of wet sand, and the booster was detonated.

A convincing failure to propagate resulted. The pipe was completely destroyed around the booster area, and ripped longitudinally for about 16 in. (40.6 cm), which is not unusual when a Titan 500 booster is used. At the end of the pipe opposite the booster, there was a black, oily residue and about 4 in. (10.2 cm) of unaffected Di-Cup.

These tests showed that Di-Cup dicumyl peroxide is very insensitive to shock. No detonation could be developed, even with heavy confinement and severe priming. [It is estimated that the Titan 500 booster develops temperatures over 4,400°K, pressures above 2 million psi (13,793 MPa), and a detonation velocity of about 24,000 ft/sec (7,317 m/sec). This represents a total energy content of about $1,740 \times 10^3$ ft-lbs (240.6×10^3 kg-m)].

EXPOSURE TO HEAT AND FLAME

Experiments on the effect of heat on Di-Cup may be divided into tests of:

- Exposure to heat
- Burning and extinguishing tests
- Exposure of standard containers to direct flame

Exposure to Heat

A 25-mg portion of Di-Cup R dicumyl peroxide was inserted in a copper shell and heated to 347°F (175°C). No visible reaction occurred. At 617°F (325°C) the Di-Cup foamed, but did not ignite or detonate. This behavior, however, could well be a function of the amount of material involved.

When 50 g of Di-Cup was heated at the rate of 1°C per min, exothermic decomposition began at 248°F (120°C) and reached a peak temperature of 356°F (180°C) in 1 min.

To subject the product to extremes of heat exposure, two large-scale heating tests were performed.

1. In the first test, approximately 100 lbs (45.5 kg) of Di-Cup T (90 to 93% dicumyl peroxide) was loaded into a container made from Schedule 40 pipe, 12 in. (30.5 cm) in diameter and 30 in. (76.2 cm) long. The wall thickness of the pipe was approximately 11/32 in. (0.84 cm). A 3/8-in. (0.95-cm) flat plate was welded to each end, and one end was fitted with a 2-in. (5.1-cm) nozzle welded into the end plate. The nozzle was capped with a 2-in. (9-cm) diameter rupture disc rated at 65 psi (0.44 MPa) at 72°F (22°C) and 53 psi (0.36 MPa) at 250°F (121°C). A thermocouple was inserted into the container and connected to a remotely located recorder. The container was mounted vertically about 15 in. (38 cm) above the ground and surrounded with piled wood soaked with kerosene. This soaked wood was ignited and observed from a distance.

The container was enveloped in flame almost immediately after ignition. After about 19 min of fire exposure, flames flickered momentarily from the end of the 2-in. nozzle, followed by complete rupture of the loosened blowout disc. Almost immediately, the bottom plate was blown off, accompanied by a jet of flame, a ball of fire, black smoke, and a rather loud report. When separation of the bottom plate occurred, the container was blown about 250 ft (76.2 m) into the air, landing about 100 ft (30.5 m) from the test site. Inspection of the test area indicated that the container ruptured because of overpressuring. Not all the contents were consumed in the reaction. The test vessel was essentially undamaged, except for removal of the bottom plate.

The temperature inside the test container rose gradually from 70 to 248°F (21 to 120°C) during flame exposure, and then soared to 482°F (250°C) almost instantaneously, at which point the thermocouple wires became separated from the test vessel. The temperature at which a violent exothermic decomposition occurred [248°F (120°C)] was the same as that previously noted in laboratory-scale tests.

2. A second test was carried out in exactly the same manner, except that the blowout disc was replaced by a threaded cap. The bottom plate was again blown off after 19 to 20 min of flame exposure. This reaction was perhaps slightly more severe, with less flame and less evidence of residual Di-Cup.

These tests showed that Di-Cup can withstand considerable exposure to heat, but once an internal temperature of 248°F (120°C) is reached, an exothermic reaction occurs that cannot be contained. The resulting expansion is not an explosion, since containers were recovered more or less undamaged, but it does cause rupture of the vessel and vigorous combustion. Similar rupture would be expected of any completely sealed vessel containing combustible matter on exposure to flame temperatures, regardless of the chemical nature of the contents.

With an organic peroxide such as Di-Cup, however, heat from external sources is supplemented by internal heat from exothermic decomposition of the peroxide, resulting in earlier rupture. This reaction is also evident in exposure of standard shipping containers to direct flame.

Burning Tests

Initial tests of the burning characteristics of Di-Cup dicumyl peroxide were carried out by the Bureau of Explosives Laboratory at South Amboy, New Jersey, with the following report:

"Di-Cup R can be ignited by contact with external flame. When so treated, it melts and burns quietly at first, then with the characteristic acceleration of organic peroxides. While this ignition or burning test was run on a comparatively small amount, the burning rate accelerated as the combustion progressed and indicated the possibility of vigorous, or perhaps violent, combustion if a large quantity should become involved in fire."

To study the burning characteristics of larger quantities of Di-Cup, two additional sets of tests were carried out with 25 and 100 lbs (11.4 and 45.5 kg), respectively, of Di-Cup T. A further purpose of these experiments was to investigate effectiveness of various extinguishers in combating fires involving Di-Cup.

In the first series of tests, 25 lbs of Di-Cup was spread on the bottom of a circular tray [24 in. (61.0 cm) in diameter] and ignited by means of a large torch. After about 30 sec of vigorous burning, the flames were extinguished with a 30-lb (13.6-kg), dry-chemical hand extinguisher, a similar standard CO₂ extinguisher, and water spray. The water spray was supplied from a 95 to 100-psi (0.65 to 0.68-MPa) hydrant system through a 1½-in. (3.8-cm) hose a combination solid-stream/water-spray nozzle.

In a second series of experiments, 100 lbs of Di-Cup T was distributed over the bottom of a 42 by 70-in. (106.7 by 177.8-cm) rectangular tray. Again, the Di-Cup was ignited by means of a torch, and after a period of vigorous burning, was extinguished with each of the extinguishers just mentioned.

Selected examples of these two sets of tests were filmed in color to demonstrate the type of fire encountered and ease of extinguishment. From these tests, it was concluded that *Di-Cup is difficult to ignite, burns vigorously, and is easy to extinguish.*

While the fire from ignition of a sizable quantity of unconfined Di-Cup must be considered a serious hazard, there were no indications that such a fire is any more hazardous than one resulting from the burning of a similar quantity of many flammable organic solvents and chemicals.

Exposure to Direct Flame

To further investigate the effect of flame exposure on confined Di-Cup in a practical way, standard 5- and 55-gal (18.9- and 208-L) containers, as shipped, were exposed to direct contact with fire. These tests were intended to explore the consequences of stored, unopened containers of Di-Cup being involved in a fire from other sources. They represent maximum hazard, in that they simulate prolonged exposure to a fire without fire-protection systems.

5-Gal (18.9-L) Tests — In early tests, 5-gal cans containing Di-Cup T were placed directly in a wood fire and allowed to remain until completely consumed. In some cases, side-by-side comparisons were made with a similar 5-gal can of turpentine. In these tests, the containers ruptured and the contents burned vigorously, but evenly, to complete consumption. The fire resulting from the burning Di-Cup was comparable to that from turpentine, and probably less violent than from a similar quantity of gasoline. No acceleration of burning rate was noted once vigorous burning had begun.

More recently, 14 such tests were performed in various types of 5-gal containers. Di-Cup was tested "as shipped," frozen, and molten. Side-by-side comparisons were made with cumene hydroperoxide and with gasoline. The surrounding fire was provided by burning isopropanol.

No significant differences were noted between the different 5-gal cans with various types of closures. There were no observable differences in test results when Di-Cup dicumyl peroxide was in its normal crystalline state and when it was premelted or prefrozen. In all cases, 5-gal containers of Di-Cup ruptured after about 4 min of exposure to fire, and burned vigorously until consumed or extinguished.

When Di-Cup and gasoline were tested in similar containers, Di-Cup overpressured the can about 2 min earlier than the gasoline. This result is attributed to exothermic decomposition of Di-Cup. Whereas gasoline was exposed to only one source of heat (the external fire), Di-Cup also generated considerable internal heat, once exothermic decomposition was initiated. However, violence of combustion when the container ruptured was much greater with gasoline than with Di-Cup.

55-Gal (208-L) Tests — In larger scale tests, very similar to the foregoing, standard 55-gal drums containing Di-Cup T were tightly sealed (ring-and-bolt closure) and suspended over burning isopropanol. After 12 min and 25 sec of direct fire exposure, Di-Cup overpressured the drum, pushed the lid off and to the side, then burned vigorously. Even on this scale, however, two men readily extinguished the fire, using 20-lb (9.1-kg) and 30-lb (13.9-kg) Ansul⁽¹⁾ dry-chemical extinguishers. After the test, about 140 lbs (63.6 kg) of molten Di-Cup, out of the 425 lbs (193 kg) originally present, remained in the drum.

Although burning of the contents is vigorous once the container is breached, no explosion occurs, as evidenced by lack of damage to the drum itself. The 55-gal drums remained upright and in the same position as when the test was started.

CONCLUSIONS AND RECOMMENDATIONS

The following conclusions and recommendations are based on the tests described in the publication:

Conclusions

1. Di-Cup dicumyl peroxide is very insensitive to mechanical shock.
2. It cannot be made to detonate even under heavy confinement and severe priming.
3. Di-Cup is relatively difficult to ignite, but once ignited it burns vigorously.
4. Burning Di-Cup is easy to extinguish. Suitable extinguishers include dry-chemical and CO₂ types and water spray.
5. When subjected to continuing heat, Di-Cup will begin to decompose exothermally at about 248°F (120°C).
6. Once exothermic decomposition begins, internal temperature will rise very rapidly, causing overpressuring of the container. Violence of rupture is proportional to the degree of confinement.
7. Standard shipping containers used for Di-Cup are breached with relatively little violence on exposure to flame. [A standard 55-gal drum of 17H construction, with ring-and-bolt closure, has been reported to fail at around 20 psi (0.13 MPa)].
8. Sealed, heavy-walled containers can result in violent pressure release.

⁽¹⁾Charles R. Teas & Co.

Recommendations

1. Treat Di-Cup with respect.
2. Consider it a highly flammable material.
3. Keep in-plant inventories to a practical minimum.
4. Provide proper fire-protection equipment. An automatic sprinkler installation supplemented with adequate extinguishers is recommended.
5. Do not store Di-Cup with other chemical materials.
6. Avoid contamination of Di-Cup with strong acids, acid-forming salts, oxidation catalysts, reducing agents, or catalysts of the Friedel-Crafts type.
7. When melting Di-Cup, use warm-water heating only, controlled to a maximum of 131°F (55°C).
8. If any quantity of Di-Cup is contained in a closed vessel, provide large-area pressure venting at 20 psi (0.13 MPa) or lower.

Technical Information

BULLETIN ORC-204E
(Supersedes ORC-204D)

DI-CUP® DICUMYL PEROXIDE AND ITS DECOMPOSITION PRODUCTS **SUMMARY OF TOXICOLOGICAL INVESTIGATIONS**

When used as a peroxidic catalyst, **DI-CUP®** dicumyl peroxide is always present in conjunction with its decomposition products (acetophenone, dimethylbenzyl alcohol, and *alpha*-methylstyrene). This bulletin, therefore, has been prepared to summarize the toxicological information obtained on all four compounds. These data are presented in detail on the following pages and are also summarized in Table I.

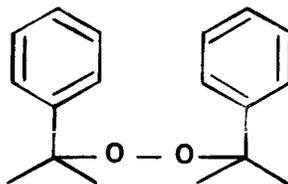
Summary

Di-Cup and its decomposition products are only slightly toxic by ingestion or inhalation. Mild eye and skin irritation may be encountered from accidental exposure to liquids or vapors.

The decomposition products of Di-Cup, however, are strong odorants. Low vapor concentrations result in persistent aromatic scents; when present in high concentrations, these odors may be objectionable.

Chemical Composition

Di-Cup is commercial dicumyl peroxide, available in four forms. The molecular weight of dicumyl peroxide is 270.36; its structural formula is shown below.



General Properties

The four forms of Di-Cup permit selection of material most convenient for the application. Di-Cup 40C and 40KE are off-white, free-flowing powders that average 40% active material supported on precipitated calcium carbonate and Burgess KE clay, respectively. Di-Cup T, with 94-97% dicumyl peroxide, is a pale yellow, low-melting, semicrystalline solid. Di-Cup R is a pale yellow to white, granular, low-melting crystalline solid, and is a more highly refined form that contains 98+% dicumyl peroxide. The specific gravity at 25/25°C is 1.57 for Di-Cup 40C and 1.55 for Di-Cup 40KE (calculated), and 1.02 for Di-Cup T and Di-Cup R. Di-Cup is used as a high-temperature catalyst in polymerization, vulcanization, and cross-linking reactions in the plastics and rubber industries.

Solubility

Dicumyl peroxide is soluble in vegetable oils and most organic solvents. It is soluble or disperses readily in natural and synthetic rubber compounds, silicone gums, and polyester resins. It is insoluble in water.

We cannot anticipate all conditions under which this information and our products, or the products of other manufacturers in combination with our products, may be used. We accept no responsibility for results obtained by the application of this information or the safety and suitability of our products, either alone or in combination with other products. Users are advised to make their own tests to determine the safety and suitability of each such product or product combination for their own purposes. Unless otherwise agreed in writing, we sell the products without warranty, and buyers and users assume all responsibility and liability for loss or damage arising from the handling and use of our products, whether used alone or in combination with other products.

Acute Oral Toxicity

The oral LD₅₀ of Di-Cup (administered as a 20% solution in corn oil) to rats was found to be 4,100 mg/kg.

Eye Irritation

Application of 0.1 ml of 50% solution of Di-Cup in corn oil to the eyes of rabbits according to the Draize method produced mild conjunctivitis, with complete recovery within 48 hours. There were no effects on the cornea.

Acute Inhalation Toxicity

Ten groups of animals (10 rats and 4 rabbits each) were exposed for 6 hours to atmospheres containing dusts of Di-Cup 40C. The concentrations ranged from 21 to 224 mg of dust/m³ of air (8 to 90 mg of dicumyl peroxide/m³ of air) and the particle size ranged from 0.4 to 18 microns. There were no observable signs of toxicity during the exposure or the 14-day postexposure observation period.

Primary Irritation and Sensitization

Guinea Pig Sensitization: Repeated intradermal injections of 0.1% suspension of Di-Cup in normal saline (according to the Draize method) into guinea pigs produced no primary irritation or sensitization.

Schwartz Prophetic Patch Test: Two hundred human subjects were patch-tested with Di-Cup T. There was slight primary irritation, but no evidence of sensitization. Two hundred human subjects also were patch-tested with two samples of rubber. One sample was cured with Di-Cup (2%) and the other with sulfur. Both produced the same incidence and degree of mild primary irritation, but did not produce sensitization. While the degree of irritation produced by Di-Cup T was essentially the same as that from cured rubber, the incidence was less.

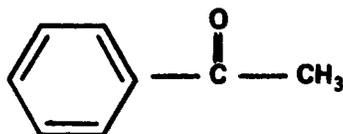
Vulcanization Fumes

In some vulcanization and cross-linking operations, there may be a secondary decomposition of cumyloxy radicals to yield acetophenone, dimethylbenzyl alcohol, *alpha*-methylstyrene, and methane. Most of the sweet, pungent odor is due to acetophenone. As with other industrial fumes, undue exposure should be avoided as much as possible, and adequate ventilation should be provided.

DECOMPOSITION PRODUCTS

ACETOPHENONE

Acetophenone, also called phenyl methyl ketone and acetylbenzene, is a low-melting solid with a sweet, pungent odor. It is used in perfume bases to impart an orange-blossomlike odor, and in numerous organic syntheses. In the past, it enjoyed limited use as an anesthetic and hypnotic. Its structural formula is shown below.



Physical Properties

Acetophenone has a molecular weight of 120.14, a melting point of 67°F (19.7°C), and a vapor pressure of 1 mm Hg at 99°F (37°C) and 100 mm Hg at 67°F (19.7°C). It is insoluble in water, but soluble in most organic solvents. It has a flashpoint (closed cup) of 180°F (82°C).

Acute Oral Toxicity

The oral LD₅₀ of acetophenone for rats has been reported as 900 mg/kg, and 3,000 mg/kg, with typical symptoms of analgesia and deep coma preceding death.

Acute Dermal Toxicity

The LD₅₀ for guinea pigs by skin absorption of undiluted acetophenone is greater than 20,000 mg/kg.

Eye Irritation

When undiluted acetophenone was placed in the eyes of rabbits, it caused conjunctival irritation and transient corneal damage comparable in degree to that caused by butyl alcohol.

Skin Irritation

Undiluted acetophenone is a primary skin irritant for rabbits. It has also been reported to cause irritation to human skin in some industrial operations.

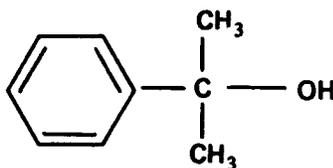
Acute Inhalation Toxicity

Because of the low vapor pressure of acetophenone, its inhalation toxicity has been given very little study. Exposure of rats to saturated vapor concentrations (about 600 ppm or 3,000 mg/m³) for 8 hours produced no deaths.

DIMETHYLBENZYL ALCOHOL (DMBA)

Chemical Composition

Dimethylbenzyl alcohol, molecular weight 136, has the following structural formula:



Acute Oral Toxicity

The oral LD₅₀ in the rat ranged from 1,400 to 3,000 mg/kg. The oral LD₅₀ in the rat of a mixture of 75% DMBA and 25% acetophenone was found to be 2,000 mg/kg.

Eye Irritation

Application of 0.1 mL to the eyes of rabbits and 0.05 mL to the eyes of guinea pigs daily for 15 days produced some irritation with redness. There was complete recovery within 7 to 14 days, with no permanent corneal damage.

Primary Irritation and Sensitization

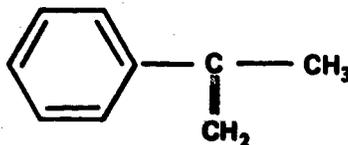
Guinea Pig and Rabbit Skin: Daily application of DMBA to the skin of rabbits and guinea pigs produced mild primary irritation.

Repeated Insult Patch Test: Fifty-five human subjects received 24-hour exposures to test patches three times weekly for 3 weeks and a challenge patch in the sixth week. There was little or no primary irritation. There was, however, a definite indication of skin sensitization in 7 of the 55 subjects, and more or less equivocal evidence of sensitization in 10 other subjects.

ALPHA-METHYLSTYRENE

Chemical Composition

alpha-Methylstyrene is a colorless liquid with a sharp, aromatic odor, a molecular weight of 118.15, and a boiling point of 330°F (165.4°C). It has the following structural formula:



Acute Oral Toxicity

The oral LD₅₀ in the rat was found to be 4,800 mg/kg.

Eye Irritation

Application of two drops in the eyes of rabbits caused slight irritation of the conjunctiva, but no corneal damage.

Skin Irritation

Repeated applications to the skin of rabbits caused moderate to marked redness. There was no indication of absorption with subsequent systemic toxic effects.

Acute Inhalation Toxicity

Vapor inhalation at 2,920 ppm (14 g/m³) for 15 to 60 minutes caused respiratory irritation and central nervous system depression in rats, mice, and guinea pigs. Mice died after about 4 hours, but rats and guinea pigs survived the 5-hour exposure.

Inhalation Toxicity

The Threshold Limit Value (TLV,⁽¹⁾ 1981) for *alpha*-methylstyrene is 50 ppm, or 240 mg/m³ (ceiling). Available toxicological information is reviewed in the TLV documentation (American Conf. of Gov. Industr. Hygienists, Cincinnati, 1980).

⁽¹⁾Registered trademark of American Conference of Governmental Industrial Hygienists.

METHANE

Methane is considered a simple asphyxiant by the ACGIH. It has no physiological action except when it lowers the partial pressure of oxygen in the air enough to cause effects due to oxygen deprivation. It has no warning odor. Because of its low toxicity, no TLV has been set.

Table I
Toxicity Summary
Di-Cup and Its Decomposition Products

	Di-Cup	Acetophenone	DMBA	<i>alpha</i>-Methylstyrene
Acute oral toxicity (LD ₅₀), mg/kg	4,100	900-3,000	1,400-3,000	4,700-4,900
Acute inhalation toxicity	90 mg/m ³ — no effect	Saturated vapor— no deaths		14 g/m ³ — produced death (mice)
Eye irritation	Mild conjunctivitis	Irritation and transient corneal injury	Irritation with redness	Irritation without corneal damage
Skin irritation (undiluted)	Mild irritation	Irritation	Mild irritation	Moderate irritation
Skin sensitization	No	No	Yes	No

Product Safety

As Hercules interprets the U.S. Occupational Safety and Health Act of 1970, Di-Cup peroxides should be considered hazardous materials because they may undergo decomposition when they are overheated or when they come in contact with copper, copper alloys, or acids. Detailed safety instructions can be obtained from Hercules Technical Data Bulletin ORC-202. Material Safety Data Sheets should be obtained prior to use of these products.



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Technical Information

BULLETIN ORC-208
 (Supersedes ORC-111D)

DI-CUP® Peroxide Semibulk Container Melting and Handling System

DI-CUP® T and DI-CUP R peroxides are commercially available in 55-gal, open-head drums and in semibulk containers (SBC's) of approximately 460-gal capacity. At normal shipping and storage temperatures, these peroxides are oily to semicrystalline solids. Means of safely handling, melting, and storing Di-Cup in 55-gal shipping containers are given in Hercules Bulletin ORC-202. (See bibliography.)

This bulletin describes an effective system for melting, handling, and storing these peroxides in SBC's, and suggests principles of design. Consult your Hercules representative for specific requirements. Arrangements for a safety engineering presentation at the customer's plant that reviews in detail the principles and guidelines outlined here can also be made through your Hercules representative.

Hercules' recommendations for the safe handling, melting, and storage of Di-Cup in SBC's are summarized herein. However, these recommendations are not a substitute for, nor an inducement to violate, applicable laws or standards related to safety, insurance coverage, pollution control, or good engineering practice.

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We cannot anticipate all conditions under which this information and our products, or the products of other manufacturers in combination with our products, may be used. We accept no responsibility for results obtained by the application of this information or the safety and suitability of our products, either alone or in combination with other products. Users are advised to make their own tests to determine the safety and suitability of each such product or product combination for their own purposes. Unless otherwise agreed in writing, we sell the products without warranty, and buyers and users assume all responsibility and liability for loss or damage arising from the handling and use of our products, whether used alone or in combination with other products.

Melting and Handling System

The semibulk container melting and handling system for Di-Cup (see Figure 4 and Tables I and II) converts Di-Cup from the solid or semisolid state to liquid for transfer to subsequent processing. It is based on a hot water heating system with the temperature automatically controlled at 122 to 131°F (50 to 55°C). When properly designed, such a system minimizes the chance of overheating.

Note: Steam, electric band heaters, or any heating medium above 131°F (55°C) should never be used for melting Di-Cup, because localized heating can cause hot spots and eventual decomposition of the peroxide. Ovens and hot rooms are other sources of heat that are not considered safe.

Hercules SBC's meet Department of Transportation Specification 57 requirements for portable tanks. The tanks are fabricated of 304L stainless steel, 45½ in. OD x 64 in. high, 460 gal total capacity. Current use capacity is 400 gal, or 3,500 lbs net (1,590 kg). Total gross weight is approximately 4,300 lbs. A forklift truck is required to transport the SBC's. Figures 1, 2, and 3 show several views of Hercules' semibulk container. It is recommended that a minimum of two SBC's be installed in parallel in the melting and handling system (Figure 4) if uninterrupted flow must be maintained.

Physical Properties

Physical properties of Di-Cup T and R that should be considered in devising safe and effective means of handling these products are given below:

	<u>Di-Cup T</u>	<u>Di-Cup R</u>
Melting point, °C	30-38	38
Heat of fusion, cal/g	18 ± 2	18 ± 2
Specific heat		
Solid	0.35	0.35
Liquid	0.42	0.42
Specific gravity at 50/50°C	1.02	1.02
Viscosity, cps ^(a)		
At 40°C	12.5	19.1
At 50°C	10.3	10.6
Flashpoint, SETA-CC, °F(°C)	170 (77)	260 (127)

^(a)Brockfield viscometer, Model LVF, Spindle No. 1, at 60 rpm.

Temperature Control Precautions

Di-Cup T or R can be safely handled with minimum precautions at ordinary temperatures and within a limited temperature range in which it is a liquid. The basis for its commercial utilization, however, requires that it decompose at elevated temperatures and at an ever-increasing rate as the temperature increases. Because of this inherent characteristic, certain precautions regarding temperature control should be observed in melting Di-Cup.

- SAFE:** At 86 to 131°F (30 to 55°C), Di-Cup T or R can be held for several days without significant decomposition.
- DANGEROUS:** The handling temperature should never exceed 131°F (55°C). Above this, the rate of thermal decomposition and heat evolution increases rapidly. At about 244°F (118°C), rapid exothermic decomposition takes place, as evidenced by flammable white smoke.
- EMERGENCY ACTION:** Should the temperature reach 158°F (70°C), cold water should be introduced into the coils of the SBC by opening valves **A** and **B** and closing valve **C**. (See Figure 4.)
- Should the temperature reach 176°F (80°C), the deluge sprinkler system serving the SBC melt station should be manually tripped.

Semibulk Melt Station Location

Ideally, the SBC melt station for Di-Cup should be located in a separate, enclosed area away from the process facilities. Often this area is an outside shed or an enclosed corner of an existing building with two outside walls. All peroxide equipment, including the semibulk containers, peroxide pump, peroxide filter, etc., should be housed inside the enclosed area. The electrical classification inside the enclosure should be Class 1, Group D, Division 2. The enclosure should be protected by an automatic deluge sprinkler system designed to deliver 0.3 gpm/ft² of floor area. The enclosure should also include provisions for ventilation, drainage, and comfort heating.

The hot water equipment, including the tank, pump, heater, and high-temperature alarms, should all be located outside the enclosure. Such an arrangement allows the electrical classification for the hot water equipment to be Nonhazardous.

Construction details of the melt station enclosure will vary, depending on the specific location chosen by the customer. Hercules should be consulted regarding potential locations and specific recommendations.

Safety Precautions for Di-Cup SBC Storage Area

The following safety precautions *must be adhered to* in the storage area for Di-Cup peroxides:

- The storage area for SBC's should be separate from the process area, important buildings, and public neighboring localities so that a fire in the storage area will not jeopardize them.
- SBC's are never to be double-tiered.
- The storage area should be under cover, protected from direct rays of sunlight (a shed with walls on the sun-exposure sides provides this protection).
- The storage area building should be constructed of noncombustible materials.
- The storage area should be sprinkler-protected.
- The storage area should have sufficient natural or mechanical ventilation to prevent the temperature of the Di-Cup from exceeding 125°F (52°C).
- The electrical classification of the storage area should be Nonhazardous for storage of unopened containers.

Safety Precautions (Continued)

- At least one fire hydrant with sufficient lengths of hose should be located preferably not closer than 50 ft nor more than 150 ft from the storage area.
- The storage area should be protected against access by unauthorized personnel.
- Smoking in the storage area is prohibited.
- Housekeeping in and around the storage area should be maintained to prevent accumulation of easily combustible materials such as dry grass or paper.
- No acids or other chemicals, such as Friedel-Crafts catalysts, reducing agents, and oxidation catalysts, are to be stored in an area used for Di-Cup.
- The storage area should be isolated from storage areas used for flammables such as solvents.

Waste Disposal

All waste Di-Cup, or materials containing appreciable quantities of it, should be disposed of in accordance with local, state, and Federal regulations.

Caution: Do not mix concentrations or solutions of Di-Cup with other chemical wastes. Do not put solutions containing Di-Cup into sewer systems.

Product Safety

This product may be considered hazardous under the U.S. Occupational Safety and Health Act of 1970. A Material Safety Data Sheet should be obtained prior to use of this product. Toxicological data are available in Hercules Technical Data Bulletin ORC-204.

Figure 1
Hercules 400-Gal Semibulk Container

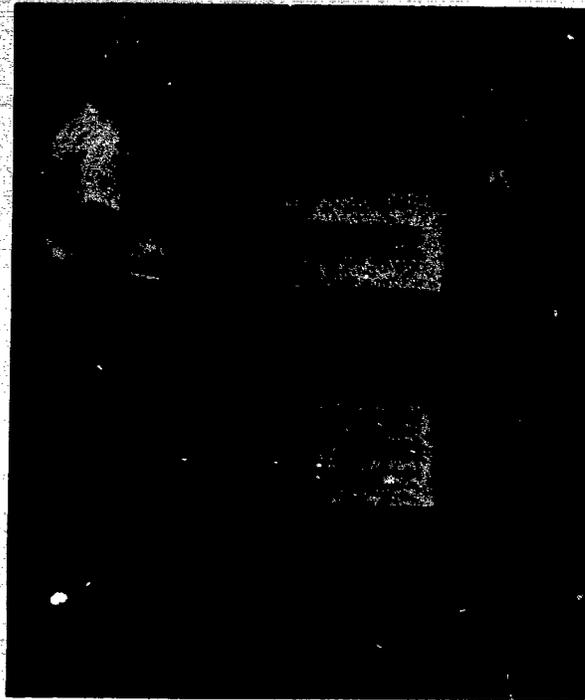


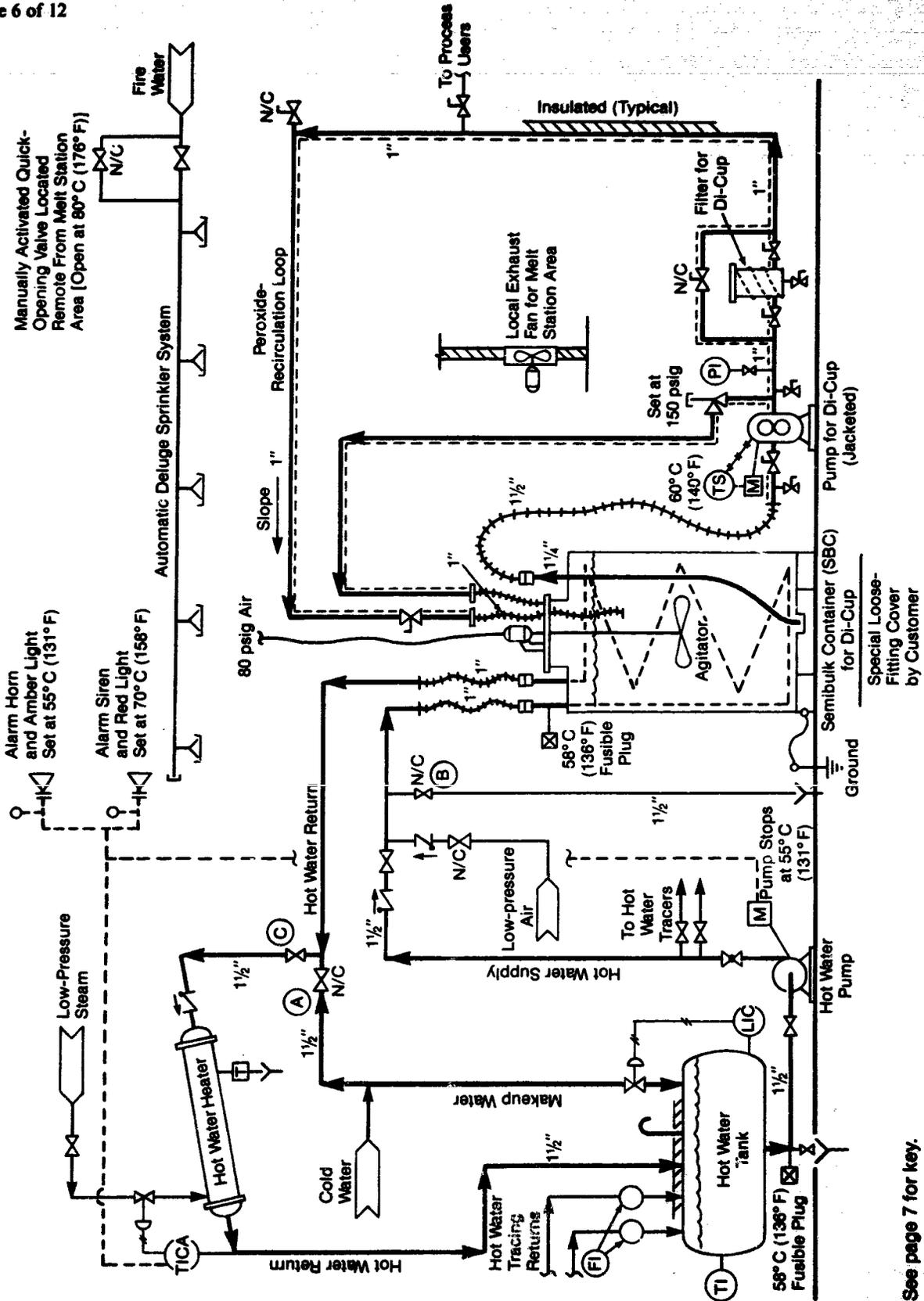
Figure 2
Top View of SBC, Showing Removable Lid,
Hot Water Connections,
and Eductor Pipe Connection for DI-Cup



Figure 3
SBC With Lid Removed,
Showing Internal Hot Water Coils



Figure 4
Melting and Handling System



See page 7 for key.

KEY:

-  Hot-water-traced piping and equipment.
-  Instrument air lines.
-  Instrument electrical lines.
-  Instrument capillary or filled system.
-  Flexible hose with quick-connect Hansen coupling.
-  Gate valve.
-  Ball valve.
-  Globe valve.
-  Check valve.
-  Air-operated control valve.
-  Temperature indicator.
-  Temperature indicator control alarm.
-  High-temperature cutout (stops pump for Di-Cup).
-  Pressure indicator.
-  Level-indicating controller.
-  Flow indicator.
-  Pump motor.
-  Valve normally closed.
-  Emergency valves. (See Table I.)

Table I
Safety Features Included in Figure 4

Triggering Temperature	Safety Feature and/or Description	Corrective Action Required
122-129° F (50-54° C)	Normal hot water control temperature	No corrective action required.
131° F (55° C)	<i>First high-temperature alarm system</i> —sounds horn, activates amber light, and stops hot water circulating pump.	Determine cause of excessive water temperature. Correct and restart water pump.
136° F (58° C)	Fusible plug (on water piping and/or SBC) melts and diverts hot water flow away from SBC.	Determine why water temperature is excessive and correct; replace fusible plug.
140° F (60° C)	High-temperature cutout on peroxide pump is activated, which stops pump.	Determine cause of pump overheating or overtemperature peroxide.
158° F (70° C)	<i>Second high-temperature alarm system</i> —sounds siren, activates red light, and stops hot water circulating pump.	Emergency action required. Introduce cold water into coils of semibulk container by opening valves (A) and (B) and closing valve (C) . Cool peroxide back to 122° F (50° C).
176° F (80° C)	Beyond this temperature it may be difficult to stop rapid decomposition.	Emergency action required. Manually trip deluge sprinkler system protecting melt station. Keep personnel away from area.

**Table II
Equipment Specifications**

Equipment used in a typical SBC melting and handling system for Di-Cup, as shown in Figure 4, is given below:

<u>Hot Water System</u>	<u>Description</u>
Hot water tank	60 to 100-gal galvanized or glass-lined atmospheric storage tank.
Hot water circulation pump	Centrifugal type, 25 gpm at 50 ft HD.
Hot water piping	Galvanized with brass valves.
Hot water tracing	<p>5/8-in. copper. Bond to peroxide piping and equipment with Thermon grade T-85 heat-transfer cement.</p> <p>Note: A special temperature-limited electrical tracing system known as Chemelex Auto-Trace is an acceptable alternative to hot water tracing. This system is manufactured by Raychem Corporation, 837 Second Avenue, Redwood City, CA 94063. It must be specified as Factory Mutual approved for use in a Class I, Group D, Division 2 electrical classified area. In addition, the Chemelex system must be engineered so that the Di-Cup temperature will not exceed 122°F (50°C).</p>
Hot water heater	Bell and Gossett type SU or equivalent, 22-ft ² carbon steel shell, 3/4-in.-OD copper tubes.
Fusible plug	Melts at 136°F (58°C). Plug has 1-in. NPT and is available from Hercules on request.
Water hoses (2)	Rubber, with 1-in. NPT end adapter.
Water hose connections (2) (Connect to semibulk container water coil)	<p>1-in. 303 stainless steel quick-connect Hansen 8-HK LL8-H36 with internal shutoff valve (socket on supply hose).</p> <p>1-in. 303 stainless steel quick-connect Hansen 8-HK LL8-36 with internal shutoff valve (plug on return hose).</p>
Hot water flow indicators	Ernst Gauge Company or equivalent, 200-psig, 200°F rotating wheel.
Insulation	Fiberglass for hot water tank, pump, heater, and piping.
Temperature indicator	Taylor or equivalent dial thermometer, 32-212°F (0-100°C) range.
Steam control valve	1-in. carbon steel; requires air to open (closes on air failure).
Temperature control	Local temperature indicator/controller, Foxboro or equivalent pneumatic type, 32-212°F (0-100°C) range.
Alarm horn, alarm siren, and warning lights	<p>Electrically tied in with above temperature controller. Alarm horn and amber light set at 131°F (55°C). Provide an alarm silence button to silence horn, but allow pilot light to remain energized.</p> <p>Alarm siren and red light set at 158°F (70°C).</p> <p>Locate horn, siren, and warning lights in a location where employees are always present.</p>

Hot Water System (Continued)

Level-indicating control Customer's choice.

Low-pressure air connection For blowing out residual water left in coils after emptying an SBC.

Hercules Semibulk Container and Auxiliaries

Semibulk container **Provided by Hercules.** DOT Specification 57 portable tank. 45½-in. OD × 64 in. high, 460-gal volume, 400-gal operating capacity, 3,500 lbs net weight of Di-Cup, 4,300 lbs approximate gross weight, 304L stainless steel construction. Has 22½-in.-OD removable lid secured by a removable weak chime closure. Lid includes a threaded Rieke fitting frangible relief device with a set pressure of 3 psig. Container has approximately 150 lin ft of 1-in. 304L stainless steel internal heating coil and a single 304 stainless steel 1¼-in. eductor pipe and well. Fittings terminate in quick-connect Hansen couplings as described below. Bottom of container has two box channels suitable for movement by a forklift truck.

Ground clamp Stewart R. Browne or equivalent VUD-100 temporary bonding and grounding device.

Dipstick Used for determining liquid level in SBC, 7-ft-long stainless steel rod.

Water coil connections (2) **By Hercules on semibulk container.** 1-in. 303 stainless steel quick-connect Hansen 8-HK LL8-K36 with internal shutoff valve (plug on coil inlet connection).

1-in. 303 stainless steel quick-connect Hansen 8-HK LL8-H36 with internal shutoff valve (socket on coil outlet connection).

Peroxide connection (dip pipe) **By Hercules on semibulk container.** 1¼-in. stainless steel quick-connect Hansen 12-HK LL12-H41 with internal shutoff valve and Viton⁽¹⁾ O-rings (socket on dip pipe outlet).

Fusible plug **By Hercules on SBC water coil inlet tee connection.** Melts at 136°F (58°C). Plug has 1-in. NPT.

Temperature indicator **By Hercules on outside of semibulk container.** Two indicating labels supplied. Color darkens at 120, 140, 160, and 180°F (49, 60, 71, and 82°C).

Agitator Variable-speed air-motor-driven portable agitator, stainless steel blades and shaft, with 4-ft shaft. Gast Manufacturing Company or equivalent.

Special cover A flat, loose-fitting stainless steel cover provided by the customer. Cover provides support for portable agitator and includes two sleeves for insertion of two flexible hoses (peroxide recirculation line and relief valve tailpipe).

Exhaust fan Depending on the arrangement and location of the semibulk melting station, the exhaust fan can be either a wall fan serving the semibulk melt room or a local exhaust fan near the top of the tank. Check with Hercules for specific application guidelines.

⁽¹⁾E. I. du Pont de Nemours & Co.

Peroxide Supply System

Peroxide unloading hose connection (to dip pipe on tank)

1½-in. stainless steel quick-connect Hansen 12-HK LL12-K41 with internal shutoff valve and Viton O-rings (plug on peroxide hose).

Peroxide unloading hose

1½-in. stainless steel flexible hose, length to suit.

Peroxide recirculating line hose

1-in. stainless steel flexible hose, length to suit.

Relief valve tailpipe hose

Stainless steel flexible hose, length to suit.

Peroxide piping and valves

1-in. Schedule 40 Type 304 or 316 stainless steel, and stainless steel ball valves.

Note: Do not use any copper, copper alloys, lead, or iron in direct contact with Di-Cup. These materials will catalyze the peroxide and could result in decomposition.

Peroxide gasketing, O-rings, and pipe dope

Teflon⁽¹⁾ (Di-Cup attacks all natural and most synthetic rubbers).

Peroxide pump

Self-priming centrifugal or rotary gear pump. Viking heavy-duty alloy pump, Model H 4724, 10 gpm, 50-psig discharge pressure, 1,200 rpm, Class I, Group D, Division 2 motor. Pump casing and all wetted parts 316 stainless steel complete with mechanical seal (stainless steel, Teflon, carbon graphite, and stellite). Pump to be completely jacketed with jacketed headplate for hot water heating and provided *without* internal relief valve.

Relief valve

Stainless steel, 150-psig set pressure. Route tailpipe to semibulk container.

Peroxide filter

Cartridge filter, Fullflow or United, 10 gpm, 316 stainless steel housing and cartridge, 10-micron cotton filter fabric. Hot water jacketing optional.

Heat tracing

Note: All peroxide piping, fittings, valves, filters, etc., must be heat-traced with hot water or electrical tracing as noted under Hot Water System.

Insulation

All peroxide piping, valves, filters, pumps, etc., should be insulated with a noncombustible, nonwicking-type insulation such as Foamglas.⁽²⁾

High-temperature cutout for peroxide pump

Temperature switch. United Electric Control, Class I, Group D, Division 2, 15 amps, normally closed. Set to open (stop pump) at 140°F (60°C).

Pressure indicator

Stainless steel, 0 to 200-psig range.

⁽¹⁾E. I. du Pont de Nemours & Co.
⁽²⁾Pittsburgh Corning Corp.

Bibliography

**Hercules
Bulletin**

- | | |
|---------|--|
| ORC-201 | Di-Cup Dicumyl Peroxide—Vulcanization Agent and Polymerization Catalyst |
| ORC-202 | Di-Cup Dicumyl Peroxide—Safety Aspects of Storing and Handling 55-Gallon Shipping Containers |
| ORC-203 | Di-Cup Dicumyl Peroxide—Summary of Hazard Tests |
| ORC-204 | Di-Cup Dicumyl Peroxide and Its Decomposition Products—
Summary of Toxicological Investigations |

Material Safety Data Sheets (MSDS)

- | | |
|--------|-------------------|
| OR-614 | Di-Cup R Peroxide |
| OR-615 | Di-Cup T Peroxide |

MATERIAL SAFETY DATA SHEET

(Approved by U.S. Department of Labor as "Essentially Similar" to Form OSHA-20)

MSDS OR 614B

DATE 3/28/83

PAGE 1 of 3

I. PRODUCT IDENTIFICATION

CHEMICAL NAME AND SYNONYMS	Dicumyl peroxide	REGULAR TELEPHONE NO. 302-678-5000
CHEMICAL FAMILY	Organic peroxide	EMERGENCY TELEPHONE NO. 302-606-3000*
FORMULA	C ₁₈ H ₃₂ O ₂	CAS NO. 80-43-3
TRADE NAME AND SYNONYMS	DI-CUM® R	MOLECULAR WEIGHT 270

II. HAZARDOUS INGREDIENTS

MATERIAL	%	TLV-TWA VALUES ADOPTED BY ACGIH 1981
Dicumyl peroxide	98-100	Not established

III. PHYSICAL DATA

BOILING POINT, 760 mm Hg	NA-Decomposes	MELTING POINT	38°C (100°F)
VAPOR PRESSURE @ 38°C	15.4 mm Hg	SPECIFIC GRAVITY (H ₂ O=1)	1.04 at 25/25°C Calculated
VAPOR DENSITY (AIR=1)	NA	PERCENT VOLATILE BY VOLUME (%)	0.03 at 20°C (68°F)
SOLUBILITY IN WATER, % BY WEIGHT @	Negligible	EVAPORATION RATE (BUTYL ACETATE=1)	0.002
APPEARANCE AND ODOR: Pale yellow to white granular solid; acrid, fruity odor.		pH	NA

IV. FIRE AND EXPLOSION HAZARD DATA

FLASH POINT (TEST METHOD)	126.7°C (260°F) SETA-CC	AUTOIGNITION TEMPERATURE	Not determined
FLAMMABLE LIMITS IN AIR PERCENT BY VOLUME	LOWER Not determined	UPPER	Not determined

EXTINGUISHING MEDIA: Water spray, dry chemical, foam, or carbon dioxide.

SPECIAL FIRE-FIGHTING PROCEDURES: Use water to keep fire-exposed containers cool. Direct hose streams from a protected location.

UNUSUAL FIRE AND EXPLOSION HAZARDS: See SECTION VI, REACTIVITY DATA

NOTES: Factory Mutual has given this product FM organic peroxide Classification IV when handled in DOT 17H drums. Class IV peroxides have moderate fire hazard characteristics that can be easily contained by normal sprinkler systems and fire walls.

NA=NOT APPLICABLE

*Use this emergency number only after normal business hours and only for emergencies involving safety and health.

We cannot anticipate all conditions under which this information and our products, or the products of other manufacturers in combination with our products, may be used. We accept no responsibility for results obtained by the application of this information or the safety and suitability of our products, either alone or in combination with other products. Users are advised to make their own tests to determine the safety and suitability of each such product or product combination for their own purposes. Unless otherwise agreed in writing, we sell the products without warranty, and buyers and users assume all responsibility and liability for loss or damage arising from the handling and use of our products, whether used alone or in combination with other products.

HERCULES INCORPORATED • WILMINGTON, DELAWARE 19899

V. HEALTH HAZARD DATA

THRESHOLD LIMIT VALUE: Not established. See Section II.

EFFECTS OF OVEREXPOSURE: Mild eye and skin irritation. Decomposition products and fumes from vulcanization and crosslinking operations may cause eye, skin, and respiratory irritation and may be skin sensitizers. See Hercules Bulletin ORC-204 for summary of toxicological investigations.

EMERGENCY AND FIRST AID PROCEDURES:

EYES: In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Call a physician.

SKIN: Wash thoroughly with soap and running water. Wash clothing before reuse.

INHALATION: Remove to fresh air and, if indicated, give artificial respiration, preferably mouth-to-mouth. If breathing is difficult, give oxygen. Call a physician. Treat symptomatically.

VI. REACTIVITY DATA

STABILITY		CONDITIONS TO AVOID: Stable at room temperatures. Avoid temperatures above 55°C (131°F) or contact with materials listed below. Higher temperatures or contact with listed materials promote exothermic decomposition.
UNSTABLE		
STABLE	X	

INCOMPATIBILITY (MATERIALS TO AVOID): Acids and acidic-type materials such as Friedel-Crafts catalysts, strong oxidizing and reducing agents, and oxidation catalysts. Copper, copper alloys, lead and iron.

HAZARDOUS DECOMPOSITION PRODUCTS: Acetophenone, cetyl alcohol, *o*-methyl styrene, methane, and ethane. Under acidic decomposition conditions, phenols may also be formed. Combustion products include CO, CO₂, and generation of smoke.

HAZARDOUS POLYMERIZATION	MAY OCCUR		CONDITIONS TO AVOID: None
	WILL NOT OCCUR	X	

VII. SPILL OR LEAK PROCEDURES

STEPS TO BE TAKEN IF MATERIAL IS RELEASED OR SPILLED: Salvage in clean, steel drums. Absorb excess in clean sand and place in clean, steel drums for disposal.

WASTE DISPOSAL METHOD: Dispose of in accordance with local, state, and Federal regulations.

VIII. SPECIAL PROTECTION INFORMATION

RESPIRATORY PROTECTION (SPECIFY TYPE) None required in normal use. Use self-contained breathing apparatus in excessive fumes from product decomposition.

VENTILATION	LOCAL EXHAUST	—	SPECIAL OTHER	—
	MECHANICAL (GENERAL)	—		Use adequate ventilation.
PROTECTIVE GLOVES: Chemical resistant			EYE PROTECTION: Safety glasses	
OTHER PROTECTIVE EQUIPMENT: —				



MATERIAL SAFETY DATA SHEET

MSDS OR 614B
DATE 3/28/83

TRADE NAME DI-CUT® R

PAGE 3 of 3

IX SPECIAL PRECAUTIONS

PRECAUTIONARY LABELING

DANGER!

HEAT OR CONTAMINATION MAY CAUSE HAZARDOUS DECOMPOSITION.

PRODUCT MAY CAUSE IRRITATION OF EYES AND SKIN.

DECOMPOSITION PRODUCTS MAY CAUSE EYE, SKIN, AND RESPIRATORY IRRITATION AND MAY BE SKIN SENSITIZERS.

Do not heat above 55°C (131°F) or permit contact with acids and acidic-type materials such as Friedel-Crafts catalysts, strong oxidizing and reducing agents or oxidation catalysts. Higher temperatures or contact with these materials cause rapid, self-accelerating heating, and decomposition that may cause container rupture and possible fire. Avoid contact with copper, copper alloy, lead, and iron as contact with these metals accelerates decomposition.

Avoid contact with eyes, skin, and clothing.

Avoid breathing vapors.

Use with adequate ventilation.

Wash thoroughly with soap and water after handling.

FIRST AID: EYES - In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Call a physician.

SKIN - Wash thoroughly with soap and running water. Wash clothing before reuse.

INHALATION - Remove to fresh air and, if indicated, give artificial respiration, preferably mouth-to-mouth. If breathing is difficult, give oxygen. Call a physician. Treat symptomatically.

STORAGE AND HANDLING: Store below 52°C (125°F) in original containers in a ventilated area out of direct sunlight and away from heat sources.

SPILL OR LEAK: Salvage in clean steel drums. Absorb excess in clean sand and place in clean steel drums for disposal. Dispose of in accordance with local, state, and Federal regulations.

BULK CONTAINER LABELS

MELTING PROCEDURE: Melt by circulating hot water at 55°C (131°F) maximum temperature through coils provided in container. Other heat sources such as ovens, hot rooms, immersion heaters, and direct flame or steam are not safe. Please refer to appropriate Hercules literature that offers a more comprehensive procedure for heating of this container.

DRUM LABELS

MELTING PROCEDURE: USE WATER BATH OR WATER COILS ONLY. Heat must be applied uniformly without creating localized hot spots that might exceed 55°C (131°F). Other heat sources such as ovens, hot plates, hot rooms, immersion heaters, and direct flame or steam are not safe. Please refer to appropriate Hercules literature that offers a more comprehensive procedure for heating of this container.



Hercules Incorporated
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Wilmington, DE 19899
(302) 575-8500

Product Data

NUMBER 420-7
(Supersedes 420-6)

RETEN® 210 and 220 Cationic Water-Soluble Polymers

RETEN® 210 and 220 are cationic, high molecular weight acrylamide-based copolymers supplied as finely divided powders. They dissolve readily in warm or cold water to produce clear, smooth, non-Newtonian solutions of high viscosity for use in a variety of industrial applications.

Typical Properties^(a)

Polymer	Reten 210	Reten 220
Cationic functionality	Low	Moderate
Bulk density, lbs/ft ³ (kg/m ³)	42 (667)	42 (667)
Volatiles, as packed, max, %	15	15
Color	← Off-white to white →	
Particle size, %, on 20 mesh, max	1	1
Through 200 mesh, max	30	30
Solution		
Viscosity (typical values), ^(b) cps		
0.5% solids solution	300	325
1.0% solids solution	700	750
pH, ^(c) 1% solids	5	5

^(a)These values are not necessarily product specifications.

^(b)Determined using a Brookfield model LVF viscometer with spindle speed of 60 rpm.

^(c)in distilled water.

Outstanding Characteristics

An outstanding property of these Reten polymers is their affinity for the surface of aqueous dispersed solids. The linearity of the Reten polymer molecule in aqueous solution, coupled with this affinity for surfaces, makes it a powerful bridging flocculant. Its high molecular weight makes it a more efficient flocculant than other commonly used products.

Applications

Reten 210 and 220 polymers are used in aqueous solution, depending on type and solution concentration, as a flocculant, slip agent, thickener, antistat, adhesive, film-former, solids-suspending agent, and/or chemical cross-linking agent. These products are also substantive to protein and demonstrate good specific adhesion to many hydrophobic surfaces.

The solutions are compatible with nonionic and most other cationic materials. Reten cationic polymers may complex and precipitate from solution in the presence of high molecular weight anionic resins and polyfunctional anions such as those found in detergents.

(over)

We cannot anticipate all conditions under which this information and our products, or the products of other manufacturers in combination with our products, may be used. We accept no responsibility for results obtained by the application of this information or the safety and suitability of our products, either alone or in combination with other products. Users are advised to make their own tests to determine the safety and suitability of each such product or product combination for their own purposes. Unless otherwise agreed in writing, we sell the products without warranty, and buyers and users assume all responsibility and liability for loss or damage arising from the handling and use of our products, whether used alone or in combination with other products.

Preparation, Viscosity, and Storage of Dilute Solutions

It is recommended that only solutions of Reten of more than 0.5% solids be stored and handled, and that they be prepared with care. Clean water, free of dissolved organics and suspended matter, should be used to avoid formation of insoluble flocs in the preparation and handling system. For best results, the makeup water should have a hardness of less than 100 ppm and residual chlorine of less than 1 ppm.

Extremes of pH will degrade the polymer and will also adversely affect the solution viscosity, as shown in Figure 1. Makeup water should be at pH 3.5 to 8 to obtain the highest solution viscosity.

To prepare solutions, the polymer should be added carefully to water under low-shear agitation, and stirring should be continued until the polymer is completely dissolved. High-shear agitation should be avoided because it can degrade the polymer. Additional information on solution preparation, together with equipment recommendations, is available in Bulletin VC-471, Preparation of Solutions of Reten® Polymers.

Reten polymer solutions can support the growth of microorganisms, so a preservative may be required if solutions are to be stored. For best storage stability, the solution pH should be in the range of 3.5 to 5.0.

Shipping and Handling

Reten 210 and 220 are shipped as dry powders in 25- and 50-pound (11.4- and 22.7-kilogram), polyethylene-lined multiwall bags. They should be stored in a cool, dry place.

Reten 210 and 220 have a low order of oral toxicity and are not primary skin irritants or sensitizers. In case of contact with the skin or eyes, rinse with running water.

Avoid spills of Reten 210 and 220 powders or solutions, as floors become slippery when wet. Use an absorbent on spills and sweep up for disposal.

FDA Status

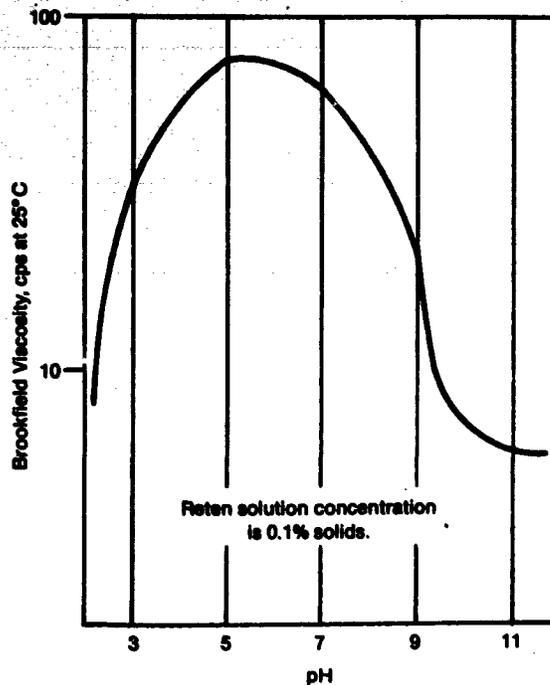
Subject to any limitations specified, Reten 210 and 220 are in compliance with the requirements of the U.S. Food and Drug Administration as specified in the Code of Federal Regulations, Title 21, under the following Sections:

- 176.170 Components of paper and paperboard in contact with aqueous and fatty food
- 176.180 Components of paper and paperboard in contact with dry food

Product Safety

Material Safety Data Sheets should be obtained prior to use of these products.

Figure 1
Typical Relationship for Solution Viscosity vs pH for Reten 210 and 220





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Toxicological Data

BULLETIN T-112C
(Supersedes T-112B)

Cationic RETEN® Products Summary of Toxicological Investigations

Chemical Composition and Properties

RETEN® 205, 210, and 220 are a family of high molecular weight synthetic polymers that possess strong cationic functionality. They differ only in cationic substitution, with Reten 220 having the highest.

These cationic Reten products are supplied as powder or granules that dissolve readily in either cold or hot water to produce viscous, non-Newtonian, shear-thinning solutions.

Results of studies with all three products are reported in this bulletin. Reten 205 was used in the initial studies. Since Reten 220 has the highest cationic substitution, it was used for the in-depth toxicological studies.

Acute Oral Toxicity

A 1% aqueous solution of Reten 205 was administered to rats at 463 milligrams/kilograms, the highest single dose possible. This was accepted without toxic manifestations.

Acute Eye Irritation

Three milligrams of powdered Reten 205 were placed in the conjunctival sac of rabbits' eyes, without subsequent washing. Shortly after application, the test material became lodged in the lower conjunctival sac to provide a continual release of Reten 205. Moderate erythema and vascularization of the sclera and nictitating membrane developed quickly, and persisted, without progressing to greater severity, for 2 to 4 days.

One hundred milligrams of Reten 210 were instilled into the eyes of albino rabbits. The left eye of each animal was rinsed for 2 minutes with tap water after a 1-minute contact period. Transient iridal and conjunctival irritation was noted in both eyes within 1 hour after instillation. Within 7 days, the ocular tissues of all animals returned to normal. Reten 210 was rated as being mildly irritating to both washed and unwashed eyes.

Acute Dermal Toxicity

Reten 205 was applied to the clipped skin of rabbits in the form of a moist aqueous paste at dosage levels up to 3,160 milligrams/kilograms for 24 hours. No systemic toxic effects were observed. Mild erythema, which subsided within 2 days, was present.

Acute Inhalation Toxicity

Mice, rats, and guinea pigs were exposed to a dust of Reten 205 for 6 hours at a concentration of 0.049 milligrams/liter. The only abnormal behavior observed was a slowed and deepened respiration. On autopsy, there was slight congestion of the lungs, but all other tissues and organs were normal.

This bulletin has been prepared as a service to Hercules customers to summarize the results of our own research and the research of others as of the date of issue. It is believed to be reliable. However, we make no guarantee that the studies and conclusions drawn from them are complete, definitive, and adequate for all purposes. Purchasers should, therefore, determine for themselves the applicability and completeness of these studies as well as the suitability and safety of the product itself for any specific use. All Hercules products are warranted to be of our standard quality; there are no other warranties, either expressed or implied.

Subacute (90-Day) Feeding Studies — Rats

Reten 205 was fed daily for 90 days to rats at concentrations of 500, 2,000, 10,000, and 50,000 ppm. There were no significant toxic effects as measured by rate of growth, general behavior or reactions, mortality, urinalysis, hematologic studies, gross pathology, and microscopic pathology at any level of Reten 205. Examination of organ weights, organ-to-body weight ratios, and organ-to-brain weight ratios did not reveal any changes at any feeding level, with the exception of a very slight increase in liver weights of female rats fed 10,000 and 50,000 ppm. In light of these very slight increases in liver weight, it was concluded that 2,000 ppm is the "no effect" level.

Reten 220 was fed daily for 90 days to rats at concentrations of 500, 2,000, and 10,000 ppm. Observations made during the course of the tests included rate of growth, mortality, general behavior or reactions, hematologic studies, and urinalysis. After sacrifice, gross and microscopic pathologic studies included gross autopsy examination, organ weights, and histologic examinations of organs. The results of these examinations showed no significant effects at any dietary level.

Subacute (90-Day) Feeding Studies — Dogs

Reten 205 also was fed daily for 90 days to beagles at concentrations of 500, 2,000 and 10,000 ppm. There were no significant toxic effects as measured by general behavior or reactions, mortality, urinalysis, hematologic studies, clinical blood chemistry studies, gross pathology, and microscopic pathology at any dietary level. The only significant effects found were a depression in weight gain among animals in the 10,000-ppm group because of partial food refusal and subsequent reduced food consumption. This caused slightly elevated organ-to-body weight ratios among the animals at the 10,000-ppm level. No other effects were revealed, and it was concluded that the "no effect" level is 2,000 ppm.

Human Patch Tests

Fifty-five subjects were patch-tested by the Draize closed-patch repeat-insult technique with 0.5 milliliter of a 2% aqueous solution of Reten 205. The subjects received 24-hour exposures 3 times a week for 3 weeks; and after a 2-week rest period, they received a challenge application. No primary irritation or sensitization was observed with any of the subjects.

Chronic (2-Year) Oral Toxicity — Rats

Two-year chronic oral toxicity studies have been conducted on Reten 220. The polymer was incorporated into ground commercial diet and fed to rats at dietary levels of 10,000, 2,000, and 500 ppm. A control group of rats received the stock diet.

The observations made during the course of the study included body weights, food consumption, mortality, behavioral reactions, hematologic studies, clinical blood chemistry studies, urinalysis, and tumor incidence. After sacrifice, gross and microscopic pathologic studies included gross autopsy examination, individual organ weight evaluation, and complete histologic examination of organs.

The results of these examinations demonstrated no significant toxic effects at any dietary level.

Chronic (2-Year) Oral Toxicity — Dogs

Two-year chronic oral toxicity studies have been conducted on Reten 220. The polymer was incorporated into ground commercial diet and fed to beagles at dietary levels of 10,000, 2,000, and 500 ppm. A control group of beagles received the stock diet.

The observations made during the course of the study included body weights, food consumption, mortality, behavioral reactions, hematologic studies, blood chemistry studies, liver and kidney function tests, and urinalysis. After sacrifice, gross and microscopic pathologic studies included gross autopsy examination, individual organ weight evaluation, and complete histologic examination of organs. The results of these examinations showed no significant toxic effects at any dietary level.

Three-Generation Reproduction Study in Rats

A 3-generation, 6-litter reproduction study in albino rats has been conducted with Reten 220. The resin was incorporated into ground commercial diet and fed to the rats at dietary levels of 2,000 and 500 ppm. A control group received the stock diet. Animals of each generation in all groups were maintained on their respective diets without interruption until their sacrifice which followed the weaning of the second litters. Weanlings from the second litter were selected as parents for the second generation and continued on their respective diets until after weaning of a second litter. A third generation was selected in the same manner. Complete gross pathology, organ weight evaluation, and microscopic pathology were performed on all 3 parental generations following the weaning of the second litters in each case. After weaning of the last litter, the weanlings were subjected to complete gross microscopic pathology.

Observations on the progeny included population data, survival, body weights, and reactions and abnormalities. The parental animals were observed for body weights, mortality, reactions, hematologic studies, urinalysis, gross and microscopic pathology of organs and tissues, organ weights, and reproductive performance.

The results demonstrated no significant differences between control and test animals fed Reten 220.