Acrylic Acid
Emergency Restabilization
with RESTAB™ Technology
Summary

BASF has developed a new technology for emergency restabilization of acrylic acid, with the goal of enhancing the safety of bulk storage facilities. RESTAB™ is being made available world-wide as part of our on-going Responsible Care® commitment. RESTAB™ meets all requirements for emergency devices and is designed as a possible second defense for situations where primary safety control devices have failed or where unforeseen circumstances, such as fires, have created a critical situation.

Outstanding features of RESTAB™ include the low weight and volume necessary to mitigate a suspected runaway polymerization. Various size mobile units are available. A 13 gallon dolly mounted unit is available for tank sizes up to 28,000 gallons and a 30 gallon dolly mounted unit is available for tank sizes up to 65,000 gallons. Trailer mounted units are available for larger tanks.

RESTAB™ is self contained and operates without the assistance of external energy sources or mixing devices. It is fast, economic, and is initiated using a simple release system from sheltered locations and safe distances.

The RESTAB™ system uses a solution with a high flash point and low toxic potential for humans and for the environment.

Two options are described for connecting RESTAB™ to individual tanks which are endangered. In one case some steel work inside the tank may be necessary. Another version involves mounting an expandable lance on the tank roof. A spare flange is the only prerequisite for this option. Lay out suggestions are available for transfer lines and coupling points.

While BASF recognizes that emergency restabilization is never a substitute for good preventive safety programs, use of RESTAB™ may minimize the impact of an unscheduled polymerization. In view of the fact that RESTAB™ will be used in critical situations, there can of course be no guarantee that it will minimize or control any particular polymerization. Many factors affect processing, application, or use. The potential user of this new technology should feel free to contact a BASF Product Steward listed on the back cover of this brochure for detailed information and guidance.

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Introduction

A risk is defined by two parameters: The probability and the impact, hence the risk is:

$$\text{Risk} = \text{Probability} \times \text{Impact}$$

Since the total risk potential may be low with one of the parameters being low the risk potential can be minimized by reducing either one or both the parameters.

In conventional risk assessments of acrylic acid storage facilities the probability factor is commonly minimized by various temperature control devices suitable to prevent inadvertent polymerization. Until very recently, manufacturers of acrylic acid assumed they had reached safer bulk storage conditions through these measures. However, careful analysis of acrylic acid incidents have led manufacturers to the conclusion that a residual risk potential for runaway polymerizations persists. In many cases it can be attributed to human failures. Certain unforeseen circumstances may also contribute to a residual risk, such as may arise from fires or catastrophes.

Considering that the impact of an uncontrolled polymerization, in particular of acrylic acid, may be very violent with extreme rates of temperature increase and build-up of pressure, the minimization of the risk potential has now expanded to searching for options to also reduce the impact factor—even though the likelihood of such an event is very small.

BASF recently developed an emergency intervention system which can minimize the impact of an uncontrolled polymerization. It can be used as a secondary prevention device for acrylic acid storage installations. The system is called RESTAB™, its name pointing to the goal of restabilization.

The RESTAB™ Concept

Radical polymerization can be suppressed by substances called inhibitors. A common example is MeHQ which is a typical inhibitor for transport and storage. It is called aerobic, since it requires oxygen to be fully effective. Anaerobic inhibitors in contrast are effective in the absence, though not necessarily under exclusion, of oxygen. Phenothiazine (PTZ) is a typical representative of anaerobic inhibitors and one of the most effective known\(^\text{(1)}\). Concentrations of 200 to 300 parts per million (ppm) are considered to be capable of completely stopping an uncontrolled polymerization of acrylic acid—even one in a progressed state. The RESTAB™ concept is based upon the highly active PTZ inhibitor.

<table>
<thead>
<tr>
<th>Chemical Formula</th>
<th>C(<em>{12})H(</em>{9})NS</th>
</tr>
</thead>
<tbody>
<tr>
<td>CAS</td>
<td>92-84-2</td>
</tr>
<tr>
<td>Melting point °Celsius</td>
<td>183-185</td>
</tr>
<tr>
<td>Boiling point (27 mbar) °Celsius</td>
<td>235</td>
</tr>
<tr>
<td>Decomposition temperature °C</td>
<td>&gt; 250</td>
</tr>
<tr>
<td>Toxicity</td>
<td>low toxic</td>
</tr>
<tr>
<td>Dermal Toxicity</td>
<td>non irritant</td>
</tr>
<tr>
<td>Aquatic environment</td>
<td>very little soluble</td>
</tr>
<tr>
<td>Solubility in acrylic acid w.%</td>
<td>2</td>
</tr>
</tbody>
</table>

Table 1: Properties of phenothiazine (PTZ)

![Specific gravity of PTZ solutions in NMP](image)

**Figure 1:** Specific gravity of PTZ solutions in NMP
BASF conducted a detailed investigation into the PTZ compound and the delivery system to make available a complete technology and to ensure its practical application. This investigation focused on five key issues: Reliability—Mobility—Ergonomics—Energy usage—Economy. Summarizing the results of the investigation, the RESTAB™ system:

- operates under extreme conditions,
- minimizes accidental activation,
- is non-dedicated and mobile,
- is simple to use with only two armatures to operate,
- requires no external energy source,
- is self-sufficient in regard to mixing technique,
- requires minimal investment costs and tank modifications, and
- requires minimal maintenance.

The PTZ Compound

Phenothiazine is a widely used chemical in the acrylic monomer industry. No particular hazards are involved with using the material under normal industrial hygiene conditions. Some of the more important properties of PTZ are listed in Table 1.

A characteristic feature of PTZ is its low solubility in most common solvents. A key task for BASF therefore was to identify solvents providing a high load solution and capable of meeting other requirements needed for the anticipated technology. Among the tested solvents, one in particular met the property profile for the intended use: N-methyl pyrrolidone or NMP(2). The NMP performance is shown in Table 2.

The evaluation of the viscosities and specific gravities of PTZ solutions as shown in Figures 1 and 2 was important for the specification of the most appropriate PTZ solution. A 50 percent weight solution was judged to best meet specific gravity and viscosity needs at low temperatures. It allows filling of the recipients for the emergency solution with a nearly 15 percent higher volume and even at cold weather conditions the 50 percent solution will flow freely. At ambient temperatures the viscosity resembles a light syrup.

<table>
<thead>
<tr>
<th>Solvent Property</th>
<th>Solvent Property Profile</th>
<th>NMP-Performance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solubility</td>
<td>minimum 40 %</td>
<td>&gt; 55 weight %</td>
</tr>
<tr>
<td>Boiling point</td>
<td>&gt; 150° C</td>
<td>&gt; 200° C</td>
</tr>
<tr>
<td>Flash point</td>
<td>high</td>
<td>91° C</td>
</tr>
<tr>
<td>Viscosity</td>
<td>low</td>
<td>see Table 4</td>
</tr>
<tr>
<td>Reactivity</td>
<td>no reaction with acryl acid</td>
<td>no reaction with acryl acid</td>
</tr>
<tr>
<td>Toxicity</td>
<td>no toxicity to humans and to environment</td>
<td>toxicity 2000 mg/kg rat non-carcinogenic non-mutagenic low environmental hazards biodegradability &gt; 90%</td>
</tr>
<tr>
<td>Availability</td>
<td>commercial chemical</td>
<td>commercial, world-wide</td>
</tr>
<tr>
<td>Reprocessability</td>
<td>reprocessable in BASF monomer processes</td>
<td>reprocessable in BASF monomer processes</td>
</tr>
</tbody>
</table>

Table 2: Solvent property profile and NMP-performance

![Figure 2: Kinematic viscosity of PTZ in NMP](Image)
Technology

The high load and low viscosity PTZ compound of 50 weight percent was the basis for further development of a technology. An average concentration of 250 parts per million of phenothiazine was another guiding value to establish appropriate lay out data. This concentration is assumed to be capable of stopping a heat induced polymerization and is likely to mitigate a radical initiated reaction. Experimental verification is difficult but most manufacturers agree that 200 to 300 ppm PTZ will stop polymerizations appropriately. This concentration is based on technical experiences in monomer processes and in inadvertent polymerizations.

The Emergency Solution Recipients

Because a 50 percent weight solution of PTZ in NMP can be achieved, emergency solution recipients can be kept small and mobile. The advantage of non dedicated, mobile RESTAB™ units is obvious. In multiple tank storage installations with several tanks and in different locations only a small number of RESTAB™ units are required. The cost savings may be considerable compared with dedicated systems. For routine inspection, recipients can be replaced by new units while the original is recharged and inspected.

The Table 3 exemplifies the relationship between tank sizes, PTZ demand and RESTAB™ recipient sizes.

<table>
<thead>
<tr>
<th>Tank Volumes (1,000 gallons)</th>
<th>5</th>
<th>10</th>
<th>25</th>
<th>50</th>
</tr>
</thead>
<tbody>
<tr>
<td>PTZ-Concentration [ppm]</td>
<td>250</td>
<td>250</td>
<td>250</td>
<td>250</td>
</tr>
<tr>
<td>PTZ-Mass [lbs]</td>
<td>11</td>
<td>22</td>
<td>54</td>
<td>109</td>
</tr>
<tr>
<td>50%-Solution Mass [lbs] *</td>
<td>22</td>
<td>44</td>
<td>109</td>
<td>218</td>
</tr>
<tr>
<td>Volume PTZ Recipient (gallons)</td>
<td>2.5</td>
<td>4.5</td>
<td>11.5</td>
<td>23</td>
</tr>
</tbody>
</table>

* referred to the specific gravity of the 50 weight percent solution

Table 3: Tank volumes and RESTAB™ recipient sizes

A dolly mounted unit is shown in Figure 3. It is completely equipped with a self-sealing coupling system (see section on Coupling Devices) and a gas bottle sufficient to convey and mix in the tank the PTZ compound through the target storage tank. The equipment is available at low cost.

Fully automatic restabilization systems are reported to have failed repeatedly and have caused false launches. This may generate high costs for rendering the storage facility and material unusable. Therefore the RESTAB™ system is intentionally designed for a manual launch.

Shelf Life of the PTZ Compound

Phenothiazine in N-methyl pyrrolidone is a fairly stable mixture. Accelerated ageing tests at 60°C have shown that the loss of activity over a period of 6 months is only marginal. A minimum shelf life of 6 years is expected after which the solution should be replaced. Replacement can be scheduled jointly with the required maintenance and pressure check of the RESTAB™ units.

Gas Mixing Technology

BASF designed the RESTAB™ unit to be completely independent from energy sources and mixing installations attached to the tank.

In a worst case scenario the beginning of an unintentional polymerization could coincide with the interruption of the electrical power supply. For example, a fire in the tank area could result in the subsequent destruction of electrical cables. In the case pumps would not be available for the transfer of short stopping solution into the target tank thus rendering unusable most emergency devices. Furthermore, a number of reports on unintentional polymerizations indicated that stationary pumps for discharge and revolving purposes had become themselves the source of a polymerization. All cases were related to heat stressing of acrylic acid. Under such circumstances, a typical transfer of restabilization solutions and mixing would be impossible. This is the reason why RESTAB™ is designed as a fully self-contained system.
The BASF Department of Technological Research and Development recently conducted investigations on the fluid dynamics and mixing characteristics of continuous gas streams of liquids in storage tanks of different sizes and shapes. These investigations indicated that a high homogenization rate can be achieved with comparatively low gas volumes and in a comparatively short period of time. The mixing mechanism is induced by air bubbles rising to the surface and generating a vertical, convective circulation of the tank contents, provided that the viscosity was sufficiently low.

The study results have a direct application in ensuring the RESTAB™ solution is thoroughly mixed throughout the target tank. Initially, this was achieved by stationary compressed gas containers. However, with the low volumes of the RESTAB™ required for restabilization, BASF developed a mode to combine gas supply bottles with the PTZ solution recipients described above to meet the total propellant and mixing gas demand.

Certain supplementary assumptions—where necessary with respect to the feed time and the length of the feed line. From studies on heat released during the polymerization of glacial acrylic acid it was established that there existed a time window of several hours for a successful restabilization. Subsequently, injection and mixing was assumed to begin within 30 minutes of positively identifying an inadvertent polymerization.

For the length of the feed line, BASF assumed that no individuals should be endangered during application and that the coupling point of the RESTAB™ unit would be kept a safe distance from a target tank. A total length of 330 feet was stipulated and the feed line should always end behind some solid construction.

The diameter of the feed line greatly influences the feed time. The diameter is limited to two parameters: pressure necessary to move the inhibitor solution and losses of the PTZ compound adhering to the line surface. Calculations show that for all tanks up to 26,000 gallons, a diameter of 1 inch is appropriate. Material losses are compensated by excess material in the RESTAB™ recipients. Similar calculations for tanks up to 364,000 gallons suggest diameters of up to 2 inches.

Table 4 shows the relationship of feed line and actual feed times. Calculations assume a 50 percent weight solution of PTZ at +10 and -10 degrees Celsius. The feed pressure was assumed to be 6 bars—corresponding to the normal operational pressure for the recipients. Feed times depend also on recipient sizes.

For minimizing material losses, the vertical branch of the feed line is suggested to be designed with half the diameter of the horizontal branch if the latter has diameters < 1 inch. Feed times are short compared with mixing times. Properly dimensioned orifices in the RESTAB™ units ensure that an optimal gas flow is maintained subsequent to the injection of the inhibitor solution.

The relationship of tank volumes and shape versus the required total gas volumes at atmospheric pressure to achieve homogenization rates better than 90 percent are shown in Table 5.

<table>
<thead>
<tr>
<th>Task Volume (000 gallons)</th>
<th>10</th>
<th>50</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ratio H2O</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>Mixing Time, [minutes] 95%</td>
<td>6.8</td>
<td>5</td>
</tr>
<tr>
<td>Required Gas Volume, ft³ [STP]</td>
<td>17</td>
<td>8.5</td>
</tr>
<tr>
<td>Required Gas Volume, ft³ [SCFM]</td>
<td>17</td>
<td>7.4</td>
</tr>
<tr>
<td>Volume of Restabilization Solution (gallons)</td>
<td>45</td>
<td>23</td>
</tr>
</tbody>
</table>

Table 4: Feed times as a function of diameters of feed lines

Table 5: Tank volumes and mixing gas, mixing time data
**Coupling Device**

Different injection systems can be used in conjunction with the RESTAB™ unit (see section on Injection Devices). One end of the feed line will be attached to the injection device with the other end hermetically closed through use of a self sealing coupling armature supplied with the RESTAB™ unit. Each individual tank in a multiple tank farm must have a stationary feed line equipped with the male fitting of the coupling system.

Where feasible, all coupling points should be routed to a common location. Clearly visible labeling of the lines should prevent from injecting to the wrong tank during an emergency. Male and female fittings should be protected against dust with plastic caps even though the construction allows a safe manipulation also if the coupling is contaminated with dust or sand.

The female fitting is mounted on the pressure hose of the RESTAB™ recipient. Self sealing of armatures is effective up to a pressure of 6 bars. Since normal operational conditions of the RESTAB™ system is below this pressure it is safe to couple even if the gas bottles were opened erroneously prior to attaching the hose to the feed line.

Routine maintenance is not required for the coupling system. After use of the RESTAB™ unit, the O-rings and the flexible hose require replacement since the NMP solvent is aggressive to synthetic material. Relifting of the unit and replacement of the plastic parts can be carried out by the supplier. Figure 4 shows an example of a coupling system[7].

**Injection Armatures**

The design of injection armatures must consider the potential of polymer formation by direct contact with the monomer. Polymer may form in stagnant compartments of equipment due to the depletion of dissolved oxygen or through condensation of monomer vapors on cold surfaces. This material is essentially uninhibited and commonly polymerizes very slowly under formation of encrustations or stalactites. If not prevented polymer formation may render the emergency device unusable.

Alterations to the tank must also be considered since it is often undesirable to remove a tank from service to make technical changes. The RESTAB™ concept allows at least two options for installing injection armatures. They have in common that the injection devices enter the tank from the tank roof, where the feed line commonly ends. Entering points below the liquid surface are vulnerable and therefore considered potentially hazardous.

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Figure 4: Self sealing coupling system (7)
The Gas Purged Injection Pipe

As mentioned above, stagnant monomer in a non-ventilated compartment may gradually deplete in dissolved oxygen. More specifically, a tube reaching to the bottom of a tank can separate monomer from normal material exchange. Under such conditions, the tube may be blocked partially or completely if not regularly flushed.

A simple injection armature can be constructed which is protected against polymer formation. Most suitable is continuous purging with a low flow of compressed air. The air supply should be controlled and alarmed to maintain a reliable gas stream. Provided that the gas stream will never be interrupted, this simple device can serve as a reliable injection armature. Since the feed line attached to the injection armature is hermetically closed by the male coupling fitting, gas escapes through the injection conduct. The air supply line should be protected by a check valve to prevent flow of the PTZ compound into the line.

During loading and recirculation of the tank contents, considerable shear forces may act on the built-in armatures. Therefore, it is necessary to stabilize the injection conduct firmly by attaching it to the tank wall. A suggested schematic of the injection armature is shown in Figure 5.

Maintenance work is not required for the purged injection pipe except for the control armatures. An inspection should be completed as a part of the routine tank inspection.

Installing a purged injection pipe may require clean-out of the tank while the unit is out of service for some time. This may become unacceptable in certain cases. The RESTAB™ system can be connected to an air purged differential pressure level indicator if such an indicator is already installed on the tank.

The Telescopic Extension Lance (TEL™)

BASF has designed an alternative method of injecting the PTZ compound which does not require mechanical work inside the tank. The Telescopic Extension Lance is a fully automated device, which is positioned above the monomer surface and protected against vapor and liquid monomer while not in use. The TEL™ is mounted on the tank roof through use of a spare flange of 4 inches in diameter. The tank may remain in service if the flange is available. The apparatus can extend to a total length of approximately 6 meters corresponding to total tank volumes up to 40,000 gallons. Custom lances up to 33 feet length are available also for large tanks (264,000 gallons). The core of TEL™ consists of several extendable, concentric, telescopic tubes. The end of the inner tube is closed by a rupture disc, adjusted to the proper operational pressure, normally 50 psig.

[Diagram: Injection armature as air purged open pipe]

Figure 5: Injection armature as air purged open pipe
After coupling and injecting the RESTAB™ unit the feed line is pressurized and the lance is activated automatically. A pneumatic lever system releases the locking device on the concentric tubes. With increasing pressure the TEL™ extends towards the tank bottom to a pre-set halt position. The latter should be around 0.5 to 1 metre above the tank bottom. If the pre-set halt position is reached the pressure inside the lance will gradually increase to the rupture disc pressure setting. The PTZ compound and mixing gas are injected and the tank contents are restabilized. Since the feed line is hermetically closed from both sides, contaminants cannot enter into system. The lance head is protected with a plastic cap in the idle position.

The TEL™ should be routinely inspected by unloading the unit from the feed line and from the roof flange. If necessary the rupture disc can easily be replaced in the dismantled position. After use the TEL™ can be repositioned by a rewinding device. The device is shown in Figure 6.

A schematic view of the device mounted on a tank is shown in Figure 7.

Rules for Using the RESTAB™ System

The RESTAB™ system is designed as a potential second defense for situations where primary safety control devices have failed or where unforeseen circumstances have created a critical situation. It should never be a substitute for good preventive safety programs.

Restabilization with PTZ inhibitor is an appropriate emergency response for an uncontrolled polymerization such as might be caused by accidental overheating or accidental contamination with initiator type materials, or such as might be caused by robust pool fires.

Early detection of unsafe conditions inside or around the tank is a key task for a timely restabilization. All tanks should be equipped with redundant temperature monitoring determining the temperature and a heating rate. A high temperature alarm to detect a overheating is highly recommended and should be mandatory. Storage temperatures greater than the industry-recommended target value of 15 to 25 degrees Celsius or an unexplained temperature increase are indications that dangerous conditions may be developing.

Figure 6: Telescopic Extension Lance
Criteria for Injecting the RESTAB™ System

Scenarios which may lead to incipient polymerizations may be very variable. They range from destabilization due to a lack of oxygen or inhibitor, to a vigorous pool fire around the tank. In all cases an early detection and timely response provide the best chances to bring the situation under control. Contamination scenarios are difficult to judge. The consequences depend mainly on the character and quantity of the contaminant. With gross amounts of initiating material there can be no assurance for the RESTAB™ system to work. Table 6 gives some guidelines on conditions under which restabilization should be actuated.

Final Remarks

Over several years BASF has devoted significant resources to a Product Stewardship Program to among other things promote safer processing, handling, and transportation of acrylic monomers including acrylic acid. BASF is making available world-wide the RESTAB™ as part of its on-going Responsible Care® commitment.

Potential users of this technology are encouraged to contact a BASF Product Steward listed on the back cover for information regarding RESTAB™. BASF can also provide the addresses of suppliers for the RESTAB™ elements. BASF is available to share its experience with suppliers, material of construction, and the design criteria used in its own restabilization systems.

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Table 6: Criteria for launching the BASF-RESTAB™ system

<table>
<thead>
<tr>
<th>Scenario for Destabilization</th>
<th>Criterion for Actuating</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Destabilization by depletion of oxygen and/or inhibitor concentration</td>
<td>T &gt; 40°-45°C (depending upon temperature/water system) or self-heating rate of &gt; 5°C per hour</td>
<td>Overstorage, inert gas purge. High storage temperatures or lack of available oxygen</td>
</tr>
<tr>
<td>Contamination with material which is known to promote polymerization</td>
<td>unconditional</td>
<td>Contaminants are: peroxides, acid, nitric acid, mineral acids, ethers, aldehydes, metal salts</td>
</tr>
<tr>
<td>Contamination with other material of unknown interaction</td>
<td>unconditional or temperature rise is observed (any rate)</td>
<td>Call BASF for consultation</td>
</tr>
<tr>
<td>Robust fire conditions pool fire or tank fire</td>
<td>immediate and unconditional</td>
<td>preventive</td>
</tr>
</tbody>
</table>

Figure 7: Telescopic Extension Lance as injection armature
Bibliography


(2) US Pat. Application No: Ser. 08/96 0356; (1997), BASF.


(4) By Courtesy of TOTAL-Feuerschutz GmbH, D-68526, Ladenburg, Germany.


(7) By Courtesy of Roman Seiger Armaturenfitabrik GmbH D-22848 Norderstedt, Germany

(8) Germ. Pat Applic. No. 19749859.0 (1997), BASF.

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