

The grant requires DuPont Merck to make available the results of the demonstration project. These results were presented in May at two seminars organized by the New Jersey Technical Assistance Program for Industrial Pollution Prevention.

Ultrasonic cleaning of reaction tanks and reactors is simple, says Angelo C. Piro, president of the U.S. subsidiary of Telsonic Ultrasonics, the Swiss producer of ultrasonic tubular resonators. A tank to be cleaned is filled with water, the water is heated to 150 °F, and 2% surfactant by weight is added. Ultrasonic power is applied for two to four hours, depending on tank size and contaminant type, and then the water is drained.

As environmental manager for DuPont Merck's R&D division until the end of April this year, Piro tested the tubular resonator in reactors ranging in capacity from 20 to 300 gal. Depending on tank size, one to three resonators were used, and cleaning times ranged from two to four hours. In Europe, however, trials have been carried out on 2,000-gal tanks, says Piro. For this capacity, six resonators were required.

Conventional cleaning requires refluxing of a solvent, usually methanol, inside tanks for four to eight hours, explains Piro. The vessel is half filled with solvent and then heated to the solvent's boiling point. At the end of the reflux period, the solvent is cooled and drained, and a sample is analyzed. The procedure is repeated until analysis shows an acceptable level of contaminants in the solvent. This process can take up to five cycles, says Piro. And spent solvent then has to be disposed of by incineration.

With the ultrasonic tubular resonator, cleaning usually takes only one cycle, DuPont Merck tests show. For a 300-gal vessel typically cleaned with methanol, savings amounted to almost \$4,900 per cleaning, representing the cost of methanol, energy for refluxing, labor, and methanol disposal. Other savings due to reduced vapor emissions on site and fewer hazards to workers also are realized. For the same vessel, the cost of the tubular resonators—about \$27,000—is paid off after six cleanings, says Piro.

Use of solvent is not completely eliminated, explains Mary F. Cooke, director of chemical process R&D facili-

Ultrasonic cleaning cuts waste, energy use

The ultrasonic tubular resonator, a device for removing surface contamination, is making its way to chemical process tanks. Cleaning tanks and reactors with this device is less expensive, less energy intensive, and more environmentally friendly than with conventional cleaning methods, according to results of a demonstration project by DuPont Merck Pharmaceutical.

DuPont Merck has been testing the technology at its facilities in Deepwater, N.J., under a grant from the National Industrial Competitiveness through Energy, Environment & Economics (NICE³) program. NICE³ is a joint cost-sharing grant program of the Department of Energy and the Environmental Protection Agency.

ties at DuPont Merck. But much smaller amounts are required, primarily to remove traces of surfactant and to verify that contaminants were adequately removed, she says. A solvent cycle is also needed to remove any water in the tank and to clean overhead piping and condensers, Piro adds.

At the heart of this technology is an ultrasonic transducer, which converts an ac voltage signal into high-frequency sound waves. The transducer is connected to a tubular resonator, which is immersed in the tank to be cleaned. High-frequency sound waves are produced in the liquid by the transducer, initiating the process of cavitation—the formation, growth, and implosive collapse of bubbles in a liquid.

As sound passes over a bubble, the expansion wave causes the bubble to expand a little, and the compression wave causes the bubble to compress somewhat, explains chemistry professor Kenneth S. Suslick of the University of Illinois, Urbana-Champaign, who specializes in sonochemistry. So the bubble oscillates. The pumping action brings in gas, causing the bubble to grow slowly. Eventually, the bubble reaches a so-called resonant size, at which point it can grow rapidly and then collapse in a single sound cycle.

When near an extended surface, such as a reactor wall, a bubble collapses asymmetrically. It compresses faster away from the wall than near the wall, generating a dimple on the back side that accelerates. "You end up forming a high-speed liquid jet that comes smashing through the bubble into the surface at a speed of a couple of hundred meters per second," Suslick says. The jet helps to cause the scrubbing action that cleans the tank wall. And the shock wave of the collapsing bubble results in a wiping action.

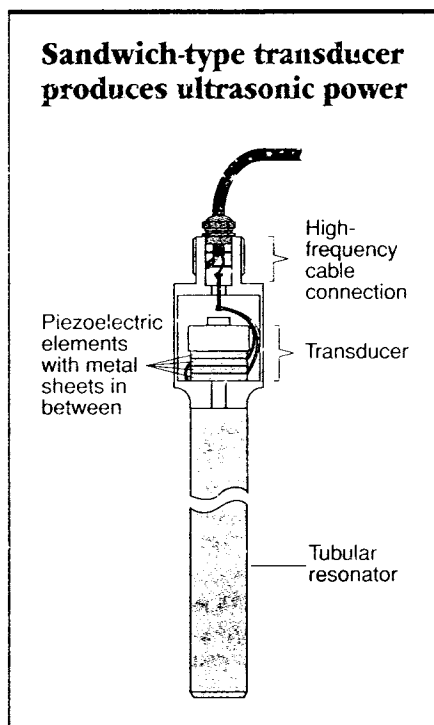
During ultrasonic cleaning, the water is heated because the higher temperature allows sound waves to travel faster, says Piro. The warmer water also enhances the cleansing ability of the surfactant, even though surfactant is not added for cleaning. Rather, it is used to decrease the water's surface tension so that waves can travel more easily. Surfactant also helps suspend contaminants and prevent them from being redeposited on the walls.

Ultrasonic cleaning is not new, explains Piro. It is used for materials with irregular shapes and hard-to-reach ar-

eas such as airplane engines and electronic components. But earlier transducers propagated waves in only one direction. To clean tank surfaces completely, one must reposition the apparatus at various intervals or use several units in different orientations. Either solution is impractical for reactor vessels. What Piro sought was an omnidirectional transducer.

Telsonic Ultrasonics already was making the device Piro needed, but the company had not thought of using it to clean chemical process vessels. When Piro heard of Telsonic, he got help from the New Jersey Division of Energy Planning & Conservation to obtain a NICE³ grant for a project to determine if Telsonic's tubular resonator could be used to clean reactors.

Sidney L. Palius, energy program representative at the New Jersey Board of Public Utilities, has been managing the DuPont Merck NICE³ grant, which started Oct. 1, 1992, and will end Dec. 31 this year. He tells C&EN: "DuPont Merck identified a problem, and they were looking at ultrasonics to solve it. We're excited and very pleased with [the ultrasonic tubular resonator] because it represents a dramatic reduction in production of hazardous wastes and in energy consumption." DuPont Merck and the utility board received DOE technology commercialization awards for this project.



DuPont Merck uses the technology to remove drug intermediates for developmental pharmaceutical products. So far, the company has found only one problem. Erosion marks measuring 1 mil (0.001 inch) have appeared in walls of some of the glass-lined reactors at DuPont Merck. Cooke says they have been trying to re-create the conditions leading to them but so far have not succeeded. Until they are explained satisfactorily, DuPont Merck will restrict use of the resonators only to metal vessels, she adds.

Piro suspects the erosion marks were caused by a surfactant batch with an unusually high amount of phosphates, which can react with glass, particularly in combination with the sonochemical effects of the resonator. But there is no analytical proof to support Piro's hypothesis, notes Cooke. Suslick, however, believes the marks are due simply to microjet impacts on the surface, causing localized erosion.

Two companies contacted by C&EN have contrasting results when using the resonator to clean paint tanks. One company, which requested not to be identified, says the device did not clean processing tanks for coatings sufficiently in the times thought reasonable. But the company explains it may not have optimized conditions for use nor exhausted possibilities for making the technology work. The company also suggests that its use of unpolished carbon steel tanks, instead of stainless steel tanks, could be a significant factor in its inability to get acceptable results from the tubular resonator. The company says it is using its resonators for other applications, which were not disclosed.

On the other hand, DuPont's process engineering department in Philadelphia tells C&EN the technology has been successful in cleaning 150-gal tanks used to prepare dispersions for automotive paint products. A supervisor in that department says savings of up to \$150,000 per year can be realized with this device.

Other companies are expressing interest in the technology. As one environmental affairs manager for another pharmaceutical company puts it, the combination of less solvent being used and less hazardous wastes being generated is a win-win scenario for pollution prevention.

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