## Scanning the Industry

## Finding Contaminants and Keeping Them Out!

By Phaedra E. McGuinness, Managing Editor

In the oil refining industry, keeping contaminants out is big business. According to Manager of Diagnostic Research, Cathy Vartuli, Intercat, of Savannah, Georgia, has the tools to do just that.

Intercat is a manufacturer of catalysts and sorbents for use in the petroleum refining industry. Some catalysts are used to selectively crack heavy hydrocarbon feed oils to lighter, more valuable products, such as propylene, gasoline and diesel. Other products are used to reduce the elements of contaminants found in the feed oil, such as sulfur oxides, nitrogen oxides, carbon monoxide and sulfur compounds in gasoline. All of these compounds result in pollutants which are harmful to the environment. Although not the worst offenders, petroleum refineries are considered a major point source of emissions.

Originally enacted by the Environmental Protection Agency (EPA) in 1970, The Clean Air Act created a national program to control the damaging effects of air pollution. Twenty years later the EPA went even further to ensure the air everyone breathes is safe, by enacting several limits to emissions of stationary and mobile sources of pollutants. (www.epa.gov).

One of Intercat's environmental catalysts is used to reduce the amount of sulfur oxides emitted from the Fluid Catalytic Cracking (FCC) process within most modern oil refineries. Sulfur oxide molecules are bound to the catalyst, thus preventing sulfur molecules from being reduced into the atmosphere and contributing to air pollution. Current EPA mandates limit sulpher oxide (SO×) emissions from FCC units to as low as 25 parts per million (ppm).

Analysis of the spent catalyst from the refining process that contains the sulfur oxide binding catalyst is critical in evaluating the performance of the catalyst and assisting in the development of next-generation products. Figure 1 shows a scanning transmission electron microscope (STEM) image of a spent catalyst particle obtained from the FCC refining process. This particle contains differences in elemental constituents due to a combination of starting catalyst composition as well as contaminants from the feed oil such as sulfur. Although the STEM is able to give relative compositional information through phase contrast, Vartuli relies on energy dispersive spectroscopy (EDS) to determine qualitative and quantitative elemental concentrations. Her team works with two EDAX systems (Mahwah, N.J., USA); one on an Hitachi HD-2000 STEM and another on an Hitachi S-4800 SEM. "In the past, I supervised a group of 12 people all running EDS systems. I've found EDAX products to be straightforward and easy to learn," Vartuli says.

According to EDAX SE salesperson Tara Nylese, "Rapid crystallographic sample scans that used to take months can



FIG. 1 Scanning transmission electron microscope image of a thin slice of refining catalyst from a spent catalyst particle obtained from the fluid catalytic cracking process.

now be accomplished in a matter of hours." Vartuli also appreciates the ability to perform a theoretical overlap. "You can miss something, for example, in semiconductors with tungsten plugs in a silicon matrix when it overlaps with the K and M peaks," she says. "The tungsten has higher energy peaks, but if one is looking at small particles that are contaminating the system, this may not be an option, and it's important to know what's in it, where it came from, and back-track from there."

Contamination can enter an environment through tool corrosion, a bad gas line, feed stock, or additives in the pollutant. According to Vartuli, "Finding a contaminant is like playing a game of Clue. You find out what you can and work backwards from there." Prior to her work at Intercat, Vartuli worked in the semiconductor industry. She said that, while the oil refining industry is challenging, the tools perform in the same manner. The EDS system used at Intercat costs approximately \$100,000, but there are less expensive versions with varying sensitivity and resolution.

The EDAX detector incorporates several techniques during detector manufacture to increase the functionality of the systems. The ice prevention design allows researchers to forget about the problem of ice build-up. "When small particles of ice form, it causes microphonics. This can increase noise and lead to work delays–with EDAX this is a nonissue, and that's really nice because we have enough to worry about," Vartuli commented.

As new impurities are introduced, researchers will continue to turn to industry for an array of equipment that locates contaminants and helps keep them out.

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