

MONITORING TBC - A POLYMERIZATION INHIBITOR BY UV ABSORBANCE



The Process

TBC (*tert*-Butyl Catechol) is usually added as a stabilizer and an inhibitor of polymerization to butadiene, styrene, and other reactive monomers. The concentration of TBC needs to be regulated at approximately 100ppm. A TBC concentration drop below this level might result in a spontaneous polymerization in pipes or shipping vessels that will require mechanical removal of the polymer and a down time for the process. The downside of working with TBC at concentration levels above the optimum is the waste of an expensive additive, as well as the difficulties that might arise during the polymerization process.

The Method

TBC shows distinct UV absorbance features, the intensity of which can be correlated to the concentration of the TBC in the process. In this application TBC in butadiene and trace amounts of toluene were

measured by an industrial diode array spectrophotometer. The significance of monitoring a complete spectrum is two fold: first, other impurities interfere with the measurement and need to be corrected for. Second, a complete spectrum overcomes the difficulties in obtaining pure liquid butadiene for blanking and calibration.

Pure liquid butadiene is very difficult to prepare and store at normal temperatures and pressures and therefore can not be used in the blanking calibration and validation procedures. Alternatively, pure isopropanol was used for blanking, and mixtures of TBC in isopropanol were utilized for calibration and validation. There are, however, some dissimilarities between the absorbance spectrum of TBC in butadiene and that of TBC in isopropanol. Analyzing the complete spectrum and correcting for the discrepancies allows for the use of isopropanol as an alternative to the butadiene for blanking, calibration, and validation. The benefit of using isopropanol can be understood in light of the difficulties in handling the explosive butadiene in an industrial environment.

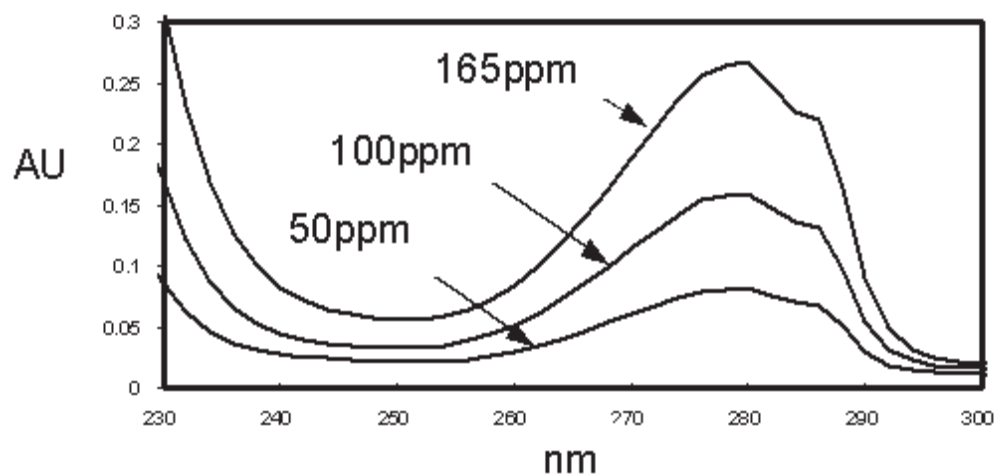
Standards were used for calibration in the 0-200ppm concentration range. A stream of TBC & trace amounts of toluene in butadiene is continuously monitored by a diode array spectrophotometer and the output - the concentration of TBC - is continuously updating the plant's main computer at 1 second intervals. The method accuracy is ± 1 ppm TBC.

The above UV spectroscopic method had replaced an HPLC system that was installed on the same fast loop. The HPLC system required high maintenance, had a significant amount of down time, and the readings for TBC were at 15 minutes interval (approximately 1000 readings of the spectrometer for a single HPLC measurement). The full spectrum method enables a faster tracking of changes in the stream, requires no maintenance and has practically no down time.

It was concluded that the diode array spectrophotometer with an appropriate background compensation method would replace an HPLC system, eliminating the need for the separation technique.

UV absorbance spectra

The following graph shows absorbance spectra of TBC (165, 100, 500 PPM) in isopropanol.



Method's Features

- Return on investment < 6 months in most plants
- Direct measurement - no sample preparation.
- Instantaneous response (<1 second)
- Continuous operation
- Automatic method determination
- Background correction
- Solid state hardware
- Low maintenance



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