

BRUKER APPLICATION NOTE

Magnettech ESR5000 for Monomer Control in Styrene Production

Magnettech ESR5000 – Electron Spin Resonance Monomer Control

Innovation with Integrity

Vinyl monomers such as styrene can undergo undesired polymerization reactions during the industrial processes in which they are synthesized and purified. To avoid this undesired phenomenon, polymerization inhibitors such as (2,2,6,6-tetramethylpiperidin-1-yl) oxidanyl (TEMPO) and its analogues are added to the monomer reaction system.

TEMPO (Figure 1.) is a free radical compound that is used to inhibit the polymerization reaction by stopping the free radical chain reaction (Figure 2.). Typical industrial monomers such as acrylates, vinyl chloride, butadiene and styrene require inhibitors for both processing and safe transport and storage. Many monomers are purified industrially by distillation, which can lead to thermally initiated polymerization. Styrene, for example, is distilled at temperatures above 100 °C whereupon it undergoes thermal polymerization at a rate of ~2% per hour.

It is important to suppress this reaction as the products can foul the fractionating tower, it is also typically exothermic which can lead to a runaway reaction and potential explosion if left unchecked. Once initiated, polymerization is free radical in mechanism and as such many polymerization inhibitors act as free radical scavengers (Kuldeep Wadhwa et. al J Polym Res (2017) 24:201).

The introduction of electron spin resonance (ESR), also referred to as electron paramagnetic resonance (EPR), allows

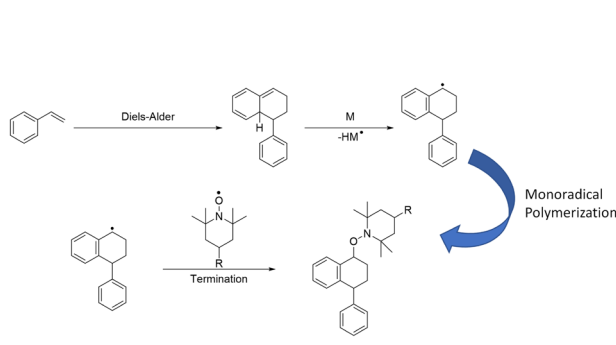


Figure 1: Structure for the TEMPO Free Radical Polymerization Inhibitor

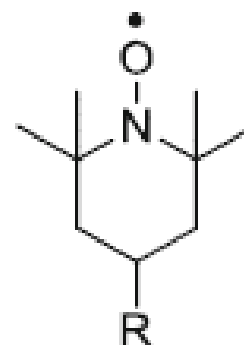


Figure 2: Mechanism for Polymerization Inhibition by TEMPO

direct measurement of TEMPO in styrene. This technique provides highly sensitive and quantitative data and gives actionable information on the TEMPO level in a styrene production vessel. Bruker's state-of-the-art benchtop ESR spectrometer, named Magnettech ESR5000, provides a rapid, quantitative and very compact instrument for monitoring TEMPO in a styrene production facility. The current TEMPO level is quantitatively monitored at various positions within the production process and proper actions are taken when levels decrease below a defined threshold. This technique introduces a significant cost-saving compared to adding TEMPO in excess whilst improving the overall safety of the production process.

The system package includes a detailed SOP and requires only very simple sample preparation. This involves only adding the styrene sample to a small tube and measuring with the Magnettech ESR5000.

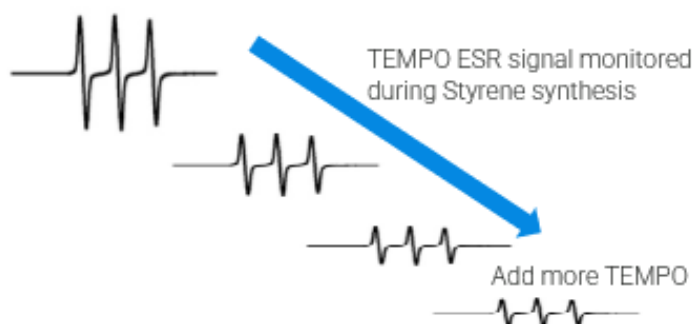


Figure 3: Monitoring of TEMPO EPR signal during styrene synthesis. TEMPO is added when signal intensity falls below a threshold value.



Benefits from the Magnettech ESR5000 Monomer Control Method

- Improved plant efficiency and product output
- Improved plant economics: Minimize inhibitor cost (TEMPO) and monomer loss
- Reduced lab expense: Simple monitoring system provides fast actionable data
- Improved plant safety: Reduces risk of "runaway" polymerization and subsequent downtime
- Increases production capacity and equipment preservation

Features of the Magnettech ESR5000 Monomer Control System

- Determine TEMPO levels in as little as 1 minute at concentrations as low as 0.5 ppm
- Includes SOP for simple sample preparation and measurement workflow
- Compact, lightweight with very small footprint
- Integrated automation and software for high throughput measurement and data analysis

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