DuPont™ EKC922™

Application Note #1

Crystallization Containment

Introduction

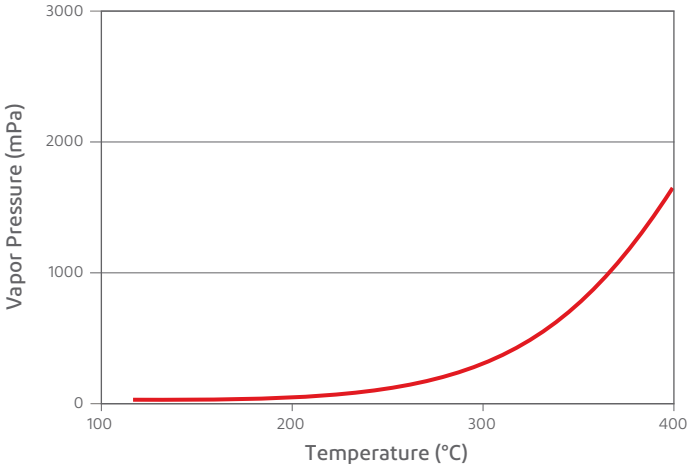
EKC922™ was commercialized in 1982. Sporadic appearance of crystals has been observed in EKC922™ since this time, as an inherent characteristic of the product. Originally this product was only offered in one gallon bottles. Generally, by simply shaking the bottle, the entire contents could be drained from the container and the crystals easily dissolved in the heated bath. Due to the very intermittent occurrence of the phenomenon, characterization of this physical change has been difficult. To date, the technical group at EKC has not been able to reproduce this crystallization event in a controlled, laboratory setting.

When crystallization events occur in a 50 gallon drum dispensing system, greater consideration must be taken in containment actions due to the complexity of the delivery system.

Table 1. Physical and Chemical Properties of Catechol

Boiling Point	245 °C
Water Solubility at 20 °C	31.2 g/100 g
Melting Point	105 °C

Figure 1. Vapor Pressure of Catechol[1]



Graph showing vapor pressure of neat catechol. The curve becomes steeper near normal operating temperatures for EKC922™. Vapor pressures for dissolved species are normally higher than for neat compounds, but these values have not been determined for catechol in EKC922™. However, this data illustrates the crystallization phenomenon seen under certain operating conditions with the product.

Analytical Capabilities

- Mobile metal ions DCP, GFAA, ICP-MS
- Anions via IC
- Component assays via GC and titration chemistry
- Physical properties characterized (e.g., refractive index, density, color and viscosity)
- Particulate analysis down to 0.2 µm

Potential Root Causes

- Extreme temperature swings can cause catechol to fall out of solution.
- Precipitates can build up from the drum unit to the day-tank, especially along the L-joints and locations whereby there is flow restriction.
- Non-heated BCD systems can cause precipitates to form.

Crystallization

Potential Containment Actions

Based on customer feedback and experience, listed below are certain potential containment actions that may help minimize crystallization in a bulk chemical delivery system. However, each customer needs to evaluate for itself the appropriateness and efficacy of using any of the potential containment actions in its own process or system. These potential containment actions are provided for your convenience only and EKC and Dupont make no guarantees or warranties of any kind with regard to the use or implementation of these potential containment actions, or their effectiveness, in your particular process or system.

For a Bulk Chemical Delivery (BCD) System

- Monitor flow rate for both the drum unit as well as the day tank periodically to detect drift early.
- Consider adding a strainer to the delivery system as a pre-filter.
- Make sure all filters, if used, are no smaller than 0.5 um. Most Fabs either use a coarse filter or do not filter at all.
- Change to point-of-use filter instead of circulation filter. This should extend the life of the filter.
- Flush lines with EKC854™ once a month to clear out crystals in the line.
- Heat parts of BCD system at risk for crystallization.
- Add in-line removable filter to drum supply.
- Add more filtration to production process.

For One Gallon Bottles

- With the cap securely on, shake the bottle vigorously and pour directly into a heated bath.

[1]VonNiederhausern, D. M.; Wilson, G. M.; Giles, N. F. Critical point and vapor pressure for Four Compounds by a Low Residence Time Flow Method. J. Chem. Eng. Data, 51 (6), 1986 -1989, 2006. 10.1021/je0602465 S0021-9568(06)00246-9 Web Release Date: October 17, 2006

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