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“1,2,4-Butanetriol Production at ATK – A Sustainable Solution”

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ATK Energetic Systems
Why Synthesis at ATK Energetic Systems

✓ History
  • ATK has operated RFAAP since inception and intends to be the operating contractor of the future
  • Recent history has shown small companies to be a risk of single point failure

✓ Commitment
  • ATK is dedicated to safety, quality, and excellence in all that we do
  • ATK has and will continue to support the DoD and NTIB contractors
  • ATK has invested heavily in upgrading & expanding facility capabilities

✓ Quality
  • ATK has a world class Specialty and Flexible Energetics Facilities
  • We met all quality requirements for BT every time
    – From initial small scale lab synthesis to 10l scale
  • Systems and talent in place to ensure quality is maintained through scale up

ATK Energetic Systems is committed to serving the warfighter: past, present, and future!
BT – What is it?

1,2,4-butanetriol (BT)

- 1,2,4-butanetriol (BT) is a straight chain polyol (similar to glycerin)
- Multiple synthetic routes to BT are known, but purification is key
- BT is the precursor to BTTN
- BTTN is an energetic plasticizer used in several propellants
- BTTN is lower melting and less sensitive than NG

BT is a simple molecule that is not so simple to obtain
The sad story of BT – an orphaned chemical

- Pre-2002: Avecia is qualified CONUS BT supplier
- 2002: Copperhead Chemical is re-qualified as BTTN supplier
- 2002-2003: Avecia sells business segment that produces BT to Cytec
  - Cytec assures Copperhead that BT production will continue
- 2003: Cytec discontinues BT production;
- May 2008: ATK Energetic Systems answers Sources Sought with a BT production solution
- November 2008: ATK Energetic Systems delivers high purity BT sample for evaluation
  - Purest BT ever evaluated by Copperhead Chemical
- March 2009: ATK begins BT process development and scale-up activities

Reliance on commercial sources outside of the NTIB puts the government at risk
ATK BT requirements

Meet or exceed Mil Spec requirements

High Purity >98% (GC)

Boiling point ca. 170°C at 8-10mmHg

No new trace contaminants

Optically inactive

Low cost reagents

Robust synthesis

• History of NG explosions from impure glycerin

Readily available materials and reagents

Readily scaled up in NTIB facilities

Plan to Identify and Meet Requirements First Time
ATK BT Process Summary

- Two step process is amenable to scale-up
  - Robust process with good yields

- BT from AES meets internal requirements

- BT process development and scale-up ongoing
  - Leveraging extensive ATK resources
  - Partnering with ABL on scale-up effort

- Rapid development at lab scale
  - Key proprietary purification step has been developed at AES
  - Several pounds of BT purified

DL-malic acid

\[ \text{1,2,4-butanetriol (BT)} \]

1. MeOH, H^+
2. NaBH_4
Esterification

Acid catalyzed esterification

- Choice of starting material, alcohol (methanol/ethanol), catalyst and reaction conditions

Desire

- Low cost reagents
- Rapid and complete reaction
- High reactor loading
- Simple work up

Result

- Low cost four carbon optically inactive natural product
- Clean reaction under simple conditions

Reaction optimized to rapidly give high isolated yield of pure ester
**Hydride reduction**

- Choice of reagents and conditions
- Wide range of solvents in literature

**Desire**

- Safe and complete reaction
- High reactor loading
- Simple work up

**Reaction optimized to give high purity in crude mixture and moderate isolated yield of pure BT**

- Crude BT may contain boron from reduction
- Expect yield to increase at larger scale
Lab studies complete

- Optimization of esterification at lab scale
- Optimization of reduction of ester at lab scale
- Purification of crude alcohol at lab scale

Scale up in pilot plant

- Esterification already conducted in pilot plant
  - No changes anticipated moving to 100 gallon reactor
- Reduction already conducted in pilot plant
  - Potential for further improvement in Pfaudler reactors
- Purification scale up to be done

Will scale all steps to 100 gallon by July 2009
• Drs. Jamie Neidert and Greg Drake (AMRDEC)

• Gregg Corley, Dr. Ron Clawson, and Dr. Scott Riley (ATK/ABL)

• Randy O’Brien and Peter Hartmann (ATK/RFAAP)

• John Schrader, Copperhead Chemical Co.