

# **Innovation ... Delivered.**

**2009 NDIA IM/EM Meeting  
Tucson, AZ – May 11-14, 2009**

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

**Dr. Andrew Sanderson, Dr. Steve Velarde  
ATK Energetic Systems**

# Why Synthesis at ATK Energetic Systems



A premier aerospace and defense company

## ✓ 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

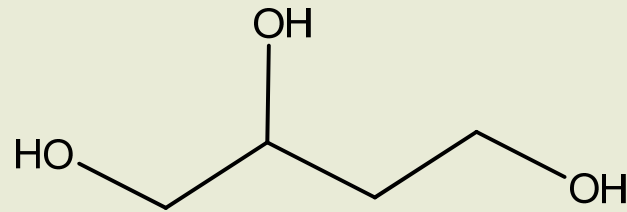
## ✓ Commitment

- 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!**



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**

**Meet or exceed Mil Spec requirements**

**High Purity >98% (GC)**

**Boiling point ca. 170C at 8-10mmHg**

**No new trace contaminants**

**Optically inactive**

**Low cost reagents**

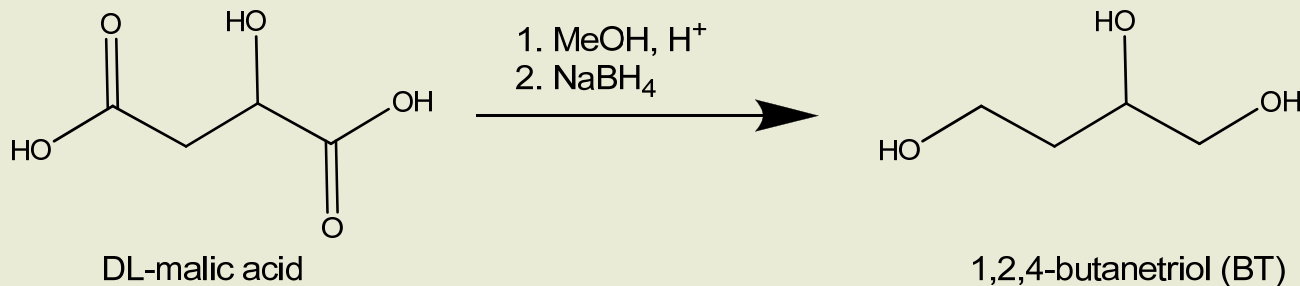
**Robust synthesis**

- 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**



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

- ✓ **BT from AES meets internal requirements**

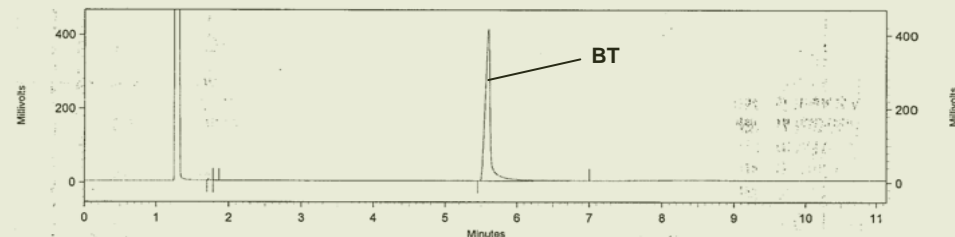
- ✓ **BT process development and scale-up ongoing**
  - Leveraging extensive ATK resources
  - Partnering with ABL on scale-up effort

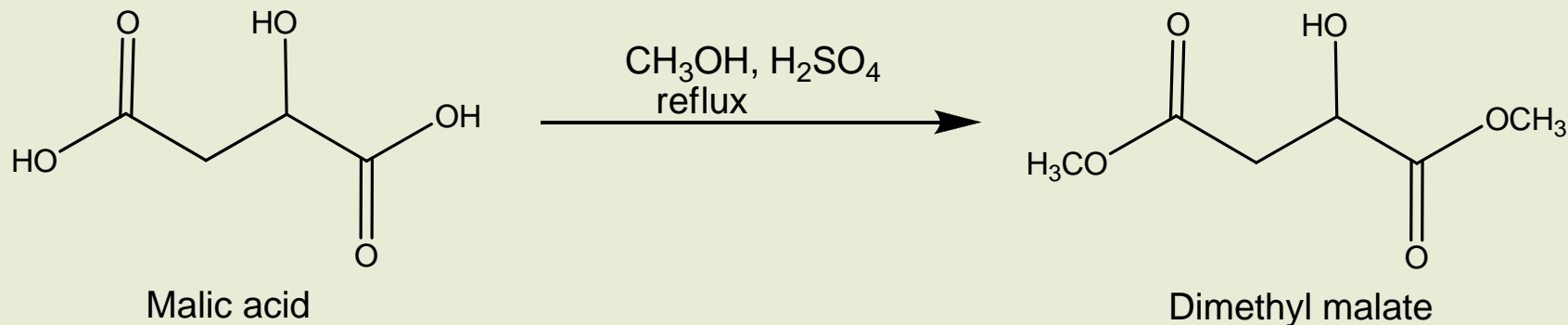
- ✓ **Rapid development at lab scale**

- Key proprietary purification step has been developed at AES
- Several pounds of BT purified

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## Acid catalyzed esterification

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

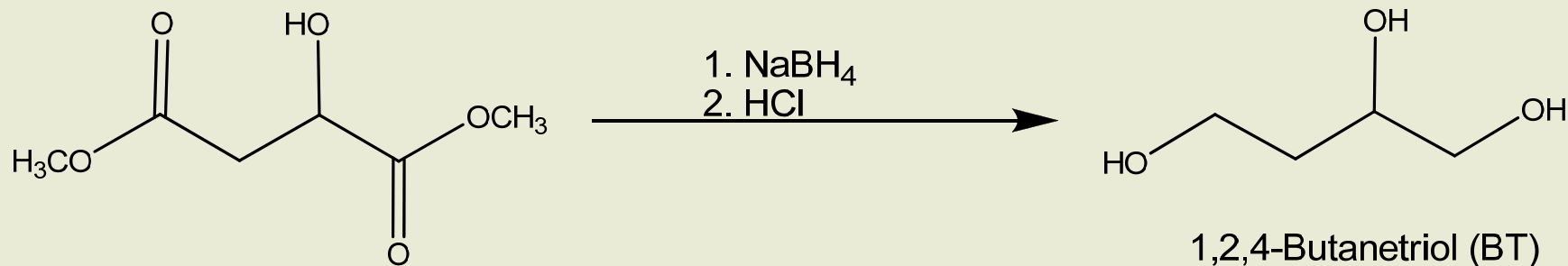
## Desire

- 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**



**All BT Requirements Met**

- **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.**