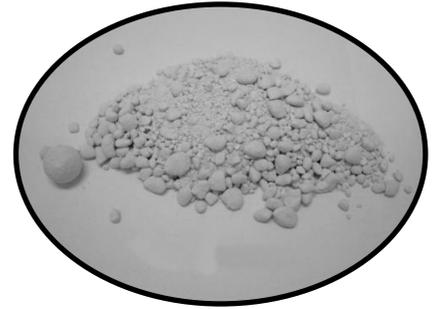
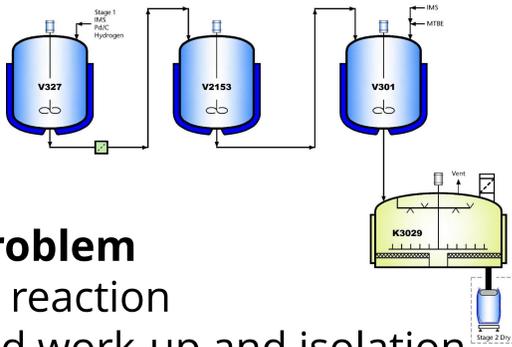




# Teamwork kicks the balls into touch

Two Britest members working together to solve a problem with an intermediate



## The Problem

- ✓ Fast reaction
- ✓ Good work-up and isolation
- ✓ High Yield
- ✓ Good product purity
- ✗ Poor product physical form

## The Product

- ✗ Large lumps or "balls"
- ✗ Gave difficulties downstream



## Joint Britest study: customer and supplier



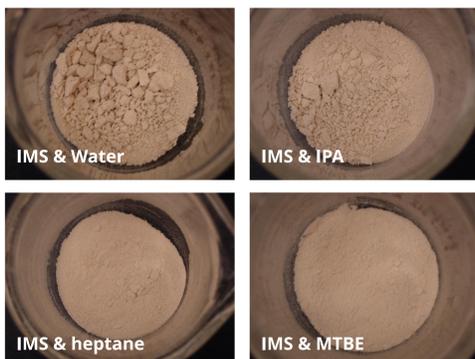
Lumps  
 Melt temp 67°C  
 Partly soluble in IMS 25mg/ml @ 25°C  
 Partly soluble in MTBE 10mg/ml @ 25°C  
 off condensation in the cooling stage size  
 appear to be product.  
 Glassy lumps  
 Hard, not friable  
 Material is 99% pure  
 Mother liquor is very dark  
 but material is pale  
 so contamination by mother liquor  
 will be obvious.  
 Mother liquor  
 IMS  
 Product  
 2% starting material long term



Calculating  
 no heat  
 25°C  
 Equipped  
 - hold @ 50°C  
 - 3 bx into 1 for cryst  
 - 3% yield by hand temp ~0°C  
 isolated.  
 in IMS/H<sub>2</sub>O  
 wash with IMS  
 TBE-washed cake is less lumpy  
 on glycol loop  
 undrying, liquid starts to  
 pool at top of the cake  
 liquid disappears. Then balls  
 start to form. Some large.  
 Other small.  
 which were quicker in 11m...  
 a source of the  
 area?  
 History of material prior to  
 parking on filter  
 Cryst temp? Exotherm.

## Action plan

- Test alternative wash solvents



Solvent	Solubility at 25 °C (mg / ml)
IMS	25
MTBE	10
IPA	5
water	< 5
heptane	< 5

## Revised process

- Two wash solvents
- Controlled filter blow down
- Controlled dryer agitation
- Defined drying profile



## Results

- ✓ Product is predominately powder
- ✓ No offload blockages
- ✓ 25% faster plant throughput
- ✓ Reduced milling losses
- ✓ Good downstream processing

Supporting organisations in gaining value from process understanding