# Integration of Particle/Powder Technology in the ChE Curriculum
# Demonstrations and Teaching Modules

ASEE Chemical Engineering Division

**Summer School**

July 21-27, 2012
University of Maine
Orono, ME

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# Integration of Particle/Powder Technology Demonstrations and Teaching Modules

- Introduce and familiarize students with technology and industrial applications  - Focus: Pharmaceutical industry

- Measurements and Analysis

  - Conductivity measurements
  - Colorimetric  measurements
  - Experimental design
  - Engineering statistics (I err therefore I am.  *St. Augustine)*

- Study of unfamiliar technology opens minds to new analysis techniques

# Agenda

- Hopper flow (video 1)
- ***Rise of large particles in bed of smaller particles***
- Particle mixing/segregation

- **Pneumatic conveying**
- *Video 2*

- **V-mixing**
  - *Concept*
  - *V-mixer design and construction (detailed instructions in pdf file provided)*
  - *Experimental design and group experiment*

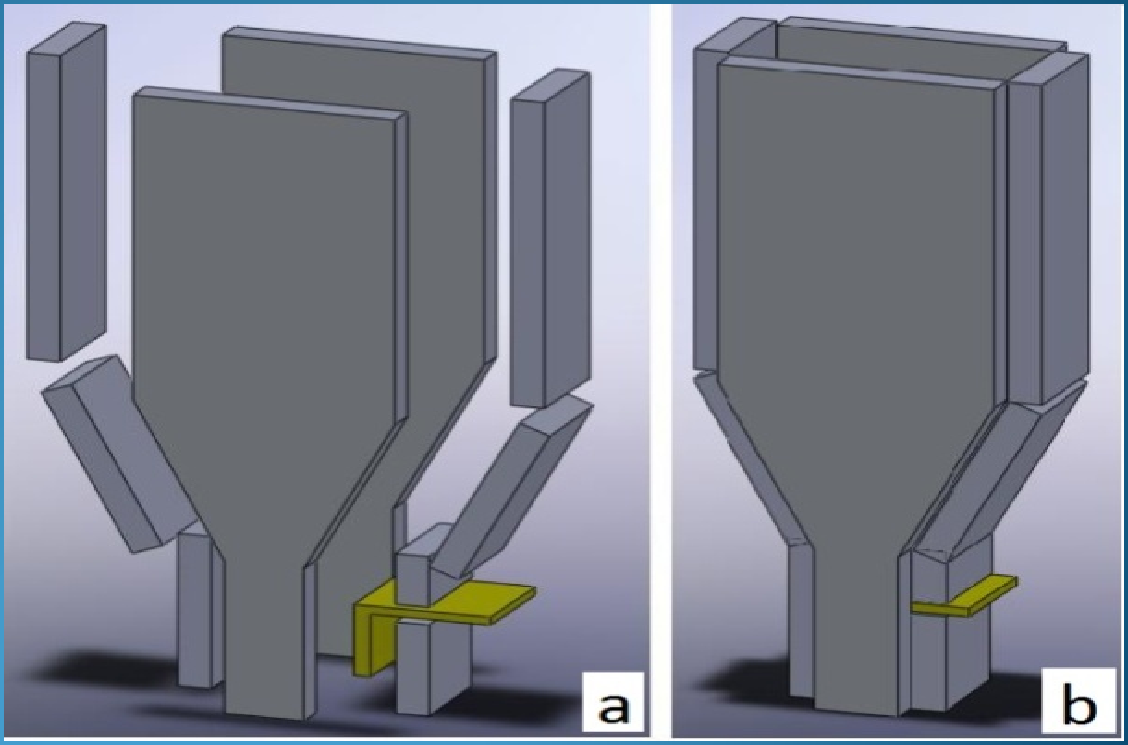
- Group Formation: pick number and join group

- References in Excel© file provided

- Additional information and tabular design format(Excel® ready) also provided

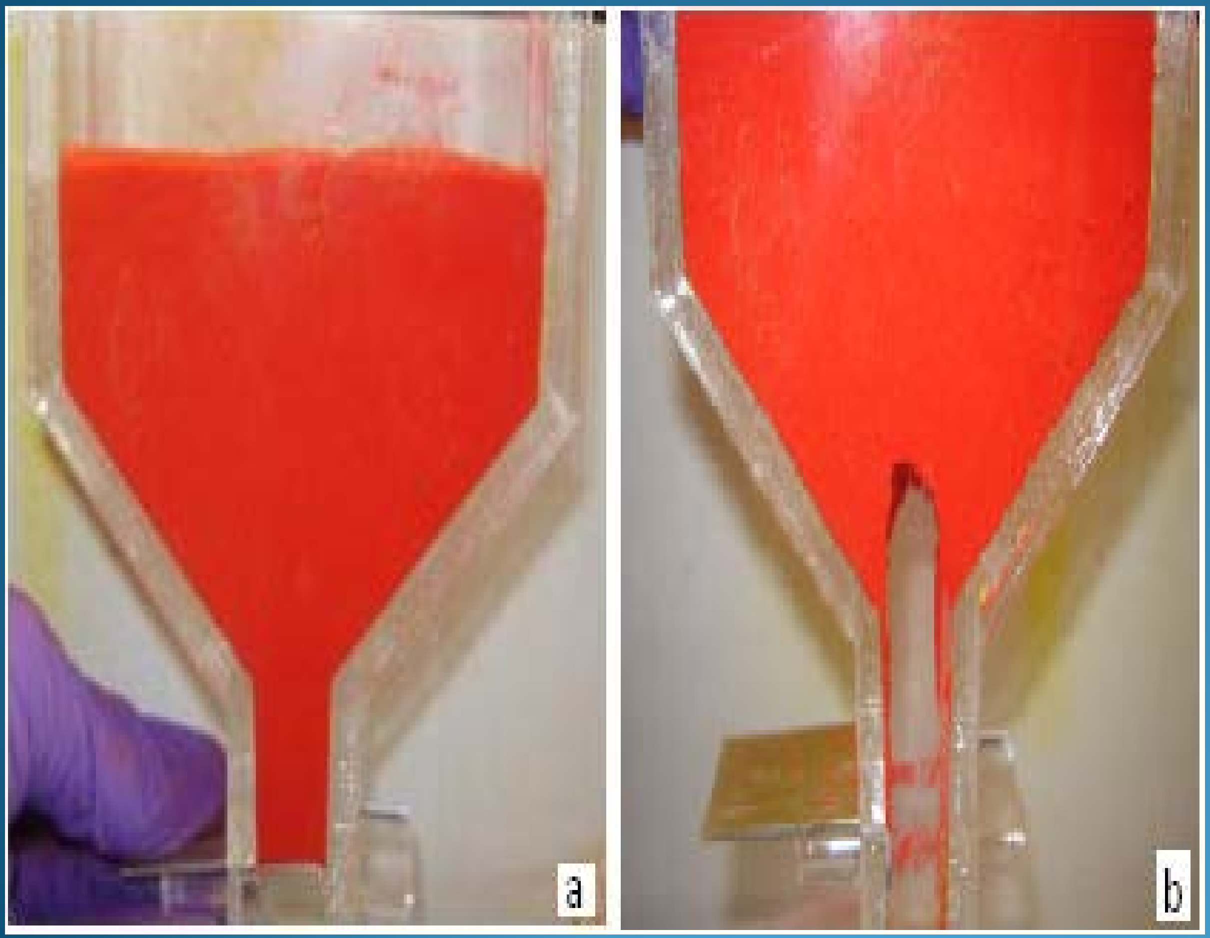
# Hopper Flow

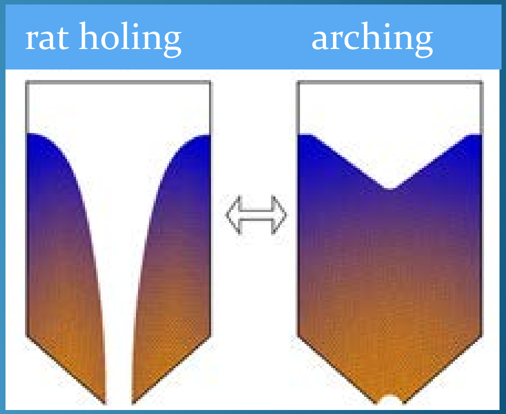
- 2-D hopper
  - ¼ in. clear polycarbonate, acrylic
  - brass hopper (18 gauge) “flow controller”
  - Angle: 40° and 60°
  - Acrylic bonding adhesive
– H = 7.75”, Max. width of 5”




**Hopper: (a) Un-assembled (b) Fully assembled**

# Hopper Flow



$ff = \frac{\sigma_C}{\sigma_D}$

# Hopper Flow Factors

$$ff = \frac{\sigma_C}{\sigma_D}$$

$\sigma_c$ = hopper compacting stress

$\sigma_D$ = stress developed in powder

$\sigma_y = f(\boldsymbol{\sigma}_C)$ unconfined yield stress of powder (arch surface)

Probability of arching decreases with flow factor

$$\boldsymbol{\sigma}_D > \boldsymbol{\sigma}_y \quad \textbf{for flow}$$

$$\frac{\boldsymbol{\sigma}_C}{ff} > \boldsymbol{\sigma}_y$$

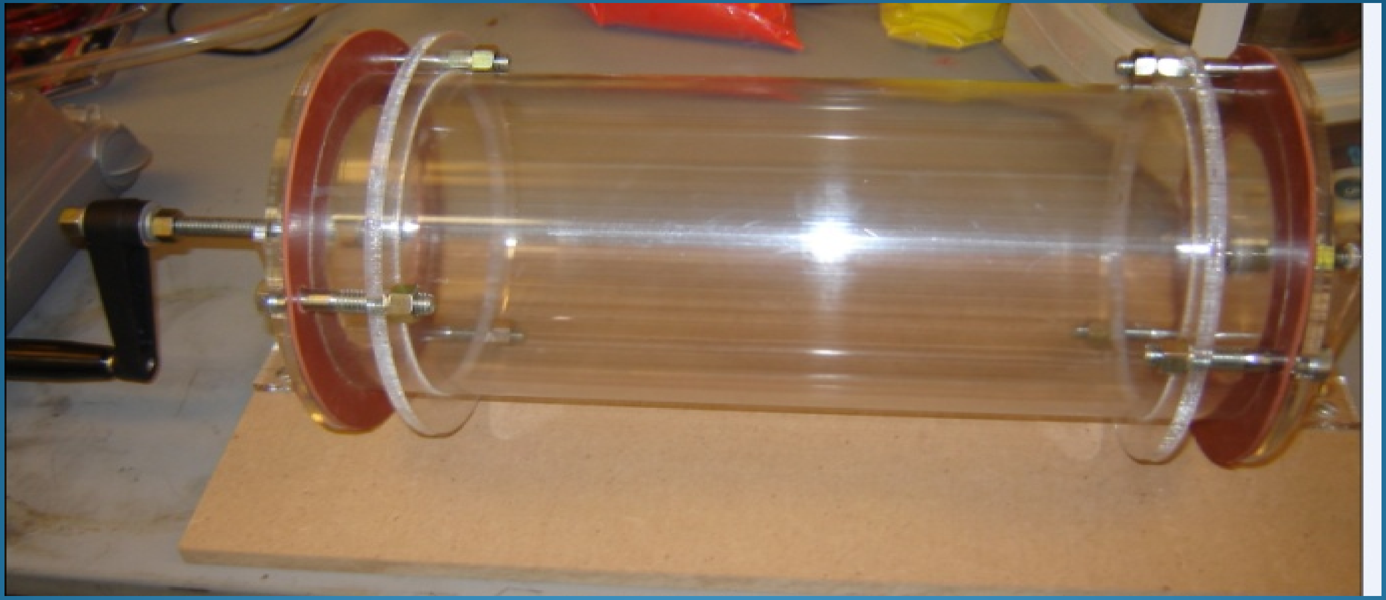
- **Arching function of material, temperature, moisture, corrosion and abrasion**

# Hopper Flow

- http://www.jenike.com/Solutions/poorflow.html

video 1

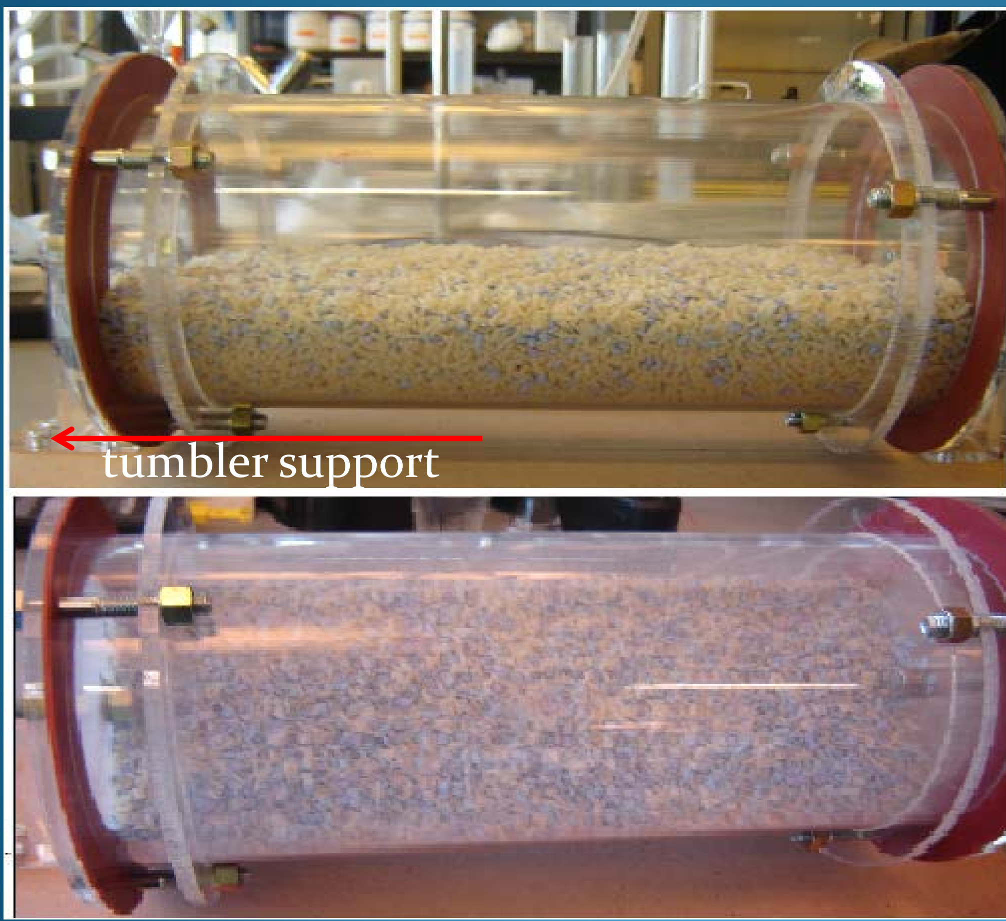
# Particle Mixing/Segregation



- Clear plastic
- Hand-crank or motor

| | |
|---|---|
| 1 | tumbler; $d_{inner}$ = 5.75", length = 12" |
| 2 | gaskets $d_{inner}$ = 6" |
| 6 | 2" long 3/8" bolts |
| 6 | 3/8" hex nuts |
| 1 | wooden base with tumbler supports |

# Particle Segregation




Particles forced toward wall.
Larger particles travel further and smaller particles "fill" spaces among larger particles

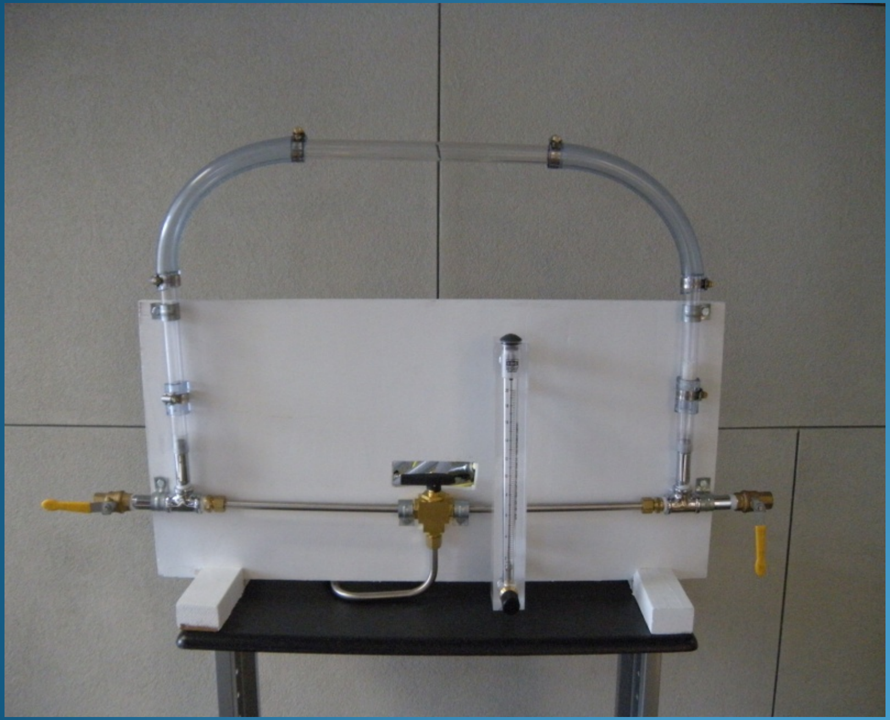
Can connect to trajectory seg./percolation

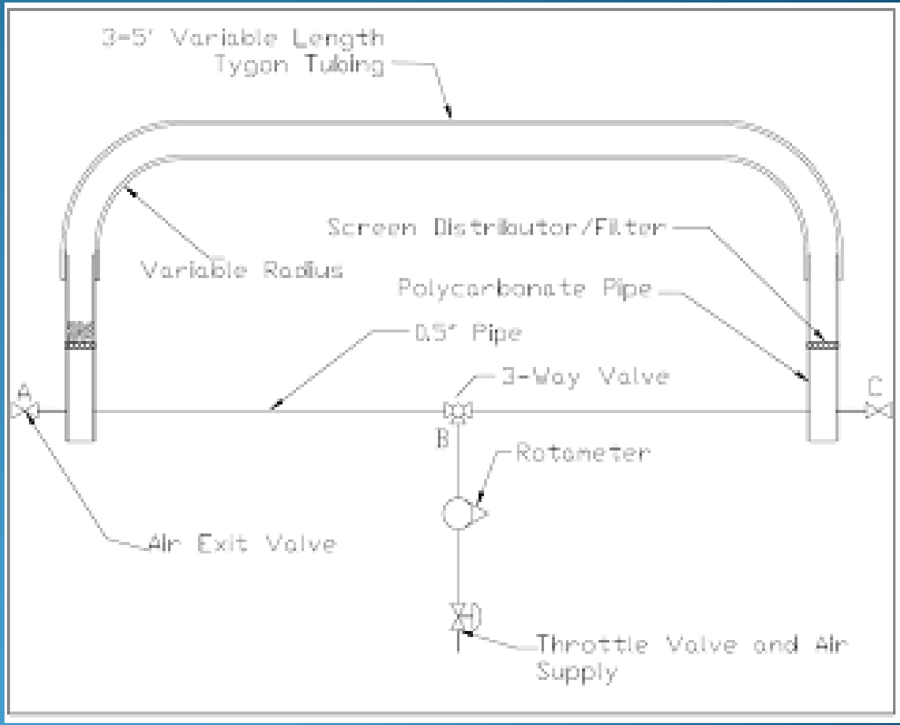
Stoke´s Law distance, D

$$D = \frac{U \rho_p x^2}{18\mu}$$

$U$ = velocity
$\rho_p$ = particle density
$x$ = particle diameter
$\mu$ = fluid viscosity

# Pneumatic Transport Apparatus (small)

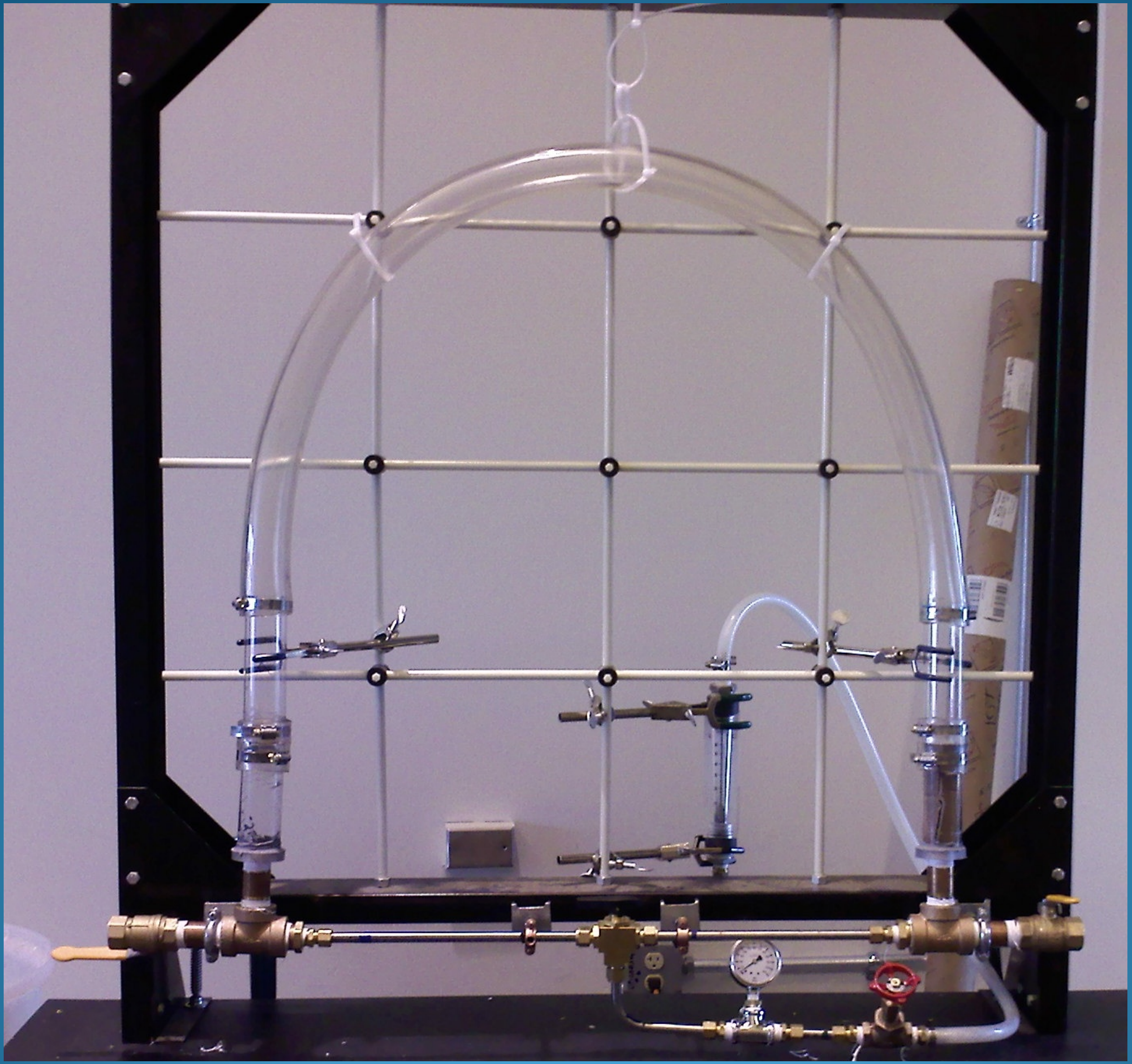




# Pneumatic Transport Apparatus Parts List (small)

| Part Inventory - Pneumatic Transport Apparatus - SMALL | | | |
|---|---|---|---|
| ITEM | QTY. | Specification | Purpose/Description |
| 1 | 6' | 1" ID | Tygon Tubing - Thick walled |
| 2 | 2' | 1" OD | Polycarbonate Round tube Clear |
| 3 | 2 | 1/2" | Threaded |
| 4 | 4 | 1/2" X 3" | Threaded Nipples |
| 5 | 1 | 0 - 40 SCFM | Flow meter |
| 6 | 1 | 1/2" | Brass Ball Valve |
| 7 | 6' | 1/2" ID | Braided Hose Air Feed |
| 8 | 1 | 1/2" Tube | Diverting 3 Port brass ball valve Yor Lock Fittings, Ultra High Pressure |
| 9 | 5 | 1/2" | Brass Compression Fitting Adapter for 1/2" Tube |
| 10 | 1 | 1/2" | Bronze Globe Valve 1" NPT Female Connections |

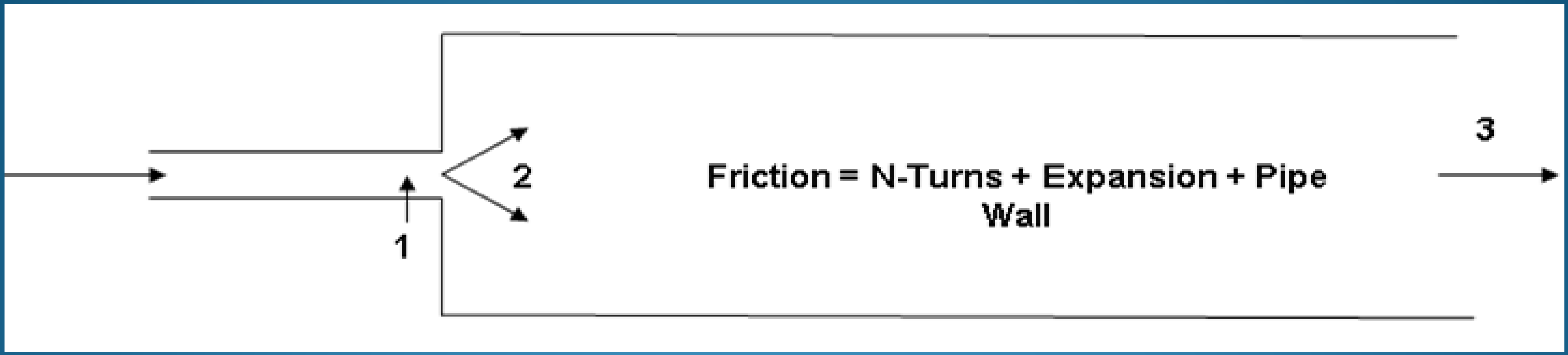
# Pneumatic Transport Apparatus (large) – <u>**Ear Protection Required**</u>



# Pneumatic Transport Apparatus Parts List (large)

| Part Inventory For Pneumatic Transport Apparatus - LARGE | | | |
|---|---|---|---|
| **ITEM** | **QTY.** | **SPECIFATION** | **DESCRIPTION** |
| **1** | 6 ft | 2" ID - 2.5" OD | Tygon PVC Tubing (0.25" wall) |
| **2** | 2 ft | 1.75" OD 1.5" ID | Poly-Carbonate Round Tube (clear - 0.125" wall) |
| **3** | 2 | 1.0" NPT | Brass Ball Valve - Female Connections |
| **4** | 2 | 1.0" | Medium-Pressure Cast Brass Threaded Pipe Fitting, TEE |
| **5** | 2 | 1" Male X 1/2" Female | Medium-Pressure Cast Brass Threaded Pipe Fitting, Hex Bushing |
| **6** | 400 | 0.125" DIA. | Nylon Spheres |
| **7** | 4 | 0.5" NPTF | Std. Brass Compression Tube Fitting Adapter Male Pipe |
| **8** | 1 | 0.5" TUBE OD | Brass Ball Valve w/ YOR-LOK Fittings Diverting 3-port Ultra High Pressure |
| **9** | 6" x 6" | 0.125" spacing | Std. Screen/Mesh Air Distributor |
| **10** | 1 | 1/2" class 150 | Bronze Globe Valve 1" NPT Female - Throttling Valve |
| **11** | N/A | 0.5" TUBE OD | Unspecified length TO BE DETERMINED |
| **12** | 4 | 1" | Thick-wall Brass Threaded Pipe Nipple, Sch 80,11/16" Thread Length |
| **13** | 1 | 10-60 SCFM @ STP | Rotameter/Flowmeter |

# Pneumatic Transport
# Energy Balance/Continuity




$$v_2 = \left[ \frac{\frac{(P_1 - P_2)(2RT)}{MW_{AIR}}}{\left[ \left(\frac{D_2^2}{D_1^2}\right)^2 \left(\frac{P_2}{P_1}\right)^2 (P_1)(K_{EXPANSION} - 1) + P_2 \right]} \right]^{0.5} = \left[ \frac{\left(\frac{kgm}{s^2 m^2}\right)\left(\frac{kgm^4}{s^2 m^2 kmolK} \frac{Kkmol}{kg}\right)}{\left[\frac{kgm}{s^2 m^2}\right]} \right]^{0.5} = \frac{m}{s}$$

**(Video 2)**

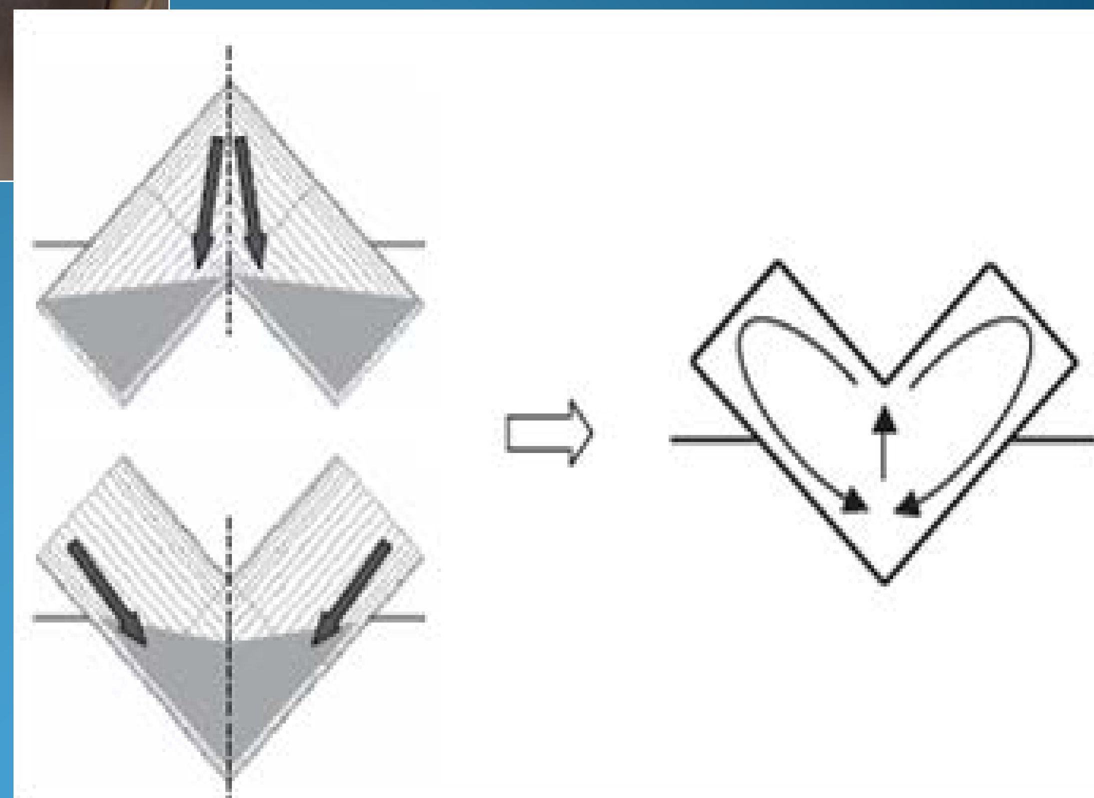
Poor estimations – why?

What energy losses are not included in analysis?

Can also do minimum fluidization velocity measurements (one side –fluidized bed)

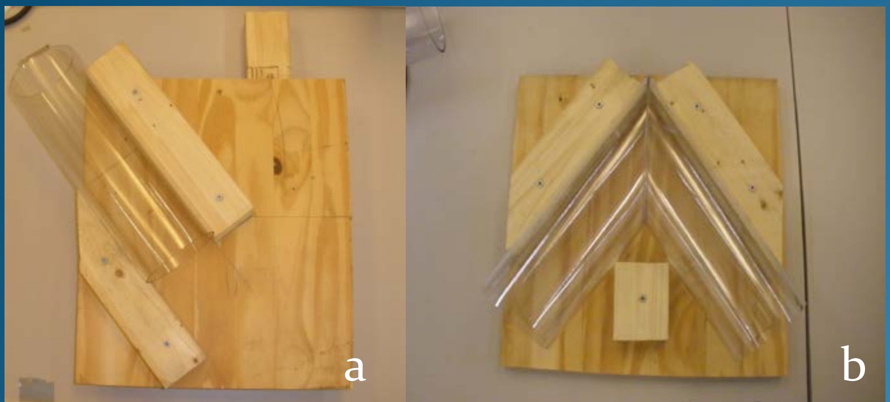
# V-Mixing

5 L  constant speed V-mixer  - www.**tabletpress.net**
$1650 – bench top model with timer switch
Loading capacity – 1 kg



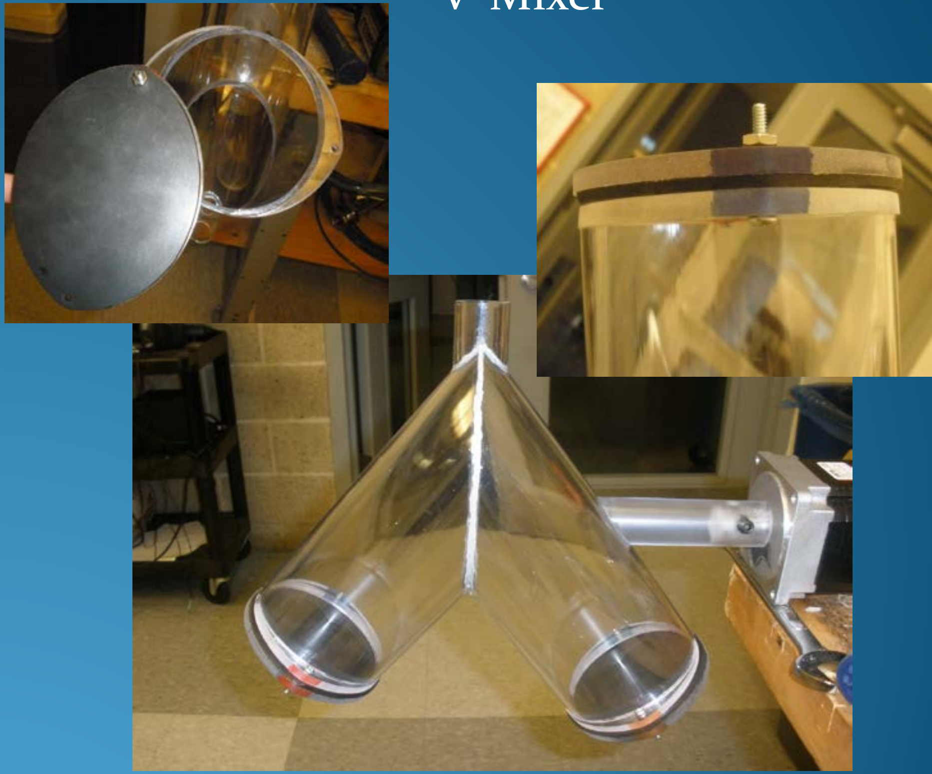
# V-Mixer Construction

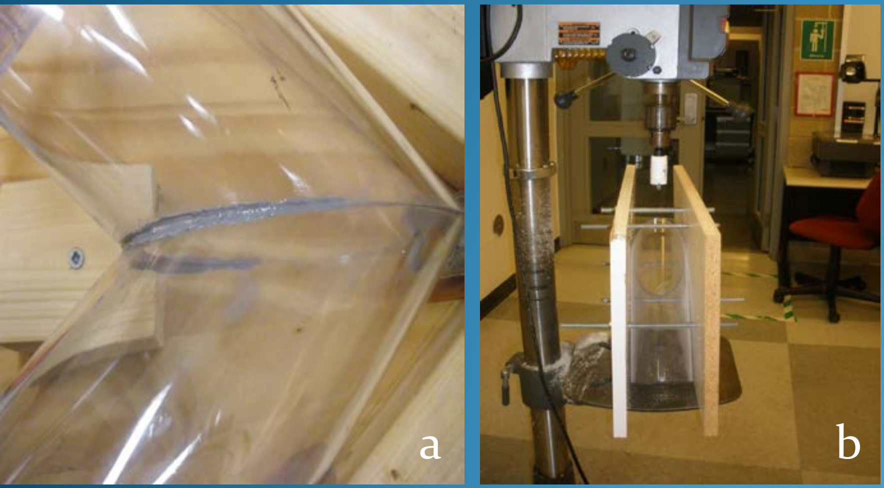
(Detailed instructions in pdf file provided)




Cutting (a) and gluing tubes (b)

V-Mixer






Epoxy (a) and drilling hole (b)

Pneumatic motor
Gast Mfg. Corp (Vendor – McMaster-Carr
Model #2AM-FCW-13 with Boston gear/speed reducer 713-60-j
$500.00

# V-mixer Demonstration

## highly visual and colorful

k-12 – engaging, ask for predictions

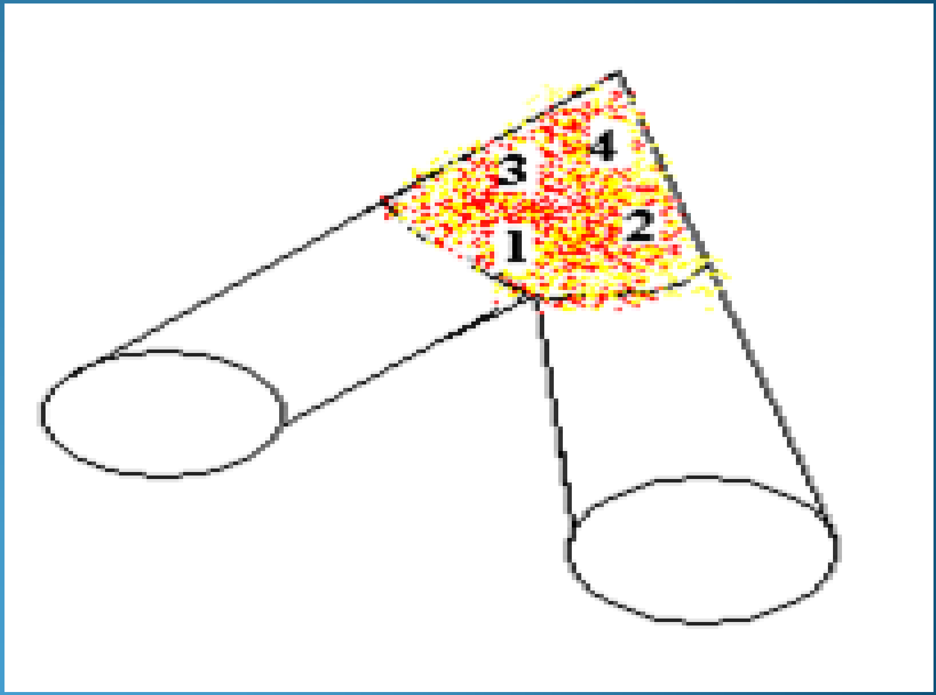
undergraduates – predictions based on physics, sampling, mixing quality measures, loading procedures

# V-Mixing Module

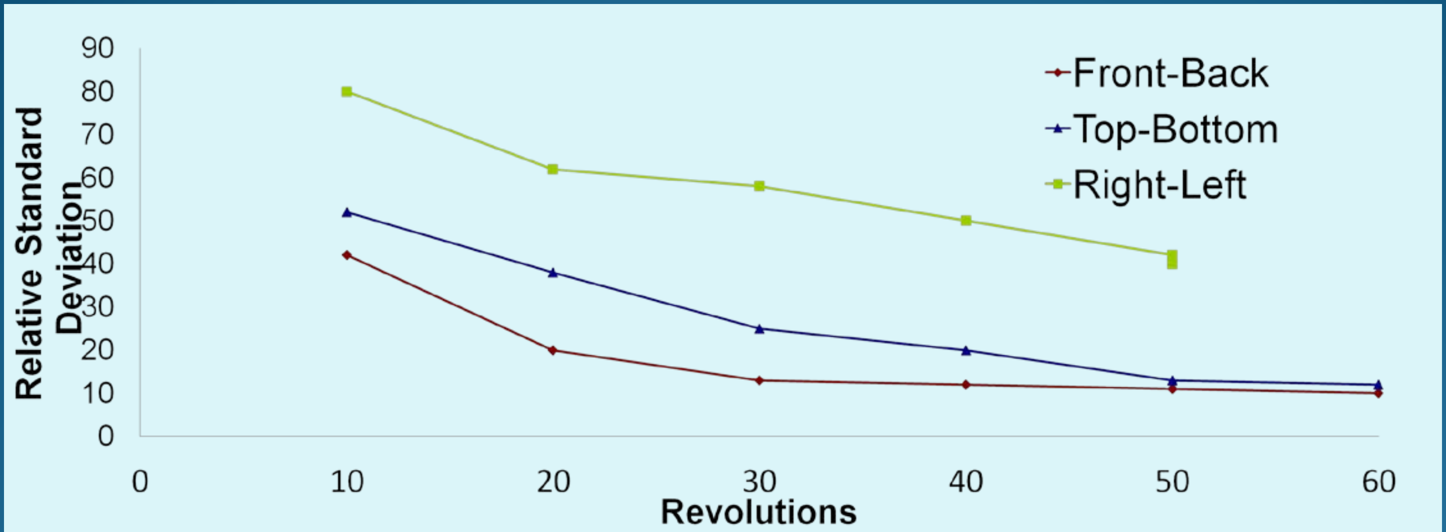
- Introduces students to mixing of solids with pharmaceutical applications
- No constitutive equations
- Introduce students to design of experiments (DOE)
- Students previously exposed to basic statistics and t distribution

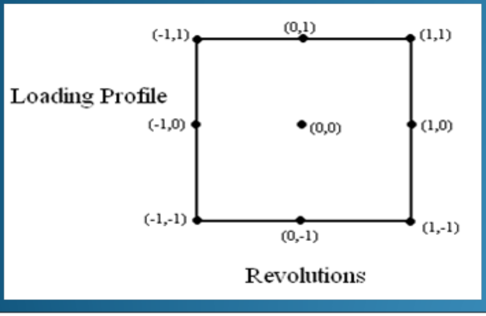
# V-Mixer Experiments (colorimetric measurement)

- 90 – 110 micron polyarmor® particles
- Red and yellow particles
- Yellow dye soluble in methanol
- Mix, sample, spectrometer measurement $\lambda = 570$ nm

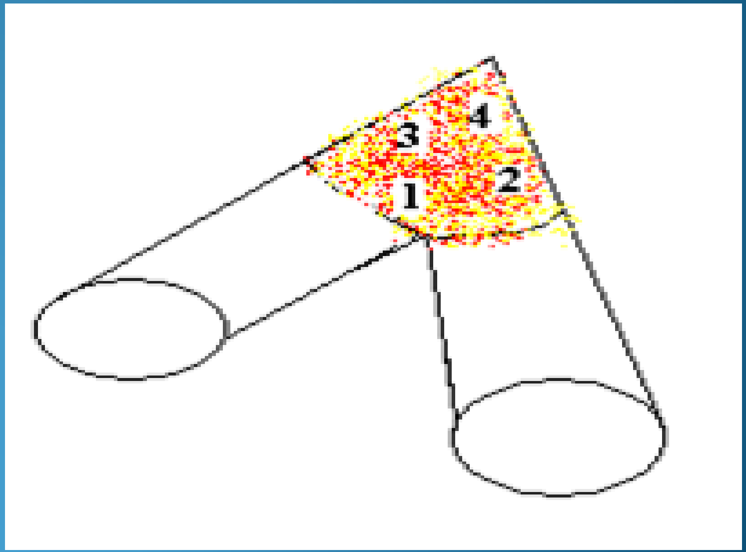


# Spectral Colorimetric Measurements - Mixing Quality



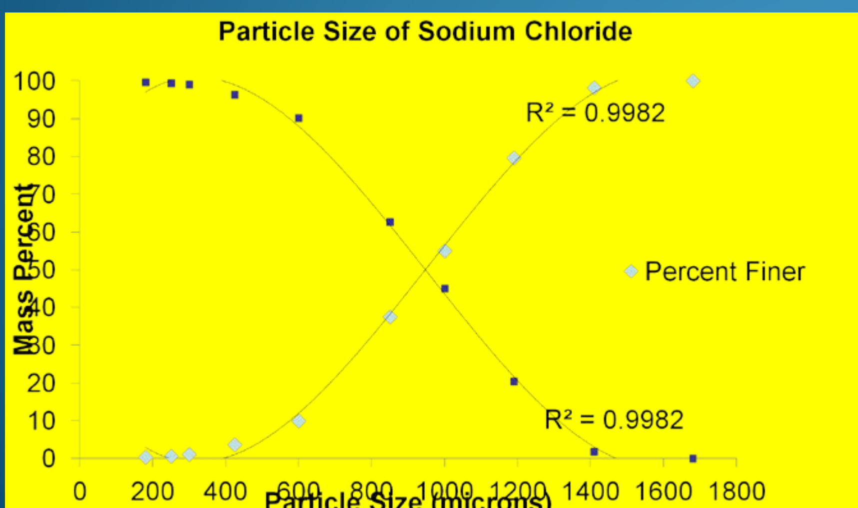



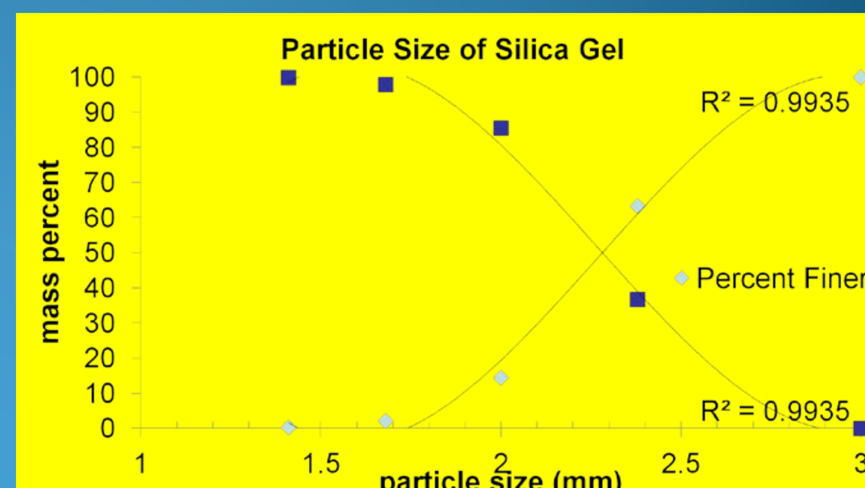

| Loading Profile | | Revolutions | |
|---|---|---|---|
| *Coded* | *Actual* | *Coded* | *Actual* |
| 1 | top-bottom | 1 | 18 |
| 0 | right-left | 0 | 10.5 |
| -1 | front-back | -1 | 3 |



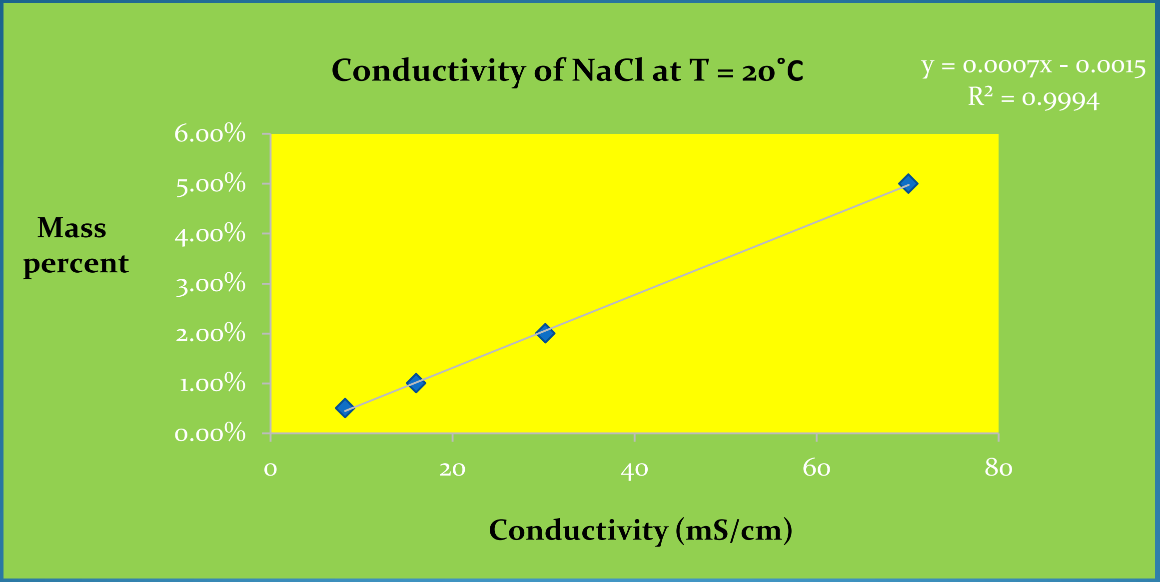
# Particle Size Distributions

**Particle size** (shaker **method or XCT scan)**
**Sodium chloride :** $\Phi_{50}$ = 950 **µm**
coefficient of uniformity ($\Phi_{60}/\Phi_{10}$) = 0.67
Silica gel: $\Phi_{50}$ = 2.27 mm
coefficient of uniformity ($\Phi_{60}/\Phi_{10}$) = 0.82



# Conductivity of NaCl Solution - Quantifies Concentration Mixing Quality

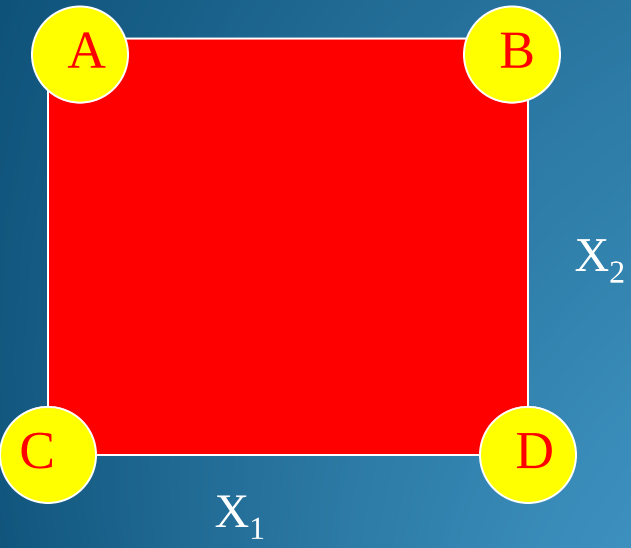



| *Source* | *Sum of Squares* | *Df* | *Mean Square* | *F-Ratio* | *P-Value* |
|---|---|---|---|---|---|
| MAIN EFFECTS | | | | | |
| A:Particle System | 10650.2 | 1 | 10650.2 | 148.15 | 0.0000 |
| B:Loading Profile | 559.77 | 1 | 559.77 | 7.79 | 0.0107 |
| C:Number of Revolutions | 609.329 | 1 | 609.329 | 8.48 | 0.0081 |
| RESIDUAL | 1581.53 | 22 | 71.8879 | | |
| TOTAL (CORRECTED) | 13380.7 | 25 | | | |

# Let´s Experiment!

- Four (4) V-mixers
- Load mixers at design points
- Variables: particle (colored spheres) size and loading profile
- Constant: mixing speed, mixing time, V-mixer fill, sphere size ratio
- Very basic experimental design concepts before forming groups

# V-Mixer Experiment
# 2 level factorial for two variables
# $2^2$ factorial



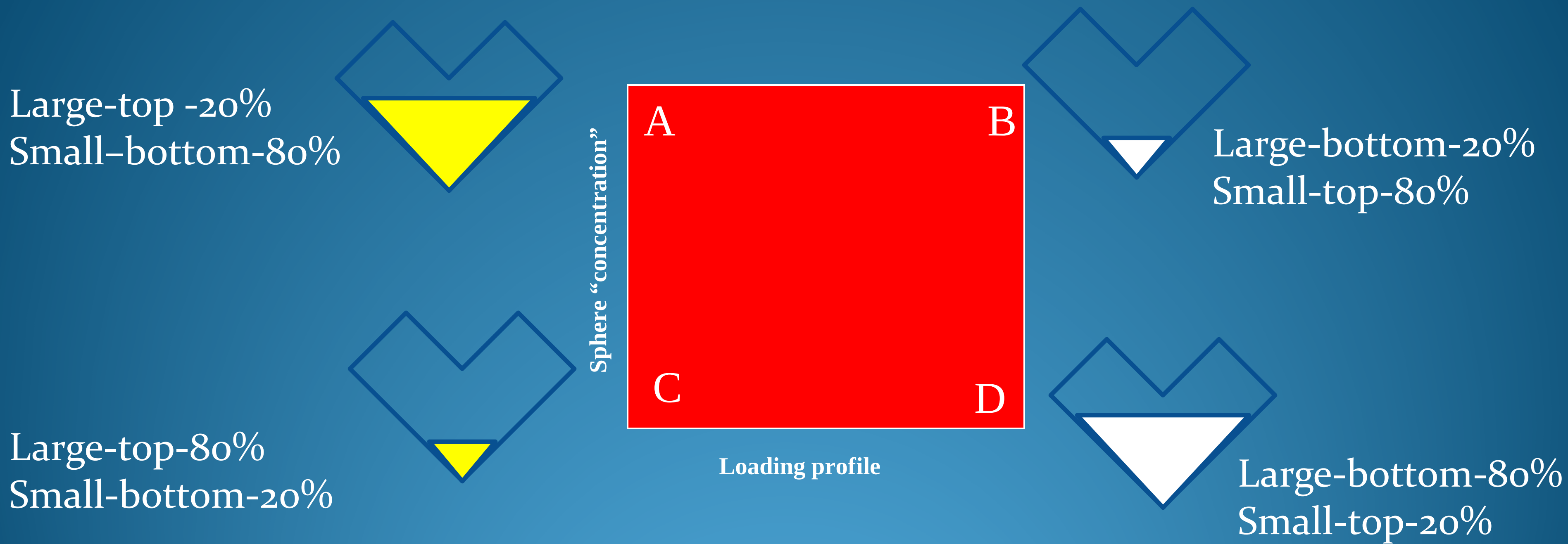

1. Average results at high and low values of $x_1$ and $x_2$
2. Compute difference between average result at high and low values of $x_1$ and $x_2$
3. Compare difference to MSFE at desired confidence level
4. Do "center point" experiments to test curvature
5. Compare "center point" average to MSCE at desired confidence level

Results:

- Identify statistically significant variables
- Develop algebraic model for measured variable as function of independent variables $x_1$ and $x_2$ and their interactions

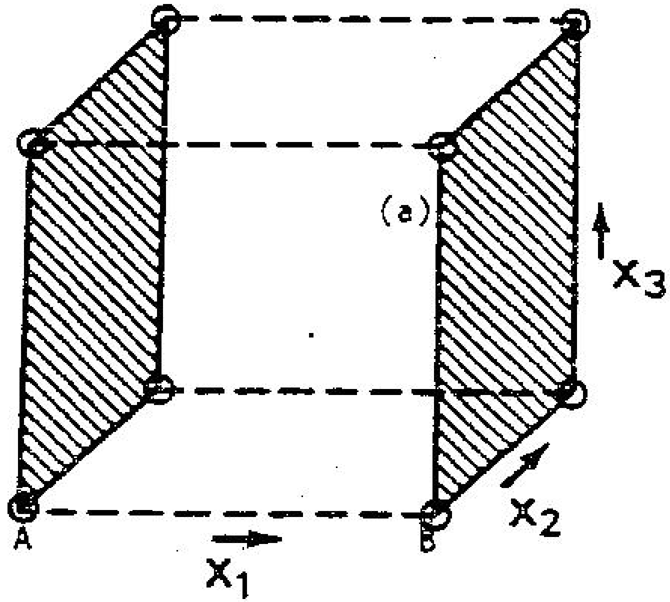
# V-Mixer Experiment (not to scale)

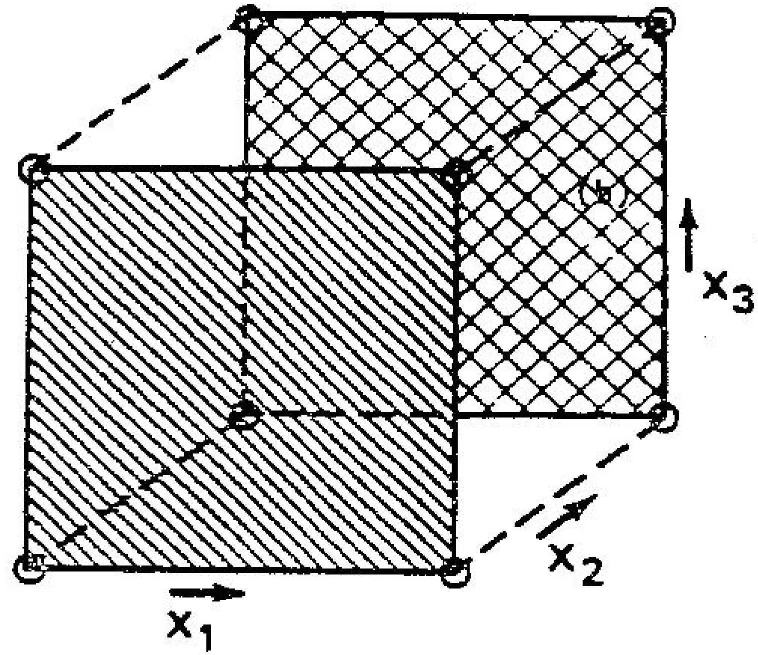
(measurements – weight %)




Spheres - particle size and location in mixer
constant ratio 80/20
constant V-mixer fill

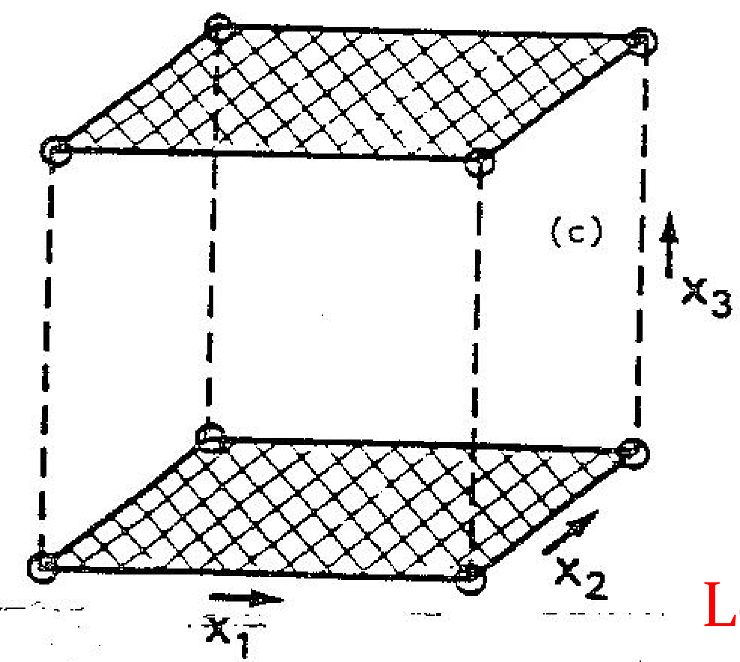
large spheres small spheres




**Experimental design**

- **Hidden replication- less experiments**
- **Orthogonality – independence among variables**




Let´s form groups

# Acknowledgements

- C. Del Vecchio
- K. Ross
- M. Glasspool
- J. Giacomelli
- M. Rodgers
- H. Diallo
- **M. Harris, ChE Technician**

Engineering Research Center for Structured Organic Particulate Systems

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uprm.edu universidad de puerto rico Recinto Universitario de Mayagüez

National Science Foundation WHERE DISCOVERIES BEGIN


**This work is part of a publication in progress.**

# Number of Experiments: OFAAT v. Design

- Extraction of herbicide from soil matrix

  \- quantification of residue and sample preparation
- Variables: solvent, co-solvent, T, P, soil matrix, operation mode
- OFAAT: 3 variable values, 2 modes of operation

  $N = (3)(3)(3)(3)(3)(2) = 486$
- Factorial design: $2(2^5) = 64$
- Design: minimum experimental runs, inclusive and accurate analysis

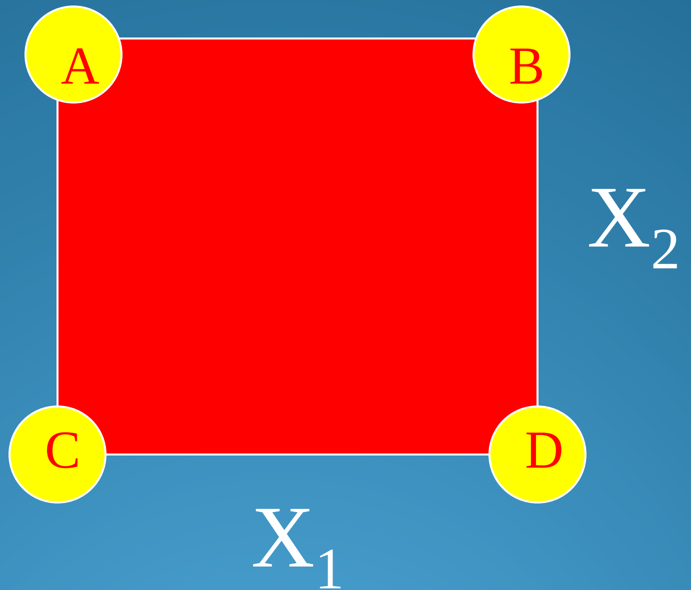
# Properties of Factorial Designs

- Hidden replication

  - all points are used to calculate effect on variables and interactions

- Orthogonal (independent measurements)

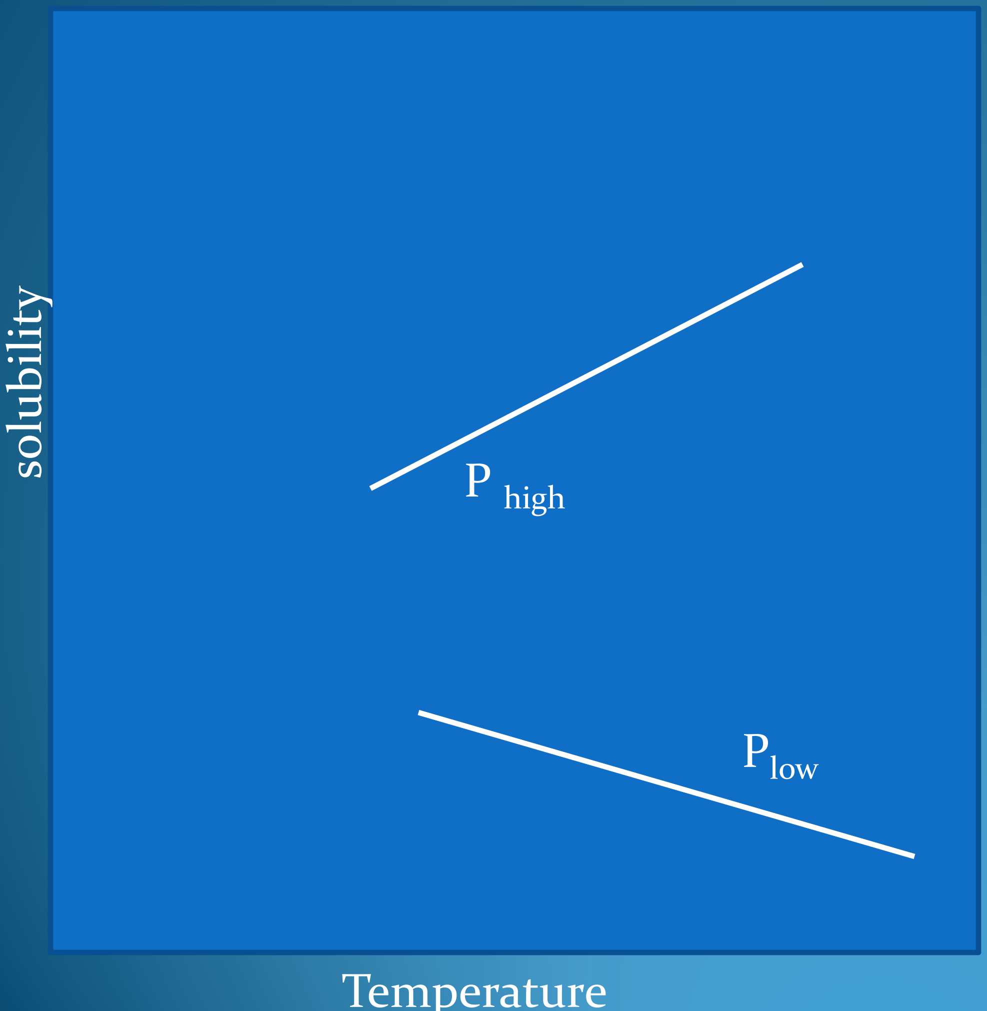
# Interactions
# Independent Variables



# Interactions
# Independent Variables

- In = ½ [(b – a) – (d – c)]
- If more than 2 factors – compare diagonally opposing planes

# Interaction Example
# Solid Solubility in SF $CO_2$




Solubility = f($CO_2$ density, solid volatility)

T ↑ volatility ↑ density ↓

$P_{high}$ - density high

$P_{low}$ - density low

# Design: Coding Factors

- Code factors for tabular analysis

  discrete: + and –

  continuous: high +

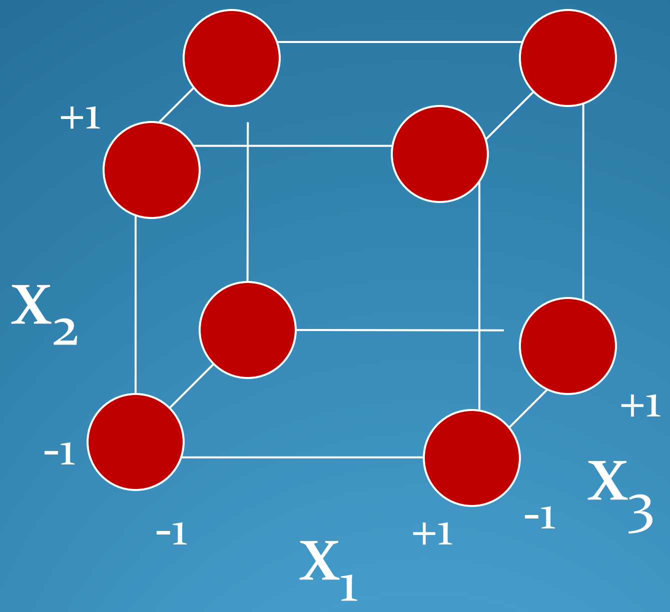
  low -

- Interpret + and – as +1 and -1

- Space widely for larger response

| Trial | Factors $x_1$ | $x_2$ | $x_3$ | $x_4$ | $x_5$ |
| --- | --- | --- | --- | --- | --- |
| 1 | − | − | − | − | − |
| 2 | + | − | − | − | − |
| 3 | − | + | − | − | − |
| $4 = 2^2$ | + | + | − | − | − |
| 5 | − | − | + | − | − |
| 6 | + | − | + | − | − |
| 7 | − | + | + | − | − |
| $8 = 2^3$ | + | + | + | − | − |
| 9 | − | − | − | + | − |
| 10 | + | − | − | + | − |
| 11 | − | + | − | + | − |
| 12 | + | + | − | + | − |
| 13 | − | − | + | + | − |
| 14 | + | − | + | + | − |
| 15 | − | + | + | + | − |
| $16 = 2^4$ | + | + | + | + | − |
| 17 | − | − | − | − | + |
| 18 | + | − | − | − | + |
| 19 | − | + | − | − | + |
| 20 | + | + | − | − | + |
| 21 | − | − | + | − | + |
| 22 | + | − | + | − | + |
| 23 | − | + | + | − | + |
| 24 | + | + | + | − | + |
| 25 | − | − | − | + | + |
| 26 | + | − | − | + | + |
| 27 | − | + | − | + | + |
| 28 | + | + | − | + | + |
| 29 | − | − | + | + | + |
| 30 | + | − | + | + | + |
| 31 | − | + | + | + | + |
| $32 = 2^5$ | + | + | + | + | + |

# $2^3$ Factorial Design



# Computational table for a $2^3$ Factorial Design

| Run | mean | $X_1$ | $x_2$ | $x_1 x_2$ | $x_3$ | $x_1x_3$ | $x_2x_3$ | $x_1 x_2x_3$ | Y |
|---|---|---|---|---|---|---|---|---|---|
| 1 | + | - | - | + | - | + | + | - | |
| 2 | + | + | - | - | - | - | + | + | |
| 3 | + | - | + | - | - | + | - | + | |
| 4 | + | + | + | + | - | - | - | - | |
| 5 | + | - | - | + | + | - | - | + | |
| 6 | + | + | - | - | + | + | - | - | |
| 7 | + | - | + | - | + | - | + | - | |
| 8 | + | + | + | + | + | + | + | + | |
| ∑+<br>∑-<br>D<br>E | D= ∑+ - ∑-<br>E= D/4 | | | | | | | | |

Y = measured variable, compare E to MSFE, average of center measurements compared
To MSCE if abs. value of E >MSFE – significant at confidence level . If center average > MSCE – nonlinearities
Significant at confidence level

Computational Table for Two-Level Factorial for up to Five Factors
(showing main effects and interactions)

| | $2^2$ | | | | $2^3$ | | | | $2^4$ | | | | | | | | $2^5$ | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Trial | Mean | $X_1$ | $X_2$ | $X_1X_2$ | $X_3$ | $X_1X_3$ | $X_2X_3$ | $X_1X_2X_3$ | $X_4$ | $X_1X_4$ | $X_2X_4$ | $X_1X_2X_4$ | $X_3X_4$ | $X_1X_3X_4$ | $X_2X_3X_4$ | $X_1X_2X_3X_4$ | $X_5$ | $X_1X_5$ | $X_2X_5$ | $X_1X_2X_5$ | $X_3X_5$ | $X_1X_3X_5$ | $X_2X_3X_5$ | $X_1X_2X_3X_5$ | $X_4X_5$ | $X_1X_4X_5$ | $X_2X_4X_5$ | $X_1X_2X_4X_5$ | $X_3X_4X_5$ | $X_1X_3X_4X_5$ | $X_2X_3X_4X_5$ | $X_1X_2X_3X_4X_5$ |
| 1 | + | - | - | + | - | + | + | - | - | + | + | - | + | - | - | + | - | + | + | - | + | - | - | + | + | - | - | + | - | + | + | - |
| 2 | + | + | - | - | - | - | + | + | - | - | + | + | + | + | - | - | - | - | + | + | + | + | - | - | + | + | - | - | - | - | + | + |
| 3 | + | - | + | - | - | + | - | + | - | + | - | + | + | - | + | - | - | + | - | + | + | - | + | - | + | - | + | - | - | + | - | + |
| 4 | + | + | + | + | - | - | - | - | - | - | - | - | + | + | + | + | - | - | - | - | + | + | + | + | + | + | + | + | - | - | - | - |
| 5 | + | - | - | + | + | - | - | + | - | + | + | - | - | + | + | - | - | + | + | - | - | + | + | - | + | - | - | + | + | - | - | + |
| 6 | + | + | - | - | + | + | - | - | - | - | + | + | - | - | + | + | - | - | + | + | - | - | + | + | + | + | - | - | + | + | - | - |
| 7 | + | - | + | - | + | - | + | - | - | + | - | + | - | + | - | + | - | + | - | + | - | + | - | + | + | - | + | - | + | - | + | - |
| 8 | + | + | + | + | + | + | + | + | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | + | + | + | + | + | + | + | + |
| 9 | + | - | - | + | - | + | + | - | + | - | - | + | - | + | + | - | - | + | + | - | + | - | - | + | - | + | + | - | + | - | - | + |
| 10 | + | + | - | - | - | - | + | + | + | + | - | - | - | - | + | + | - | - | + | + | + | + | - | - | - | - | + | + | + | + | - | - |
| 11 | + | - | + | - | - | + | - | + | + | - | + | - | - | + | - | + | - | + | - | + | + | - | + | - | - | + | - | + | + | - | + | - |
| 12 | + | + | + | + | - | - | - | - | + | + | + | + | - | - | - | - | - | - | - | - | + | + | + | + | - | - | - | - | + | + | + | + |
| 13 | + | - | - | + | + | - | - | + | + | - | - | + | + | - | - | + | - | + | + | - | - | + | + | - | - | + | + | - | - | + | + | - |
| 14 | + | + | - | - | + | + | - | - | + | + | - | - | + | + | - | - | - | - | + | + | - | - | + | + | - | - | + | + | - | - | + | + |
| 15 | + | - | + | - | + | - | + | - | + | - | + | - | + | - | + | - | - | + | - | + | - | + | - | + | - | + | - | + | - | + | - | + |
| 16 | + | + | + | + | + | + | + | + | + | + | + | + | + | + | + | + | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 17 | + | - | - | + | - | + | + | - | - | + | + | - | + | - | - | + | + | - | - | + | - | + | + | - | - | + | + | - | + | - | - | + |
| 18 | + | + | - | - | - | - | + | + | - | - | + | + | + | + | - | - | + | + | - | - | - | - | + | + | - | - | + | + | + | + | - | - |
| 19 | + | - | + | - | - | + | - | + | - | + | - | + | + | - | + | - | + | - | + | - | - | + | - | + | - | + | - | + | + | - | + | - |
| 20 | + | + | + | + | - | - | - | - | - | - | - | - | + | + | + | + | + | + | + | + | - | - | - | - | - | - | - | - | + | + | + | + |
| 21 | + | - | - | + | + | - | - | + | - | + | + | - | - | + | + | - | + | - | - | + | + | - | - | + | - | + | + | - | - | + | + | - |
| 22 | + | + | - | - | + | + | - | - | - | - | + | + | - | - | + | + | + | + | - | - | + | + | - | - | - | - | + | + | - | - | + | + |
| 23 | + | - | + | - | + | - | + | - | - | + | - | + | - | + | - | + | + | - | + | - | + | - | + | - | - | + | - | + | - | + | - | + |
| 24 | + | + | + | + | + | + | + | + | - | - | - | - | - | - | - | - | + | + | + | + | + | + | + | + | - | - | - | - | - | - | - | - |
| 25 | + | - | - | + | - | + | + | - | + | - | - | + | - | + | + | - | + | - | - | + | - | + | + | - | + | - | - | + | - | + | + | - |
| 26 | + | + | - | - | - | - | + | + | + | + | - | - | - | - | + | + | + | + | - | - | - | - | + | + | + | + | - | - | - | - | + | + |
| 27 | + | - | + | - | - | + | - | + | + | - | + | - | - | + | - | + | + | - | + | - | - | + | - | + | + | - | + | - | - | + | - | + |
| 28 | + | + | + | + | - | - | - | - | + | + | + | + | - | - | - | - | + | + | + | + | - | - | - | - | + | + | + | + | - | - | - | - |
| 29 | + | - | - | + | + | - | - | + | + | - | - | + | + | - | - | + | + | - | - | + | + | - | - | + | + | - | - | + | + | - | - | + |
| 30 | + | + | - | - | + | + | - | - | + | + | - | - | + | + | - | - | + | + | - | - | + | + | - | - | + | + | - | - | + | + | - | - |
| 31 | + | - | + | - | + | - | + | - | + | - | + | - | + | - | + | - | + | - | + | - | + | - | + | - | + | - | + | - | + | - | + | - |
| 32 | + | + | + | + | + | + | + | + | + | + | + | + | + | + | + | + | + | + | + | + | + | + | + | + | + | + | + | + | + | + | + | + |

# Accounting for Curvature

- Non-linear effects
- Run center points ( +1=150 and -1= 50 then center point, 0 = 100)
- Severity of curvature quantified by minimum significant curvature effect

# Factor Effect Significance: Minimum Significant Factor Effect

- MSFE = $t \cdot s(2/mk)^{1/2}$

  t= student's t at desired confidence level
  and appropriate degrees of freedom

  s= standard deviation

  m = number of + signs in column

  k= number of replicates

# Curvature Effects:Minimum Significant Curvature Effect

- $$MSCE = t \cdot s \left[ \frac{1}{mk} + \frac{1}{c} \right]^{1/2}$$

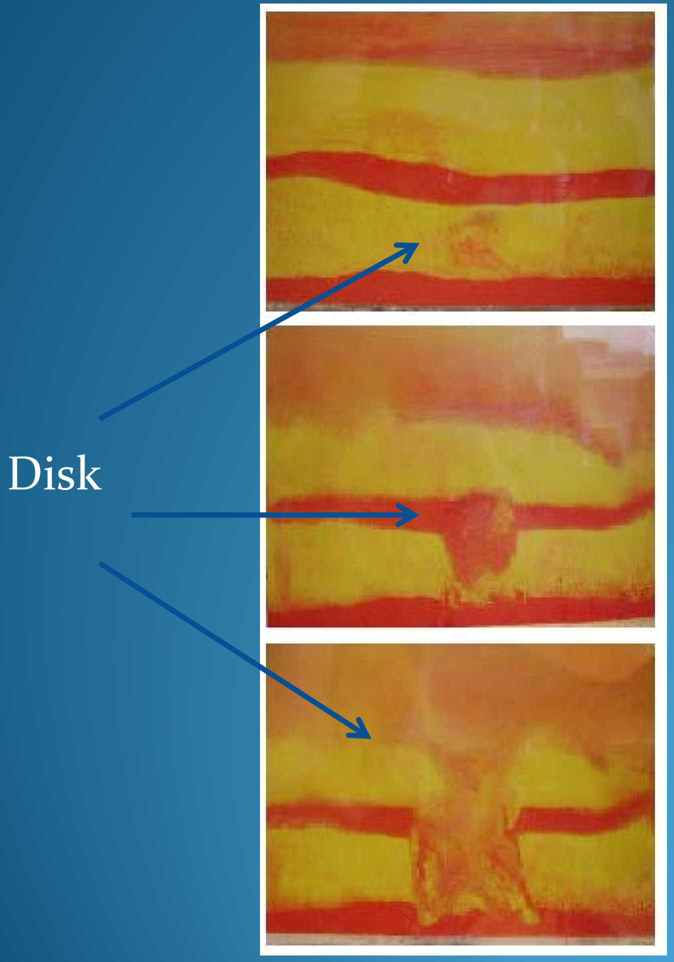
all variables as defined and c = number of center points

# Coding and Model from Experimental Data

- Code $$X_{(coded)} = \frac{X - (high + low)/2}{(high - low)/2}$$

X = the value of the independent variable at which a prediction is desired

# Large Particle Rise in Smaller Particle Bed




| | |
|---|---|
| 8 | 6-32 thread 7/64 hex head screws |
| 2 | 10" by 10" acrylic plastic sheets |
| 4 | 10" by 1" acrylic plastic sheets |
| 1 | 10" by 1" gasket |

- 2-D square vessel (10”)
- Sealed
- Flat metal disc d= 1”