Determining Critical Water Activities for Glass Transitions Using the Dynamic Dewpoint Isotherm Method

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Glass Transition

- Glassy state characterized by a meta-stable “frozen” state with low mobility
- Has some characteristics of 2nd order transition but also appears to be kinetic
- Low molecular weight carbohydrates in amorphous state have relatively low glass transition temperatures
- Above glass transition, mobility is increased and time-dependent processes speed up

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Glass Transition

- Glass transition can be induced with temperature or increased plasticizer
- Water most common plasticizer
- Water activity influences plasticization and has been related to changes in Tg
- Water activity is more easily measured than Tg
- Can glass transition be determined using Dynamic Sorption Isotherms instead of Differential Scanning Calorimetry?

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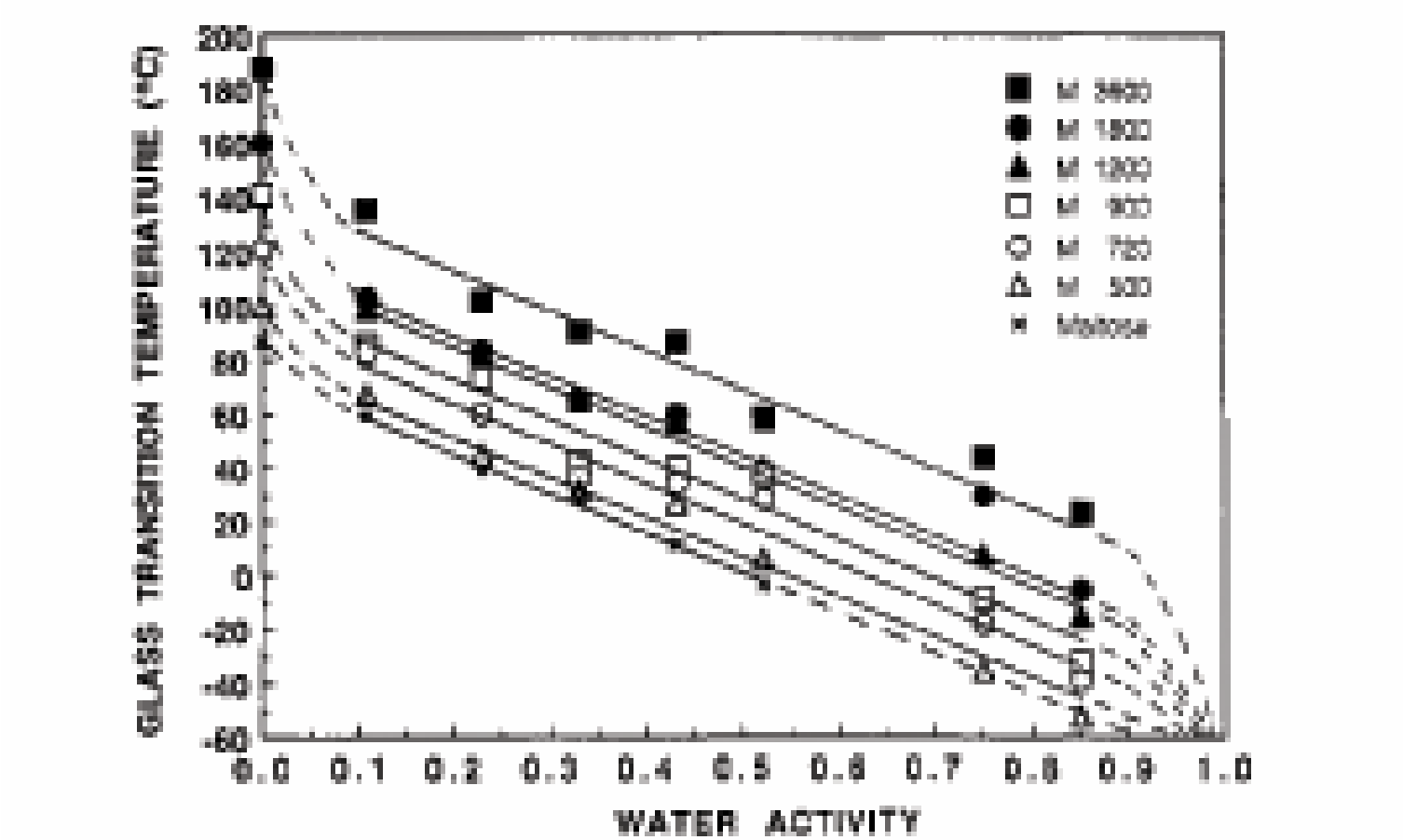
Water Activity and Glass Transition



Roos and Karel 1991 Biotechnol. Prog. 7:49-53

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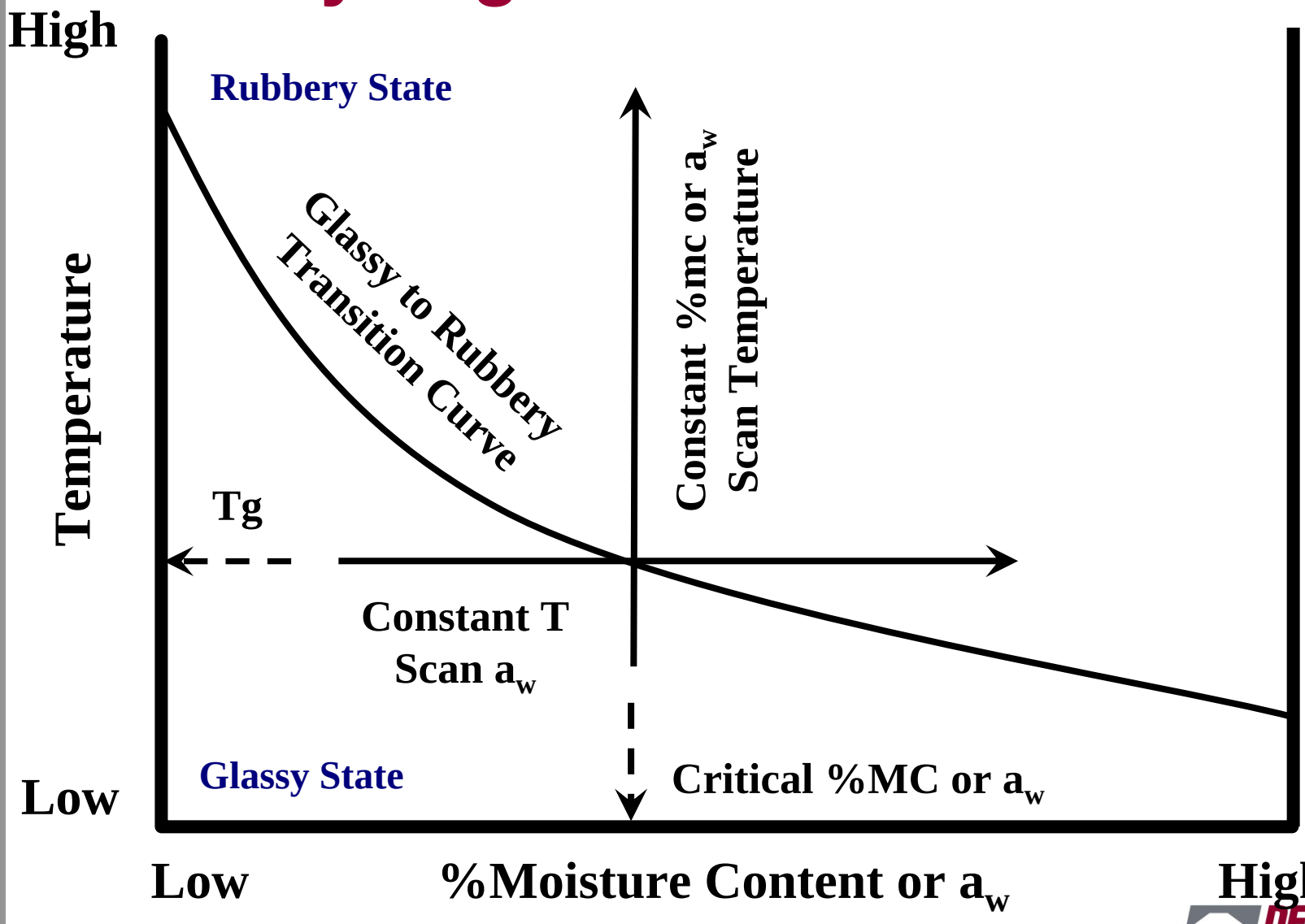
Analyzing Glass Transitions



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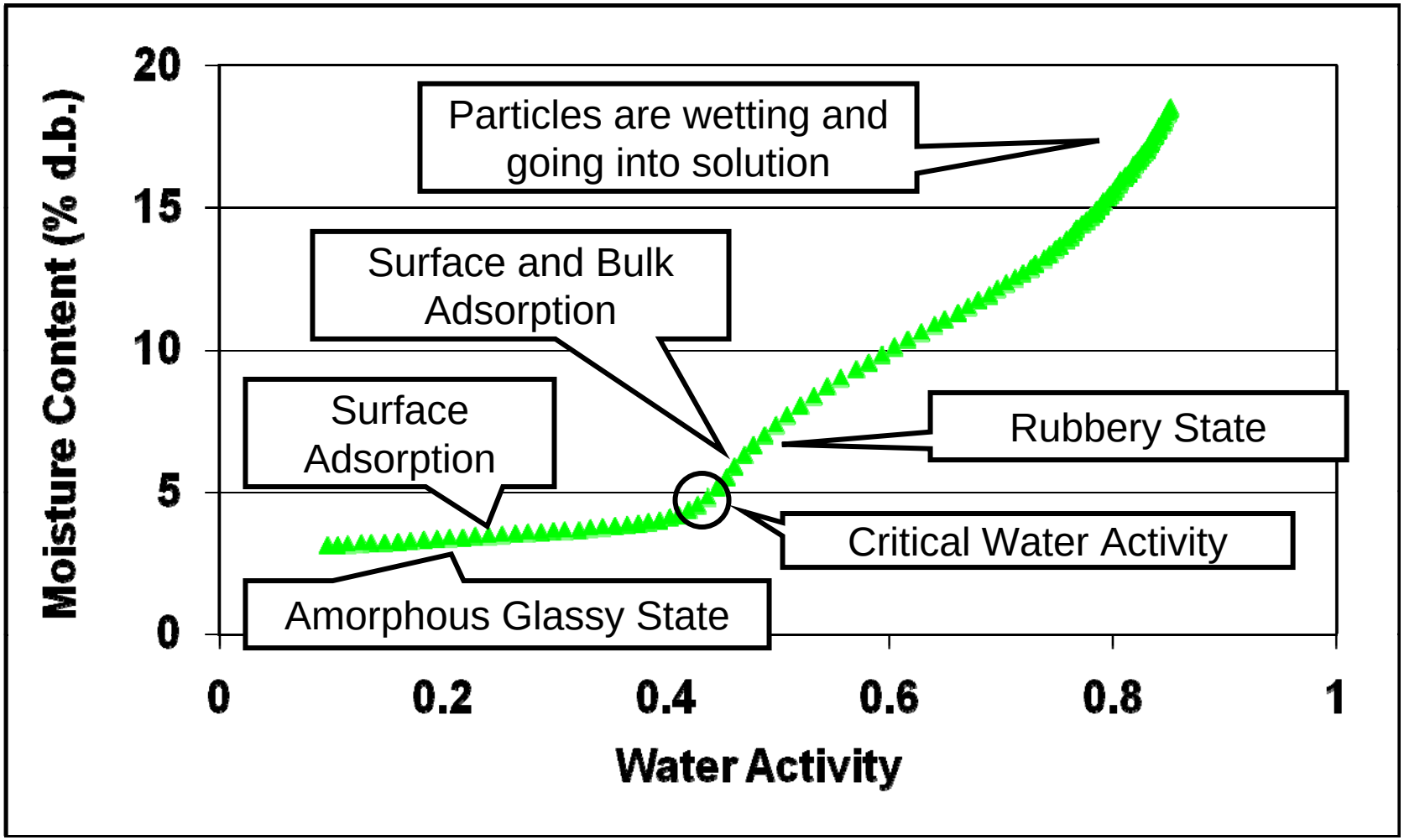
Dynamic Sorption Isotherms and Glass Transition



*Data are for Spray Dried Milk Powder

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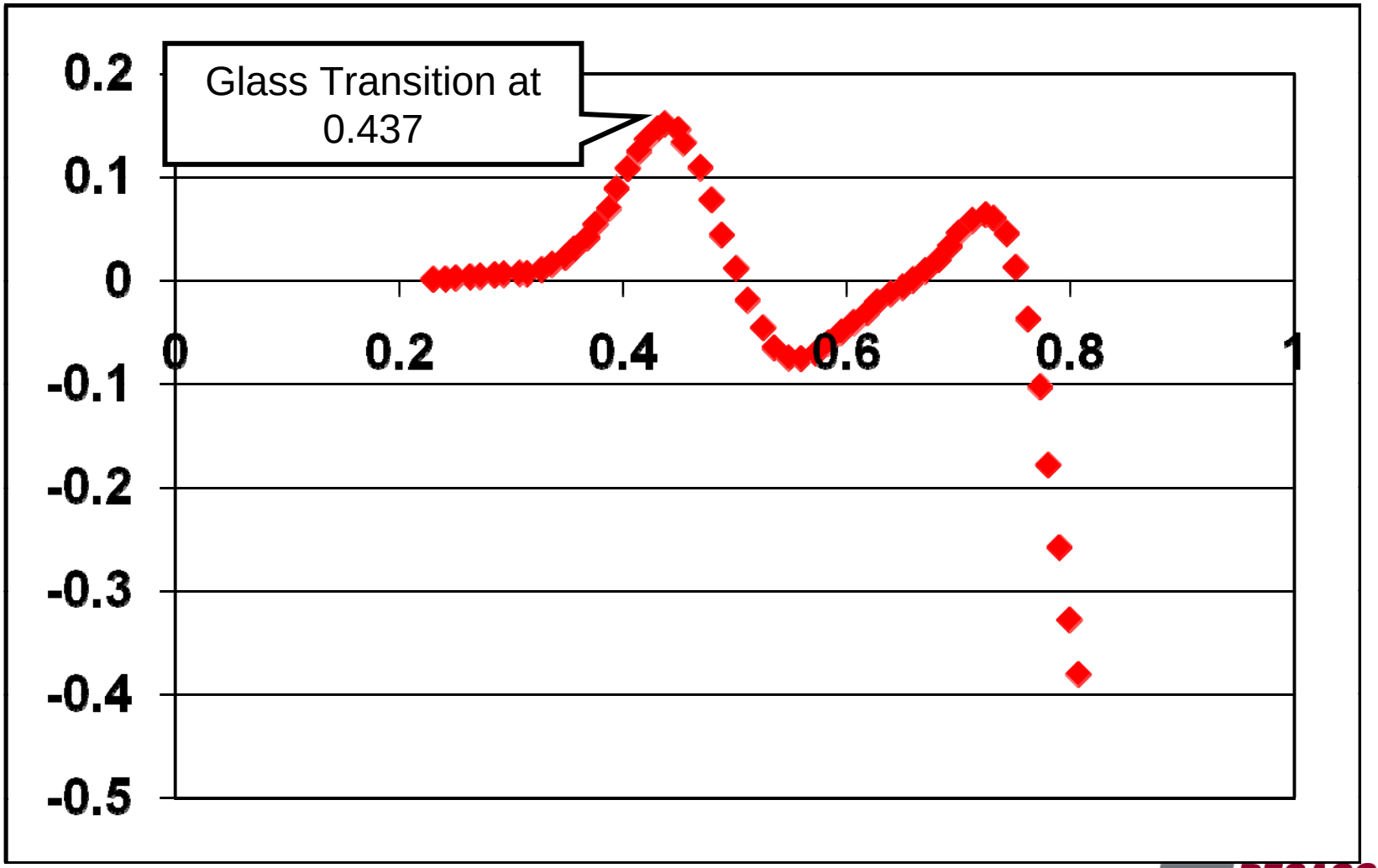
Determining Critical Water Activity



*Data are for Spray Dried Milk Powder

*2nd derivative determined using Savitsky-Golay curve smoothing

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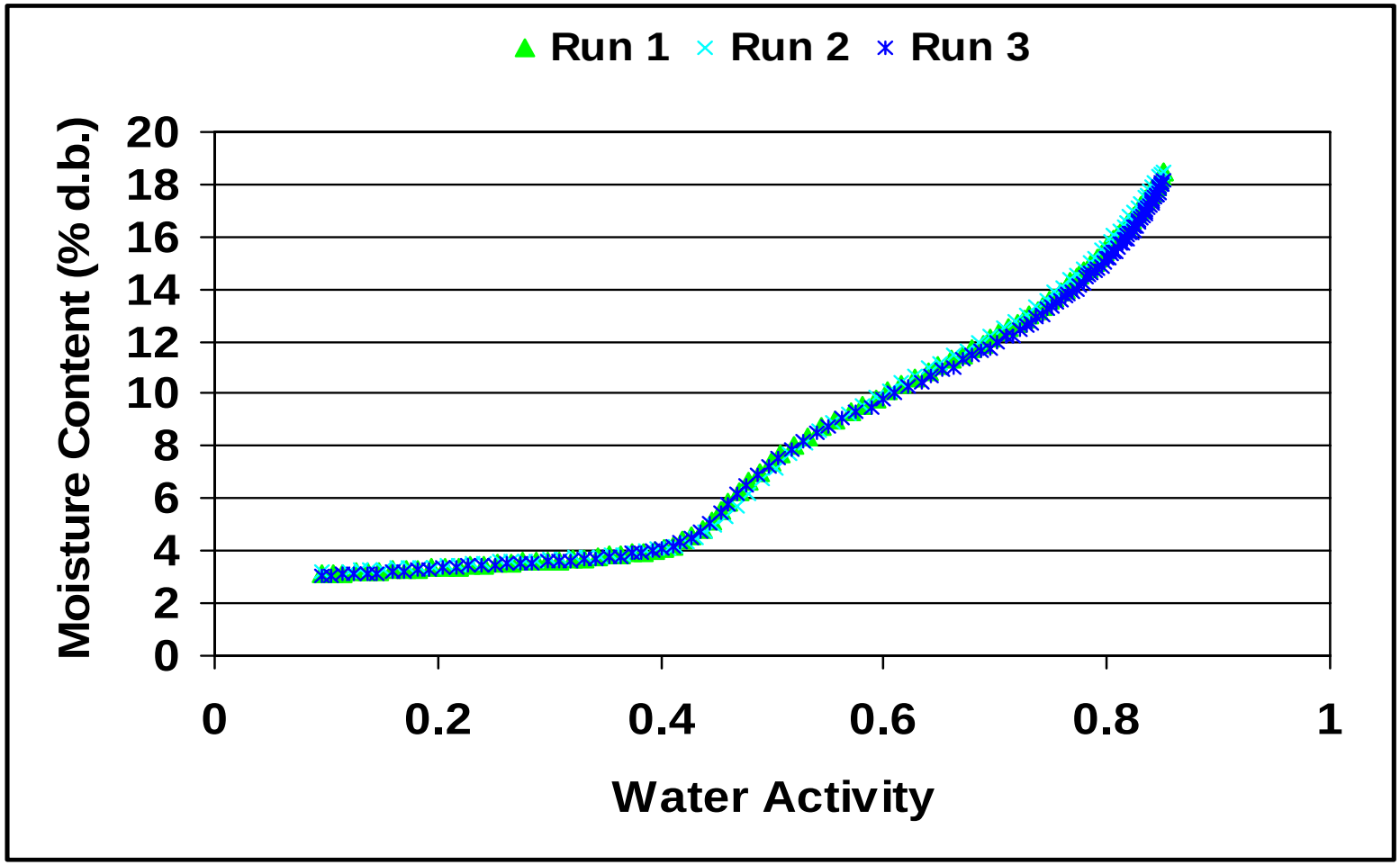
Transition is Repeatable



*Data are for Spray Dried Milk Powder

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Materials and Methods

- Commerical Spray-dried Skim Milk Powder
- Polydextrose 90% Purity
- Dynamic Dewpoint Isotherms
  - AquaSorp with flow rate at 80ml/min
  - Inflection points determined using Savitsky-Golay 2nd derivative
- Differential Scanning Calorimetry on Polydextrose
  - Q2000 DSC, Nitrogen Purge gas, Temperature scanned at 10°C/min
  - Midpoint Tg

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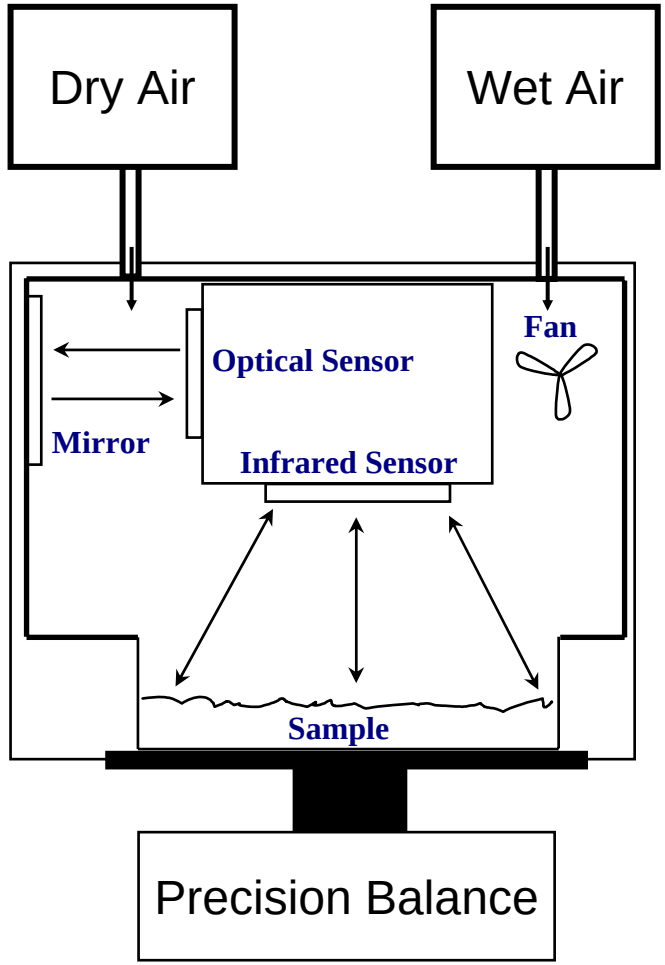
Dynamic Dewpoint Isotherm (DDI) Method

- Wet air for adsorption, dry air for desorption
- Water activity directly measured
- Equilibration to known water activity not necessary
- Fast with unequalled data resolution
- Provides a Dynamic Sorption Isotherm
- Does not provide kinetics of sorption



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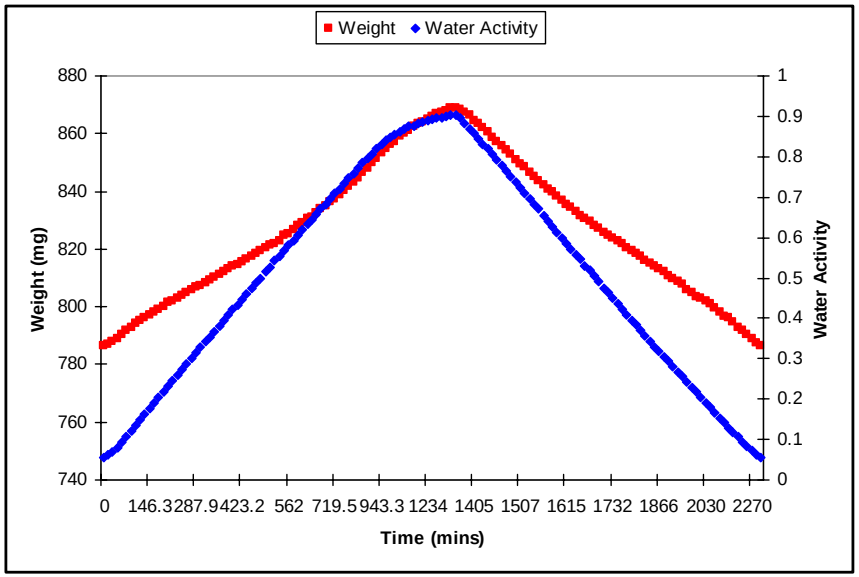
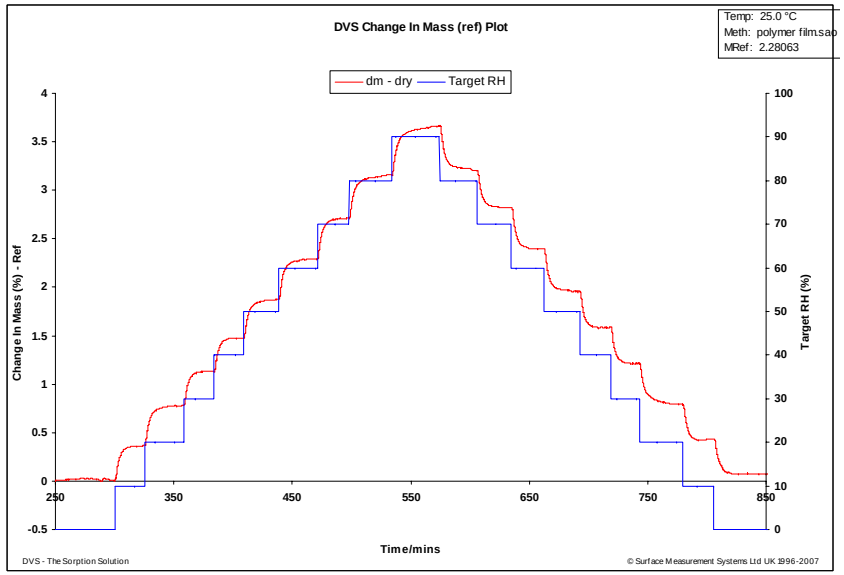
Comparing Isotherm Methods

DDI



DVS



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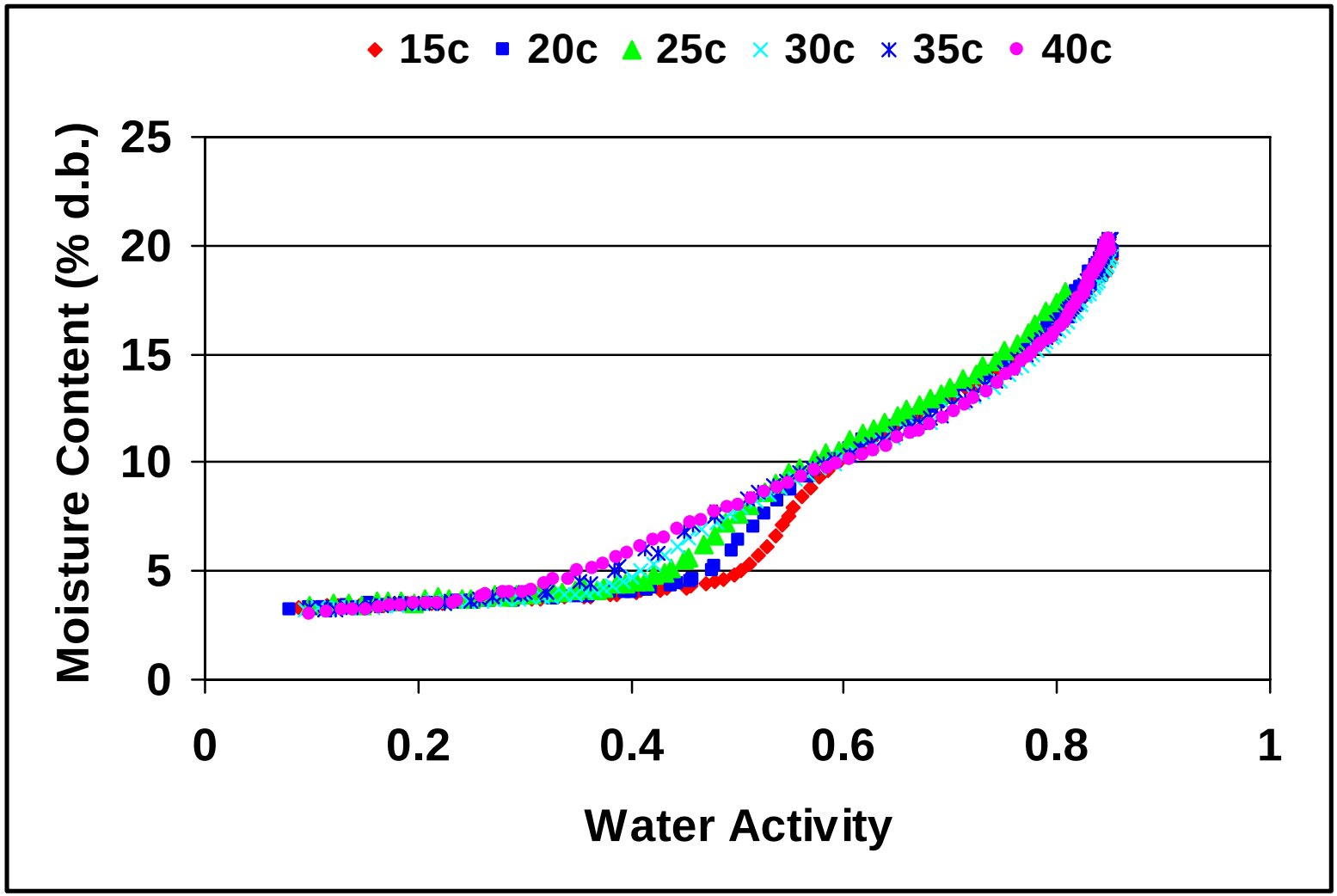
Milk Powder at Different Temperatures



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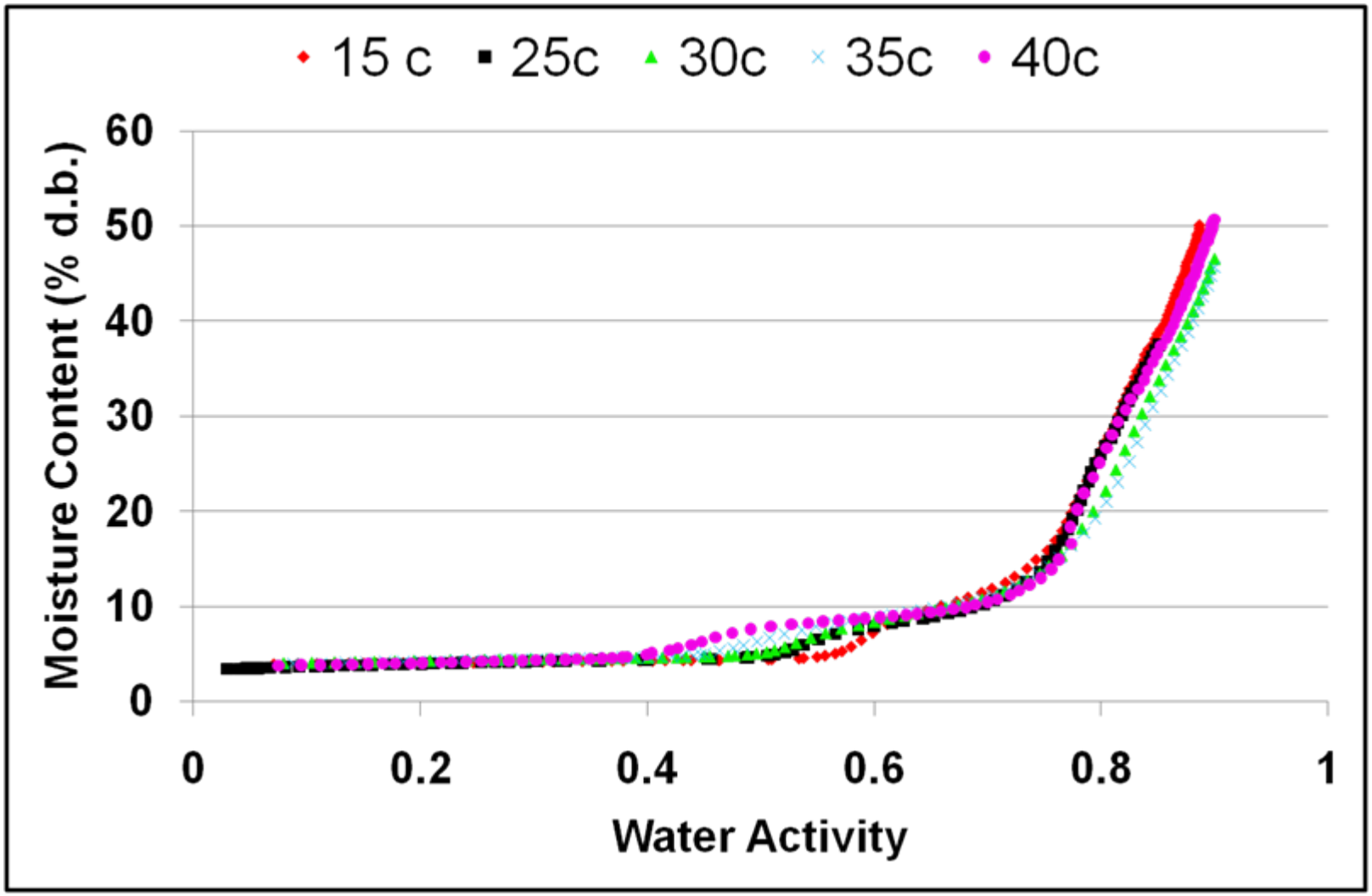
Polydextrose at Different Temperatures



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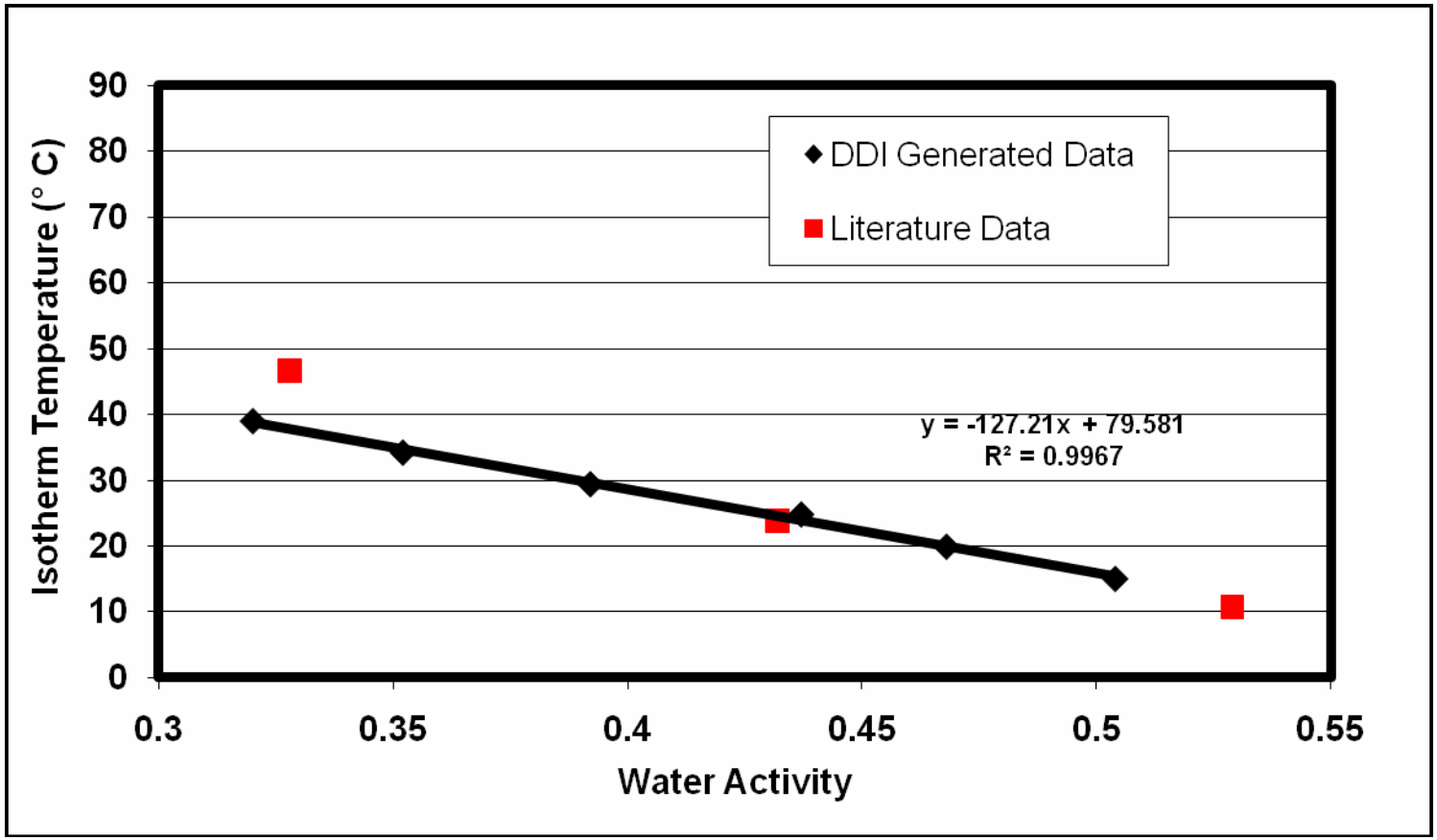
Critical Water Activity vs. Glass Transition for Spray Dried Milk Powder



*Literature values adopted from Shrestha et al 2007. Water sorption and glass transition properties of spray dried lactose hydrolyzed skim milk powder. LWR 40:1593-1600

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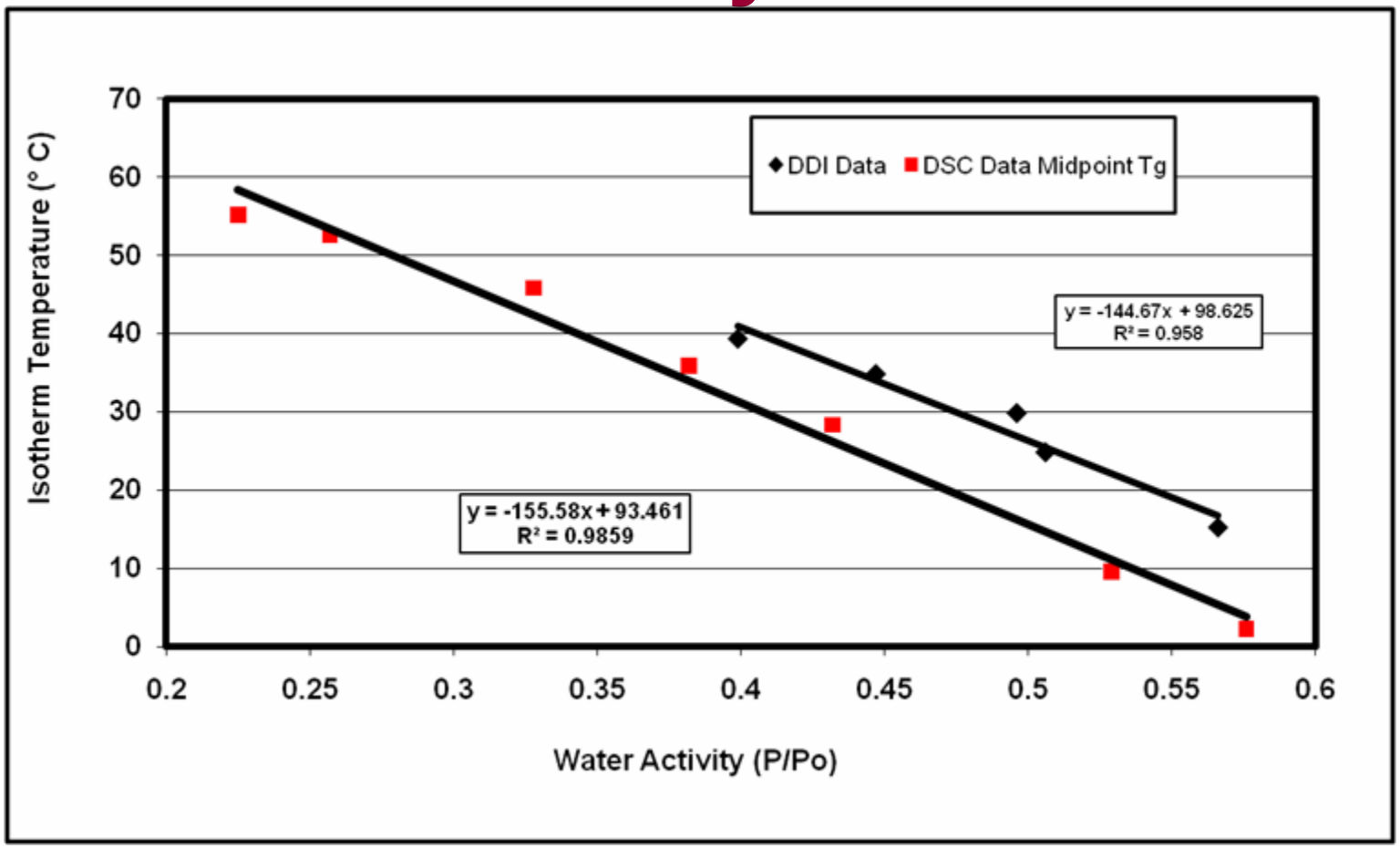
Critical Water Activity vs. Glass Transition for Polydextrose



*DSC Data taken from Xiaoda 2007. Investigation of the relationship between the critical relative humidity and the glassy to rubbery transition in polydextrose. Masters Thesis , Purdue University, Page 106.

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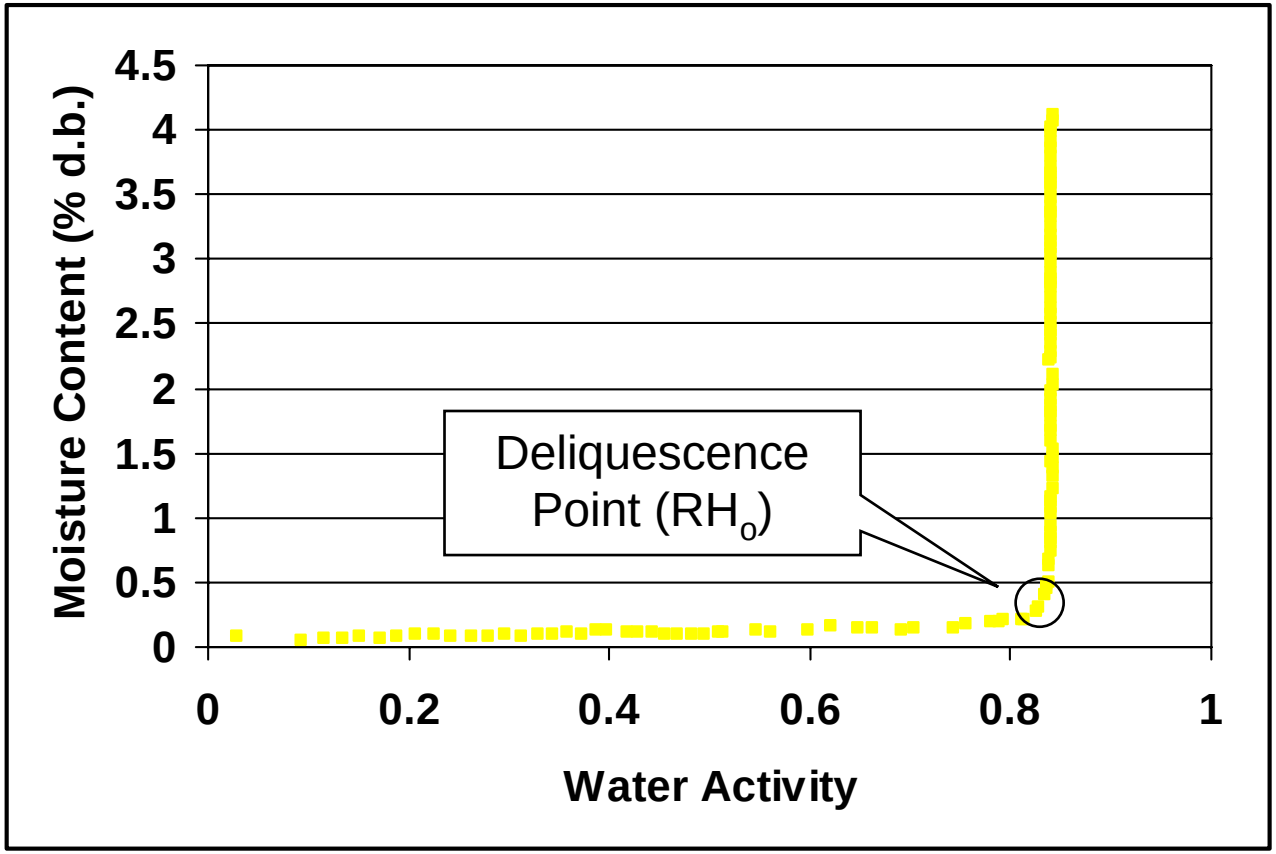
Determining Deliquescence Point



*Data are for Crystalline Sucrose

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Conclusion

- Dynamic Sorption Isotherms show distinct changes in sorption properties in the area of a glass transition
- These changes in sorption properties may not be the actual glass transition event, but a physical consequence of the transition
- Critical water activities for moisture or temperature induced glass transition can be determined
- Critical water activity at different temperatures correlates well with DSC Tg values
- There appears to be a rate dependency to the critical water activity values.
- Critical water activity can be used as a practical control measure for meta-stable amorphous materials

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Thank you

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