

# WATER ACTIVITY: REACTION RATES, PHYSICAL PROPERTIES, AND SHELF LIFE

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# SHELF LIFE AND STABILITY

- Consumers expect quality, freshness, nutritive value, and accurate label information.
- Shelf life indicates the extent of allowable product change due to
  - Time
  - Environment
- Understanding stability and factors that affect stability, such as moisture, can lead to improving shelf life and shelf life predictions.

# TYPES OF STABILITY

- Microbial
- Sensory
- Chemical
- Physical

# FOOD PRODUCT TYPES

- Dry (powders, cereals)
- Intermediate Moisture (bars)
- Solutions (beverages)

# THE STABILITY QUESTION

- A food has a sensitive “attribute”
  - Loss of sensitive ingredient
  - Formation of an undesirable product
  - Undesirable change in texture
- How does this “attribute” change due to:
  - Processing
  - Distribution
  - Storage
    - Commercial
    - Consumer
- Answers come from modeling kinetic data

# REACTION KINETIC OVERVIEW

Active Compound  $\longrightarrow$  Degradation Product(s)

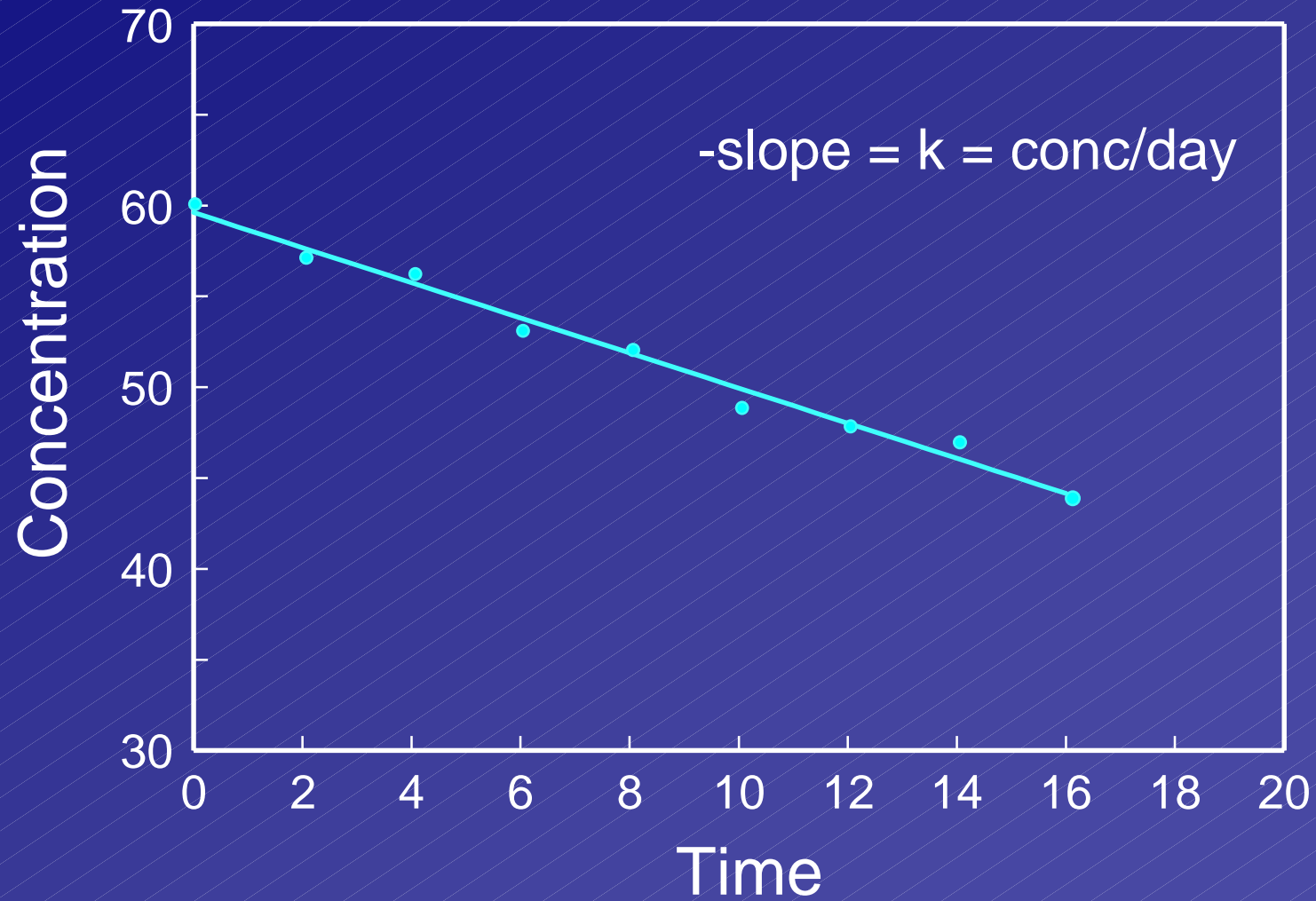
# REACTION TYPES

- Hydrolysis
- Oxidation
- Rearrangement reactions
- Amine-Carbonyl (Maillard reaction)
- Enzymatic

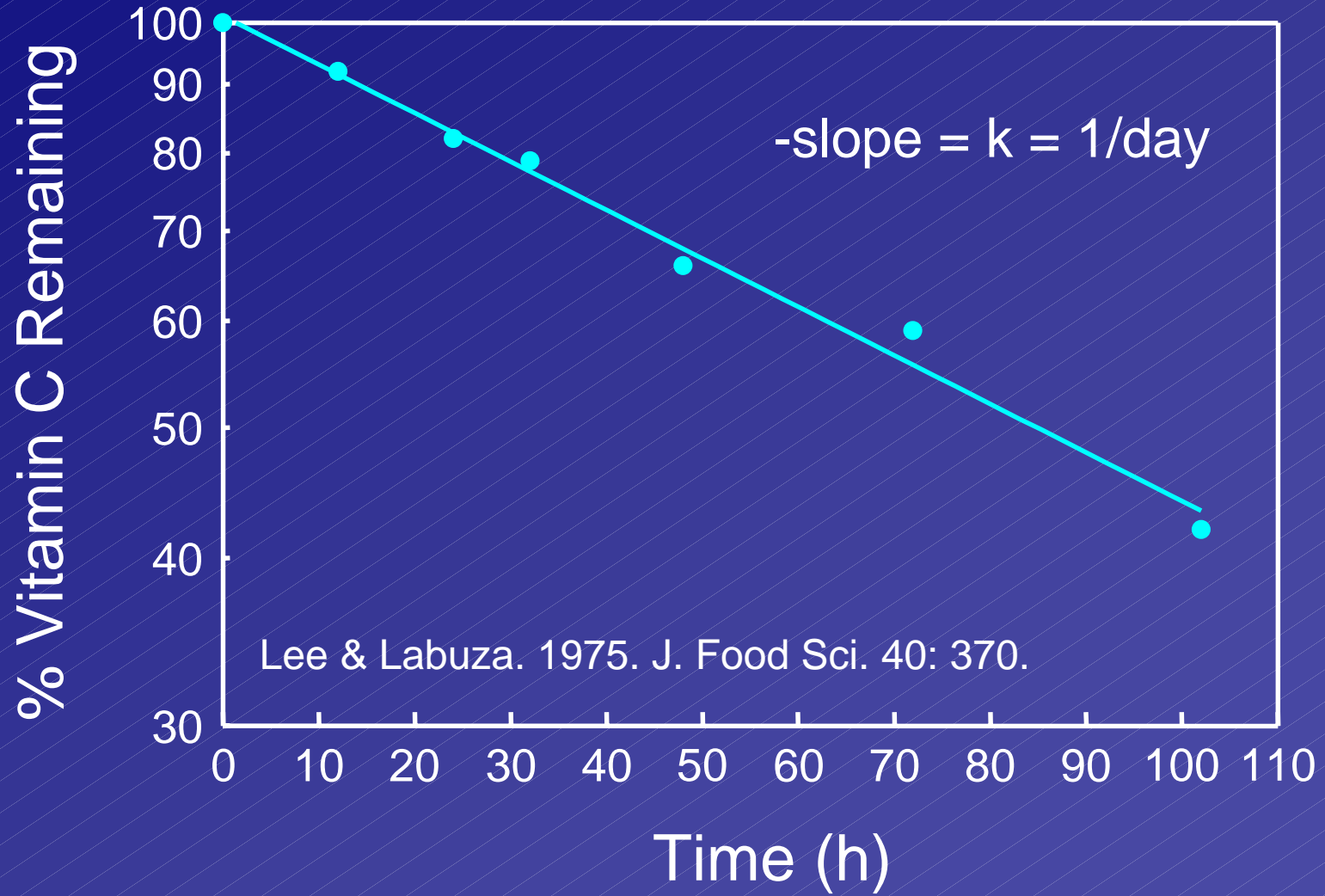
# KINETIC DATA

- zero order model:  $[A] = [A]_0 - kt$
- first order model:  $\ln([A]/[A]_0) = -kt$
- Terms
  - $k$  = rate constant
  - $t$  = time
  - $[A]$ ,  $[A]_0$  = concentration

# ZERO ORDER PLOT



# FIRST ORDER PLOT



# FACTORS INFLUENCING STABILITY

- Temperature
- Moisture
- pH
- Ingredients
- Oxygen

# TEMPERATURE

- Reaction rates increase with increasing temperature
- Models
  - Arrhenius Equation

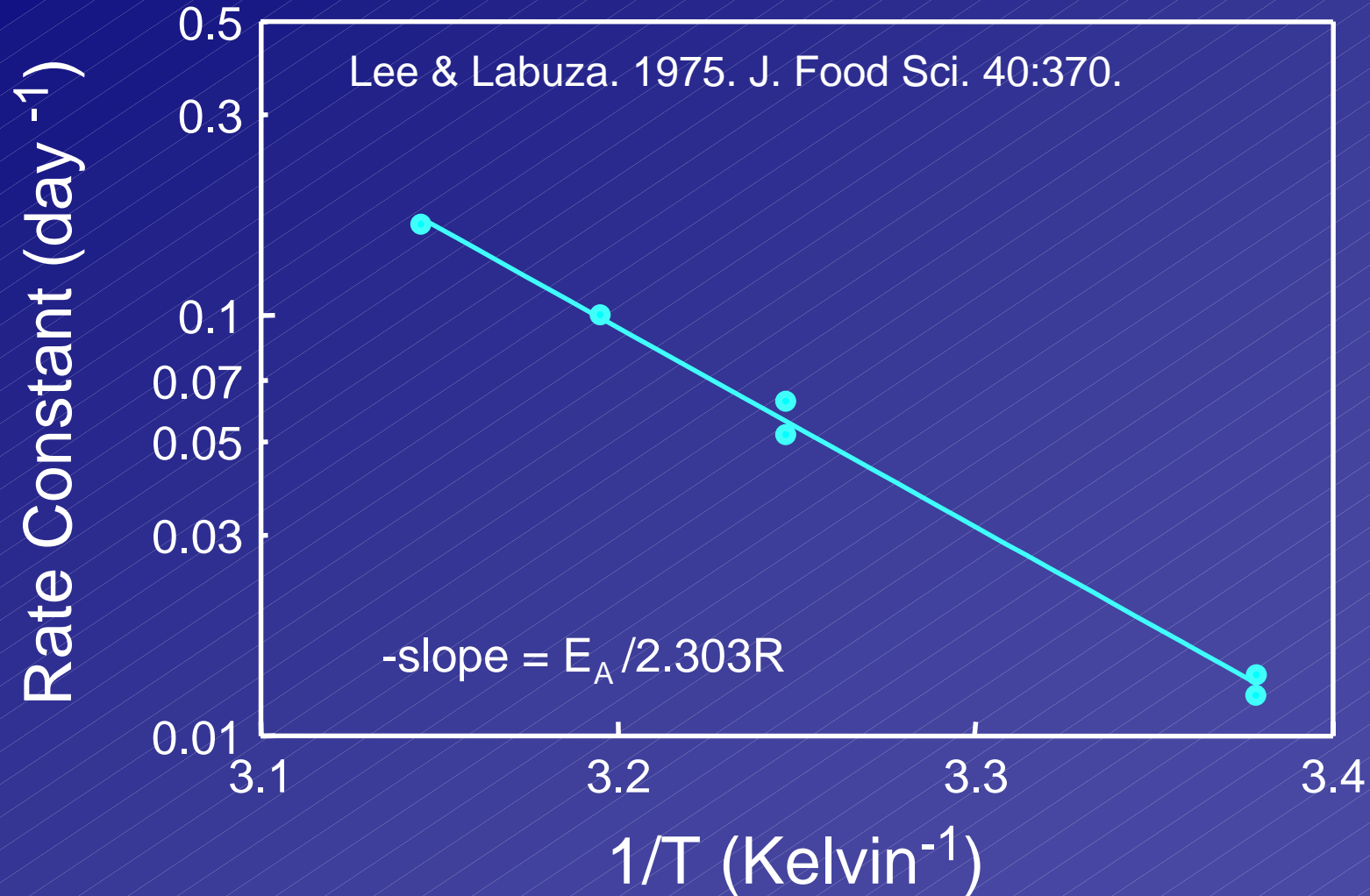
$$\ln(k) = \ln(A) - E_A / RT$$

$$\log(k) = \log(A) - E_A / (2.303RT)$$

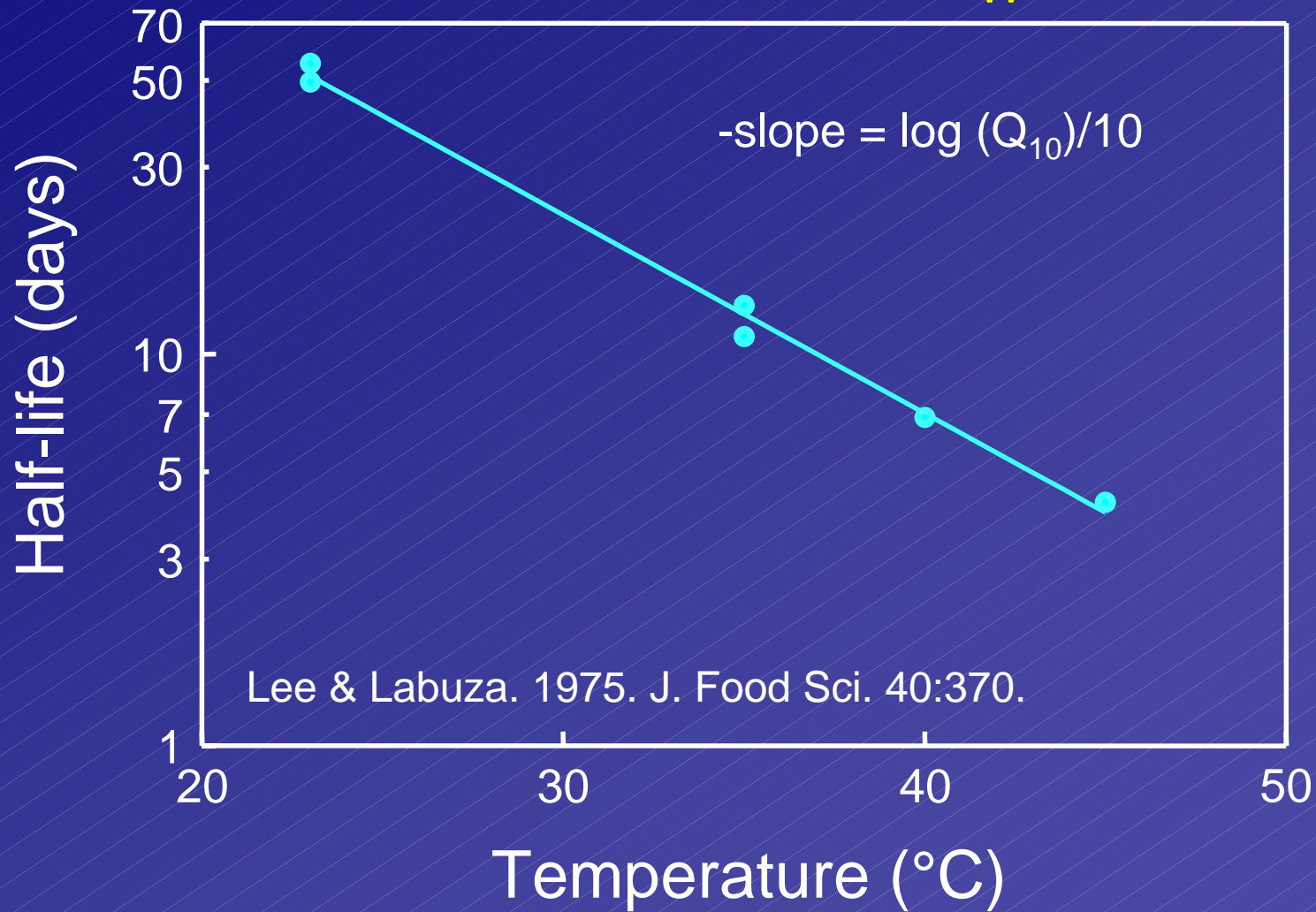
- $Q_{10}$  Approach

$$Q_{10} = (\theta_{s,T}) / (\theta_{s,T+10})$$

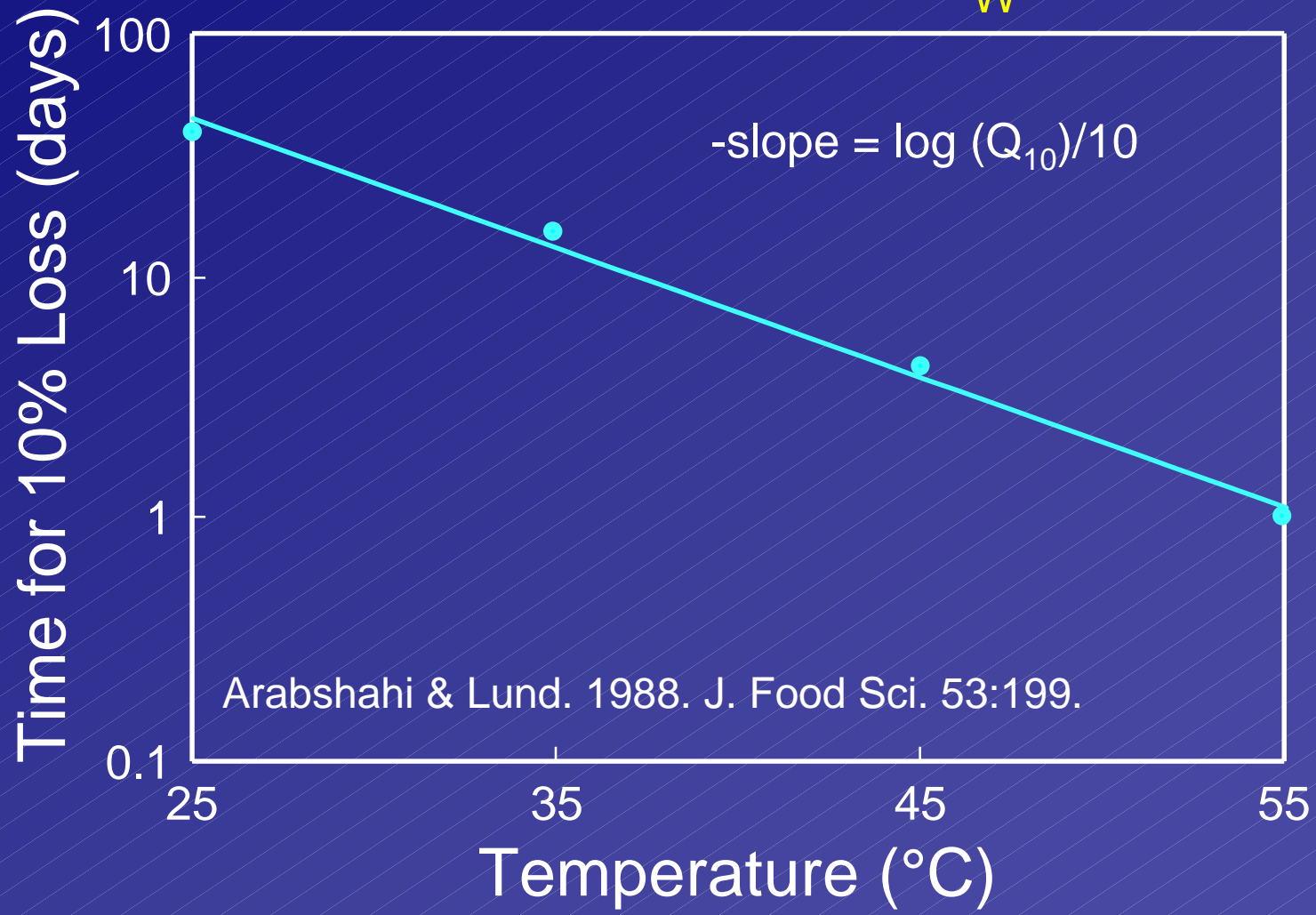
# ARRHENIUS PLOT FOR VITAMIN C DEGRADATION AT $a_w$ 0.32



# SHELF LIFE PLOT FOR VITAMIN C DEGRADATION AT $a_w$ 0.32



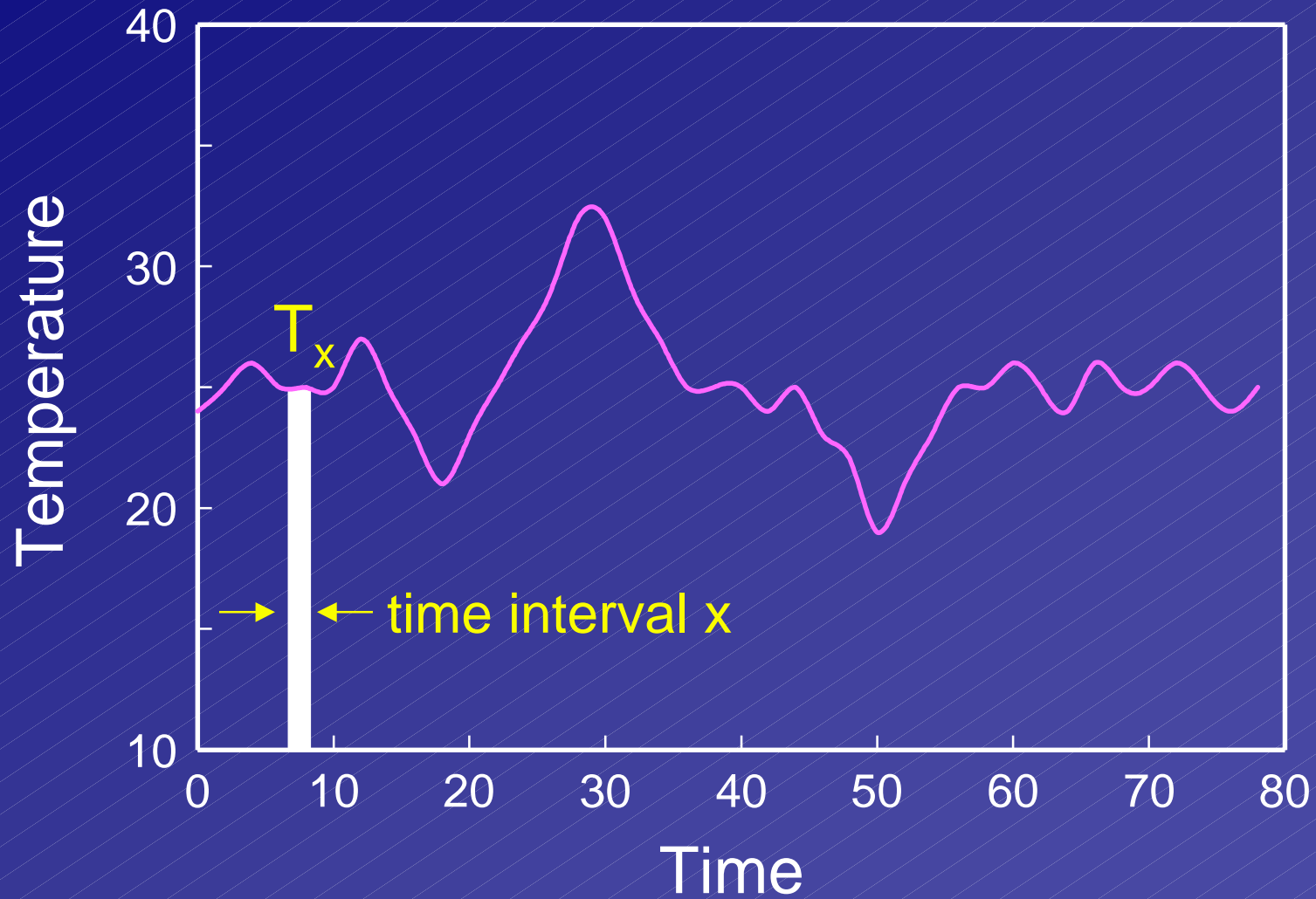
# SHELF LIFE PLOT FOR THIAMIN DEGRADATION AT $a_w$ 0.75



# SHELF LIFE AND TEMPERATURE

- Define shelf life as the time for 10% thiamin loss.
- Shelf life at 25°C is 38 days.
- Shelf life increases by 3.3 for each 10°C decrease in temperature.
- Thus, the predicted shelf life at 20°C is ≈80 days.
- Small temperature changes can have large effects on product shelf life.

# TEMPERATURE FLUCTUATIONS



# TEMPERATURE FLUCTUATIONS

## Fraction of Shelf Life Consumed ( $f_{con}$ )

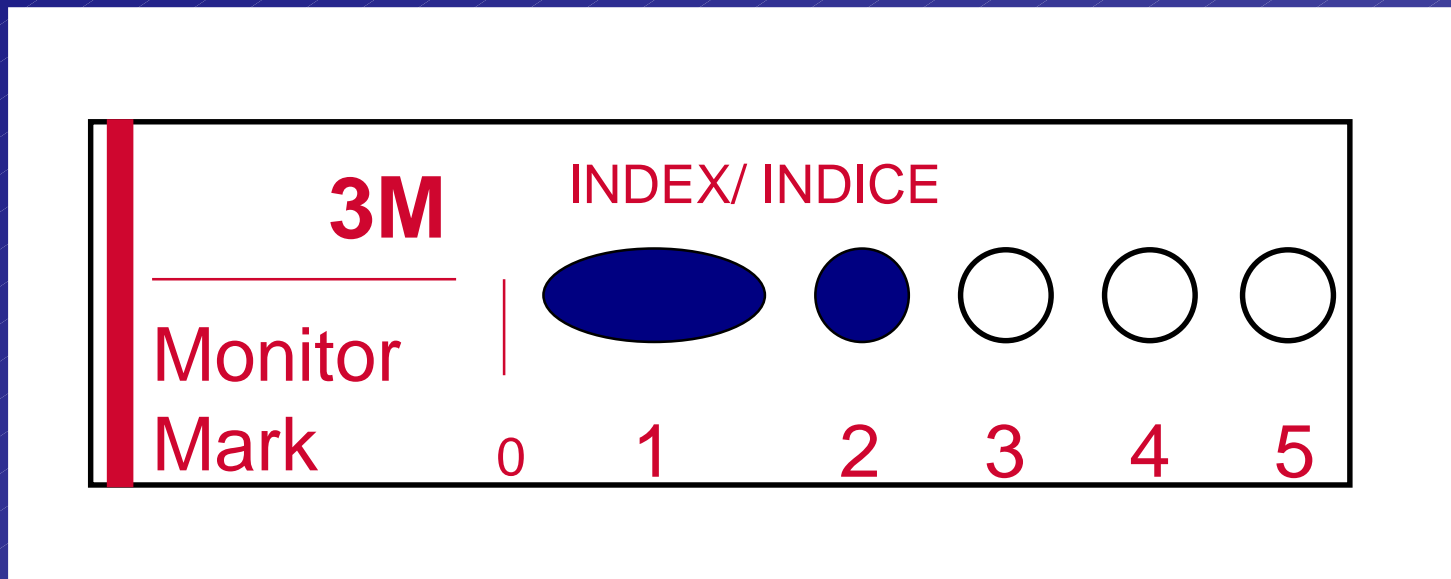
$$f_{con} = \sum \frac{\text{time at temperature } x_i}{\text{total shelf life at temperature } x_i}$$

$^{\circ}\text{C}$	Shelf Life	Time	f
20	150 d	33 d	0.22
30	75 d	7 d	0.093
40	38 d	2 d	0.053

$$f_{con} = 0.366$$

# TIME TEMPERATURE INDICATORS

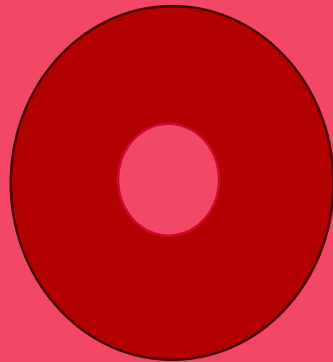
- Gives a visual indication of net exposure to time and temperature



MonitorMark is a registered trademark of 3M.

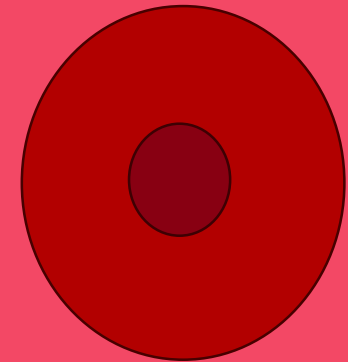
# TIME TEMPERATURE INDICATORS

## ***Quality Indicator***



Use or freeze before center is darker than outer ring.

## ***Quality Indicator***



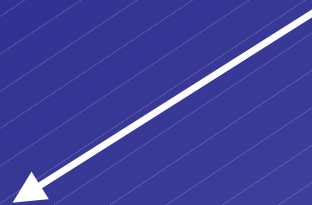
Use or freeze before center is darker than outer ring.

# WATER & CHEMICAL STABILTY

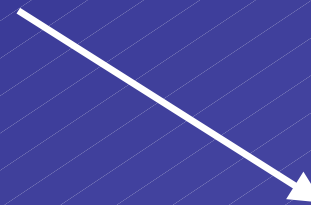
MOISTURE  
DIFFUSION



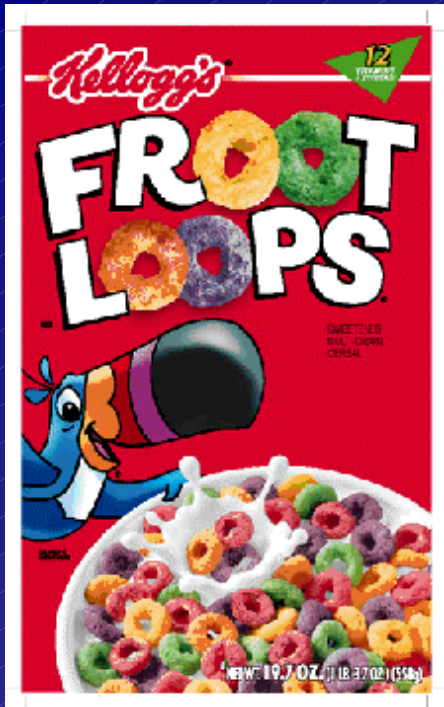
MOISTURE  
DIFFUSION



CHEMICAL  
DETERIORATION



PHYSICAL  
DETERIORATION



OPEN →



← WATER

↙  
CHEMICAL  
DETERIORATION

↘  
PHYSICAL  
DETERIORATION

# PERSPECTIVES ON WATER

- Moisture Content
- Water Activity (relative vapor pressure)
- Glass Transition (plasticizer effect)
- Water Mobility

# WATER ACTIVITY

- Determines direction of moisture transfer
- Most reaction rates increase with increasing water activity
- Most rates correlate better with water activity than moisture content
- Moisture sorption isotherms are useful

# GLASS-RUBBER TRANSITION

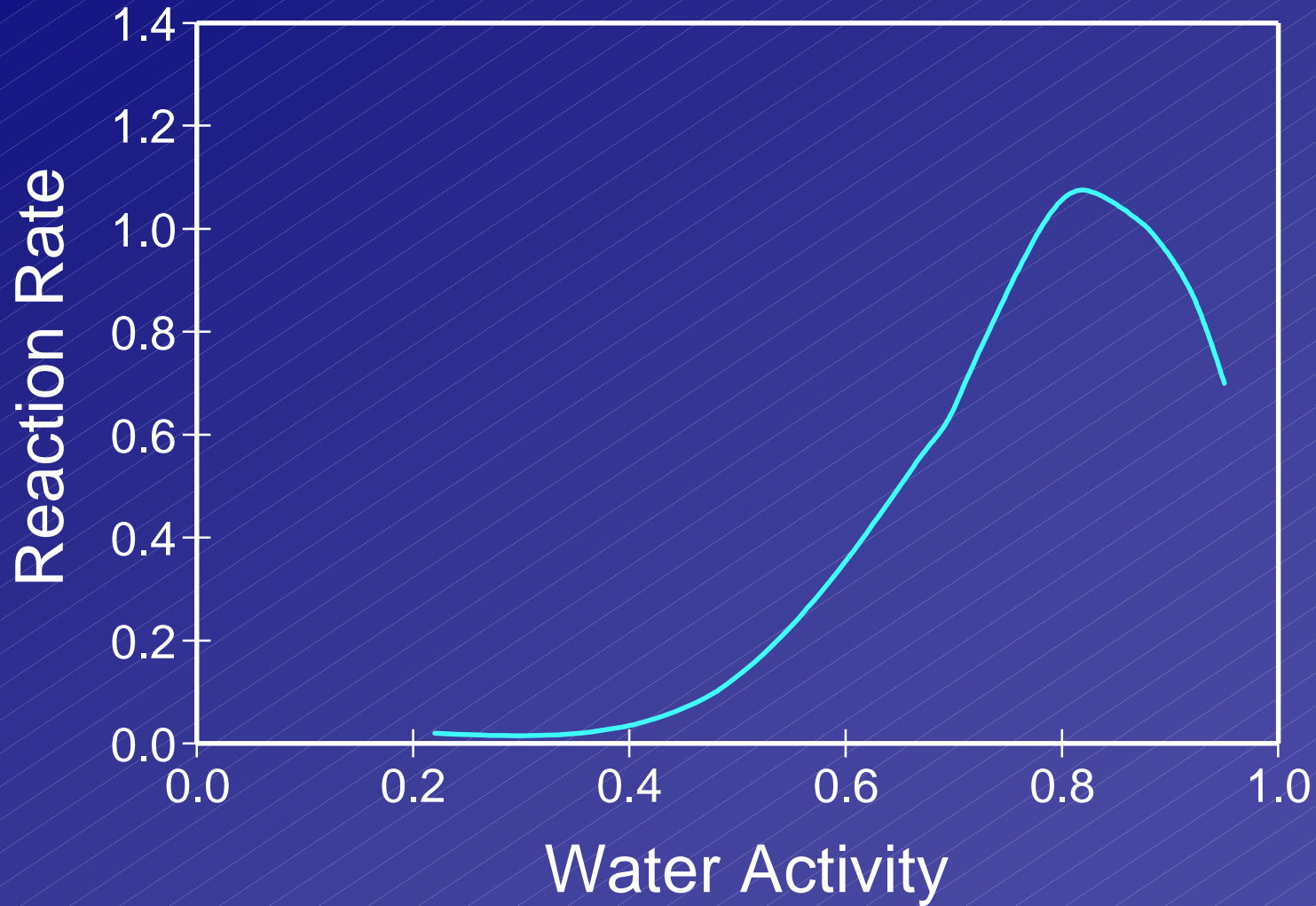


- Transition due to:
  - Temperature
  - Moisture
- Depicted by state diagram
- Relates to physical properties of system
  - Rigid glassy matrix converts into soft rubbery matrix
- Affects reactant mobility

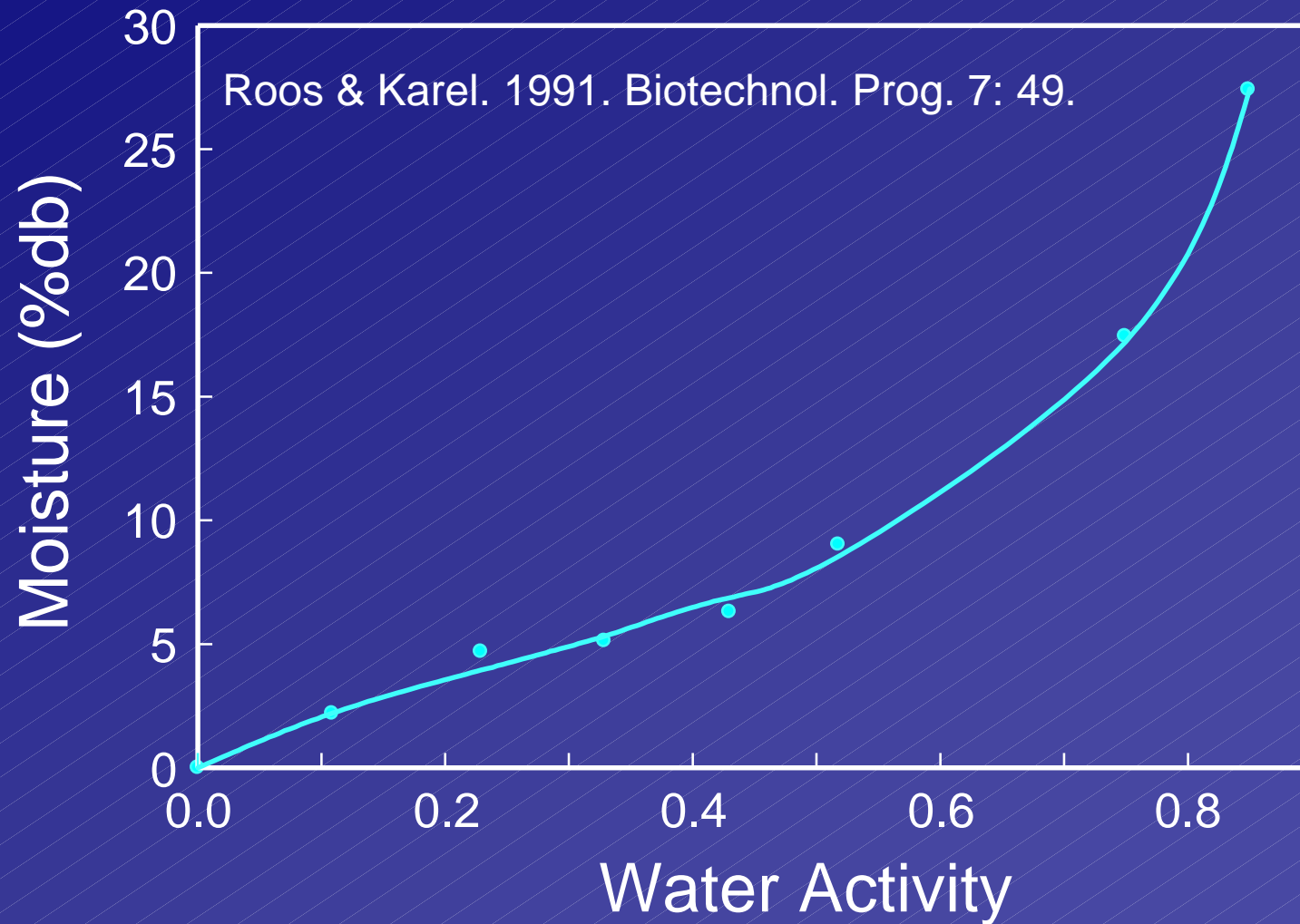
# QUESTION OF THE 1990's

Does moisture affect reactions by water activity or by increasing reactant mobility with its plasticizing ability?

# GENERAL EFFECT OF WATER ACTIVITY ON REACTION RATES



# MOISTURE SORPTION ISOTHERM FOR MALTODEXTRIN (DE 25) AT 25°C



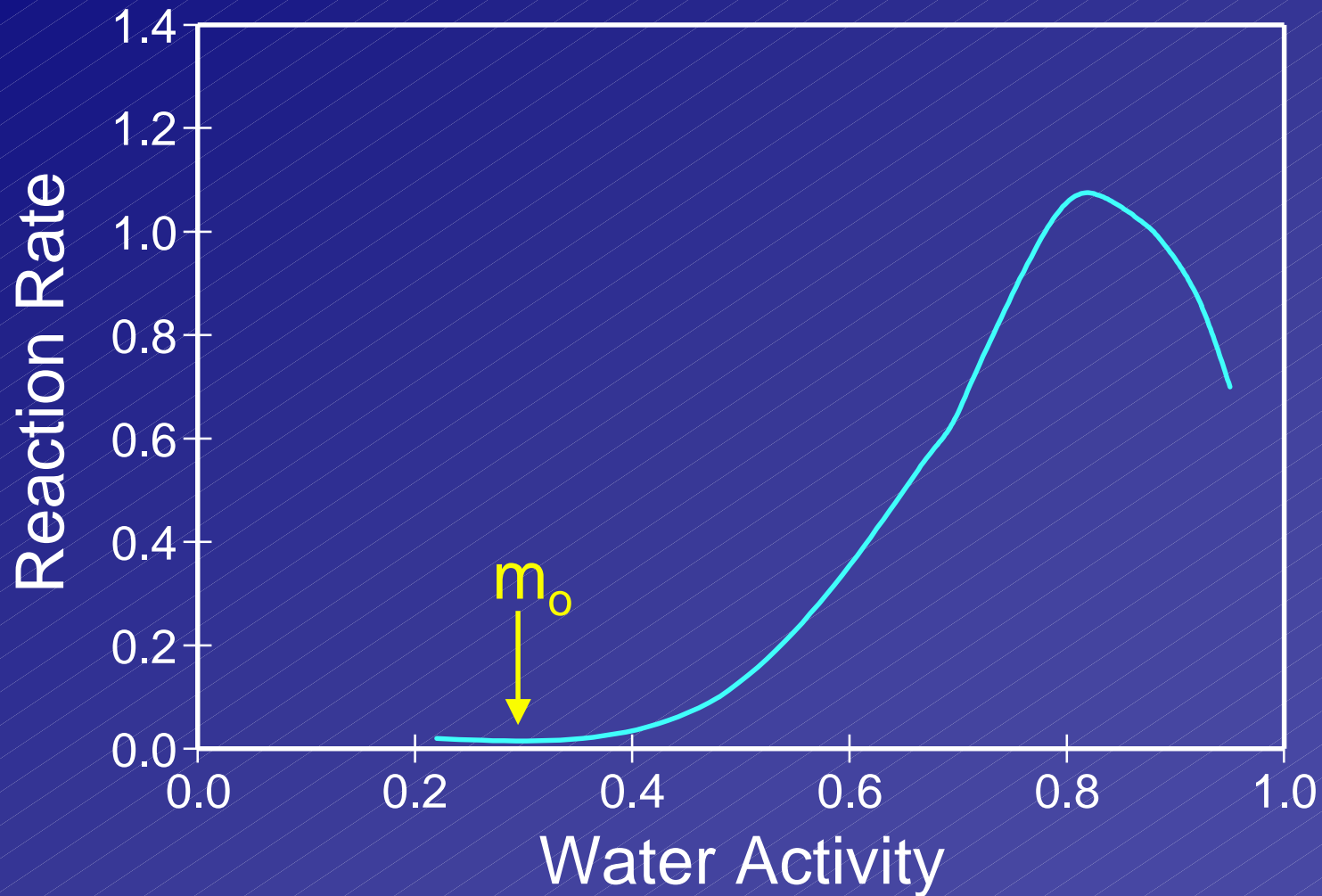
# MODELING MOISTURE SORPTION ISOTHERM DATA

- GAB equation

$$m = \frac{m_o k C a_w}{(1 - k a_w)(1 - k a_w + k C a_w)}$$

- Terms
  - $m_o$  = monolayer
  - $k$  = multilayer factor
  - $C$  = heat constant

# GENERAL EFFECT OF WATER ACTIVITY ON REACTION RATES



# GLUCOSE MOVEMENT IN DRIED CARROTS MONOLAYER = 7.2%(db)

Moisture % (db)	Movement Detected
5.9	No
8.1	Yes
10.2	Yes
13.9	Yes
18.1	Yes
21.7	Yes
26.3	Yes

Duckworth & Smith. 1963. Recent Advances in Food Science-3.  
London: Butterworths. pp 230-238.

# GLUCOSE MOVEMENT IN DRIED CARROTS MONOLAYER = 7.2%(db)

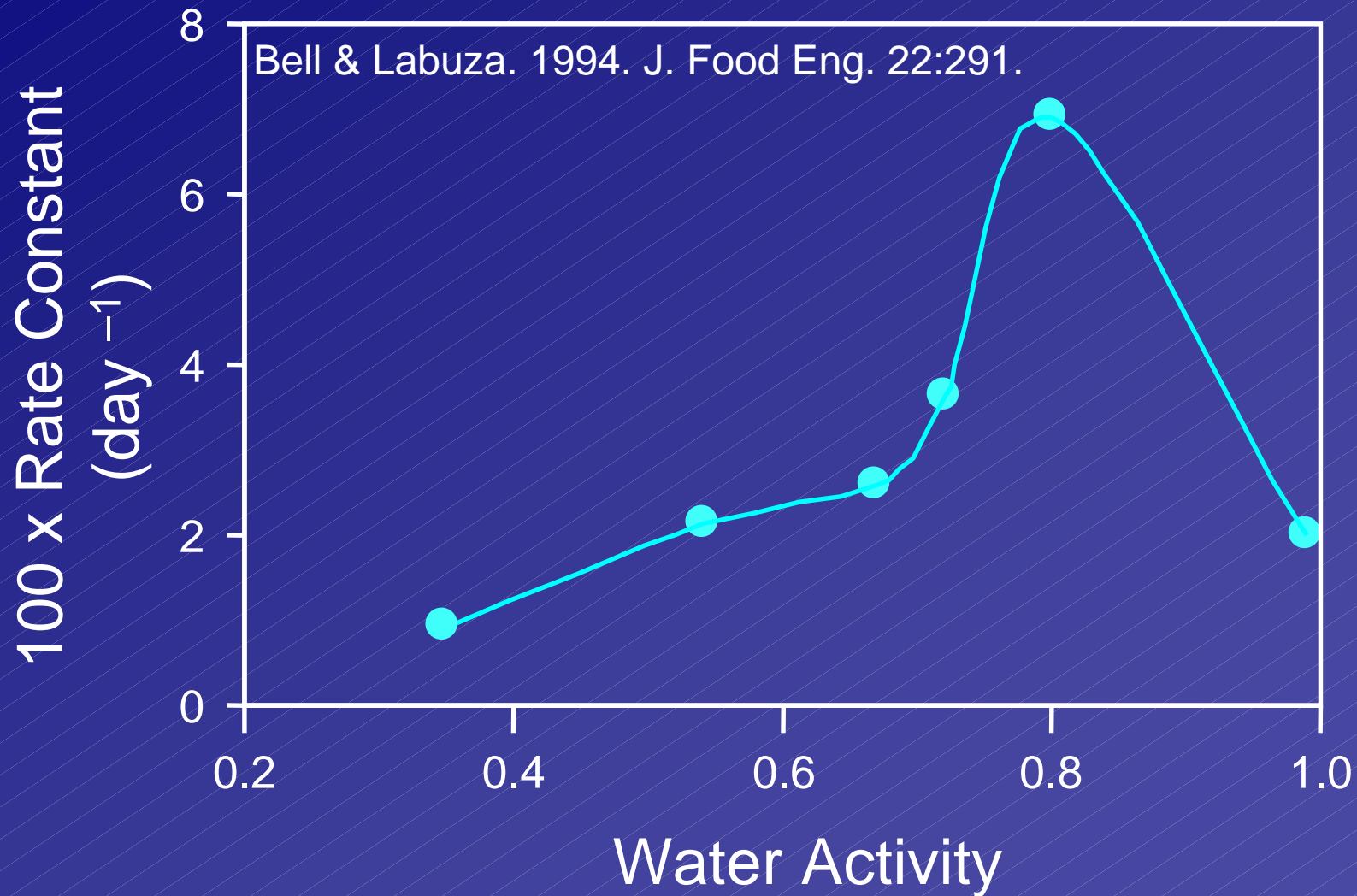
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# ASPARTAME DEGRADATION

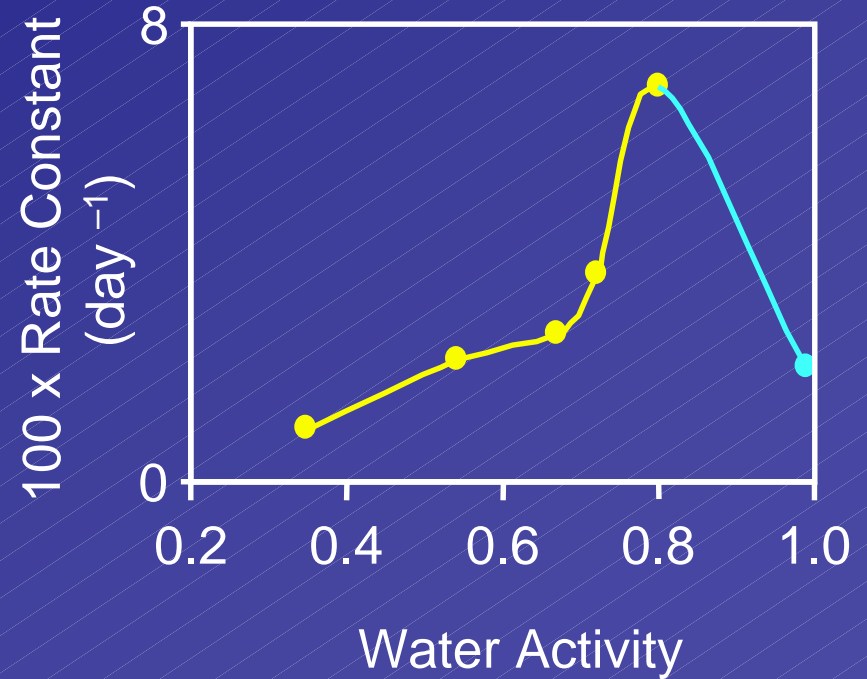
- Rearrangement reaction at  $\text{pH} > 6$
- Hydrolysis reaction at  $\text{pH} < 4$
- Both reaction types between  $\text{pH} 4$  and  $6$
- Catalyzed by buffer salts
- Follows pseudo-first order kinetics

# ASPARTAME DEGRADATION AS A FUNCTION OF WATER ACTIVITY AT pH 3 AND 30°C



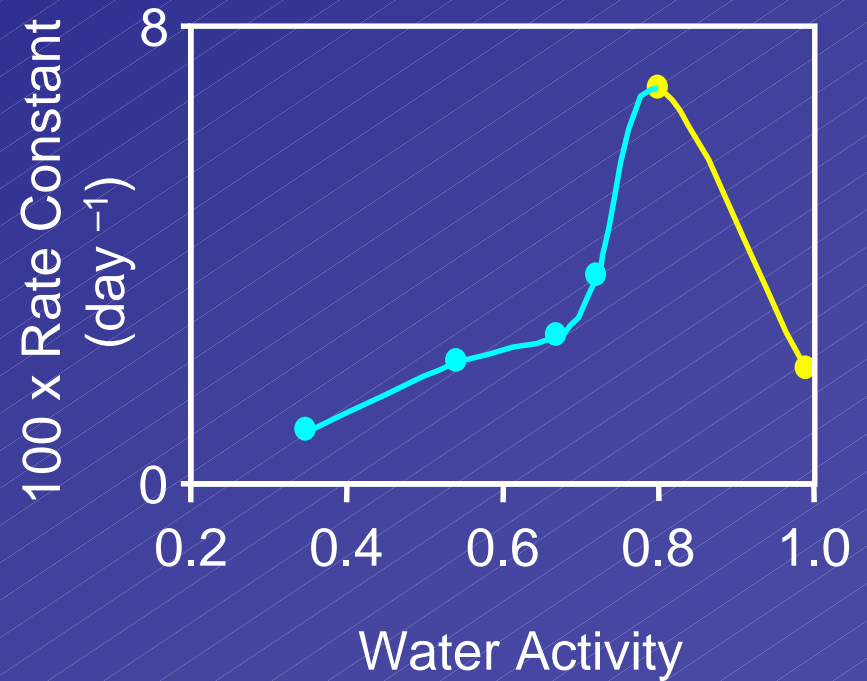
# ASPARTAME DEGRADATION

- Water activity 0.3 to 0.8
  - Solutes dissolving
  - Mobility increasing
  - Reactivity increasing



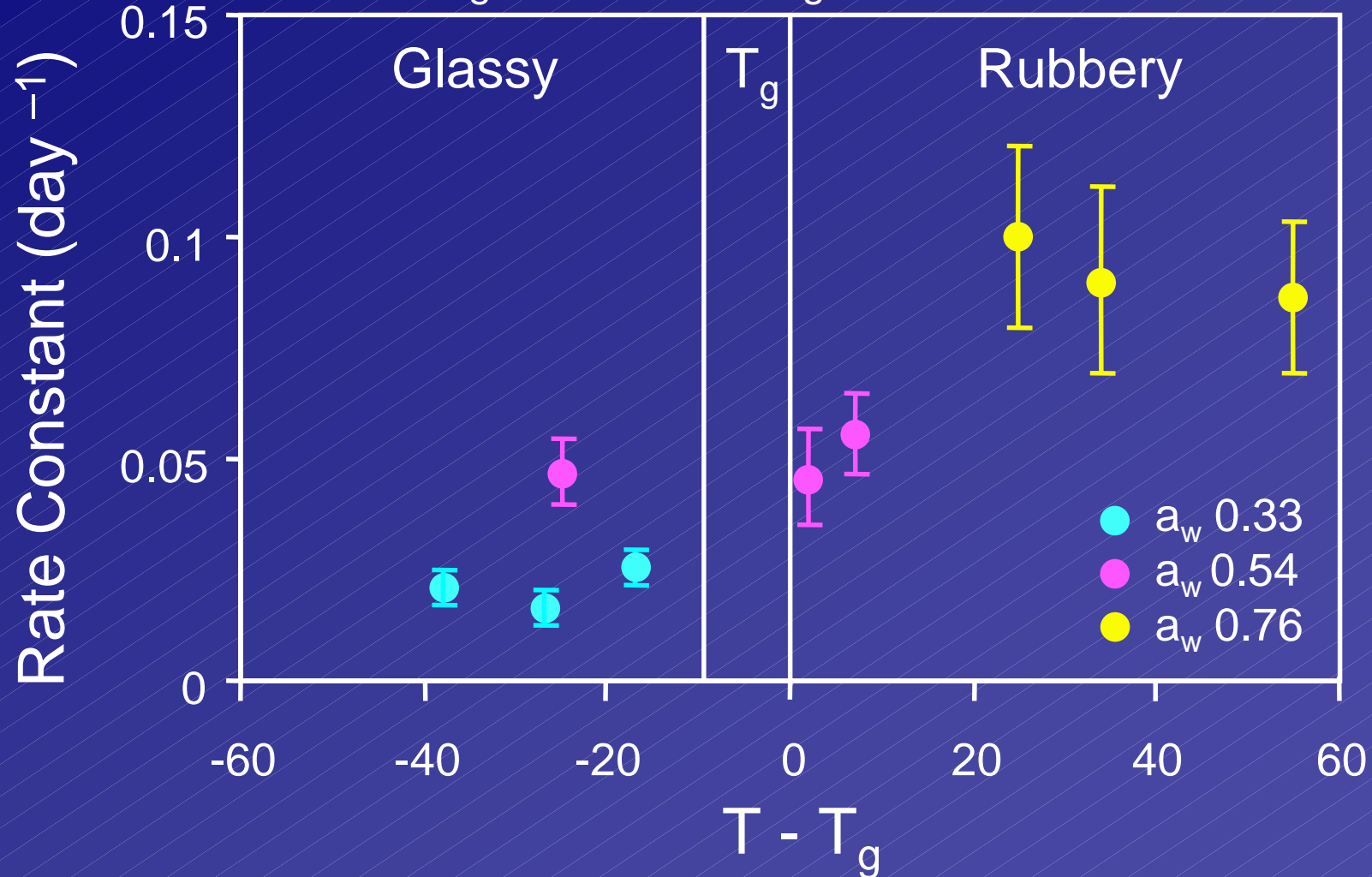
# ASPARTAME DEGRADATION

- Water activity 0.8 to 1.0
  - Solutes becoming diluted
  - Reactivity decreasing

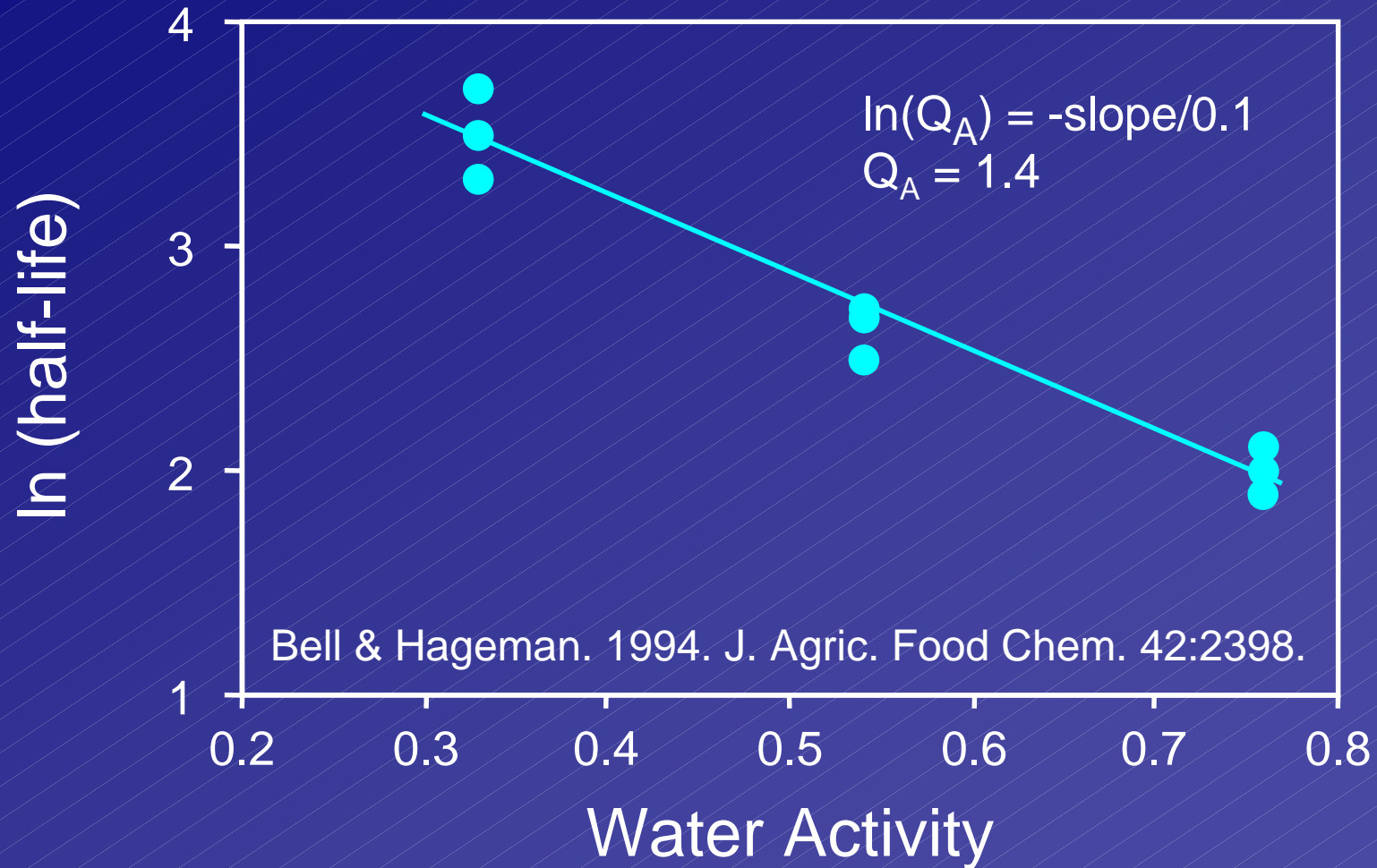


# EFFECT OF $T_g$ ON ASPARTAME DEGRADATION IN POLYVINYLPIRROLIDONE (PVP) AT 25°C AND pH 7

Bell & Hageman. 1994. J. Agric. Food Chem. 42:2398.



# $Q_A$ PLOT FOR ASPARTAME DEGRADATION IN PVP AT 25°C AND pH 7

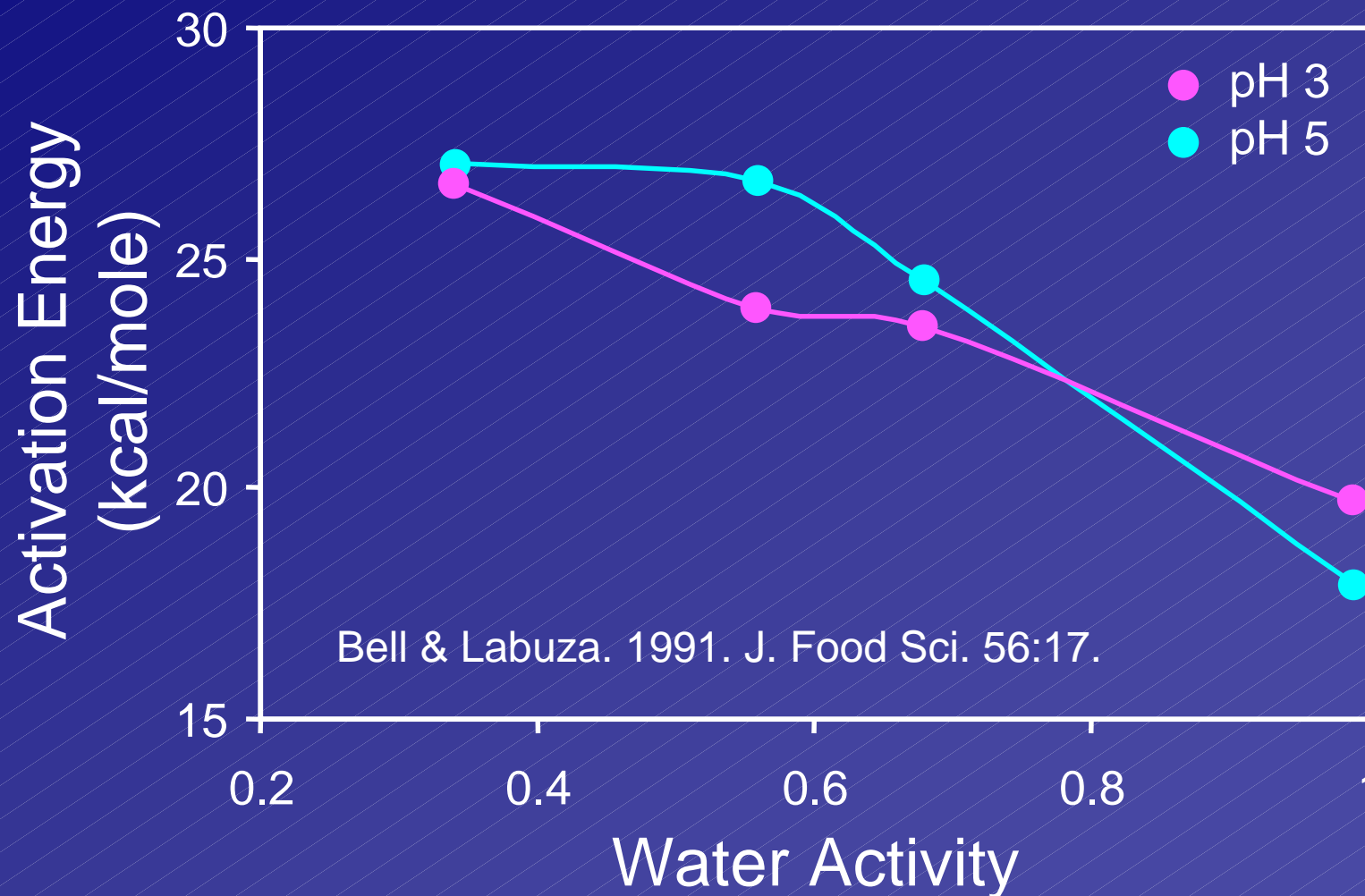


# $Q_A$ CONCEPT

$$Q_A = \frac{\text{half-life at } a_w}{\text{half-life at } a_w + 0.1}$$

- $Q_A = 1.4$  means half-life decreases by 40% for each 0.1  $a_w$  increase
- If  $a_w$  decreases by 0.1, half-life increases by 1.4
  - 33 days at  $a_w$  0.33 (experimental)
  - 46 days at  $a_w$  0.23 (predicted)
  - 65 days at  $a_w$  0.13 (predicted)

# RELATION BETWEEN ACTIVATION ENERGY OF ASPARTAME DEGRADATION AND WATER ACTIVITY



# WATER ACTIVITY EFFECTS ON $Q_{10}$ VALUES OF ASPARTAME DEGRADATION AT pH 5

$a_w$	$Q_{10}$
0.33	4.3
0.55	4.2
0.65	3.7
0.99	2.6

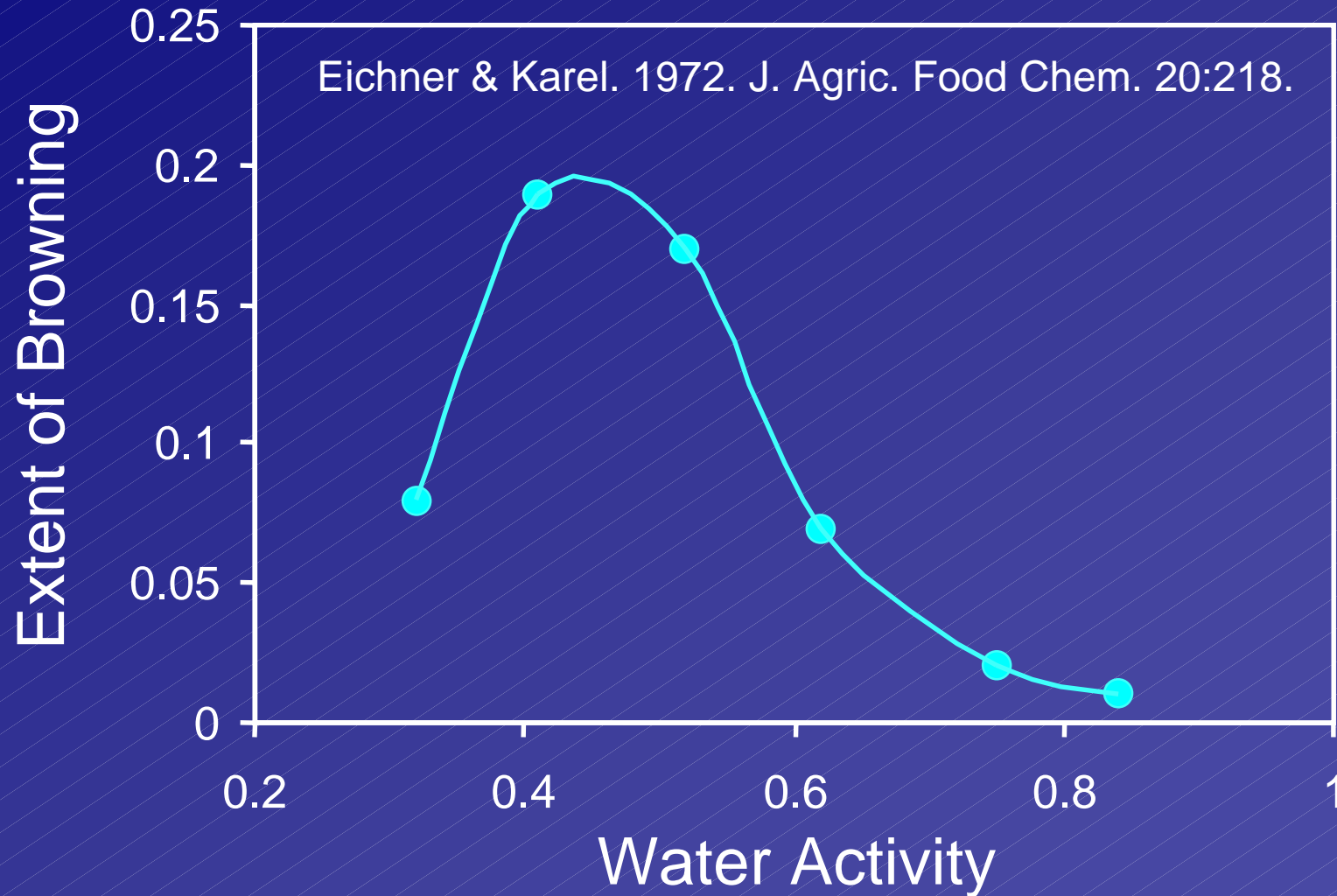
# WATER ACTIVITY AND TEMPERATURE

- Activation energies often decrease as water activity increases
- Sensitivity of a reaction to temperature changes as  $a_w$  changes
- A temperature increase has a larger effect (higher  $Q_{10}$ ) on a reaction in low moisture food than in a high moisture food
- However, the “dryness” adds stability, compensating for the food being more temperature sensitive

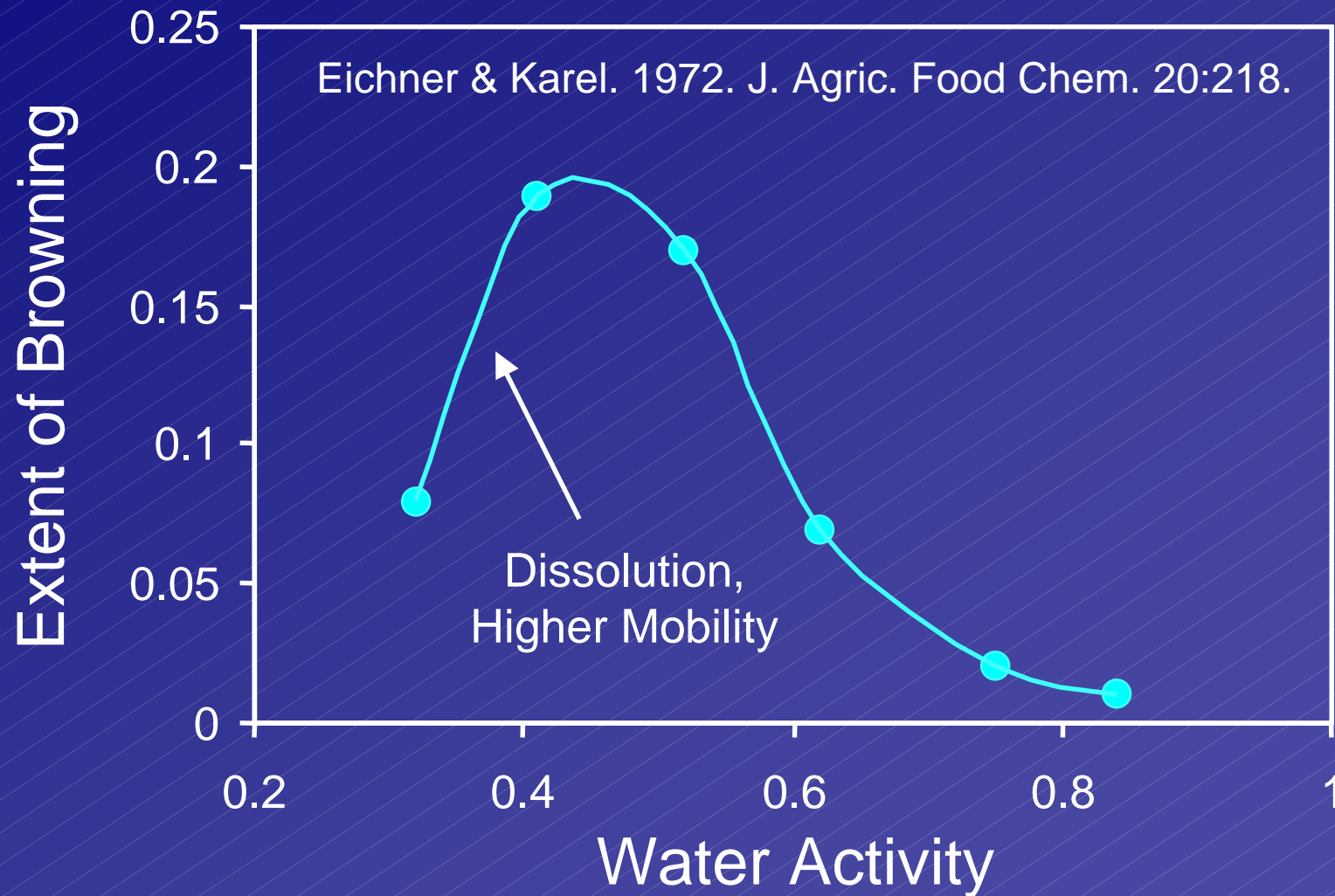
# MAILLARD REACTION (NONENZYMATIC BROWNING)

- Bimolecular reaction involving:
  - unprotonated amines (amino acid)
  - carbonyl compounds (reducing sugars)
- Reactant loss follows second order kinetics
- Brown pigment formation modeled via pseudo-zero order kinetics

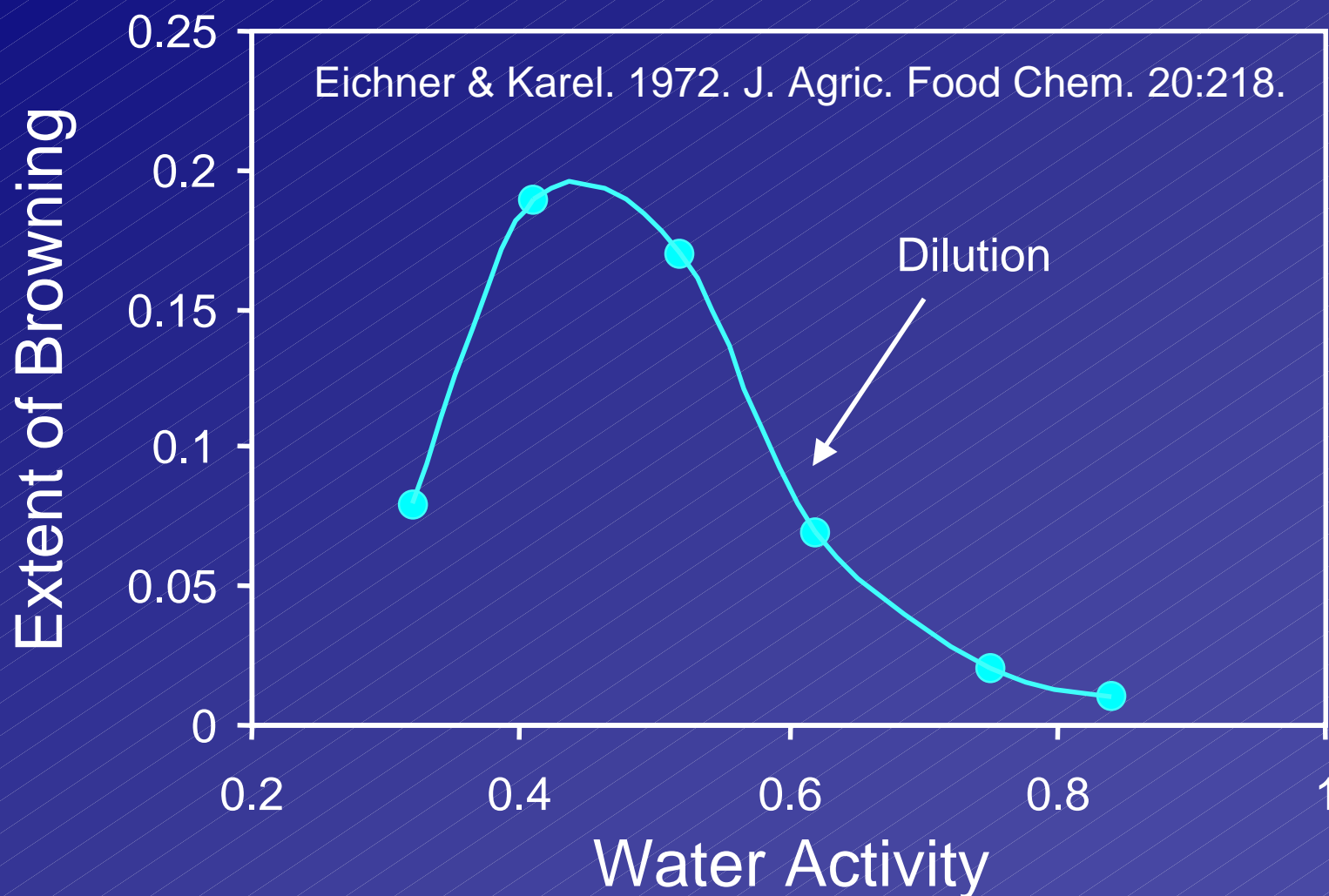
# BROWN PIGMENT FORMATION IN GLUCOSE-GLYCINE MODEL SYSTEM AT 37°C



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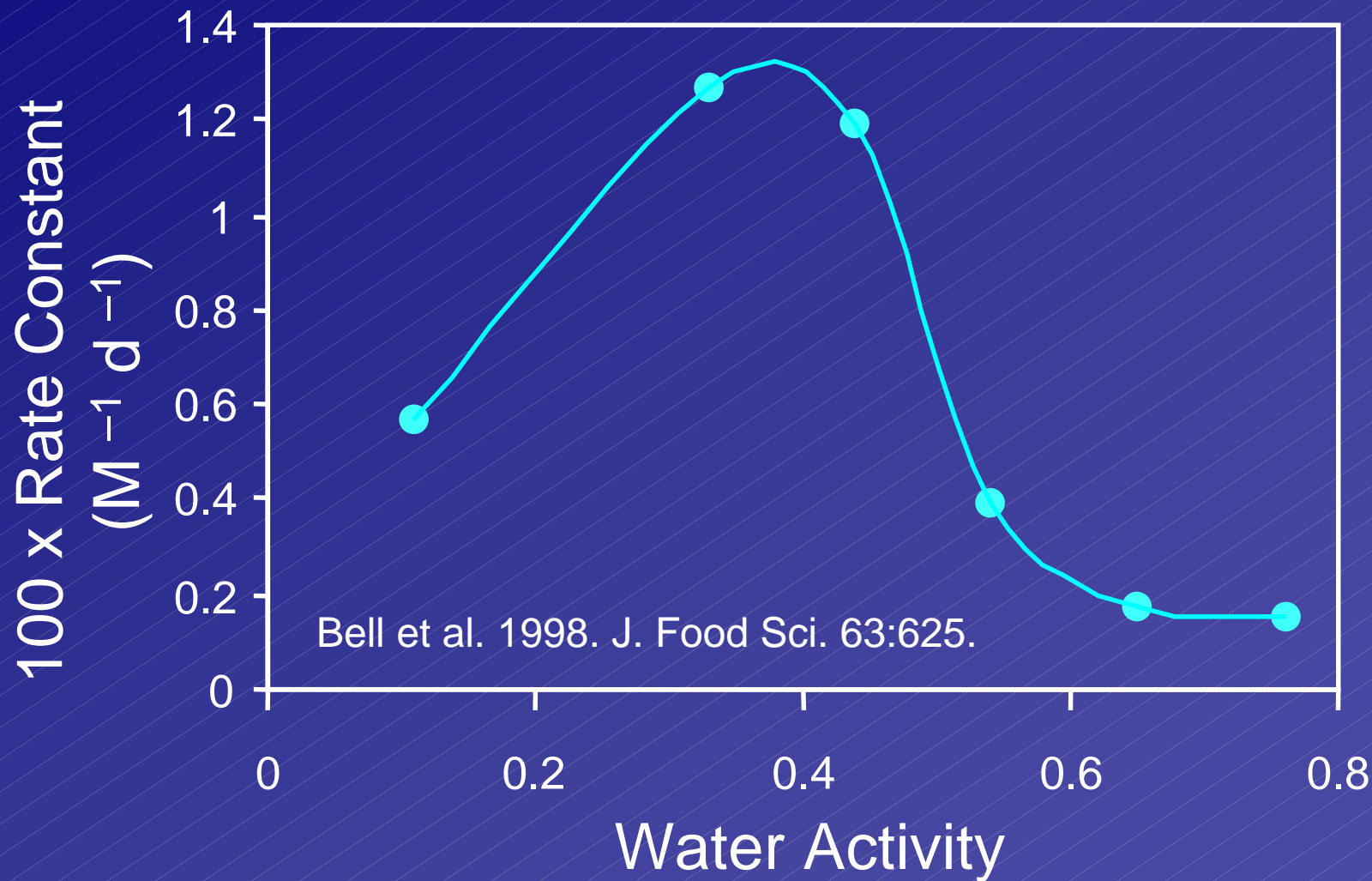
# BROWN PIGMENT FORMATION IN GLUCOSE-GLYCINE MODEL SYSTEM AT 37°C



# EXPERIMENTAL DESIGN TO ELIMINATE CONCENTRATION EFFECT

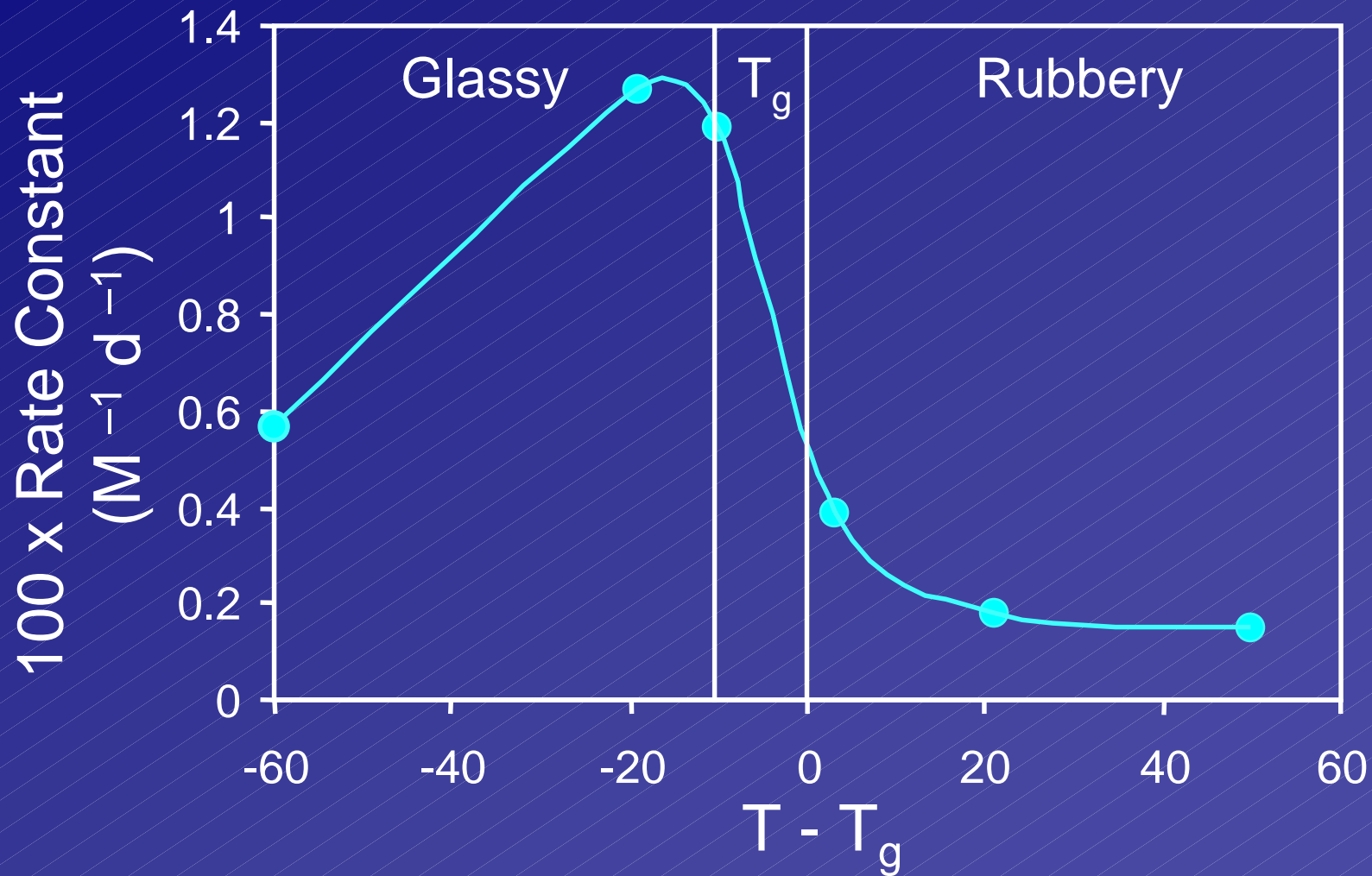
- Prepare each system with pre-determined reactant concentration so that after moisture sorption, all have the same reactant concentration in the aqueous phase (Bell et al. 1998. J. Food Sci. 63:625).
- For example
  - 10% moisture = 10 mg added reactants/g
  - 20% moisture = 20 mg added reactants/g

# GLYCINE LOSS IN PVP-K15 AT 25°C AS A FUNCTION OF WATER ACTIVITY



# GLYCINE LOSS IN PVP-K15 AT 25°C AS A FUNCTION OF GLASS TRANSITION

Bell et al. 1998. J. Food Sci. 63:625.

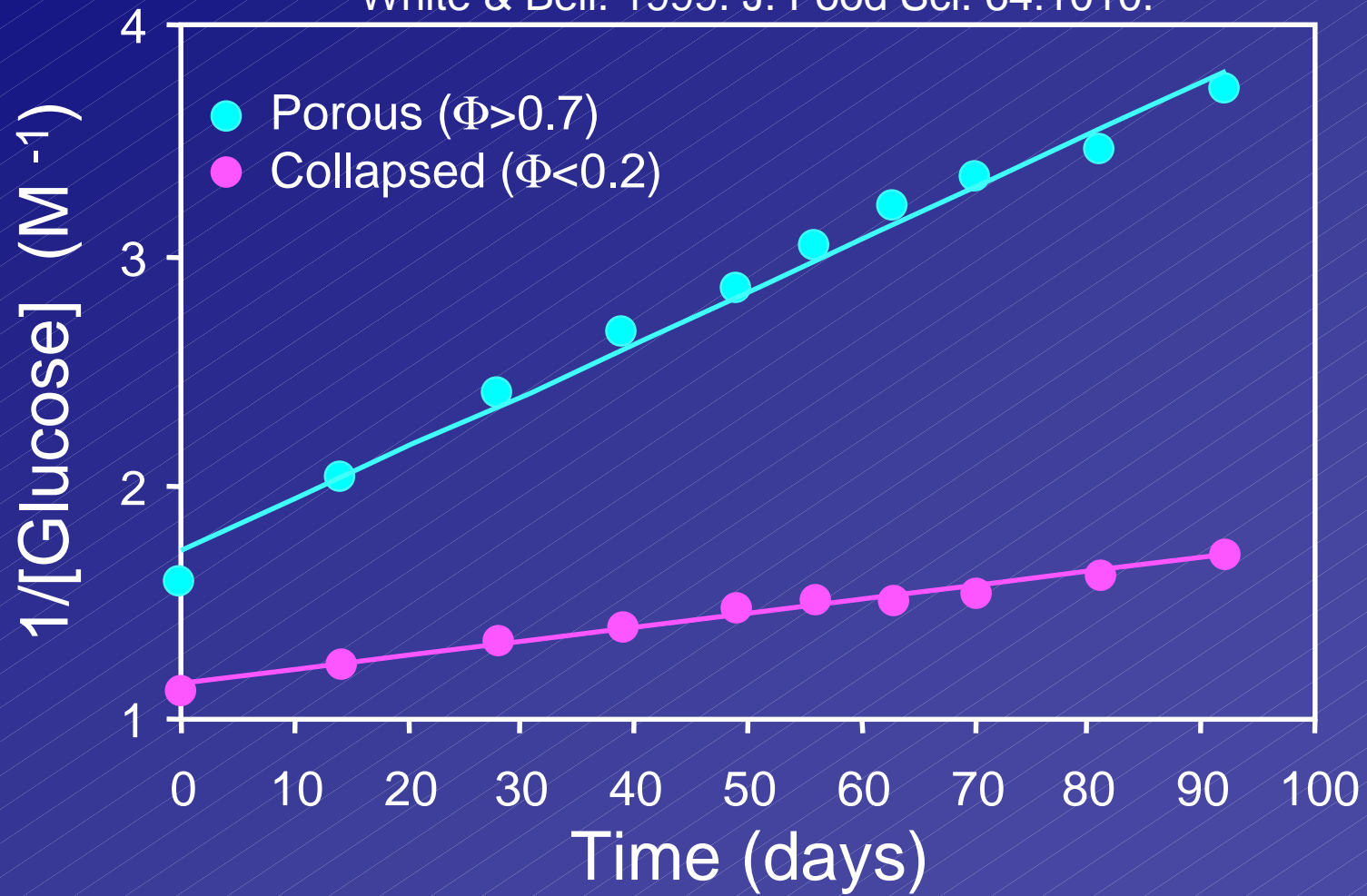


# GLYCINE LOSS VIA MAILLARD REACTION

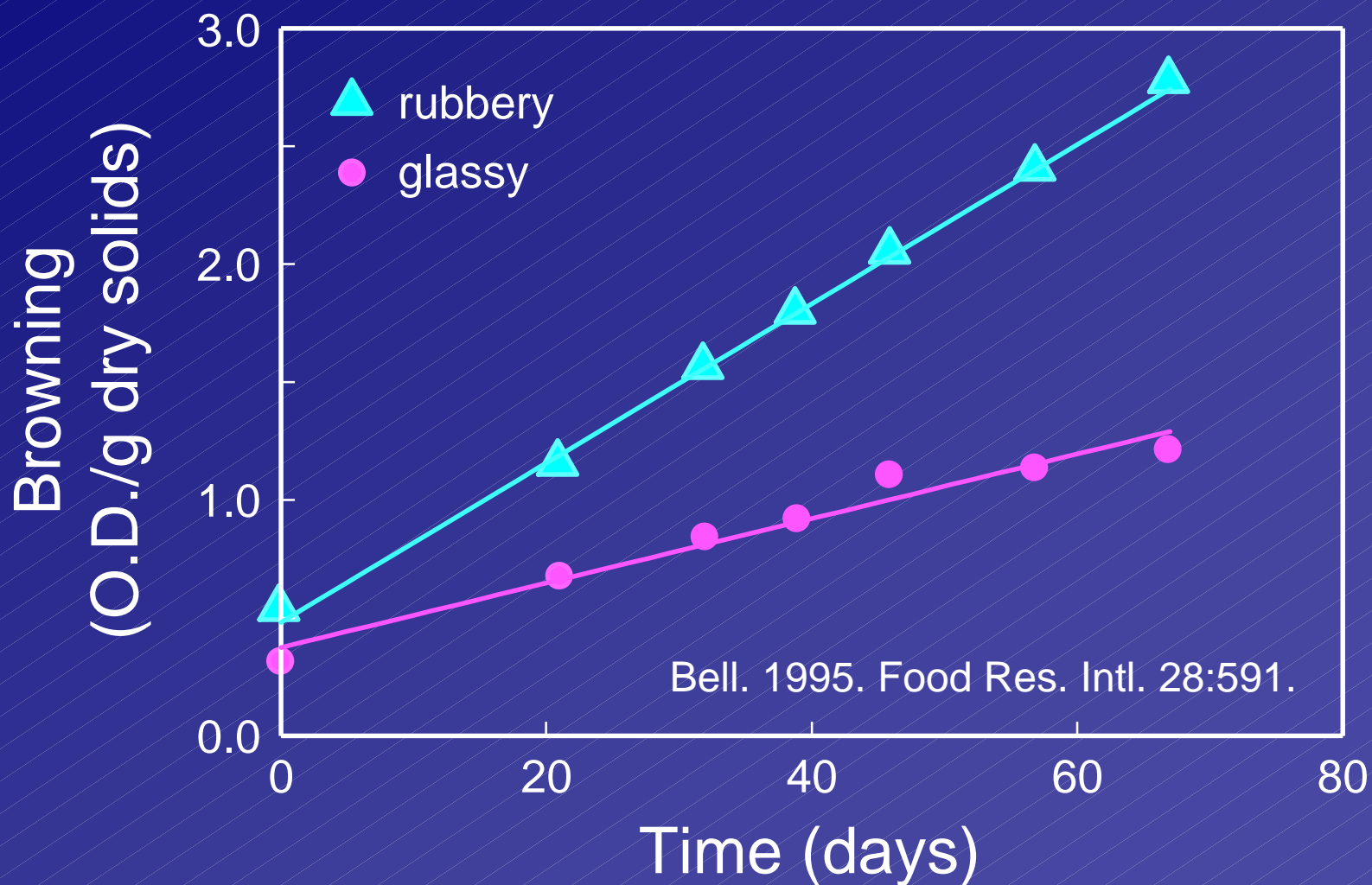
- Concentration effects eliminated
- Water activity 0.1 to 0.4
  - Mobility increasing
  - Reactivity increasing
- Water activity 0.4 to 0.7
  - Pass through glass transition
  - Structural collapse
  - Reactivity decreasing

# GLUCOSE LOSS IN PVP AT $a_w$ 0.33 AND 25°C AS AFFECTED BY POROSITY AND COLLAPSE

White & Bell. 1999. J. Food Sci. 64:1010.

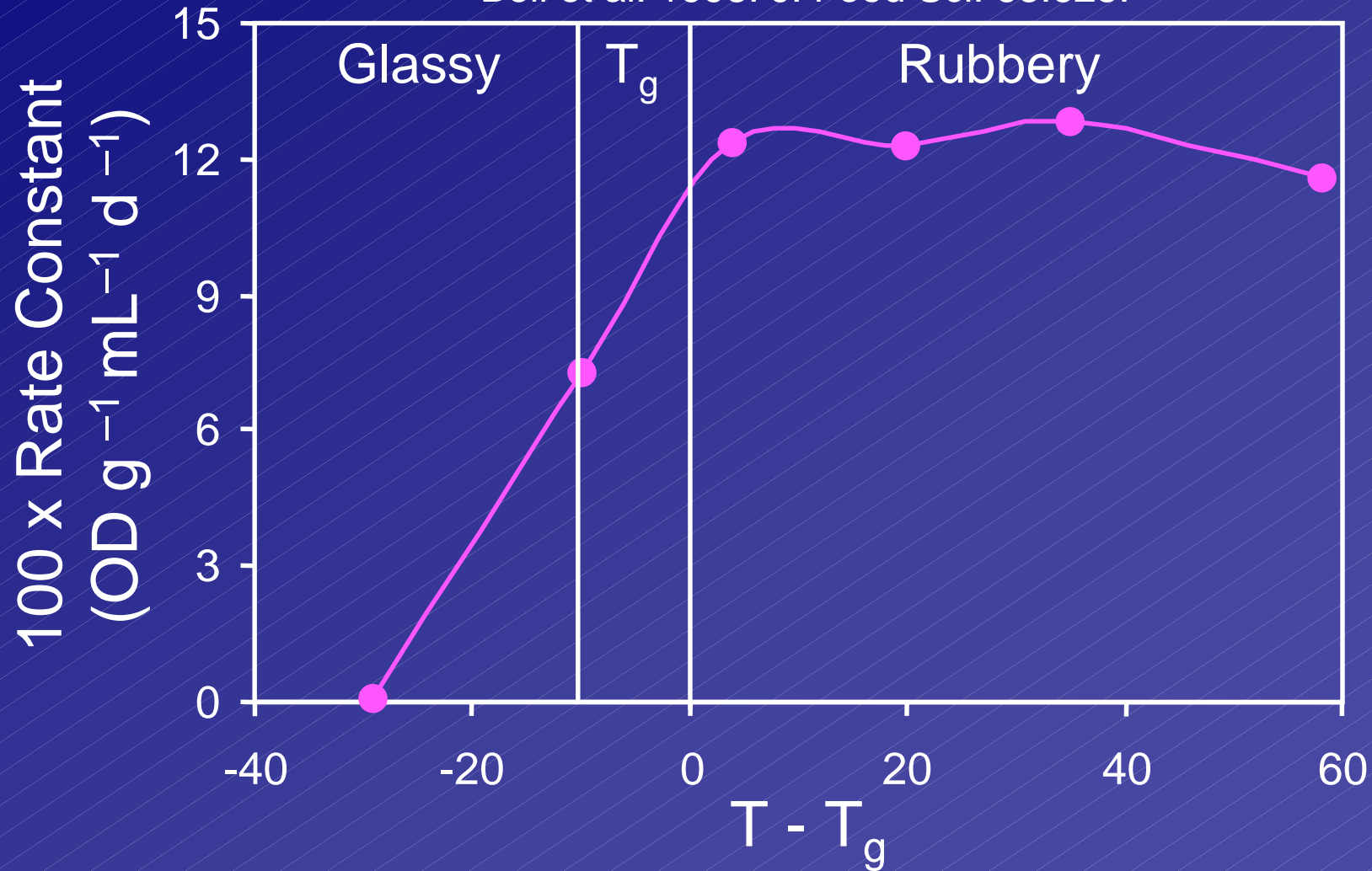


# EFFECT OF GLASS TRANSITION ON BROWNING IN PVP AT pH 7, $a_w$ 0.54, and 25°C



# BROWNING RATES IN PVP AT 25°C AS AFFECTED BY THE GLASS TRANSITION

Bell et al. 1998. J. Food Sci. 63:625.



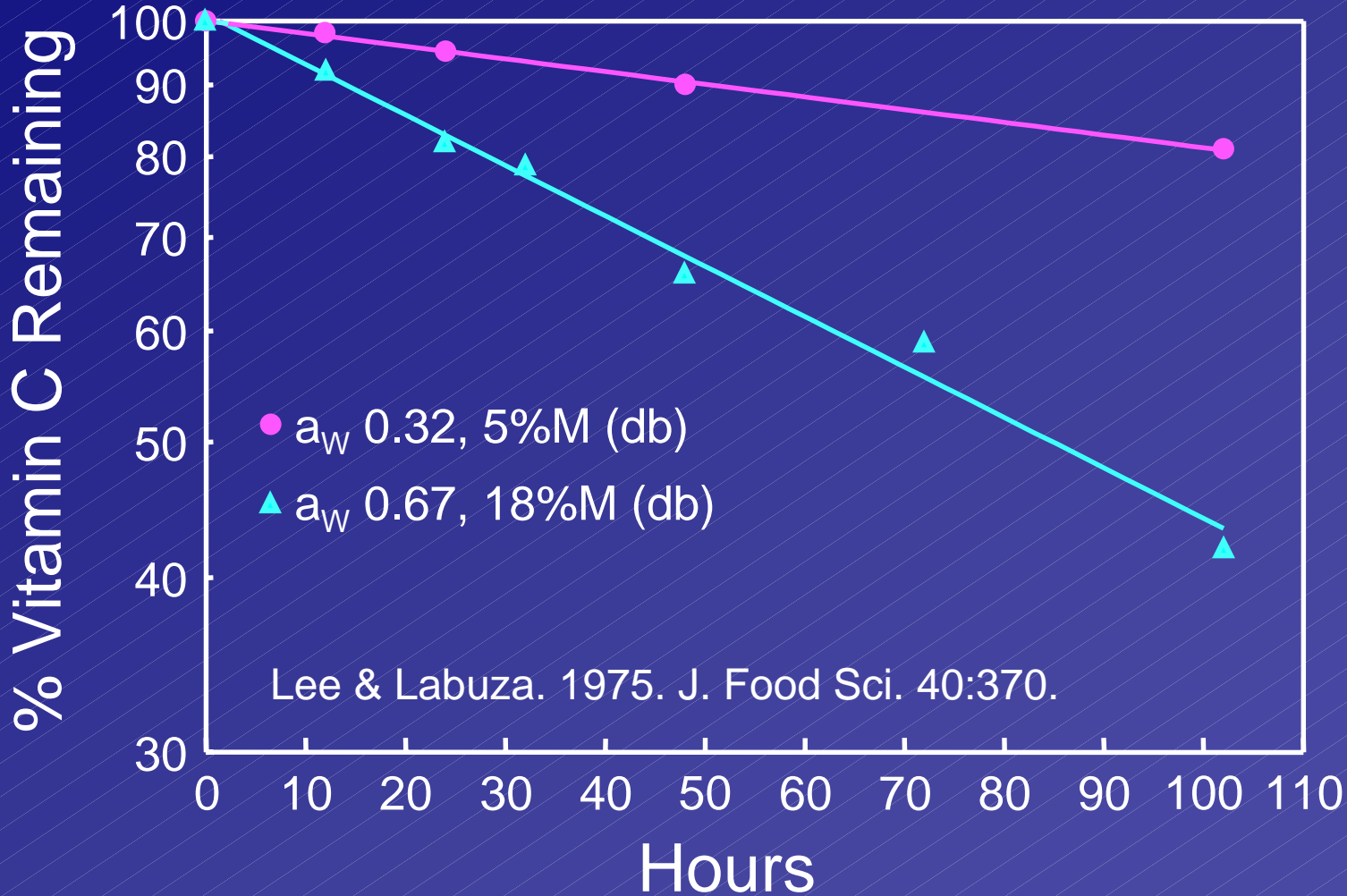
# WATER AND THE MAILLARD REACTION

- Moisture directly impacts this reaction primarily by dissolving and/or diluting the reactants
- Moisture indirectly impacts this reaction by its effect on the glass transition.
- Various researchers have shown the glass transition affects the Maillard reaction
  - Karmas et al. 1992. J. Agric. Food Chem. 40:873.
  - Buera & Karel. 1995. Food Chem. 52:167.
  - Lievonen et al. 1998. J. Agric. Food Chem. 46:2778.

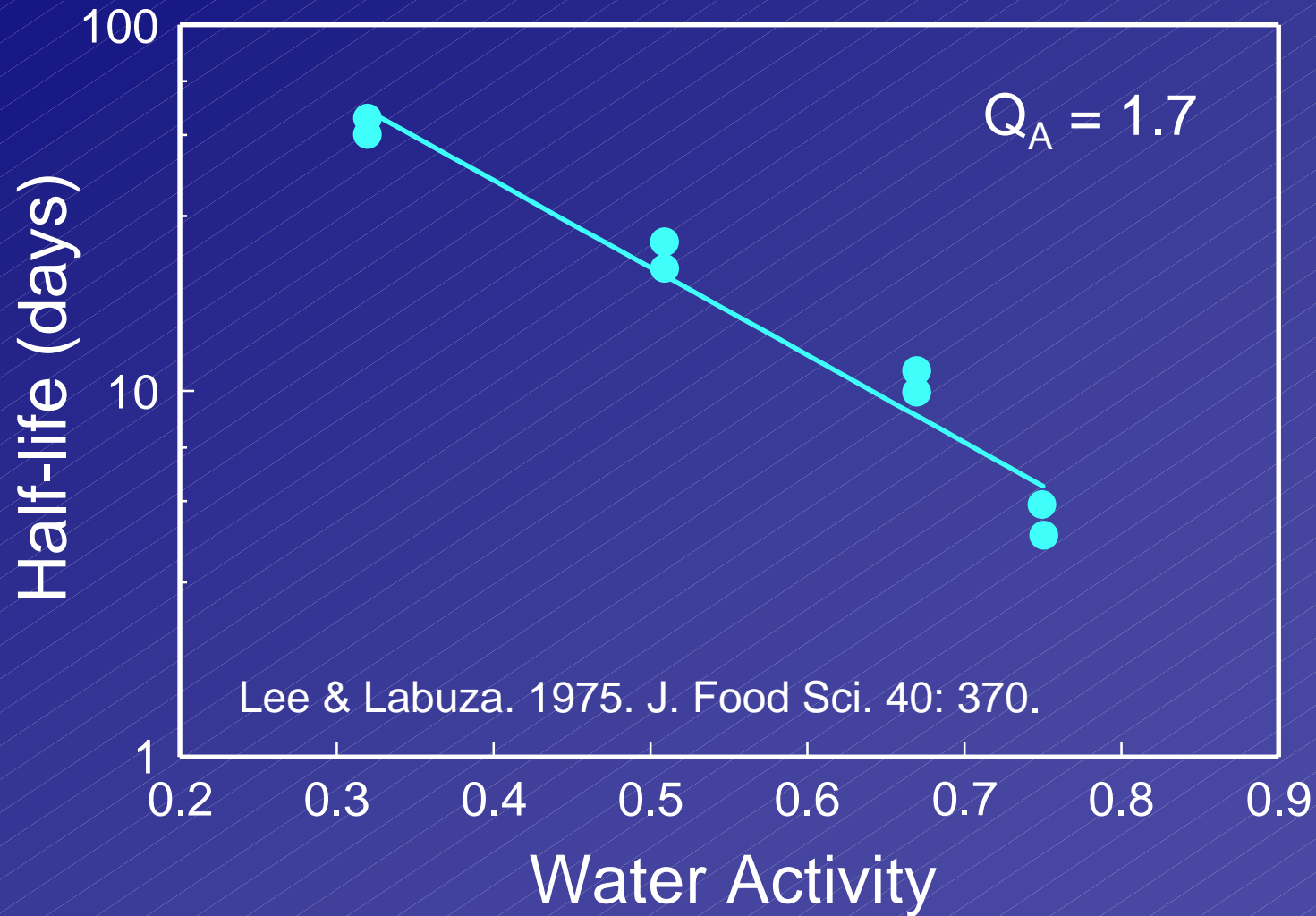
# VITAMIN STABILITY

- Ascorbic acid (vitamin C)
- Riboflavin
- Thiamin

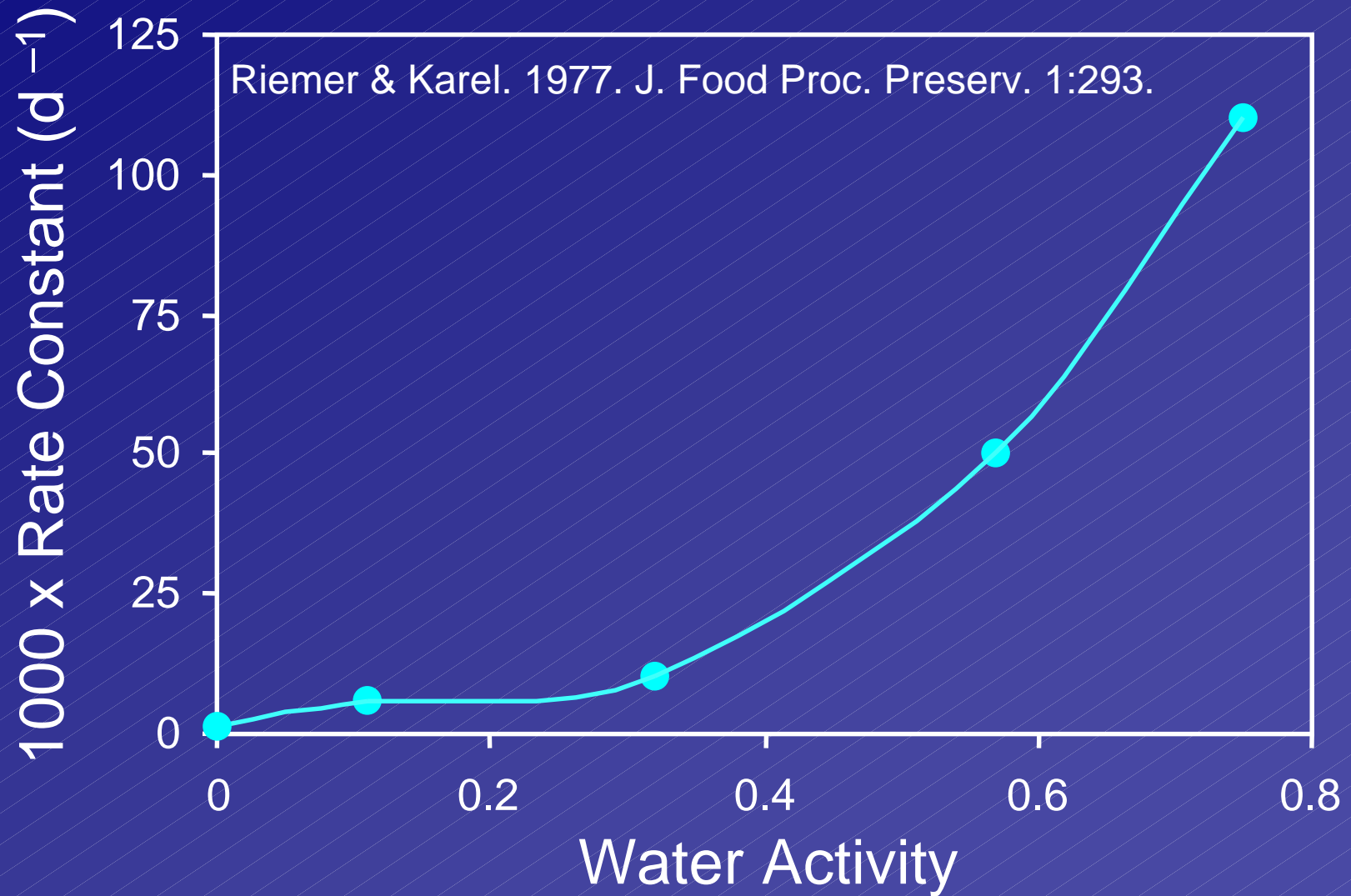
# VITAMIN C DEGRADATION AT 35°C AS INFLUENCED BY MOISTURE



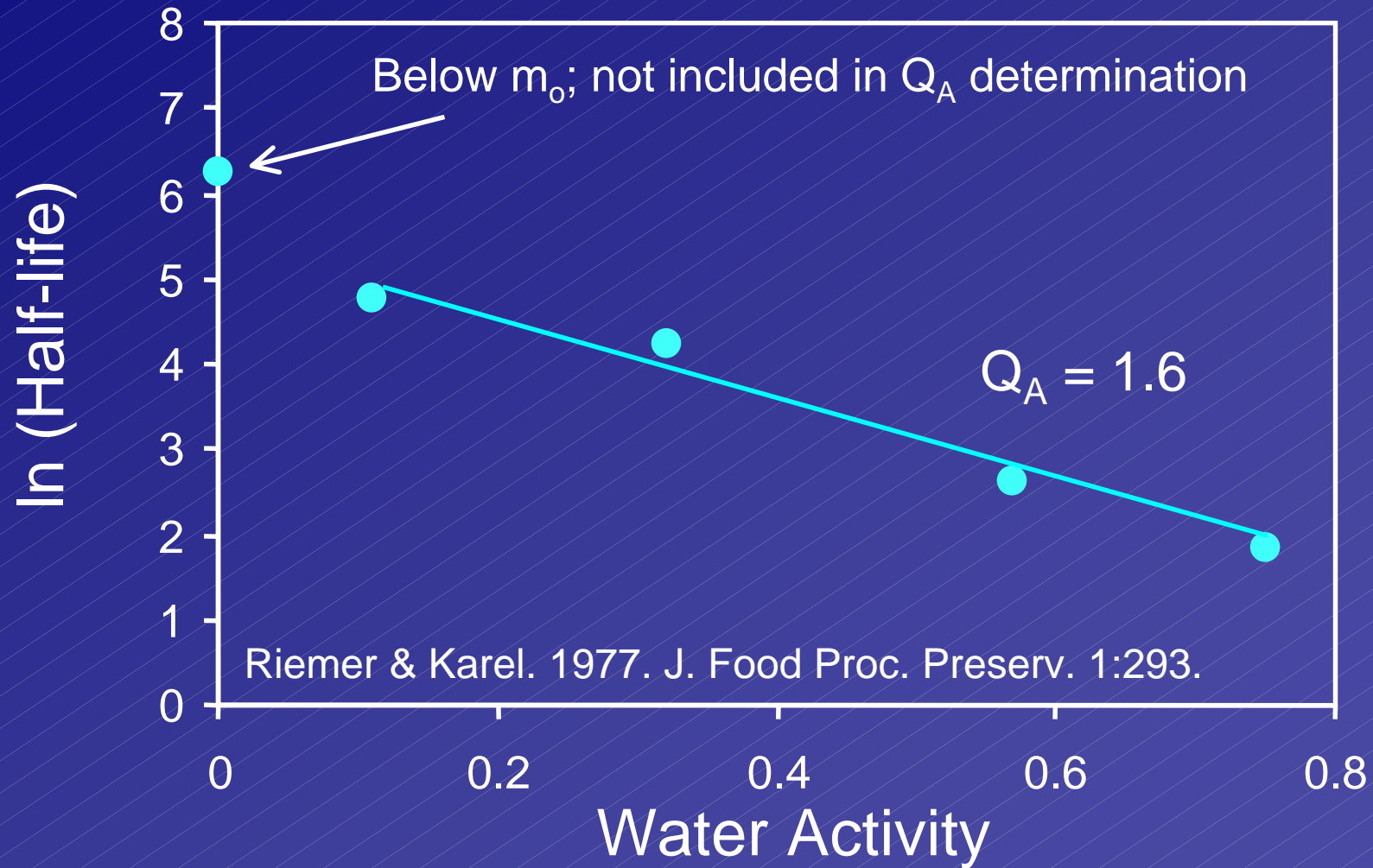
# $Q_A$ PLOT FOR VITAMIN C DEGRADATION AT 23°C



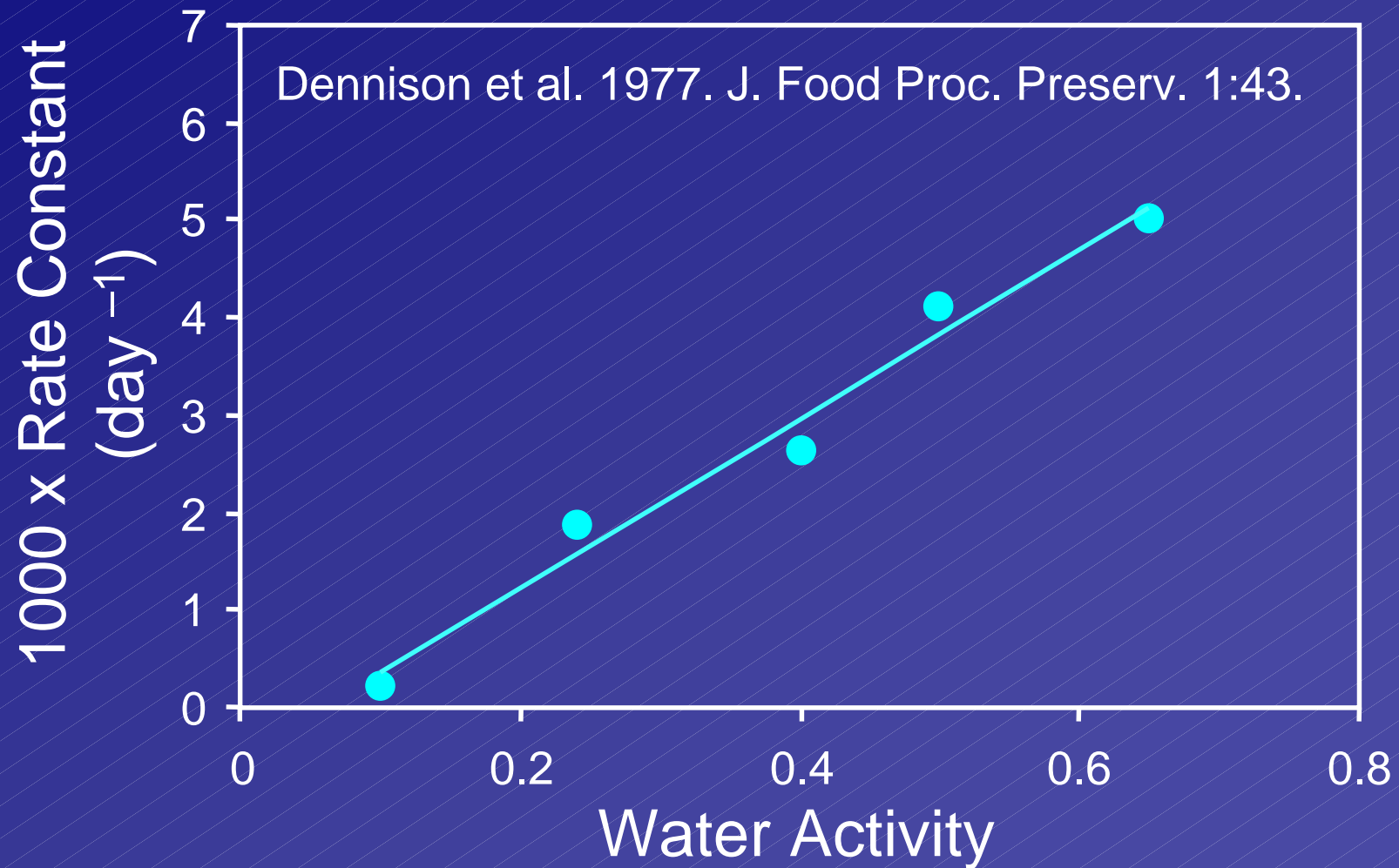
# VITAMIN C DEGRADATION IN DRIED TOMATO JUICE AT 20°C



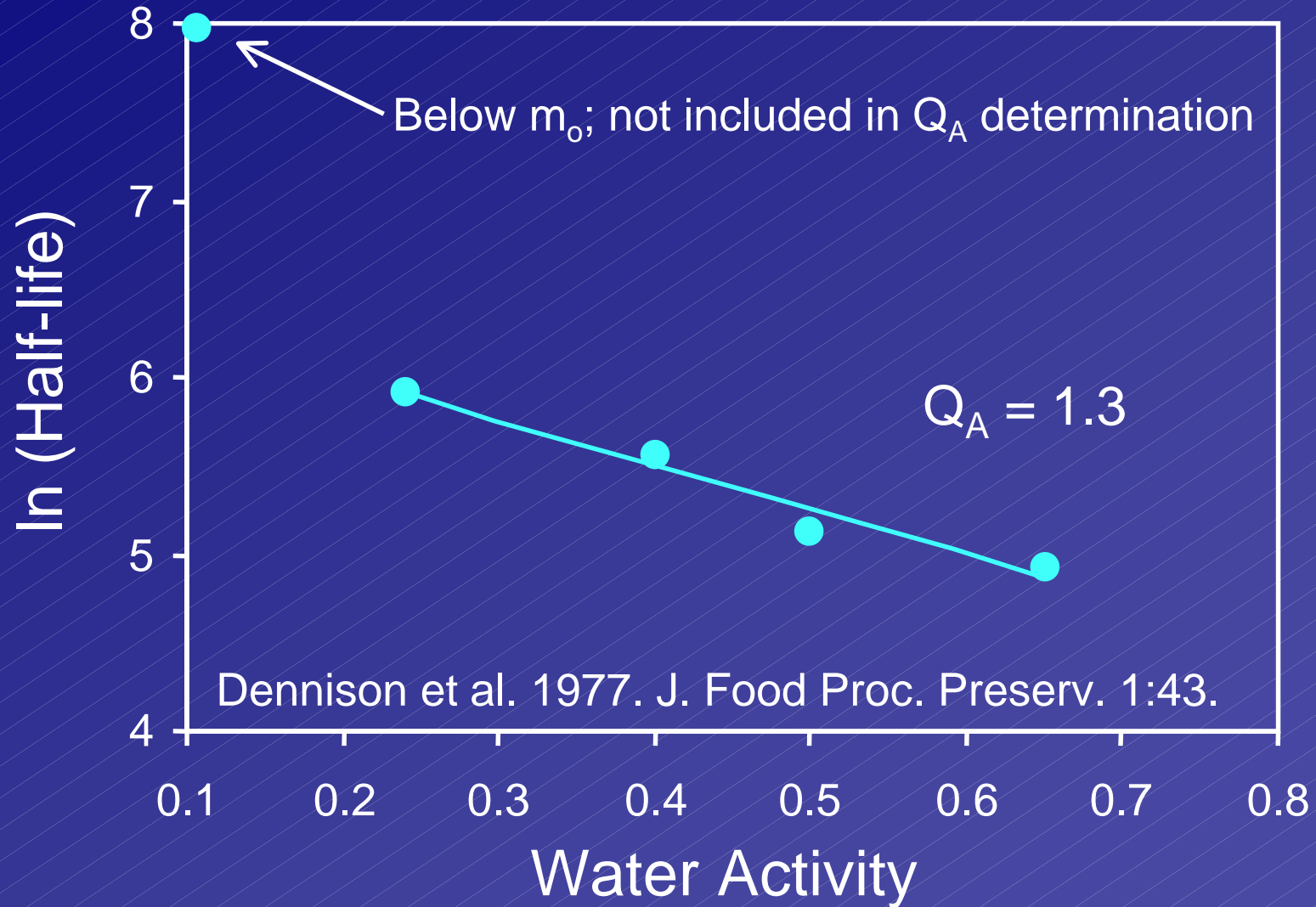
# $Q_A$ PLOT FOR VITAMIN C DEGRADATION AT 20°C IN DRIED TOMATO JUICE



# RIBOFLAVIN DEGRADATION AT 37°C IN MODEL SYSTEMS AS AFFECTED BY WATER ACTIVITY

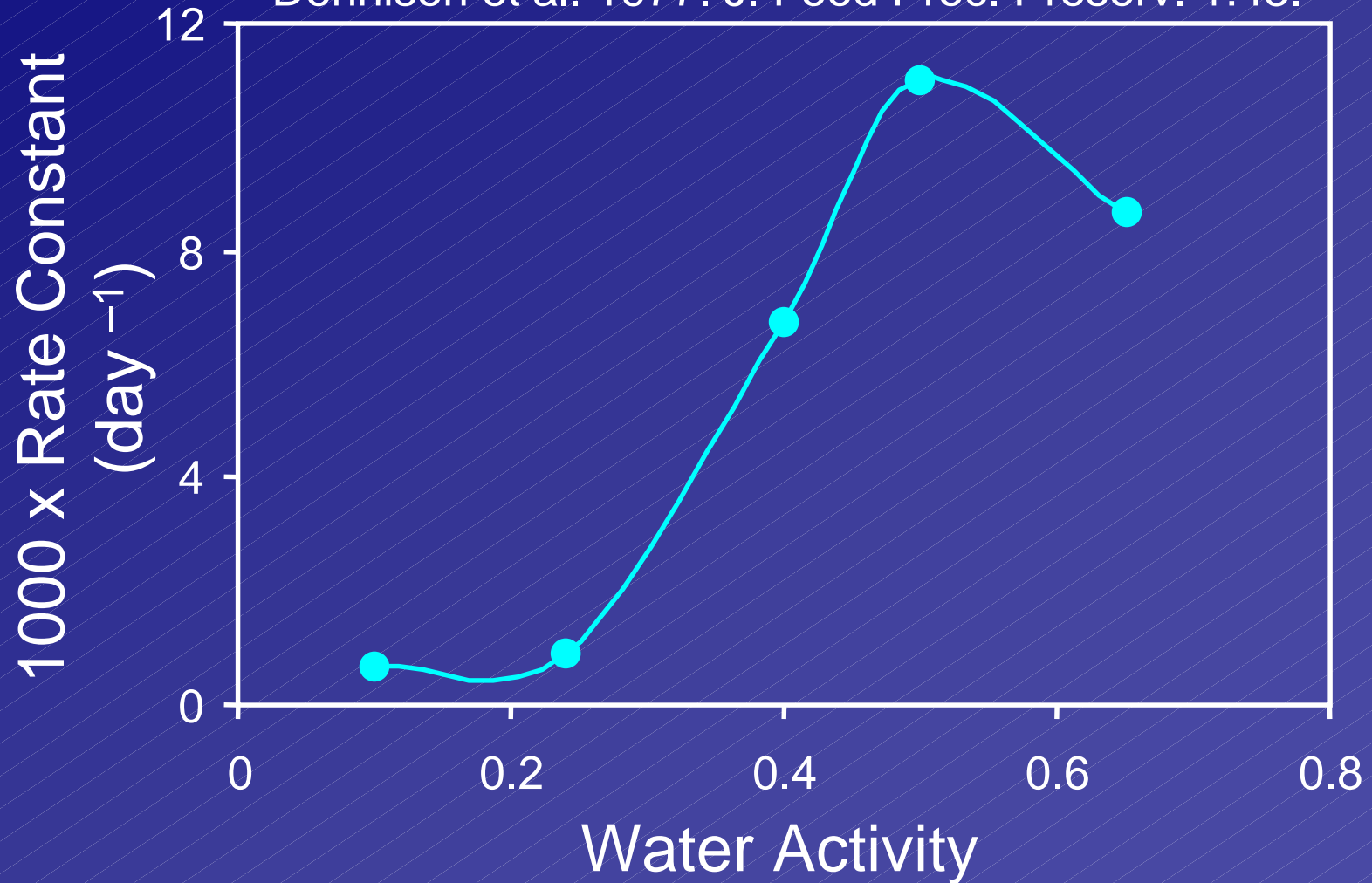


# $Q_A$ PLOT FOR RIBOFLAVIN DEGRADATION AT 37°C

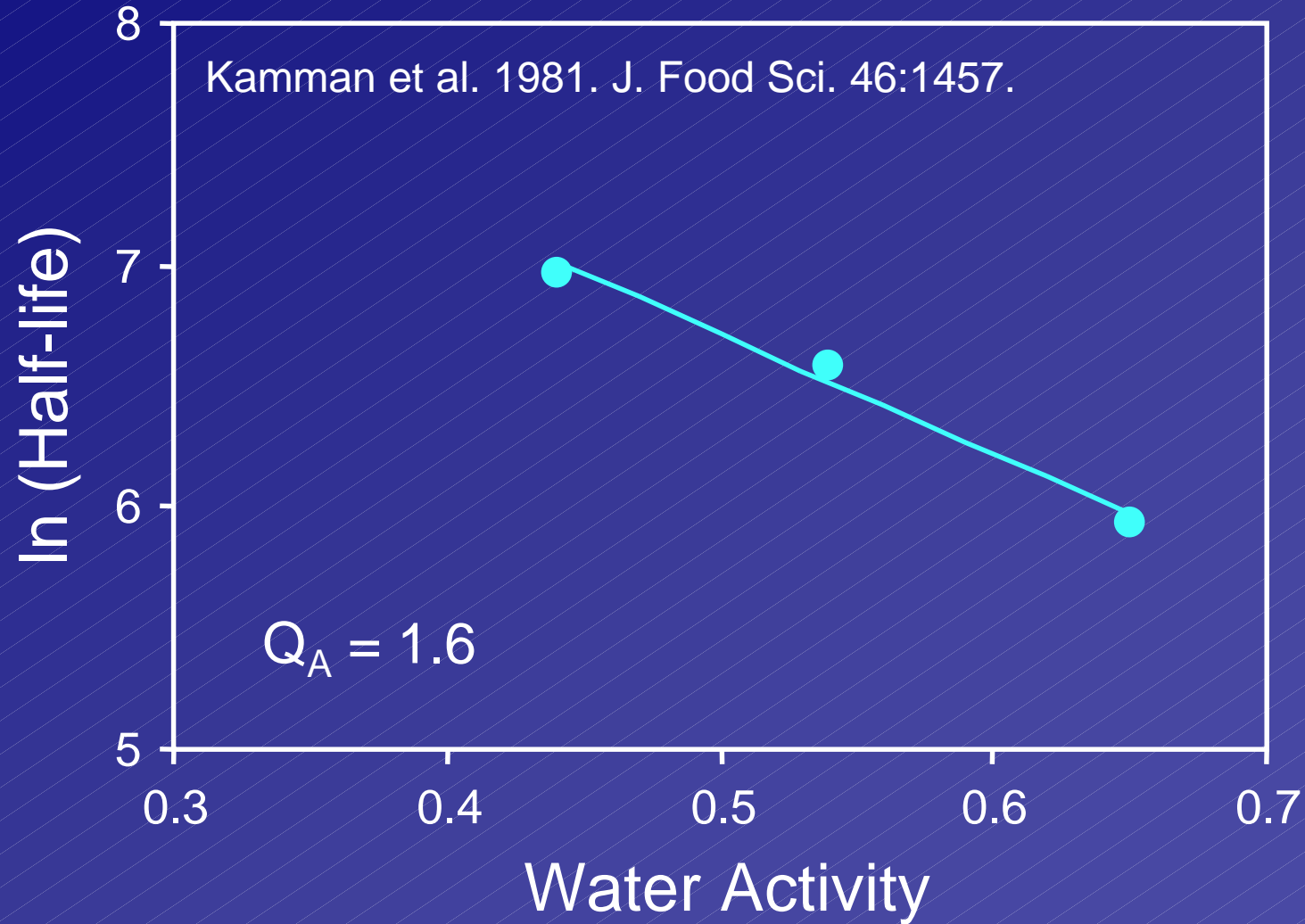


# THIAMIN DEGRADATION AT 45°C IN MODEL SYSTEMS AS AFFECTED BY WATER ACTIVITY

Dennison et al. 1977. J. Food Proc. Preserv. 1:43.



# $Q_A$ PLOT FOR THIAMIN DEGRADATION IN PASTA AT 35°C

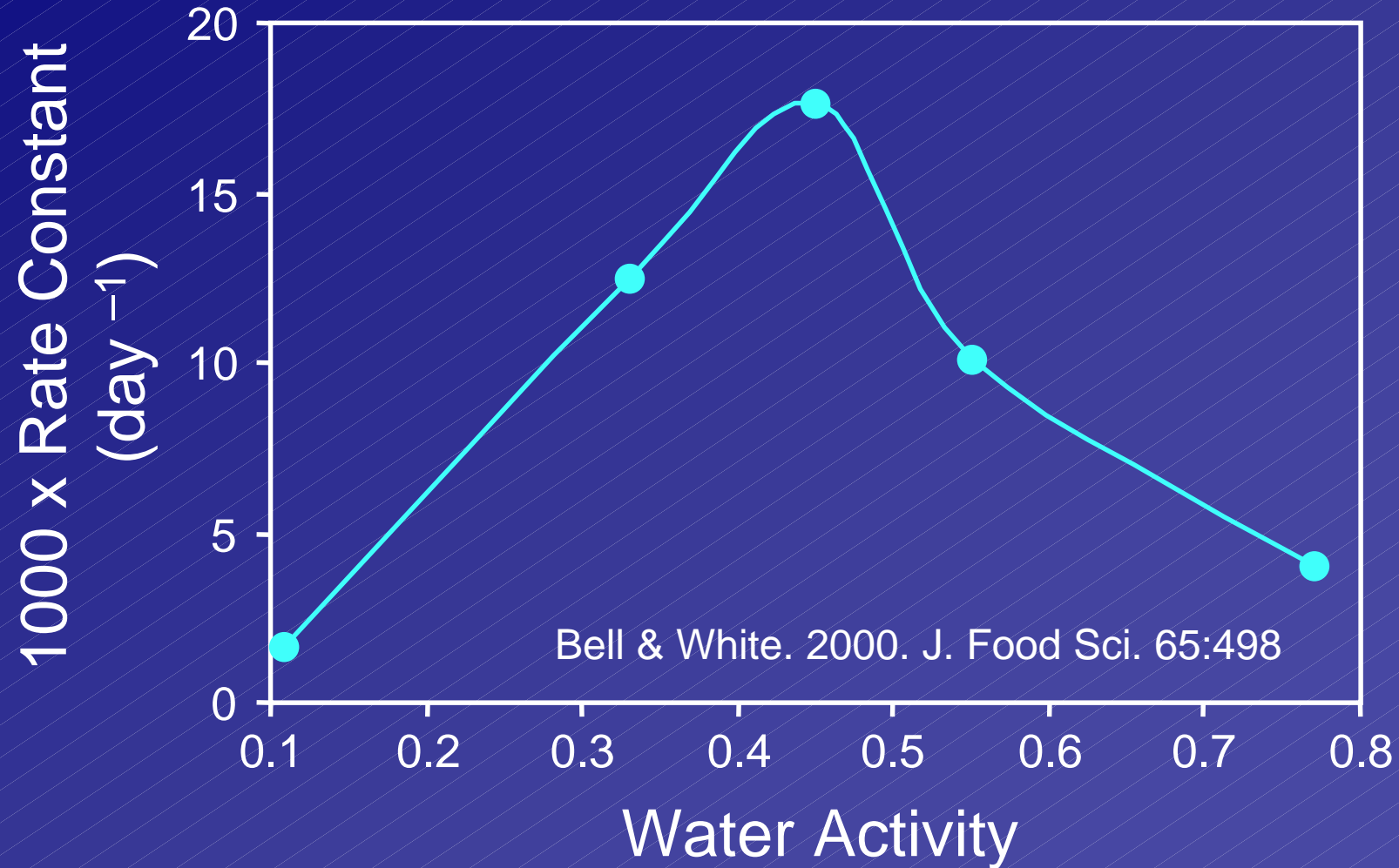


# EFFECT OF MOISTURE ON THE ACTIVATION ENERGY OF THIAMIN DEGRADATION

$a_w$	$E_A$ (kcal/mole)
0.44	30.8
0.54	29.8
0.65	26.6

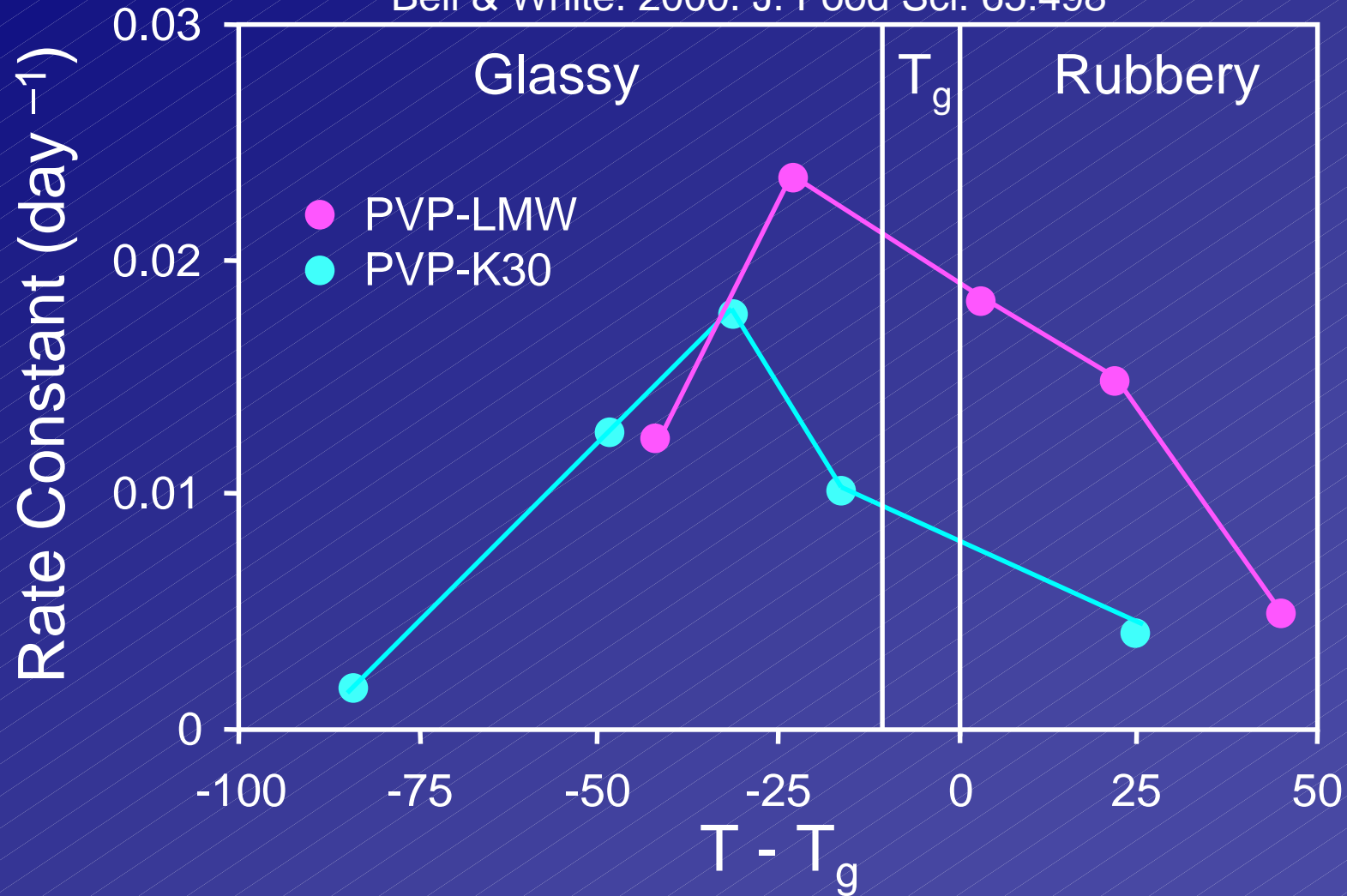
Kamman et al. 1981. J. Food Sci. 46:1457.

# THIAMIN DEGRADATION IN PVP AT 20°C AS AFFECTED BY WATER ACTIVITY



# THIAMIN DEGRADATION IN PVP AT 20°C AS AFFECTED BY $T_g$

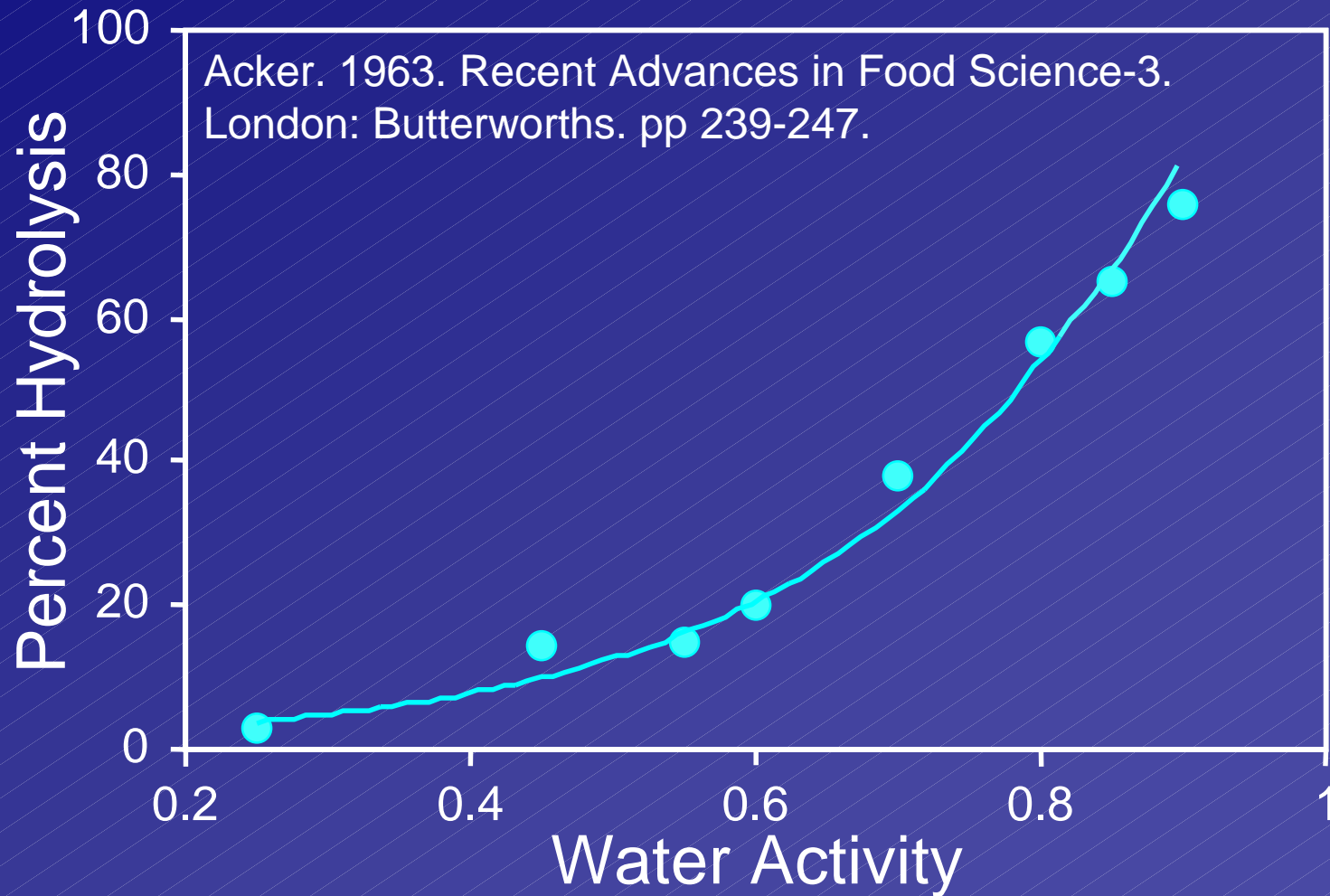
Bell & White. 2000. J. Food Sci. 65:498



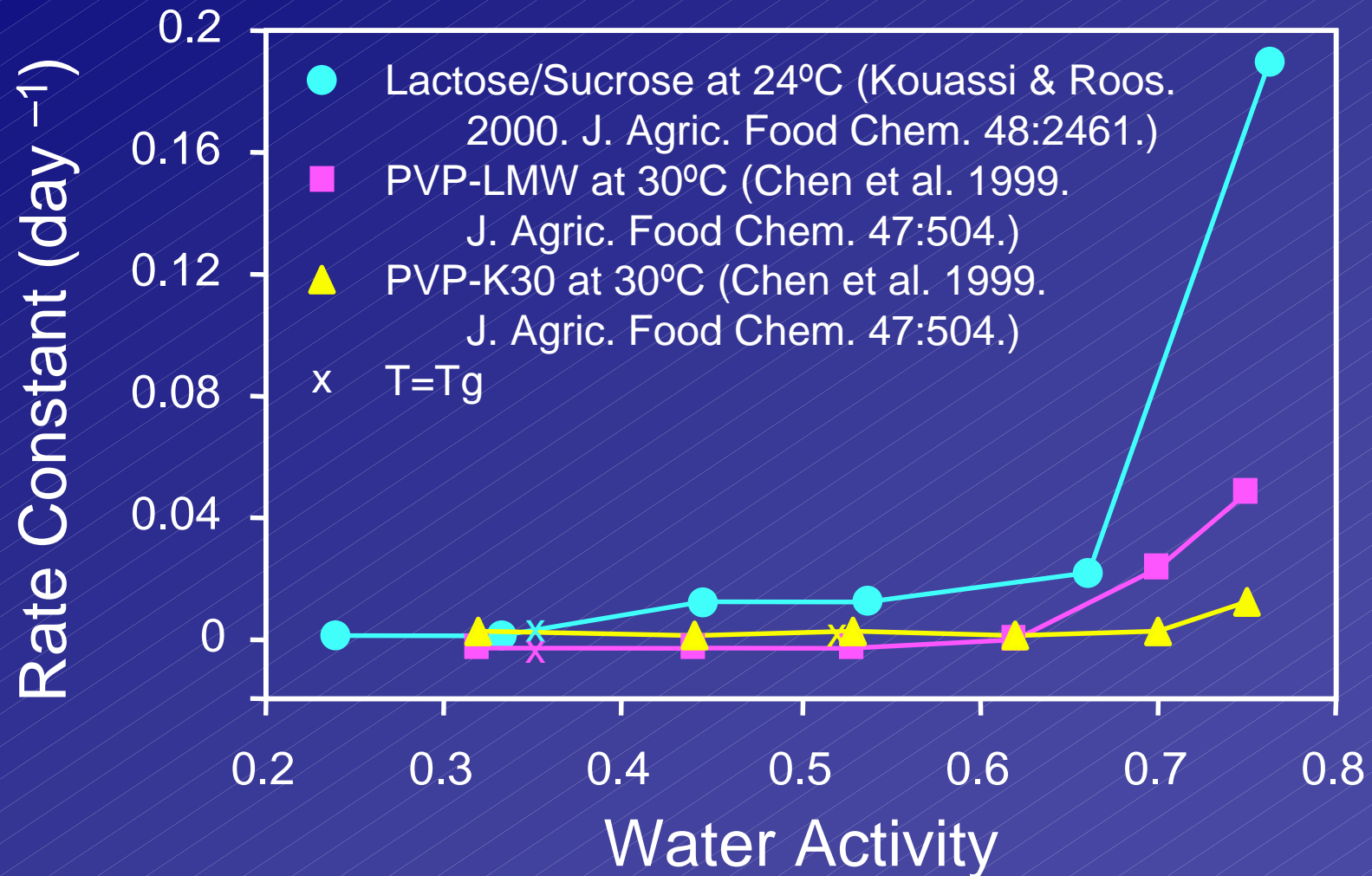
# ENZYMES AND WATER

- Enzyme activity
  - Water can:
    - Dissolve substrate
    - Increase substrate mobility
    - Be a reactant
- Enzyme stability
  - Moisture can influence “denaturation”
    - Hydrolysis
    - Deamidation
    - Oxidation

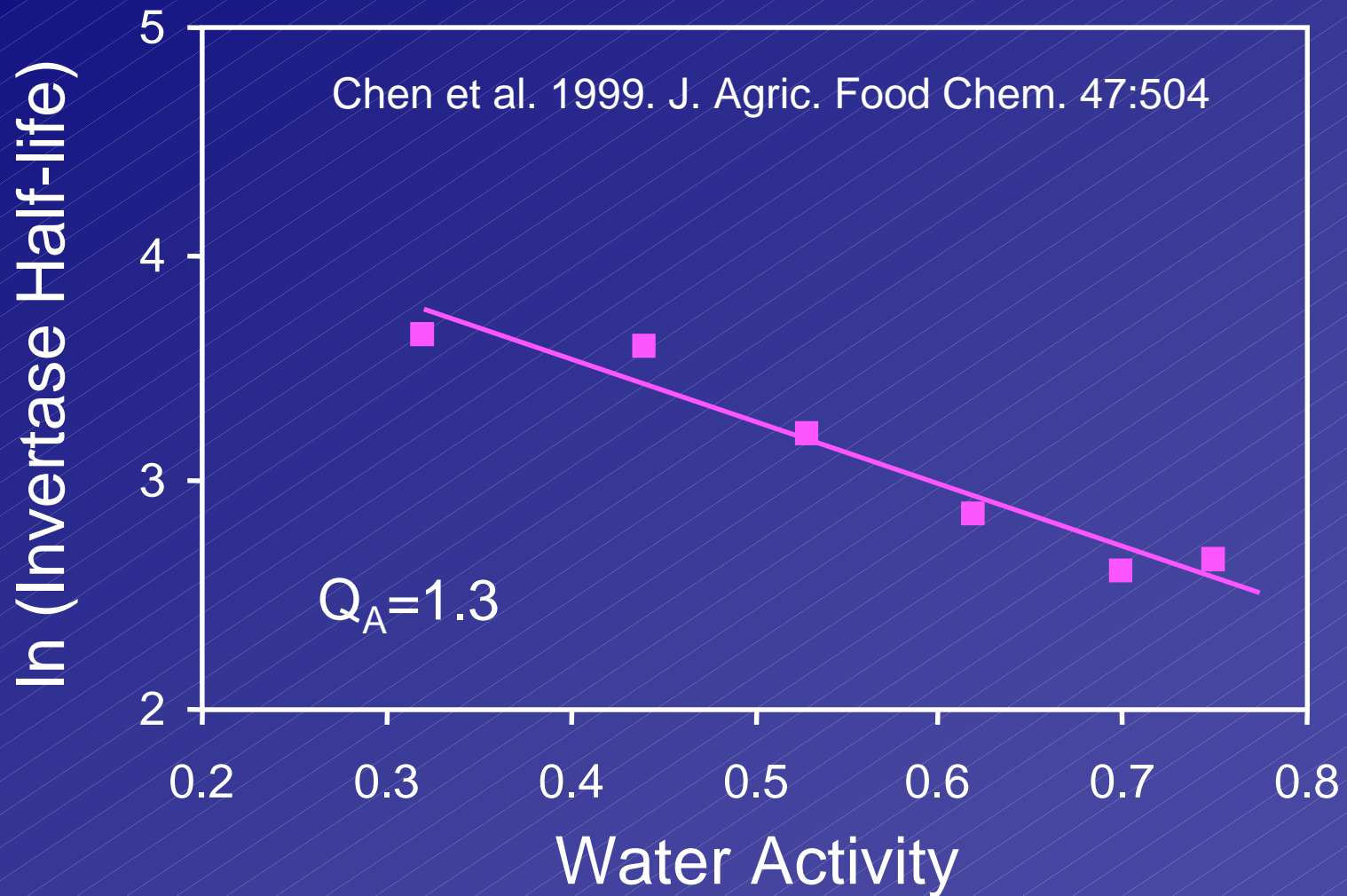
# EXTENT OF LECITHIN HYDROLYSIS BY PHOSPHOLIPASE AFTER 12 DAYS



# SUCROSE HYDROLYSIS BY INVERTASE AS A FUNCTION OF WATER ACTIVITY

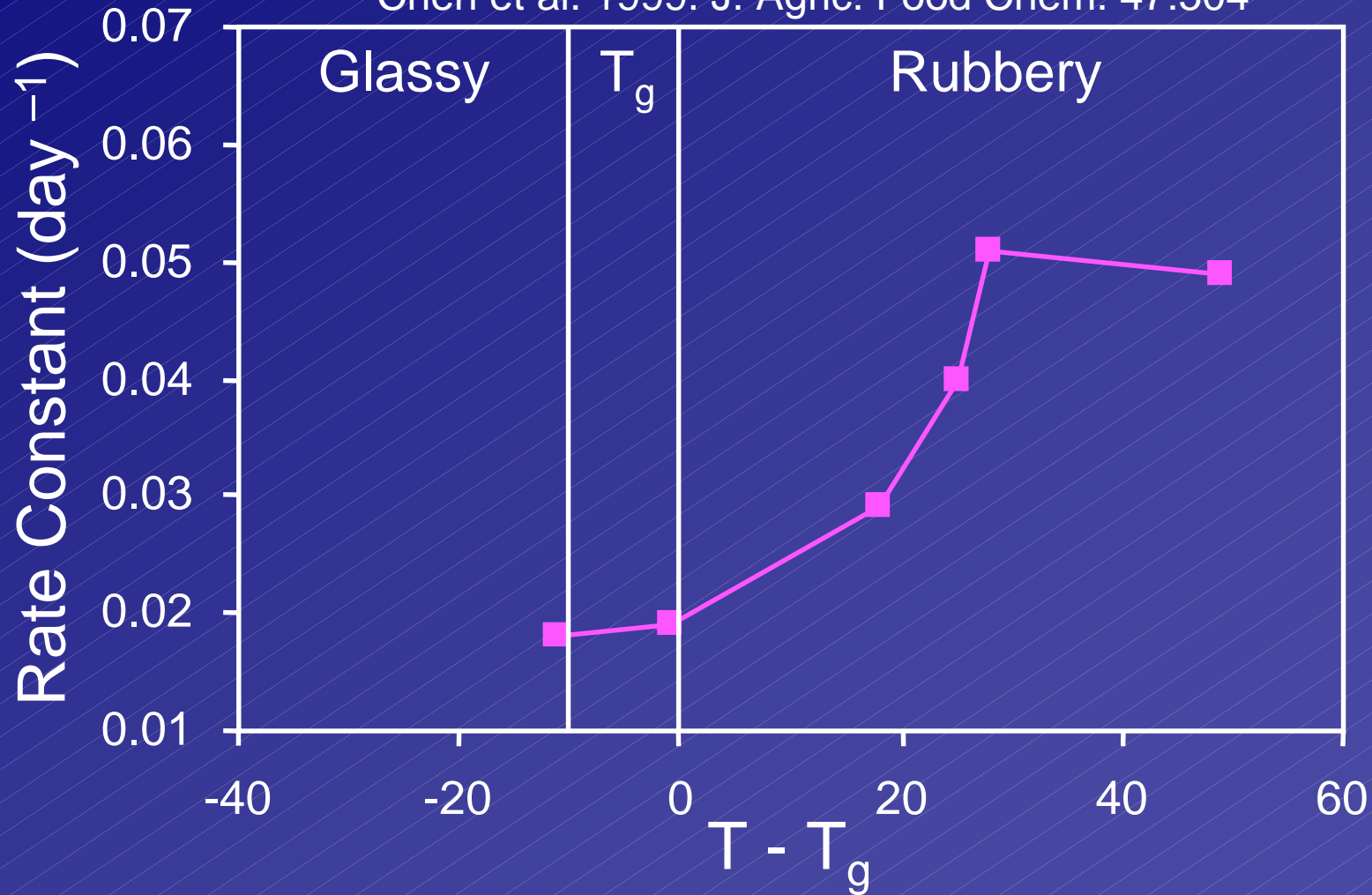


# $Q_A$ PLOT FOR INVERTASE STABILITY IN PVP AT 30°C

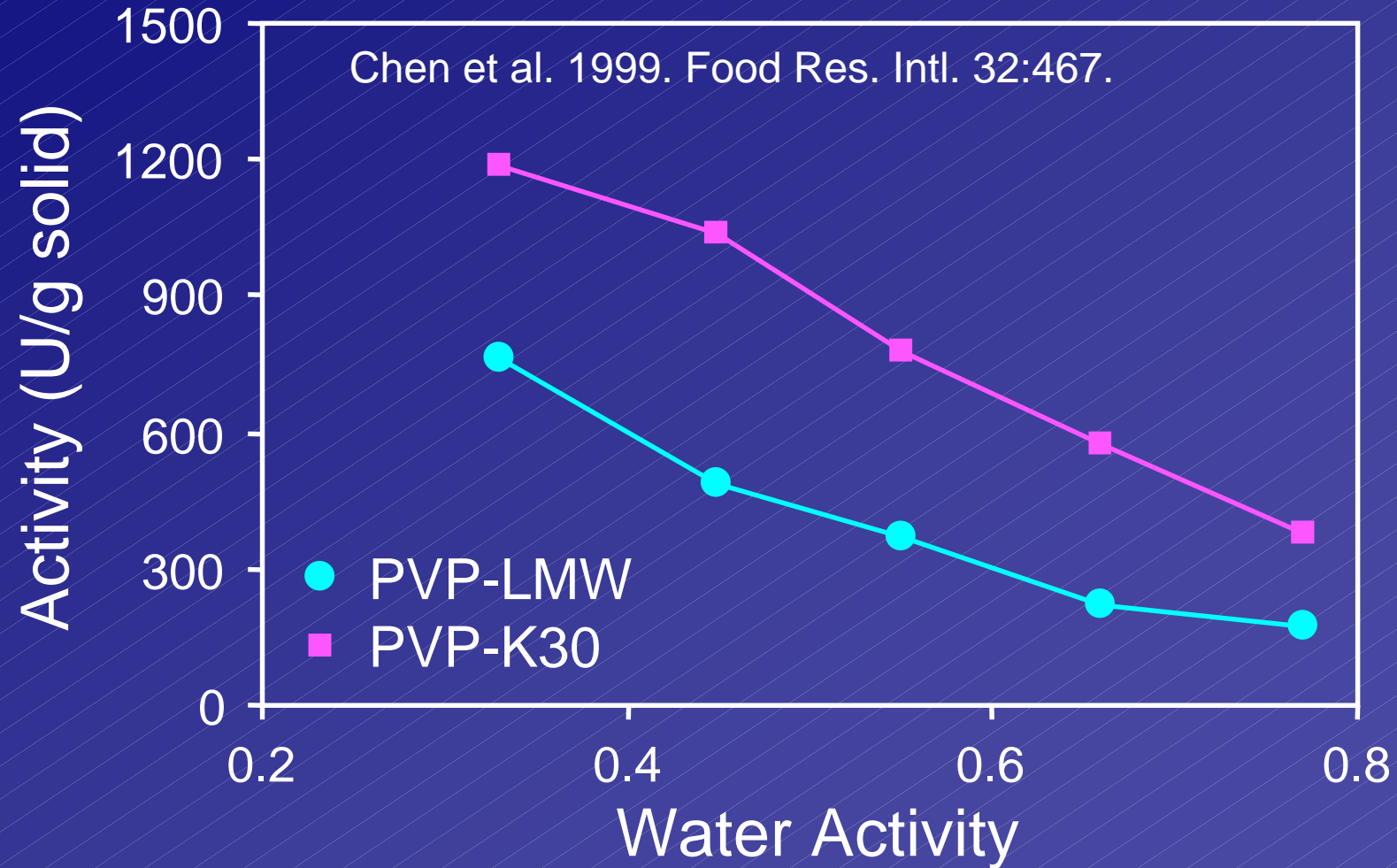


# INVERTASE STABILITY IN PVP AT 30°C AS AFFECTED BY THE GLASS TRANSITION

Chen et al. 1999. J. Agric. Food Chem. 47:504

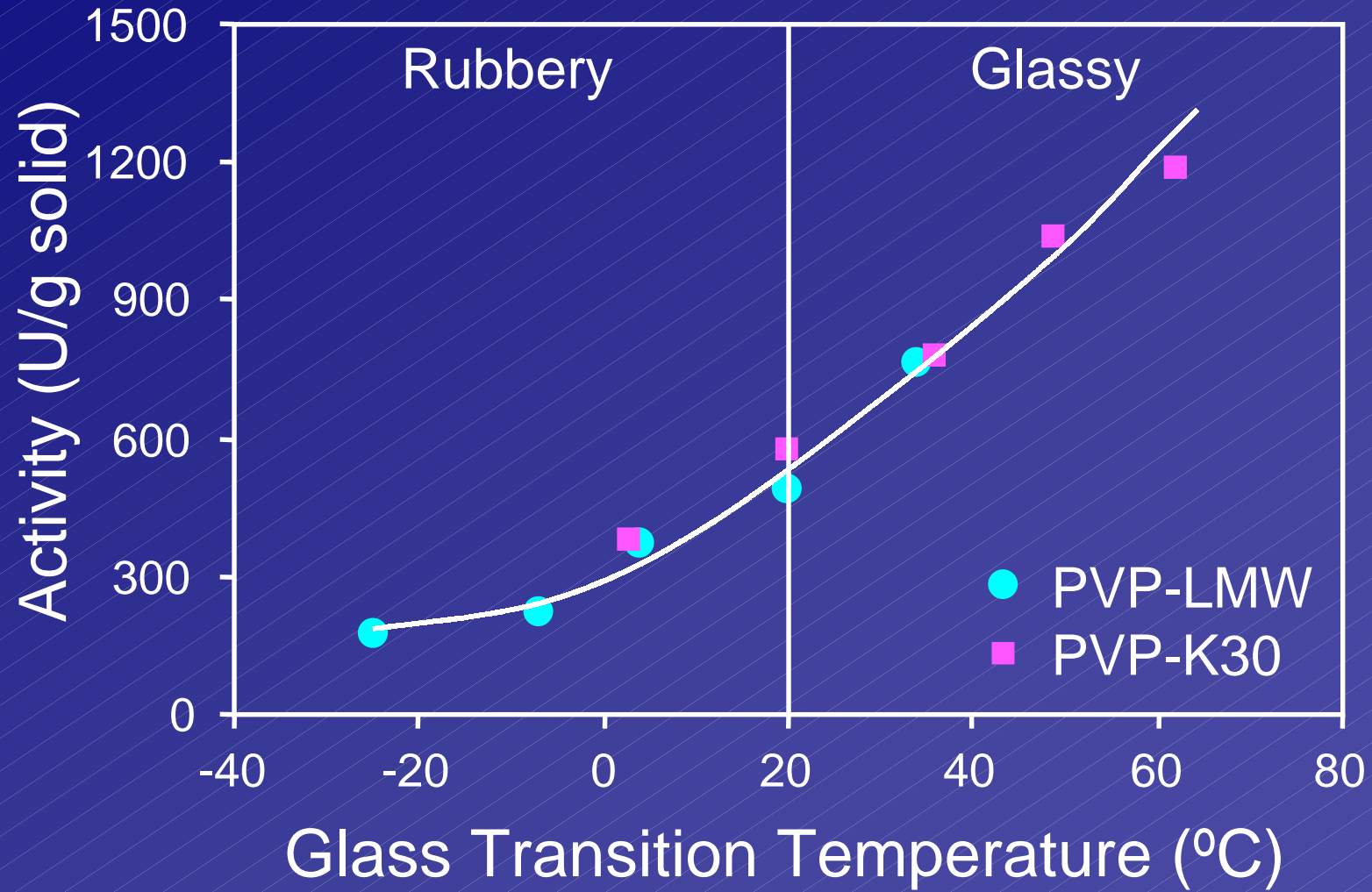


# RESIDUAL TYROSINASE ACTIVITY IN PVP AFTER 3 DAYS EQUILIBRATION AT 20°C

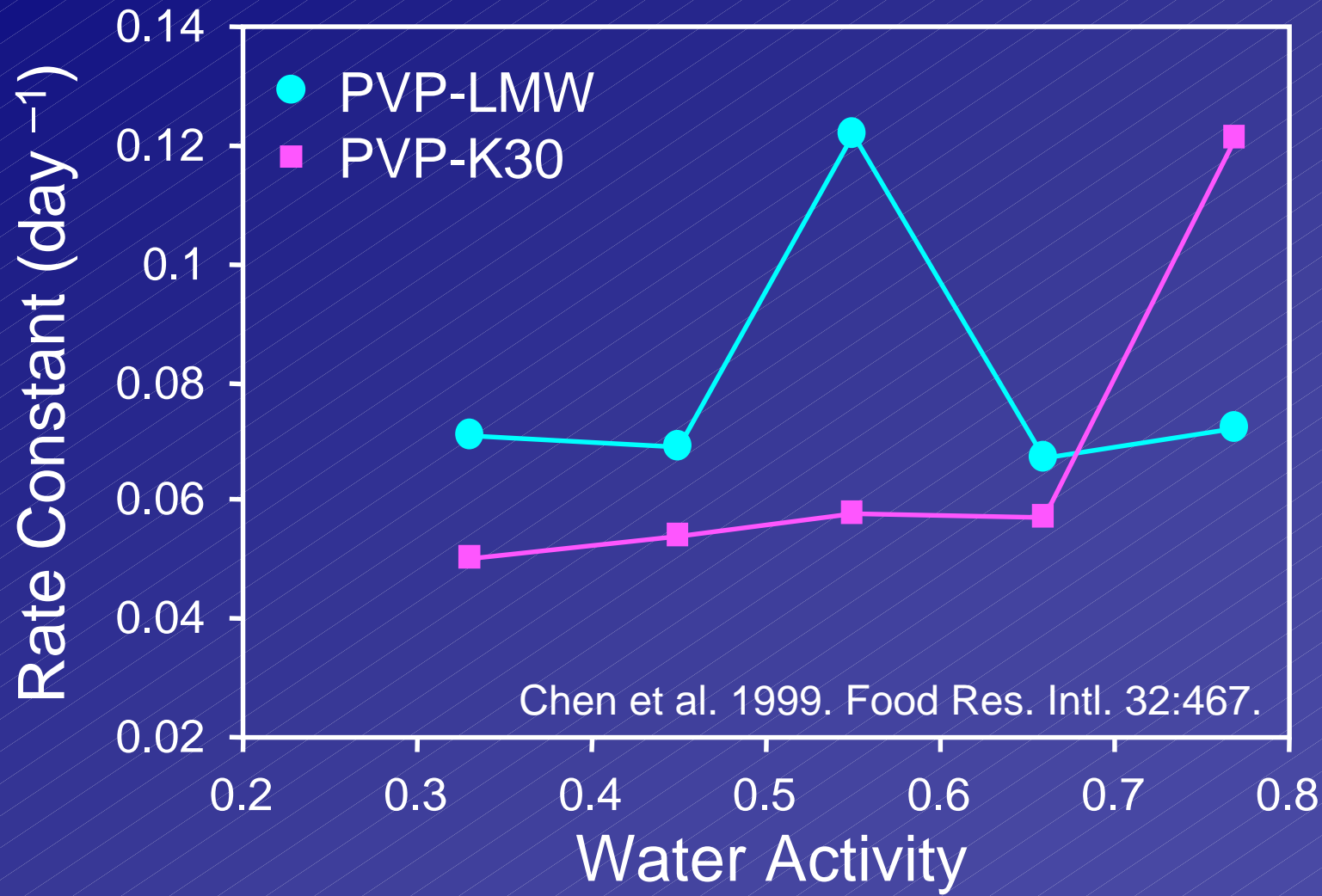


# RESIDUAL TYROSINASE ACTIVITY IN PVP AFTER 3 DAYS EQUILIBRATION AT 20°C

Chen et al. 1999. Food Res. Intl. 32:467.

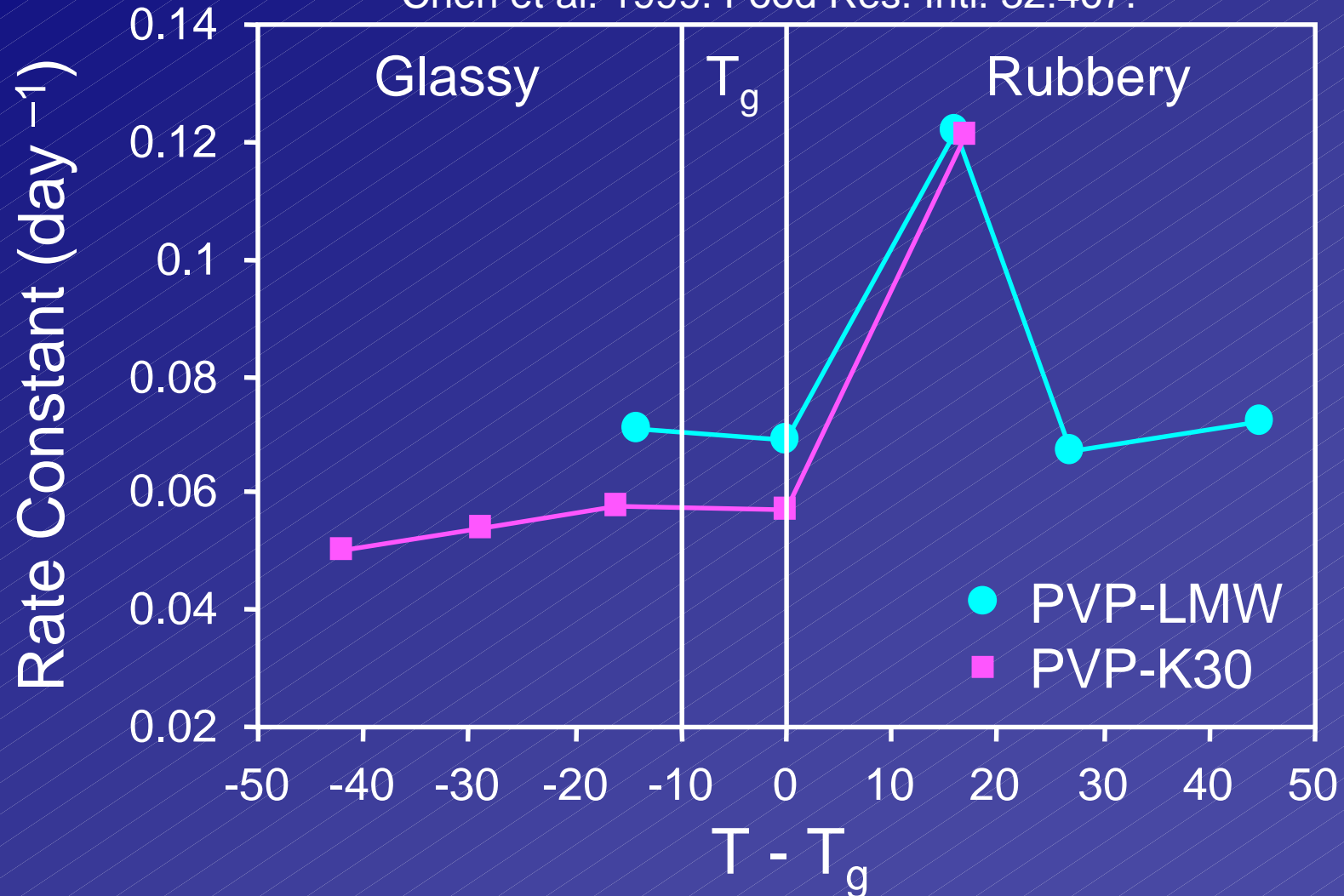


# RATE CONSTANTS FOR TYROSINASE ACTIVITY LOSS AT 20°C AS AFFECTED BY $a_w$



# RATE CONSTANTS FOR TYROSINASE ACTIVITY LOSS AT 20°C AS AFFECTED BY $T_g$

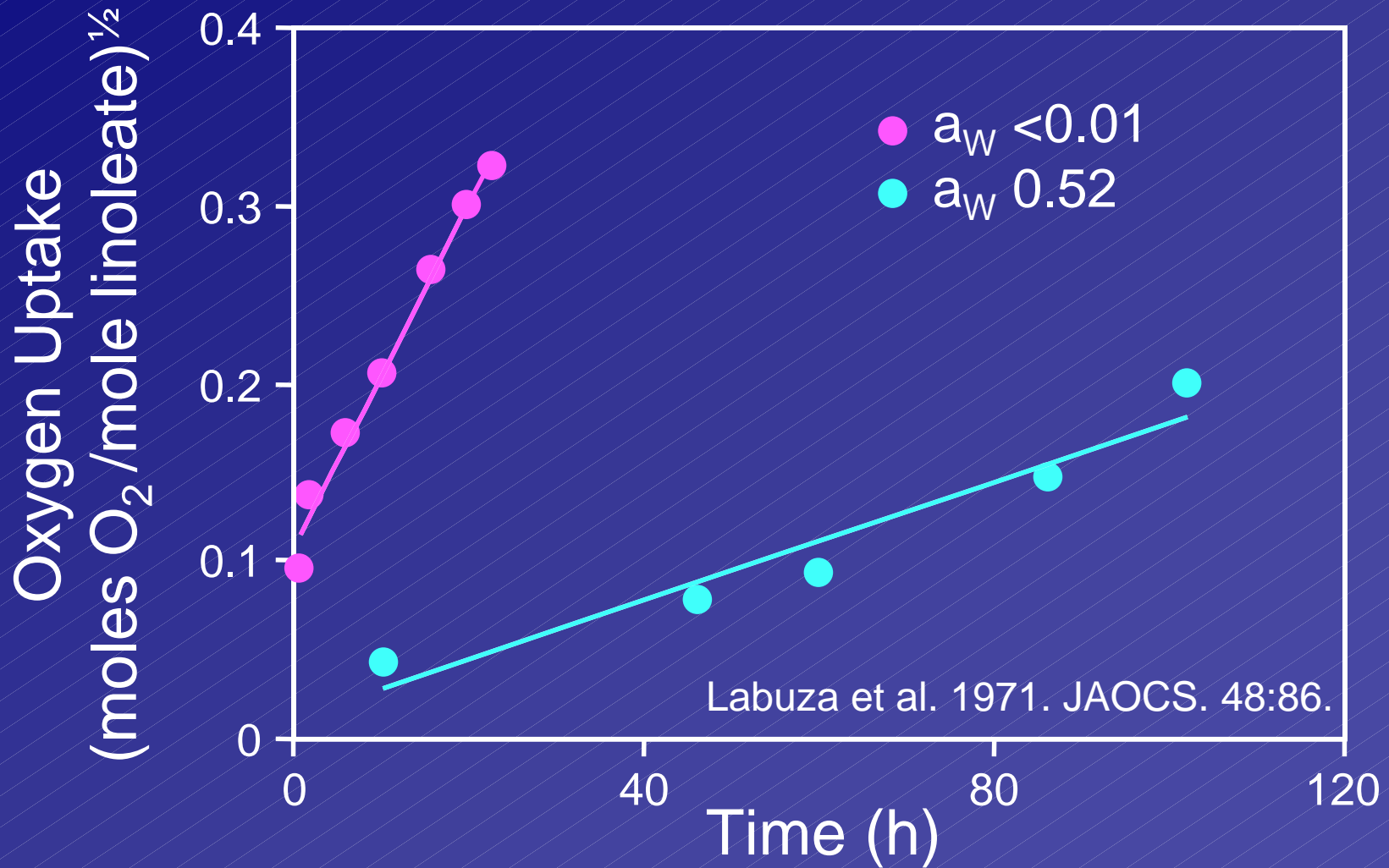
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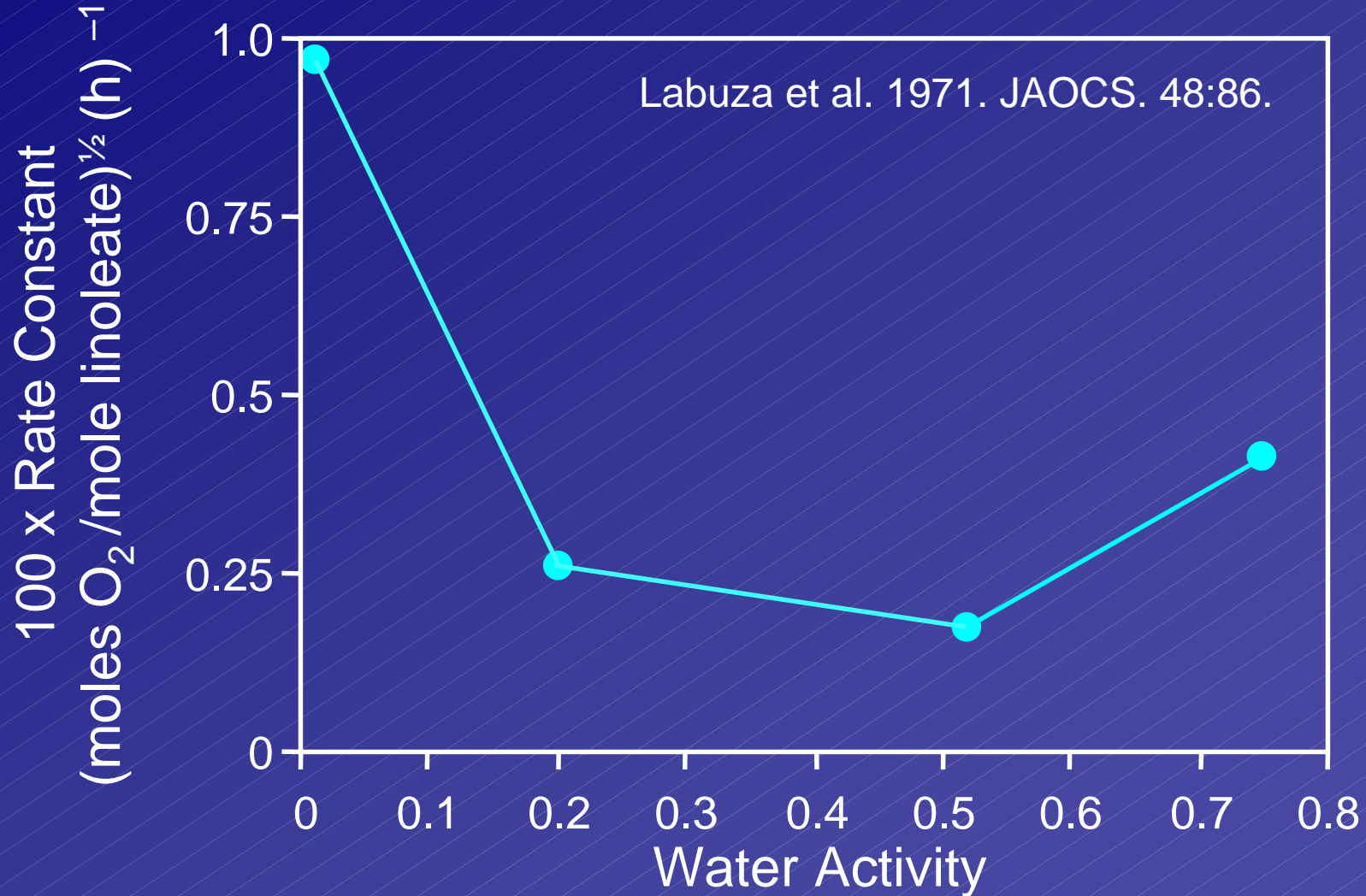
# LIPID OXIDATION

- Initiated by metal ions
- Measured by
  - Oxygen consumption
  - Peroxide values
  - Hexanal formation
- Complex kinetic modeling

# OXYGEN UPTAKE IN CELLULOSE/LINOLEATE MODEL SYSTEMS AT 37°C

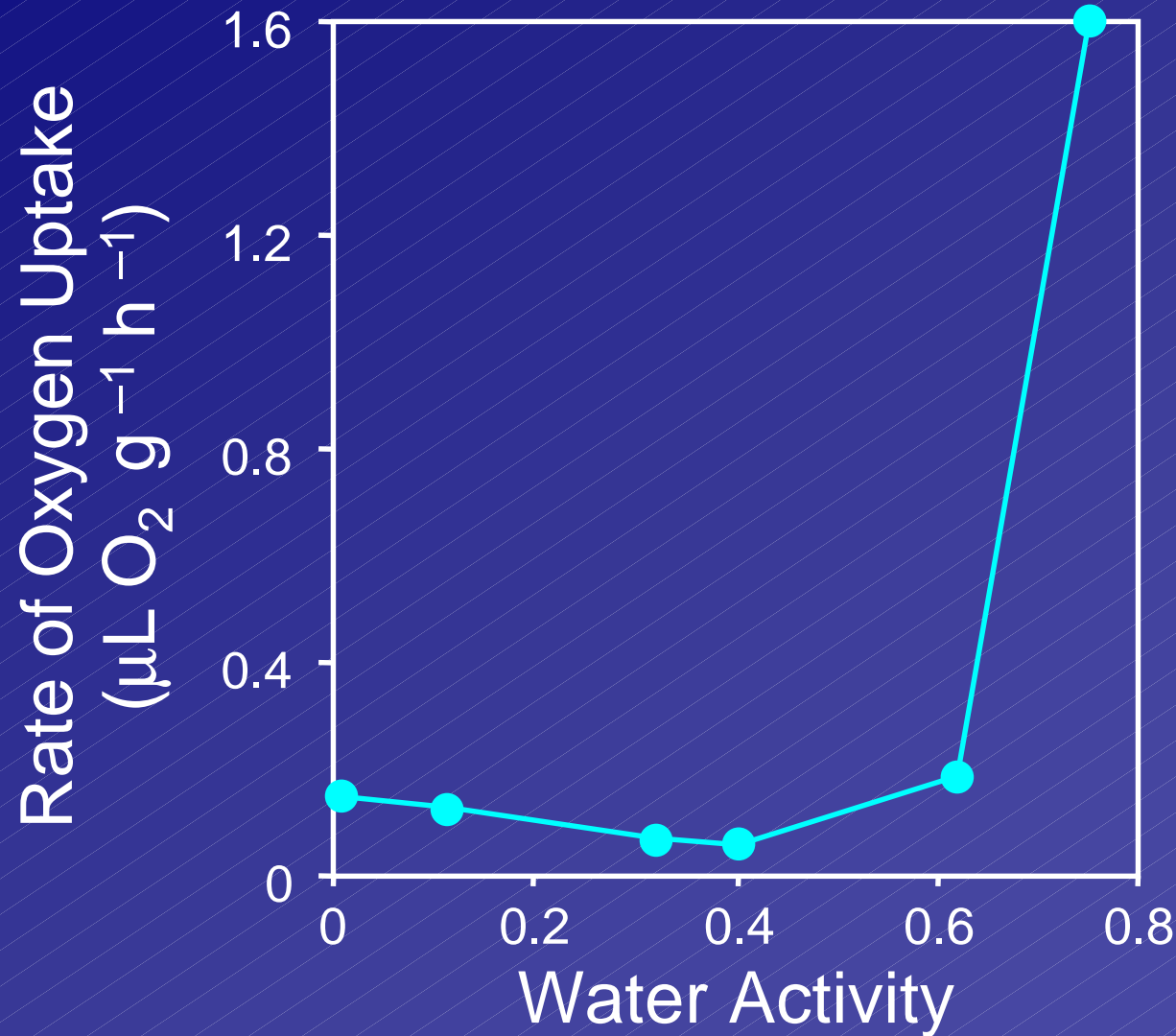


# RATE CONSTANT FOR OXYGEN UPTAKE BY LINOLEATE IN CELLULOSE MODEL SYSTEMS AT 37°C AS AFFECTED BY WATER ACTIVITY



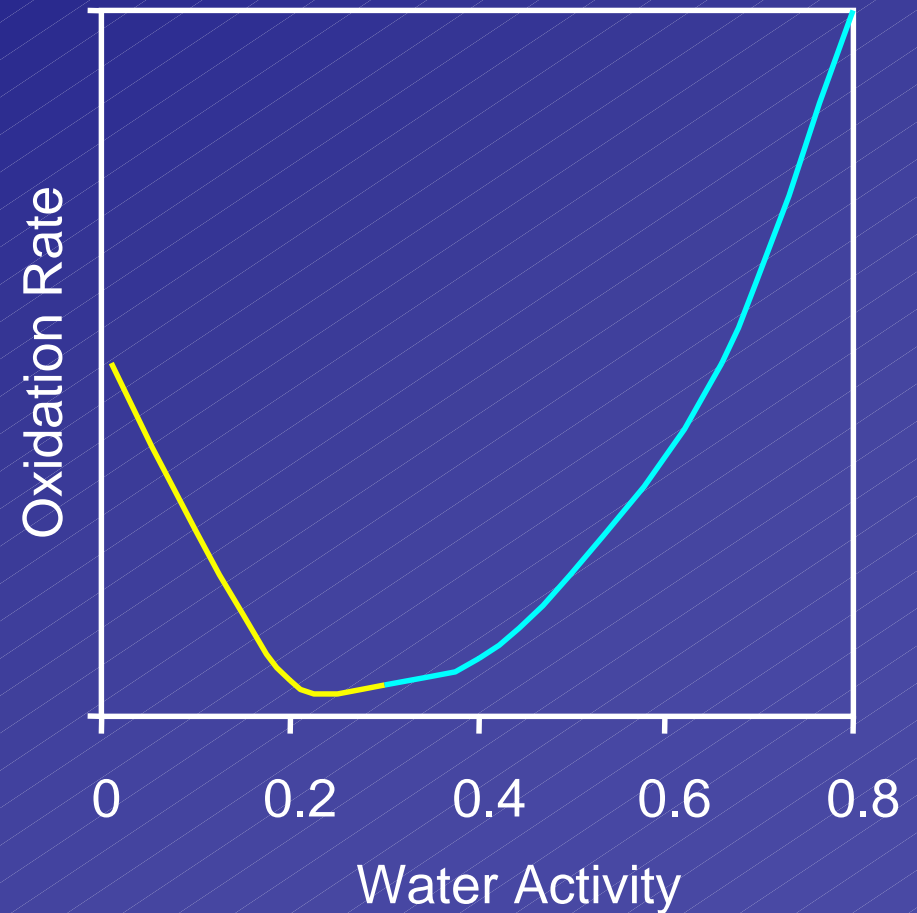
# LIPID OXIDATION IN POTATO CHIPS AT 37°C

Quast & Karel. 1972. J. Food Sci. 37:584.



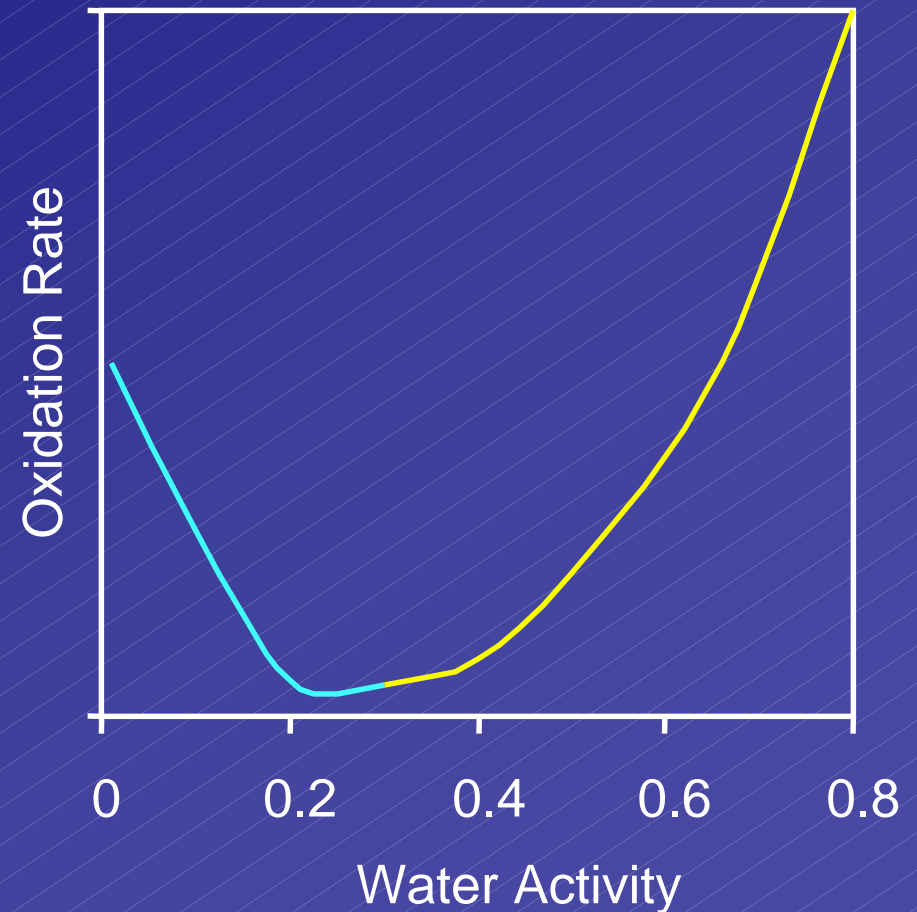
# WATER AND LIPID OXIDATION

- Increase  $a_w$  from 0 to 0.3
  - Less available metal ions due to hydration spheres
  - Reduced oxygen diffusion
  - Free radical quenching
- Rate decreases



# WATER AND LIPID OXIDATION

- Increase  $a_w$  from 0.3 to 0.8
  - Increased dissolution of catalysts
  - Increased mobility of oxygen and metal ions
- Rate increases



# LIPID OXIDATION AND GLASS TRANSITION

- Glassy encapsulants decrease oxidation by reducing oxygen diffusion
- Conversion to a rubbery matrix can increase oxygen diffusion and thus oxidation
- Structural collapse can:
  - Reduce oxidation of entrapped lipid by eliminating pores
  - Enhance oxidation if lipid becomes exuded
- For additional information about water and lipid oxidation:
  - Nelson & Labuza. 1992. Lipid Oxidation in Food. Washington, D.C.: ACS, pp. 93-103.

# pH EFFECTS

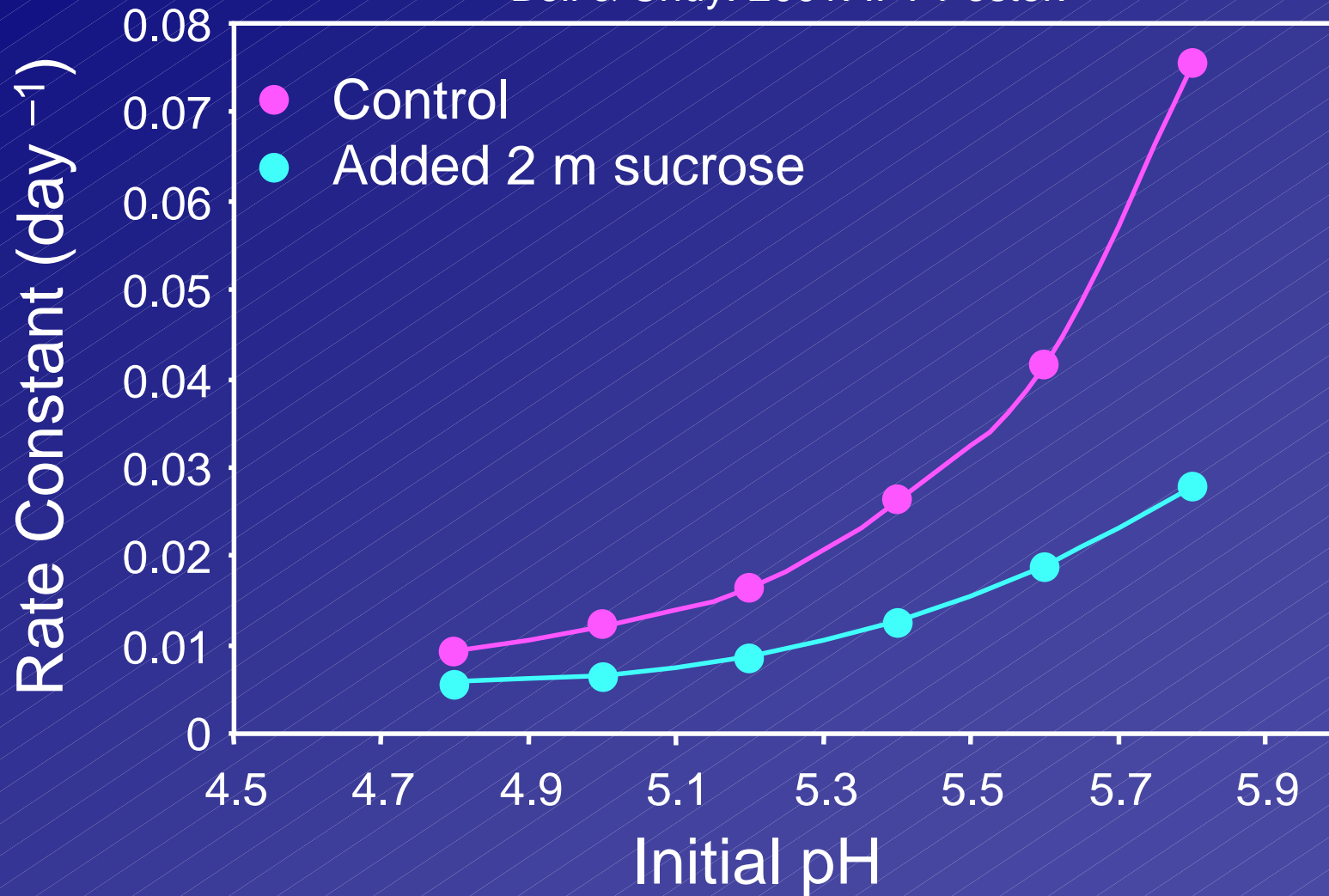
- Reactions affected by pH
  - Aspartame degradation
  - Maillard reaction
    - non-enzymatic browning
    - amino acid destruction
  - Vitamin degradation
- The importance of pH is not limited to solutions
  - Reducing water activity/moisture content can result in a pH change
  - pH changes can affect food chemical stability

# pH ESTIMATION OF REDUCED-MOISTURE SOLIDS USING A CHEMICAL MARKER

- Bell & Labuza. 1991. Cryo-Letters. 12:235.
  - At  $a_w$  0.99, 0.1 M phosphate buffer in agar/MCC has a pH of 6.5
  - After lyophilization and equilibration to  $a_w$  0.34, system behaves as pH 5
  - Generally attributed to increased concentration of buffer salts and their selective precipitation
- Results supported by other methods of estimating reduced-moisture pH
  - Bell & Labuza. 1992. J. Food Proc. Preserv. 16:289.

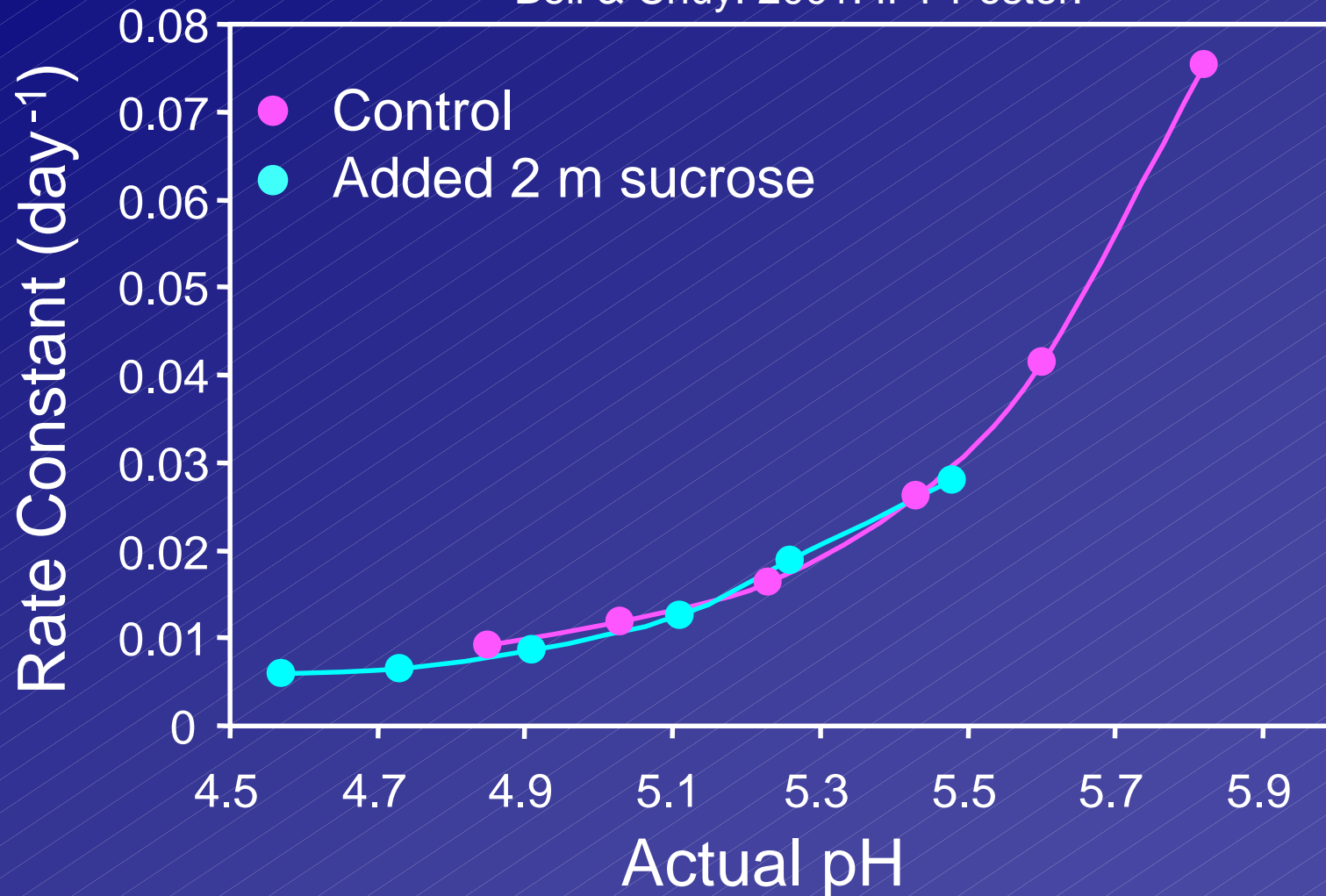
# ASPARTAME DEGRADATION IN 0.1 m PHOSPHATE BUFFER AT 25°C AS AFFECTED BY pH

Bell & Chuy. 2001. IFT Poster.



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Bell & Chuy. 2001. IFT Poster.



# STABILITY TESTING

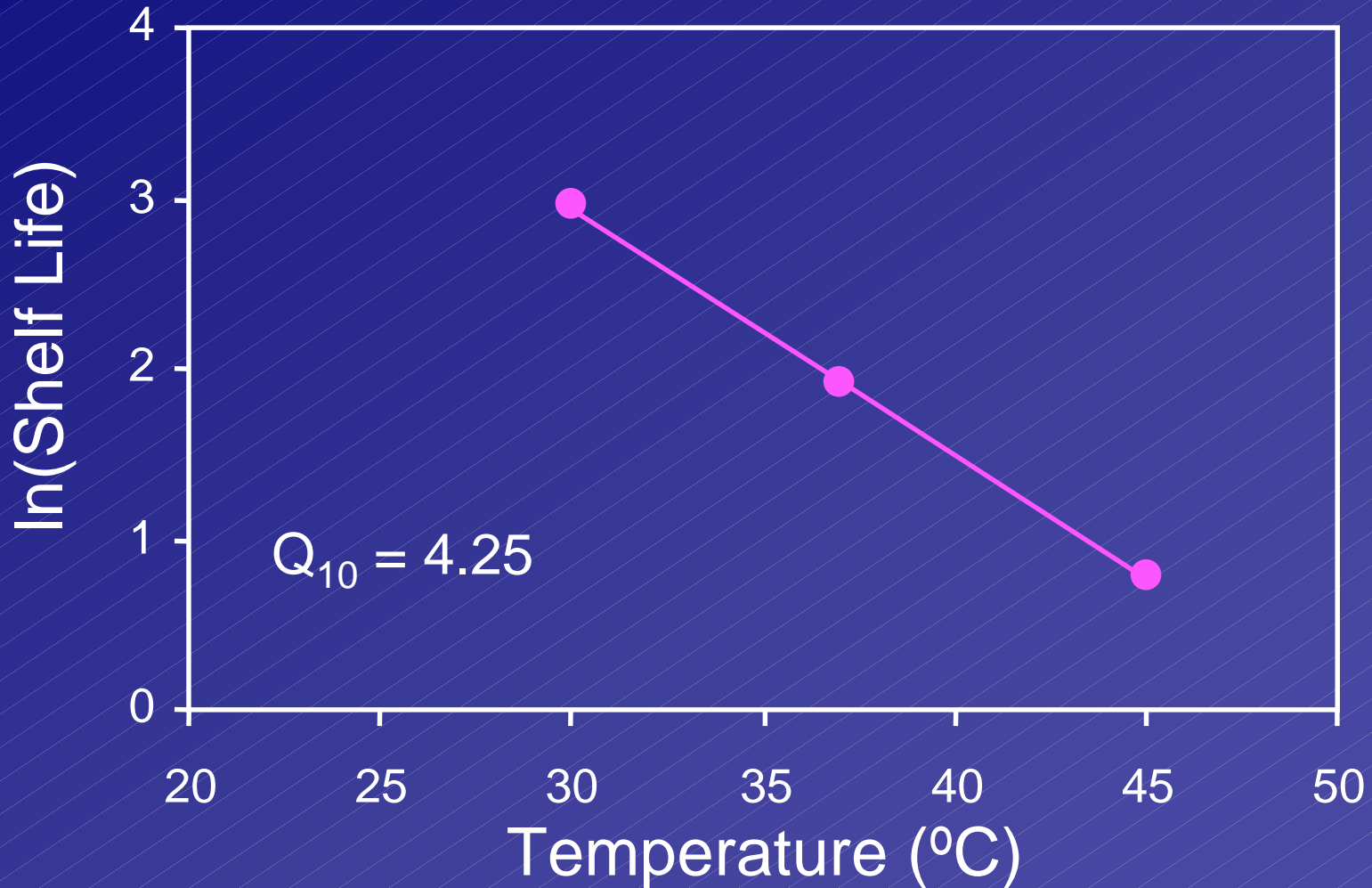
- System as close to commercial product as possible
  - formulation
  - processing
  - packaging
- Method of analysis
- Number of data points/extent of reaction
  - 8 points minimum
  - 50% change in reactant concentration
- Three temperatures
- Three water activities
- Extreme conditions may be used to accelerate testing

# SHELF LIFE TESTING EXAMPLE

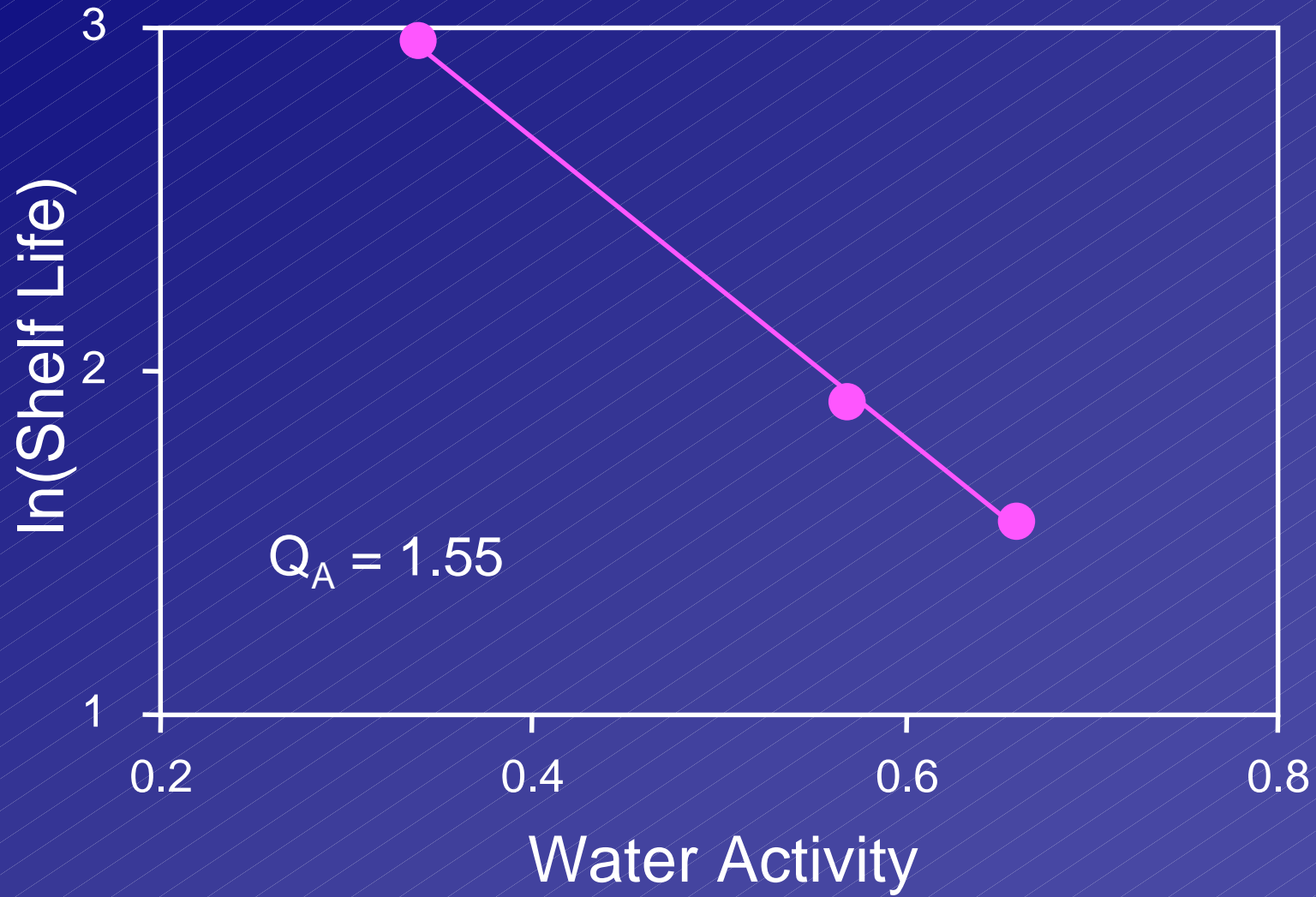
Given the following aspartame degradation data, estimate the shelf life at 20°C and  $a_w$  0.15

Conditions	Rate Constant (d <sup>-1</sup> )	$t_{10\% \text{ loss}}$ (days)
$a_w$ 0.34, 30°C	0.00548	19.2
$a_w$ 0.35, 37°C	0.01538	6.9
$a_w$ 0.42, 45°C	0.04828	2.2
$a_w$ 0.34, 30°C	0.00548	19.2
$a_w$ 0.57, 30°C	0.01574	6.7
$a_w$ 0.66, 30°C	0.02224	4.7

# SHELF LIFE PLOT FOR ASPARTAME DEGRADATION AT $a_w$ 0.34



# $Q_A$ PLOT FOR ASPARTAME DEGRADATION AT 30°C



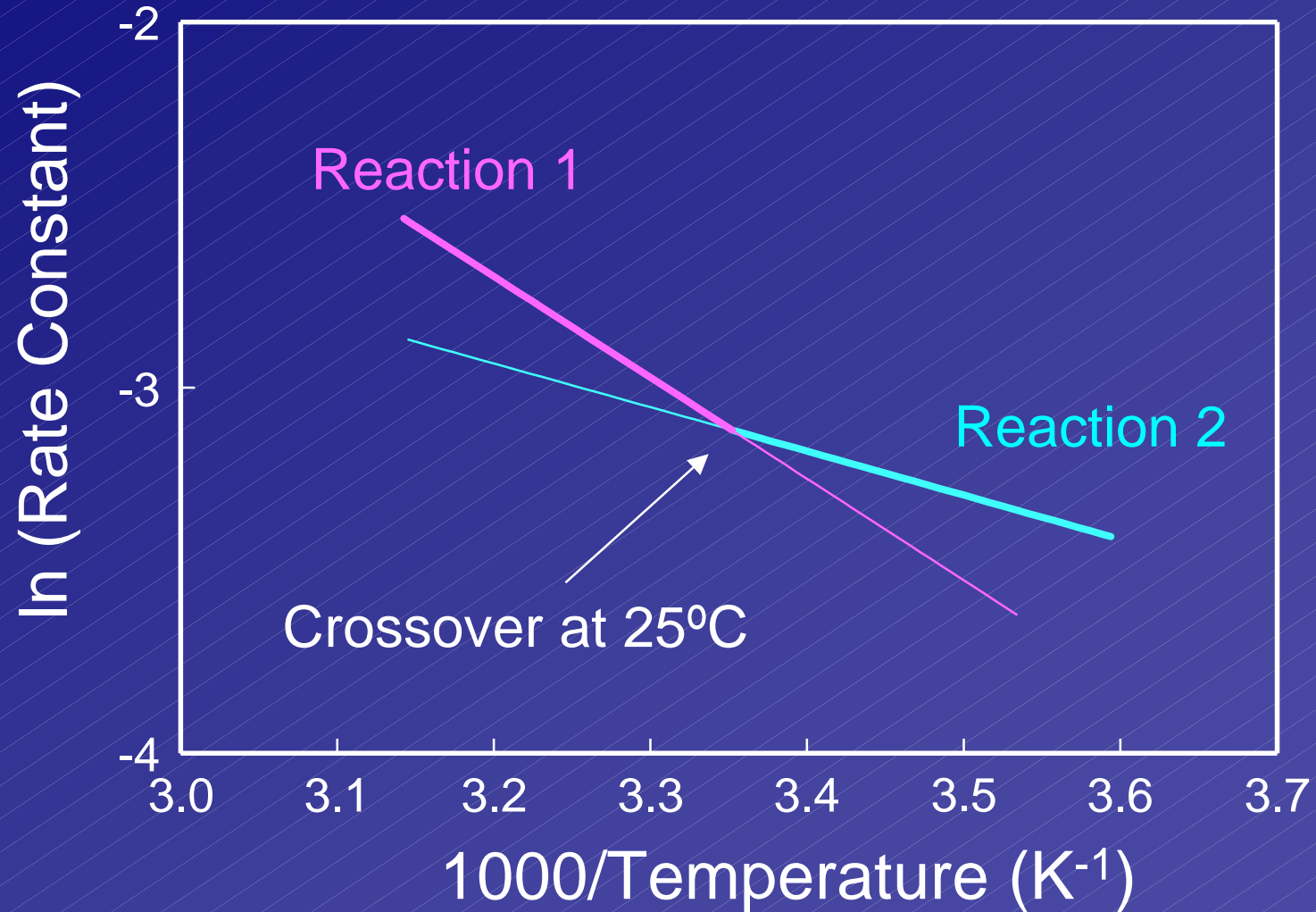
# SHELF LIFE CALCULATION

- From kinetic data:
  - At  $a_w$  0.34 and 30°C,  $t_{10\%} = 19.2$  days
  - $Q_{10} = 4.25$
  - $Q_A = 1.55$
- Want to know shelf life at  $a_w$  0.15 and 20°C
  - $(t_{10\%, a_w 0.34, 30^\circ\text{C}}) \times (Q_{10})^{\Delta T/10} \times (Q_A)^{\Delta a/0.1}$
  - $\Delta T =$  change in temperature from 30°C
  - $\Delta a =$  change in water activity from 0.34
  - $(19.2) \times (4.25)^{10/10} \times (1.55)^{0.19/0.1} = 188$  days

# PROBLEMS OF ACCELERATED SHELF LIFE TESTING AT EXTREME TEMPERATURES

- Water activity changes with temperature
- pH changes with temperature
- Solubility of reactants change
- Phase changes
  - Glass transition
  - Crystallization
- Competing chemical reactions

# SIMULTANEOUS DEGRADATIVE REACTIONS WITHIN A FOOD



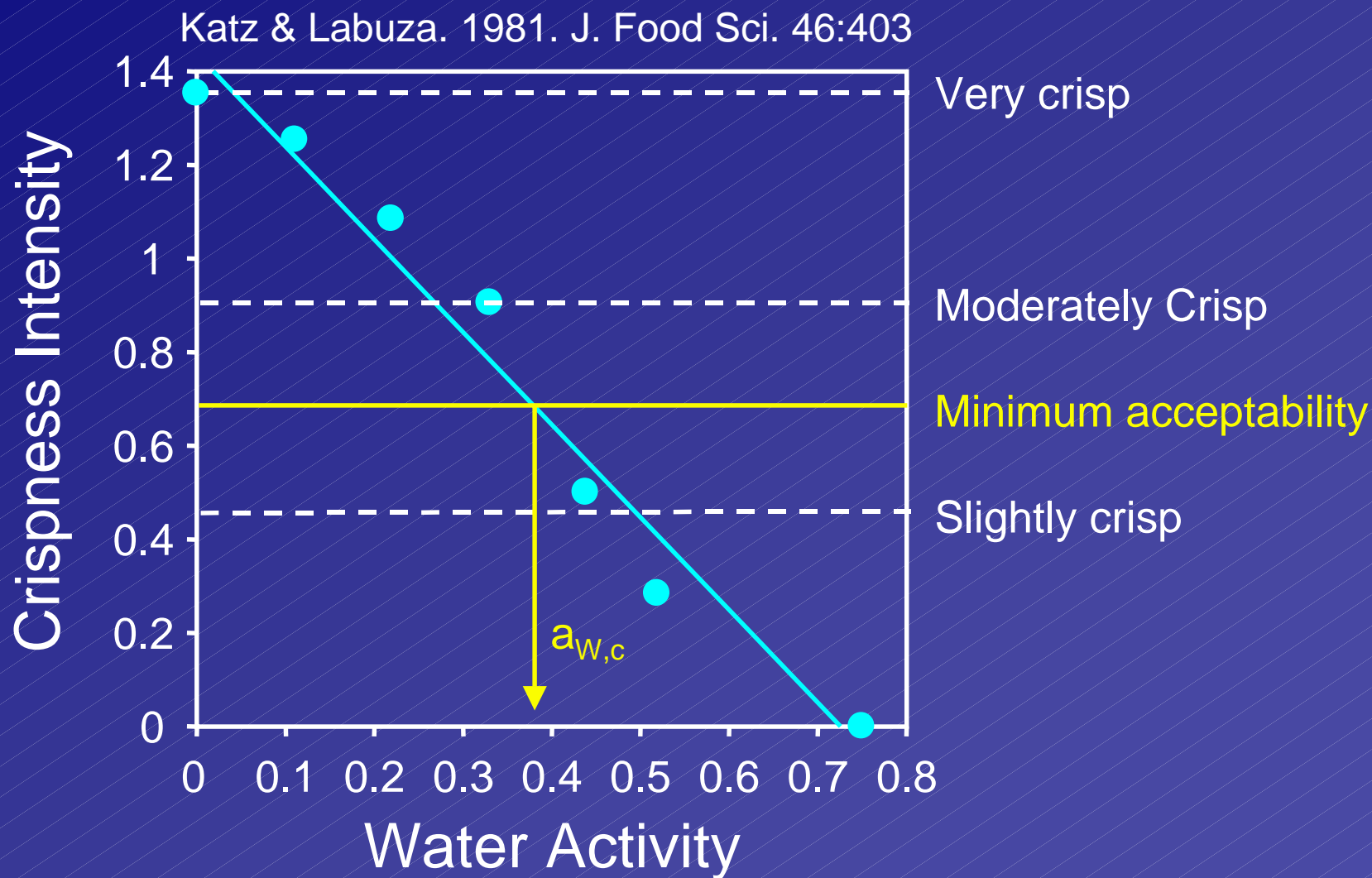
# WATER & PHYSICAL STABILITY

# PHYSICAL STABILITY

- Solutions
  - separation
  - crystallization
- Low and intermediate moisture systems
  - hardening or softening\*
  - caking and sticking of powders\*
  - crystallization\*

\* Influenced by conversion of a glassy matrix into a rubbery matrix

# SENSORY CRISPNESS INTENSITY OF SALTINE CRACKERS AS A FUNCTION OF $a_w$



# TEXTURAL ISSUES

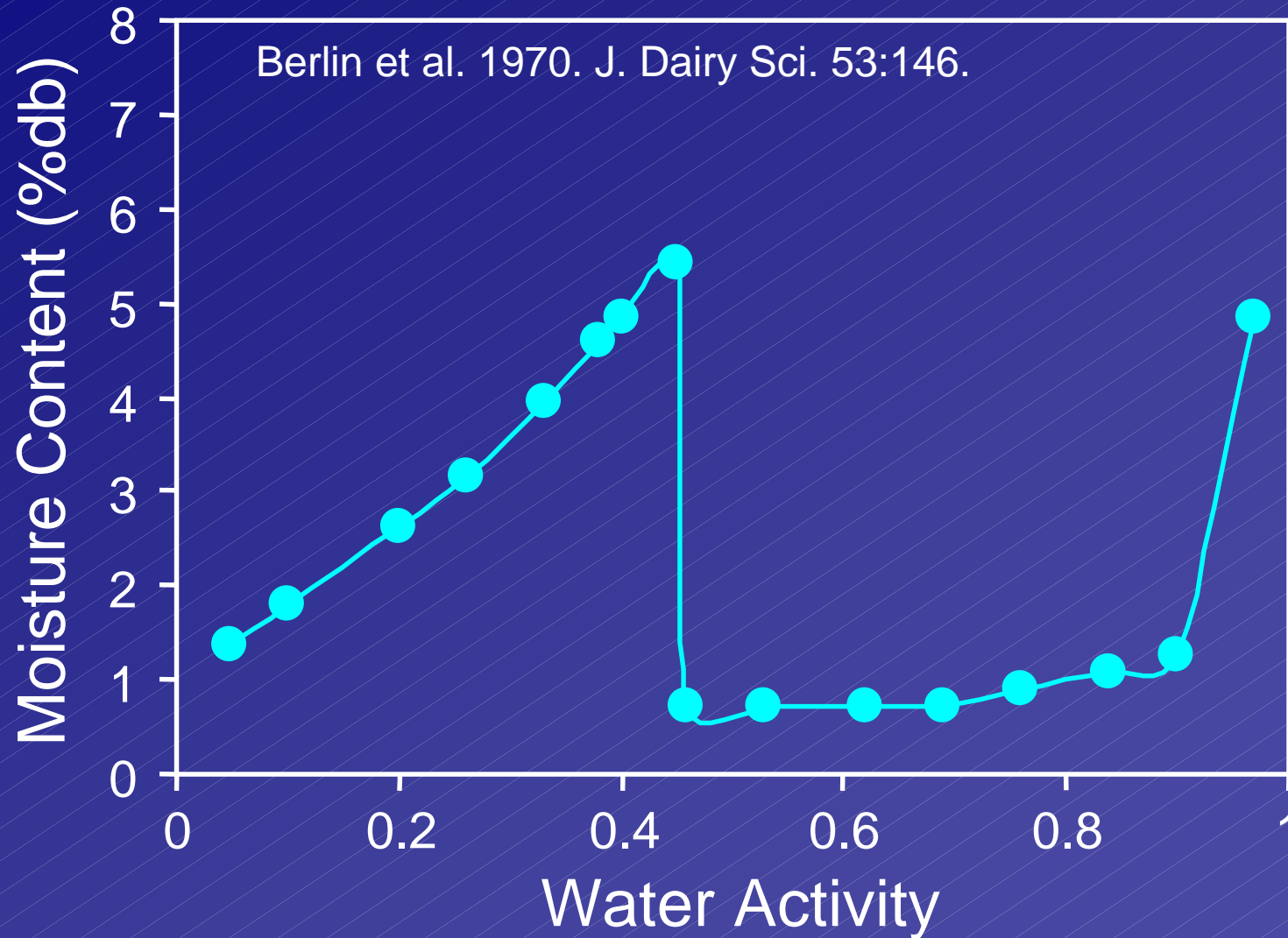
- Staling (softening) of cereal products
  - Crispness, crunchiness lost if  $a_w > 0.4$
  - Aim to keep  $a_w < 0.4$
- Hardening of fruit pieces
  - Softness, chewiness lost if  $a_w < 0.5-0.6$
  - Aim to keep  $a_w > 0.5-0.6$
- Contradictory goals for a fruit-containing cereal product



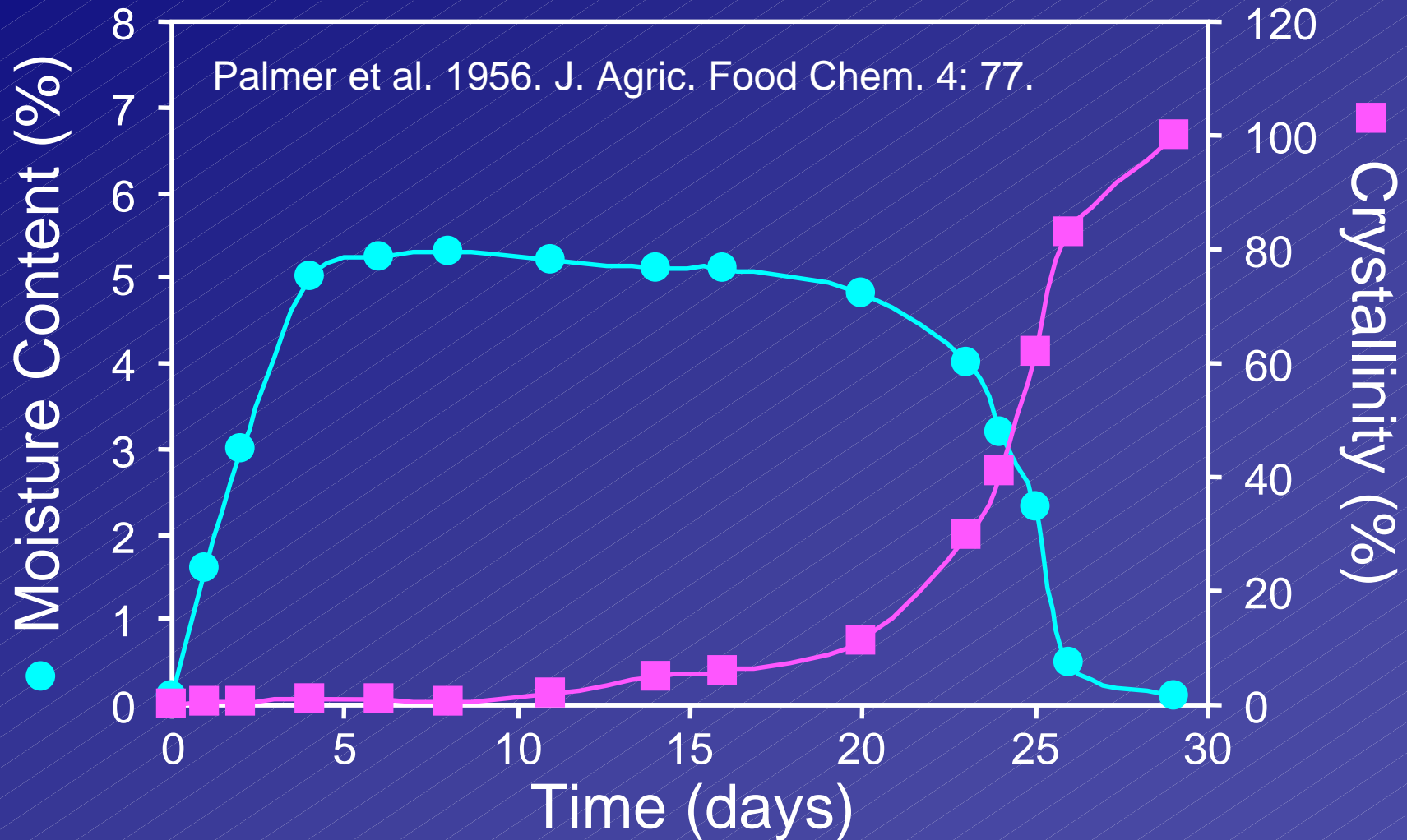
# CAKING AND STICKING OF POWDERS

- Powders in the glassy state become rubbery from:
  - Moisture sorption
  - Temperature abuse
- Rubbery powders flow together under the force of gravity
- Over long periods of time, the net result is formation of a caked mass
- Supporting literature
  - Aguilera et al. 1993. Biotech. Prog. 9:651.
  - Chuy & Labuza. 1994. J. Food Sci. 59:43.
  - Lloyd et al. 1996. J. Food Eng. 31:305.
  - Netto et al. 1998. Int. J. Food Prop. 1:141.

# MOISTURE SORPTION ISOTHERM OF FREEZE-DRIED LACTOSE AT 34°C



# EFFECT OF EXPOSURE TIME AT $a_w$ 0.3 AND 24°C ON MOISTURE CONTENT AND CRYSTALLINITY OF AMORPHOUS SUCROSE



# EFFECTS OF CRYSTALLIZATION

- Amorphous ingredients may crystallize from:
  - Moisture gain
  - Temperature abuse
  - Storage (time)
- Crystalline materials hold less moisture so upon crystallization water is released.
- This water is redistributed in the food, causing:
  - Water activity to increase
  - Glass transition temperature of remaining amorphous regions to decrease
  - Further chemical and physical deterioration!
- Literature
  - Roos & Karel. 1990. Biotech. Prog. 6:159.

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# USING MOISTURE SORPTION ISOTHERMS AND PHASE DIAGRAMS TO ESTIMATE CRITICAL WATER ACTIVITY VALUES

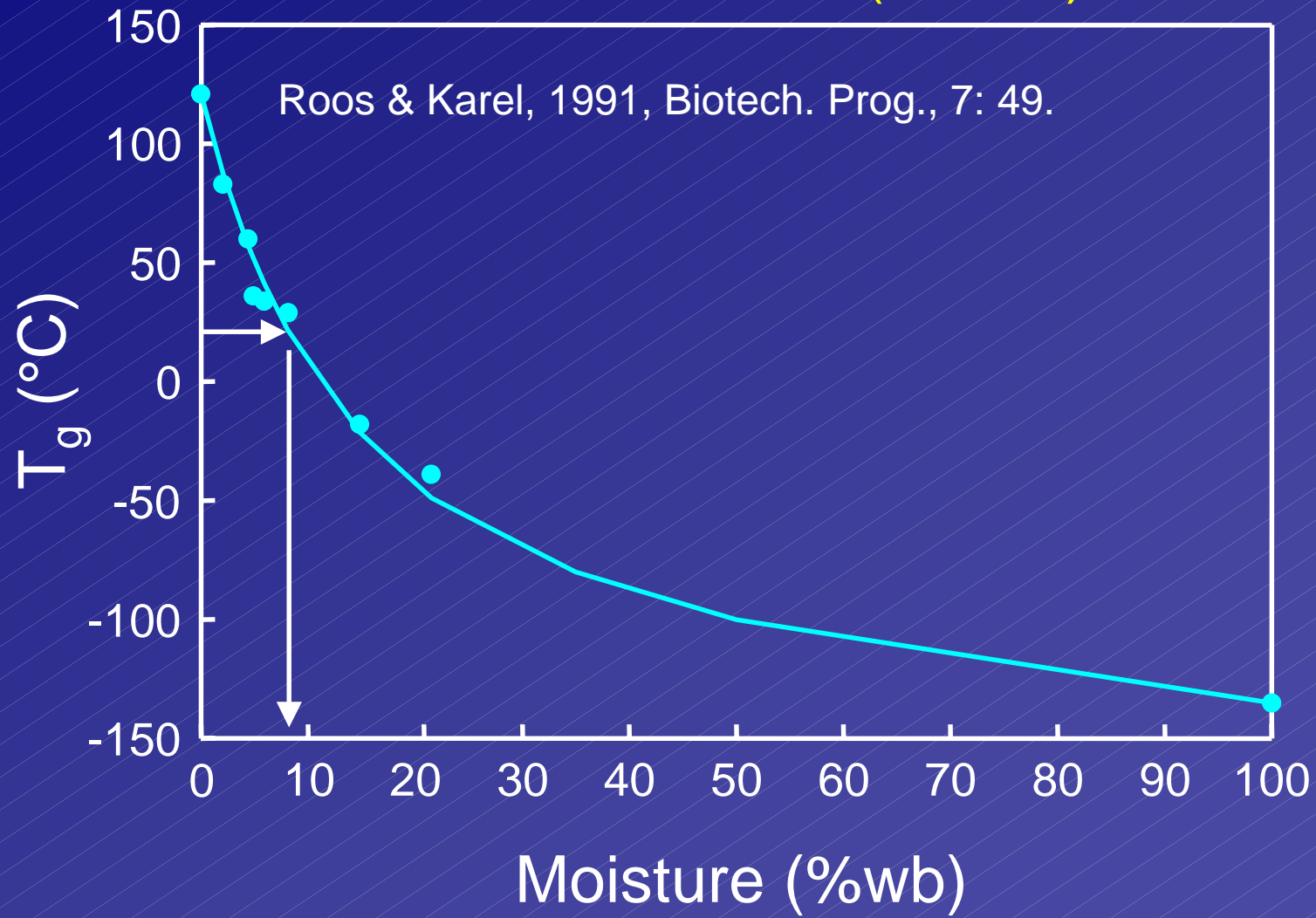
# APPLICATION OF PHASE TRANSITION TO A MALTODEXTRIN POWDER

- Caking of powders occurs due to the conversion of glassy particles into rubbery particles which stick together.

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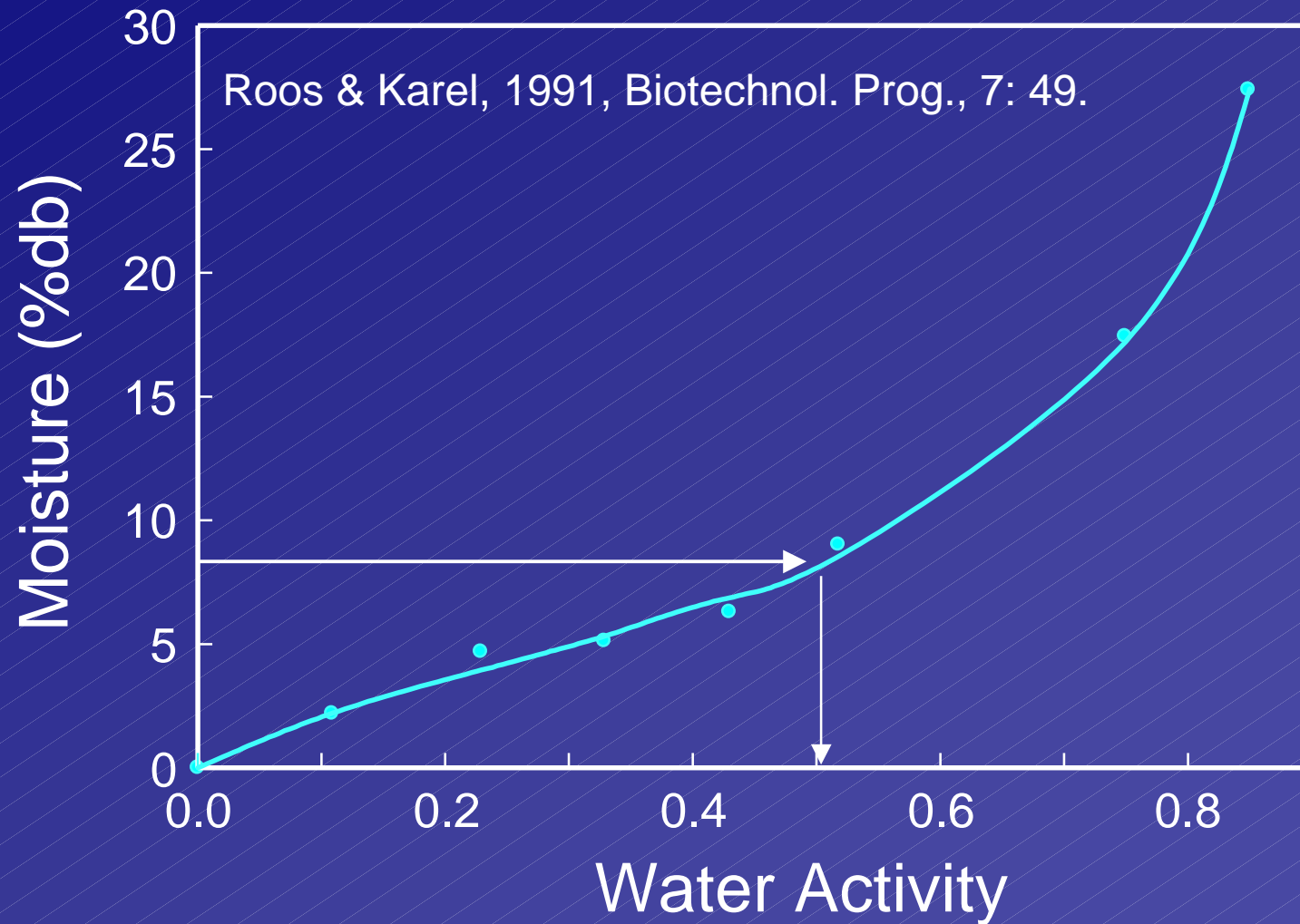
# GLASS TRANSITION STATE DIAGRAM FOR MALTODEXTRIN (DE 25)



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- From moisture sorption isotherm, maltodextrin holds 8% water at  $a_w$  0.5.
- Moisture gain from an environment when  $a_w > 0.5$  will promote caking of the maltodextrin.

# SUGGESTIONS FOR CONTROLLING MOISTURE TRANSFER (GAIN OR LOSS)

- Understand moisture sorption isotherms
- Formulation approaches
  - Humectants
  - Anticaking agents (e.g. calcium silicate)
- Packaging approaches
  - Select to minimize water permeation
  - Resealable packaging
- Handling instructions

# OVERVIEW: WATER AND STABILITY

- Chemical Stability
  - No universal physicochemical parameter describes the effects of water
  - Water activity and plasticization by water affects chemical stability differently depending upon:
    - Reaction type
    - Matrix
- Physical Stability
  - Water affects physical stability primarily by plasticizing glassy systems into rubbery systems