Primary Nucleation of Alpha Lactose Monohydrate: The Effect of Supersaturation and Temperature

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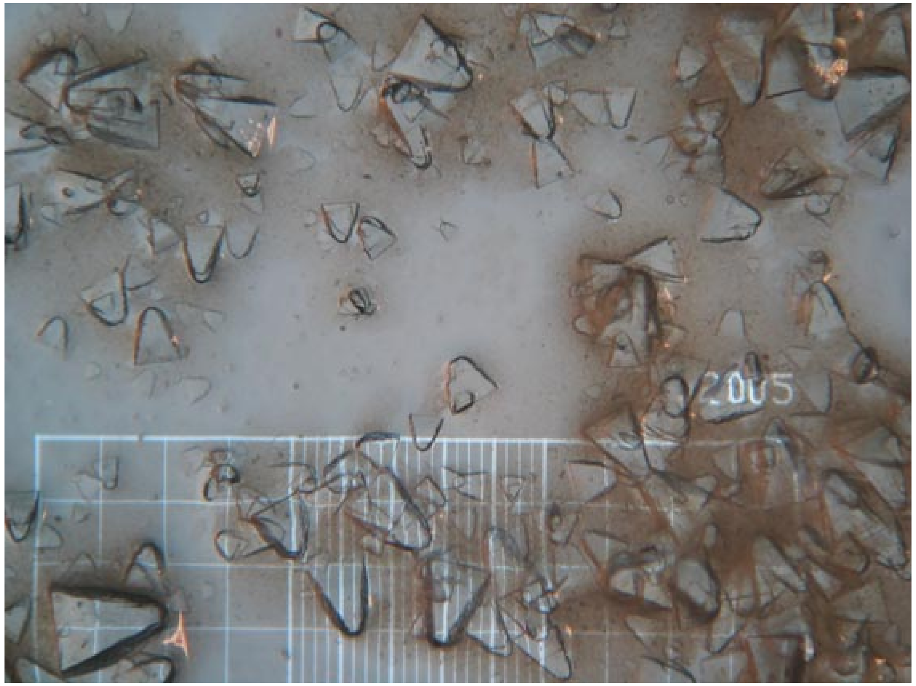
Nucleation – The effect on crystal size



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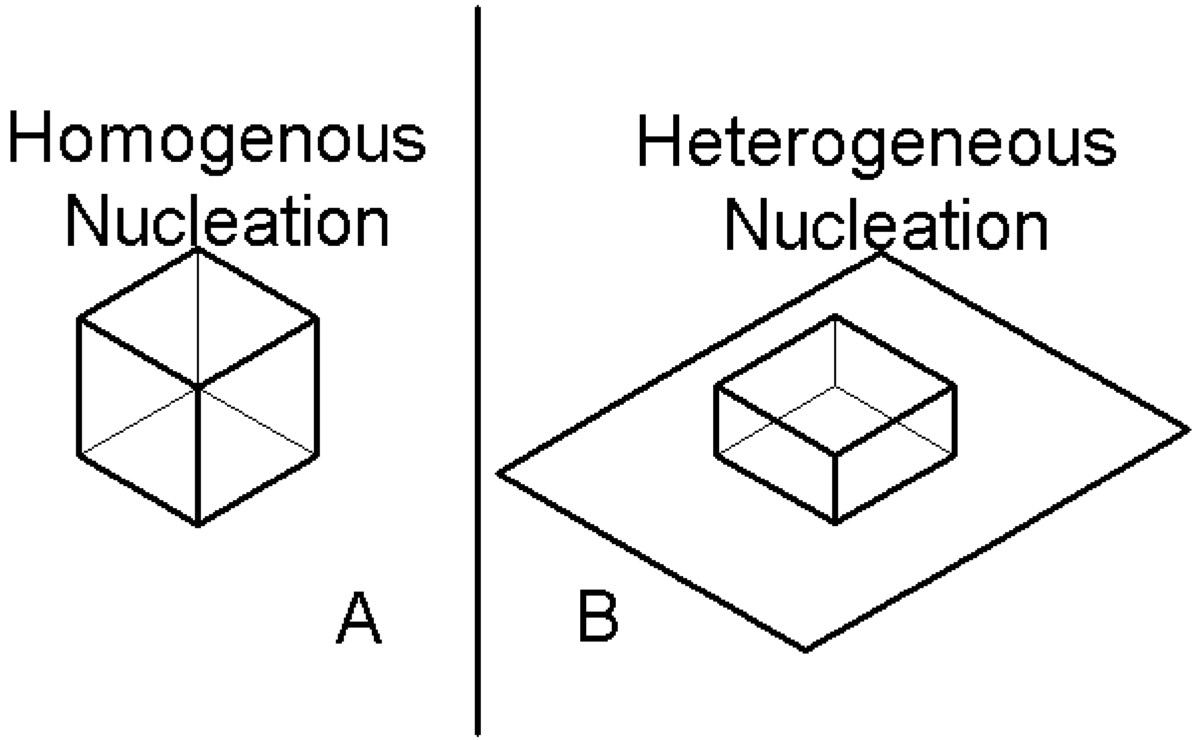
Nucleation Mechanisms

- Primary
  - Homogeneous
  - Heterogeneous
- Secondary
  - Initial Breeding
  - Polycrystalline Breeding
  - Macro abrasion
  - Dendritic
  - Fluid Shear
  - Contact

Primary Nucleation Mechanisms



Primary nucleation equations

$$J_{Hom} = A\exp\left(-\frac{16\pi\sigma^3 V_m^2}{3(kT_K)^3(\ln S_R)^2}\right)$$

$$J_{Hen} = A\exp\left(-\frac{16\pi\sigma^3 V_m^2(q)}{3(kT_K)^3(\ln S_R)^2}\right)$$

$$J_T = J_{Hom} + J_{Hen}$$

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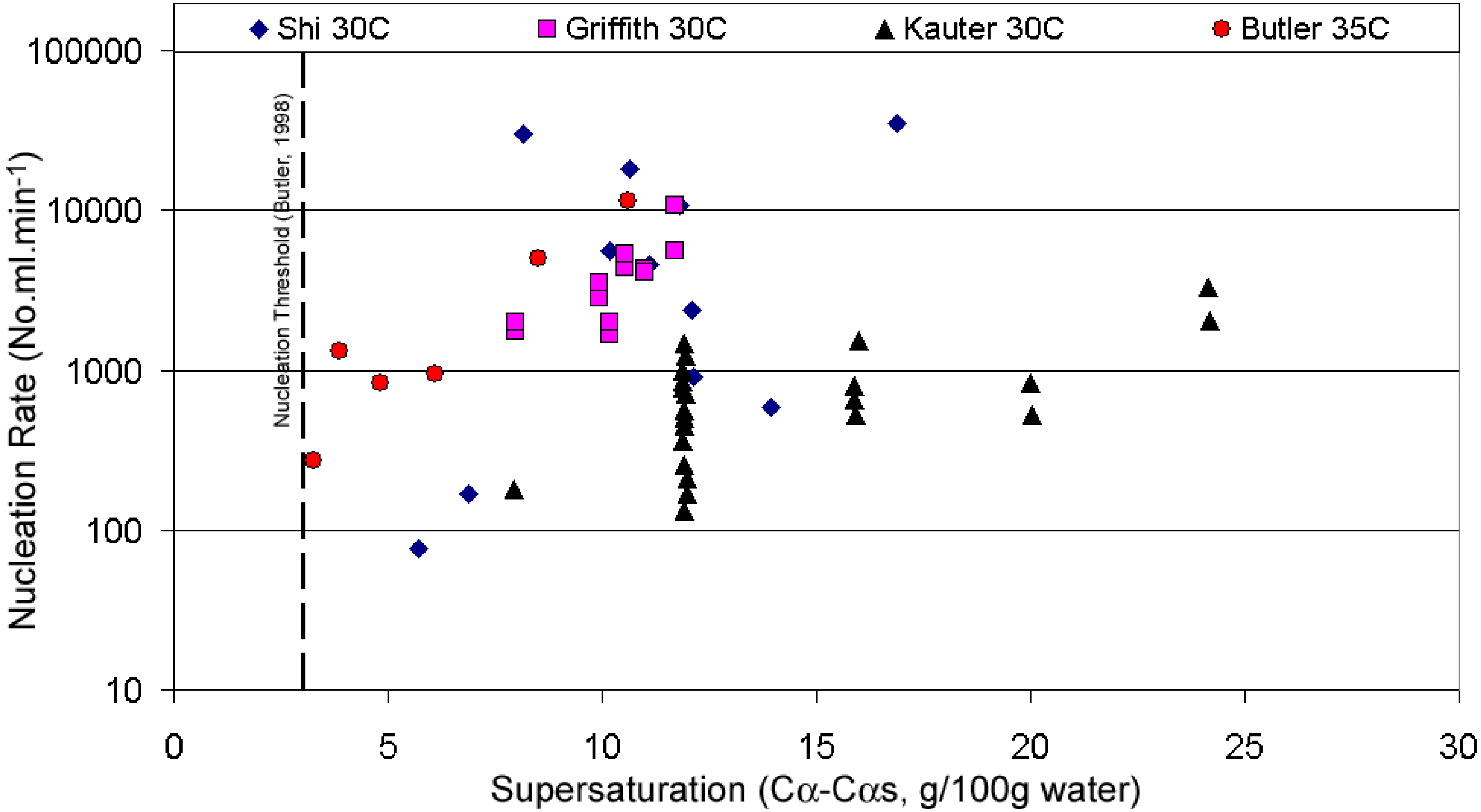
Lactose Nucleation – Previous Work

"In the manufacture of lactose it is desirable to secure a maximum yield of crystals in a minimum time, and to secure crystals which may be readily washed with a minimum of loss." Herrington, (1934)

Effect of Supersaturation on Nucleation

Shi, (1990), Griffith, (1982), Kauter, (2003), and Butler (1998)



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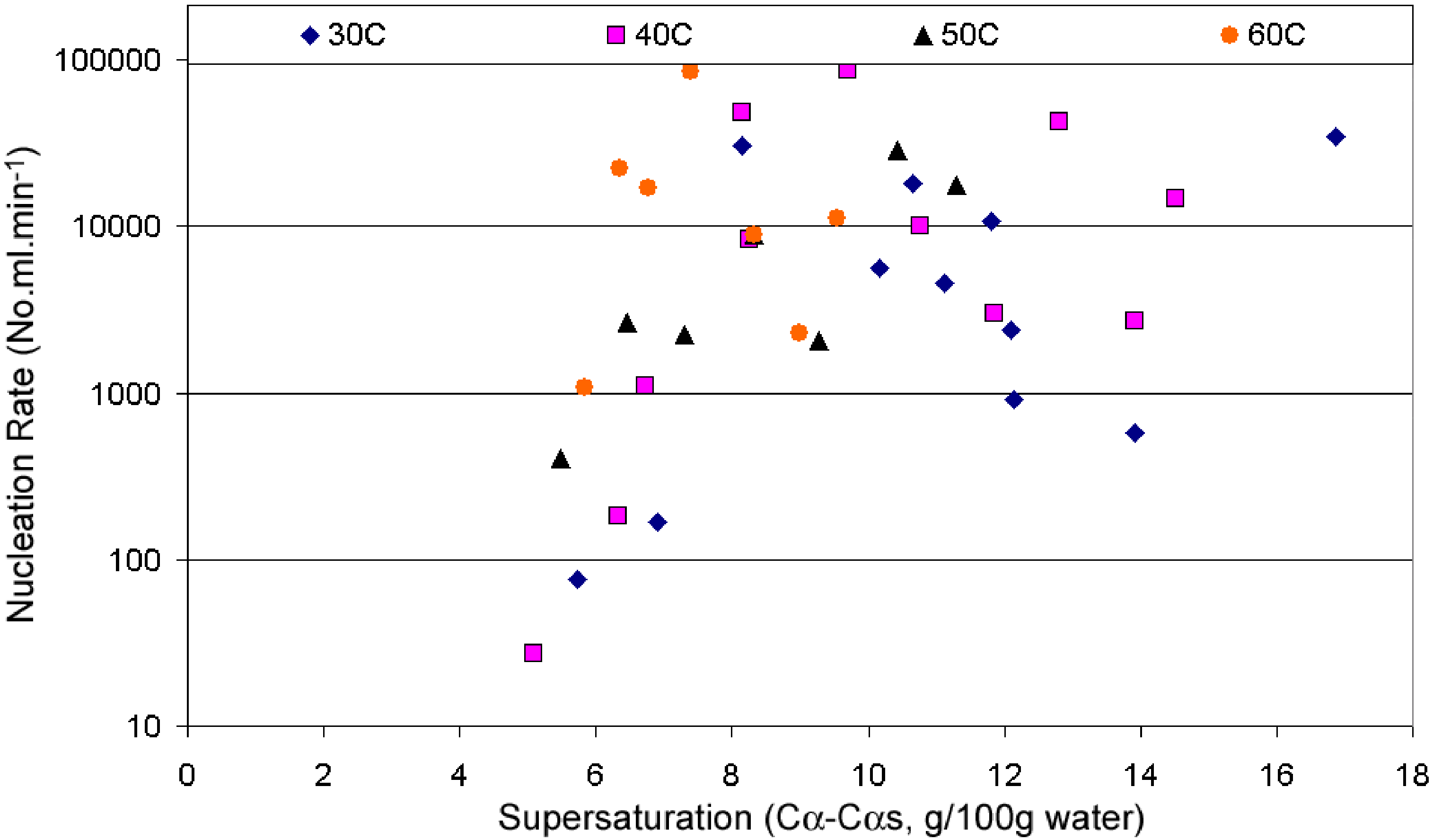
Effect of temperature on nucleation
(Shi, 1990)



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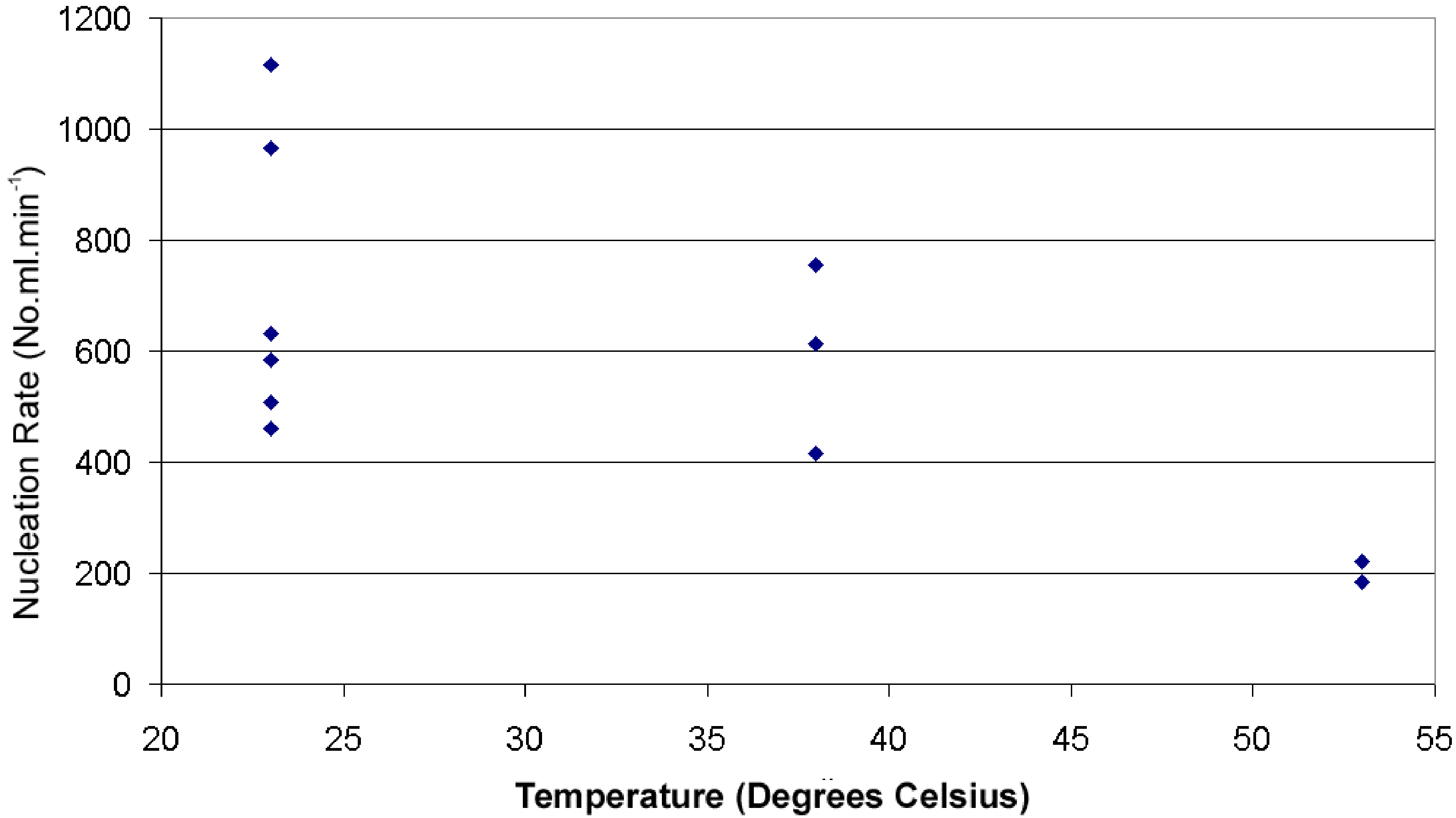
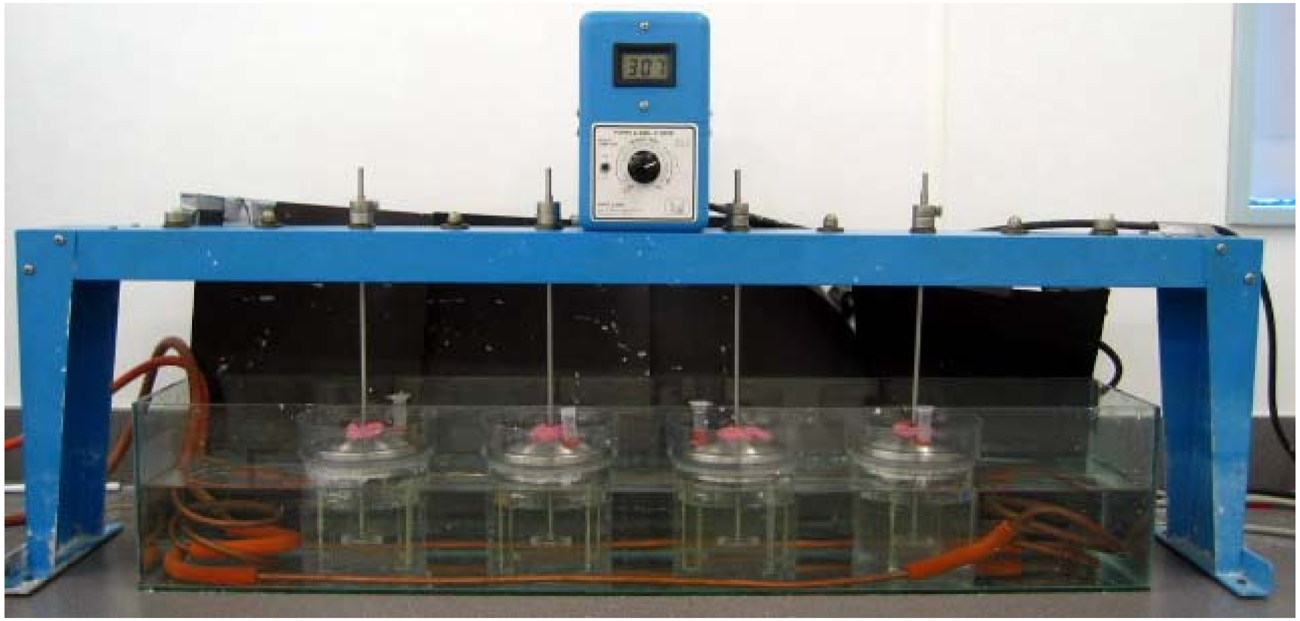
Effect of temperature on nucleation
(Kauter, 2003)



Experimental Setup



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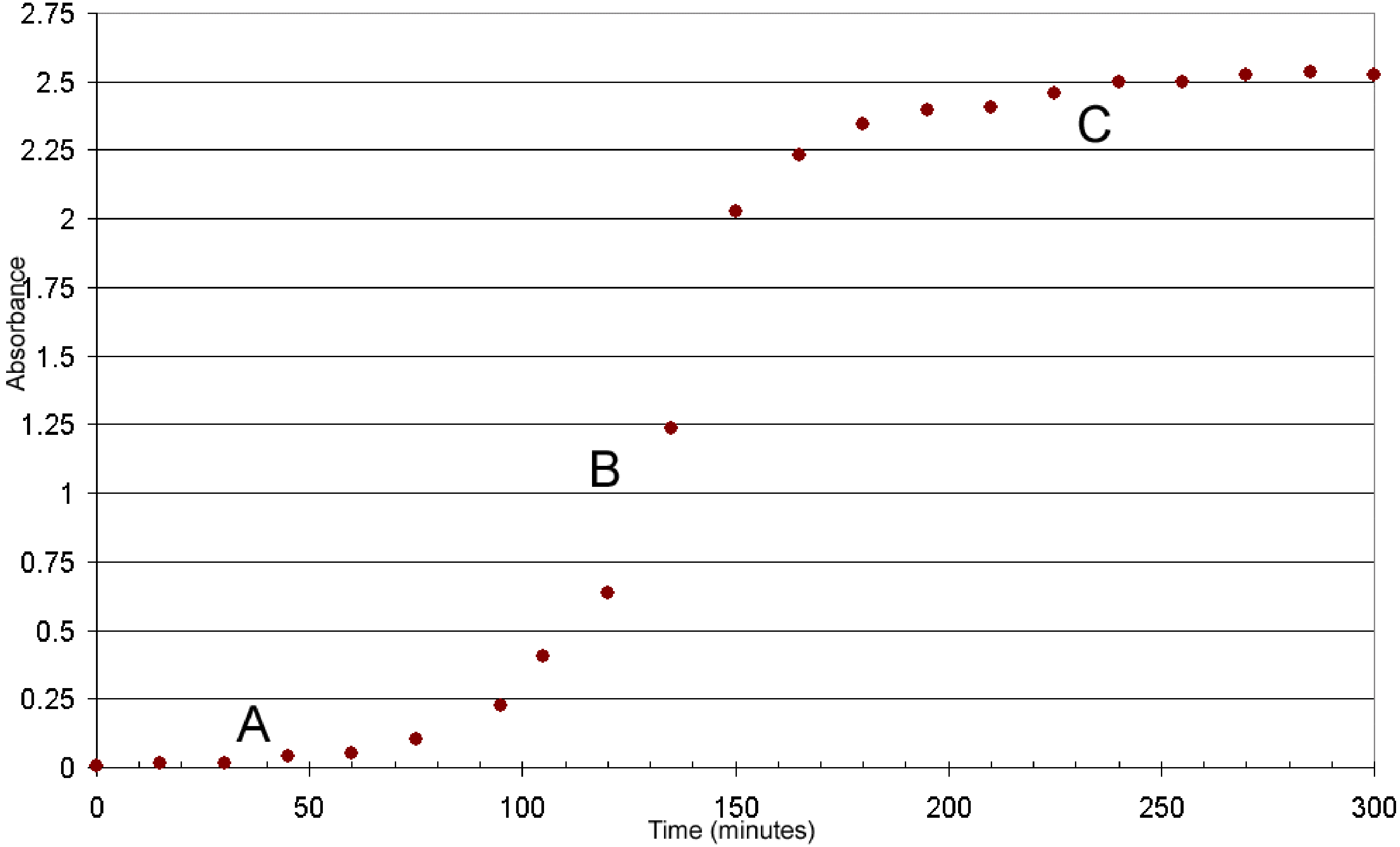
Relating induction time to the nucleation rate

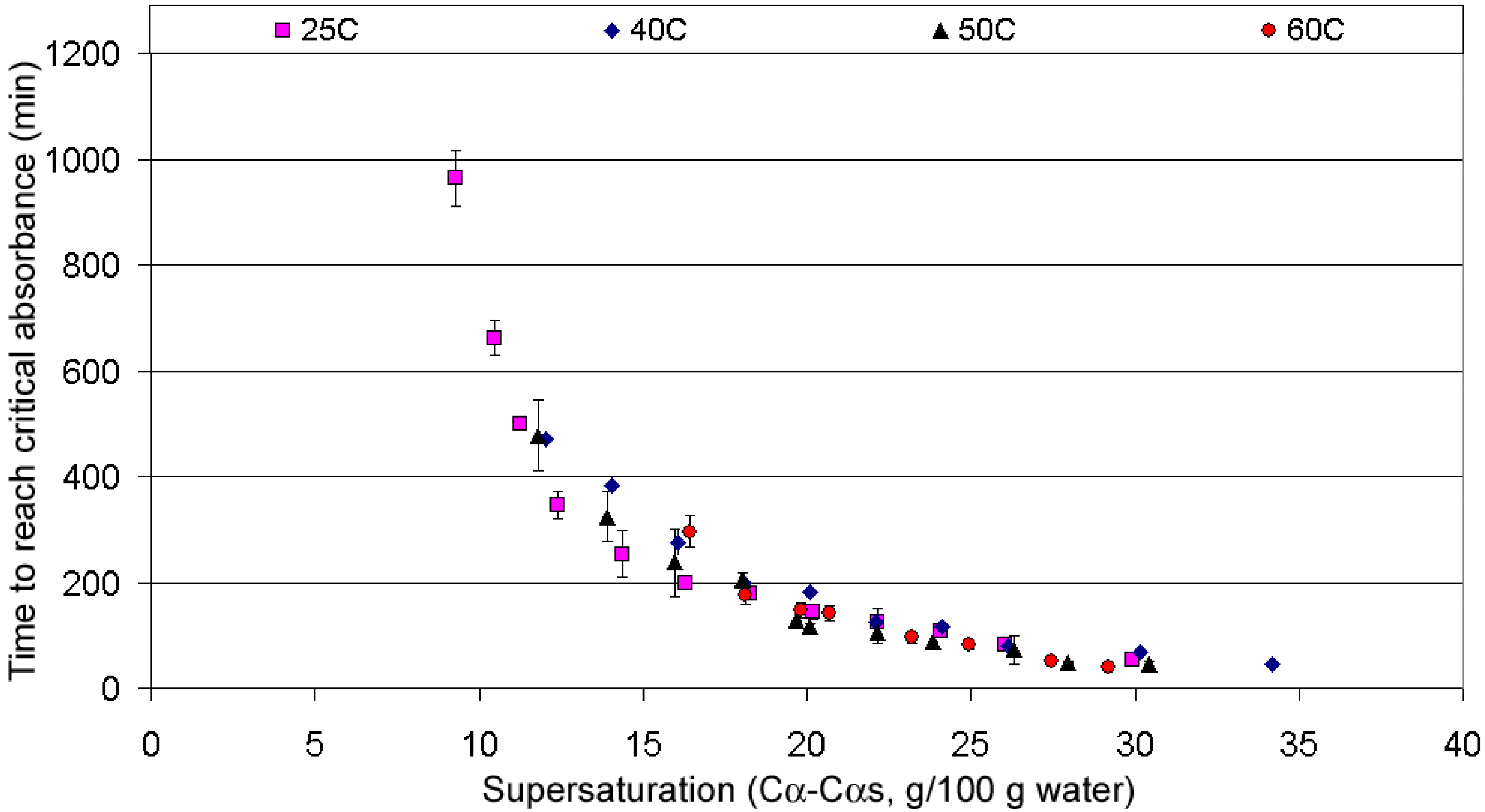
$$t_N \propto \frac{N_N}{J_T}$$

$$t_N = \frac{N_N}{J} = \frac{N_N}{A} \exp\left(-\frac{16\pi\sigma^3 V_m^2}{3(kT_K)^3 (\ln S_R)^2} \right)$$

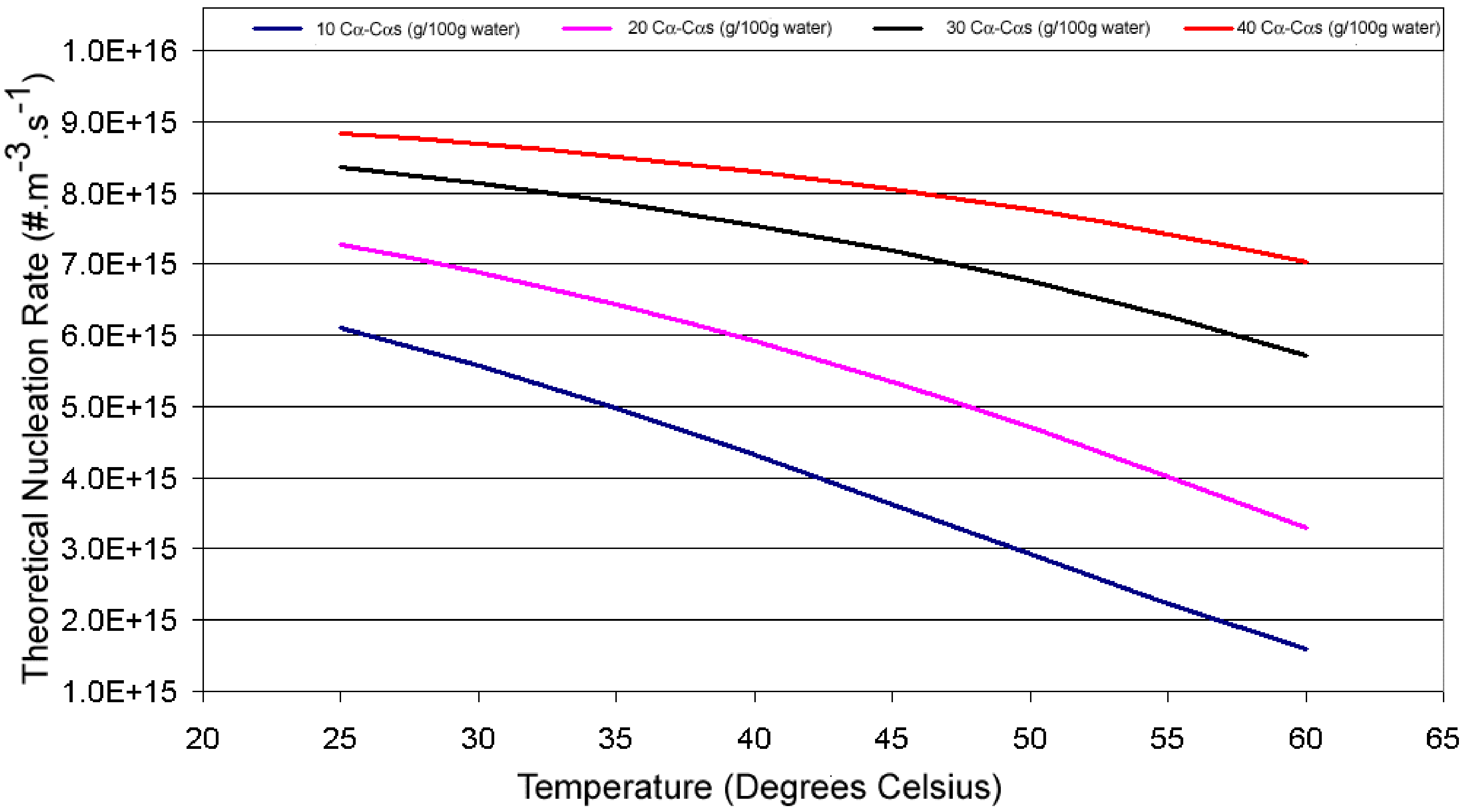
Typical absorbance curve for nucleation



Time to reach critical absorbance vs. supersaturation



The effect of temperature on nucleation rates at different absolute supersaturations



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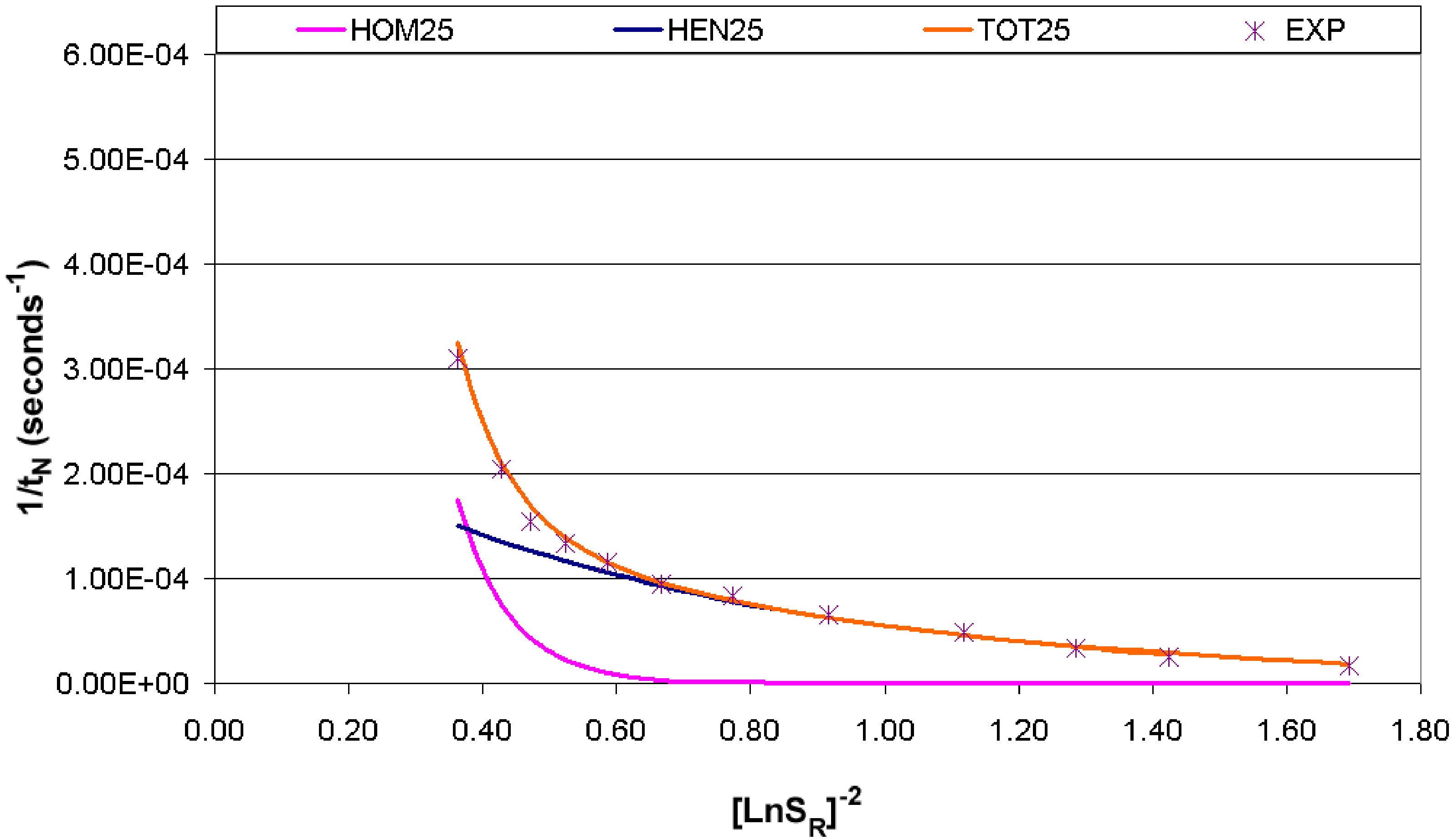
Primary Nucleation Mechanisms

$$t_N = A_{Hen} \times \exp^{(-B_{Hen} \times [\ln S_R]^{-2})} + A_{Hom} \times \exp^{(-B_{Hom} \times [\ln S_R]^{-2})}$$

Nucleation Results 25 Degrees



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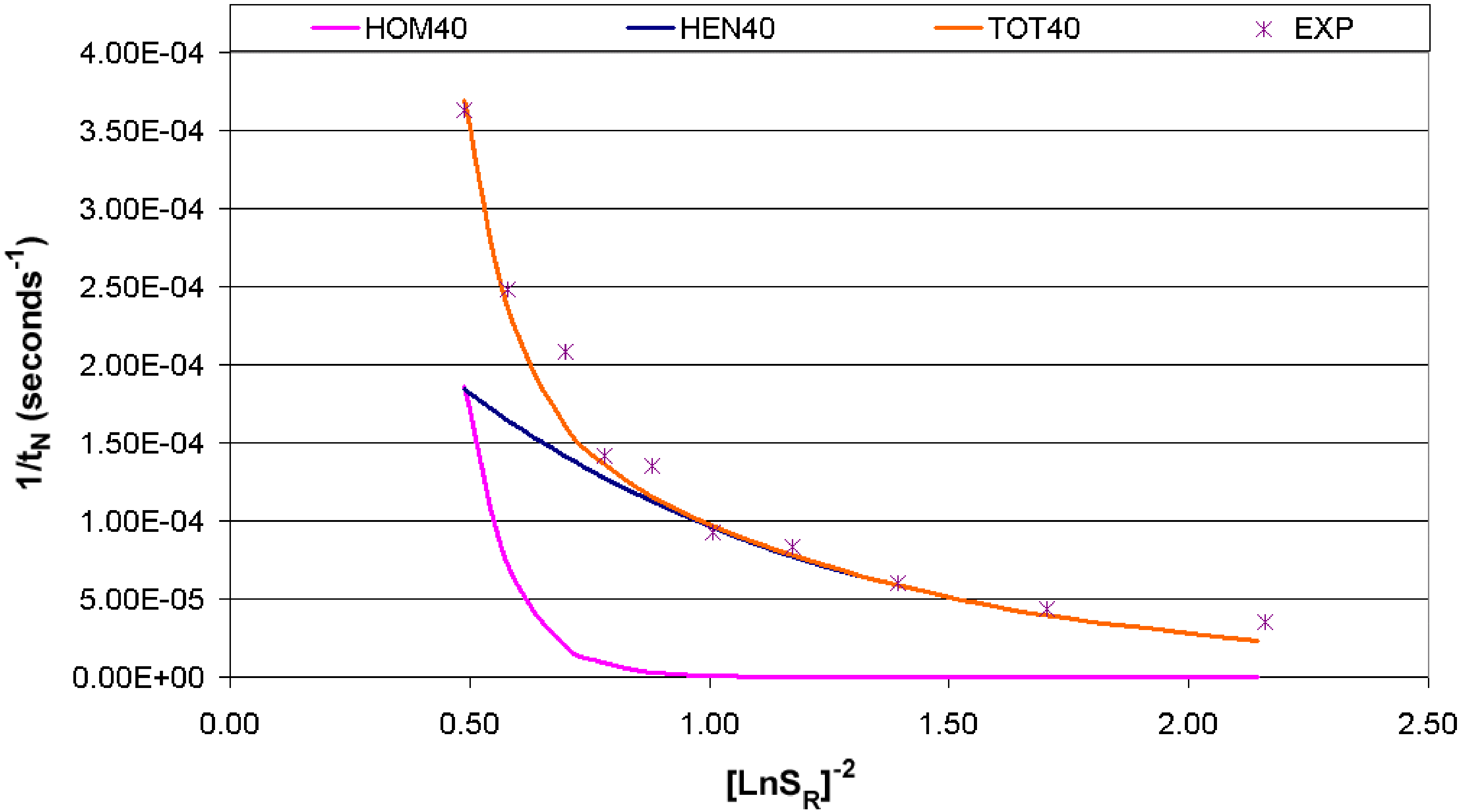
Nucleation Results 40 Degrees



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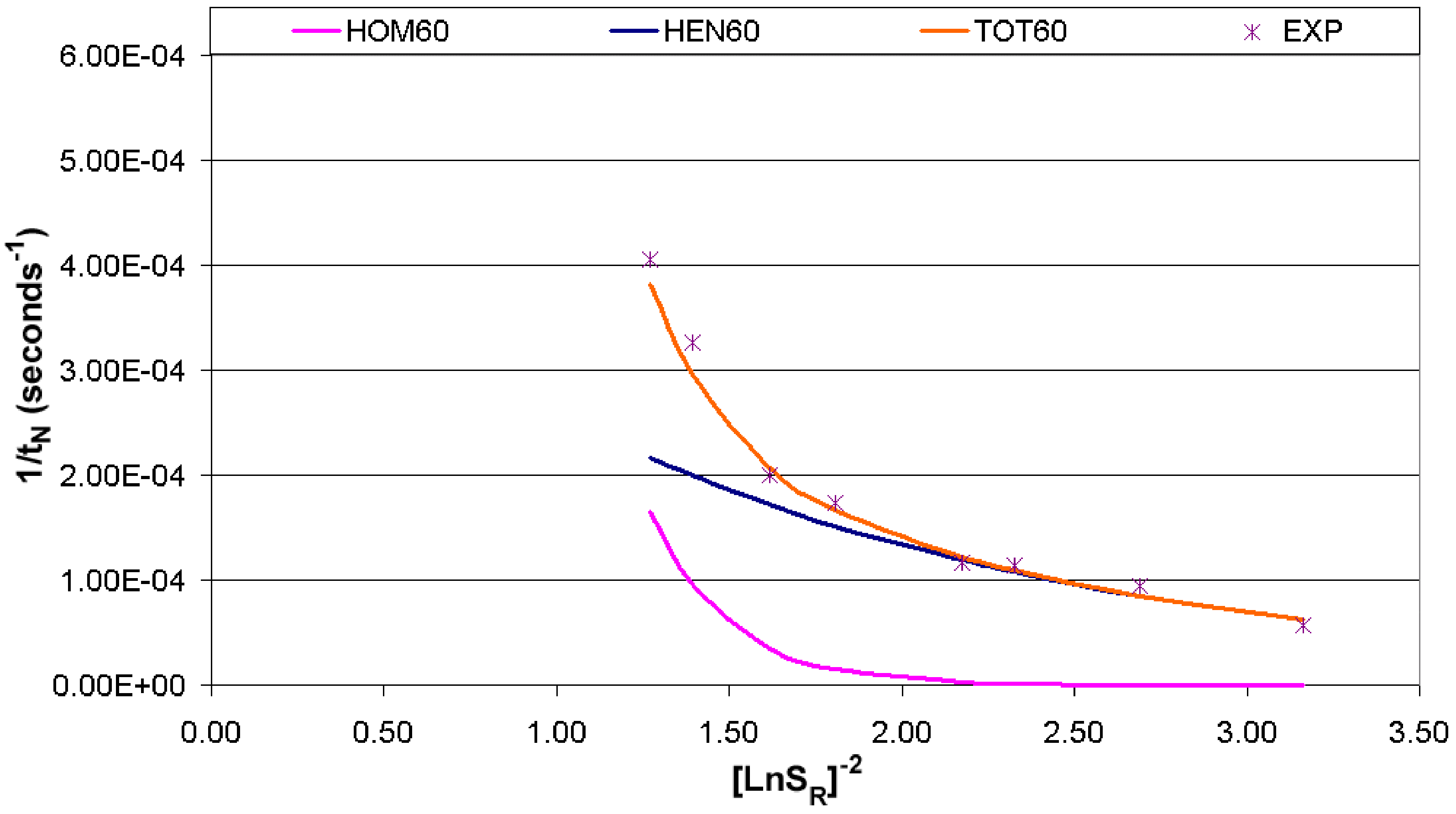
Nucleation Results 60 Degrees



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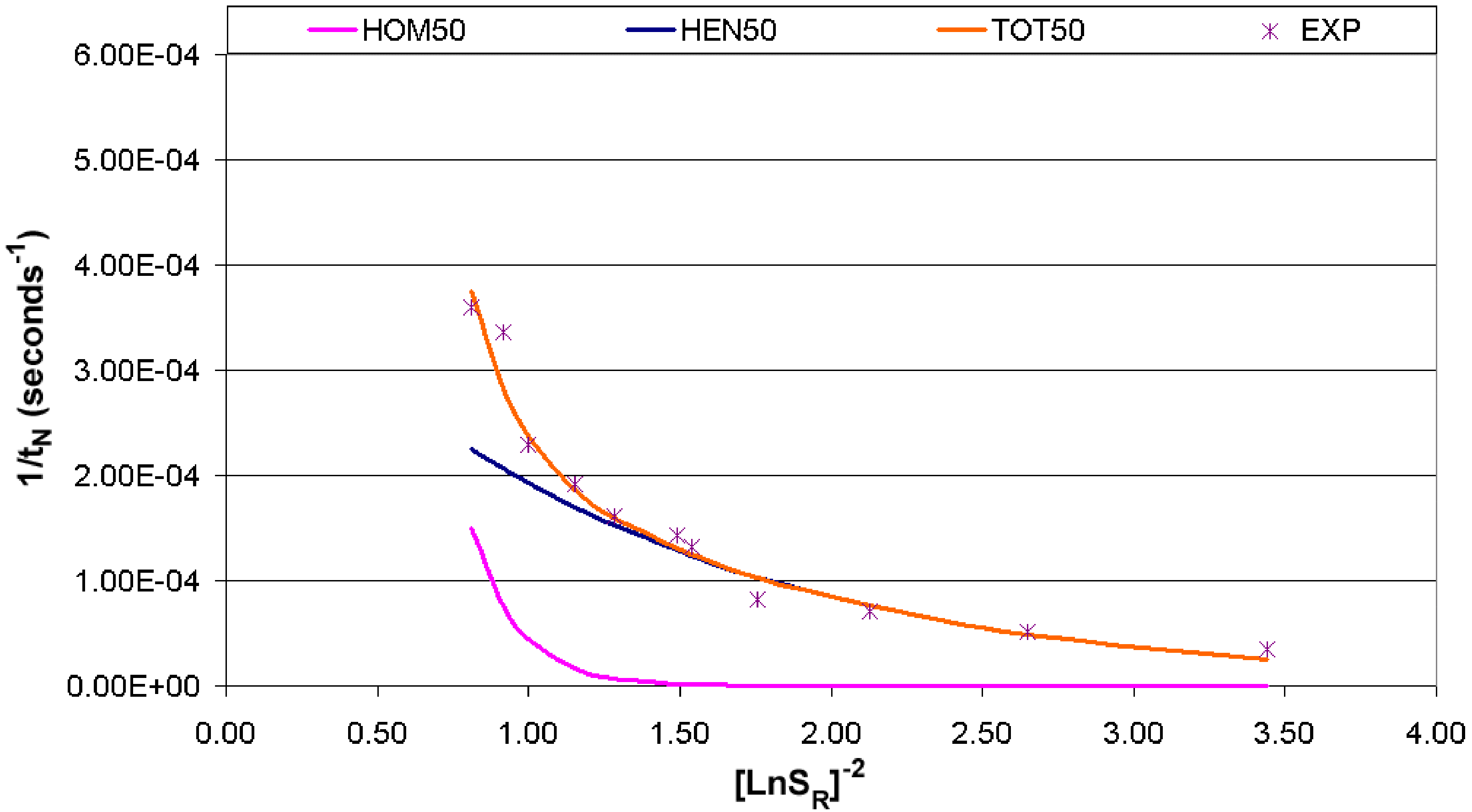
Nucleation Results 50 Degrees



Effect of temperature on the point where the homogenous nucleation begins to appear

Temperature °C	$(lnS_R)^{-2}$	**Relative Supersaturation (Cα/Cαs)**	**Absolute Supersaturation Cα-Cαs (g /100 g water)**
25	0.59	3.69	20.20
40	1.01	2.71	20.12
50	1.48	2.27	20.10
60	2.17	1.97	20.67

Conclusions

- Increasing the supersaturation increases the nucleation rate of alpha lactose monohydrate
- The effect of temperature on the nucleation rate at high supersaturations is negligible – temperature becomes more important as supersaturation is decreased
- The point at which homogenous nucleation appears as an important mechanism is independent of temperature (when viewed in absolute supersaturation).

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THANK YOU

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