

Singularities developed by the solutions of two Ostwald ripening models in reactive batch crystallizers

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1. Introduction

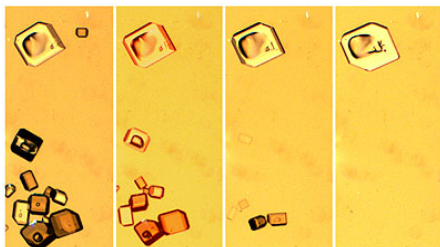
The study of crystal precipitation attracted the attention of many specialists, specially after the pioneering works by the Nobel Prize F. W. Ostwald (1853-1932) on the so called *Ostwald ripening* in reactive batch crystallizers (persistence of a single crystal size for very large values of time). Several mathematical models can be introduced to this respect. Here we shall follow the so called Tavares model [Tavares 1985], later study from the mathematical point of view in [Friedman-Ou 1989], [Friedman-Hu-Ross 1989] and [Gobi-Palpacelli-Spigler 2008].

Some other completely different models (the so-called Lifshitz-Slyozov-Wagner, ...) have been mathematically studied by several authors (see, e.g. [Boistelle-Astier 1988], [Ratke-Voorhees 2002], [Velázquez 2006] and their references).

A good illustration arises with the photographic emulsion a suspension of small particles in aqueous gelatin. A crystal is a three-dimensional atomic or molecular structure consisting of periodically repeated unit cells. The method of manufacturing silver halide grains of a given (approximate) size is based on a process called "Ostwald ripening."

Applications to traditional photography are nowadays of much less importance, in view of digital photography, but there are many other applications, e.g. to colloidal dispersions, where Ostwald ripening is an important phenomenon [Boistelle-Astier 1988].

The phenomenon of Ostwald ripening is based on the following fact: for a "very long time," either all crystal grains will dissolve into solution, or all the grains in the solution become the same size.



In this presentation we shall consider two different models. The first one (a simplified model in which the number of crystals is assumed to be prescribed for any time) which can be stated as a system of nonlinear ODEs and a more realistic model arising when the number of crystals of each size μ_j (now in number $N \geq 2$) is not prescribed but given by the "solution" of a suitable nonlinear and nonlocal hyperbolic problem. In both cases, our special interest is to search for conditions implying that the Ostwald ripening phenomenon ends completely after a finite time (and not only asymptotically when $t \rightarrow +\infty$) as in previous papers. Works in progress with R. Gómez (UCM) and with J. Jimenez (Pau, France).

2. On Tavare's model with a prescribed number of crystals.

Given a volume of fluid containing an amount of dissolved matter (*solute*), there will be in equilibrium a saturation concentration c^* , which is the maximum solute per unit volume of fluid that the system can hold. If the actual concentration $c(t)$ exceeds c^* , then the excess precipitates out in solid form, i.e., in crystal form.

Actually, to cause precipitation, $c(t)$ must be larger than a quantity c_L ($c_L > c^*$), which also depends on the size of the grain, as will be explained below.

We denote the edge of crystal grains by L (we assume that all crystal grains have the same shape and that they differ only in size. For definiteness, we assume that all the grains are cubes with variable diameter and variable orientation). We assume that the distribution of grains of edge L is roughly uniform throughout the solution. Then c_L is given by the Gibbs-Thomson relation,

$$c_L = c^* e^{\Gamma/L}$$

where Γ is a physical quantity that depends on the shape of the crystals, on its material properties, and on the temperature (which is assumed fixed).

If $c(t) > c_L$, then material will come out of the solution and deposit onto the crystals characterized by L , and if $c(t) < c_L$ then material will dissolve from the crystals. Set

$$L^*(t) = \frac{\Gamma}{\log \frac{c(t)}{c^*}}$$

According to semi-empirical law, the crystal size L will grow or dissolve at the rate

$$\frac{dL}{dt}(t) = G(L(t), c(t)) \quad (1)$$

where

$$G(L(t), c(t)) = \begin{cases} k_\gamma (c(t) - c^* e^{\Gamma/L(t)})^\gamma & \text{if } L(t) > L^*(t), \\ -k_\delta (c^* e^{\Gamma/L(t)} - c(t))^\delta & \text{if } L(t) < L^*(t), \end{cases} \quad (2)$$

where the main changes, with respect to the formulation proposed in [Friedman-Hu-Ross 1989], concerns the assumptions $\gamma > 0$ and $\delta > 0$ instead $\gamma \geq 1$ and $\delta \geq 1$.

We mention that it is well known (see, e.g. [Aris 1975]) that in many chemical reactions the kinetics leads to exponents $\gamma \in (0, 1)$ and $\delta \in (0, 1)$. As a matter of fact, the limit cases $\gamma = 0$ and $\delta = 0$ are also relevant in the applications [Aris 1975] but they must be suitably formulated in terms of multivalued functions [Díaz 1985] and we shall not discuss them here. On the rest of parameters we assume that k_γ , k_δ , Γ and c^* are given positive numbers.

Observe that

if $c(t) > c_L$ (or $L(t) > L^*(t)$) then $\frac{dL}{dt}(t) > 0$, i.e. the crystal grows,

if $c(t) < c_L$ (or $L(t) < L^*(t)$) then $\frac{dL}{dt}(t) < 0$, i.e. the crystal shrinks,

Let us assume that initially there are N different sizes of crystals, characterized by sizes $L = x_j^*$ in numbers μ_j^* per unit volume, where

$$0 < x_1^* < x_2^* < \dots < x_N^*.$$

These sizes will then evolve in time to $x_1(t) < x_2(t) < \dots < x_N(t)$, according to (1),

$$\frac{dx_j}{dt}(t) = G(x_j(t), c(t)). \quad (3)$$

The concentration $c(t)$ of the solute at time t is given by

$$c(t) := c_0 + \rho k_v \sum_{j=1}^N \mu_j^*(x_j^*)^3 - \rho k_v \sum_{j=1}^N \mu_j^*(x_j(t))^3,$$

where c_0 is the initial concentration, k_v is a geometric parameter connecting L^3 to the crystal volume (in the case of cubic crystals, $k_v = 1$), and ρ is the mass density of the solid phase.

If we substitute $c(t)$ into (3), we obtain a system of differential equations

$$\frac{dx_j}{dt}(t) = G_j(x_1(t), \dots, x_N(t)), \quad j = 1, \dots, N.$$

We also have initial conditions $x_j(0) = x_j^*$. Set

$$\mu_j := \rho k_v \mu_j^*, \quad c_1 := c_0 + \rho k_v \sum_{j=1}^N \mu_j^*(x_j^*)^3.$$

Note that c_1 represents the total amount of silver halide per unit volume in either crystal or solution form. Note also that for $N = 2$ the problem can be reformulated as the system The first one (a simplified model in which the number of crystals is assumed to be prescribed for any time) can be stated (after important simplifications) as the nonlinear ODEs system

$$\begin{cases} \frac{dx_1}{dt} + |f_1(x_1, x_2)|^{\delta-1} f_1(x_1, x_2) = 0 \\ \frac{dx_2}{dt} + |f_2(x_1, x_2)|^{\delta-1} f_2(x_1, x_2) = 0 \\ x_1(0) = x_{0,1} \\ x_2(0) = x_{0,2} \end{cases}$$

where $\delta > 0$ (here we are assuming $\gamma = \delta$ for simplicity) and $f_i(x_1, x_2) = \mu_1 x_1^3 + \mu_2 x_2^3 + e^{\Gamma/x_i} - \hat{c}$ for $i = 1, 2$ and for some positive constants μ_1, μ_2, Γ and \hat{c} , mentioned in the abstract of this lecture.

In the case $N = 1$

$$\frac{dx}{dt}(t) = G(x(t)), \quad (4)$$

$$G(x) = \begin{cases} k_\gamma (c_1 - \mu x^3 - c^* e^{\Gamma/x})^\gamma & \text{if } c_1 - \mu x^3 > c^* e^{\Gamma/x}, \\ -k_\delta (c^* e^{\Gamma/x} - (c_1 - \mu x^3))^\delta & \text{if } c_1 - \mu x^3 < c^* e^{\Gamma/x}. \end{cases} \quad (5)$$

Typical physical constants are $c^* = 4 \times 10^{-6} \text{ kmol / m}^3$, $\Gamma = 4 \times 10^{-9} \text{ m}$, $\rho = 6473 \text{ kg / m}^3$, $k_\gamma = k_\delta = 5 \times 10^{-2}$, and $c_0 = 1.05c^*$, $x^* = 10^{-7} \text{ m}$ or 10^{-8} m and $\mu = 10^{19}$ or 10^{16} .

3. Crystals of single size

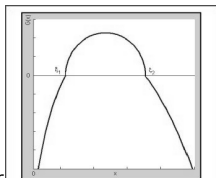
Consider the situation when all crystal grains have initially the same size x^* . The crystals will evolve according to (4), (5). The points x where G changes sign will play a crucial role. That is

$$\mu x^3 + c^* e^{\Gamma/x} = c_1 \quad (6)$$

Lemma 1. *There exist at most two positive solutions ξ_1 and ξ_2 (with $\xi_1 \leq \xi_2$) of (6).*

Proof. Function $f(x) := \mu x^3 + c^* e^{\Gamma/x}$ has a positive second derivative for $x > 0$, and $f(x) \rightarrow \infty$ if $x \rightarrow 0$ or if $x \rightarrow \infty$.

G bis



Equation (6) may have no positive solutions, but for definiteness we concentrate on the case where there are two distinct positive solutions.

Theorem 1. *i) If $x^* = \zeta_2$ then the solution of (4) is $x(t) = x^*$. If $x^* = \zeta_1$ then $x(t) = x^*$ is a solution of (4) (which, in fact, it is the only one if $\gamma \geq 1$ and $\delta \geq 1$).*

ii) If $x^ > \zeta_2$ then $x(t)$ is strictly decreasing and $\lim_{t \rightarrow \infty} x(t) = \zeta_2$ (with $x(t) = \zeta_2$ after a finite time if $\delta \in (0, 1)$).*

ii) If $x^ \in (\zeta_1, \zeta_2)$ then $x(t)$ is strictly increasing and $\lim_{t \rightarrow \infty} x(t) = \zeta_2$ (with $x(t) = \zeta_2$ after a finite time if $\gamma \in (0, 1)$).*

iii) If $x^ \in (0, \zeta_1)$ then $x(t)$ is strictly decreasing for some interval $[0, t_0]$ and then $x(t_0) = 0$ with $\lim_{t \rightarrow t_0} \frac{dx}{dt}(t) = -\infty$.*

- Remarks 1.** Note that Theorem 2 asserts that the crystal's size will decrease to zero in finite time t_0 ; thereafter the crystal is entirely dissolved in the solution (there is no differential equation once that $x(t) = 0$).
2. The proof of the existence and uniqueness (for $x^* \neq \zeta_1$) of the solution uses the decomposition of G as a locally Lipschitz function + monotone function and the different sign of $\frac{dx}{dt}(t)$ according the initial condition x^* (see, e.g. [Di-Thelin 1994] for some related results).
3. A singularity on $\frac{dx}{dt}(t)$ arises at the extinction time t_0

$$t_0 = \int_0^{x^*} \frac{ds}{G(s)}.$$

4. Crystals of several (prescribed) sizes

We consider the general case of crystals with N sizes. We assume that the initial concentration is larger than the critical concentration c^*

$$c_0 > c^*.$$

Lemma 2. We have $c(t) > c^*$ for any $t > 0$.

Proof. Indeed, if $c(t_1) = c^*$ then $\frac{dc}{dt}(t_1) \leq 0$. But, on the other hand,

$$\frac{dc}{dt}(t) = -3 \sum_{j=1}^N \mu_j(x_j(t))^2 \frac{dx_j}{dt}(t) = -3 \sum_{j=1}^N \mu_j(x_j(t))^2 G(x_j(t), c(t)) > 0, \quad (7)$$

which leads to a contradiction at $t = t_1$.

Lemma 3. We $c(t) < c_1 := c_0 + \rho k_v \sum_{j=1}^N \mu_j^*(x_j^*)^3$ for any $t > 0$. And, in particular $x_N(t) \leq \left(\frac{c_1}{\mu_N}\right)^{1/3}$ for any $t > 0$.

Proof. Use the definition of $c(t)$.

As in the case of a single-size crystal, some of the crystals may dissolve entirely in finite time, and they disappear thereafter from the differential equations

Lemma 4. *The size ordering of the solutions $x_j(t)$ continues to hold as long as the $x_j(t)$ are positive.*

Proof. We have

$$\frac{d}{dt}(x_{j+1}(t) - x_j(t)) = G(x_{j+1}(t), c(t)) - G(x_j(t), c(t)) > 0.$$

This implies that $x_{j+1}(t) - x_j(t)$ is strictly monotone increasing.

Remark. The curve $x = L^*(t) := \frac{\Gamma}{\log \frac{c(t)}{c^*}}$ determines whether a crystal grows or shrinks. If $x_j(t) > L^*(t)$ then $x_j(t)$ is growing, whereas if $x_j(t) < L^*(t)$ then $L^*(t)$ is shrinking.

We denote by k the maximal number of crystal sizes that have disappeared in finite time, say by the time $t = t^*$. Thus for $t > t^*$ there are only crystal grains with sizes $x_{k+1}(t), x_{k+2}(t), \dots, x_N(t)$ present in the differential systems (the $x_1(t), \dots, x_k(t)$, dropped out).

Theorem 2. *All crystals that do not have the largest size $x_N(t)$ will dissolve in finite time, i. e., $k + 1$ cannot be smaller than N for t^* large enough.*

Proof. We suppose that $k + 1 < N$ and derive a contradiction. A crystal of size $x_j(t)$ may possibly intersect the curve $x = L^*(t)$ several times. However, $x_N(t)$ can intersect $x = L^*(t)$ at most once. Indeed, at a point of intersection $t = t_N$, we have

$$\frac{dx_N}{dt}(t_N) = G(x_N(t_N), c(t_N)) = 0$$

whereas

$$\frac{dL^*}{dt}(t_N) < 0 \text{ since } \frac{dc}{dt}(t_N) < 0$$

as seen from (7) (the sum ranges over $k + 1 < j < N$, and each G with $k + 1 < j < N$ is negative). We conclude that for some $t_N > t^*$ either

$$x_N(t) > L^*(t) \text{ for all } t > t_N \quad (8)$$

or

$$x_N(t) < L^*(t) \text{ for all } t > t_N. \quad (9)$$

In the last case (9) $\frac{dx_N}{dt}(t_N) < 0$ for all $t > t_N$ whereas in the first case (8) $\frac{dx_N}{dt}(t_N) > 0$ for all $t > t_N$.

In both cases $\lim_{t \rightarrow \infty} x_N(t)$ exists (recall that $x_N(t)$ is bounded, by Lemma 3).

From Lemma 4, with $j + 1 = N$, it follows that $\lim_{t \rightarrow \infty} x_{N-1}(t)$ also exists, and similarly $\lim_{t \rightarrow \infty} x_j(t)$ exists for all $N > j > k + 1$.

From the definition of $c(t)$ it also follows that $\lim_{t \rightarrow \infty} c(t)$ exists. Set

$$x_j(\infty) = \lim_{t \rightarrow \infty} x_j(t) \text{ and } c(\infty) = \lim_{t \rightarrow \infty} c(t).$$

Again, by Lemma 4

$$x_{k+1}(\infty) < x_N(\infty).$$

Hence

$$G(x_{k+1}(\infty), c(\infty)) < G(x_N(\infty), c(\infty)).$$

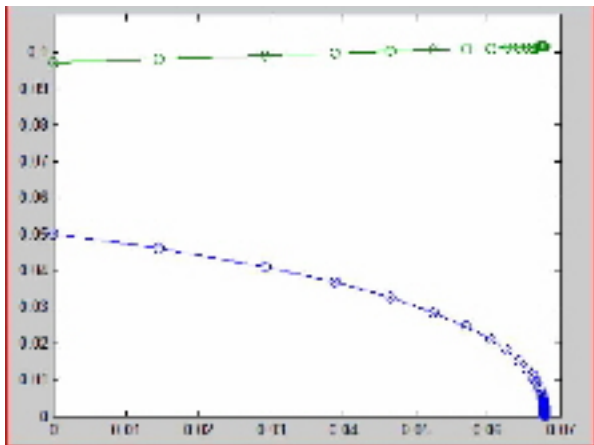
It follows that at least one of these two numbers is different from zero; say $\eta := G(x_{k+1}(\infty), c(\infty))$. Since for very large t

$$\frac{dx_{k+1}}{dt}(t_N) = G(x_{k+1}(t), c(t)) \sim \eta,$$

we deduce that $\lim_{t \rightarrow \infty} x_{k+1}(t)$ does not exist, which is a contradiction.

The contradiction can be avoided only if $k + 1 = N$, and thus Theorem 2 follows.

As conclusion, we now know that $x_1(t)$ dissolves in time τ_1 , $x_2(t)$ dissolves in time τ_2 , ..., $x_{N-1}(t)$ dissolves in time τ_{N-1} . For $t > \tau_{N-1}$ we are back into the one-size crystals situation studied before. Moreover $x_N(t)$ attains its asymptotic value in a finite time if $\delta \in (0, 1)$ or $\gamma \in (0, 1)$ according if $\frac{dx_N}{dt}(t)$ is negative or positive after τ_{N-1} . We have concluded that after a finite time $t = \tau_{N-1}$ all but the largest size crystals will have dissolved in the solution. Thereafter, the remaining crystals, having uniform size, will either (i) shrink to zero size (and hence dissolve) in finite time; or (ii) converge to one of two sizes $\tilde{\zeta}_1$ and $x^* = \tilde{\zeta}_2$, depending on the conditions existing at time $t = \tau_{N-1}$. Numerical methods allow us to compute the limiting size of the crystals.



5. On the case of non prescribed sizes crystals.

A more realistic model arises when the number of crystals of each size μ_j (now in number $N \geq 2$) is not prescribed but given by $n(x, t)$ the "solution" of the nonlinear and nonlocal hyperbolic problem

$$\begin{cases} \frac{\partial n}{\partial t} + \frac{\partial}{\partial x}(Gn) = 0 & x > 0, t > 0, \\ n(x, 0) = n_0(x) & x > 0, \end{cases} \quad (10)$$

where

$$G(x, t) = \begin{cases} k_\gamma (c(t) - c^* e^{\Gamma/x})^\gamma & \text{if } x > x^*(t), \\ -k_\delta (c^* e^{\Gamma/x} - c(t))^\delta & \text{if } x < x^*(t), \end{cases} \quad (11)$$

$$x^*(t) = \frac{\Gamma}{\log(c(t)/c^*)} \text{ and } c(t) = c_0 + \beta \int_0^{+\infty} x^3 n_0(x) dx - \beta \int_0^{+\infty} x^3 n(x, t) dx \quad (12)$$

The natural modelling of the problem leads to the assumption $n_0(x) = \sum_{m=1}^N \mu_m \delta(x - x_{m,0})$, where μ_m are some given positive constants, $\delta(x)$ denotes the Dirac measure with unit mass at $x = 0$ and the values $0 < x_{1,0} < x_{2,0} < \dots < x_{N,0} < \infty$ are N given positive numbers representing the sizes of the initial crystals.

Besides its relevance in the applications, which made specially interesting such a problem is that the corresponding solution is not a L^1 -valued function but a measure-valued function $n(\cdot, t)$. We shall prove that, under suitable additional conditions on G (for instance $\gamma, \delta \in (0, 1)$) the Ostwald ripening phenomenon takes place not only asymptotically (when $t \rightarrow +\infty$) but in a finite time.

As mentioned, our approach follows closely the pioneering paper [Friedman-Hu-Ross 1989]. Their arguments can be easily extended to prove the existence and uniqueness of a solution of problem (10), (11), (12), for such initial datum.

Theorem 3. *Assume $\gamma > 0$ and $\delta > 0$. Then, given such a $n_0(x)$, there exists a unique entropy solution $n(x, t)$ of problem (10), (11), (12), $n \in C([0, +\infty) : \mathcal{M}(0, +\infty))$. More precisely, we have the*

representation formula

$$n(x, t) = \sum_{m=1}^N \mu_m \delta(x - x_m(t)) \quad (13)$$

for some functions $x_m(t)$ satisfying that $x_m(0) = x_{m,0}$.

Idea of the proof. It is natural to start by approximating the initial datum by

$$n_{0,j}(x) = \sum_{m=1}^N \mu_m \rho_j(x - x_{m,0})$$

where $\rho_j(x)$ is a smooth function such that

$$\rho_j \geq 0, \quad \rho_j(x) = 0 \text{ if } |x| > \frac{1}{j} \text{ and } \int_{-\infty}^{+\infty} \rho_j(x) dx = 1.$$

The existence of a solution $(n_j(x, t), c_j(t))$ for this class of initial datum is an easy modification of the arguments of [Friedman-Ou 1989] since the *local existence* is built through the solution of the ordinary differential equation

$$\begin{cases} \frac{dx}{dt} = G_j(x, t) & t > 0, \\ x(0) = x_0. \end{cases}$$

It is clear that, in general, function G is not globally Lipschitz continuous but it is locally Lipschitz continuous and monotone near the singular points. So, by well known results (see, e.g. [Di-Thelin 1994]) we know the existence of a global solution $x(t)$. Then

$$\frac{d}{dt}n_j(x_j(t), t) = -\frac{\partial G_j(x_j(t), t)}{\partial x}n_j(x_j(t), t)$$

and thus

$$n_j(x_j(t), t) = n_{0,j}(x) e^{-\int_0^t \frac{\partial G_j(x_j(s), s)}{\partial x} ds}.$$

Then we get that

$$c_j(t) = c_{1,j} - \beta \int_0^{+\infty} x^3 n_j(x, t) dx,$$

$$\frac{dc_j(t)}{dt} = -3\beta \int_0^{+\infty} x^3 n_j(x, t) \widehat{G}_j(x, t) dx,$$

$$\widehat{G}_j(x, t) = \begin{cases} k_\gamma (c_j(t) - c^* e^{\Gamma/x})^\gamma & \text{if } x > x_j^*(t), \\ -k_\delta (c^* e^{\Gamma/x} - c_j(t))^\delta & \text{if } x < x_j^*(t), \end{cases}$$

with $x_j^*(t) = \frac{\Gamma}{\log(c_j(t)/c^*)}$. If we denote by $x_j(t) \equiv x_j(t; x)$ the solution of

$$\begin{cases} \frac{dx_j}{dt} = \widehat{G}_j(x, t) & t > 0, \\ x_j(0) = x_0, \end{cases} \quad (14)$$

we obtain that $x_j(t)$ is well defined (even if $\gamma \in (0, 1)$ and/or $\delta \in (0, 1)$) and that

$$\widehat{G}_j(x, t) \leq C, \quad \frac{dx_j}{dt} \leq C \quad \text{and} \quad \int_0^{+\infty} n_j(x, t) dx \leq \int_0^{+\infty} n_{0,j}(x) dx \leq C,$$

for a suitable positive constant C . Then we can extract a subsequence $c_j(t)$, $x_j(t)$ and $n_j(x, t)$ which are pointwise convergent to the searched solution satisfying the representation formula (13). Once more, the uniqueness of such a solution is consequence of the uniqueness of solution of the Cauchy problem obtained passing to the limit in (14).

Remark. It is possible to prove (something which is not analyzed in [Friedman-Hu-Ross 1989]) that the obtained solution $n(x, t)$ satisfies the hyperbolic equation in the weak entropy sense, $n \in C([0, +\infty) : \mathcal{M}(0, +\infty))$ as introduced in [DiPerna 1985] (see also, e.g., the exposition made in [Malet et al 1996]).

Thanks to an extra assumption, we can prove that the Ostwald ripening phenomenon (persistence of a single crystal size for very large values of time) takes place not only asymptotically (when $t \rightarrow +\infty$) but in a finite time.

Theorem 2. *Assume that*







$$\gamma \in (0, 1) \text{ and } \delta \in (0, 1). \quad (15)$$







Then there exists $\hat{t} > 0$ such that $n(x, t) = \mu_N \delta(x - x_N(t))$ for any $t > \hat{t}$. Moreover, $n(x, t) \rightarrow \mu_N \delta(x - \zeta_2)$ as $t \rightarrow +\infty$, where ζ_2 is one of the zeros of the transcendent equation

$$\beta \mu_N \zeta^3 + c^* e^{\Gamma/\zeta} = c_0 + \beta \sum_{m=1}^N \mu_m x_m^3 \equiv c_1.$$

Idea of the proof. We use the arguments of the precedent section to show that the solutions of the limit problem associated to (14) vanishes in a finite time (except for the last size x_N), thanks to the condition (15). The asymptotic behaviour follows similar arguments to the ones introduced in [Friedman-Hu-Ross 1989].

THANKS FOR YOUR ATTENTION

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