Mathematical Aspects of Electroinitiated Free Radical Polymerization

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ABSTRACT

With few simplifying but reasonable assumptions, some mathematical aspects of electroinitiated polymerization has been discussed in this research. The mathematical relationship between electrolysis current and the initial rate of free radical polymerization was derived. In the case of free radical polymerization, the initial rate was proportional to the half power of electrolysis current. The relationship between electrolysis current and the average degree of polymerization was also derived. Here, the mathematical relationship between electrolysis current and kinetic chain length for direct current control of a polymerizing system was also deduced.

Keywords: Mathematical derivation, Electroinitiated polymerization, Free radical electrosynthesis.

INTRODUCTION

Electroinitiated polymerization has several distinct advantages over traditional chemical polymerization and it can be controlled at will. By driving the oxidation of monomers via electrical potential or current rather than chemical reagants, this process also controls the polymer film's properties and spatial orientation [1]. This method is also widely recognized for synthesis of polymers by applying electric potential or current to a solution containing monomers. The process allows for precise control of film thickness, morphology and porosity by adjusting electrochemical parameters like potential, current density and polymerizing time. The synthesis of polymers is controlled by the applied potential, leading to cleaner synthesis, with fewer hazardous products, working with the principles of green chemistry approach for functional polymers[2-7]. No excess amounts of chemical oxidants such as ammonium persulphate or ferric chloride are used which leave behind ionic impurities or unreacted residues that degrade polymer performance.

The synthesizing or functional coatings on electrodes in electroinitiated polymerization, mathematical and kinetic derivation offers critical advantages across fundamental research, process engineering and application design. The mathematical aspects or kinetics reveals whether the overall polymerization rate is limited by charge transfer, monomer diffusion to the electrode or chemical coupling i.e., dimerization. These derivation provide explicit functional relationship between experimental inputs like potential, current density, monomer concentration, solvent viscosity, etc and output material properties like

(a) Film thickness and growth rate

(b) Molar mass distribution and chain length

(c) Morphology and nucleation control

To understand the mathematical analysis or kinetic derivation of EIP, we must consider the various reaction steps, their nature, rate and temporal sequence. This task is conveniently handled by discussing limiting cases which are summarized below

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Limiting EIP Schemes

a. Electrode transfer with electrode

i. Monomer

ii. Initiator

iii. Catalyst

b. Chemical Reaction

i. Dimerization of radical ions

ii. Addition of Initiator to Monomer

iii. Addition of Monomer to Growing Chain

iv. Electron transfer between Catalyst and Monomer

c. Sorption at Electrode Surface

i. Adsorption of Electroactive Species

ii. Desorption of non-electroactive species

iii. Desorption of Electrochemical products – activated monomer, Initiator, catalyst or polymers (dead or alive)

d. Diffusion near Electrode

i. Electroactive species to Electrode

ii. Dead Polymer from Electrode

iii. Counter-diffusion in reaction layer

iv. Surface-diffusion to active sites on electrode.

The overall reaction may be separated into (a) two electrodes reactions and (b) bulk solution reactions. In order to characterize the electrode kinetics, it is necessary to obtain experimental relationships between (a) the independent variables like current density, electrode potential, concentration of reactants, temperature, cell geometry etc. and (b) the dependent variables like rate of polymer formation and reactant consumptions, degree of polymerization, molecular weight distribution, etc. In addition, the following quantities may be determined like electron transfer co-efficient, reaction order, limiting diffusion, sorption, reaction currents, stoichiometric factor and overpotentials [8]. Here we shall confine ourself to analyze those aspects of kinetics which are more typical of EIP, such as electron transfer steps involved in the initiation of polymerization and the influences of chemical reactions on the electrode processes.

RESULTS AND DISCUSSION

We shall assume that monomer molecules and electrons utilized to produce polymer and all monomer reacted becomes part of the ultimate polymer produced as well as all electrons transferred between electrode and solution initiate at least one polymer chain. The rate of monomer consumption is therefore:

$$-\frac{d[M]}{dt} = \frac{d[P]}{dt} = \frac{i}{FV} + K_p[M][P^*] + K_t[M][P^*] + K_i[M][N] \quad \ldots \quad \ldots \quad \ldots(1)$$

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Where,

i = constant current in Ampere

V = Volume of the electrolyte

If the current is a function of time, then

$$i = \int i_t \, dt,$$

K_t = termination constant,

K_i = initiation constant,

[M] = monomer concentration,

[I] = initiator concentration,

And

P* = active polymer – growing site.

It is likely that all the terms in equation (I) are involved in many EIP systems under certain conditions. However, it is reasonable and desirable to expect that EIP conditions may be regulated for any particular system so as to eliminate one or more of the terms in equation (I).

The rate of chain growth is

$$R_p = K_p [M] [P^*] \quad \ldots \quad \ldots \quad \ldots \quad (2)$$

and the rates of initiation, R_i and termination, R_t are

$$R_t = K_i [M][I] + \frac{i}{FV} \quad \ldots \quad \ldots \quad \ldots \quad (3)$$

$$R_t = K_{tc} [P^*]^2 + K_{td} [P^*]^2 + K_{trm} [M] [P^*] + K_{trs} [S] [P^*] \quad \ldots \quad \ldots \quad (4)$$

Where,

K_{tc} = termination constant for combination

K_{td} = termination constant for disproportionation,

K_{ktrm} = transfer constant to monomer,

and K_{trs} = transfer constant to solvent

The molecular weight distributions obtained in EIP under conditions of ideal mixing i.e., no concentration gradients are similar to those of non-EIP processes in which the initiation rate is of the same form.

According to steady-state assumption the total concentration of all active sites [P*] remains constant during essentially the entire duration of polymerization, so that

$R_i = R_t$ … … … [5]

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Degree of polymerization, $DP = \frac{R_p}{R_t + R_{tr}} = \frac{R_p}{R_i}$ … … … [6]

The values of [M], [P*], and [I] in the latter equations for a particular EIP system depends on:

a) The types of initiation, termination, and transfer mechanism.

b) The relative speeds of the various kinetic steps in the EIP process.

c) The duration of polymerization and

d) The distribution of reacting species through the reaction species of the system. The latter is determined by the efficiency of mixing and the ease of diffusional processes in the reaction space.

In the analyses of EIP processes, it is assumed that the reaction rate constants are independent of polymer size and that the concentrations are uniform throughout the polymerization volume. Hence, diffusion is rapid and mixing is efficient with respect to the reaction rates of all the various mechanistic steps involved in the EIP.

Free Radical Electroinitiated Polymerization

To illustrate the analysis and behaviour of EIC processes, we shall discuss the case of free radical polymerization mechanism in which termination is second order, combination and / or disproportionation, and initiation is the result of a non-catalytic additive generated electrochemically and no chain transfer occurs.

For any combination of current-time in this system, one has:

$R_i = K_i [M] [I^\bullet]$ … … … [7]

$R_p = K_p [M] [P^\bullet]$ … … … [8]

$R_t = K_{tc} [P^\bullet]^2$ … … … [9]

and the concentrations are related by

$$-\frac{d[M]}{dt} = K_i[M][I^\bullet] + K_p[M][P^\bullet] \quad \ldots \quad \ldots \quad \ldots \quad [10]$$

$$\frac{d[I^\bullet]}{dt} = \frac{i}{FV} - K_i[M][I^\bullet] \quad \ldots \quad \ldots \quad \ldots \quad [11]$$

And

$$\frac{d[P^\bullet]}{dt} = K_i[M][I^\bullet] - K_{tc}[P^\bullet]^2 \quad \ldots \quad \ldots \quad \ldots \quad [12]$$

There relations are not much different from those of a similar homogeneous chemically initiated polymerization [9], the notable exception being the electrolytic current term in equation1). In equation (11), it has been assumed that only one electron is required to activate the non-catalytic initiator, I, to the free radical species, $I^\bullet$. For example, this is true in the case of electroinitiated oxidation of carboxylate anions in the Kolbe's synthesis of free radicals.

$$R - CO_2^- \rightarrow e^-(anode) + RCO_2^\bullet \quad \ldots \quad \ldots \quad \ldots \quad [13]$$

$$R - CO_2^{\bullet} \rightarrow R^{\bullet} + CO_2 \quad \ldots \quad \ldots \quad \ldots \quad [14]$$

$$R^{\bullet} + M \rightarrow P^{\bullet} \quad \ldots \quad \ldots \quad \ldots \quad [15]$$

For the particular instance of constant current and steady state equation (11) gives,

$$[I^{\bullet}] = \frac{i}{K_i FV[M]} \quad \ldots \quad \ldots \quad \ldots \quad [16]$$

And from equations (5), (7), (9) and (16)

$$[P^{\bullet}]_{ss}^2 = \frac{K_i}{K_{tc}}[M][I^{\bullet}] = \frac{K_i M}{K_{tc}} \frac{i}{K_i FV[M]} = \frac{i}{K_{tc} FV}$$

Therefore

$$[P^{\bullet}]_{ss} = \left(\frac{i}{K_{tc} FV}\right)^{1/2} \quad \ldots \quad \ldots \quad \ldots \quad [17]$$

Equation (10) implies:

$$-\frac{d[M]}{dt} = \frac{i}{FV} + K_p[M]\left(\frac{i}{K_{tc} FV}\right)^{\frac{1}{2}} \quad \ldots \quad \ldots \quad \ldots \quad [18]$$

Or, $$\frac{d[M]}{\frac{i}{FV} + K_p[M]\left(\frac{i}{K_{tc} FV}\right)^{\frac{1}{2}}} = -dt$$

On integration

$$\left[\frac{l\,n\left\{\frac{i}{FV} + Kp[M]\left(\frac{i}{K_{tc} FV}\right)^{\frac{1}{2}}\right\}}{K_p\left(\frac{i}{KtcFV}\right)^{\frac{1}{2}}}\right]_{M_0}^{M} = [-t]_0^t$$

Or $$l\,n\left[\frac{i}{FV} + Kp[M]\left(\frac{i}{K_{tc} FV}\right)^{\frac{1}{2}}\right]_{M_0}^{M} = -Kp\left(\frac{i}{K_{tc} FV}\right)^{\frac{1}{2}}$$

Or $$l\,n\left[\frac{i}{FV} + Kp[M]\left(\frac{i}{K_{tc} FV}\right)^{\frac{1}{2}}\right]_{M_0}^{M} = -l\,n\left[\frac{i}{FV} + Kp[M]_0\left(\frac{i}{K_{tc} FV}\right)^{\frac{1}{2}}\right] = Kp\left(\frac{i}{K_{tc} FV}\right)^{\frac{1}{2}} t$$

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Or $$l\,n\left[\frac{i}{FV}+Kp[M]\left(\frac{i}{K_{tc}FV}\right)^{\frac{1}{2}}\right]=-l\,n\left[\frac{i}{FV}+Kp[M]_0\left(\frac{i}{K_{tc}FV}\right)^{\frac{1}{2}}\right]=Kp\left(\frac{i}{K_{tc}FV}\right)^{\frac{1}{2}}t$$

Or

$$l\,n\frac{\frac{i}{FV}+Kp[M]\left(\frac{i}{K_{tc}FV}\right)^{\frac{1}{2}}}{\frac{i}{FV}+Kp[M]_0\left(\frac{i}{K_{tc}FV}\right)^{\frac{1}{2}}}=-Kp\left(\frac{i}{K_{tc}FV}\right)^{\frac{1}{2}}t$$

Or $$\frac{\frac{i}{FV}+Kp[M]\left(\frac{i}{K_{tc}FV}\right)^{\frac{1}{2}}}{\frac{i}{FV}+Kp[M]_0\left(\frac{i}{K_{tc}FV}\right)^{\frac{1}{2}}}=\exp\left\{-Kp\left(\frac{i}{K_{tc}FV}\right)^{\frac{1}{2}}t\right\}$$

Or $$\frac{i}{FV}+Kp[M]\left(\frac{i}{K_{tc}FV}\right)^{\frac{1}{2}}=\left\{\frac{i}{FV}+Kp[M]_0\left(\frac{i}{K_{tc}FV}\right)^{\frac{1}{2}}\right\}\bullet\exp\left\{-Kp\left(\frac{i}{K_{tc}FV}\right)^{\frac{1}{2}}t\right\}$$

Or

$$Kp[M]\left(\frac{i}{K_{tc}FV}\right)^{\frac{1}{2}}=\left\{\frac{i}{FV}+Kp[M]_0\left(\frac{i}{K_{tc}FV}\right)^{\frac{1}{2}}\right\}\bullet\exp\left\{-Kp\left(\frac{i}{K_{tc}FV}\right)^{\frac{1}{2}}t\right\}-\frac{i}{FV}$$

Or

$$[M]=\left\{\frac{\frac{i}{FV}}{Kp\left(\frac{i}{K_{tc}FV}\right)^{\frac{1}{2}}}+\frac{Kp[M]_0\left(\frac{i}{KtcFV}\right)^{\frac{1}{2}}}{Kp\left(\frac{i}{KtcFV}\right)^{\frac{1}{2}}}\right\}$$

$$\exp\left\{-Kp\left(\frac{i}{K_{tc}FV}\right)^{\frac{1}{2}}\right\}-\frac{\frac{i}{FV}}{Kp\left(\frac{i}{KtcFV}\right)^{\frac{1}{2}}}$$

Or $$[M]=\left\{[M]_0+\frac{Ktc}{Kp}\left(\frac{i}{FV}\right)^{\frac{1}{2}}\right\}\exp\left\{-\frac{Kp}{Ktc^{\frac{1}{2}}}\left(\frac{i}{FV}\right)^{\frac{1}{2}}t\right\}-\frac{Ktc}{Kp}\left(\frac{i}{FV}\right)^{\frac{1}{2}}\quad\ldots(19)$$

If $$\frac{Ktc^{\frac{1}{2}}}{Kp}=K\ and\ \phi=\frac{i}{FV}$$

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Then

$$[M]=\left\{[M]_0+K\phi^{\frac{1}{2}}\right\}\exp\left\{-\frac{\phi^{\frac{1}{2}}}{K}t\right\}=K\phi^{\frac{1}{2}} \quad \ldots \quad \ldots \quad \ldots(20)$$

This result may be simplified further whenever high molecular weight polymers are formed. In this instance, monomer consumed by initiator is negligible, and equation (10) becomes:

$$-\frac{[dM]}{dt}=Kp[M][P^{\bullet}] \quad \ldots \quad \ldots \quad \ldots \quad \ldots \quad (21)$$

Or

$$-\frac{[dM]}{dt}=Kp[M]\left(\frac{i}{KtcFV}\right)^{\frac{1}{2}} \quad \ldots \quad \ldots \quad \ldots \quad (22)$$

Therefore, rate of polymerization $\alpha i^{\frac{1}{2}}$

The rate of EIP of methyl methacrylate has been found experimentally to be proportional to half power of current [10]

From equation (22)

$$-\frac{[dM]}{Kp[M]\left(\frac{i}{KtcFV}\right)^{\frac{1}{2}}}=dt$$

Which on integration gives

$$\left[\frac{LnKp[M]\left(\frac{i}{KtcFV}\right)^{\frac{1}{2}}}{Kp\left(\frac{i}{KtcFV}\right)^{\frac{1}{2}}}\right]_{M_0}^{M}=\left[-t\right]_0^t$$

Or

$$\left[LnKp[M]\left(\frac{i}{KtcFV}\right)^{\frac{1}{2}}\right]_{M_0}^{M}=Kp\left(\frac{i}{KtcFV}\right)^{\frac{1}{2}}$$

Or, $$LnKp[M]\left(\frac{i}{KtcFV}\right)^{\frac{1}{2}}-l\,\mathrm{n}\,Kp[M]_0\left(\frac{i}{KtcFV}\right)^{\frac{1}{2}}$$

$$=-Kp[M]_0\left(\frac{i}{KtcFV}\right)^{\frac{1}{2}}t$$

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Or $$Ln\frac{Kp[M]\left(\frac{i}{KtcFV}\right)^{\frac{1}{2}}}{Kp[M]_0\left(\frac{i}{KtcFV}\right)^{\frac{1}{2}}}=-Kp\left(\frac{i}{KtcFV}\right)^{\frac{1}{2}}t$$

Or $$l\,n\frac{[M]}{[M]_0}=\frac{Kp}{Ktc^{\frac{1}{2}}}\left(\frac{i}{FV}\right)^{\frac{1}{2}}t \qquad \ldots \quad \ldots \quad \ldots \quad (23)$$

Then the ratio $\frac{Kp}{Ktc^{\frac{1}{2}}}$ is the slope of a plot of $l\,n\frac{[M]}{[M]_0}$ *versus* $\left(\frac{i}{FV}\right)^{\frac{1}{2}}$ at constant current. At low conversation, $[M]=[M]_0=$constant, the average degree of polymerization is then:

$$\overline{DP}=\frac{Rp}{Ri}=\frac{Kp[M]_0}{Ktc^{\frac{1}{2}}\left(\frac{i}{FV}\right)^{\frac{1}{2}}} \qquad \ldots \quad \ldots \quad \ldots \quad (24)$$

$\frac{Kp}{Ktc^{\frac{1}{2}}}$ may be obtained by several experiments at different constant currents. Hence two methods may be followed to evaluate $\frac{Kp}{Ktc^{\frac{1}{2}}}$ from the same experimental data.

Since in practice, it is usually a simple matter to limit conversions to low values and still obtain useful data, there is no advantage to consider the more complicated higher conversion case. However, it is interesting to note that at constant electrolysis current the initiation rate in constant even though initiation is not a direct result of the electrode process.

$$Ri=Ki[M]\left(\frac{i}{Ki[M]FV}\right)=\frac{i}{FV} \qquad \ldots \quad \ldots \quad \ldots. \quad (25)$$

Consequently, steady-state, once established may maintained regardless of [M] and as long as the termination rate remains constant, as it is often true for certains chemically initiated polymerizations. For example, whenever the rate of decomposition of the initiator into free radicals is constant and the initiator concentration remains essentially constant. However, these conditions are uncommon for most chemically initiated polymerizations so that EIP occupies special position in this regard.

At significant conversions, [M] departs from $[M]_0$ and the average DP at the end of polymerization time is obtained by averaging equation (24) over γ :

$$\overline{\overline{DP}}=\frac{Kp}{Ktc^{\frac{1}{2}}\left(\frac{i}{FV}\right)^{\frac{1}{2}}\int_0^{\gamma}[M]\,d\tau}$$

And using equation (23),

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$$\overline{\overline{DP}} = \frac{Kp}{\tau Ktc^{\frac{1}{2}}\left(\frac{i}{FV}\right)^{\frac{1}{2}}}\int_0^{\gamma}[M]_0 \exp\left\{-\frac{Kp}{Ktc^{\frac{1}{2}}}\left(\frac{i}{FV}\right)^{\frac{1}{2}}\tau\right\}d\tau$$

or $$\overline{\overline{DP}} = \frac{Kp[M]_0}{\tau Ktc^{\frac{1}{2}}\left(\frac{i}{FV}\right)^{\frac{1}{2}}}\left\{\frac{\exp\left[-\frac{Kp}{Ktc^{\frac{1}{2}}}\left(\frac{i}{FV}\right)^{\frac{1}{2}}\tau\right]}{\frac{Kp}{Ktc^{\frac{1}{2}}}\left(\frac{i}{FV}\right)^{\frac{1}{2}}}\right\}_0^{\tau}$$

or $$\overline{\overline{DP}} = \frac{[M]_0}{\tau\left(\frac{i}{FV}\right)}\left[1-\exp\left\{-\frac{Kp}{Ktc^{\frac{1}{2}}}\left(\frac{i}{FV}\right)^{\frac{1}{2}}\tau\right\}\right] \quad \ldots \quad \ldots. \quad (27)$$

To determine at what time the average DP is largest, let

$$K = \frac{Kp}{Ktc^{\frac{1}{2}}}\left(\frac{i}{FV}\right)^{\frac{1}{2}} \text{ then}$$

or $$\overline{\overline{DP}} = \frac{[M]_0}{\left(\frac{i}{FV}\right)\frac{1}{\tau}}\left[1-\exp\left(-K\tau\right)\right]$$

or $$= \frac{\left(\frac{i}{FV}\right)\overline{\overline{DP}}}{[M]_0}\frac{1}{\tau}\left[1-\exp(-K\tau)\right] = \frac{1}{\tau}-\frac{1}{\tau}\exp(-K\tau)$$

and $$= \frac{\left(\frac{i}{FV}\right)}{[M]_0}\frac{\overline{\overline{DP}}}{dt} = 0 = \frac{1}{\tau^2}+\frac{1}{\tau^2}1-\exp(-K\tau)+\frac{K}{\tau}\exp(-K\tau)$$

or $$-1+e^{-K\tau}+\tau Ke^{-k\tau} = 0$$

or $$-\left(K\tau+1\right)e^{-K\tau} = 1$$

or $$e^{-K\tau} = K\tau+1$$

or $$1+K\tau+\frac{(K\tau)^2}{L^2}.....-K\tau+1 = 0$$

or $$\frac{(K\tau)^2}{L^2}..... = 0$$

hence, $\overline{\overline{DP}}$ is maximum when $\tau = 0$ and has the value:

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$$\left(\overline{\overline{DP}}\right)\max = \frac{M_0}{\frac{i}{FV}} \lim_{\tau \to 0} \frac{1}{\tau}\left[1 - e^{-K\tau}\right]$$

$$= \frac{[M]_0}{\left(\frac{i}{FV}\right)} K = \frac{[M]_0 Kp}{Ktc^{\frac{1}{2}}\left(\frac{i}{FV}\right)^{\frac{1}{2}}} \quad \ldots \quad \ldots \quad 28$$

Which is the same result as equation (24), as one would intuitively expect. However, polymers made before steady-state is obtained are larger than this value because the rate of termination is slower.

As shown by equation (27) $\overline{\overline{DP}}$ has a maximum value at $\tau = 0$, and it continues to decrease with time until ultimately it is zero, because it is physically impossible to maintain constant current beyond the point of total monomer and / or initiator consumption, this limit being realized when

$$t_{\max} = \frac{FV[M]_0}{i} \quad \ldots \quad \ldots \quad \ldots \quad [29]$$

Therefore, the DP never becomes lower than:

$$\left(\overline{\overline{DP}}\right)_{\min} = 1 - \exp\left\{-\frac{Kp[M]_0}{Ktc^{\frac{1}{2}}\left(\frac{i}{FV}\right)^{\frac{1}{2}}}\right\} \quad \ldots \quad \ldots \quad [30]$$

Equation (3) states that the average DP at full conversion is highest for high $[M]_0$ and low i, as one would intuitively expect.

The practical limit of conversion is determined by the initiator concentration since the electrode potential required to maintain constant current is related to [I] and has an upper limit corresponding to the decomposition potential of other species in the system.

The above results are analogous to those of free radical polymerizations initiated chemically or photochemically. However, the outstanding features of EIP are

a) The initiation rate may be varied, at will, simply by controlling the applied electrolysis current.

b) The initiation may be achieved by different initiators and mechanism than those of photopolymerization, thereby making it possible to determine the absolute reaction rate constants of polymerizations previously not possible by the photo methods.

The above mathematical derivations are just a theoretical concept based on which the kinetic expressions of different electroinitiated polymerizations can be deduced, as published earlier[11-12]. By changing the current density, monomer concentration, supporting electrolyte concentration and temperature, the derived kinetic expression may be experimentally verified to substantiate the mathematical aspects. It is pertinent to mention that the applied current density will affect the rate of initiation, as in the case of initiator concentration in chemical or photochemical polymerization. Here also, termination occurs via disproportionation or coupling and electrodes or supporting electrolytes used in the process also play an important role, as explained in the previous paper[13].

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CONCLUSION

An attempt was therefore made to derive mathematical relationships in order to gain a better understanding of the kinetics of electroinitiated polymerization. The discussions are primarily confined to important and significant aspects of the polymerization such as initiation steps, polymer growth process and the degree of polymerization.

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