

Theoretical Description for Lugduname and Perillartin Electrochemical Determination by Cathodic Route

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Abstract: In this work, the possibility of lugduname and perillartin electrochemical determination by cathodic process has been given. Both substances are reduced, forming the amino group, which may become protonated in an acidic medium, affecting the DEL ionic force and thereby affecting the analytical signal interpretation. Although even in the acidic medium, the system is electroanalytical efficient, it is recommended to use the neutral or mildly acidic medium for the electroanalytical process. This permits us to use the electrochemical sensor to monitor the sweetener concentration in most soft beverages.

Keywords: lugduname; perillartin; sugar substitutes; electrochemical sensor; electrochemical oscillations; stable steady-state.

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1. Introduction

Lugduname (Figure 1) is one of the sweetest substances in the world [1]. It is estimated to be up to 300 000 (three hundred thousand) times as sweet as ordinary sugar, 37,5 times as sweet as neotame, the sweetest sugar substitute currently in widespread use, 300 times as sweet as sucralose, and 600 times as sweet as saccharine. It was developed by the Université de Lyon (France) in 1996, and its name is derived from the Latin name of the city (Lugdunum). It is a part of the family of the guanidine derivatives of acetic acid, which are found to be potent sweeteners. It is also used to study taste responses in animals [2].

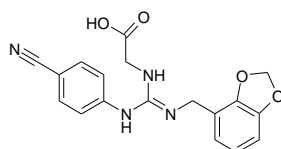


Figure 1. Lugduname.

It is limitedly used as a sweetener, as its toxicity essays have not been finished yet. Moreover, it contains toxic groups, like nitrile [3,4], the toxicity of which is dose-related. Therefore, developing a method for lugduname determination is actually [5], and electrochemical methods could provide a good service.

Adding two or more sweeteners to the beverage is frequently used to obtain certain taste combinations and/or mimic the real taste of certain beverage components. In this aspect, using lugduname and sucralose together in perspective would become widespread in regulating the organoleptic properties of beverages and pharmaceutical forms. Nevertheless, the products tend to substitute synthetic sweeteners with natural ones, which are more biocompatible and maybe even more sweet than some of the most used synthetic sugar substitutes.

One of them is perillartine (Figure 2) (perillaldehyde anti-aldoxime), a natural compound from Japanese perilla (*shiso*) leaves [6]. It is considered twice as sweet as sucralose, four times as sweet as saccharine, and 2000 times as sweet as sucrose (common sugar).

Not only the proper perillartine but also its ether derivative are used as sweeteners, although the ether is much less sweet (being compared in this relation to aspartame).

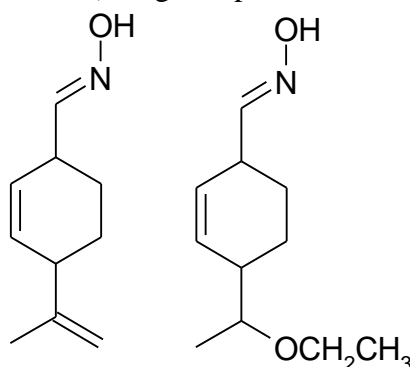


Figure 2. Perillartine and its ether derivative.

Contrarily to synthetic sweeteners like aspartame, saccharin, and sucralose, perillartine is biodegradable and bioaccessible. It is not considered toxic or dangerous for the environment.

Nevertheless, it may be allergic to people who are allergic to shiso herb [7], which has to be taken into consideration for those who tend to use perillartin instead of synthetic sweeteners. Also, some toxic nitrogen derivatives like hydroxylamine may form during its metabolism in some people. It is important to mention that perillaldehydealdoxyme and its

derivatives are rarely used as sweeteners outside Japan, so this statement may be used to investigate the falsification of allegedly Japanese products claimed to contain perillartine. Thus, developing an efficient, exact, and rapid method for perillartine determination is an actual task, and the electroanalytical methods would give it a good service.

Both compounds possess redox-active functional groups [9–14] and are electrochemically active. For this reason, both cathodic and anodic processes may be detected in an electrochemical way. In the cathodic process, the analyte will receive protons, and the presence of the proton-transfer mediator as an electrode modifier [15–21] is desirable for the analytical signal to be easily interpreted.

Therefore, using vanadium(III) oxyhydroxide as an electrode modifier may facilitate the electrochemical detection of both sweeteners in neutral and mildly acidic media, which corresponds to most soft drinks.

Nevertheless, both of the substances are reduced, yielding basic compounds, which become ionized in an acidic medium and influence the double electric layer (DEL) electrophysical properties. This may affect strongly the behavior of the electroanalytical process. For this and other reasons, the *a priori* theoretical investigation, aimed to analyze the behavior of the electroanalytical system with sucralose and lugdunam electrochemical determination from the mechanistic point of view, including the comparison to the similar processes [15–21], is necessary and this is the goal of the present study.

2. Materials and Methods

Both lugdunam and perillartine possess easily reducing groups, yielding basic amino groups, which remain less ionized in neutral solutions and more ionized in more acidic solutions. This begins to affect the DEL ionic force, conductivity, and capacitance, which becomes responsible for the instabilities in the acidic medium.

The mechanism for the electrochemical reduction of both natural and synthetic sugar substituents may be described in Figure 3:

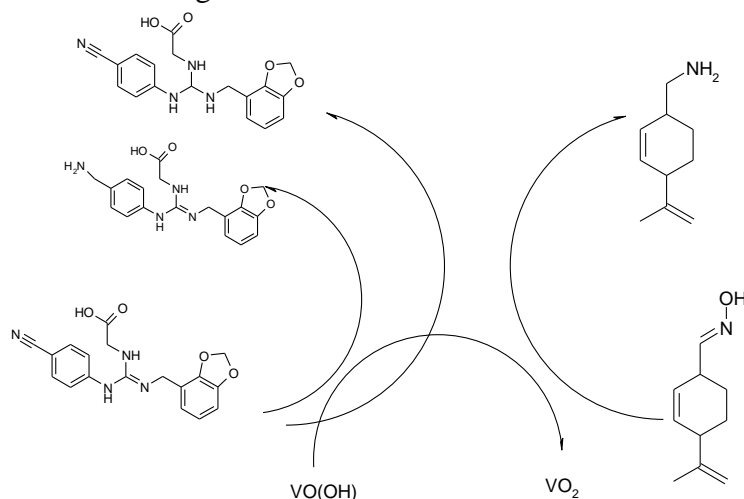


Figure 3. Perillartine and its ether derivative.

Taking this into account and considering certain assumptions [17], we describe the behavior of this system by a trivariate balance differential equation set as (1):

$$\begin{cases} \frac{dl}{dt} = \frac{2}{\delta} \left(\frac{L}{\delta} (l_0 - l) - r_{l1} - r_{l2} \right) \\ \frac{dp}{dt} = \frac{2}{\delta} \left(\frac{P}{\delta} (p_0 - p) - r_p \right) \\ \frac{dv}{dt} = \frac{1}{V} (r_{r1} + r_{r2} + r_p - r_r) \end{cases} \quad (1)$$

Herein, δ is the diffusion layer thickness, l , and p are the pre-surface concentrations of each of the sugar substitutes, L and P stand for the correspondent diffusion coefficients, l_0 and p_0 stand for their bulk concentrations, v is the VO(OH) surface coverage degree, V stands for its maximal surface concentration, and the parameters r correspond to the correspondent reaction rates, calculated as:

$$r_{l1} = k_{l1} l (1 - v)^4 \exp(-\alpha l) \quad (2)$$

$$r_{l2} = k_{l2} l (1 - v)^6 \exp(-\alpha l) \quad (3)$$

$$r_p = k_p p (1 - v)^4 \exp(-\beta p) \quad (4)$$

$$r_r = k_r v \exp\left(-\frac{F\varphi_0}{RT}\right) \quad (5)$$

In which the parameters k are the correspondent reaction rate constants, α , and β are parameters describing the impact of the chemical stages on the polymer matrix and DEL electrophysical properties correspondently (6, 7):

$$\alpha \begin{cases} = 0, \text{ if } pH = 7 \\ \neq 0, \text{ if } pH \neq 7 \end{cases} \quad (6)$$

$$\beta \begin{cases} = 0, \text{ if } pH \geq 7 \\ \neq 0, \text{ if } pH < 7 \end{cases} \quad (7)$$

F is the Faraday number, φ_0 is the zero-charge-related potential slope, R is the absolute gas constant, and T is the absolute temperature.

In this system, both of the analytes affect the DEL during the chemical stage in an acidic medium and do not affect it in neutral solutions. For this reason, the acidic medium may lead to somehow more unstable behavior. Despite this, this system is an efficient electrochemical process in both media, as shown below.

3. Results and Discussion

We investigate the electroanalytical process of VO(OH)-assisted lugdunane and perillartin electrochemical determination, analyzing the equation-set (1) alongside the algebraic relations (2 – 7) by means of linear stability theory. The steady-state Jacobian matrix members may be calculated as (8):

$$\begin{pmatrix} a_{11} & a_{12} & a_{13} \\ a_{21} & a_{22} & a_{23} \\ a_{31} & a_{32} & a_{33} \end{pmatrix} \quad (8)$$

In which:

$$a_{11} = \frac{2}{\delta} \left(-\frac{L}{\delta} - k_{l1} (1 - v)^4 \exp(-\alpha l) + \alpha k_{l1} l (1 - v)^4 \exp(-\alpha l) - k_{l2} (1 - v)^6 \exp(-\alpha l) + \alpha k_{l2} l (1 - v)^6 \exp(-\alpha l) \right) \quad (9)$$

$$a_{12} = 0 \quad (10)$$

$$a_{13} = \frac{2}{\delta} (4k_{l1} l (1 - v)^3 \exp(-\alpha l) + 6k_{l2} l (1 - v)^5 \exp(-\alpha l)) \quad (11)$$

$$a_{21} = 0 \quad (12)$$

$$a_{22} = \frac{2}{\delta} \left(-\frac{P}{\delta} - k_p (1 - v)^4 \exp(-\beta p) + \beta k_p p (1 - v)^4 \exp(-\beta p) \right) \quad (13)$$

$$a_{23} = \frac{2}{\delta} (4k_p p (1 - v)^3 \exp(-\beta p)) \quad (14)$$

$$a_{31} = \frac{1}{v} (k_{l1}(1-v)^4 \exp(-\alpha l) - \alpha k_{l1} l(1-v)^4 \exp(-\alpha l) + k_{l2}(1-v)^6 \exp(-\alpha l) - \alpha k_{l2} l(1-v)^6 \exp(-\alpha l)) \quad (15)$$

$$a_{32} = \frac{1}{v} (k_p(1-v)^4 \exp(-\beta p) - \beta k_p p(1-v)^4 \exp(-\beta p)) \quad (16)$$

$$a_{33} = \frac{1}{v} \left(-4k_{l1} l(1-v)^3 \exp(-\alpha l) - 6k_{l2} l(1-v)^5 \exp(-\alpha l) - 4k_p p(1-v)^3 \exp(-\beta p) - k_r \exp\left(-\frac{F\phi_0}{RT}\right) + \xi k_r v \exp\left(-\frac{F\phi_0}{RT}\right) \right) \quad (17)$$

Avoiding the cumbersome expression during the determinant analysis, we introduce new variables and rewrite the determinant as (18):

$$\text{Det } J = \frac{4}{\delta^2 C} \begin{vmatrix} -\kappa - \Xi & 0 & -\Lambda \\ 0 & -\phi - P & -\Phi \\ -\Xi & -P & -\Omega - \Lambda - \Phi \end{vmatrix} \quad (18)$$

Considering that:

$$-\text{Det } J \begin{cases} > 0, \text{ for steady-state stability} \\ = 0 \text{ monotonic instability} \end{cases} \quad (19)$$

Opening the brackets, applying the $\text{Det } J < 0$ requisite, salient from the criterion, and changing the signs to the opposite, we rewrite the condition set as (20):

$$\Omega(\kappa\phi + \kappa P + \Xi\phi + \Xi P) + \Lambda(\kappa\phi + \kappa P) + \Phi(\kappa\phi + \Xi\phi) \begin{cases} > 0, \text{ curve linearity} \\ = 0, \text{ detection limit} \end{cases} \quad (20)$$

If $-\text{Det } J > 0$, the Routh-Hurwitz stability criterion is valid, and the steady-state is thereby stable, providing an efficient bisphenols electrochemical determination. Moreover, the wide stability region allows us to use this system as an electroanalytical for sensing purposes.

This criterion is readily satisfied if the kinetic parameters P , Λ , Φ , and Ω are positive. In the vast majority of the cases, they both have positive signs and considering that the other variables in the determinant are positive, it indicates the vast steady-state stability topological region. The electroanalytical process is both diffusion and kinetically controlled, with the prevalence of kinetic factors. Nonetheless, in a neutral medium, in which P , Λ , and Φ are always positive (due to the conditions imposed in (6) and (7)), the diffusion factor will be more definitive for the steady-state stability.

In the absence of the side reactions or other factors capable of compromising the analyte and (or) modifier stability, excluding the reactions foreseen by the mechanism, the linearity between the electrochemical parameter and concentration is observed, providing an efficient analytical signal interpretation, which is important for sweetener concentration monitoring. Again, the analytical signal interpretation will become more efficient in a neutral medium.

The condition $\text{Det } J = 0$ corresponds to the detection limit, manifested by the *monotonic instability*. It may be seen as an N-shaped part of the steady-state voltammogram, which depicts the margin between the stable steady-states and unstable states and corresponds to the steady-state multiplicity. In other words, multiple steady-states, each one unstable, coexist at this point.

As for the oscillatory behavior, it is realized beyond the detection limit in the case of the Hopf bifurcation realization, and its probability will be different in acidic and neutral mediums. Its realization requires the presence of the positive-feedback related positive addendums in main diagonal elements.

If we analyze the main diagonal elements (9), (13), and (17), we may observe four elements capable of being possible:

$\alpha k_{l1} l(1-v)^4 \exp(-\alpha l) - k_{l2}(1-v)^6 \exp(-\alpha l) > 0$, $\alpha k_{l2} l(1-v)^6 \exp(-\alpha l) > 0$ if $\alpha > 0$, like also $\beta k_p p(1-v)^4 \exp(-\beta p) > 0$ if $\beta > 0$, describing the impact of the cyclic

changes in DEL caused by ionic forms (trans)formations during the chemical stages, which will cause the cycles of increase and decrease in DEL conductivity, manifested by the oscillatory behavior, like also the similar impacts of the surface, described by the positivity of $\xi k_r v \exp\left(-\frac{F\varphi_0}{RT}\right) > 0$, if $\xi > 0$.

Again, in a neutral medium, in which α and β are nil, only the last factor for the oscillatory behavior remains. Therefore, it is possible to conclude that, despite the efficiency of the sensor in mildly acidic solutions, the neutral medium is the most favorable for quantifying both sweeteners, such as in similar systems [17].

4. Conclusions

From the theoretical description of lugdunane and perillartin electrochemical cathodic determination on trivalent vanadium oxyhydroxide-modified electrode, it has been possible to conclude it may be an excellent modifier for the quantification of both of the sweeteners in neutral and mildly acidic media. The electroanalytical process is both diffusion and kinetically controlled, with the prevalence of the kinetic factor in an acidic medium and diffusion factor in a neutral medium. The oscillatory behavior in this system may be caused by both DEL and matrix influence by electrochemical and chemical stages. Nevertheless, the chemical stage impacts the electrochemical oscillations only in an acidic medium. The system may be efficiently used to quantify both sugar substitutes in beverages.

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Conflicts of Interest

The authors declare no conflict of interest.

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