

On quality assurance of winemaking components

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Abstract. This report examines product quality assurance issues arising when technological aids and food additives are utilized in winemaking. L(+)-tartaric acid, the acidity regulator, is used as an example to describe the in-depth state of modern regulatory frameworks applied at both national and international levels to protect consumer rights and the interests of bona fide manufacturers of high-quality and authentic products. One aspect of quality assurance is synthetically derived L(+)-tartaric acid present on the market, which contradicts the International Oenological Codex (IOC), describing in its COEI-1-LTARAC Monograph natural L(+)-tartaric acid obtained from plant-based raw materials, namely grapes. To ensure the quality and authenticity of L(+)-tartaric acid at the interstate level between the CIS countries, in a harmonisation effort with regard to the IOC, the Interstate Standard GOST 21205-2024 for plant-derived L(+)-tartaric acid (food additive E334) has been developed. According to the new standard, plant-derived L(+)-tartaric acid is permitted for use in food production, including winemaking. At the same time, tartaric acid produced using a combined chemical and biotechnological synthesis process from hydrocarbon raw materials, including coal, oil, and its processed products, such as intermediates like maleic anhydride, benzene, butane, epoxysuccinates, etc., is not considered an acceptable component for the food industry. The interstate standard GOST 21205-2024 contains a new parameter, isotope ratio of carbon $^{13}\text{C}/^{12}\text{C}$ ($\delta^{13}\text{C}_{\text{VPDB}}$), ‰, added to ensure product authenticity, enabling a reliable assessment of the origin of L(+)-tartaric acid. The practical application of this parameter within the new standard aligns with research done by the International Organisation of Vine and Wine (OIV), particularly with a new method developed for analysing tartaric acid preparations based on mass spectrometry of the ratios of stable carbon and oxygen isotopes (OENO-SPECIF 21-691). A logical progression in methodological support for the quality and authenticity of components used in winemaking is the development and standardization of a method employed to determine the nature of organic acids, including L(+)-tartaric acid, contained directly in wine. This work creates a systematic approach to improving the harmonised regulatory framework for protecting the rights of manufacturers, consumers, and fair competition, ultimately contributing to the production and market circulation of high-quality and authentic wine products.

1. Introduction

Modern winemakers face a challenging task of creating a product that meets market demands and evolving consumer preferences while following traditions and maintaining authenticity. Certainly, they have at their disposal a wide range of food additives and technological aids designed to help achieve this goal. These components not only influence the quality and stability of the final product but also play a crucial role in implementing environmentally sustainable practices and adhering to stringent safety standards. This makes the issue of quality and authenticity of food additives and

technological aids, as well as consumer awareness of their properties, even more significant. Consumer rights are safeguarded by the regulatory framework consisting of various legal acts, aiming to provide transparent guidance on permissible and prohibited components while also legislating reliable methods for assessing quality parameters. However, in certain cases, the regulatory framework fails to keep up with the rapidly changing reality. For example, advances in chemistry and biotechnology have led to the creation of a synthetic analogue of L(+)-tartaric acid, which can be used as an acidity regulator. Yet, there is currently no legal

distinction between natural and synthetic L(+)-tartaric acid, despite the fact that the production and commercial distribution of the synthetic product have been ongoing for several decades. In this regard, it seems appropriate to align the regulatory framework to ensure that consumers are guaranteed with access to high-quality products.

2. Problem Localisation

In order to protect consumer rights to high-quality and safe products, the Eurasian Economic Union (EAEU) has established a regulatory framework, consisting of the following regulatory documents:

1) Technical Regulations of the Customs Union TR CU 029/2012 Safety Requirements for Food Additives, Flavourings and Technological Aids.

The Technical Regulations have been developed to establish unified mandatory requirements within the Customs Union for the use and implementation with regard to food additives, flavourings, and technological aids, as well as their content in food products. The Regulations permit the use of L(+)-tartaric acid as a food additive, labelled according to the international standard CODEX STAN 192-1995 Codex Alimentarius INS334/E334. The information is provided in Appendix No. 2 to the Technical Regulations, Safety Requirements for Food Additives, Flavourings and Technological Aids (TR CU 029/2012): List of Food Additives Approved for the Use in Food Production.

This is consistent with research findings presented in the twenty-first report of the Joint FAO/WHO Expert Committee on Food Additives, which confirms that the L(+) isomer of tartaric acid is safe for human consumption.

2) Federal Law No. 468-Φ3 On Viticulture and Winemaking in the Russian Federation dated 27 December 2019.

The Federal Law regulates relationships between organisations, agricultural consumer cooperatives, peasant (farm) enterprises, individuals, government authorities of the Russian Federation, government authorities of the constituent entities of the Russian Federation, and local self-governing bodies in viticulture and winemaking. Federal Law No. 468-Φ3, among the key objectives of state policy related to viticulture and winemaking, in Part 2 of Article 4 establishes, among other things:

1. Enhancing the quality of viticulture and winemaking products that are produced and distributed within the Russian Federation;

2. Increasing the competitiveness of viticulture and winemaking products made from grapes grown in the Russian Federation;

3. Ensuring and protecting civil rights to safe viticulture and winemaking products, as well as access to reliable information regarding the origin, composition, and characteristics of winemaking products.

To achieve these objectives, the Federal Law defines authorised technological methods for processing grapes intended for winemaking, as well as approved technological techniques and operations for producing wine, fortified wine, sparkling wine, cognac, and cognac distillate used in its production. Additionally, it specifies the raw and other materials permitted for use in the production of wine, fortified wine, and sparkling wine.

This Federal Law allows for the use of tartaric acid for acidification purposes. However, there is currently no clear definition of this technological aid that would enable consumers to accurately identify it.

At the same time,

1) there are two main technologies for the production of tartaric acid:

- the production of natural plant-derived L(+)-tartaric acid, made from by-products of grape processing and winemaking (e.g., grape pomace, tartar, etc.);
- the production of synthetic L(+)-tartaric acid using a combined chemical and biotechnological synthesis process from hydrocarbon raw materials, including coal, oil, and its processed products, such as intermediates like maleic anhydride, benzene, butane, epoxysuccinates, etc.

2) The Classifier of Foreign Economic Activity Commodity Nomenclature, as part of the commodity classification system for customs clearance purposes in the EAEU countries, applies a single code for all types of tartaric acid: 2918120000.

3) The market offers both natural and synthetic L(+)-tartaric acid for use and free consumption for winemaking purposes. However, this is in direct contradiction with the International Oenological Codex (IOC), describing in its COEI-1-LTARAC Monograph only natural L(+)-tartaric acid obtained from plant materials (grapes).

4) Currently, there are no regulatory documents governing the use of food additive E334, L(+)-tartaric acid, including in winemaking, that define identification parameters based on its origin.

Thus, there is a need for a legal instrument to differentiate between synthetic and natural L(+)-tartaric acid within existing regulations and to provide clear physical identification criteria for products.

3. Solution

3.1. Legal Aspect

To ensure the quality and authenticity of L(+)-tartaric acid at the interstate level between the EAEU (CIS) countries, in a harmonisation effort with regard to the IOC, the Interstate Standard GOST 21205-2024 for plant-derived L(+)-tartaric acid (food additive E334) has been developed.

DEVELOPED by V.M. Gorbatov Federal Research Centre for Food Systems of RAS (Gorbatov Research Centre for Food Systems) and Chemical Leaders Limited Liability Company (Chemical Leaders LLC).

ADOPTED by the Interstate Council for Standardization, Metrology, and Certification (Protocol No. 178-II dated 31 October 2024).

The following votes were given for the adoption: Azstandard (Azerbaijan), National Body for Standardisation and Metrology, CJSC of the Republic of Armenia (Armenia), State Standard of the Republic of Belarus (Belarus), State Standard of the Republic of Kazakhstan (Kazakhstan), Kyrgyzstandard (Kyrgyzstan), Rosstandard (Russia), Tajikstandard (Tajikistan), Uzstandard (Uzbekistan).

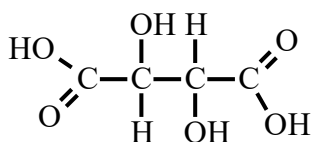
By Decree of the Federal Agency on Technical Regulating and Metrology No. 1662-cr dated 13 November 2024, the interstate standard GOST 21205-2024 was enacted as the National Standard of the Russian Federation from 1 December 2025, with the right to early application. The Standard replaces the outdated standard GOST 21205-83.

The Standard applies to the food additive, plant-derived crystalline tartaric acid L(+) E334, intended for use in food production as an acidity regulator, antioxidant, or flavouring agent.

Characteristics: Tartaric acid L(+) E334 is L(+)-tartaric acid obtained from raw materials of plant origin.

Formulas:

- empirical - C₄H₆O₆
- structural



Molecular weight – 150.09.

Chemical name – L(+)-tartaric acid; L-2,3-dihydroxybutanedioic acid; d- α , β -dihydroxysuccinic acid, CAS number 87-69-4.

Table 1. Physical and chemical parameters of L(+)-tartaric acid

Parameter	Standard
Identification of L(+)-tartaric acid E334	Passed
Mass fraction of the main substance, % min	99.5
Mass fraction of ash, % max	0.3
Mass fraction of free sulfuric acid, % max	0.03
Mass fraction of chlorides, % max	0.01
Mass fraction of sulphates, % max	0.20
Acetic calcium oxalate test	Passed
Barium test with sulfuric acid	Passed
Ferric chloride ferrocyanide test	Passed
Specific optical rotation angle $[\alpha]^{20}_D$, deg	11.5° to 12.5° (20% (w/v) aqueous solution)
Isotope ratio of carbon $^{13}\text{C}/^{12}\text{C}$ ($\delta^{13}\text{C}$), ‰	- 24.0 to - 21.0

According to the new standard, plant-derived L(+)-tartaric acid is permitted for use in food production, including winemaking. At the same time, tartaric acid produced using a combined chemical and biotechnological synthesis process from hydrocarbon raw materials, including coal, oil, and its processed products, such as intermediates like maleic anhydride, benzene, butane, epoxysuccinates, etc., is not considered an acceptable component for the food industry.

3.2. Method for Determining the Isotope Ratio of Carbon $^{13}\text{C}/^{12}\text{C}$ in Tartaric Acid Preparations

The interstate standard GOST 21205-2024 contains a new parameter, isotope ratio of carbon $^{13}\text{C}/^{12}\text{C}$ ($\delta^{13}\text{C}$), ‰, added to ensure product authenticity, enabling a reliable assessment of the origin of L(+)-tartaric acid.

The isotope ratio of carbon $^{13}\text{C}/^{12}\text{C}$ ($\delta^{13}\text{C}$) in L(+)-tartaric acid E334 is determined using mass spectrometric measurement of the isotope ratio of carbon $^{13}\text{C}/^{12}\text{C}$ in carbon dioxide (CO₂) molecules ($m/z = 44$, $m/z = 46$), formed during high-temperature oxidation-reduction decomposition of the initial sample of L(+)-tartaric acid with a purity of at least 95%.

3.2.1. Measuring Instruments, Glassware, Materials, and Reagents

- Helium for analysis (CAS No. 07440-59-7).
- Oxygen for analysis (CAS No. 07782-44-7).
- Reagents for oxidation, reduction for the furnace and combustion system.
- Drying agent to eliminate water produced during combustion, such as magnesium perchlorate (CAS No. 10034-81-8).
- Disposable tin capsules.

- Carbon dioxide CO₂ (CAS No. 00124-38-9) with a purity of at least 99.998%.
- Working and control reference standards with $\delta^{13}\text{C}$ values calibrated against international reference standards.
- IRMS/SIRA isotope ratio mass spectrometer, allowing measurements of CO₂ carbon dioxide isotopomers produced during combustion with an accuracy of $\pm 0.1\%$ for C (measurement accuracy is the difference between two measurements of the same gas sample). The mass spectrometer used must be equipped with a series of current collectors for simultaneous measurement of ion currents: $m/z = 44$, 45, 46. The carbon isotope ratio $^{13}\text{C}/^{12}\text{C}$ is determined using the intensity ratio of $m/z = 45$ and $m/z = 44$, with corrections for isobaric processes of $^{12}\text{C}^{17}\text{O}^{16}\text{O}$, the influence of which can be calculated as a function of the current intensity measured for $m/z = 46$ and the relative abundance of ^{18}O and ^{17}O (Craig correction). The isotope ratio mass spectrometer must be equipped with a dual-inlet system for alternating measurement of the sample and standard, or a continuous flow system that supplies the mass spectrometer with the amount of gas generated during combustion of samples and working reference standards.
- An elemental analyser for high-temperature oxidation-reduction decomposition (combustion) that can ensure quantitative conversion of the sample into carbon dioxide (during combustion), as well as separate gases and remove water without isotope fractionation. The equipment can be either a continuous helium flow system integrated into the IRMS/SIRA mass spectrometer or a standalone combustion system. In the latter case, gases are collected in special containers, which are then connected to the IRMS/SIRA isotope ratio mass spectrometer.
- Analytical microbalances for weighing samples in the range of 0 to 100 mg with an accuracy of ± 0.01 mg.
- Porcelain mortar and pestle as per GOST 9147.

3.2.2. Preparation for Analysis

The analysis is carried out on a sample of dry L(+)-tartaric acid E334 with a purity of at least 95%, without prior sample preparation. To obtain a homogeneous mass, the samples are ground using a mortar and pestle.

3.2.3. Analysis

The procedure described below generally applies to sample combustion using commercial automated combustion systems. Alternative methods may also be used, provided they enable the quantitative conversion of samples into carbon dioxide without losses due to evaporation.

A clean tin capsule is picked up using micro tweezers and placed in a capsule holder. Using a special spatula,

the required amount of the L(+)-tartaric acid E334 original sample is placed into the capsule.

Note: The required amount of the L(+)-tartaric acid E334 original sample placed in the tin capsule is determined based on the difference between the CO₂ signal intensity during sample combustion and the CO₂ signal obtained during combustion of the working reference standard (or reference material), which should not exceed 50%. To ensure the specified measurement acceptability limit, a preliminary measurement must be conducted to determine the required amount of L(+)-tartaric acid E334 original sample.

The required sample weight of the L(+)-tartaric acid E334 original sample is measured using an analytical microbalance.

The capsule is hermetically sealed using micro tweezers. At least two capsules are prepared for each L(+)-tartaric acid E334 sample. The capsules are placed in an autosampler rack of the elemental analyser (if available). All capsules must be sequentially numbered. The capsules containing the working reference standard are systematically placed at the beginning and end of the sample series.

The hermetically sealed tin capsules containing the samples are sequentially introduced into the automatic sampler of the elemental analyser. The gases produced during combustion of each sample are progressively fed into the IRMS/SIRA mass spectrometer, which measures ion currents. A computer connected to the IRMS/SIRA mass spectrometer, along with corresponding software, ensures the registration of ion currents and the calculation of $\delta^{13}\text{C}$ values for each sample.

3.2.4. Calculation and Expression of Results

The $^{13}\text{C}/^{12}\text{C}$ values are calculated and expressed on the delta scale $\delta^{13}\text{C}_{\text{VPDB}}$, ‰, relative to the

international standard V-PDB (Vienna Pee Dee Belemnite standard for carbon isotope ratio) using the formula:

$$\delta^{13}\text{C}_{\text{VPDB}} = [({}^{13}\text{C}/{}^{12}\text{C})_{\text{проба}} - ({}^{13}\text{C}/{}^{12}\text{C})_{\text{VPDB}} / ({}^{13}\text{C}/{}^{12}\text{C})_{\text{VPDB}}] \cdot 1000,$$

where

$({}^{13}\text{C}/{}^{12}\text{C})_{\text{VPDB}}$ is the international reference standard for $^{13}\text{C}/^{12}\text{C}$ carbon isotope ratio measurements;

$({}^{13}\text{C}/{}^{12}\text{C})_{\text{sample}}$ is the isotope ratio of carbon in the analysed sample of L(+)-tartaric acid E334;

1000 is the correction factor.

The measurement results are calculated automatically by the computer software connected to the IRMS/SIRA mass spectrometer. At least two international reference standards or working reference standards must be placed at the beginning and at the end of the sequence of sample capsules. Based on the two reference points provided by the international reference standards (or working reference standards), an interpolation line is plotted, and the corresponding equation is derived, which is then used to correct all measurement results.

3.2.5. Control of Determination

The mean value obtained for the working reference standards used in the laboratory must fall within the reliability range established by the laboratory during the calibration of the mass spectrometric measurement system. The absolute difference between the results of two repeated measurements of the same L(+)-tartaric acid E334 sample must not exceed 0.3 ‰.

3.3. Application

Using the above method, studies were conducted on samples of L(+)-tartaric acid available for sale in the Russian Federation market and offered by suppliers as food additive E334, a technological aid for winemaking for acidification purposes.

During the study, 8 samples of L(+)-tartaric acid currently in commercial circulation were analysed. The results are presented in Table 2.

Table 2. Result Summary of Comprehensive Analytical Study on Tartaric Acid

Item No.	Isotope ratio of carbon $\delta^{13}\text{C}$, ‰	Specific angle of rotation $[\alpha]_D^{20}$	Content of tartaric acid in the preparation, % (HPLC) to the reference standard
1	2	3	4
<i>Specific reference</i>			
1	-22.96 ± 0.12	+12.47°	110.6
2	-22.66 ± 0.15	+12.49°	112.3
<i>Samples of products on the Russian market</i>			
3	-22.18 ± 0.07	+12.54°	107.9
4	-28.03 ± 0.10	+12.50°	105.0
5	-28.14 ± 0.10	+1.30°	101.0
6	-28.50 ± 0.07	+12.50°	104.2
7	-27.75 ± 0.13	+12.40°	103
8	-28.11 ± 0.11	+12.47°	102.8
9	-29.68 ± 0.10	+12.50°	101.6
10	-28.48 ± 0.16	+12.39°	101.8

Samples numbered 4, 5, 6, 7, 8, 9, and 10 were clearly obtained through a microbiological process from mesotartaric acid, which was converted into D/L-tartaric acid with the microbiological removal of the D-isomer, as evidenced by the isotope ratio of carbon $\delta^{13}\text{C}$ ranging from -27.75 to -29.68.

Thus, the study established that L(+)-tartaric acid available on the Russian market is partially represented by a synthetic product, yet it is being offered by suppliers as a technological aid for winemaking. This directly contradicts the International Oenological Codex (IOC) and the Interstate Standard GOST 2205-2024, thereby violating consumer rights to access high-quality and authentic products.

4. Conclusion

Food additives and technological aids play a crucial role in modern winemaking, ensuring the stability, safety, and high quality of the final product. However, it is important to maintain a responsible approach to their use, adhering to recommendations and regulations to preserve the natural character and uniqueness of wine. In this context, the adequacy of the regulatory framework will contribute to the balance between innovative technologies and authentic methods. The practical application of the isotope ratio of carbon $\delta^{13}\text{C}$ within the new standard aligns with research conducted by the International Organisation of Vine and Wine (OIV), particularly with the development of a new method for analysing tartaric acid preparations using mass spectrometry of stable carbon and oxygen isotopes (OENO-SPECIF 21-691). A logical progression in methodological support for the quality and authenticity of components used in winemaking is the development and standardization of a method employed to determine the nature of organic acids, including L(+)-tartaric acid, contained directly in wine. This work creates a systematic approach to improving the harmonised regulatory framework for protecting the rights of manufacturers, consumers, and fair competition, ultimately contributing to the production and market circulation of high-quality and authentic wine products.

5. Conflict of Interest

The authors declare no conflict of interest.

6. References

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