

Comparison of three different methods for the determination of sulphur dioxide in fruit and vegetable products

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Abstract: Sulphite is a food additive used worldwide. Globally, for concentrations above 10 mg·kg⁻¹, sulphite compounds must be labelled as sulphur dioxide (SO₂) on the packaging due to their potential health risks. This study compares spectrophotometric (S), titration (T) (modified optimised Monier-Williams, OMW), and reflectoquant (R) methods for measuring sulphur dioxide in twenty fruit and vegetable products. The samples comprise sulphited, un-sulphited, and naturally sulphur-containing products (*Allium* genus such as garlic and onion). The article discusses the strengths and weaknesses of the method used. *Allium* genus samples yielded false-positive results, especially in fresh garlic samples with average SO₂ concentrations of 46, 1 152, and 40 mg·kg⁻¹ obtained by titration, spectrophotometric, and reflectoquant methods, respectively, therefore, none of the methods is suitable for testing this type of vegetables or products containing a low proportion of them. For other types of samples, the methods showed acceptable working characteristics. Recovery tests showed 89.5, 82.0, and 75.2% recovery with 2.8, 3.9, and 13.2% repeatability and the limit of quantification of 1, 10, and 25 mg·kg⁻¹ in the spectrophotometric, titration, and reflectoquant methods. The result highlights the importance of method selection based on sample characteristics and regulatory compliance.

Keywords: sulphite; false positive; *Allium* genus samples; spectrophotometric method; titration method; reflectoquant method

Sulphites are a class of food additives subject to global regulations for their usage in food products. Due to their versatile nature, they possess extensive applications. Synthetic chemical preservatives are widespread within the food sector due to their affordability and potent preservation capabilities. Recently, there has been increasing consumer concern about the adverse health effects of some synthetic preservatives.

This concern is particularly prominent among people with respiratory sensitivities, which cause a noticeable shift in consumer preference towards fresh fruits and vegetables, mostly driven by their perceived health benefits and nutritional value (Baselice et al. 2017; Dwivedi et al. 2017).

Sulphur dioxide and sulphites, specifically designated as food additives signed from E220 to E228, are

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commonly applied in food, in the form such as sulphur dioxide, sodium sulphite, calcium sulphite, sodium bisulphite, potassium bisulphite, sodium metabisulphite, and potassium metabisulphite. According to Robbins et al. (2016), within the food business, these substances have applications as antimicrobials, bleaching agents, reducing agents, and inhibiting enzymatic and non-enzymatic browning. Sulphites are commonly employed in preserving dried fruits and fermented drinks due to their antioxidant properties (Gunther et al. 1998). To prevent mould and yeast growth and to inhibit discolouration, preservatives are added to concentrated fruit and vegetable juices at a concentration not exceeding $50 \text{ mg}\cdot\text{kg}^{-1}$ (Codex-Alimentarius Commission 2018). During the initial years of the 1980s, there were documented accounts of individuals with heightened sensitivity exhibiting pronounced allergic-like responses after the consumption of food products that had undergone sulphite treatment. As a result, the European Commission Regulation (EU) No. 1129/2011 implemented a regulation mandating the inclusion of sulphite content (measured as SO_2) on the packaging of any product containing sulphite levels over $10 \text{ mg}\cdot\text{kg}^{-1}$, and the permissible quantities are determined by the food items specified in Annex II or the specific way the component is incorporated, as outlined in Annex III European Commission regulation (EU) No. 1130/2011. The permitted amount range goes from $10 \text{ mg}\cdot\text{kg}^{-1}$ (e.g. table grapes) to $10\,000 \text{ mg}\cdot\text{kg}^{-1}$ in papain in solid form [European Commission regulation (EU) No. 1130/2011].

Certain food items inherently contain elevated concentrations of sulphur compounds, plants contain these natural biologically active molecules that serve as a defence mechanism and proper functioning. These molecules are classified as glucosinolates, alliin, thiosulphinates, and sulfoxides (Petropoulos et al. 2017). There is currently no evidence to suggest that sulphur dioxide and sulphites exist naturally in raw materials. Nevertheless, certain existing regulatory techniques used for sulphite detection, such as the optimised Monier-Williams (OMW), have been found to yield inaccurate positive outcomes when applied to vegetables belonging to the *Allium* (garlic, onions, etc.) and *Brassica* (cabbage, mustard, etc.) genera (Robbins et al. 2016).

Allium plants, including garlic, onions, and leeks, are renowned for their characteristic sulphur-containing chemicals, particularly thiosulphinates, which occur naturally (Iberl et al. 1990). The compounds in *Allium* vegetables are responsible for the characteristic aroma and

taste exhibited by these vegetables. Thiosulphinates can interact with enzymes, including alliinase, accelerating the conversion of alliin to allicin. This conversion is typically triggered by mechanical damage or processing, which leads to allicin decomposing and liberating various sulphur compounds, including SO_2 (Mochizuki et al. 1997; Borlinghaus et al. 2014; Bosca et al. 2023). According to a study by Lafeuille et al. (2007), allicin produces a significant amount of SO_2 when exposed to pH levels below 2.4 and boiling water temperatures, leading to false positive outcomes in samples.

Regulatory agencies in the United States and the European Union have been searching for an approach to eliminate false positives in *Allium* and *Brassica* vegetable sulphite analysis. Although there have been attempts at identifying the difference between extra sulphite and naturally occurring sulphur compounds, no single method has been proven to work across multiple species. The problem is thought to be caused by the method's extraction conditions, which are thought to cause endogenous sulphur compounds to be released, causing SO_2 to be produced (Robbins et al. 2016). Robbins et al. (2016) and Kim et al. (2000) analysed different kinds of vegetables from *Allium* and *Brassica* genera using various methods and found that the *Allium* samples produced much higher SO_2 concentrations than the *Brassica* samples. The *Brassica* samples evaluated by the OMW titration method exhibited values ranging from $7.0 \text{ mg}\cdot\text{kg}^{-1}$ to $12.6 \text{ mg}\cdot\text{kg}^{-1}$ SO_2 in broccoli and cauliflower, respectively. Similar results were obtained for the OMW gravimetric, where the concentrations of SO_2 in broccoli and cauliflower were $6.5 \text{ mg}\cdot\text{kg}^{-1}$ and $11.8 \text{ mg}\cdot\text{kg}^{-1}$, respectively. The liquid chromatography with tandem mass spectrometry (LC-MS/MS) method detected SO_2 as the least ranging from $1.4 \text{ mg}\cdot\text{kg}^{-1}$ to $3.9 \text{ mg}\cdot\text{kg}^{-1}$ SO_2 in kale and cauliflower, respectively. The *Allium* genera produced the most false positive results and made regulatory compliance the most challenging. Specifically, the garlic showed SO_2 values of around $90 \text{ mg}\cdot\text{kg}^{-1}$ as determined by the OMW method. This has also been demonstrated in the literature, where particular garlic samples have been discovered to have SO_2 values higher than $200 \text{ mg}\cdot\text{kg}^{-1}$ (Perfetti et al. 2003). The studied *Allium* family members have different concentrations of SO_2 . And OMW titration results for garlic were $86.5 \text{ mg}\cdot\text{kg}^{-1}$ SO_2 and for chives were $17.4 \text{ mg}\cdot\text{kg}^{-1}$ SO_2 . As stated previously, the concentrations and patterns of the OMW gravimetric analysis results matched those of the OMW titration results. The LC-MS/MS method showed significantly lower

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concentrations compared to the OMW titration method which is below $10 \text{ mg} \cdot \text{kg}^{-1}$ across all the measured *Brassica* samples.

Because these fruits and vegetables are employed as ingredients in products, there must be a method for regulatory assessments that limits the false-positive result as much as possible. To the best of the author's knowledge, limited research has been published investigating the false-positive response in various vegetable kinds or using various methodologies. Chung et al. (2008) used high-performance liquid chromatography (HPLC) with fluorometric detection to evaluate false positives caused by certain matrices. They analysed three distinct *Brassica* samples but did not look into any *Allium* species, where most false positives occur. Because of the discrepancy in the literature, the goal of this study is to learn more about the degree of false positives caused by three distinct methodologies in an *Allium* species and the other non-challenging samples to explore the strengths and weaknesses of the method.

MATERIAL AND METHODS

Material and its preparation

A total of twenty samples of various foods are listed in Table 1, five samples labelled as sulphited, ten challenging samples and five unsulphited were collected from stores and local markets and kept in a normal laboratory condition. The garlic and onion samples were prepared for analysis just before they were tested. The samples were placed in a Retsch GM 200 homogeniser (Retsch, Germany), and run for about 15 to 20 s at 2 500 rpm (revolutions per minute) until homogenised, and analysis was performed in replication.

Determination methods of SO_2

Titration (modified optimised Monier-Williams) method (T). The analytical conditions were based on ČSN EN 1988-1 (560025) with some modifications on optimised Monier-Williams such as using more concentrated acid to reduce the boiling time. The sample is added into the double neck round bottom flask, and 350 mL of distilled water and 20 mL of 35% hydrochloric acid (HCl) are added. After that, the flask is placed into the heating mantle, and the system is attached with two dreschel bottles containing 10 mL of 3% hydrogen peroxide (H_2O_2) in each. The sample is boiled for 30 min in an acidic condition; sulphur contained in the sample will be released as SO_2 , which is then carried along with the flow of nitrogen gas

Table 1. Sample list

Sample name/ number	Product name	Geographical origin
DF1*	papaya	Thailand
DF2*	pineapple	Thailand
DF3*	raisin	USA
DF4*	raisin	Chile
DF5*	apricot	unknown
DF6	raisin	Germany
DF7	raisin	Türkiye
DF8	bio raisin	Türkiye
DF9	cranberry	Canada
DF10	cranberry	USA
G1	bio garlic	Italy
G2	bio garlic	Spain
G3	garlic	unknown
G4	garlic	Czech Republic
G5	powder garlic	Czech Republic
O1	shallot	France
O2	yellow onion	Netherlands
O3	yellow onion (bio)	Czech Republic
O4	shallot	Czech Republic
O5	red onion	Czech Republic

*Means samples with SO_2 labelled; DF – dry fruit; G – garlic; O – onion

and reacts with H_2O_2 in two dreschel bottles, forming sulphuric acid. The amount of acid formed is measured by titration against a diluted $0.05 \text{ mol} \cdot \text{dm}^{-3}$ sodium hydroxide (NaOH) solution, which is determined by adding three drops of bromophenol blue, an indicator.

Spectrophotometric method (S). Davídek et al. (1981) primarily carried out the method of study. The distillation Unit K-355 BÜCHI (BÜCHI, Switzerland) has been set at steam: 50% and time: 14.5 min. Samples were weighed into a beaker and distilled water (40 mL) was added, which was further homogenised by ultraturax and transferred to a distillation tube; $4 \text{ mol} \cdot \text{dm}^{-3}$ NaOH (10 mL was added to the beaker and then to the distillation tube and after 5 min 20 mL of 25% phosphoric acid (H_3PO_4) was added. The addition of 50 mL water + 10 mL $4 \text{ mol} \cdot \text{dm}^{-3}$ NaOH + 20 mL 25% H_3PO_4 in a distillation tube results in the blank preparation. On the receiving side, a 250 mL volumetric flask is placed, and 25 mL of $0.16 \text{ mol} \cdot \text{dm}^{-3}$ NaOH is added before distillation. The flask should be filled to the mark with distilled water after distillation, and the distillate sample should be diluted based on the prejudged concentration. Then,

a 10 mL sample solution is pipetted into a test tube, followed by the addition of 2 mL of pararosaniline and 2 mL of 0.2% formaldehyde. The absorbance (560 nm) was measured on spectrophotometer SPEKOL 1300 (Analytik Jena, Germany), after waiting for 30 min. Six-point calibration of concentration 0.5, 1.0, 1.5, 2.0, 2.5, 3.0 mg·kg⁻¹ is prepared from 10 mg·kg⁻¹ standard solution of SO₂ which is prepared by the quantitative measurement and the dilution of sodium metabisulphite salt for the quantification.

Reflectoquant method (R). The analysis was performed according to the procedure of RQflex[®] 20 Reflectoquant[®] (Merck KGaA, Germany), Test strip reflectometer 10–200 mg·L⁻¹ sulphite ions. Reflectometry is the principle for the R method, which determines the amount of sulphite (SO₃²⁻) in water. Test strips coated with a mixture of chemicals (potassium hexacyanoferrate II, zinc sulphate, and sodium nitroprusside) that react with sulphite ions to form a red compound that is determined reflectometrically. The concentration of sulphite ions in the sample directly influences the intensity of this colour alteration [Test for Determination of Sulfites (116987; Merck, Germany)]. The R method requires a separate preliminary sample preparation procedure for the raw material and the dry product. For the dry product, 50 mL of 0.01 mol·dm⁻³ NaOH is added to the homogenised sample and gently shaken for 5 min. The pH is maintained between 9 and 11 with a 1 mol·dm⁻³ NaOH solution. In addition, for the raw material, 25 mL and 0.01 M NaOH are added to a 25 g crushed sample and added to 25 mL of distilled water to mix thoroughly. The sample is allowed to settle, centrifuge (if necessary), and use supernatant liquid for analysis. The reflectometer is started, and the test strip is dipped into the sample for 2 s to cover both response zones. The strips are inserted into the reflectometer to inspect, once 30 s have passed and saving is automatically done (mg·L⁻¹).

Statistical analysis

The data collected were used for statistical evaluation in STATISTICA software (version 12.0). First, before the statistical evaluation, the Dean-Dixon test was performed to exclude outliers. The statistical significance between different determination methods of SO₂ was statistically determined at first by one-way analysis of variance (ANOVA) applied to collected data. Then, a post hoc HSD Tukey multiple comparison test was used to see the different data groups. The significance level was established at $P < 0.05$. All resulting data in the table are given as mean ± standard deviation (SD).

RESULTS AND DISCUSSION

Analytical assurance. Twenty different types of fruits and vegetables were tested for sulphur dioxide using the spectrophotometric (S), titration (T), modified optimised Monier-Williams), and reflectoquant (R) methods. The foods included sulphited, unsulphited, and naturally sulphur-containing items like garlic and onion. Prior studies have indicated that the OMW method produces a false-positive result for several of these species. Additional investigation was required to identify the level of false positives observed using these three methods. 100 mg·kg⁻¹ of SO₂ standard solution is prepared by the quantitative measurement and the dilution of sodium metabisulphite salt for the quantification, which is further diluted and used for the limit of detection (LOD) and the limit of quantification (LOQ) analysis of the three methods. The LOD was determined to be 1, 2, and 10 mg·kg⁻¹ and LOQ was 1, 10, and 25 mg·kg⁻¹ for the S, T, and R methods, respectively. The utilization of sulphites in recovery studies remains inappropriate due to the reactivity of added sulphite, which leads to a significant amount becoming irreversibly associated with the food matrix. Robbins et al. (2016) recorded a similar situation. The authors suggest that there could be an interaction between the added sulphite and the internal components, resulting in the sulphite being bound or reacting in a manner that prevents the production of SO₂ gas during distillation. Therefore, a water recovery study was conducted to demonstrate proficiency. All methods used sodium metabisulphite (Na₂S₂O₅) in concentrations of 100 mg kg⁻¹ and 10 mg·kg⁻¹ for the recovery test. The results of the water recovery analysis of 100 mg·kg⁻¹ and 10 mg·kg⁻¹ are 89.5% and 85.3%, 82.0% and 84.7%, 75.2% and below the limit of detection, for S, T, and R methods, respectively. Repeatability (RSD) at 100 mg·kg⁻¹ (Na₂S₂O₅) was found to be 2.8% in the S method, 3.9% in the T method, and 13.2% in the R method across five duplicate samples. The findings of this study demonstrate the precision of the methodology used in analysing the food matrix.

Results of sulphite in fruit and vegetable products. When evaluating the three methods for determining total sulphite, it is crucial to consider the variation in the process of sulphite liberation from the samples. The T method utilizes a process of strong acid refluxing, which results in the liberation of all sulphite forms and the subsequent creation of sulphuric acid through a reaction with H₂O₂. The S method employs both acid and base treatments to liberate SO₂. Acidification through

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steam distillation releases the free SO_2 , while alkaline hydrolysis liberates the total SO_2 . The liberated SO_2 then reacts with pararosaniline to form a purple-red complex, which can be quantitatively measured for colour intensity using spectrophotometry at a wavelength of 560 nm. The R method involves the reaction of sulphite ions with a combination of potassium hexacyanoferrate (II), zinc sulphate, and sodium nitroprusside, resulting in the formation of a red compound that is subsequently determined reflectometrically (Test for Determination of Sulfitess). Due to the different functionalities and restrictions of methods, we have created two different categories to test out the efficiency of the methods, which are dry food samples (unsulphited and sulphited samples), and challenging samples (garlic and onions).

Table 2 demonstrates a comparison of the results in ten samples using each method for sulphur dioxide presence. Due to the different functionalities and restrictions of the R method, we have decided not to use it for our further analysis because of the false positive results in the unsulphited sample. Because the measurement is dependent on the creation of colour on the strip, samples that are coloured or that release colour during dilution may result in false positive results. Another disadvantage can be the sample's homogeneity, or any suspended particles in the test liquid, which can affect the result. The reflectoquant approach, on the other hand, is economical, quick, compact, and simple (Test for Determination of Sulfitess). The measurements obtained from the samples are within the allowed legal limit range of 50 to 2000 $\text{mg}\cdot\text{kg}^{-1}$ Commission Regulation (EU) No. 1129/2011.

All sulphited samples were positive for SO_2 presence across T and S methods with an R^2 of 0.99 indicating a strong positive linear relationship between them. The SO_2 concentration in the samples varied from 5.9 $\text{mg}\cdot\text{kg}^{-1}$ to 431.5 $\text{mg}\cdot\text{kg}^{-1}$ and from 6.0 $\text{mg}\cdot\text{kg}^{-1}$ to 338.6 $\text{mg}\cdot\text{kg}^{-1}$ in the S and T methods, respectively. In addition to, neither technique was able to detect SO_2 in five unsulphited samples. The statistical evaluation in Table 2 indicates that there was no significant difference ($P < 0.05$) between the T and S methods in sulphited samples DF1*, DF2*, DF4*, and DF5* and unsulphited samples DF6, DF7, DF8, DF9, and DF10. However, significant differences were observed in samples DF3* ($P < 0.05$), where the concentration in the DF3* by S method was higher than the other two methods. Whereas, sample DF4 by R method yield less than half of the SO_2 concentration than S and T method. In our analysis, we observe the sulphur-positive result in unsulphited sample DF9 (cranberry) by R method with concentrations of 80.2 $\text{mg}\cdot\text{kg}^{-1}$, which is believed to be caused by the presence of suspended particles and colour leach out from samples.

Table 3 is classified under the problematic category because of the false positive results associated with the *Allium* and *Brassica* vegetable genera. The results obtained from all the samples demonstrate the apparent existence of SO_2 through the testing methods. According to the statistical findings, the S method is significantly higher than the T and R methods throughout the challenging samples, except in the O2 sample and consistently produces higher concentration values. This is supported

Table 2. Determination of sulphur dioxide content in the dry fruit sample ($n = 5$ for each sample)

Sample name/ number	Titration method	Spectrophotometric method	Reflectoquant method	ANOVA (P -value)
DF1*	6.00 ± 0.55	5.94 ± 0.12	–	n.s.
DF2*	7.71 ± 0.33	7.09 ± 0.32	–	n.s.
DF3*	$116.70^b \pm 1.22$	$142.00^{ac} \pm 11.34$	$119.92^b \pm 7.10$	0.0228
DF4*	$338.56^c \pm 7.93$	$368.61^c \pm 15.84$	$172.29^{ab} \pm 11.18$	1.29×10^{-6}
DF5*	324.70 ± 48.42	431.50 ± 11.75	–	n.s.
DF6	< LOD	< LOD	< LOD	n.s.
DF7	< LOD	< LOD	–	n.s.
DF8	< LOD	< LOD	< LOD	n.s.
DF9	< LOD	< LOD	$80.21^{ab} \pm 7.63$	1.55×10^{-12}
DF10	< LOD	< LOD	–	n.s.

*Means samples with SO_2 labelled; ^{a–c}means in a row (difference between determination method) with a different superscript letter differing statistically ($P < 0.05$) as analysed by one-way analysis of variance (ANOVA); data are expressed as mean $\pm$ SD (standard deviation); n.s. – not significant; (–) – not analysed; DF – dry fruit; n – number of replicates; LOD – limit of detection

Table 3. Sulphur dioxide concentration in challenging samples (garlic and onion) ($n = 4$ for each sample)

Sample name/ number	Titration method	Spectrophotometric method	Reflectoquant method	ANOVA (P -value)
G1	$29.73^b \pm 2.72$	$662.54^{ac} \pm 37.03$	$54.08^b \pm 10.70$	2.97×10^{-8}
G2	$48.94^b \pm 7.10$	$939.46^{ac} \pm 219.14$	$26.24^b \pm 2.42$	3.36×10^{-5}
G3	$56.06^b \pm 4.96$	$1\,517.21^a \pm 54.48$	–	0.0007
G4	$35.85^b \pm 24.63$	$1\,363.90^a \pm 63.82$	–	5.28×10^{-5}
G5	$73.51^b \pm 5.54$	$1\,398.82^a \pm 79.01$	–	0.0018
O1	$7.54^{bc} \pm 0.71$	$52.46^a \pm 3.18$	$35.52^a \pm 2.63$	5.84×10^{-6}
O2	7.45 ± 0.66	10.67 ± 0.66	< LOD	n.s.
O3	$10.12^b \pm 1.62$	$271.15^a \pm 0.61$	–	2.20×10^{-5}
O4	$10.18^b \pm 0.99$	$289.86^a \pm 0.83$	–	1.06×10^{-5}
O5	$8.02^b \pm 0.38$	$301.81^a \pm 22.15$	–	0.0028

*Means samples with SO₂ labelled; ^{a-c}means in row (difference between determination method) with a different superscript letter differing statistically ($P < 0.05$) as analysed by one-way analysis of variance (ANOVA); data are expressed as mean $\pm$ SD (standard deviation); n.s. – not significant; (–) – not analysed; G – garlic; O – onion; n – number of replicates; LOD – limit of detection

by a strong coefficient of determination R^2 of 0.94 between the S and T techniques. Whereas, the T and R methods showed non-significant differences in samples G1 and G2 but produced significant differences in sample O1, with the R method producing higher concentration.

The measured concentration of SO₂ in garlic varies between 30 mg·kg⁻¹ and 1 517 mg·kg⁻¹. Among the garlic samples, four exceeded 1 000 mg·kg⁻¹, while sample G1 measured 663 mg·kg⁻¹ using the S method. In the T method, all measured values of the samples were found to be below 100 mg·kg⁻¹, with the highest concentration obtained in garlic powder being 74 mg·kg⁻¹ with an average of around 52 mg·kg⁻¹. Robbins et al. (2016) and Perfetti et al. (2003) have reported in their scientific literature that the content of SO₂ in the samples was determined at 90 mg·kg⁻¹ and over 200 mg·kg⁻¹ in certain garlic samples using the T method.

The concentration of SO₂ in onions ranges from 7 mg·kg⁻¹ to 302 mg·kg⁻¹. Notably, the S method yielded a significantly wider range of values, extending from 10 mg·kg⁻¹ to 302 mg·kg⁻¹ O2 and O5, respectively. Conversely, the results obtained using T methods demonstrated a relatively narrow concentration range, falling between 7 mg·kg⁻¹ and 10 mg·kg⁻¹ O2 and O4. Robbins et al. (2016) reported similar results, reporting 10 mg·kg⁻¹ SO₂ in an onion sample. The O2 exhibited comparable results in terms of its SO₂ concentration, with the S and T methods yielding the lowest values of 10 mg·kg⁻¹ and 7 mg·kg⁻¹, respectively.

This disparity in the obtained result can be attributed to the unsuitability of the S method for accurately

determining the concentration of SO₂ in the samples, particularly those containing endogenous sulphur compounds such as garlic. These extraction conditions and their interactions with the chemicals, or variants of them, exist in *Allium* species, explaining false positives and concentration differences (Block 1985). Robbins et al. (2016) compared the LC-MS/MS method with other titration methods for sulphite determination and demonstrated a reduced level of false positives and higher recovery from the LC-MS/MS method. Furthermore, Perfetti et al. (2003) and Lafeuille et al. (2007) have reported a close link between the creation of SO₂ and the pH of the analytical condition, as well as its interaction with the enzyme alliinase in garlic.

CONCLUSION

In conclusion, the R method is regarded as a simple and quick method for analysis due to its compact size, which facilitates ease of handling and cost-effectiveness. In contrast, this approach yields false positive results in coloured samples, even when they are free of sulphur.

The T method has a lower level of complexity in comparison to the S method. The method has good reproducibility and is acceptable for samples with an SO₂ level over 10 mg·kg⁻¹, except for samples suspected to include endogenous sulphur compounds. These samples pose a challenge in determining whether sulphite has been added to the sample. Another issue with this method is that it is very difficult to estimate the titration endpoint based only on the analyst's expe-

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rience. As a result, it might be challenging to assess the recovery of a low-concentration spike in a sample with a low SO₂ concentration.

The S method is efficient in terms of time consumption, which is 15 min for one sample compared to 30 min of just boiling at constant heat in the T method. This method demonstrates high levels of repeatability. This method offers greater flexibility from an analytical perspective, as the concentration may be readily altered to suit our preferences through the process of dilution. The low LOD and low LOQ of this procedure exceeds that of the R and T methods. Conversely, this method is not suitable for analysing endogenous sulphur-containing samples such as *Allium* and *Brassica* species. More research into more species, as well as additional commercially sulphited goods, would be required before this technology could be widely used for regulatory assessments. Once completely confirmed, these more specific, and robust techniques should aid in more efficient and dependable compliance with global sulphite regulations.

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