Application Note 54

Determination of Total and Free Sulfite in Foods and Beverages

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Key Words
Dionex IonPac ICE AS1 Column, Disposable Platinum Electrode, Ion-Exclusion Chromatography, Electrochemical Detection, PAD

Introduction

In the food and beverage industries, sulfites are a group of compounds that includes sulfur dioxide and sulfite salts. Sulfites can occur naturally in some foods and beverages due to fermentation. For centuries, sulfiting agents—such as sodium sulfite, sodium bisulfite, and sodium metabisulfite—have been used as preservatives to prevent microbial spoiling and browning reactions in a wide variety of food and beverage products.[1] Sulfiting agents can undergo a series of different reactions in food/beverage matrices, producing various species—including sulfite, bisulfite, metabisulfite, and other sulfite-related forms—that are either reversibly or irreversibly bound to food/beverage constituents, depending on the pH of the food/beverage.

Sulfites have been implicated as the cause of allergic reactions that range in severity from minor to life threatening. Sulfites also have been reported as the cause of some asthmatic responses in certain people.[2] Therefore, since 1986, the U.S. Food and Drug Administration (FDA) has required labeling of any food or beverage containing a sulfite concentration >10 ppm.[3] Since July 29, 2011, California has listed sulfur dioxide under Proposition 65 as a chemical known to cause reproductive toxicity.[4] To facilitate regulatory compliance, the food and beverage industries need a simple yet reliable method that offers the required selectivity and sensitivity to monitor low concentrations of sulfites in complex food/beverage matrices.

In foods and beverages, reversibly bound sulfite consists primarily of adducts with carbonyl compounds and hydroxysulfonates. These adducts are stable at low-to-intermediate pH and completely dissociate into free sulfite above pH 8.[5] The combined free and reversibly bound sulfite is referred to as total sulfite. Both free and total sulfite are of interest to the food and beverage industries. For instance, sulfite in wine combines with acetaldehyde to form the hydroxyl/sulfonate adduct with only a small proportion of the sulfite present as free sulfite.[6] Because the concentration of free sulfite is an important parameter in the wine industry, an analysis method is needed that accurately differentiates free from bound sulfite.

The optimized Monier-Williams Method (AOAC Method 990.28) has been widely used for determining the concentration of total sulfite in various food/beverage products.[7] However, the procedure is time-consuming, labor-intensive, and reportedly shows false positive responses.[8] A more efficient and accurate method (AOAC Method 990.31) to determine total sulfite was developed by Kim and Kim using alkaline extraction followed by ion-exclusion chromatography with direct current amperometric detection.[9, 10] However, loss of detector sensitivity frequently occurs, as the working electrode becomes contaminated. In order to minimize errors, the calibration standards and sample solutions must be analyzed sequentially.[11]

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The previous, now archived, version of Dionex (now part of Thermo Scientific) Application Note (AN) 54 describes a modification of AOAC Method 990.31 that uses pulsed amperometric detection (PAD) to overcome the working electrode fouling problem.[9] Although PAD extends the lifetime of a working electrode, when a decrease in detection response does occur, time-consuming and labor-intensive electrode polishing is required to restore response. In this updated study, a disposable platinum (Pt) working electrode is used in place of a conventional electrode for the detection of sulfite.[12] Other improvements include replacing the 9 mm Thermo Scientific™ Dionex™ IonPac™ ICE-AS1 column set with a 4 mm column set to decrease eluent consumption, and replacing the sulfuric acid eluent with less corrosive methanesulfonic acid (MSA).

Goal

To develop an improved ion-exclusion chromatography method for the determination of total and free sulfite in foods and beverages

Equipment

- Thermo Scientific™ Dionex™ ICS-5000 system, including:
  - DP Dual Pump*
  - DC Detector/Chromatography Compartment
  - Dionex™ AS-AP Autosampler with tray temperature control option; Sample Syringe, 250 µL (P/N 074306); 1200 µL Buffer Line (P/N 074989); Vial Kit, Polystyrene, 10 mL (with caps and blue septa, P/N 074228)
  - ED Electrochemical Detector (without cell, P/N 072042)
  - ED Cell (no reference electrode or working electrode, P/N 072044)
  - pH, Ag/AgCl Reference Electrode (P/N 061879)
  - Platinum Disposable Electrodes (P/N 064440)
  - Knitted Reaction Coil, 375 µL (P/N 043700)
- ¼-28 to 10-32 Union (P/N 042806)
- Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System software version 7.1

** An SP Single Pump can also be used.*

Reagents and Standards

- Deionized (DI) Water, Type I reagent grade, 18 MΩ-cm resistance or better
- Sodium Sulfite, 98+%, anhydrous, ACS reagent (Fisher Scientific P/N AC42443)
- D-Mannitol, 98+% (Fisher Scientific P/N AC12534)
- Sodium Phosphate, dibasic, 99+%, anhydrous (Fisher Scientific P/N AC20485)
- Methanesulfonic Acid, 99%, extra pure (Fisher Scientific P/N AC12561)

Consumables

- Thermo Scientific™ Nalgene™ Rapid-Flow™ Sterile Disposable Filter Units with Nylon Membrane, 1 L, pore size: 0.2 µm (Fisher Scientific P/N 09-740-46)
- Fisherbrand™ Easy Reader™ Plastic Centrifuge Tubes, polypropylene, 15 mL (Fisher Scientific P/N 07-200-886)
- Nalgene Syringe Filters, acrylic housing, 25 mm, pore size: 0.2 µm (Fisher Scientific P/N 09-740-61A)
- AirTite™ All-Plastic Norm-Ject™ Syringes, 5 mL, sterile (Fisher Scientific P/N 14-817-28)
- WHEATON® 20 mL High-Density Polyethylene (HDPE) Liquid Scintillation Vials (Fisher Scientific P/N 03-341-72C)

Samples

- Red Wine
- Rosé Wine
- Coconut Water
- Dried Apricot
- Nutritional Bar

Conditions

Columns:	Dionex IonPac ICE-AS1 Guard, 4 × 50 mm (P/N 067842) Dionex IonPac ICE-AS1 Analytical, 4 × 250 mm (P/N 064198)
Eluent:	20 mM MSA
Temperature:	25 °C (upper compartment, detector) 30 °C (lower compartment, column)
Tray Temp:	4 °C
Detection:	PAD, disposable Pt working electrode
System Backpressure:	~1760 psi
Background:	50–60 nC
Noise:	0.04 nC peak-to-peak
Run Time:	25 min

Waveform for the ED:

Time (s)	Potential (V)	Last Step*	Ramp*	Gain Region*	Integration
0.00	0.8	Off	On	Off	Off
0.40	0.8	Off	On	On	On
0.60	0.8	Off	On	On	Off
0.61	1.2	Off	On	Off	Off
0.70	1.2	Off	On	Off	Off
0.71	0.1	Off	On	Off	Off
1.00	0.1	On	On	Off	Off

**These settings are required in the Dionex ICS-3000/5000 system but not used in older Dionex systems; the reference electrode is in AgCl mode (Ag/AgCl reference electrode).*

Preparation of Solutions and Reagents

MSA Eluent, 20 mM

Add 1.92 g of concentrated (99%) MSA to a 1 L volumetric flask containing 900 mL of degassed DI water. Bring to volume with degassed DI water and mix thoroughly. Transfer the solution to a glass eluent bottle and pressurize it with helium or nitrogen.

Acidic Extraction Solution, 20 mM MSA/10 mM Mannitol, pH 2

Dissolve 1.82 g of D-mannitol in 800 mL degassed DI water in a 1 L volumetric flask, add 1.92 g concentrated MSA, and bring to volume with degassed DI water. Filter through a Nalgene 0.2 µm filter unit. Store at 4 °C for up to a week.

Note: This solution tends to reabsorb oxygen from the air. An oxygen dip is observed in the chromatograms of samples prepared with extraction solution that is older than a week.

Alkaline Extraction Solution, 20 mM Na_2HPO_4/10 mM Mannitol, pH 9

Dissolve 2.84 g of sodium phosphate dibasic and 1.82 g of D-mannitol in 900 mL of degassed DI water in a 1 L volumetric flask, then bring to volume with degassed DI water. Filter through a Nalgene 0.2 µm filter unit. Store at 4 °C for up to a week.

Note: This solution tends to reabsorb oxygen from the air. An oxygen dip is observed in the chromatograms of samples prepared with extraction solution that is older than a week.

Sulfite Stock Solution, 1000 mg/L

Prepare a 1000 mg/L stock solution of sulfite by accurately weighing 158 mg of sodium sulfite. Transfer to a 100 mL volumetric flask and dilute to volume with alkaline extraction solution for determination of total sulfite or with acidic extraction solution for determination of free sulfite. Prepare the stock solution daily.

Preparation of Sulfite Standard and Samples

Prepare fresh standards daily and handle all samples and standards with caution to reduce air exposure and minimize oxidation. In addition, analyze unpreserved samples immediately to improve the accuracy of the results. When preparing sulfite standards or samples, remove an aliquot from the interior central portion of the stock solution or food/beverage sample, where it is least exposed to oxygen.

Sulfite Primary Standard, 100 mg/L

Transfer 10 mL of 1000 mg/L sulfite stock solution to a 100 mL volumetric flask and dilute to volume with the alkaline extraction solution for total sulfite determination, or with the acidic extraction solution for free sulfite determination.

Sulfite Calibration Standard Solutions

Prepare calibration standard solutions daily by diluting the 100 mg/L sulfite primary standard as required with the alkaline extraction solution for total sulfite determination, or with the acidic extraction solution for free sulfite determination.

Sample Preparation

Red Wine

Transfer 19.8 mL of alkaline extraction solution and 0.2 mL of red wine to a 20 mL HDPE vial for total sulfite determination. Transfer 19.6 mL of acidic extraction solution and 0.4 mL of red wine to a 20 mL HDPE vial for free sulfite determination.

Rosé Wine

Transfer 19.8 mL of alkaline extraction solution and 0.2 mL of rosé wine to a 20 mL HDPE vial for total sulfite determination. Transfer 19.2 mL of acidic extraction solution and 0.8 mL of rosé wine to a 20 mL HDPE vial for free sulfite determination.

Coconut Water

Transfer 9 mL of alkaline extraction solution and 1 mL of coconut water to a 20 mL HDPE vial for total sulfite determination. Transfer 9 mL of acidic extraction solution and 1 mL of coconut water to a 20 mL HDPE vial for free sulfite determination.

Dried Apricot

Homogenize 100 g of dried apricot with a blender at high speed. Weigh 0.4 g of the homogenized dried apricot in a 15 mL centrifuge tube. Add 10 mL of alkaline extraction solution. Centrifuge the sample at 6600 rpm at 2 °C for 5 min. Mix 1 mL of the resulting supernatant with 4 mL of alkaline extraction solution before injection for total sulfite determination. For free sulfite, follow the same procedure as for the total sulfite determination but use 10 mL of acidic extraction solution in place of the alkaline extraction solution and inject the resulting undiluted supernatant.

Nutritional Bar

Homogenize 100 g of the nutritional bar with a blender at high speed. Weigh 1 g of the homogenized nutritional bar in a 15 mL centrifuge tube. Add 10 mL of alkaline extraction solution. Centrifuge the sample at 6600 rpm at 2 °C for 10 min. Filter the supernatant through a 0.2 µm polyethersulfone syringe filter before injection for total sulfite determination. Follow the same procedure using 10 mL of acidic extraction solution in place of the alkaline extraction solution for free sulfite determination.

System Preparation and Configuration

Install and configure the Dionex AS-AP Autosampler in Push Mode. Follow the instructions in the Dionex AS-AP Autosampler Operator's Manual (Document No. 065361) to calibrate the sample transfer line to ensure accurate and precise sample injections. Prepare a 30 µL sample loop by measuring approximately 5.8" (14.7 cm) of 0.020" i.d. tubing. To verify the volume of the loop, weigh the empty tubing, fill the tube with DI water, then reweigh the filled tube, and calculate the volume. The total sample volume must be 30 µL ± 5%. Install the sample loop on the injection valve in the lower compartment of the DC Detector/Chromatography Compartment.

Install the Dionex IonPac ICE-AS1 guard and analytical columns in the lower compartment of the DC Detector/Chromatography Compartment. Connect a 375 µL reaction coil at the outlet of the Dionex IonPac ICE-AS1 analytical column using 0.005 in. i.d. PEEK™ tubing and a ¼-28 to 10-32 union. Install the electrochemical cell in the upper compartment and connect the cell inlet to the outlet of the reaction coil. Use 0.005" i.d. PEEK tubing for all the analytical flow paths and keep the lengths of the connecting tubing to a minimum. Refer to Dionex ICS-5000⁺ Ion Chromatography System Installation Instructions (Document No. 065447) for detailed instructions for assembling the electrochemical cell. Refer to the Dionex Disposable Platinum Electrode Installation Guide (Document No. 065139-01) for detailed instructions for installation of disposable working electrodes. After installing a disposable Pt electrode, install a pH, Ag/AgCl reference electrode and perform a calibration procedure using pH 7 and pH 4 buffers (refer to Document No. 065447, Sec. B.1.4, for detailed instructions).

Results and Discussion

The selectivity of this method for both separation and detection is demonstrated. The separation using the Dionex IonPac ICE-AS1 column is achieved by a combination of Donnan exclusion, partitioning, and size-exclusion mechanisms.[13] The Donnan exclusion mechanism causes stronger acid anions to elute before weaker ones according to increasing pKa. When a strong acid eluent is used, sulfite is converted to a weak acid (i.e., H_2SO_3) and therefore is retained on the column, while the stronger acid anions from the matrix are unretained.

Electrochemical detection is more selective than conductivity detection because only compounds that are oxidized at a selected potential are detected. Therefore, this method is less subject to interference from complex food/beverage matrices. The pulse sequence of PAD constantly cleans the working electrode and minimizes electrode fouling. However, once the electrode is fouled by contaminants, the electrode surface requires reconditioning through proper polishing.

To eliminate the need for time-consuming and labor-intensive polishing, a disposable Pt electrode was used instead of the conventional Pt electrode described in the archived version of AN 54.[12] The performance of a conventional Pt electrode was also examined under the same experimental conditions in this study, and better sensitivity was obtained using the disposable electrode. Installation of a reaction coil after the column removed trace amounts of oxygen in the eluent and generated a lower background signal. The reduction in oxygen decreased the oxygen dip caused by the difference in oxygen concentration between the sample and the eluent, which would otherwise interfere with the quantification of sulfite.

Method improvement was also achieved through the choice of eluent. The sulfuric acid used in the archived version of AN 54 was replaced by MSA. No significant difference in separation and sensitivity was observed when using the same eluent concentration of the two acids. Because MSA is relatively safer to handle than sulfuric acid, MSA was used for this study. Under the suggested experimental conditions, the equilibrated system typically shows a background charge of 50–60 nC and a retention time of ~13.6 min for sulfite (Figure 1).

Columns:	Dionex IonPac ICE-AS1 Guard/Analytical, 4 mm set
Eluent:	20 mM MSA
Flow Rate:	0.2 mL/min
Inj. Volume:	30 µL
Temperature:	30 °C
Detection:	PAD, disposable Pt electrode
Samples:	A) Sulfite standard in acidic extraction solution B) Sulfite standard in alkaline extraction solution

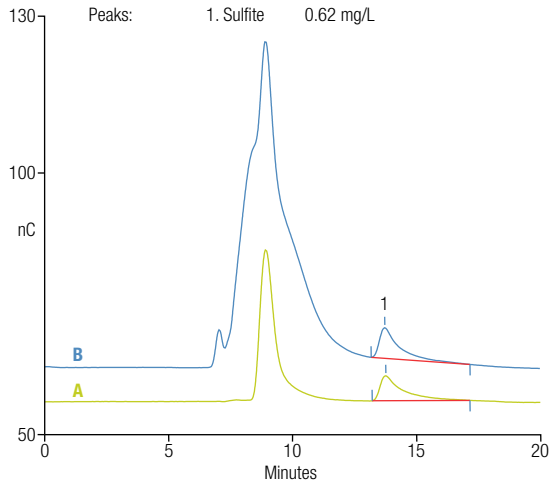

Figure 1. Sulfite standard (0.62 mg/L) in acidic and alkaline sample buffers. A 4% signal offset has been applied.

Sample Preservation

In solution, sulfite is rapidly oxidized to sulfate. Therefore, mannitol was used to reduce the oxidation of sulfite, allowing solution stability for ~8 h. However, stability can be affected by other sample components and solution pH.[14]

One study demonstrated that carbonyl/sulfite adducts are most stable at pH ~2, whereas dissociation of the adducts occurs at pH >8.[5] Therefore, free sulfite is determined by treating the food sample with acid at room temperature (treatment using acid and heat can release bound sulfite, as in the Monier-Williams method) and total sulfite is determined by releasing the bound sulfite in alkaline extraction solution. Here, alkaline extraction was used to treat the food/beverage samples for determination of total sulfite, and an acidic extraction solution (pH 2) was used for determination of free sulfite.

Calibration, Limit of Detection, Limit of Quantification

Sulfite has a slight response difference when prepared in alkaline, compared to acidic, extraction solutions. To ensure measurement accuracy, the determinations of total and free sulfite were calibrated separately using sulfite standards prepared in alkaline and acidic extraction solutions, respectively. The sulfite concentration in this method was quantified by peak height. The baseline noise was determined by measuring peak-to-peak noise in a representative 1 min segment of the baseline where no peaks elute. The signal of 0.2 mg/L sulfite standard was used to determine the limit of detection (LOD) and limit of quantification (LOQ) for total and free sulfite, respectively. The summary of the calibration, LOD, and LOQ is shown in Table 1. Chromatograms of sulfite standards prepared in alkaline and acidic extraction solutions are shown in Figure 1, Traces A and B, respectively.

Table 1. Calibration data and method detection limits of sulfite.

Analyte	Range (mg/L)	Coefficient of Determination (r^2)[a]	LOD (mg/L)[b]	LOQ (mg/L)[c]
Total Sulfite[d]	0.2–10	0.9960	0.039	0.13
Free Sulfite[e]	0.1–5	0.9994	0.028	0.093

[a]Quadratic fit
[b]LOD = 3 × signal-to-noise ratio (S/N)
[c]LOQ = 10 × S/N
[d]Prepared in alkaline extraction solution
[e]Prepared in acidic extraction solution

Sample Analysis

To minimize any sample matrix effect and thereby obtain good sulfite recoveries, an appropriate sample dilution is required. Wine samples are relatively acidic due to the presence of naturally occurring organic acids and acidic byproducts from fermentation. Proper dilution with alkaline extraction solution is therefore needed to achieve the effective release of the bound sulfite in the sample extract of pH 9.[15]

The red and rosé wines in this study were treated with a 100-fold dilution of the alkaline extraction solution to obtain an extract of approximately pH 9 for accurate determination of total sulfite. The total sulfite was 72.4 mg/L and 145 mg/L in the red and rosé wines, respectively. Using a 50-fold dilution with the acidic extraction solution, the free sulfite was 43.6 mg/L in the red wine. Using a 25-fold dilution in the acidic extraction solution, 38.1 mg/L free sulfite was found in the rosé wine, which is considerably lower than the concentration of total sulfite. Figure 2 shows the chromatograms of total and free sulfite in the red wine sample. The chromatograms of total sulfite in unspiked and spiked rosé wine are shown in Figure 3.

Columns: Dionex IonPac ICE-AS1 Guard/Analytical, 4 mm set
Eluent: 20 mM MSA
Flow Rate: 0.2 mL/min
Inj. Volume: 30 µL
Temperature: 30 °C
Detection: PAD, disposable Pt working electrode

Samples: A) Total sulfite, 100-fold dilution
B) Free sulfite, 50-fold dilution

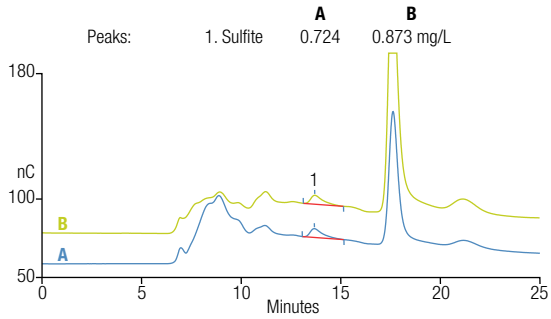

Figure 2. Chromatograms of A) total and B) free sulfite in red wine. A 15% signal offset has been applied.

Columns: Dionex IonPac ICE-AS1 Guard/Analytical, 4 mm set
Eluent: 20 mM MSA
Flow Rate: 0.2 mL/min
Inj. Volume: 30 µL
Temperature: 30 °C
Detection: PAD, disposable Pt working electrode

Samples: A) Rosé wine, 100-fold dilution
B) Rosé wine, 100-fold dilution spiked with 1.44 mg/L sulfite

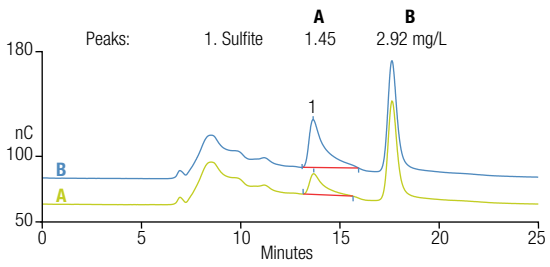

Figure 3. Chromatograms of total sulfite in A) unspiked and B) spiked rosé wine. A 15% signal offset has been applied.

Coconut water contains a variety of nutrients including vitamins, minerals, amino acids, and other organic compounds. The pH of the coconut water in this study was ~5. Therefore, a 10-fold dilution in the alkaline extraction solution was suitable to increase the pH to 9 and extract the total sulfite, which was 3.80 mg/L. Using the same dilution factor for the acidic extraction solution, free sulfite was 3.71 mg/L. Figure 4 shows the chromatograms of total and free sulfite in coconut water.

In the dried apricot sample, the concentration of free sulfite was 16.7 mg/L, which is significantly lower than 304 mg/L for total sulfite. Chromatograms of the total and free sulfite in dried apricot are shown in Figure 5.

A nutritional bar fortified with vitamins and minerals was also subjected to free and total sulfite analyses. Due to the nature of the sample, filtration was required to remove the small particles from the supernatant following centrifugation. Only 3.97 mg/L total sulfite and 2.67 mg/L free sulfite were found in the sample. Figure 6 shows the separation of sulfite and other sample components in the nutritional bar.

The results of total and free sulfite found in the selected food/beverage samples are summarized in Table 2.

Table 2. Summary of total and free sulfite contents in food and beverage samples.

Sample	Total Sulfite		Free Sulfite	
	Calculated Concentration (mg/L)	Dilution	Calculated Concentration (mg/L)	Dilution
Red Wine	72.4	1:100	43.6	1:50
Rosé Wine	145	1:100	38.1	1:25
Coconut Water	3.80	1:10	3.71	1:10
Dried Apricot	304	1:125	16.7	1:25
Nutritional Bar	3.97	1:10	2.67	1:10

Columns: Dionex IonPac ICE-AS1 Guard/Analytical, 4 mm set
Eluent: 20 mM MSA
Flow Rate: 0.2 mL/min
Inj. Volume: 30 µL
Temperature: 30 °C
Detection: PAD, disposable Pt working electrode

Samples: A) Free sulfite, 10-fold dilution
B) Total sulfite, 10-fold dilution

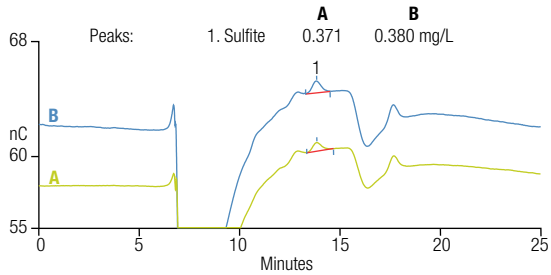

Figure 4. Chromatograms of A) free and B) total sulfite in coconut water. A 50% signal offset has been applied.

Columns: Dionex IonPac ICE-AS1 Guard/Analytical, 4 mm set
Eluent: 20 mM MSA
Flow Rate: 0.2 mL/min
Inj. Volume: 30 µL
Temperature: 30 °C
Detection: PAD, disposable Pt working electrode

Samples: A) Free sulfite, 25-fold dilution
B) Total sulfite, 125-fold dilution

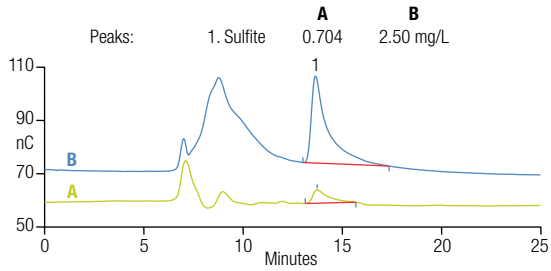

Figure 5. Chromatograms of A) free and B) total sulfite in dried apricot. A 20% signal offset has been applied.

Columns: Dionex IonPac ICE-AS1 Guard/Analytical, 4 mm set
Eluent: 20 mM MSA
Flow Rate: 0.2 mL/min
Inj. Volume: 30 µL
Temperature: 30 °C
Detection: PAD, disposable Pt working electrode

Samples: A) Free sulfite, 10-fold dilution
B) Total sulfite, 10-fold dilution

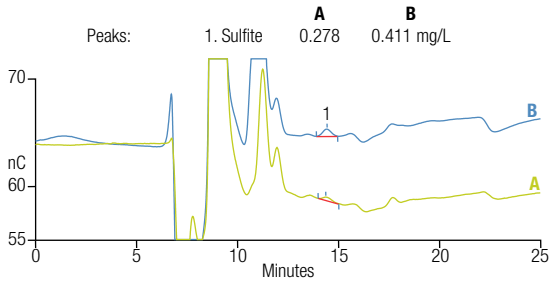

Figure 6. Chromatograms of A) total and B) free sulfite in a nutritional bar. A 30% signal offset has been applied.

Table 3. Sulfite recoveries in food and beverage samples.

Sample	Total Sulfite				Free Sulfite			
	Found (mg/L)	Added (mg/L)	Total Found (mg/L)	Recovery (%)	Found (mg/L)	Added (mg/L)	Total Found (mg/L)	Recovery (%)
Red Wine	0.724	0.713	1.48	105	0.873	0.895	1.90	115
Rosé Wine	1.45	1.45	2.92	102	1.52	1.52	3.12	106
Coconut Water	0.380	0.372	0.685	82.1	0.371	0.395	0.762	98.8
Dried Apricot	2.50	2.36	4.48	84.2	0.704	0.728	1.50	109

Recoveries and Precision Study

To validate this method, recovery of sulfite from the different samples was studied. Each sample was spiked with a sulfite concentration comparable to the endogenous concentration. For the wine and coconut water samples, a known sulfite concentration was added to the sample before diluting with extraction solution. For the solid samples, a known sulfite concentration was added to the solid before adding the extraction solution. The effect of the matrix on recoveries from the samples was examined and the optimum dilution conditions are reported in Table 2.

Recoveries in the range of 82.1–115% were obtained for all selected food/beverage samples (Table 3) except the nutritional bar, which produced no sulfite recovery. This is possibly due to the combination of a matrix effect and instability of the low concentrations of sulfite. Sufficient dilution was required to minimize interference from the complex matrix components, and the resulting low sulfite concentration is more prone to oxidation during sample preparation. Overall, these experiments demonstrate that all results are accurate, with the exception of the nutritional bar.

Precision was evaluated from the peak height RSDs of sulfite standards and selected food/beverage samples from seven successive injections. The intraday precision of peak height RSDs for 0.5 mg/L sulfite prepared in alkaline and acidic extraction solutions were 0.29% and 2.14%, respectively. The peak height RSDs of red wine spiked with 0.895 mg/L sulfite and rosé wine spiked with 1.52 mg/L sulfite were 1.97% and 2.40%, respectively. These data indicate good method precision.

Electrode Stability

This study used the same waveform described in the archived version of AN 54, which was originally developed using a conventional Pt electrode. Therefore, stability of the disposable Pt electrode was evaluated using a 1.2 mg/L sulfite standard that was prepared fresh daily (Figure 7). The response of this standard was continuously evaluated over two weeks while total and free sulfite were determined in various food/beverage samples. Considering the interday variations in the sulfite standard preparation and the manual preparation of the eluent, an RSD of 15.7% (n = 6) for the peak height precision of free sulfite and an RSD of 8.63% (n = 4) for the peak height precision of total sulfite demonstrates good stability of the Pt disposable electrode used with this waveform.

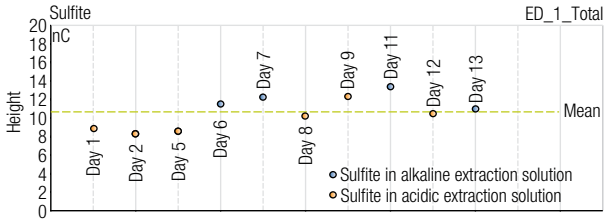

Figure 7. Electrode stability study over two weeks of continuous use. Signal of 1.2 mg/L sulfite standard.

Conclusion

This study demonstrates the determination of free and total sulfite in a selection of food and beverage samples. This method replaces the conventional Pt working electrode described in the archived version of AN 54 with a disposable Pt working electrode, which demonstrates good stability and does not require polishing. A smaller-dimension column set is used, which operates with a lower flow rate that significantly reduces eluent consumption. This updated method also can be applied to food and beverage samples with lower sulfite concentrations than those addressed in the archived version of AN 54.

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