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## Measurement of Trace Amounts of Beryllium in Olive Leaves by Graphite-Furnace Atomic Absorption Spectrophotometry Using Acetylacetonone as a Chelating Agent

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### Abstract

Trace amounts of Be (0.006 - 0.76 ng) in a dried olive leaf sample (20 mg) could be accurately determined by GFAAS after treating with two-stage microwave digestion (HNO<sub>3</sub>/H<sub>2</sub>O<sub>2</sub>) at 85°C for 10 min and using acetylacetonone as a chelating agent in the presence of an acetate buffer (pH 6.0). The method detection limit (MDL, 3σ) for Be was found to be 0.3 ng/g; the calibration graph was linear up to 38.0 ng/g. The Be contents measured in five olive leaf samples (BCR CRM No. 62 and four samples collected in Taiwan) were between 3.6 and 28.0 ng/g. Good spiked recoveries (97.2 - 101.0%) were obtained for these five olive leaf samples with a relative standard deviation (RSD, n = 3) < 3.0%. The proposed method could be applied to measurements of Be in olive leaves.

**Keywords:** olive leaves, beryllium, acetylacetonone, graphite-furnace atomic absorption spectrophotometry

### 1 Introduction

Olive plants provide biophenols [1] which may function as antioxidants for peroxy radicals and play a role for human skin protection. The antioxidants extracted from olive leaves are about 30 times more effective than that from orange or tea leaves [2]. The contents of beryllium (Be) in the earth's crust [3], in soil [4], and in coal [5] are about 2 - 6, 1.2 - 2.1, and 2.5 μg/g, respectively. Pure Be and its metal alloys (such as Cu, Al, Mg,

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Ni) have been widely used for aircraft, electrical equipment, and electronic instrumentation [6,7]. By way of rain, Be and its compounds in the earth's crust, in soil, or in gaseous vapor and fly ash [8,9] discharged from coal-burning or metal alloy factories may be dissolved in water. Thus, trace amounts of Be may be absorbed by the roots and finally accumulate in olive leaves.

Be and its compounds may cause lung disease (berylliosis) through inhalation, or cause body weight loss through diet. The concentration of Be in air is regulated not to exceed  $2 \mu\text{g}/\text{m}^3$  for the 8-h average permissible exposure limit by OSHA [10], Japan [6], and Taiwan government [11]; while in fish tissue, the risk level is  $84 \text{ ng}/\text{g}$  suggested by USEPA [12]. The levels of Be in olive leaf samples may be very low and have not been reported in olive leaf certified reference material (e.g. BCR CRM No. 62) nor in the literature until present. In the present work, a small sample size (20 mg) of dried olive leaf powder was completely dissolved in  $\text{HNO}_3/\text{H}_2\text{O}_2$  by two-stage microwave digestion. The dissolved Be(II) in the sample was reacted with acetylacetone [6, 13 -17] to form the chelate,  $[\text{Be}(\text{acac})_2]$ , preconcentrated on two home-made oasis cartridges, and then eluted with methanol. Hence trace amounts of Be in an olive leaf sample could be accurately determined by graphite-furnace atomic absorption spectrophotometry (GFAAS).

## 2 Experimental

### 2.1 Apparatus

A Hitachi Z-2700 graphite-furnace atomic-absorption spectrophotometer, equipped with a Zeeman background corrector, was used for an atomic-absorption measurement of Be at  $234.9 \text{ nm}$  with a slit width of  $1.3 \text{ nm}$ . A hollow-cathode lamp of Be (Hitachi Co., Japan) was operated at  $10 \text{ mA}$ . Pyrolytic graphite tube cuvettes (No. 7J0-8880, Hitachi Co., Japan) were purchased. A MARS-5 microwave accelerated reaction system (CEM Co., USA), equipped with a temperature-controlled sensor, was used for the microwave digestion of Be in olive leaf samples. During microwave digestion, each  $7\text{-mL}$  teflon microvessel was placed in a  $90\text{-mL}$  teflon PFA vessel that contained about  $9.3 \text{ mL}$  of pure water for samples (or  $10.0 \text{ mL}$  of pure water for a temperature-controlled sensor).

### 2.2 Reagents and solutions

All chemicals used were of analytical reagent grade or better. Nitric acid (double distilled) was purchased from Fisher Chemical Co., USA. Acetylacetone (99%, w/w) was purchased from Tokyo Chemical Industrial Co., Japan. Methanol and hydrogen peroxide (35%, w/w) were purchased from Merck, Germany. A stock standard solution of  $1000 \text{ mg}/\text{L}$  of Be(II), which was an ICP standard traceable to SRM 3105a (lot 892707) from NIST, was obtained from Merck. Its composition was beryllium acetate  $[\text{Be}_4\text{O}(\text{C}_2\text{H}_3\text{O}_2)_6]$  in superpure  $\text{HNO}_3$  (2 - 3%), certified as  $1001 \pm 8 \text{ mg}/\text{L}$  of Be by ICP-OES. A working standard solution ( $100 \mu\text{g}/\text{L}$ ) of Be(II) was prepared by diluting the stock solution with methanol. Ammonium hydroxide (microelectronic grade) was

purchased from J. T. Baker, Inc. (Phillipsburg, NJ, USA). Ammonium acetate (Merck) and acetic acid (99.99%, Sigma and Aldrich) were used to prepare an acetate buffer in an aqueous solution and in methanol, respectively.

### 2.3 Samples of olive leaves

An olive leaf BCR CRM No. 62 with an uncertified value for Be was purchased from the Commission of the European Communities (Luxembourg). Four real samples of olive leaves were collected from central and northern Taiwan. They all belong to *Elaeocarpus Serratusl.* No. 1 was from Tunghai University campus (Taichung, in the middle of Taiwan); No. 2 was from Tenwei (Changhua, about 30 km in the south of Taichung); No. 3 was from Paoshan (Hsinchu, about 60 km north of Taichung); No. 4 was from Dashi (Taoyuan, about 80 km north of Taichung). Among them, No. 3 is about 6 km southeast of the Hsinchu Industrial Science Park which may have factories manufacturing Be-alloy equipment for electronics and instrumentation.

### 2.4 Sample preparation

The procedure used was referred to that of BCR CRM No. 62 and was slightly modified. The real samples collected were thoroughly washed with pure water, air dried, and then placed in an oven (110 °C) for 4 h. After cooling to room temperature, the treated olive leaves were ground into powder with a mortar and pestle to pass through a 420 µm (40 mesh) sieve stainless-steel screen in the laboratory. Each of the powdered samples was stored in a brown glass vial (10 mL) with a teflon-lined screw cap. The BCR CRM No. 62 powder was used as provided without further treatment. In order to make sure that a dry basis was employed, all samples (about 1 g) were placed in a vacuum desiccator at room temperature over magnesium perchlorate (Merck) for at least 24 h before weighing.

### 2.5 Oasis cartridge preparation and pretreatment

Oasis particles (HLB, Waters Association Co.) are copolymers [18] consisting of hydrophilic (N-vinyl pyrrolidinone) and lypophilic (divinylbenzene) monomers with a 1:1 ratio. The polarity of Oasis particles is relatively small comparing with water and is classified as reversed-phase.

Each Oasis cartridge was prepared by packing 150 mg Oasis particles in a 1 mL polypropylene pipet tip. Prior to preconcentrating for Be(acac)<sub>2</sub> in an aqueous solution, each Oasis cartridge was conditioned with methanol (2 mL) and then pure water (10 mL).

### 2.6 Analytical procedure for Be in olive leaves

An amount (20 mg) of dried olive leaf sample was accurately weighed to ± 0.1 mg and placed in a 7-mL teflon microvessel. For spiked recovery tests or the standard addition method, appropriate amounts (0 - 0.300 ng) of Be [100 µg/L of Be(II) prepared in methanol] were added to the samples. After being left standing overnight to allow the methanol to evaporate, a two-stage microwave digestion procedure using conc. HNO<sub>3</sub> and H<sub>2</sub>O<sub>2</sub>, respectively, was performed.

After cooling to room temperature, each digested sample was transferred to a beaker (200 mL) and its pH was adjusted to about 5 - 6 by NH<sub>3</sub>(aq). Appropriate amounts of

ammonium acetate buffer and acetylacetone were added to the solution. The mixture was allowed to react at room temperature for about 1 h [14,16,17] to form a chelate of  $[\text{Be}(\text{acac})_2]$ . The chelate was preconcentrated on two home-made Oasis cartridges in series. Each cartridge was eluted with methanol, and adjusted to 1.00 mL in a small graduated glass test tube (2.00 mL). In order to prolong the use of the pyrolytic tube, usually a 1:1 ratio of methanol solution in cartridge 1 and cartridge 2 was mixed before introducing 20  $\mu\text{L}$  of the methanol solution into a graphite tube. A suitable temperature program was used to atomize Be. The peak heights in absorbance multiplied by a factor of two equal the sum of the absorbance in cartridges 1 and 2. The net absorbance values were used for a quantitative analysis after correction for each corresponding blank.

### 3. Results and Discussion

Since the Be content in BCR CRM No. 62 was small (about 0.20 ng) for a 20 mg dried olive leaf sample, an appropriate amount (0.200 ng) of Be(II) in methanol was spiked on each sample in order to compare the effects of the following parameters more clearly.

#### 3.1 Temperature program used for GFAAS

In order to prevent the splashing of the sample and to remove ammonium salts during the heating processes, very slow drying step (60 - 110°C, 40 s) and first ashing step (120 - 400°C, 40 s) were performed. The effect of the second ashing temperatures (600 - 1000°C for 40 s) and the atomization temperatures (2000 - 2600°C for 2 s) on the absorbance with and without a chelating agent (acetylacetone plus pH 6.0 acetate buffer) was tested with 4.0 pg Be(II) in 20  $\mu\text{L}$  of a methanol solution [which was prepared by spiking 0.200 ng of Be(II) on 20 mg of olive leaves BCR No. 62, and finally concentrating to 1.00 mL in methanol for cartridge 1 and cartridge 2, respectively. A 1 : 1 ratio of the methanol solution was mixed and 20  $\mu\text{L}$  was used]. Figure 1 indicates that when acac was not added into the digested olive leaf sample solution, the  $\text{Be}(\text{acac})_2$  chelate could not form in the sample solution. Thus, Be could not be adsorbed on the Oasis cartridges when the sample solution passed through them. After the Oasis cartridges were eluted with methanol, Be was not included in the methanol solution. Hence, no absorbance signal was observed during the pyrolysis. In the presence of acac and acetate buffer, the  $\text{Be}(\text{acac})_2$  chelate was formed. During ashing, the absorbance was the same from 600°C to 650°C for 40 s and decreased above 650°C. During atomization, the absorbance increased as the temperature increased from 2000 to 2500°C for 2 s and was the same at 2600°C. Hence, suitable ashing (650°C) and atomization (2500°C) temperatures were used, as tabulated in Table 1.

#### 3.2 Conditions used for microwave digestion

Concentrated  $\text{HNO}_3$  (650  $\mu\text{L}$ ) and  $\text{H}_2\text{O}_2$  (50  $\mu\text{L}$ ) in one-stage and two-stage microwave digestion procedures were compared. The results indicate that the color of the sample solution was not as clear as that in the two-stage digestion, and the absorbance

was about 10% lower than the two-stage digestion. Hence, two-stage microwave digestion was used.

The effect of the amounts (500 - 700  $\mu\text{L}$ ) of concentrated  $\text{HNO}_3$  and  $\text{H}_2\text{O}_2$  (0 - 70  $\mu\text{L}$ ) for digesting a spiked olive leaf sample, BCR No. 62 (mentioned above), on the absorbance was tested with 4.0 pg Be(II) in 20  $\mu\text{L}$  of the methanol solution. Figure 2 indicates that when 50  $\mu\text{L}$  of  $\text{H}_2\text{O}_2$  was used, the relatively large absorbance value was observed at 650  $\mu\text{L}$ ; further, when 650  $\mu\text{L}$  of conc.  $\text{HNO}_3$  was used, the absorbance increased as the amount of  $\text{H}_2\text{O}_2$  was increased from 0 to 50  $\mu\text{L}$ , and decreased as the amount of  $\text{H}_2\text{O}_2$  was increased from 50 to 75  $\mu\text{L}$ . Hence, 650  $\mu\text{L}$  conc.  $\text{HNO}_3$  and 50  $\mu\text{L}$  of  $\text{H}_2\text{O}_2$  were selected for use in this study.

The effects of the digestion temperatures (80 to 90°C holding for 10 min) and the digestion times (5 - 20 min at 85°C) using concentrated  $\text{HNO}_3/\text{H}_2\text{O}_2$  on the absorbance were tested with 4.0 pg Be in 20  $\mu\text{L}$  of the methanol solution. The results indicate that the absorbance increased when the digestion temperature was increased from 80 to 85°C, and then decreased above 90°C; further, the absorbance increased when the digestion time increased from 5 to 10 min at 85°C, and then decreased when the digestion time was longer than 10 min. Hence, digestion at 85°C for 10 min was used.

### 3.3 Amounts of acetate buffer and acetylacetone used

The effect of the pH (5.0 - 7.0) of ammonium acetate buffer (5.0 M) on the absorbance of Be was tested with 4.0 pg of Be in 20  $\mu\text{L}$  of the methanol solution. Figure 3 indicates that the relatively large absorbance value of Be was observed at pH 6.0. This may be explained as follows: When the pH increases from 5 to 6, the predominant species [19] of beryllium is changed from Be(II) to  $\text{Be}(\text{OH})_2$ . Acetylacetone is predominant [13] in an enol form [ $\text{CH}_3\text{C}(\text{OH})=\text{C}(\text{H})\text{COCH}_3$ ] when the pH is in the range of 6 to 10. Hence at pH 6.0, the enol form of acac is readily to react with  $\text{Be}(\text{OH})_2$  to form the stable  $\text{Be}(\text{acac})_2$  chelate [13]. When the pH increases to 6.5 and 7.0, some portion of  $\text{Be}(\text{OH})_2$  may be precipitated and the absorbance values of Be become smaller. Hence, an acetate buffer pH of 6.0 was used. Similarly, the amounts of acetate buffer (15 - 30 mmol of pH 6.0) and acetylacetone (0 - 70  $\mu\text{L}$ ) were varied. Figure 4 indicates that when 50  $\mu\text{L}$  of acac was used, the relatively large absorbance values were observed in the range of 22.5 to 30 mmol. When 22.5 mmol of acetate buffer was used, the absorbance increased as the amount of acac was increased from 0 to 50  $\mu\text{L}$ , and decreased as the amount of acac was increased from 60 to 70  $\mu\text{L}$ . This might have been due to the excess acac, which would not be completely removed during the ashing step, and would interfere with the atomization of Be in the atomization step. Hence, 22.5 mmol of acetate buffer and 50  $\mu\text{L}$  of acetylacetone were selected for use.

### 3.4 Calibration graphs, method detection limit (MDL), and recovery tests for Be in olive leaves

Two sets of calibration graphs were compared. In the first set, a typical calibration graph for Be from the standard addition method was  $y = 2.35 \times 10^{-1} x + 4.78 \times 10^{-2}$  with a correlation coefficient of 0.9993 when 0 - 0.300 ng of Be(II) was spiked to olive leaf sample BCR CRM No. 62 (20 mg). The second set was prepared by adding Be(II) (0 -

0.800 ng) directly to a methanol solution (1.00 mL) containing acetylacetone (50  $\mu$ L) and an appropriate amount of ammonium acetate buffer (0.50 mmol, in methanol). A typical calibration graph from the second set was  $y = 2.36 \times 10^{-1} x + 6.88 \times 10^{-4}$  with a correlation coefficient of 0.9996. The relative error of six slopes obtained from these two sets was within 4.3%. By comparing the slopes of fifteen linear equations obtained from the standard addition method (0 - 0.200 ng Be) for five olive leaf samples with the above three calibration graphs from the second set, the relative error was within 4.3%. These results indicate that the olive leaf matrices do not significantly interfere with the measurement of Be after microwave digestion.

Following the proposed method, the MDL was determined as the amount corresponding to three times the standard deviation of twelve replicates using 20  $\mu$ L of a methanol solution containing 2.1 pg of Be prepared from cartridge 1 of olive leaf sample No. 1 (Tunghai University campus). The MDL ( $3 \sigma$ ) value of Be in olive leaves from the mean of four determinations was found to be  $(0.006 \pm 0.001)$  ng for a 20 mg olive leaf sample, or  $(0.3 \pm 0.0)$  ng/g.

Since no CRMs for Be in olive leaves are available at present, spiked recoveries were used for evaluating the performance of the proposed method. Five samples [BCR No. 62 and four real samples (No. 1 to No. 4)] were analyzed according to the proposed method. The Be content in these five samples was found in the range of 0.071 - 0.560 ng (or 3.6 - 28.0 ng/g), with relative standard deviation (RSD,  $n = 3$ ) between 1.8 and 5.6%, as listed in Table 2. Among them, the level of Be in the Paoshan (No. 3, 28.0 ng/g) was slightly higher than that of BCR No. 62 (10.2 ng/g). Others (No. 1, No. 2, and No. 4) were lower than that of BCR No. 62. This might be because Paoshan is near the Hsinchu Industrial Science Park where some electronic or instrumental factories might use alloys containing Be. However, the levels of Be analyzed in these five olive leaf samples were all below the risk level (84 ng/g) of Be in fish tissue [12] suggested by USEPA.

Appropriate amounts (0.100 and 0.200 ng) of Be(II) in methanol were spiked on each of the above five olive leaf samples. Table 3 indicates that the recoveries from the mean of three determinations were 97.2 - 101.0%, with the RSD ( $n = 3$ ) being within 3.0%.

#### 4. Conclusion

Since no certified reference materials (CRM) for Be in olive leaves are available at present, spiked recoveries were used to evaluate the performance of the proposed method. Good spiked recoveries were observed for five olive leaf samples [including one BCR CRM No. 62, but with uncertified value for Be, and four olive leaf samples in Taiwan]. It is concluded that the contents (0.3 - 38.0 ng/g) of Be in a dried olive leaf sample (20 mg) can be accurately determined by the proposed method.

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**Table 1** Suitable temperature program for beryllium in olive leaf samples by GFAAS

Step	Temperature (°C)	Time (s)	Flow rate of Ar (mL/min)
Drying	60 - 110	40	200
Ashing	120 - 400	40	200
	650 - 650	40	200
Atomization	2500 - 2500	2	0
Cleaning	2600 - 2600	5	200

**Table 2** Beryllium content in olive leaves measured by the proposed method

Sample <sup>a</sup> Correlation  coefficient	Measured Be content <sup>b</sup>		Typical linear equation  from std. addn. method	
	Amount (ng)	Conc. (ng/g)		
BCR No. 62	0.203 ± 0.004	10.2 ± 0.2	$y = 0.235 x + 0.0478$	0.9993
No. 1	0.118 ± 0.005	5.9 ± 0.3	$y = 0.240 x + 0.0291$	0.9998
No. 2	0.071 ± 0.003	3.6 ± 0.2	$y = 0.240 x + 0.0170$	0.9996
No. 3	0.560 ± 0.010	28.0 ± 0.5	$y = 0.236 x + 0.133$	0.9997
No. 4	0.090 ± 0.001	4.5 ± 0.1	$y = 0.233 x + 0.0483$	0.9995

<sup>a</sup>. 20.0 mg of dried olive leaf sample was used.

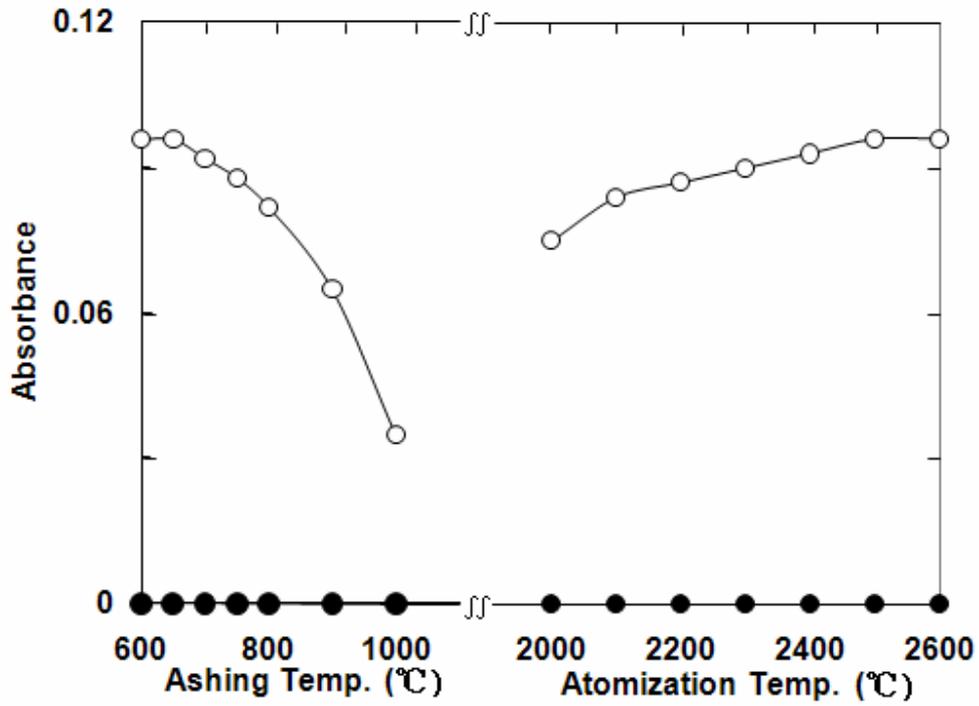
<sup>b</sup>. Mean of three determinations with standard deviation.

**Table 3** Recovery tests for beryllium in olive leaf samples

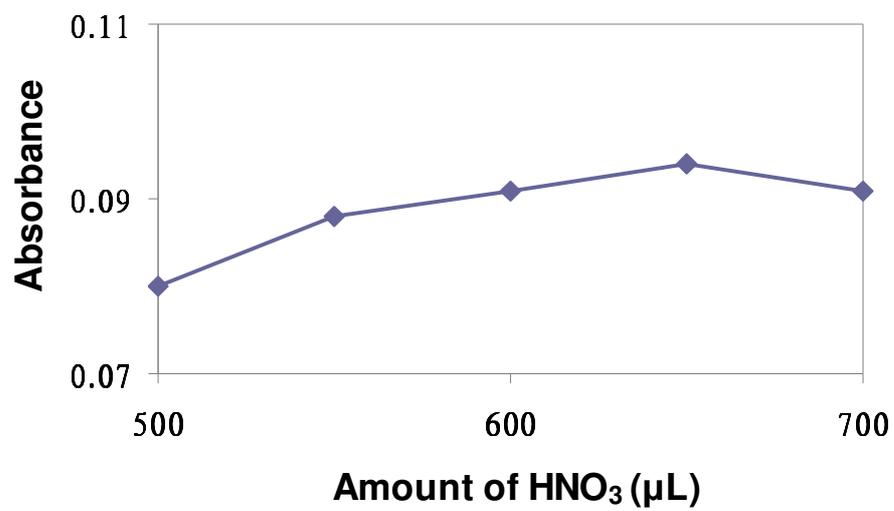
Sample <sup>a</sup>	Amount of Be (ng)		Recovery <sup>b</sup> (%)
	Added	Found <sup>b</sup>	
BCR No. 62	0.100	0.101 ± 0.004	101.0 ± 0.4
	0.200	0.195 ± 0.003	97.5 ± 1.5
	0.300	0.303 ± 0.002	101.0 ± 0.7
No. 1	0.100	0.0988 ± 0.0019	98.8 ± 1.9
	0.200	0.201 ± 0.001	100.5 ± 0.5
No. 2	0.100	0.0972 ± 0.0002	97.2 ± 0.2
	0.200	0.201 ± 0.002	100.5 ± 1.0
No. 3	0.100	0.0982 ± 0.0028	98.2 ± 2.8
	0.200	0.201 ± 0.002	100.5 ± 1.0
No. 4	0.100	0.101 ± 0.003	101.0 ± 3.0
	0.200	0.199 ± 0.002	99.5 ± 1.0

<sup>a</sup>. 20.0 mg of olive leaf sample was used.

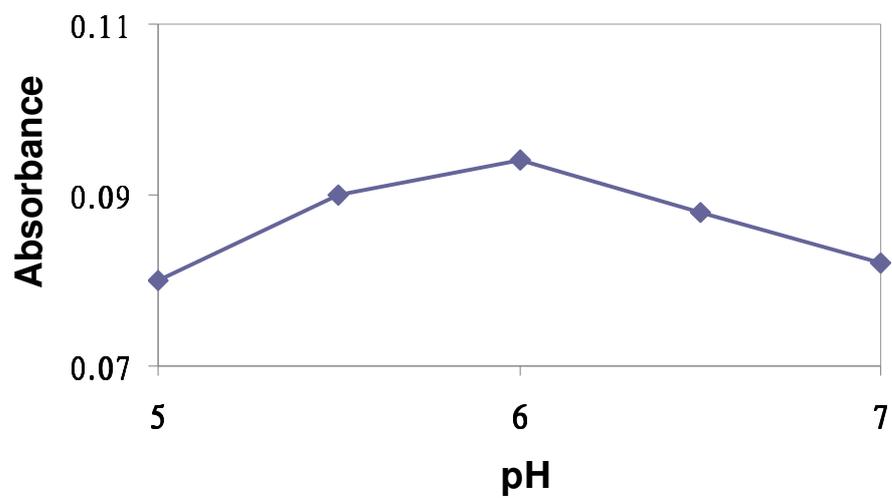
<sup>b</sup>. Mean of three determinations with standard deviation.



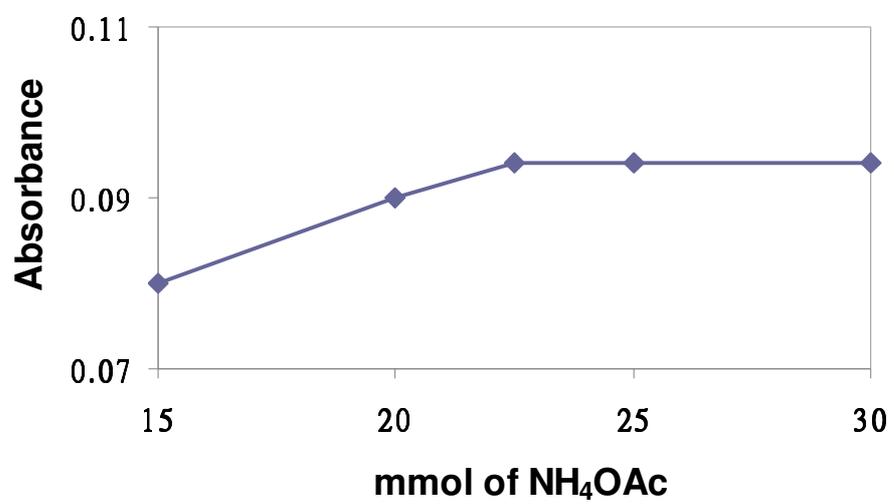
**Fig. 1** Effect of the ashing and atomization temperatures on the absorbance of Be (a) with (open symbols) and (b) without (solid symbols) a chelating agent for 4.0 pg Be in 20  $\mu$ L of the concentrated methanol solution.



**Fig. 2** Effect of amount of HNO<sub>3</sub> on absorbance for 4.0 pg Be in 20 µL of the concentrated methanol solution.



**Fig. 3** Effect of pH of ammonium acetate buffer on absorbance for 4.0 pg Be in 20  $\mu$ L of the concentrated methanol solution.



**Fig. 4** Effect of amount of ammonium acetate buffer on absorbance for 4.0 pg Be in 20 μL of the concentrated methanol solution.

## 以乙醯丙酮作為螯合劑及使用石墨式原子吸光法 測定橄欖葉中鉍的含量

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顏佳新、沈佳俞、王俊傑、郭茂松\*

### 摘要

取20 mg橄欖葉乾粉樣品(含鉍0.006至0.76 ng)，分別經濃硝酸和過氧化氫作兩階段的微波消化(85°C，10 min)後，將消化液之pH值調整至5 - 6之間，加入適量醋酸鉍緩衝溶液(pH 6.0)和乙醯丙酮，使形成鉍的螯合物[Be(acac)<sub>2</sub>]，經預濃縮後，用甲醇沖洗出。取出部分(20 μL)注入石墨式原子吸光儀測定鉍的含量。本方法的偵測極限值(MDL, 3σ)約為0.3 ng/g，線性可達38.0 ng/g。在五種橄欖葉樣品(歐洲BCR CRM No.62及四種台灣的橄欖葉)中，測得鉍的濃度介於3.6至28.0 ng/g之間，添加回收率介於97.2至101.0%之間，相對標準偏差(RSD, n=3)在3.0%以內。本方法應可應用於橄欖葉中鉍的測定。

**關鍵字:**橄欖葉、鉍、乙醯丙酮、石墨式原子吸光法