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Determination of bismuth in pharmaceutical products using methyltriphenylphosphonium bromide as a molecular probe by resonance light scattering technique

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Abstract

A method for the determination of bismuth in pharmaceutical products using methyltriphenylphosphonium bromide as a molecular probe based on the resonance light scattering (RLS) technique was developed. In the presence of Tween-20, bismuth reacts with a large excess of I^- to form $[BiI_4]^-$, which further reacts with methyltriphenylphosphonium bromide (MTPB) to form an ion-association compound. This resulted in a significant enhancement of RLS intensity and the appearance of the corresponding RLS spectral characteristics. The enhanced RLS intensity was directly proportional to the concentration of Bi(III) in the range of 0.001–1.50 μg/ml for the system. The detection limit was 0.98 ng/ml. The characteristics of RLS spectra of the complex, the optimum conditions and the influencing factors were investigated. The method has high selectivity and was applied to the determination of Bi(III) in pharmaceutical products with satisfactory results, which were in agreement with those of the official method and atomic absorbance spectrometry (AAS).

Keywords: Resonance light scattering (RLS); Methyltriphenylphosphonium bromide (MTPB); Bismuth; Pharmaceutical products

1. Introduction

Bismuth is the active ingredient of various pharmaceutical products. It can form a mucosa which is able to prevent the surface of ulcer from eroding by stomach acidity and proteinase. As a result, bismuth-containing pharmaceuticals can be used for different medical purposes, especially for the treatment of gastrointestinal tract disturbances, such as gastritis and peptic ulcer [1,2]. A variety of techniques were described for the determination of bismuth in pharmaceutical products, such as polymeric membrane ion-selective electrode [3], spectrophotometric determination [4–7], sequential injection method [8], flow injection analysis [9–12], UV–vis spectrophotometry [13] and colorimetric method using a sensor coupled to a multicommutated flow system [14]. However, most of these methods suffered from lower sensitivity, large volume of toxic solvents and complicated operations in the procedure. Therefore, a new method is required for the inexpensive and rapid determination of Bi(III) in the pharmaceutical manufacture and quality control process.

Recently, a promising spectral technique, which was based on the measurement of enhanced resonance light scattering (RLS) [15,16], have attracted much attention and generated a great deal of research interest [17–21]. RLS, a special elastic scattering, is produced when the wavelength of Rayleigh scattering (RS) is located at or close to the molecular absorption band. According to the macroscopic fluctuation theory, scattering light originates from the fluctuations of the solution refractive index which consists of real and imaginary parts. In a transparent isotropic medium, since the imaginary part of the refractive index originating from molecular absorption can be neglected, the Rayleigh scattering law of $I \propto 1/\lambda^4$ [22,23] is obeyed for the light scattering of the molecular particles 20-fold smaller than the wavelength of the incident beam [22]. However, if the wavelength of the incident beam is close to that of the absorption band of the molecular particles which exist as aggregates, Rayleigh scattering will deviate from the law and the intensity of some wavelengths will rapidly increase. By using a common spectrofluorometer to measure the RLS, Pasternack et al. [15,16]

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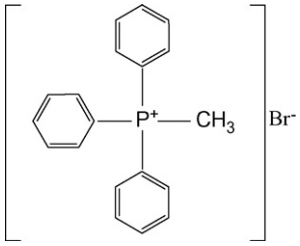

Fig. 1. The structure of MTPB.

studied the aggregations of porphyrines. Later on, RLS became a useful technique for the determination of the traces of inorganic ions [24,25], nucleic acids [26,27], surfactants [28,29], drugs [30–32], proteins [33,34], as well as for the studies of interactions between biopolymers and their probes [35]. Compared with other analytical methods, the RLS method possesses the distinct advantages of speed, convenience, sensitivity, and can be accomplished with a common fluorescence spectrometer by using inexpensive and safe reagents.

Methyltriphenylphosphonium bromide (MTPB, Fig. 1), is one of quaternary phosphonium salts. It can be used as the electrolyte to synthesize $[MePh_3P]$-$[Pd(dmit)_2]_3$ ($dmit = C_3S_5^{2-}$ = 1,3-dithiole-2-thione-4,5-dithiolate) and $[Ph_4P]$-$[Pd(dmit)_2]_3$ [36]. Its derivatives also have extensive application in syntheses, for example, 1,3-dioxolan-2-ylmethyltriphenylphosphonium bromide was used under phase transfer conditions when aldehydes were efficiently transformed into allylic dioxolanes with a Wittig-type reaction [37]; (2-naphthoyl) methyltriphenylphosphonium bromide in the presence of K_2CO_3 in methylene chloride with methyl 2-perfluoroalkynoates at room temperature afforded methyl 4-(2-naphthoyl)-2-triphenylphosphoranylidene-3-perfluoroalkyl-3-butenoates [38].

A method of rapid determination of Bi(III) by using MTPB has not yet been reported so far. In this paper, the characteristics of RLS spectra of Bi(III)-KI-MTPB system were studied. The optimum reaction conditions of the system, the affecting factors, as well as the influences of some coexisting substances were investigated. In Tween-20 solution, $[BiI_4]^-$ reacts with MTPB to form an ion-association compound which results in significant enhancement of RLS intensity and the appearance of new RLS spectra. The enhanced RLS intensity is proportional to the concentration of Bi(III). Little interference from most coexisting substances was observed. The presented method was applied to the determination of Bi(III) in pharmaceutical products with satisfactory results, which were in agreement with those of the official method [39] and atomic absorbance spectrometry (AAS).

2. Experimental

2.1. Apparatus

An FP-6200 spectrofluorometer (JASCO, Japan) was used for recording and measuring the RLS spectra by scanning simultaneously the excitation and emission monochromators from 300 to 600 nm. Both the excitation and emission slits were set at 5.0 nm. A Z-5000 atomic absorption spectrometer (Hitachi Ltd., Tokyo, Japan) was used for carrying out the atomic absorption spectrometric measurement. All glass and plastic wares were kept in 10% (v/v) HNO_3 for at least one night and then rinsed with doubly distilled water before used.

2.2. Reagents

Stock solution of bismuth ion (1.0 mg/ml) was prepared by dissolving 1.000 g of high-purity bismuth in 50 ml (v/v = 1:1) nitric acid, evaporating the solution to remove the excess acid and diluting with (v/v = 1:100) nitric acid to 1000 ml. The standard working solution of 10 μg/ml was prepared by appropriately diluting the stock solution. Cetyltrimethylammonium bromide (CTMAB), 2.0 g/l; cetylpyridinium bromide (CPB), 2.0 g/l; *p*-octyl polyethylene glycol phenyl ether (OP), 2.0 g/l; Tween-20, 2.0 g/l; sodium dodecyl sulfate (SDS), 2.0 g/l; sodium lauryl sulfonate (SLS), 2.0 g/l; KI, 10% (m/v); MTPB (Sigma), 0.015 M/l. All the reagents were of analytical reagent grade and doubly distilled water was used throughout.

2.3. General procedure

Into a 10 ml volumetric flask, 1.4 ml of KI solution, a certain volume of Bi(III), 0.4 ml of MTPB and 0.8 ml of Tween-20 were added successively and diluted to the mark with doubly distilled water. After mixed thoroughly, the RLS spectra of the system were recorded after 10 min with synchronous scanning at $\lambda_{ex} = \lambda_{em}$ (i.e. $\Delta\lambda = 0$). The RLS intensity, I_{RLS} for the ion-association compound and I^0_{RLS} for the reagent blank (KI + MTPB + Tween-20) at the maximum RLS wavelength were measured, $\Delta I_{RLS} = I_{RLS} - I^0_{RLS}$.

3. Results and discussion

3.1. Resonance light scattering spectra

The RLS spectra of the system were recorded by synchronous scanning from 300 to 600 nm. The results are shown in Fig. 2. It can be observed that under the given experimental conditions the RLS intensity of the blank was very small and nearly constant. When Bi(III) was added, the RLS intensity was greatly increased and new RLS spectra appeared with three peaks at 323, 402 and 520 nm, respectively. The maximum located at 402 nm. Therefore, 402 nm was selected as the analytical wavelength in order to obtain higher sensitivity.

When MTPB is added, it reacts with $[BiI_4]^-$ to form an ion-association complex and greatly increases the volume of the molecule. According to the following formula of RLS [40,41],

$$I_{RLS} = \frac{32\pi^3 V^2 n^2 N}{3\lambda_0^4}[(\delta_n)^2 + (\delta_k)^2]$$

where n is the refractive index of the medium, N the molarity of the solution, λ_0 the wavelength of the incident and scattered light, V^2 the square of molecular volume, δ_n and δ_k are the fluctuations in the real and imaginary components of the refractive

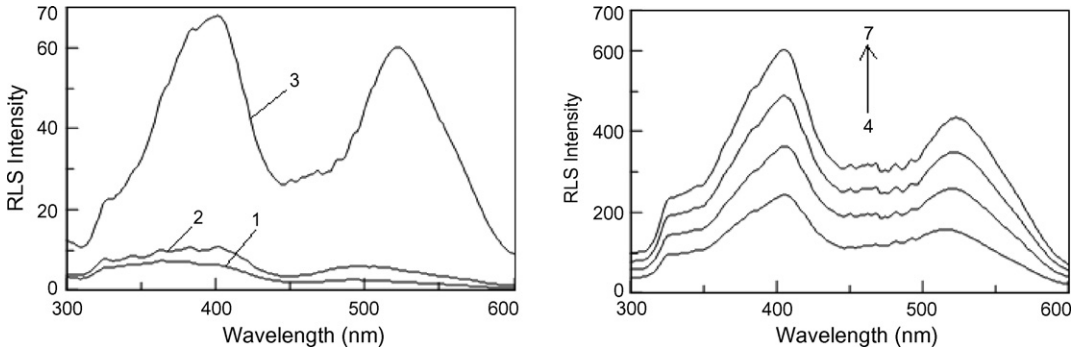

Fig. 2. Resonance light scattering spectra of the MTPB-KI-Bi(III) system. (1) Bi(III) 0.80 μg/ml; (2–7) MTPB 0.020 mol/l; Bi(III) from 2 to 7: 0, 0.05, 0.20, 0.40, 0.60 and 0.80 μg/ml. *Conditions*: KI, 0.8%; Tween-20, 0.16 g/l.

index of the particle, respectively. When other factors are constant, I_{RLS} is related to the size of the formed particle and directly proportional to the square of molecular volume (i.e. V^2). Apparently, with the increase of molecular volume, I_{RLS} is enhanced obviously.

3.2. Optimum conditions for the reactions

3.2.1. The sequence of reagents addition

The effects of the adding sequence on the sensitivity of this method were studied. The results indicated that the sequence of KI + Bi(III) + MTPB + Tween-20 gave the maximum and constant RLS intensity. The reason of this phenomenon was that Bi(III) reacts with a large excess of I^- firstly to form $[BiI_4]^-$ which further reacts with MTPB to form an ion-association compound. So this sequence was used to the further determination.

3.2.2. Effects of surfactants on the RLS intensity

The effects of cationic surfactants (CTMAB and CPB), anionic surfactants (SLS and SDS) and non-ionic surfactants (Tween-20 and OP) on the RLS intensity of the system were investigated. The addition of cationic surfactants resulted in the appearance of precipitation because cationic surfactants reacted with KI. The effects of anionic surfactants and non-ionic surfactants are listed in Fig. 3. The RLS intensity of the system increased with the addition of these surfactants and the sensitivity was the highest when Tween-20 was added. So, the surfactant of Tween-20 was employed and its optimum concentration was 0.16 g/l.

3.2.3. Effect of KI concentration

The influence of KI concentration on ΔI_{RLS} was studied, as shown in Fig. 4. ΔI_{RLS} of the system gradually increased with the increasing of KI concentration at first. When the concentration of KI was higher than 1.4%, RLS intensity kept relatively stable. The reason might be the complete formation of the ion-association compound. Hence, 1.4% KI was chosen for the system.

3.2.4. Effect of MTPB concentration

Fig. 5 shows the effect of MTPB concentration in the range 0–0.015 mol/l on this assay. When the MTPB concentration was low, due to the incompletion of reaction of MTPB cation with $[BiI_4]^-$, the relative intensity of RLS was low. ΔI_{RLS} reached maximum when the MTPB concentration was 0.006 mol/l. If MTPB concentration increased further, ΔI_{RLS} decreased gradually. Hence, 0.006 mol/l of MTPB was selected for further research.

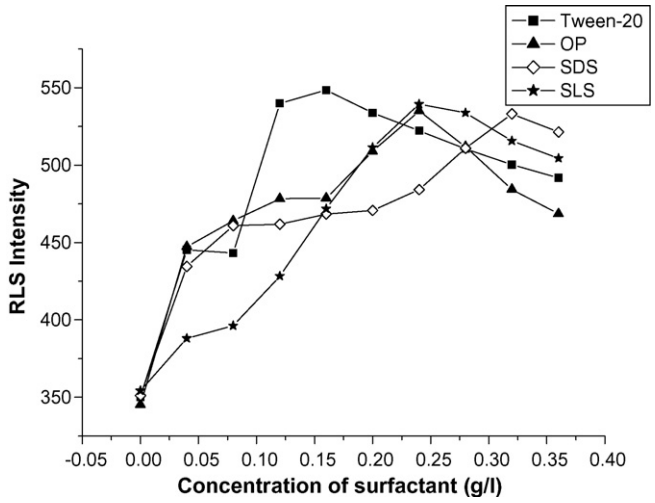

Fig. 3. Effect of surfactant concentration on the RLS intensity. MTPB, 0.020 mol/l; KI, 0.8%; Bi(III), 1.0 μg/ml.

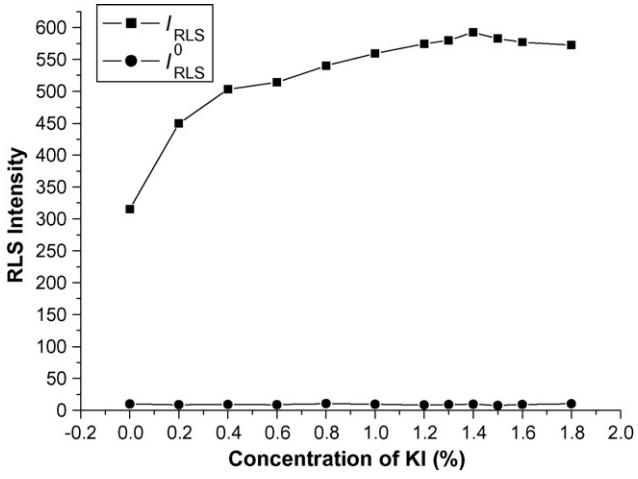

Fig. 4. The effect of KI concentration on RLS intensity. MTPB, 0.020 mol/l; Tween-20, 0.16 g/l; Bi(III), 1.0 μg/ml.

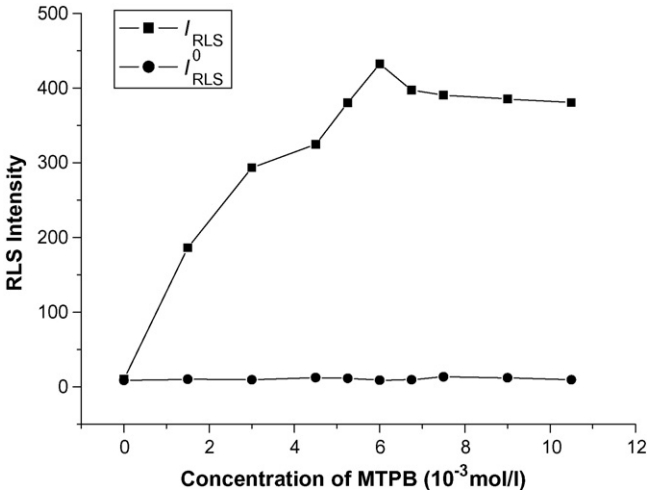

Fig. 5. The effect of MTPB concentration on RLS intensity. KI, 0.8%; Tween-20, 0.16 g/l; Bi(III), 1.0 μg/ml.

3.2.5. Stability of the system and effect of temperature

At room temperature, the reacting time was studied by measuring the RLS intensity values of 1.0 μg/ml of Bi(III) at 402 nm for 2.0 h after mixing. The results showed that the reaction was completed within 7 min and RLS intensity remained constant for at least 1.5 h. When the temperature change was within 5 °C, ΔI_{RLS} remained constant. Thus, this assay did not require crucial temperature and the stability of this assay was reasonable and acceptable for analytical application. When the temperature increased further, ΔI_{RLS} decreased. The reason might be that the ion-association complex was formed by electrostatic attraction and hydrophobic interaction force. With further increasing of temperature, the molecular thermal motion accelerated and surmounted electrostatic attraction and hydrophobic interaction force. So the ion-association complex was dissociated, then ΔI_{RLS} of the system was decreased.

3.2.6. Effect of ionic strength

The effect of ionic strength on the RLS intensity of the system was investigated. It was obtained by keeping the MTPB, KI, Bi(III) and Tween-20 concentrations constant and increasing the concentration of NaCl (0.5 mol/l). It was observed that I_{RLS} decreased with the increasing of ionic strength. It revealed that the increase of salt concentration decreased the combination of MTPB and $[BiI_4]^-$. This effect might be explained as a competition between Cl^- and $[BiI_4]^-$ for the same binding sites on MTPB. Therefore, in the further experiments, no NaCl solution was added in order to simplify the experiment.

3.2.7. Effect of organic solvent

Organic solvents are widely known to affect analytical spectroscopic measurements. Here, the effect of organic solvent on the RLS intensity by increasing of the concentration of ethanol was investigated. The RLS intensity of the system decreased with the addition of ethanol. This might be due to a change in microenvironment for the binding reaction.

3.3. Effects of foreign ions

According to the procedure, the effects of foreign substances on the system were examined at a concentration of 1.0 μg/ml of Bi(III). The tolerance limit was defined as the content of substance that gave relative error not more than ±5%. The results are summarized in Table 1. Most of the ions have little interference on the determination of Bi(III). Some metal ions, such as Fe^{3+} and Hg^{2+} were allowed only at relatively low concentration, but their contents are very low in the pharmaceutical products. Therefore, the present method had good selectivity and could be applied to the direct analysis of samples.

3.4. Calibration graph and sensitivity of the method

Under the optimum conditions, the ΔI_{RLS} values of the complex were measured at the maximum scattering wavelength, and the calibration graph of ΔI_{RLS} against concentration of Bi(III) was constructed. The enhanced RLS intensity was directly proportional to the concentration of Bi(III) in the range of 0.001–1.50 μg/ml and the regression equation was calculated as $\Delta I_{RLS} = 122.57 + 601.93C$ (μg/ml) with regression coefficient $r = 0.9999$. The detection limit was 0.98 ng/ml, which was calculated according to $3S_0/S$, where S_0 was the standard deviation (S.D.) of the blank measurements ($n = 11$) and S was the slope of the calibration curve. The R.S.D. for 11 determinations of 1.0 μg/ml of Bi(III) was 2.05%.

Table 1
Tolerance to foreign ions (1.0 μg/ml Bi(III)

Foreign ion	Tolerance limit (μg/ml)	Relative error (%)	Foreign ion	Tolerance limit (μg/ml)	Relative error (%)
F^-	100	−3.98	Ni^{2+}	37.5	5.49
NO_3^-	80	3.36	Hg^{2+}	0.4	5.75
SO_3^{2-}	50	−5.08	Al^{3+}	80	5.26
CO_3^{2-}	5	−3.48	Cu^{2+}	3	−4.17
Ba^{2+}	200	−4.85	Fe^{3+}	0.5	4.58
SO_4^{2-}	200	5.43	Zn^{2+}	100	−4.50
NH_4^+	600	3.89	Pb^{2+}	4	4.56
HSO_3^-	50	5.13	Ca^{2+}	90	−3.62
$C_2O_4^{2-}$	20	4.79	Mg^{2+}	70	−4.71
Br^-	100	−3.98	Cd^{2+}	7	4.21
Mn^{2+}	300	5.30	Co^{2+}	400	3.94

Table 2
Determination results of Bi(III) in samples and comparison with the results obtained by official method and AAS method

Samples	Official method (μg, $n=5$)	AAS method (μg, $n=5$)	RLS method			
			Found (μg, $n=5$)	R.S.D. (%)	Added (μg, $n=5$)	Recovery (%)
1[a]	0.42	0.43	0.44	2.85	0.5	97.2
2[b]	0.69	0.67	0.68	0.89	0.5	102.3
3[c]	0.34	0.37	0.35	3.21	1.0	99.8
4[d]	0.97	1.05	1.00	2.38	0.3	104.2

[a] Chenxianglu-Bailu Tablets.
[b] Bismuth Nitras Basicus Tablets.
[c] Colloidal Bismuth Pectin Capsules.
[d] Compound Bismuth Aluminate Tablets.

From the results above, it can be concluded that this method had a low detection limit and high sensitivity. Compared with the reported analytical methods for Bi(III) [3–14], the RLS method was highly sensitive and selective.

3.5. Analysis of real samples

Four pharmaceutical products containing Bi(III) compounds, Chenxianglu-Bailu Tablets, Bismuth Nitras Basicus Tablets, Colloidal Bismuth Pectin Capsules, and Compound Bismuth Aluminate Tablets, were analyzed. According to the manufacturer, each tablet of Chenxianglu-Bailu Tablets (0.30 g) contains 0.066 g of bismuth subnitrate ($4BiNO_3(OH)_2{\cdot}BiO(OH)$), glycyrrhiza uralensis, rheum palmatum, citrus reticulata, and other unspecified Chinese traditional medicine. Bismuth Nitras Basicus Tablets contains 0.24 g of Bi_2O_3 approximately per tablet (0.30 g) and other unspecified excipients. The main component of Colloidal Bismuth Pectin Capsules is $[KBiC_{12}H_{10}O_8(OH)_6]_n$. Compound Bismuth Aluminate Tablets contain 0.20 g of bismuth aluminate ($Bi_2(Al_2O_4)_3{\cdot}10H_2O$), 0.40 g of magnesium carbonate, 0.20 g of sodium bicarbonate, and other unspecified compounds.

Ten tablets or capsules of each kind of pharmaceutical product containing Bi(III) were weighed, ground in a mortar made of agate about 0.50 h and finally homogenized. An accurately weighed amount from each sample was heated at 700 °C for 4.0 h in order to remove the interference of organic substances. After cooled for 2.0 h, the residue was dissolved by appropriate amount of 1:1 HNO_3, filtered and was transferred to a 100 ml volumetric flask, and diluted to the mark with distilled water. The solution was diluted 100-fold before determination. The results of determination are listed in Table 2. In addition, recovery tests were performed and the recoveries were in the range of 97.2–104.2%, which demonstrated that the proposed procedure could be used satisfactorily for the analysis of Bi(III) in the pharmaceutical products. The accuracy of the developed method was checked by comparison of the results obtained by RLS method with those got by the official procedure and AAS (Table 2), no obvious differences were observed.

4. Conclusion

A novel method for the determination of bismuth in pharmaceutical products using MTPB as a molecular probe based on the resonance light scattering (RLS) technique was developed. Compared with other common methods for determination of Bi(III), the RLS technique is rapid, convenient, sensitive, and can be accomplished with a common fluorescence spectrometer by using inexpensive and safe reagents. The simplicity of procedure and the precision of results justified the use of MTPB with RLS technique as a useful method for the quantitative analysis of Bi(III) in pharmaceutical products. The obtained results were satisfactory, and were in agreement with those of the official method and AAS.

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