Pergamon

Talanta, Vol. 41, No. 2, pp. 171–178, 1994

0039-9140/94 \$6.00 + 0.00

SEPARATION OF ANTIMONY(III) WITH IODIDE AND DITHIZONE BY SORPTION ON POLYURETHANE FOAM FROM SULPHURIC ACID MEDIUM FOR ITS SPECTROPHOTOMETRIC DETERMINATION IN GLASSES

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(*Received* 15 *June* 1993. *Revised* 30 *August* 1993. *Accepted* 30 *August* 1993)

Summary—A method for quantitative separation of antimony(III) by sorption on polyether based polyurethane foam and its spectrophotometric determination has been described. The method involves formation of a pink–red complex of antimony(III) with iodide (0.045M) and dithizone ($2.3 \times 10^{-5}M$) in 0.25–0.75M H_2SO_4 medium, sorption of the complex on polyurethane foam (within 45 min) at room temperature followed by its elution with acidified acetone (acetone containing 0.008% H_2SO_4) and spectrophotometric measurement at 507.2 nm ($\epsilon = 2.56 \times 10^4$ l mol cm). The method obeys Beer's law from 0.1 to 6.0 μg antimony(III). Tolerance limits of other ions are Co (100 μg), Ni (100 μg), Fe (10 μg), Cu (0.5 μg), Sn (20 μg), Zn (100 μg), As (100 μg), Mn (200 μg), Pb (50 μg), Ti (100 μg), V (50 μg), *etc.* Interference by iron and copper have been eliminated by treating with KOH prior to the extraction of antimony. The method has been standardized with glass samples spiked with known amounts of antimony and applied to the determination of antimony in various glasses.

Various methods for separation of antimony(V) and antimony(III) by extraction of its halide complexes with suitable solvents have been reported[1–6] for determination of antimony using spectrophotometry and AAS. Iodide complexes of antimony(III)[1–5] have been utilized for the spectrophotometric determination of antimony, but due to poor sensitivity the determination level cannot be extended below 10 μg ml Sb. Numerous other spectrophotometric methods[7–12] have also been reported for determination of low concentrations of antimony involving extraction of Sb(III) and Sb(V) with various azo dyes and their derivatives. The methods mostly suffer from high blank values due to the colour of the dyes. As a result, the determination of lower concentration of antimony poses a problem. Besides these, chelating reagents like diphenylcarbazone[13] and diethyldithiocarbamate,[14] have been utilized for extraction of antimony for its determination by AAS. Reaction of antimony with dithizone has been reported[1,15] and some authors claimed that the extraction with benzene at pH4 and from ~ 1M HCl (red–brown colour) is satisfactory. But those methods could not be utilized for determination of antimony due to the instability of the complex in benzene medium. It has also been reported[16] that Sb(III) forms a mixed ligand complex with dithizone (H_2Dz) and diethyldithiocarbamate (DDC) in CCl_4 through an exchange reaction between Sb $(DDC)_3$ and Cd $(HDz)_2$ but this has not been utilized for spectrophotometric determination of antimony.

The use of resilient open-cell polyether type polyurethane foam (PUF) membranes as sorbents is a relatively novel advance in the field of separation science.[17,18] Owing to the membrane-like structure, outstanding rapid sorption and mass-transfer properties of PUF has enabled the replacement of liquid–liquid extraction systems by the open-pore quasi-spherical solid liquid system with enhanced separation and preconcentration efficiency.[18] Using a manual batch technique Bowen[19] reported that antimony(V) could be extracted with polyether PUF from 6M HCl solution. More recently, the extraction of antimony(III) and (V) from aqueous HCl acid solution by polyether PUF has been investigated[20] using an automatic batch squeezing device. Pre-concentration methods of antimony have also been reported from HCl–LiCl,[21] fluoride[22] and thiocyanate[23,24] medium. A method for the separation of antimony(III) from antimony(V) using unplasticized diethyldithiocarbamate foam has been described using batch and column mode of extraction.[25] Separation of antimony from indium and mercury have also been reported using DDC-loaded polyester PUF in batch experiments.[26] PUF loaded with a 1% solution of 1, 2 ethanediol in benzene has been examined for separation and preconcentration of antimony from natural waters.[27] But none of the methods were utilized for spectrophotometric determination of antimony in practical analysis of glass and ceramic materials.

Using the extraction technique with unloaded PUF, a new approach has been made in this communication to establish optimum conditions for the formation of a mixed ligand complex of antimony(III) with iodide and dithizone, its quantitative sorption on PUF, elution and the stability of the complex. The results of the study have also been utilized to develop a spectrophotometric method for determination of antimony in various glasses.

EXPERIMENTAL

Instrumentation

A Shimadzu UV-2100 spectrophotometer was used for absorbance measurement.

Reagents and chemicals

All reagents and chemicals were of analytical grade unless otherwise stated. Bidistilled water was used all through-out.

Standard antimony(III) solution (100 *µg/ml*)

A 0.13714 g amount of potassium antimony(III) oxide tartarate, $K(SbO)\ C_4H_4O_6\ 1/2\ H_2O$ (M.W.333.93) was dissolved in water and diluted to 500 ml in a calibrated flask. Standard working solutions were prepared by diluting the above stock solution.

Potassium iodide–ascorbic acid solution

A KI solution (0.45*M*) was prepared in water containing 5% (W/V) ascorbic acid.

Dithizone

Dithizone solution ($2.3 \times 10^{-4}M$) was prepared in methanol and standardized by extractive titration with standard silver solution.[28]

Eluent

The eluent was prepared by adding 1 ml of 0.8% (V/V) H_2SO_4 to 99 ml of acetone.

Preparation of foam

Polyether type polyurethane foam (commercial 'U' foam, M/s U-Foam Ltd, India) was cut in the form of cylinders (6 mm diameter and 2 cm long) of average weight 12 mg/chip. The foam cylinders were soaked in 3*M* HCl with occasional squeezing for 24 hr, then washed several times by squeezing with water until free from acid and finally refluxed with acetone in a Soxhlet apparatus for 6 hr. The washed foam cylinders were dried at 40°C and stored in dark coloured bottles in the dark.

Procedure

Calibration. Standard antimony(III) solutions (1–10 ml) containing 1–60 µg of Sb were pipetted into 50 ml glass beakers; 3 ml of 5*M* H_2SO_4 was added followed by the addition of 2 ml of 0.45*M* KI–ascorbic acid solution; 2 ml of $2.3 \times 10^{-4}M$ dithizone was then added and the volume of each solution was made up to 20 ml with water. Five pieces of foam chips were added to each solution and squeezed thoroughly with a glass plunger. The solutions were equilibrated with foam for 45 min by squeezing the foam at regular intervals of 10 min. The foam chips were then removed from the solution and transferred into a 5 ml disposable syringe. The foam chips were thoroughly washed by squeezing four times with 4 ml of water. Finally the sorbed complex was eluted from the foam by washing four times with 2 ml portions of acidified acetone (acetone containing 0.008% H_2SO_4 acid) into a 10 ml calibrated flask and diluted up to the mark with acidified acetone added from the same syringe. The absorbance of each solution was measured at 507.2 nm against a reagent blank and a calibration curve (absorbance *vs* concentration) was plotted.

Preparation of sample solution. A 0.1–0.5 g glass sample (depending upon the amount of antimony) was dried at 110°C, placed in a PTFE basin and digested on a sand bath with 2 ml sulphuric acid (1 + 1) and 10 ml of HF to near dryness. The basin was cooled, a further 5 ml of HF was added and the process was repeated until fumes of SO_3 were evolved. The residue was taken up in dil. sulphuric acid + tartaric acid (1 ml of 5% W/V) mixture, boiled and made alkaline with potassium hydroxide solution. Any precipitate formed was filtered off through Whatman No. 41 filter paper and washed with hot water. The filtrate was just acidified with dilute sulphuric acid, transferred into a 100 ml calibrated flask, cooled and diluted up to the mark with water.

A 5 ml aliquot of the above sample solution was transferred into a 50 ml beaker and then the determination procedure was applied.

Calculation

The absorbance of the test solution was compared with the calibration curve and converted to the quantity of antimony in μg. from which its concentration in the sample is calculated.

$$Sb = 2 \times 10^{-2}\, A/W\%$$

where, A is the number of μg of Sb corresponding to the absorbance measured and the sample weight is in g.

RESULTS AND DISCUSSION

It reveals from literature[1,15] that Sb(III) reacts with dithizone in acidic media (pH 4 and 1M HCl) to form a red–brown complex which can be extracted with benzene. But no further information is available for its spectrophotometric measurement. In the present study it was observed that Sb(III) reacts with dithizone in 0.5M H_2SO_4 to form a red complex in aqueous medium. The absorption maximum of the complex in aqueous medium has been found to be 505.6 nm as shown in Fig. 1 (curve a). Further it is known that Sb reacts with KI in H_2SO_4 acid medium to form a yellow Sb–iodide complex but interestingly it was observed that the yellow iodide complex again reacts with dithizone to form a pink–red complex in the same aqueous H_2SO_4 acid medium. The absorption of this complex was also measured in aqueous medium and found to be maximum at 506.8 nm as shown in Fig. 1 (curve b). Again it was observed that both the complexes can be sorbed on PUF. But the rate of sorption of the complex on PUF in batch squeezing mode is shown in Fig. 1 (curve b) was faster than the complex shown in Fig. 1 (curve a). It was further observed that the colour of the sorbed Sb(III)–HDz complex on PUF was green while the Sb(III)–I–HDz complex sorbed on PUF was pink–red as observed in the aqueous medium. The absorption spectra of both eluted solutions (the complexes in both cases were eluted with acidified acetone) was obtained from 700 to 450 nm and presented in Fig. 2 (a: Sb(III)–HDz and b: Sb(III)–I–HDz). It can be seen from curve a that there is a distinct peak at 610 nm in the green region of the spectrum but on proceeding towards shorter wavelengths there is a small hump with its absorption maximum at 510 nm. Again there is an increase in absorption beyond 490 nm. In curve b, however, the green peak is absent and there is a single absorption peak at 507.2 nm and also there is a sharp decrease in absorption up to 450 nm. Therefore, from the nature of the curves it can be concluded that the extent of formation of the red Sb(III)–HDz (Fig. 2 curve a) complex in acetone medium is less, while that of Sb(III)–I–HDz complex in curve b is distinctly higher.

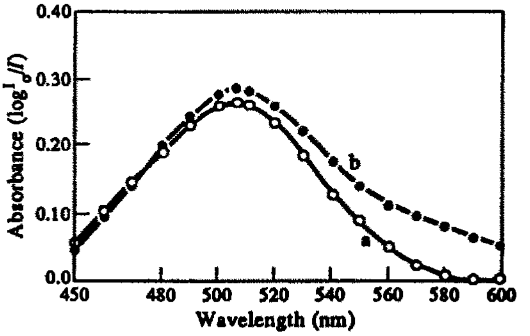

Fig. 1. Absorption spectra of (a) Sb(III)–HDz and (b) Sb(III)–I–HDz complexes in aqueous sulphuric acid (0.5M) media.

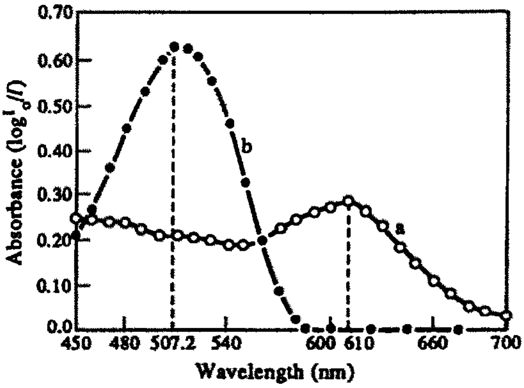

Fig. 2. Absorption spectra of (a) Sb(III)–HDz and (b) Sb(III)–I–HDz complexes after extraction of PUF and subsequent elution with acidified acetone.

The net outcome of the above studies is that Sb(III) forms a pink–red complex with potassium iodide and dithizone (visually different from the red Sb(III)–HDz complex) in aqueous sulphuric acid medium which can be extracted on PUF and subsequently eluted quantitatively with acidified acetone for measurement of absorbance at 507.2 nm. These observations lead to further investigation for establishing different parameters affecting the sorption of the Sb(III)–I–HDz complex on PUF, its quantitative elution and stability of the eluted complex *i.e.* acid concentration during sorption, concentration of potassium iodide and dithizone, time of equilibration and final acid concentration in acetone medium.

Effect of sulphuric acid concentration

The formation of a stable pink–red complex of Sb(III)–I–HDz and its quantitative sorption on PUF was found to be primarily dependent on the sulphuric acid concentration in the reaction medium. Therefore to optimize the acid concentration a series of experiments was carried out by taking 30 μg of antimony(III) in each beaker containing 0.045*M* potassium iodide, $2.3 \times 10^{-5}M$ dithizone and different concentrations of sulphuric acid (0.05–0.87*M*) in a total volume of 20 ml. Five pieces of foam were added in each beaker and solutions were equilibrated with foam chips for 45 mins followed by washing the foam with water and elution of the sorbed complex with acidified acetone according to the procedure. The absorption spectra of each eluted solution is presented in Fig. 3. It can be seen from Fig. 3 that at acid concentrations below 0.25*M* H_2SO_4 two different complexes of different colours (pink–red and green) were formed in which the green colour is also prominent (after correction of the absorption value of the green colour exhibited by the blank). However, on increasing the acid concentration, it was observed that the absorption peak due to green colour at 610 nm gradually diminished on the other, the absorption at 507.2 nm increased considerably and at 0.25*M* H_2SO_4 a single peak due to pink–red colour at 507.2 nm was obtained. Moreover, the absorbance was found to be maximum in the acid range 0.25–0.75*M* H_2SO_4 (which also indicated the maximum sorption of the complex on PUF). But, on further increasing the acid concentration >0.75*M* the extent of sorption of Sb(III)–I–HDz on PUF gradually decreased probably due to less formation of the extractable species at higher acid concentration. Therefore, the concentration of sulphuric acid was maintained at 0.25–0.75*M* for subsequent experiments. These observations clearly indicated that there must be some optimum concentrations of iodide and dithizone for the formation of the extractable complex of ideal stoichiometry at this specified acid range (0.25–0.75*M* H_2SO_4).

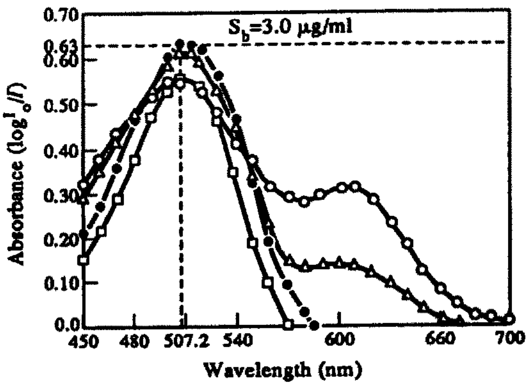

Fig. 3. Absorption spectra of Sb(III)–I–HDz complex at different H_2SO_4 acid concentrations (0.045*M* KI and $2.3 \times 10^{-5}M$ dithizone), ○–○ 0.05*M*, △–△ 0.15*M*, ●–● 0.25–0.75*M*, □–□ 0.87*M*.

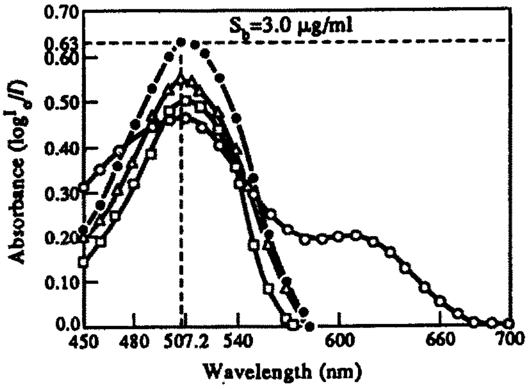

Fig. 4. Absorption spectra of Sb(III)–I–HDz complex at different KI concentration (H_2SO_4-0.75*M* and H_2Dz-$2.3 \times 10^{-5}M$); ○–○ 0.006*M*, △–△ 0.03*M*, ●–● 0.045*M*, □–□ 0.06*M*.

Effect of potassium iodide

The influence of potassium iodide on the formation and sorption of Sb(III)–I–HDz complex on PUF was studied by taking a series of 30 μg Sb(III) in 50 ml beakers containing 0.75*M* H_2SO_4, $2.3 \times 10^{-5}M$ H_2Dz and varying concentrations of potassium iodide (0.006–0.06*M*) in a total volume of 20 ml. The above procedure was followed for recording the absorption spectra of each solution. It can be observed from Fig. 4 that the complex sorbed on PUF at iodide concentrations below 0.03*M* exhibited two absorption peaks (507.2 and 610 nm) in acidified acetone medium as observed in the previous study. However, it was also observed that with the gradual increase of potassium iodide concentration the absorbance at 610 nm gradually diminished and simultaneously the absorbance at 507.2 nm increased to reach its maximum value at 0.045*M* potassium iodide. But on further increasing the potassium iodide concentration the absorbance value at 507.2 nm decreased probably due to the formation of a stable Sb(III)-iodide complex in presence of higher concentrations of potassium iodide.

Therefore, for further studies potassium iodide concentration was kept at 0.045*M*.

Effect of dithizone

The dithizone concentration was similarly optimized by equilibrating, with foam, a series of 20 ml solutions containing 30 µg of Sb(III), 0.75*M* H_2SO_4, 0.045*M* potassium iodide and various amounts of dithizone. The optimum concentration of dithizone was found to be in the range 2.0–2.3 × 10^{-5}*M* (Fig. 5). Above this concentration the absorbance gradually decreases probably due to the competitive sorption of the complex and free dithizone on PUF.

Effect of ascorbic acid

The effect of ascorbic acid was studied on the formation and sorption of Sb(III)–I–HDz complex on PUF by varying the ascorbic acid concentration from 0.5 to 15% in KI solution, so that its final concentration in the extracting medium may vary from 0.05 to 1.5% (0.75*M* H_2SO_4, 0.045*M* KI and 2.3 × 10^{-5}*M* H_2Dz). Results indicated that ascorbic acid concentrations between 2 and 15% in KI solution have no effect on the formation and sorption of the desired complex. However, at ascorbic acid concentration below 2% in KI solution resulted lower sorption of the complex probably due to preoccupation of the sorption sites of the foam cavities by iodine itself.

Effect of equilibration time

The sorption of the Sb(III)–I–HDz complex with PUF under the above mentioned conditions was found to be maximum after 35 min and no further sorption took place after that period (Fig. 6). The sorption of the Sb(III)–I–HDz complex was found to ~99% (calculated from the difference of absorbances of the aqueous solution before and after extraction) under the experimental conditions. In practical analytical applications the equilibration time was extended up to 45 min to ensure complete sorption of the complex on PUF.

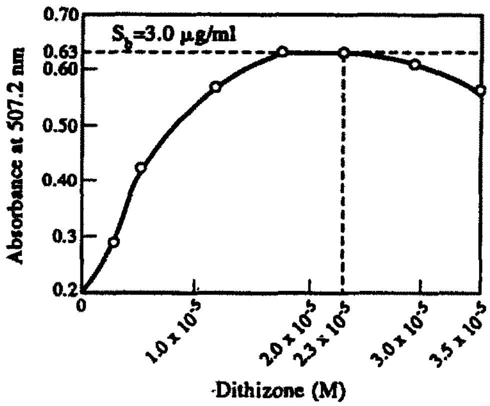

Fig. 5. Effect of dithizone concentration on the formation and sorption of Sb(III)–I–HDz complex on PUF (0.75*M* H_2SO_4 and 0.045*M* KI).

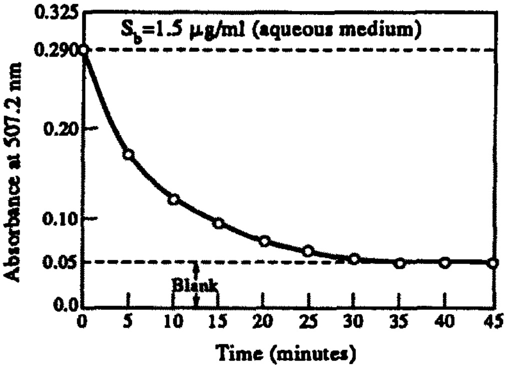

Fig. 6. Sorption of Sb(III)–I–HDz complex on PUF as a function of time (0.75*M* H_2SO_4, 0.045*M* KI and 2.3 × 10^{-5}*M* H_2Dz). Absorbance at 507.2 nm.

Elution and stability of the eluted complex in acetone

It was observed during the study that the complex can only be quantitatively eluted from the foam matrix with acidified acetone, *i.e.* acetone protonated with sulphuric acid. The optimum acid concentration for quantitative elution and stability of the eluted complex was found to be 0.008%–0.01% sulphuric acid (V/V) in acetone (Fig. 7). It can further be seen from Fig. 7 that below 0.008% sulphuric acid the elution of the complex from foam is not quantitative while at >0.01% sulphuric acid the complex tend to dissociate and yield lower

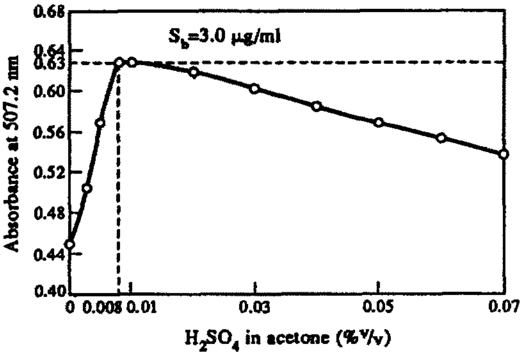

Fig. 7. Effect of H_2SO_4 acid concentration in acetone for quantitative elution and stability of Sb(III)–I–HDz complex. Absorbance at 507.2 nm.

absorbance value. However, at an acid concentration of 0.008–0.01% V/V H_2SO_4 (in acetone) the eluted complex was stable for 2 hr.

It can therefore be suggested from Fig. 2 that Sb(III) probably forms a mixed ligand complex containing iodide and dithizone. It is known[1] that antimony(III) forms all six Sb(III)–I^- species depending on the acidity and KI concentration. Again from the results obtained in Figs 3 and 4 it can be suggested that there is a close relation between optimum concentrations of H_2SO_4 and KI for the formation of a desired Sb(III)–I^- complex after which a reaction with dithizone forms a stable Sb(III)–I–HDz complex of unknown stoichiometry which is yet to be established. Moreover, the elution behavior of the complex suggests that the sorbed mixed ligand complex of Sb(III) with iodide and dithizone is anionic in nature since protonated acetone was necessary for its quantitative elution. The sorption mechanism was not studied in the present content. But, on the basis of present observation and the studies made by earlier workers[17,29] it can be suggested that the mixed ligand anionic species were sorbed on PUF through a cation chelation mechanism.[29] According to this mechanism, complexation of cations, *e.g.* K^+ or H_3O^+ takes place in the PUF cavities through ion-dipole interaction or hydrogen bonding or both. Then the anionic species are extracted as counter ions to the captured cations in the PUF cavities.

Validity of Beer's law

The effect of antimony concentration was studied under the experimental conditions set up and Beer's law was found to be obeyed in the range 0.1–6.0 μg ml antimony. The molar extinction coefficient for antimony has been calculated to be 2.56×10^4 l mol cm at 507.2 nm. From the regression analysis of the calibration plot of absorbance against Sb concentration, the correlation coefficient was found to be 0.9998 and the standard error of the mean, calculated from the equation of the line ($y = mx + b$, where y is the absorbance and x is the amount in μg of Sb) was found to be ± 0.029. The Sandell's sensitivity was calculated to be 0.0048 μg cm^2 against 0.001 absorbance.

Effect of foreign elements

The effect of diverse ions on the determination of 3 μg ml Sb(III) was examined following the procedure and is presented in Table 1. Among the interfering elements studied, tin and cadmium also form extractable complexes with iodide and dithizone and can be tolerated up to 20 μg ml under the experimental conditions. Iron and copper interfere since they react with iodide under the experimental conditions and can be tolerated up to 10 μg ml and 0.5 μg ml, respectively. Interferences of iron and copper when present in larger amounts can be eliminated by separating from antimony by treatment with caustic potash. Most of the other elements present in glass, ceramic systems do not interfere.

Table 1. Effect of foreign elements on the sorption of antimony. Conditions: antimony(III)—3 μg ml, acid—0.75*M* H_2SO_4, KI—0.045*M*, dithizone—$2.3 \times 10^{-5}M$, sorbent—PUF, eluent—acidified acetone (acetone containing 0.008% v/v H_2SO_4)

Foreign ion	Amount added *μg ml*	Absorbance
Nil	—	0.63
Fe(III)	10	0.62
Cu(II)	0.5	0.62
Co(II)	50	0.63
Ti(IV)	100	0.63
V(V)	100	0.63
Al(III)	200	0.63
Mn(II)	200	0.62
Ni(II)	50	0.63
Ca(II)	200	0.63
Mg(II)	200	0.63
Sn(II)	20	0.63
Cd(II)	20	0.63
As(III)	100	0.63
Zn(II)	100	0.63

Application of the method

The developed method was standardized with glass samples (without antimony) spiked with known amounts of antimony (Table 2). The method was then applied to the determination of antimony in glass samples after preparation of sample solutions by following the procedures mentioned before. Results are presented in Table 3.

CONCLUSIONS

The results of the above study establish for the first time the sorption characteristics of a coloured complex of antimony with iodide and dithizone and its application to the spectrophotometric determination of antimony in glass samples. The method is simple and accurate compared to the existing spectrophotometric methods and effective for determination of antimony down to 0.1 μg ml.

Table 2. Standardization of method for determination of antimony in simulated glass samples* (results are averages of five determinations)

Sample	Antimony added (%)	Antimony found (%)	Standard deviation	95% Confidence limit $CL = \bar{X} \pm A(\Sigma R)$
Optical Glass	0.02	0.019	±0.004	0.019 ± 0.0043
Soda-lime Glass 1	0.05	0.048	±0.014	0.048 ± 0.014
Soda-lime Glass 2	0.10	0.102	±0.015	0.102 ± 0.012
Borosilicate Glass 1	0.30	0.304	±0.026	0.304 ± 0.026
Borosilicate Glass 2	0.50	0.498	±0.027	0.498 ± 0.036

*Glass compositions: SiO_2—69.26–80.60%, Al_2O_3—0.32–1.94%, Fe_2O_3—0.008–0.22%, Tio_2—0–0.027%, B_3O_3—0.06–8.32%, Cao—0.51–20.64%, MgO—0.02–2.60%, BaO—0–2.96%, Na_2O—7.5–22.80%, K_2O—0.37–4.38%, SO_3^{2-}—0.009%, Cl^-—0.036%, As_2O_3—0.14%.

†Known amounts of antimony were added during decomposition of each glass sample.

Table 3. Determination of antimony in different glass samples (results are averages of five determinations)

Sample	Antimony present (%)	Antimony found (%)	Standard deviation	95% confidence limit $CL = \bar{X} \pm A(\Sigma R)$
Opal glass	1.07	1.067	±0.0044	1.067 ± 0.0057
Barium crown glass 1	0.16	0.164	±0.0098	0.164 ± 0.0046
Barium crown glass 2	0.148	0.142	±0.0037	0.142 ± 0.0041
Bulb glass	0.43	0.422	±0.018	0.422 ± 0.026
Bulb glass	0.10	0.104	±0.0021	0.104 ± 0.0026
Borisilicate crown glass	0.01	0.0096	±0.00089	0.0096 ± 0.00098

Acknowledgements—The authors wish to thank Dr B. K. Sarkar, Director, Central Glass and Ceramic Research Institute for his kind permission to publish this paper.

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