

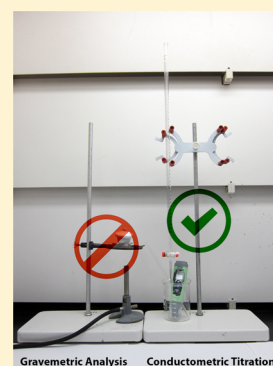
# Determination of Sulfate by Conductometric Titration: An Undergraduate Laboratory Experiment

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## S Supporting Information

**ABSTRACT:** The classic technique for sulfate analysis in an undergraduate quantitative analysis lab involves precipitation as the barium salt with barium chloride, collection of the precipitate by gravity filtration using ashless filter paper, and removal of the filter paper by charring over a Bunsen burner. The entire process is time-consuming, hazardous, and frequently yields poor results. Titrimetric methods are much faster, but a good indicator has not been found for sulfate titrations. However, because the relative concentrations of ions in solution change during a titration sequence, monitoring conductivity during the progress of a titration produces a signal which can be used to indicate the equivalence point. A conductometric titration protocol has been developed which is relatively rapid, inexpensive, and can produce results as accurate as the classic technique in an undergraduate laboratory.

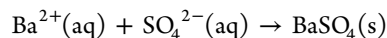


**KEYWORDS:** First-Year Undergraduate/General, Second-Year Undergraduate, Analytical Chemistry, Laboratory Instruction, Hands-On Learning/Manipulatives, Problem Solving/Decision Making, Conductivity, Instrumental Methods, Titration/Volumetric Analysis

## INTRODUCTION

The sulfate ion is of major environmental significance, as it is the third most abundant ionic species in seawater.<sup>1</sup> Sulfate concentration is also of major importance in soil science and acid mine drainage. However, although a wide variety of methods, both wet and instrumental, exist for quantitation of sulfate, the classic gravimetric technique is still widely used, largely unchanged over the past century.<sup>2,3</sup> Factors contributing to its endurance include its low cost, simplicity, and accuracy in the hands of an experienced analyst. These qualities have also made it a common addition to undergraduate chemistry laboratory sequences.

The method is based upon insolubility of  $\text{BaSO}_4$ ,  $K_{\text{sp}} = 1.1 \times 10^{-10}$ .<sup>1</sup> The basic procedure involves a soluble sulfate unknown dissolved in water, the solution is acidified with HCl, and the sulfate is precipitated by addition of  $\text{BaCl}_2$  to yield  $\text{BaSO}_4$  as per the equation below.



A slight excess of the precipitating agent ensures completeness of precipitation utilizing the common ion effect, and the precipitate is allowed to digest overnight. This is followed by collection of the precipitate by gravity filtration using ashless filter paper and removal of the filter paper by charring over a Bunsen burner.<sup>4</sup>

The method can be prone to numerous errors, including contamination of the precipitate with occluded and coprecipitated impurities and reduction of the sulfate to sulfide during the charring process.<sup>5</sup> Although the use of high quality

commercially analyzed unknowns and low pH minimizes most of the former problems, many students still experience difficulties with the latter. It should be noted that many crucibles are broken during this experiment, and the use of open flames in an undergraduate laboratory is always something to be avoided if possible. The outcome of the experiment is often that students perform a lengthy, tedious experiment, only to obtain unsatisfactory results.

The reaction between barium and sulfate ions is rapid, quantitative, and of known stoichiometry, so it would seem to be an excellent candidate for a titrimetric method of analysis. Unfortunately, a difficulty arises in finding a suitable way of detecting the end point of the titration. Numerous indicators have been tried, with varying success, including Alizarin Red S, Tetradoxiquinone, Thorin, and dimethylsulfonazo III.<sup>4–8</sup> Problems encountered include the necessity of including an organic solvent, such as methanol, to dissolve the indicator, the necessity of prior removal of interfering substances, cost, sensitivity, and toxicity. Other indicators exhibit similar limitations. Methods using potentiometric titrations and turbidity measurements have been more successful, but may require precise and costly equipment, which can present a barrier to some student laboratories.<sup>9</sup>

Another titrimetric procedure used for sulfate determination is conductometric titration, also known as conductometry, one

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of the earliest developed methods of instrumental analysis. This method monitors the conductivity (reciprocal of resistance) of a solution as a titration proceeds. The conductance will vary with the number, size, and charge of the ions involved, and also with the characteristics of the solvent. Therefore, when one ion is replaced by an ion of significantly different conductance during a titration, the conductance will change in a linear manner until the replacement is complete, after which time the line will change to a different slope due to the continued inclusion of additional ions of different conductance ( $\lambda_{\pm}^0$ ).<sup>10,11</sup> As the titration progresses, added barium ion ( $\lambda_{+}^0 = 63.6$  for  $\text{Ba}^{2+}/2$ ) will precipitate immediately, causing no net change in conductivity. However, the sulfate ion ( $\lambda_{-}^0 = 79.8$  for  $\text{SO}_4^{2-}/2$ ) is also precipitated, while the concentration of the added chloride ion ( $\lambda_{-}^0 = 76.3$  for  $\text{Cl}^-$ ) will increase. Because the ratio of  $\text{Cl}^-/\text{SO}_4^{2-}$  is 2:1, net change in conductivity will be minimal. However, after the equivalence point, precipitation is complete, so added titrant will result in an increase in conductivity, which can be seen as a change in slope of a plot of volume of titrant added vs conductivity. The point at which this change in slope occurs signals the end point.<sup>10</sup> Several aspects and applications of conductometric titrations have been the focus of recent articles in this *Journal*.<sup>12,13</sup>

One of the earliest articles to focus on the conductometric titration of sulfate with barium was authored by Kolthoff and Kameda in 1931.<sup>14</sup> These authors pointed out a number of problems with the procedure, including a systematic error consistently resulting in early end point detection, especially in more concentrated solutions. They also observed that conductivity is affected by concentration, and results could be reproducible within 1% in more dilute solutions. Therefore, the technique could be very useful if standardization was made under the same conditions as titrations of unknowns.<sup>14</sup> Because conductivity is dependent upon concentration, a dilution factor of  $(V + V_0)/V_0$ , where  $V_0$  is the initial volume and  $V$  is the volume of titrant added, should also be applied to the conductance at each reading prior to plotting the titration curve.<sup>10,15</sup> Other factors to consider are the solubility of the precipitate, which reaches its maximum value at the end point of the titration and the ionic strength of the solution, which will also affect the solubility of the precipitate and shape of the curve in the vicinity of the end point.<sup>16</sup> For this reason, Pungor recommends that values obtained in the vicinity of the end point be excluded from determination of the end point.<sup>16</sup>

The majority of the errors associated with conductometric titrations of sulfate involve coprecipitation of spectator ions, which is a problem that can be minimized by the use of dilute solutions, and by incorporation of dilution factors as described above. The absence of indicators also removes the need to incorporate organic solvents into the titration solutions. Conductivity meters suitable for performing conductometric titrations are available with a wide range of sensitivities and prices from many suppliers. A highly sensitive, inexpensive (costing less than \$50) hand-held conductivity meter is adequate for student laboratories and sophisticated software for plotting titration curves is universally available for determining the end points of titrations, so sulfate analysis by conductometric titration becomes a viable candidate for inclusion in an undergraduate chemistry laboratory sequence.

## ■ EXPERIMENTAL OVERVIEW

The goal of this project was to develop a simple, inexpensive procedure for sulfate analysis utilizing a conductometric

titration which was suitable for an undergraduate laboratory and validate the results against those obtained using the classic gravimetric method. As previously noted, this is a well-established procedure, and an online search locates numerous descriptions of similar experiments and also applications by instrument manufacturers. However, many of these experiments and applications lack a thorough theoretical discussion and/or analysis of results. As a result, the method is still relatively unappreciated and not widely used.

The accuracy of student results cannot be measured unless the true value of the "unknown" is known. Therefore, the assumption was made that a commercial unknown would be used. These usually have an analysis between that of  $\text{K}_2\text{SO}_4$  (55.12%  $\text{SO}_4^{2-}$  or 45.94%  $\text{SO}_3$ ) and  $\text{Na}_2\text{SO}_4$  (67.63%  $\text{SO}_4^{2-}$  or 56.36%  $\text{SO}_3$ ) and are free of contaminants that would interfere with the analytical procedure. A low concentration of unknown was desired, both to minimize interference by contaminants and to minimize production of toxic barium salts. However, if concentrations were too low, the change in slope at the end point would be too small to detect. A concentration of approximately 3–4 mM was found to be optimal. If an unknown of a markedly different composition was used, the amounts of reagents required would need to be adjusted accordingly.

Quantitative Analysis students first performed a gravimetric sulfate analysis of a commercial sulfate unknown using a classic procedure described in their current textbook, which involved precipitation of the sulfate by an excess of barium chloride, digesting the precipitate overnight, collection of the precipitate by gravity filtration through ashless filter paper, and removal of the paper by charring over a Bunsen burner. The dried precipitate was weighed, and the % $\text{SO}_3$  in the sample was calculated.

Approximately one month later, the students were again issued a commercial sulfate unknown sample to analyze using the conductometric titration procedure developed in this laboratory. (A copy of this handout can be found in [Supporting Information](#).) They were informed that these unknowns were similar in composition to those used for the gravimetric analysis, and the precipitation reaction involved would be the same. The students dried their unknowns and samples of standard  $\text{K}_2\text{SO}_4$ , which was used to standardize their  $\text{BaCl}_2$  titrant.  $\text{BaCl}_2 \cdot 2 \text{H}_2\text{O}$  is not a primary standard because it decomposes above 100 °C, while  $\text{K}_2\text{SO}_4$  meets all of the requirements for a primary standard. Students prepared solutions containing approximately 1.5 g of  $\text{BaCl}_2 \cdot 2 \text{H}_2\text{O}$  per 500.00 mL. Each student also prepared 500.00 mL solutions containing approximately 0.30–0.35 g of their unknowns (in duplicate) and one of their standard  $\text{K}_2\text{SO}_4$ . Each student was issued an Oakton Eco Testr EC Low Pocket Conductivity Tester. The student then filled a buret with his/her  $\text{BaCl}_2$  solution and proceeded to titrate a 100.00 mL aliquot of the standard and each unknown solution, recording the conductivity at 1.00 mL increments. The data was then graphed using MS Excel and splitting the data into two segments at the minimum conductivity and obtaining a trendline for each segment. A typical student titration curve is shown in [Figure 1](#) below.

The titration curve in [Figure 1](#) has not been corrected for dilution. The end point was determined by setting the two trendline equations equal to each other and solving for  $x$ , which is the point at which the two lines intersect. This was calculated to be 28.04 mL.

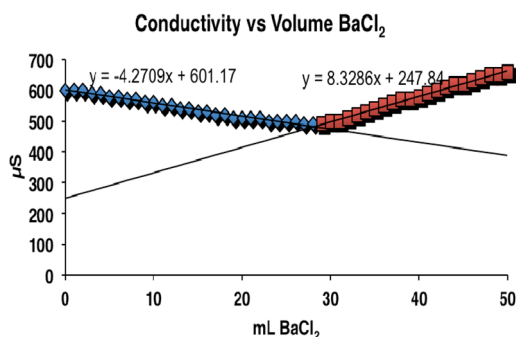


Figure 1. Titration curve showing trendlines and equations.

The same data, corrected for dilution, is shown in Figure 2.

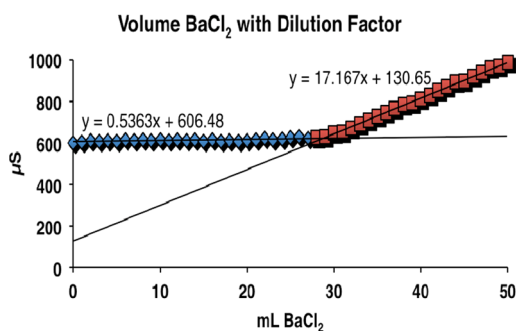


Figure 2. Titration curve, corrected for dilution, showing trendlines and equations.

Note that incorporation of the dilution factor resulted in an almost constant conductivity value prior to the end point. Also, this resulted in a larger titration volume, 28.61 mL for this sample, which yielded a more accurate result. Dilution corrected titration curves were used as the basis for all later student calculations.

Each student then calculated the molarity of his/her  $\text{BaCl}_2$  solution based on the weight of the standard  $\text{K}_2\text{SO}_4$  titrated and the volume of the end point. This molarity and the end point volumes for the unknown titrations were then used to calculate the  $\%\text{SO}_3$  in the unknown samples. (A copy of a typical data set and calculations can be found in [Supporting Information](#).)

All students wrote and submitted Lab Reports for both experiments in the standard manner required for all experiments for this course.

As per university policy for all experimental procedures involving students, both authors underwent IRB training and an IRB approval form was submitted to the University Institutional Review Board for review. The application was approved as Exempt (under 45 CFR 46.101b1), but IRB protocols were followed throughout the duration of the experiment.

## EXPERIMENTAL PROCEDURES

These two experiments were carried out by 12 students in an undergraduate Quantitative Analysis course. Previous laboratory experience ranged from 2 semesters of General Chemistry to Qualitative Organic Analysis and Instrumental Analysis. Students are assigned two laboratory sessions per week, for a total of 6 h, although days and times vary according to individual student schedules and laboratory space constraints.

Students receive a handout on the laboratory procedure the week previous to the scheduled lab. This handout includes

information on the approximate concentration range of the unknowns for gravimetric analysis so that the students can calculate the theoretical volume of precipitating reagent solution required for complete precipitation of their unknowns (plus 10% excess). On Monday the procedure is reviewed in class and they may check out unknowns, if they have already copied the procedure into their lab notebooks, so that unknowns can be oven-dried (if necessary) at  $110\text{ }^\circ\text{C}$  for at least 18 h prior to the experiment. It is also appropriate to remind the students during the review that these “unknowns” are relatively free of interfering substances and have a discussion of how the procedure should be modified for “real” unknowns.

For the gravimetric procedure, Day 1 included precipitation and cleaning of crucibles. Although most students only required 2 h, a few used the entire lab period. Day 2 included filtration and charring of the precipitate. Filtration is a slow process, but students could filter three samples simultaneously, although a few samples required refiltration when precipitate was observed in the filtrate. Samples were charred individually, in a hood, and some students were in the lab for as long as 6 h. Several students had difficulty with samples flaming, and three crucibles were broken during the process. Crucibles were then placed in the oven for at least overnight, and the students returned to the lab whenever they could find time for a final weighing. Total lab time ranged from about 6 to over 10 h for this experiment.

Because the conductometric titration had never before been utilized in an actual formal student laboratory at this institution, a decision was made to attempt to run the entire experiment in one 3 h lab session. If problems occurred, the second period would be used for reruns. Upon the basis of previous experience with students initially performing potentiometric titrations, 1 h was allowed per titration, although experience with research students familiar with the procedure showed that these titrations could be done in half of the allotted time. Each student worked individually to perform three titrations: one for standardization of their  $\text{BaCl}_2$  titrant and two for their unknown. However, each student shared a common  $\text{BaCl}_2$  solution with one or two other students. Since 500.00 mL of these solutions was prepared and each student only required three 50 mL amounts for their titrations, this presented no issues and had the advantages of providing a backup value if something went wrong with a standardization, monitoring consistency of results among different students using the same solutions, and minimizing  $\text{BaCl}_2$  usage. This turned out to be a wise precaution, because two students used sample weights at the upper end of the suggested sample weight range for their standardization titrations. When results were analyzed, it was found that the resulting titrant volume at the end point was too large to allow for a good end point determination. These students then used the average standard concentration values of their “partners” for their unknown calculations, and the suggested sample weight range in the protocol was reduced to a maximum of 0.35 g for future experiments. It was also later discovered that one student accidentally used her sample of standard  $\text{K}_2\text{SO}_4$  for one unknown titration instead of her unknown, so that result was graded against the  $\%\text{SO}_3$  in pure  $\text{K}_2\text{SO}_4$ . No other problems were noted. All students completed the experiment within the allocated 3 h.

Students were instructed to apply the correction factor for dilution prior to determining the end point of the titration graphically as described in the previous section. As shown in Figure 2, this results in an almost flat line prior to the end point



Table 1. Comparison of Individual Student Results for Titrimetric and Gravimetric Analysis

ID No.	Conductometric Titration (% SO <sub>3</sub> )				Gravimetric Method (% SO <sub>3</sub> )		
	BaCl <sub>2</sub> (M)	Unknown	Avg	Error	Unknown	Avg	Error
1	0.01313	51.94	52.00	0.06	55.91	54.70	-1.21
2	0.01283	49.68	49.23	-0.46	49.68	48.16	-1.52
3	0.01327	50.99	50.10	-0.89	49.68	56.90	7.22
4	0.01293	53.11	52.82	-0.29	53.11	52.25	-0.86
5	0.01260	54.56	53.86	-0.70	54.56	53.32	-1.24
6	0.01277	52.27	52.52	0.24	51.94	50.02	-1.92
7	0.01351	52.89	51.99	-0.90	50.99	54.38	3.39
7'	0.01351	45.94	44.29	-1.65			
8	0.01239	51.94	50.29	-1.66	51.94	50.11	-1.83
9	0.01289	50.28	52.15	1.86	53.38	52.56	-0.82
10	0.01318	53.11	52.99	-0.13	53.11	51.64	-1.47
11	0.01384	53.38	54.95	1.57	55.05	50.84	-4.21
12	0.01251	50.28	49.04	-1.25	50.28	48.91	-1.37
AVG				0.90			2.26

and an increase in slope after the end point. However, some titrations exhibited minor fluctuations in conductivity near the end point region which made it difficult to decide where to separate the data for the two trendlines, which would have a marked effect on the final results. Therefore, students were instructed to utilize the computer to decide where to separate the data. The initial "cut" was made at the point where the conductivity was definitely beginning to increase, and  $R^2$  was added to the trendline options. Then, points immediately adjacent to the intersection of the trendlines were selectively removed, one at a time, and the effect on  $R^2$  was noted. As soon as a decrease in  $R^2$  was noted, that point was restored and the process was repeated for the second trendline. When  $R^2$  had been maximized, indicating that the linearity of both trendlines was optimal, the resultant trendline equations were used to calculate the volume at the end point. This only resulted in the exclusion of a few points, but results were improved over those obtained by a random decision by the analyst. It should be noted that this is consistent with the observations and recommendations of Pungor.<sup>16</sup>

## EQUIPMENT

Oakton Eco Testr EC Low Pocket Conductivity Testers, range 0.0–1990  $\mu$ S and resolution 10  $\mu$ S, were obtained from Cole-Parmer and used per instructions.

## CHEMICALS

Barium chloride dihydrate, CAS 10326-27-9, ACS, crystalline, was obtained from Alfa Aesar and was used without further purification. Potassium sulfate (Powder/Certified ACS), CAS 7778-80-5 was obtained from Fisher Chemical and used without further purification except for oven drying at 110 °C overnight prior to use. All solutions were prepared using regular deionized water shortly before use.

Analyzed quantitative sulfate unknowns were obtained from Thorn Smith Laboratories.

## HAZARDS

Care must be taken in handling all chemicals. Hydrochloric acid (gravimetric procedure) is corrosive to skin and eyes and an irritant to the respiratory tract if inhaled. Barium chloride is hazardous in the case of skin or eye contact and toxic if ingested or inhaled. Severe overexposure can be fatal. Potassium sulfate

(titrimetric procedure) is only slightly hazardous, but contact with skin and eyes should be avoided. Barium sulfate is slightly hazardous in the case of skin or eye contact, inhalation, or ingestion. It is toxic to lungs and mucous membranes, and repeated exposure can cause tissue/organ damage. Therefore, the charring process in the gravimetric lab should be carried out in a hood, and solid product should be collected from the crucibles prior to cleaning for proper disposal. Proper clothing, shoes, gloves, and goggles with splash protection are required, and caution must be utilized in handling Bunsen burners.

## RESULTS AND DISCUSSION

Student results for the conductometric and gravimetric sulfate methods are summarized in Table 1. Students 7 and 9 used their partners' average values for their BaCl<sub>2</sub> titrant concentrations, and the value for Student 7' is for standard K<sub>2</sub>SO<sub>4</sub> as the unknown. The average errors are the averages of the absolute errors.

Examination of the students' results reveals that conductometric titrations results were generally low (9 out of 13) compared to the accepted values, as predicted by Kolthoff.<sup>14</sup> However, the same trend is observed in the gravimetric procedure results, where 10 out of 12 results were low. Although individual student outcomes varied, the average absolute error associated with the conductometric titration was less than that associated with the classic gravimetric technique. However, with a sample pool this small, a few extreme results can greatly influence the average. It should be noted that the extreme results were more prevalent in the gravimetric method (standard deviation = 1.86% SO<sub>3</sub>) than in the titrimetric method (standard deviation = 1.09% SO<sub>3</sub>). The ratio of the variances showed that the two methods were statistically different ( $F = 2.92$ ), which makes a comparison of the accuracy of the two methods problematic. The few modifications made to the conductometric protocol as a result of the outcomes of this experiment, such as reducing sample weights of the K<sub>2</sub>SO<sub>4</sub> standard, should improve this in future studies. Also, since the time required for the titrations was less than had been anticipated, unknowns can be run in triplicate in future trials to obtain better precision and allow more meaningful statistical comparison of the two methods.

This experiment provided students with experience in using a technique which is not part of the standard undergraduate lab experience and of obtaining titrimetric data which were

processed and interpreted using computer techniques which they had not yet encountered. This generated more interest in the experimental process than usual and is probably responsible for an unanticipated side result observed when the lab reports were graded.

The two sulfate analyses had the highest average grades on lab reports of all of the experiments written for the course, and the conductometric method outscored the titrimetric method by 5 points, even though it was a more challenging topic. Although this is not a statistically large difference, it should be noted that eight out of the 12 students had increased scores. The lab report grade is based upon a rubric which evaluates multiple factors, including quality of writing, discussion of all physical and chemical phenomena in the experiment, safety precautions, data analysis, and references. Several of the students became very engaged, researched the literature thoroughly, and submitted reports that were notably above their usual quality, although two students had about the same grade and two other students found the lack of “easy” references too challenging and, as a result, submitted weaker reports.

## CONCLUSION

Analysis of an unknown sulfate containing sample by undergraduate students in a Quantitative Analysis laboratory by conductometric titration with barium chloride produced results as accurate as those obtained using the classic gravimetric procedure. The method was also less time-consuming, more predictable, and less hazardous than its gravimetric analogue. A less tangible benefit was allowing the students the opportunity to experience an analytical technique not commonly encountered in undergraduate laboratories and further develop computer skills. These factors, combined with the availability of inexpensive hand-held conductivity meters, make the conductometric titration of sulfate by barium chloride a strong candidate for inclusion in an undergraduate chemistry laboratory sequence.

## ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: [10.1021/acs.jchemed.5b00941](https://doi.org/10.1021/acs.jchemed.5b00941).

Student laboratory handout, notes for instructors, sample student results (PDF, DOC)

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### Notes

The authors declare no competing financial interest.

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