Spring 5-14-2015

Removal of Pb and H2S from ACS Test Procedure For Meta-Phosphoric Acid

Christopher Atlas
Chapman University, atlas102@mail.chapman.edu

Ron Lacock
Chapman University

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Atlas, Christopher., Lacock, Ron.
Schmid College of Science and Technology/Chemistry, Chapman University, Orange, CA

Abstract

In the pursuit of green chemistry, adequate substitutes for lead acetate and hydrogen sulfide (H_2S) were investigated for use in the American Chemical Society test procedure for stabilizer (Na_2PO_3) in meta-phosphoric acid. In the current ACS procedure, lead acetate ($Pb(C_2H_3O_2)_2$)) is used to form insoluble lead phosphate. To remove lead from the solution, lanthanum chloride was investigated with positive results. Hydrogen sulfide is currently used to precipitate lead by forming insoluble lead sulfide. Several substitutes, including oxalic acid and sulfuric acid, were investigated. Preliminary testing showed that lanthanum chloride and oxalic acid are sufficient as replacements for lead acetate and hydrogen sulfide.

Hypothesis and Objective

Hypothesis: General knowledge of chemistry and solubility suggest that lanthanum chloride and oxalic acid may be suitable substitutes to replace lead acetate and hydrogen sulfide, and will be equivalent in their ability to precipitate lead and phosphate out of the sample solution.

Objective: Prove that lanthanum chloride and oxalic acid show equivalent results to the current ACS method.

Experimental Method

Weigh accurately 20.0 g of sample, dissolve in water, and dilute to 1 L. To 50.0 mL of this solution in a 200 mL volumetric flask add 50 mL of DI water and 50 mL of 5% $LaCl_3$ reagent solution, dilute with water to 200 mL, mix thoroughly and decant through a filter. Treat 100 mL of the clear filtrate with 5% oxalic acid and mix well, filter, and wash with about 20 mL of DI water. Add 2 mL of sulfuric acid, evaporate to dryness in a tarred preconditioned dish, and ignite to a constant weight at 800 +/- 25 degrees Celsius. Calculate the weight of stabilizer (Na_2PO_3) using the following expression:

$$\text{Wt } Na_2PO_3 = \text{Wt } NaSO_4 \times 1.4356$$

Reagent	Lot Number	Catalogue Number
Meta-Phosphoric Acid	1AA0591 (#1)	P1098
Meta-Phosphoric Acid	1DE0230 (#2)	P1098
Meta-Phosphoric Acid	1DI0480 (#3)	P1098
Lead Acetate	1CJ0354	L1040
Oxalic Acid	1DE0049	01025
Lanthanum Chloride	1DH0437	L1021
Ammonium Hydroxide	N/A	N/A

Table 1: Reagents

Test	Values
Assay (HPO_3)	33.5-36.5%
Stabilizer (Na_2PO_3)	57.0% - 63.0 % (Maximum allowable)
Nitrate (NO_3)	0.001%
Sulfate (SO_4)	0.005%
Chloride (Cl)	0.001%
Heavy Metals (as Pb)	0.005%
Iron (Fe)	0.005%

Table 2: ACS test specifications for meta-phosphoric acid.

Results

The ACS specifies the acceptable percentage of stabilizer (Na_2PO_3) in meta-phosphoric acid to be between 57.0 and 63.0% (See Table 2). The current ACS test procedure was used on three different lots in order to have a base of data for comparison. (See Table 3) Lot 1 produced an average of 58.76% which is within the accepted ACS specification range. Lots 2 and 3 produced averages of 53.23% and 52.93% respectively. These results were low due to sample loss during ignition, therefore Lots 2 and 3 were excluded from comparison.

Lot 1 produced valid ACS results with the current method and was therefore chosen as a comparison point. The proposed experimental method was run on Lot 1. (see Table 3) This produced an average of 85.04% which is about 30% higher than the accepted ACS value range.

To determine the cause of the increase in % weight for Na_2PO_3 a phosphate test was run on all three samples from Lot 1. All three samples failed the phosphate test. These results indicate that lanthanum chloride did not fully precipitate phosphate out of the sample solution.

Table 3: Test Results.

Lot Number	Method	Results	Average	Notes
1	Original ACS Method	58.65% 59.04% 58.60%	58.76%	Values are in accepted range
2*	Original ACS Method	52.04% 59.60 % 45.06%	52.23%	Sample Lost. Values lower than accepted range
3*	Original ACS Method	N/A 60.00% 45.85%	52.93%	Sample Lost. Values lower than accepted range
1	Proposed Method 1	84.95% 85.04% 85.13%	85.04%	30% higher than accepted
1	Method 1 (Adjusted)	77.90% 78.10% 77.87%	77.95%	25% higher than accepted

It was proposed that increasing the rate of reaction would improve the amount of phosphate precipitated out of the solution. The proposed method was adjusted. Before the $LaCl_3$ was added to the sample solution, the sample was brought to a gentle boil and the $LaCl_3$ was added. The sample solution was then allowed to digest for 5 minutes..

Raising the temperature of the sample solutions before adding the $LaCl_3$ lowered the % weight of Na_2PO_3 by approximately 5%. However the % weight of Na_2PO_3 remained 25% higher than the allowed ACS specifications.

Subsequently a test for phosphate and lanthanum was then run to ensure that both phosphate and lanthanum were precipitated out of solution. The samples tested positive for phosphate. They did however test negative for lanthanum. This indicates that lanthanum chloride may not be a suitable replacement for lead acetate. Due the failure of lanthanum chloride in the first step, it was not possible to determine if oxalic acid was a suitable substitute for H_2S.

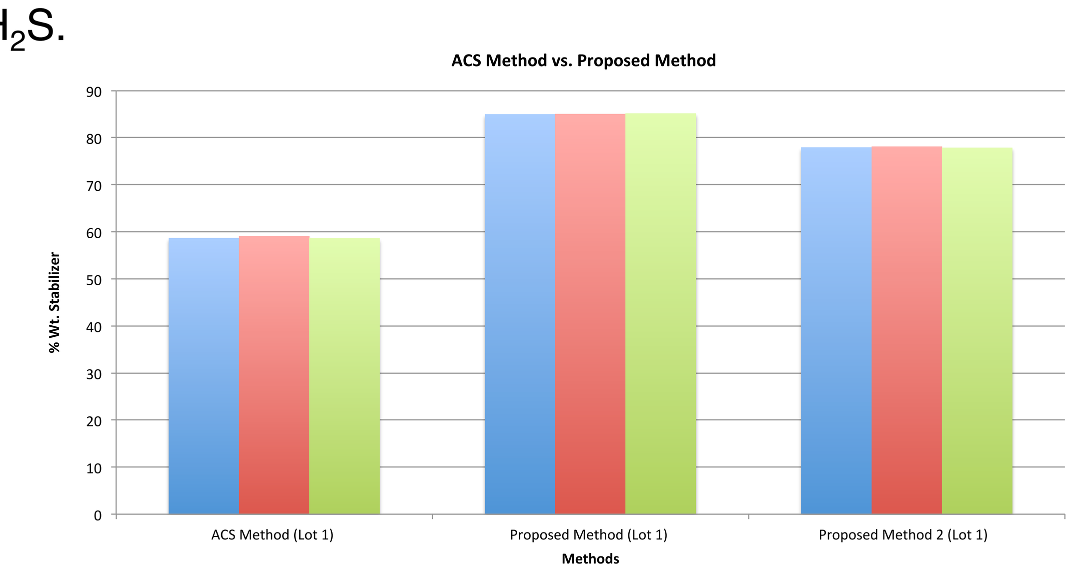

Figure 1: Current ACS method compared to proposed method.

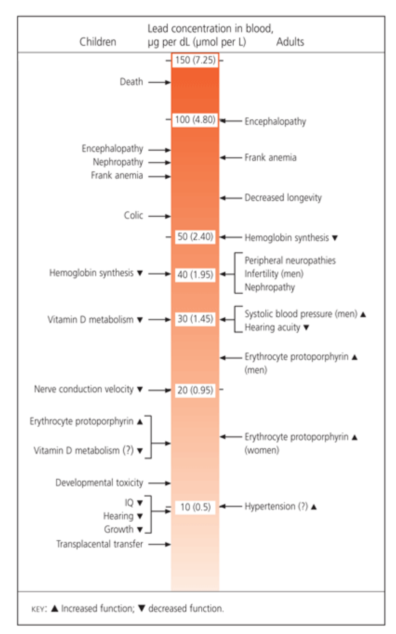

Figure 2: Lead toxicity in children and adults.

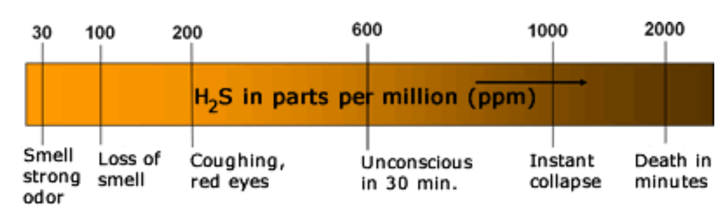

Figure 3: Hydrogen Sulfide toxicity (ppm).

Conclusion:

The purpose of this research project was to find suitable substitutes for lead and hydrogen sulfide in the hopes of removing two harmful substances from an ACS test procedure. Lead is well known to be toxic to humans and the environment, and expensive to dispose. (Figure 2) Hydrogen Sulfide is a deadly, foul smelling gas that can cause unconsciousness and possible death at concentrations as low as 600 ppm. (Figure 3) Due to the health concerns and fiscal liability in the handling and disposal of these compounds it has become imperative to find safer alternatives.

Our hypothesis was proven to be wrong. These substitutes produced results that were far above the accepted ACS specifications for stabilizer in meta-phosphoric acid.

Preliminary work, not included in this study, suggests that adjusting the pH and temperature of the sample solutions yields positive, reproducible results that indicate $LaCl_3$ and oxalic acid may be viable substitutes for lead acetate and hydrogen sulfide. Preliminary testing also

Conclusion (cont.)

indicates that adjusting the current ACS method by substituting concentrated sulfuric acid for hydrogen sulfide may be a successful alternative to precipitate lead out of solution therefore eliminating hydrogen sulfide from the test procedure.

Future Research

Below are suggested potential variations that may improve the results of the experimental method and allow for the complete removal of lead and/or hydrogen sulfide

1) Addition of ammonium hydroxide to the adjusted Method 1, in order to adjust pH of the sample solution.

2) Substituting concentrated sulfuric acid for hydrogen sulfide in the current ACS method in order to precipitate lead.

Acknowledgements

This project could not have been completed without the help and guidance of my faculty advisor Dr. Dan Wellman and Spectrum Chemical Senior Chemist Ron Lacock. Ron's expertise proved to be invaluable through out this process. I would also like to give a huge thanks to V.P of Quality and Technical Service Tom Tyner, who allowed me to use the Spectrum Chemical laboratory, and to all of the chemists at Spectrum who never hesitated to help me achieve my goal.

A special thanks to Spectrum Chemical for allowing me to conduct my research in their amazing laboratory.

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