

CH 2212

Colorimetric Determination of Iron in Food Products and Supplements

Ref: R.C. Atkins, "Colorimetric Determination of Iron in Vitamin Supplement Tablets - A General Chemistry Experiment" *J. Chem Educ.* (1975) 52, 550.

Introduction

It has been long recognized that certain trace organic compounds (vitamins) are essential for proper biological function. So, too, is the case for certain minerals necessary for enzymatic activity, cellular function and structural maintenance. Since the human body cannot synthesize these compounds, we are required to ingest them from exogenous sources.

It is common to encounter dietary supplements in a variety of foods. Vitamins and minerals necessary for human physiology are commonly added during the manufacturing and packaging processes to augment the amounts naturally present in the raw foodstuff materials. Witness the ingredient labels on your "fortified" breakfast cereal, for instance (as well as the FDA "Minimum Daily Adult Requirement").

The vitamin and mineral contents of fortified food, vitamin supplements, etc. are typically verified on a lot-by-lot basis by quality control laboratories maintained by the product's manufacturer. Occasionally, an outside contract laboratory is retained to perform the quality control tests. In a quality control environment, time is critical - the product doesn't generate revenue for the manufacturer sitting in a warehouse waiting for the QC results.

Iron is a common dietary supplement in a variety of foods. While there are many ways to quantitate iron in foodstuffs, one of the simplest (and therefore least time-consuming) methods is the colorimetric determination of the iron-phenanthroline complex at 508 nm. The concentration of the iron can be determined from a standard colorimetric calibration curve.

The degree to which other ingredients interfere is not known (at least for the purposes of this exercise).

Your group (lab) has been asked to bid on a contract with Bozo Foods, Inc. involving the quantitative determination of iron in their products as an integral part of Bozo Food's outstanding long-term commitment to bringing healthy and wholesome products to the nation's table. Bozo Foods has specified that you adapt the referenced procedure to the determination of iron in the food products and/or supplements provided (competitor's products, but representative of those produced by Bozo).

You will again work in your assigned groups. Each group is to function as an independent testing laboratory. It should have a safety officer, a quality control officer and a lab manager who is responsible for overseeing all operations of the "lab". The distribution of duties is done by the group.

Each group (lab) will have one bench and one hood for its exclusive use. This group has sole responsibility for cleaning up the assigned bench and hood at the end of each lab period.

Safety

Prior to the start of any laboratory work, a complete safety plan must be formulated, written and submitted for approval. Since it takes time to evaluate each lab's safety plan, do not expect to submit it at the beginning of a lab period and start working immediately. The safety plan should be submitted a day prior to the anticipated start of lab work (at the latest).

The safety plan should address:

- storage/handling/disposal of samples and reagents
- copies of all pertinent MSDS should be included (c.f. <http://www.msdsonline.com/>)
- cleanup of spills
- use of balances
- personal protection needed
- procedures to follow in case of exposure to hazardous chemicals
- where operations involving undiluted samples will be performed

Quality Control

Your testing laboratory is expected to provide quantitative results that are within 2 percent of the true value. Assume a 95% confidence interval. A QA/QC (quality assurance/quality control) protocol should be formulated and submitted for approval prior to the start of careful laboratory work. (Some preliminary testing will likely be needed to establish rough concentrations, etc. The method development phase should also precede formulation of a final QA/QC protocol.)

Your written QA/QC protocol should address:

- do you need to calibrate your balance?
- do you need to calibrate your volumetric glassware?
- how are you going to evaluate analytical precision?
- calibration of spectrophotometers
- method of calculating the 95% confidence interval
- measurement of precision of each step in the analysis
- calculation of overall method standard deviation
- calculation of the number of experimental results needed to report your answer to $\pm 2\%$ with 95% certainty

Method Development

The requirements are few:

- Your quantitative results must be based on the colorimetric method provided.
- Weighing of primary standards on the analytical balances must be done by the "weighing by difference" method.
- A thorough investigation of sample interferences must be made to satisfy the (somewhat irrational?) demands of the QC department at Bozo Foods.

There will likely be an "inter-laboratory quality assurance sample" given to each of the labs after your procedures are established. The results for each "lab" will be posted so that you can compare your method/technique to the other "labs".

The success of your laboratory's bid for this contract will be enhanced greatly (translation: extra credit) by the development of a colorimetric procedure for the determination of iron in Total breakfast cereal. (The QC staff at Bozo Foods have not been able to find a reliable procedure to quantitatively extract the iron from the cereal flakes.)

Report

Calculate the cost of this analysis to a hypothetical paying customer, assuming a laboratory personnel rate of \$50/hour per person (cheap in the real world). Include this calculation in your report.

Turn in (second class period after ceasing lab work) a hard-copy report detailing your method and stating your results. Report all raw data and justify your results by indicating all statistical evaluations performed. All data tables, charts and graphs should be amenable to electronic publishing (see below).

Turn in a digital copy of your report.

Turn in the group notebook.