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The WCAS Analytical Digest

WEST COAST ANALYTICAL SERVICE, INC.

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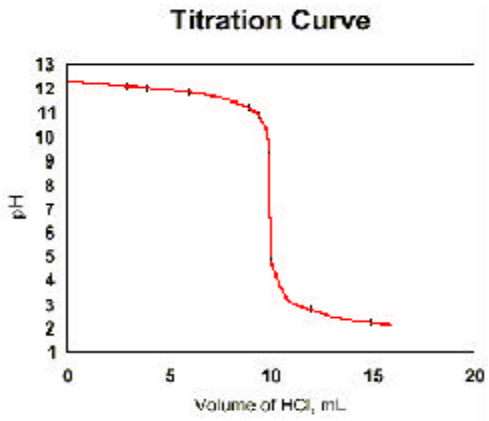
wcas.com

Titrations 101

Titrations are generally used in analytical chemistry for very accurate and precise determinations of % level components as opposed to trace amounts (>1 ppm). The most common example of a titration is determining the concentration of acid or base in solution by measuring how much of the other (base or acid) it takes to neutralize the sample. The precision and accuracy of titrations make it an excellent choice for performing assays with tight specifications.

Lets take a closer look at a titration of a basic sample, say a potassium hydroxide solution. We want to determine the concentration of hydroxide in the sample, so we will measure out a small volume (50 mL) and add, very slowly with constant swirling, small amounts of hydrochloric acid. Now the acid has been previously standardized so we know its exact concentration (0.100 molar). And we measure the amount of acid added to the sample using a long, volumetric device called a buret, able to measure volumes of 0.1 mL or less. Before we add anything to the sample, the potasium hydroxide solution has a very high pH (>12). As we add acid, the pH does not change very rapidly until we get close to the end point, the point at which the moles of hydrochloric acid almost equals the moles of hydroxide in the sample. Then the pH changes very rapidly. If we continue to add acid, the pH will become low (<3) with only a slight excess in the number of moles of acid.



Now we don't need to take sub-samples out of the acid-base mixture to measure the pH with a meter. Instead, we add a very small amount of an indicator that changes color near the pH range of the end point. In this case the end point has a pH of 7.00. As soon as we get close to the end point, we will note that the new color lasts longer with each drop of titrant added.

So how do you determine how much hydroxide was in your original sample?

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Cadmium in Plastics

WCAS is pleased to announce that we participated in an international, interlaboratory study on cadmium (Cd) in PVC during October 2001. The study was conducted by the Institute for Interlaboratory Studies (Dordrecht, NLD). The study consisted of 3 commercial samples of PVC plastic with varying amounts of Cd. The purpose of the study was provide precision and accuracy data in support of EC restrictions on total Cd to 100 mg/Kg. Thirty-two laboratories from 19 countries participated.

Since we routinely test for Prop 65 and environmental regulations in many products, especially lead (Pb), cadmium (Cd), and other toxic, heavy metals, we were quite interested in participating in this study. Over the years WCAS has participated in studies from USEPA, USGS, NIOSH, ASTM, CA ELAP and CDFA, and others, some required for accreditation and others strictly voluntary.

While the entire report can be viewed at http://www.iisnl.com/pdf/Iis99p01.pdf, the results are summarized below:

Sample	Mean Result mg/kg	WCAS Reported	Z*
0105	596.6	505	-1.72
0106	166.2	173	0.46
0107	Very low	0.8	

* |Z| < 1 good, 1 < |Z| <2 satisfactory, 1 < |Z| < 3 questionable, |Z| > 3 unsatisfactory

Sample 0107 was chosen to have little or no Cd. Many labs reported ND. With ICPMS, our detection limit was <0.01 mg/kg.

Quick Quotes

Politeness is the art of selecting among one's real thoughts.

Madame de Stael

Always do what you say you are going to do. It is the glue and fiber that binds successful relationships.

Jeffry A. Timmons

AAPS Show Giveaway winner!!

We attended the American Association of Pharmaceutical Scientists (AAPS) show in Denver, Colorado from October 22 thru October 24, 2001. Will Harcum at Hercules Inc in Wilmington, Delaware won the lightning piece of art. Congratulations and we hope you enjoy.

WCAS • 9240 Santa Fe Springs Road • Santa Fe Springs, CA 90670 • 562.948.2225 • fax 562.948.5850

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Sulfite by IC

Sulfite (SO_3^{-2}) in solution is easily oxidized by air to sulfate (SO_4^{-2}). Since sulfate is very common, this complicates the direct analysis of sulfite. This sulfite test is used to test for free sulfite in solution as well as for sulfur dioxide trapped in impingers or on impregnated filters.

We have recently found an improved method for analysis of sulfite in aqueous samples. The method is based on NIOSH 6004, and uses 2% glycerol to stabilize the sulfite ion, minimizing oxidation to sulfate. We have seen good stability of a sulfite standard sitting overnight at room temperature. Sulfite elutes between phosphate and sulfate in our standard EPA 300.0 inorganic anion system.

Some researchers have reported improving the stability of sulfite by using alcohols such as glycerol, saccharides, and formaldehyde. The latter is reported to work even in the presence of ferric ions.

This suggests that one might consider adding a preservative in the field to environmental samples to stabilize the sulfite, before shipping to WCAS.
The chromatogram at wcas.com shows rain water samples spiked with 150 µg/L sulfite and sulfate, with and without glycerol added, and allowed to stand overnight. With glycerol, sulfite was recovered at almost 80%.

Titrations 101

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Here's how:

Molarity (number of moles per liter of hydroxide) = $\frac{\text{Volume of acid } \mathbf{x} \text{ acid conc.}}{\text{Volume of Sample}}$

So, if it takes 10.0 mL of 0.100 molar HCl to titrate 50 mL of hydroxide solution to its end point (pH = 7), then the original sample contains 0.020 molar hydroxide (molecular weight 17), or 0.85 g/L of hydroxide.

Now this is an example of a very simple acid-base titration. Other examples include oxidation-reduction or redox (e.g. iodine-thiosulfate), precipitation (e.g. chloride-silver nitrate and calcium-EDTA), potentiometric, and a large number of variations and combinations involving all kinds of exciting and interesting chemistry.

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Our Moving Saga

On November 1, 2001 we moved into our new location at 9240 Santa Fe Springs Road in the city of Santa Fe Springs, California. It took us about a week to move the 16,500 square foot lab with 17 years of accumulation to our new 25, 000 square foot facility. Most of the lab furniture and hoods are new with five of the hoods designed to handle our acid digestions. Our new location has plenty of parking and room for expansion of our services. We have installed an alarm system with CCTV monitoring and a T1 to the Internet.

The saga began three years ago. It took a year to locate potential buildings and to settle on the most likely prospect. It took another year to negotiate a lease agreement. This was quite unexpected; there were lots of road blocks in coming to a fair agreement. After the lease it took another six months for designs and permits. The next five months (originally estimated for 10 weeks) was occupied with the tenant improvements.

But finally we received our occupancy permit on November 1. We notified our staff immediately and the move began. Client services, phone and network, wet area and IC/LC were up and running Monday November 5th. Other areas accomplished most of their moving that weekend and spent the rest of the week supervising the moving of large equipment. We were up and running within a week of the occupancy announcement.

For those familiar with Southern California we are located in the old 'Fedco' corporate building. We have enhanced the location with security, installed network and phone system with a universal cable plan, plus an excellent patch panel layout that made network and phone setup easy.

We have plenty of space and parking. With the new lab furniture and facilities, we hope to service our clients even better. We're working hard to do so and are planning for future growth.

Our thanks to the many friends, staff members and contractors who helped along the way.

Especially....

- Shelley Stuart, Air Products who came through with needed supplies.
- King Office Relocation, for moving the office furniture, files, and lab equipment.
- Mark Watkins and Marsh Tanner of Shepard, Mullin, Richter, and Hampton.
- Genie Scientific who planned the lab furniture move and provided all the new furniture.
- JMG Security for the alarm and CCTV system.
- Mike Hovanec who helped complete the gas and DI services.
- Greg Shaw and CableLan for the network and phone cabling and patch panel lay out.
- Source One for helping move the Altigen phone server.
- Rreef Management, our prior landlord, who didn't rush us out of our old location.
- Clients who were willing to work with us during this time.

Thanks again,

Jack and Ramona Northington