Experiment 36

Determination of Nicotine in Tobacco: A Non-Aqueous Acid-base Titration

The theory applied to acid-base titrations can also be applied to non-aqueous acid-base systems. For example, cigarettes contain several organic bases known as alkaloids? Nicotine is the most well known and abundant of these alkaloids and it has a molecular weight of 162.12 g/mol. In fact 90% of the alkaloid content in cigarettes is from nicotine or nornicotine.

In water the basicity of nicotine is too weak to permit an accurate acid-base titration. However, in an acidic non-aqueous solvent such as acetic acid, nicotine is readily quantitated by an acid base titration according to the following equation:

Experimental

(i) Into an Erlenmeyer flask accurately weigh a 6 gm sample of tobacco (6-9 cigarettes without the paper and filter components). Record this data in your lab notebook as well as the brand name of the cigarettes.

(ii) To the flasks add approximately 50 ml of the saturated aqueous $Ba(OH)_2$ solution and 2 gm of granular $Ba(OH)_2$. Insure that the tobacco is thoroughly wetted. Into the flask, pipet 100.00 ml of toluene, add a stirring bar, stopper the flask, and magnetically stir for 20 minutes.

(iii) After 20 minutes filter most of the organic layer through a whatman No.2v folded filter paper into another clean, DRY Erlenmeyer flask. The aqueous layer should not be poured into the filter.

(iv) Into a clean, DRY Erlenmeyer flask pipet 20.00 ml of the filtered solution. Add 4-5 drops of crystal violet indicator. Using your burette filled with your standardized 0.1M $HClO_4$ titrate to the characteristic greenish yellow endpoint. Repeat Step 4 two more times for reproducibility.

Factor : Each ml of 0.1 M $HClO_4$ = 0.0162 gm of nicotine.

Note : The perchloric acid is standardized by titration against potassium hydrogen phthalate.

(vi) Add the little ethyl alcohol and at once evaporate off, and dry the residue, consisting of strychnine and brucine, at 100^0C and weigh.
Record the weight of the alkaloidal mixture ————————

(vii) The process for separating strychnine from brucine depends on the greater readiness by which brucine is nitrated with HNO_3.(*Note. The separation of brucine from strychnine is most conveniently effected by exposing the mixed alkaloids to the action of diluted nitric acid, which destroys brucine very rapidly while having no appreciable action on strychnine).*

(vii) Dissolve the alkaloidal mixture in 10ml of dilute H_2SO_4. Using 50ml of H_2O, filter into a graduated cylinder. Pour the cylinder content into a flask and add exactly 5ml of concentrated HNO_3. The addition of HNO_3 should cause the solution to attain a crimson color. After standing for exactly 15 minutes, transfer the liquid to a separatory funnel and at once make alkaline with NaOH solution, and extract the strychnine with three portions of chloroform in a usual way. After boiling off the chloroform in an evaporating dish, add a little ethyl alcohol and evaporate to dryness. After drying at 100^0C, weigh the residue of strychnine.

Note : *Brucine may be separated from strychnine by virtue of the lesser solubility of its oxalate in dehydrated alcohol or of its hydriodide in water, or by the insolubility of strychnine chromate in water. Potassium ferrocyanide precipitates the strychnine from a hydrochloric acid solution of the alkaloids, the brucine remaining in solution. Brucine is crystallizable from aqueous alcohol, the crystals then contain $4H_20$. It is without odor, but of a permanent, harsh, very bitter taste; is sparingly soluble in water; very soluble in alcohol, whether hot or cold; it dissolves in 4 parts of chloroform, 440 parts of ether, 60 parts of benzene, and 120 parts of petroleum benzin. It is permanent in the air. The hydrated crystals melt at 105° C., while the anhydrous base melts at 178° C., changing color, and depositing carbon. It forms crystallizable salts with acids.*

Result : Color tests are available to differentiate between strychnine and brucine. Conduct the following tests and record results.

To a 0.1gm of strychnine, add 1ml of a 50:50 mixture of concentrated HNO_3 and H_2O and record result of spot test : ———

There is no appreciable action on strychnine.

Perform the same test with 0.1 gm of brucine.

Concentrated nitric acid produces with brucine or its salts an intense crimson color, which changes to yellow by heat.

Now add a few drops of the water to the brucine and add a few drops of stannous chloride.

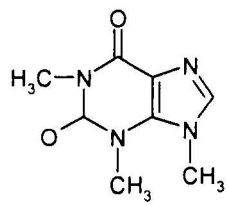
Results : ————————

The yellow liquid becomes violet upon the addition of stannous chloride or ammonium or sodium sulphide.

Experiment **45**

The Chemistry and Isolation of Caffeine from Tea

Caffeine ($C_8H_{10}N_4O_2$) is the common name for trimethylxanthine (systematic name is 1,3,7-trimethylxanthine or 3,7-dihydro-1,3,7-trimethyl-1H-purine-2,6-dione). The chemical is also known as coffeine, theine, mateine, guaranine, or methyltheobromine. The structure of caffeine is:



Caffeine

When purified, caffeine is an intensely bitter white powder. It is added to colas and other soft drinks to impart a pleasing bitter note. It is soluble in water, alcohol, acetone, chloroform, benzene, and ether. Solubility in water is increased by the presence of alkali, benzoates, cinnamates, citrates, or salicylates.

Caffeine is found in tea leaves, coffee beans, kola nuts, and cocoa beans. For the plants, caffeine acts as a natural pesticide. It paralyzes and kills insects that attempt to feed on the plants. The Table below gives the amount of caffeine in the various beverages prepared from these natural products.

One can develop both a tolerance and a dependence on caffeine. The dependence is real, and a heavy [> 5 cups of coffee per day] user will experience lethargy, headache, and perhaps nausea after about 18 hrs abstinence. An excessive intake of caffeine may lead to restlessness, irritability, insomnia, and muscular tremor. Caffeine can be toxic, but it has been estimated that to achieve a lethal dose of caffeine one would have to drink about 100 cups of coffee over a relatively short time.

Because of the central nervous system effects that caffeine causes, many persons prefer to use decaffeinated coffee. The caffeine is removed from coffeeby extracting the whole beans with

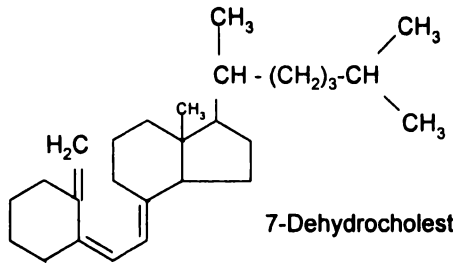
9. Fill a clean cuvet with methylene chloride. Wipe the unfrosted sides with a Kimwipe. Place the cuvette in the sample compartment in U.V spectrophotometer with the unfrosted sides facing the front and back. Close the lid.
10. Press the green button under "Collect Baseline" and wait for the collection to be completed. (The baseline will be stored in the instrument, but will not appear on the screen. "Baseline Collected" will appear when the scan is complete).
11. Remove the blank and dump the contents into a waste container. Rinse the cuvette into the waste container twice with small amounts of sample. Fill the cuvette ¾ full with sample. Return the cuvette to the sample compartment and measure the absorbance at 260nm.
12. Repeat same steps with the other standards and unknown solutions. Remember to label the printouts with the sample name.
13. Plot a Beer's law curve of absorbance (y) vs concentration (x) for the caffeine standards. From the standard curve, read out the concentration of various soft drink.

Observation Table

Caffeine standards			Soft drinks	
Concentration (ppm)	Absorbance	Brand	Concentration Absorbance (ppm)	Caffeine mg/ml
0.00				
50.00				
100.00				
150.00				
200.00				
250.00				

Calculations

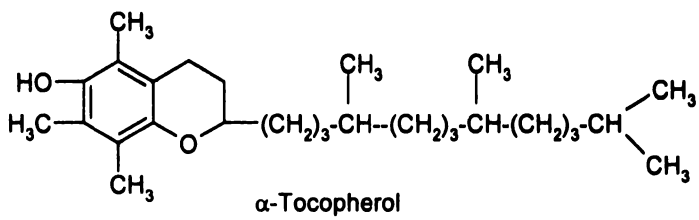
1. Using the graph, determine the concentration of caffeine in each soda in ppm.
2. Calculate the mg of caffeine in a 12 oz serving (253 ml) of soft drinks.



7-Dehydrocholesterol

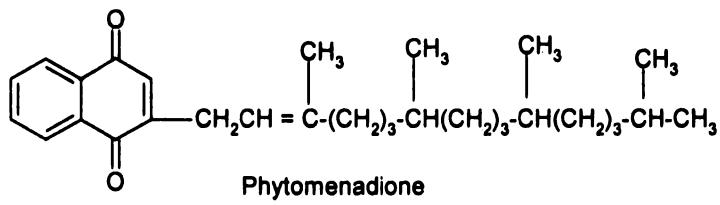
It is same as ergosterol except that the side chain in position is that of cholesterol.

The E vitamins are known chemically as tocopherols, which are designated as α, β, and γ tocopherols. Their most striking chemical features is their antioxidant property. Wheat germ oil (particularly rich in the E vitamins),milk, eggs, cereals and leafy vegetables are sources for Vitamin E. The structure of alpha tocopherol is:

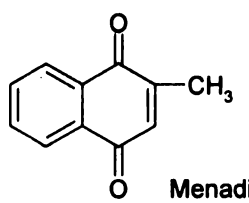


α-Tocopherol

The K vitamins are related to 2-methyl 1,4-naphthoquinone and include phytomenadione (vitamin K1) and menadione(vitamin K3). They are required for the synthesis of prothrombin in the blood, which promotes proper clotting as well as being an essential component of the phosphorylation process involved in photosynthesis. Their structures are:



Phytomenadione



Menadione

The chemical structure of vitamin C (ascorbic acid) resembles that of a monosaccharide. Ascorbic acid functions as a reducing agent in both plant and animal tissues. Its structure is:-