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## Spectroscopic and Quantitative Determination of Platinum

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SPECTROSCOPIC AND QUANTITATIVE DETERMINATION  
OF PLATINUM

This is a thesis submitted in partial fulfillment of  
the requirements for department honors in Chemistry.

Submitted by John W. Manning 3rd.

Approved by R. P. Sturgis.

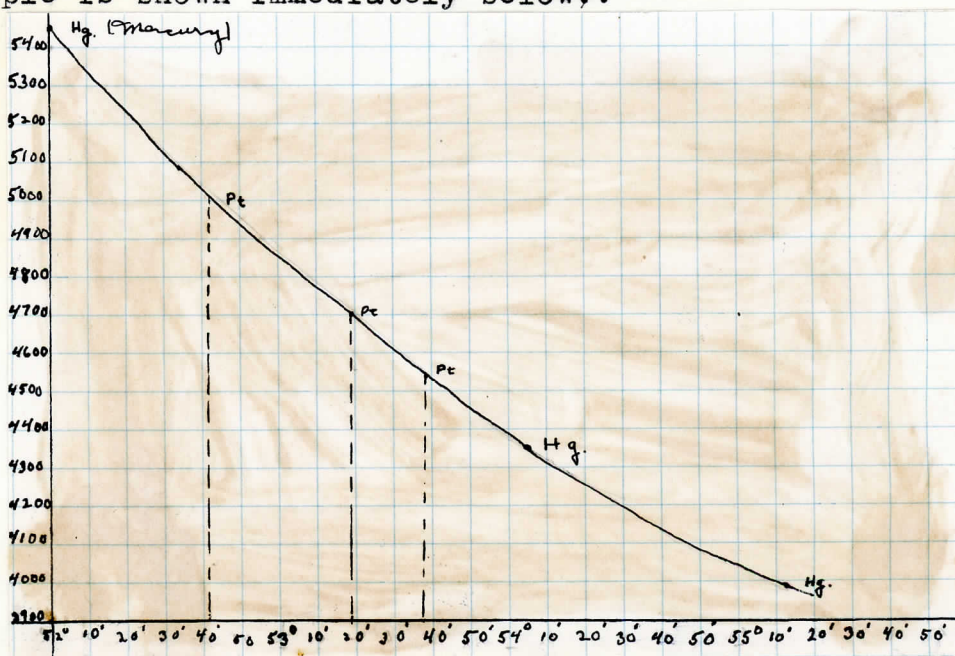


## SPECTROSCOPIC METHOD

The first method we used was the spectroscopic analysis of platinum so that we may ascertain whether it is really present or not, and thus if present by quantitative methods the exact amounts may be found.

The equipment used for this analysis consisted of: a spark coil into which we passed ten volts, two samples of our unknown which approximately weighed .05gms. were pounded into thin discs and inserted into two clamps so that a spark could jump between them, and a typical spectroscope set up so that this spark could be viewed.

Many methods may be used in the seeking of platinum lines and two methods were tried. The first using mercury as a standard and thus by finding the angle light is bent by the lines of wavelength 4046.56, 4358.34, and 5460.73, plot a graph and thus the wavelength of any other line may be found merely by it's angle of deviation from a straight beam of light. These degrees are then plotted on the graph where they intersect the mercury graph, that is their wavelength. (an example is shown immediately below).



The method we discussed above however is not entirely satisfactory because of the inaccuracy both in reading the degree of deviation and in compiling the graph, so instead of adhering strictly to this method the photographic method was also used.

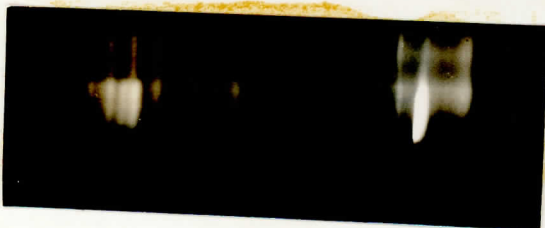
The first pictures taken were of the spectrum of the unknown metals at  $52^{\circ} 42'$ , and  $54^{\circ} 48'$ . Then at the exact same degrees two pictures were taken using platinum instead of our unknown. We then compared these pictures and found a marked similarity in the locations of the lines.

As a confirmatory test we used one electrode of platinum and one of our unknown metal. These lines because of their exact location met perfectly and thus we can conclude platinum to be present. ( photos below )

unknown



known



Crosshairs at  $52^{\circ} 42'$

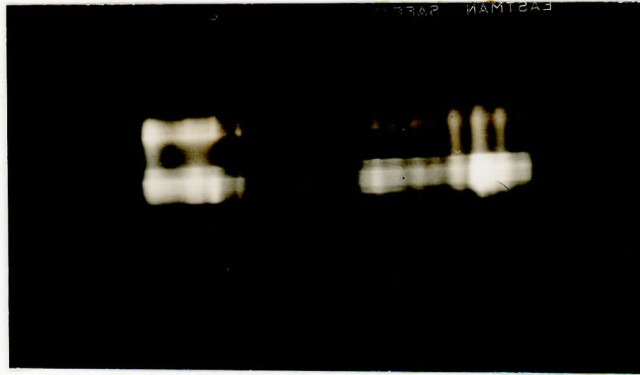
known



unknown

Crosshairs at  $54^{\circ} 48'$





Top electrode  
of platinum  
bottom of  
unknown.

In the photographic work Tri X Panchromatic film was used with a twenty minute exposure and a four to five minute developing period.

#### QUANTITATIVE METHOD

We first used the method set forth by R. Gilchrist,<sup>I</sup> however it was modified according to our needs. The procedure was then the following. The unknown which was obtained as pellets was washed in dilute hydrochloric, pounded into thin discs, dried and weighed. They were then placed into aqua regia and placed on a steam bath. With the exception of silver, which separates as the insoluble chloride, and of iridium, which separates as metal, all the constituents of the unknown pass into solution. The silver chloride was broken up from time to time to insure complete disintegration.

When decomposition of the unknown was complete the solution was diluted to a volume of 150 to 200cc. and allowed to stand on the steam bath for about one hour and then set aside to cool. Filter, wash and weigh the residue as silver chloride and if iridium is possibly present dissolve the precipitate in hot diluted ammonium hydroxide, filter off any residue (iridium), then reprecipitate silver

by making the filtrate acid with nitric acid, filter, dry and weigh residue as silver chloride.

The original filtrate obtained from the decomposition of our sample was neutralized with sodium hydroxide to the red-orange end point of thymol blue (about 1.5). Heat the solution to boiling and add to it 10ml. of a filtered 10 percent solution of sodium nitrite. Boil the solution for 2 or 3 minutes to coagulate the precipitated gold. Again neutralize to the red-orange end point of thymol blue.

Add 20ml. of the nitrite reagent and then neutralize the hot constantly stirred solution by adding dropwise a diluted solution of sodium hydroxide until the alkalinity reaches that which corresponds to a faint blue color of thymol blue. Boil the solution for five minutes, again taking care that the solution does not bump. Under these conditions gold, indium, copper, zinc, and nickel will be quantitatively precipitated.

The filtrate from the above procedure will contain any platinum which may be present. In order to precipitate this platinum the filtrate was boiled down with hydrochloric acid, the acidity regulated to .3N and hydrogen sulfide passed into this solution. The platinum will come down as the sulfide.

With the unknown we used however there was such a small amount that it could not really be considered as conclusive proof of its presence.

The method outlined by Scott<sup>3</sup> was a little more simplified however no more accurate. It proceeds as follows. The samples were weighed dissolved in dilute nitric acid and the residue filtered off. The residue will contain platinum and gold if present.

The residue was then taken dissolved in aqua regia, and finally the nitric acid was removed by evaporation and additions of small amounts of hydrochloric acid. Then it was further diluted with water and filtered to remove any silver chloride which may be present.

We then passed sulfur dioxide through the filtrate to reduce any gold or palladium which may be present. We then filtered it again and then added 15cc. of sulfuric acid to the filtrate, evaporated by heating just to the point of heavy fuming and then cooled. This solution was then diluted to 200cc. with water and boiled for several minutes. The solution was then filtered, removing all of the precipitated gold and lead sulphate, however this was not necessary with our samples because of the absence of these metal.

The solution was then heated and hydrogen sulfide passed into it and this should precipitate the sulfides of platinum and palladium. The unknown analyzed obviously contained only traces of platinum, because we obtained only a very slight precipitate of dubious nature which showed platinum to be present in amounts of less than one percent.



## SUMMARY

In the photographic work which we have included in this paper the proofs were presented, because of their more visible nature, however the negatives are vastly superior in being much more detailed and confirmatory. Also by not including the negatives they may be kept in a less perishable state.

It may also be of interest to note that our unknown sample was obtained from deposits in Montgomery County by an Ursinus alumnus. These samples which we received from him contained about 80% silver.



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