

A Study of the Incandescent Gas Mantle;
also
A Scheme for the Separation of Thorium,
Zirconium and Cerium.

Submitted as a Thesis for the Degree of Bachelor
of Science in Chemical Engineering.

E. S. Freed.

University of Tennessee.

May 1912.

The Mantle

The base of the incandescent gas mantle is a knit fabric. This fabric in the past has been made of cotton fibre, but the cotton is now being replaced by artificial silk. The knitting or weaving varies in the different mantles; for example, there are single weave, double weave, light weave and heavy weave mantles.

The cotton which goes into the mantle must contain a very small percentage of mineral residue, otherwise the impurities introduced might seriously effect the efficiency of the mantle. Ordinary cotton contains about .5 percent of mineral residue. The cotton before it can go into the mantle must first be passed through a suitable solvent which removes much of the mineral constituents.

The fibre is woven into long cylindrical tubes and these are cut into uniform lengths. One end of these lengths or pieces is tied with an asbestos cord in such a manner that the mantle ^{may} easily be supported. The mantle is then saturated with the lighting fluid and dried. After drying the company's name is branded on the mantle. This is done by means of a rubber stamp which conveys a solution of Didymium Nitrate to the mantle. Didymium Nitrate when heated is changed to the oxide and emits a yellow light while the other portions of the mantle emit a more nearly white light.

After the mantle has been shaped and branded it is fixed on a support and burned. The cotton fabric burns out leaving only the ash of the lighting fluid. In the fact that this ash should have cohesive power enough to hold together and retain the original shape of the fibre, lies the greatness of the discovery which led to the invention

of the incandescent mantle. When Dr. Auer, the inventor of the incandescent mantle, explained his idea to Professor Bunsen, he replied, "It is extremely doubtful if the ash can be made to hold together." His opinion was based upon his knowledge of the metallic oxides in general.

The mantle after the cotton has been burned out is in a soft and flabby condition. It is then intensely heated and the ash is semi fused after which it remains in a hard and resilient state.

The mantle is then dipped into a solution of soluble cotton. This gives it a coating which protects it from injury during the process of packing and shipping and when the mantle is ready for use it is readily burned off.

The Lighting Fluid.

The lighting fluid is obtained from a mineral known as Monazite Sand. Monazite is found in its original formation in minute quantities in rocks of the granite class and on account of its isolated occurrence it was named "Monazite" which means "to be solitary". The commercial sources of Monazite are secondary, resulting from the decomposition of the granite rocks and the concentration of the heavy granular particles by the action of the wind and water.

Monazite is a heavy mineral and occurs in various colors. The following is atypical analysis of a sample of Monazite.

Phosphoric Anhydride-----	28%
Cerium Oxide-----	30%
Lanthanum Oxide-----	14%
Didymium Oxide-----	16%
Thorium Oxide-----	5%
Yttrium Oxide-----	2%
Iron Oxide, Calcium Oxide-----	5%

The commercial source of Monazite Sand is from the low creek beds of North and South Carolina and from the sandy shores of Brazil. Most of the Brazilian Monazite supplies the European market while the United States utilizes its own product.

The sand is shipped to the mantle factory where it requires a treatment for many months to recover the thorium present in the Monazite in the form of a nitrate. The lighting fluid is a mixture of thorium and cerium nitrates. The standard formula is 99% thorium and 1% cerium. However it varies from one half to two percent.

As an experimental study of the incandescent gas mantle, the properties of twelve different mantles were observed. First the candle power of the mantle was determined, then its physical strength and finally its chemical constituents.

The candle power of the mantle was determined in the usual way; that is, by comparing the square of the distances from two sources of light, one of which the candle power is known and the other is the light to be examined, at which a grease spot on a piece of paper appears as strongly illuminated on one side as the other.

The light from the ignited mantle was compared with that of a standardized, 60 watt, tungsten lamp emitting 36 candle power of light at 108 volts. Since the candle power of an electric lamp changes with the voltage it is necessary that the voltage be kept constant. To maintain a constant voltage, the current was taken from a storage battery and as a further protection against a change in voltage, ^{a voltmeter} was always in the circuit, so that a slight change in voltage could easily be detected and corrected by means of a suitable resistance. In comparing the gas light with the electric light it was

found that the grease spot in the photometer appeared green on one side and yellow on the other. At first, accurate results were not obtained, because it was difficult to determine the exact point at which the two different colors mentioned above were illuminated to the same intensity. However after considerable practise this difficulty was overcome and good results were obtained.

In estimating the physical strengths, only the relative strengths were determined. This was done by means of a bumping machine in the form of an electric buzzer. The support or gas fixture on which the mantle was supported was vibrated by small blows delivered by the buzzer until the mantle had broken down. Some of the mantles, as can be seen from a following table, were shaken down in a very short time while others lasted much longer time, indicating a marked difference in the strength of the mantles.

Scheme for the Separation of Thorium, Cerium & Zirconium

From Fresenius' Qualitative Analysis the behavior of thorium cerium and zirconium oxalates was noted.

- 1st. Zirconium oxalate is soluble in an excess of ammonium oxalate even in a cold solution.
- 2nd. Cerium oxalate is slightly soluble in an excess of boiling ammonium oxalate but reprecipitates on cooling.
- 3rd. Thorium oxalate is not soluble in ammonium oxalate except in a boiling solution and is not reprecipitated on cooling.

It was found, however, that thorium oxalate is soluble not only in excess of boiling ammonium oxalate but in warm ammonium oxalate and if the excess be great it is soluble in the cold solution. It was also found that if only four times as much cold ammonium oxalate be used as is necessary to precipitate the thorium and the zirconium, the zirconium oxalate will go into solution while the thorium oxalate will remain as a precipitate.

The Separation.

To the cold solution of these elements add three or four times the amount of cold ammonium oxalate necessary to precipitate them. Let stand several hours. Decant through a filter, wash and decant again. Then add water to the precipitate and boil. This converts the fine oxalates, which would easily go through the filter, into a coarse granular precipitate. Since most of the excess ammonium oxalate has been removed there is little danger of any of the thorium oxalate going into solution when it is boiled. After cooling filter and wash. Combine the filtrate with the liquid which had been previously decanted. In this solution is found the zirconium. On the filter remains the oxalates of the thorium and cerium.

To the solution containing the zirconium add ammonia until precipitation is complete. Filter, burn and weigh.

Remove the thorium and cerium oxalates from the filter and place in a beaker. Add an excess of ammonium oxalate and boil. The thorium now goes into solution. Let stand until cold. Then filter and wash. The filtrate contains the thorium. The cerium remains on the filter.

To the solution containing the thorium add ammonia until

precipitation is complete. Filter burn and weigh.

The cerium oxalate remaining on the filter is burned and weighed as an oxide.

To check the accuracy of the separation, .5654 grams of thorium nitrate, .2664 grams of zirconium nitrate and .6386 grams of cerium nitrate were weighed out. These nitrates were previously subjected to analysis and were found to contain the following percent of the oxide.

Thorium nitrate----48.04%

Zirconium nitrate--44.60%

Cerium nitrate----42.00%

The method employed was that of burning the nitrates to a constant weight.

Then by calculation, .2716 grams of thorium oxide, .1188 grams of zirconium oxide and .2682 grams of cerium oxide had been weighed out. By analysis, using the above scheme, .2508 grams of thorium oxide, .1160 grams of zirconium oxide and .2470 grams of cerium oxide were found.

The Analysis of the Gas Mantle..

Mix about a half a gram of the mantle ash with five grams of potassium bi-sulphate in a platinum dish. At first heat gently to avoid splattering and then heat strongly until thoroughly fused. After the mass has cooled dissolve it in~~x~~ the beaker with warm water and a little hydrochloric acid. Then proceed as in the separation of thorium, zirconium and cerium given above.

~~#~~ Note-- Much of the information regarding Monazite Sand and the manufacture of the gas mantle was obtained from bulletins issued by the Welsbach Company.

Table I. Showing Results of Candle Power Determinations.

Number	Candle Power	Minimum Cu. Ft. Gas per Hour Cor- rected for Press. and Temp. *	Candle Power Corrected to 15 Cu. Ft. of Gas per Hour.
1.	90.0	3.58	125.7
2.	87.5	5.18	84.4
3.	92.2	4.36	107.2
4.	59.0	3.04	97.0
5.	63.0	4.07	77.4
6.	92.5	4.43	104.4
7.	52.6	3.53	74.4
8.	43.2	2.69	80.3
9.	48.6	2.83	85.8
10.	68.4	2.94	116.2
11.	61.2	3.60	85.0
12.	51.5	2.91	88.4

* The volume of the gas is corrected to a pressure of 30 inches of mercury and to a temperature of 60° F.

Table II. Showing the Relative Resistance of the Mantles
to Vibration.

Number.	Time during which the mantle lasted while subjected to vibration.
1.	12 minutes
2..	35 "
3.	3 "
4.	30 "
5.	2 "
6.	28 "
7.	150 seconds
8.	80 "
9.	15 "
10.	210 "
11.	10 "
12.	One hour (unbroken)

Note. The first six mantles mentioned above are of the upright type while the others are of the inverted type.

Table III. The Analysis of the Mantle.

Number.	Percent of Cerium Oxide	Percent of Thorium and Zirconium Oxides.
1.	1.75	97.40
2.	1.70	97.33
3.	1.61	98.02
4.	1.01	97.51
5.	1.98	96.39
6.	2.03	97.46
7.	.76	98.27
8.	1.31	96.88
9.	.85	97.10
10.	1.12	96.47
11.	1.12	97.84
12.	.97	98.12