# DETERMINATING THE WATER OF CRYSTALLIZATION



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Aspect	Design	DCP	CE
1			
2			
3			
Total			

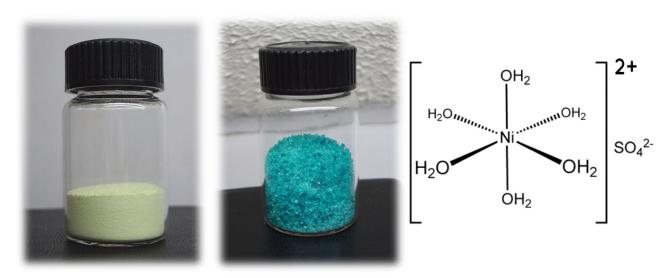
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## Introduction

There are some salts in the world, which have water in their crystalline structure, and most of them are in the nature around us. Scientists customarily know these salts as hydrated salts. Our aim is to find out how much or the number of moles of water in basic crystallization. One of these hydrated salts is called Nickel (II) Sulphate, and it is mainly used in electroplating of nickel. The chemical formula of the salt is:

It can be seen or found it two types of forms, one is anhydrous and the other is hexahydrate. This is they look like along with the chemical structure of it:



Picture 1: Anhydrous Nickel (II) sulphate

Picture 2: Hexahydrate Nickel (II) sulphate

Picture 3: Structure of Nickel (II) Sulphate

The basic water in the substance is known as water of crystallization. Our experiment focuses on how to determine how much of it is there, and by doing that we need to create an endothermic reaction. If the crystal is heated, an endothermic reaction is made:

$$NiSO_4 \cdot xH_2O \rightarrow NiSO_4 + 6H_2O$$

When it is heated, it starts to break the bonds within itself. When there is enough heat to do that, water vapor is formed from the water of crystallization. For the salt it creates an anhydrous form, when the water is removed. The number of moles of water existing in one mole of crystalline structure can be written as the formula of the hydrated salt. After we have heated the Nickel (II) Sulphate, we can easily determine how much moles of water weighing lost before and after heating several times. Then the x number of water can be calculated.

## **Research Question**

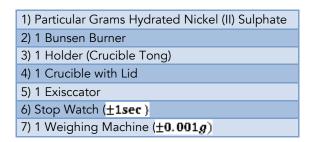
#### What is the amount of water of crystallization in hydrous Nickel (II) Sulphate?

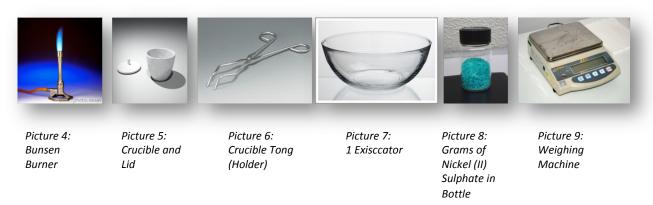
There is obvious loss of water at the starts, but then, the amount of water of crystallization is constant so after several heatings the mass of solid will not be changed.

Table 1 — List of Variables

Independent Variable	Number of Moles of Salt	
Dependent Variable	Mass of Evaporated Water	
Controllable Variable	Temperature, Time, Initial Mass of Salt	

#### <u>Table 2 — List of Materials</u>





## Risk Assesment

The procedure requires usage of fire, being careful will all method, wearing safety goggles, apron, and medical gloves is very important. No use of other material other than involved in experiment is also important.

## Method

- 1. Set up the materials needed for the experiment.
- 2. Weighed an empty crucible with its lids and without its lid and notes down the results accurately.
- 3. Filled the crucible with Nickel (II) Sulphate of about just less than half, and then weighed it with and without the lid.
- 4. Heated the crucible in top of the burner for 10 minutes, and let it cool down for 7 minutes in the bowl
- 5. Then when it is cool, weighed it with the weighing machine.
- 6. Heated again for 10 minutes, and let it to cool again for 7 minutes and again weighed it.
- 7. Recorded the results and the data throughout the experiment
- 8. Did Step 6 or Step 4, for until the mass was stable and there was no loss, about 9 times.

## **Data Collection**

#### **Qualitative Data**

The first colors of the salt and the crystal were observes are dark green/organgish but mainly green. When heated it start to change color and a bit of substance. After heating each time it changed and becoming granular and lighter in the shades of green. After the first few heating's, we saw that the sides or the circular corners were becoming lighter and whiter. Then later, the whole substance started to turn sandy and fine-grained.





#### **Quantitative Data**

Mass of Empty Crucible and Lid (in grams {g})

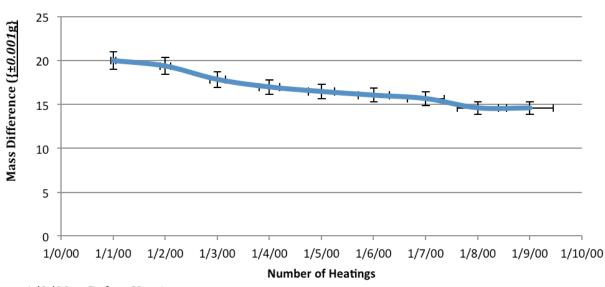
48.366 (±0.001)

Table 3 — Mass of Salt after Successive Heatings {g}

Crucible, Lid and Hydrated Salt:	Mass [ g <u>±</u> 0.001 ]
Before Heating:	68.325
After 1 <sup>st</sup> Heating:	67.697
After 2 <sup>nd</sup> Heating:	66.176
After 3 <sup>rd</sup> Heating:	65.314
After 4 <sup>th</sup> Heating:	64.800
After 5 <sup>th</sup> Heating:	64.382
After 6 <sup>th</sup> Heating:	63.991
After 7 <sup>th</sup> Heating:	62.940
After 8 <sup>th</sup> Heating:	62.930
After 9 <sup>th</sup> Heating:	62.930

Basically, heated it 9 times, until it reaches stability or very small or no change in the temperature. The results records have an uncertainty of 0.001 grams. All the units of measurement are in grams.

Graph 1 — Changes in the Mass of Salt after Successive Heatings  $\{\pm 0.001g\}$ 



1/0/00 = Before Heating

1/100 - After 1st Heating and so on...

#### **Mass of Hydrated Salt**

$$m_{hydrated} = m_{initital} - m_{crucible} = 68.325 - 48.366 = 19.959 g$$

uncertainty =  $\Delta m = 0.001 + 0.001 = 0.002 g$ 

Result:  $19.959 \pm 0.002 \,\mathrm{g}$ 

#### Mass of Anhydrous Salt

$$m_{NISO_{4}} = m_{final} - m_{crucible} = 62.930 - 48.366 = 14.024$$

uncertainty =  $\Delta m = 0.001 + 0.001 = 0.002 g$ 

Result:  $14.024 \pm 0.002 \,\mathrm{g}$ 

#### **Number of Moles of Anhydrous Salt**

$$n = \frac{m}{M} = \frac{14.024}{154.77} = 0.0906 \text{ moles}$$
uncertainty =  $\Delta n = \left(\frac{\Delta m}{m} + \frac{\Delta M}{M}\right) \times n = \frac{0.002}{14.024} + \frac{0.01}{154.77} \times 0.0906$ 
= 0.000148

Result:  $0.0906 \pm 0.0001$  mul

#### Mass of Water Evaporated / Lost

$$m_{H_2O} = m_{initital} - m_{final} = 68.325 - 62.930 = 5.395 g$$
  
uncertainty =  $\Delta m = 0.001 + 0.001 = 0.002 g$ 

Result:  $5.395 \pm 0.002$  g

#### **Number of Moles of Lost/Evaporated Water**

$$n = \frac{m}{M} = \frac{5.395}{18.00} = 0.2997 \text{ moles}$$

uncertainty = 
$$\Delta n = \left(\frac{\Delta m}{m} + \frac{\Delta M}{M}\right) \times n = \frac{0.002}{5.395} + \frac{0.01}{18.00} \times 0.2997$$
  
= 0.000537

X = 0.2997/0.0906 = 3.30

uncertainty = 
$$\Delta x = \left(\frac{\Delta n1}{n2} + \frac{\Delta n1}{n2}\right) \times X = \frac{0.000148}{0.0906} + \frac{0.000537}{0.2997} \times 3.30$$
  
= 0.007546

**Result: 3.30**  $\pm$  0.01 mol

#### **Overall Percentage Uncertainty**

% Uncertainty hydrated salt = 
$$\frac{0.002}{19.959}$$
 x 100% = 0.01%.

% Uncertainty anhydrous salt = 
$$\frac{0.092}{14.024}~x~100\% = 0.014\%$$

% Uncertainty Mass of Water = 
$$\frac{0.002}{5.395}$$
  $x$   $100\% = 0.037$  %

% Uncertainty Time = 
$$\frac{5}{1090}x \ 100\% = 0.49 \%$$

Mass of Anhydrous Salt (g)	14.024 ±0.002 g	
Number of Moles of Anhydrous salt (mol)	0.0906 $\pm$ 0.0001 mol	
Mass of Water loss (g)	$5.395 \pm 0.002g$	
Number of Moles of Lost water (mol)	<b>0.2997</b> ± 0.0005 <i>mol</i>	
Final Formula	NISO <sub>4</sub> · 3H <sub>2</sub> O	
Actual Formula	NiSO <sub>4</sub> · 6H <sub>2</sub> O	

Error: (theoretical number – actual number) / theoretical number x  $100\% = (6-3)/6 \times 100\% = 50\%$ 

### Conclusion and Evaluation

From all the calculations from the previous pages, the formula which we determined of the salt was  $NiSO_{4\times}3H_{2}O$  but originally, in fact it was  $NiSO_{4\times}6H_{2}O$ . The percentage error was about 50%, so it was clear that the result would not be exact. The real number of moles of water does not lie in the uncertainty range as well. The result is also not precise, because overall percentage uncertainties were high, especially then time uncertainty. We can also make a conclusion that the results and experiment was fairly practical.

The water within the crystal structure was not as found as it was after our calculations. Basically the result of the number of moles, mass were eventually correct, but overall result, the amount of moles per 1 mole of salt, was not precise being half than it should be was due to percentage error.

#### Evaluation

Every single step was done in such a way, that it limits all the negative events that could take place affecting the results of the experiment. Everything was carefully looked at, and precision was what it required. Most steps were taken in order to minimize the negative effects of external factors, such as placing the lid on the crucible, or measuring the same time before each weighing, more procedures should be undertaken to make the result more accurate. The overall accuracy could have been increased if things like, measuring the exact split second of the time, and amount of fire from Bunsen burner were looked at closely.

There were areas in which involved the systematic errors, and some random error. Both of these are always there in each experiment you conduct, and especially in experiments involved with Chemistry.

Type of Error	What is the error?	Improvement
Systematic Error	Measuring the weight after the time when we are able to move the crucible, because it is not very precise, same amount of time should be waited to avoid differences caused by temperature.	Having more than one person to handle the time process and not only using stopwatch, and carefully measuring the time.
Systematic Error	The crystals should be powered before putting into crucible, so water would be evaporated more evenly, and results would be closer to the original ones.	Powdering the crystal with a mixer or masher before using it in the experiment. So, a mortar could be used.
Systematic Error	We could have easily mixed the salt whenever we decided to do before doing the experiment, because then the water would evaporate from whole substance and not closet to the surface.	Mixing the salt while on the Burner and as well as on the weighed and in exicattor.
Systematic Error	A different method could be put into the concept, however the method was taken by looking at the materials available only.	Different method and different equipment could be used. It could bring different results.
Systematic Error	The weighing scale uncertainty and overall using more sophisticated technology can lower weighing scale.	Higher level of technology in weighing mass scale, and a one with more significant figures.
Systematic Error	Bigger amounts of substances can be used in order to lower the percentage error, and more amounts and not in a crucible lid could be a bigger bowl.	Take larger amount of surface area of the crucible a huge one, and take more amounts of the salt
Random Error	The temperature in the room throughout the day would have not been exactly the same no that could make the difference.	Keep measuring the room temperature with a thermometer.
Random Error	Temperature of the Bunsen Burner burning would have not been the same through the day as well, fluctuating averages.	Keep measuring the burner temperature with sophisticated technology, keeping it stable.
Systematic Error	The whole experiment should be conducted at least 5 times, so the results could be more reliable and precise.	Repeating the experiment as many time as possible according to time.

We can conclude that there are more systematic errors rather than random errors. That is why there is a high percentage error. There is a difference between percentage error and percentage uncertainty. Percentage error measures by how much the experiment by out by, in this case 50%. Percentage uncertainty, on the other hand, measures, the amount of errors in calculating or measuring. Percentage uncertainty would be used for testing a hypothesis and seeing if your results support it within a range. Percentage error is used for something you know the value of, for example when testing a piece of equipment's accuracy.

#### Crystalline Structure and Thermogravimetry

A new generation: A new thought. Sophisticated and advanced technology would have been the key to the lock. If we had access to more intense technology we would have been in profit. Thermogravimetry would have been used, it we were in an official doctor's science lab. It is a very special technique, which allows us to measure the mass and the temperature with a time change, an with an absolutely exact result, so the most annoying and problematic errors in our measurements, which would be temperature differences while taking it from one place to another would have been deleted. If we had thermogravimetry, other factors could have been controlled as well, things like pressure, which were not needed, and unknown throughout the experiment. With those precise measurements we could have made sophisticated graphs involved curves and algebra. We would have easily made more calculations, and more drawing would be conducted. It is basically an analysis theory that would make life easier for the overall experiment.



Picture 11: Thermogravimetry

different There are structures to substance in the world. There are different structures to this acid as well. This means that the different structures do have a effect on the amount of water evaporated. If we think closely about it, if there is some water stuck inside the structure, and there is some outside of it, it is obvious that the water outside would evaporate more. So, this means that not all water would be evaporated equally which would give un even results, which means it has effected the amount of water evaporated. On the other hand, if there is a lot of water inside the structure, it means that there would be less amount of water evaporated. So, different types of structures have different effects on the amount of water evaporated. The Nickel Sulphate was on average, not a lot outside, not a lot inside, so basically it was even, and it did not have a large effect on the result.

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#### Pictures:

Picture 1:

http://upload.wikimedia.org/wikipedia/commons/b/b4/Nickel\_sulfate\_anh.jpg

Picture 2:

http://upload.wikimedia.org/wikipedia/commons/0/08/Nickel\_sulfate\_hydrate.jpg

Picture 3:

http://upload.wikimedia.org/wikipedia/commons/f/f8/N9%28H2O%296S O4.png

Picture 4:

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Picture 5:

http://preview.turbosquid.com/Preview/2010/12/08\_\_18\_00\_00/crucible-c01.jpg7c87ad9c-cdf0-4c13-8b65-3fa6a2c26687Larger.jpg

Picture 6:

http://spellboundlabs.co.za/images/crucibletongs.jpg

Picture 7:

http://www.le-tom.com/img/duralex-bowl-medium.jpg

Picture 8:

http://upload.wikimedia.org/wikipedia/commons/0/08/Nickel\_sulfate\_hydrate.jpg

Picture 9:

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Picture 10:

http://i36.photobucket.com/albums/e41/truffula/Webmaze%20Pics/countdownends1.jpg

Picture 11:

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