Video « The Kjeldahl method – Demonstration »

Time	Text
00 :09	The first step is to prepare the sample. It requires to use concentrated sulphuric acid, so work under a hood, wear the strong acid resistant gloves and mind the safety glasses.
00 :30	You will have to weigh your sample. If the sample is liquid, you can directly weigh it in the beaker. But if the sample is solid, you have to crush it first, with the adapted grinder according to the type of sample. Then, you can put your sample into the mineralisation tube which will be used for the second step.
00 :53	In order to transform the protein, you will have to add a Kjeldahl catalyst during this preparation step. This catalyst allows to accelerate the mineralisation reaction.
01 :06	To avoid any foam formation during the mineralisation reaction, mind to add an anti-foam pill.
01 :18	Finally, you can add very carefully 10 mL of concentrated sulphuric acid in the Kjeldahl tube.
01 :48	Then, you can put your tube in the mineralisation rack and bring your sample to the mineralizing device.
02 :02	After the preparation of your sample, you put it in the mineralizing device. Mind to check if there are as much samples as free spaces in the block. Then, you can put down the smoke collector on your tubes and you check that the system is well sealed in order to collect all the smoke which will be formed during the mineralisation reaction.
02 :27	You can turn on the smoke collector and open the water valve.
02 :33	Everything is now ready for the mineralisation. The mineralizing device can be turned on. First, let's set the temperature around 150 °C in order to progressively increase the temperature. After 10 minutes, you can rise the temperature to 250 °C. Again, you have to wait for 10 minutes and then, finally, you can set the mineralisation temperature, which is 350 °C, and leave it for 5 hours.
03 :07	Mind to regularly check if nothing goes wrong during the mineralisation.
03 :11	After 5 hours, a ring will inform you that the mineralisation is over. You can shut down the system at this moment, but leave the smoke collector and the water on, in order to let the system cooling down for 30 minutes/1 hour.
03 :33	Once the system is cooled down, you can stop the water. Take off the mineralisation rack and take back your sample.
03 :52	Now that the sample is mineralized, the protein nitrogen is now transformed into mineral nitrogen. You can get this nitrogen as ammonium sulphate. The aim of this distillation step is to transform the nitrogen from NH_4^+ to NH_3.
04 :12	For that, you can put your sample into the distiller by checking if the system is well sealed, so be careful at the top of the tube.
04 :24	Then, the sodium hydroxide will be injected into the sample. This NaOH will transform NH_4^+ into NH_3. The ammoniac vapours will be condensed and the ammonia will be trapped into this Erlenmeyer filled with 25 mL of 0.1 mol/L hydrochloric acid with methyl red, which will be useful for the titration, and 10 mL of water, just to be sure that this tube is well immersed in the hydrochloric acid solution to avoid any loss of the ammonia vapours.
05 :02	Before starting the distillation, it is important to check if there is enough sodium hydroxide ready to be injected into the system. On the side of the device, you can check if the level in the tank is sufficiently high. Also, to ensure a good condensation in the condenser, you need to open the water valve. The water will flow in the condenser when you will start the distiller.
05 :27	Then, you can shut the door of the device and turn it on.
05 :36	To inject the 50 mL of sodium hydroxide necessary for the distillation, you have to push the NaOH button, and there will be exactly 50 mL injected in the system.
05 :53	To start the distillation, you have to push the steam button which will start the heating.

Video « The Kjeldahl method – Demonstration »

06 :13	At the ring, the distillation stops and you can open the door. Let the system cooling down before taking off the sample.
06 :26	So, now your ammonia is trapped as NH_4^+ and you have to titrate the excess of hydrochloric acid which did not trap the ammonia. To do that, you will perform a titration with 0.1 mol/L sodium hydroxide.
06 :45	Once the system is cooled down, you can take the Erlenmeyer back and, as I explained earlier, you can start the titration of the excess of HCl. Concerning the mineralisation tube, once it is cold, you can throw its content into the base tank.
07 :06	Now the titration of the excess of HCl with 0.1 mol/L sodium hydroxide which has been previously put in the burette can begin. The NaOH is added drop by drop.
07 :33	Here you can observe the switch in your indicator, which is the methyl red. It means that all the HCl has been neutralised by the NaOH and so, only water and NaCl are left.
07 :46	With this titration, you are able to know the quantity of excess HCl which did not trap the ammonia. Moreover, you have done a blank sample without any product, which you have also put in the distiller, and which gives you by titration the total quantity of HCl. Therefore, by making the difference between the blank and the sample, you will get the quantity of HCl which has reacted and is directly proportional to the quantity of trapped ammonia, so to the quantity of nitrogen in your sample.