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# Method for low-cost synthesis of nitric acid by oxidation of ammonia derived from its compounds

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## Abstract
**Objective:** Nitic acid is one of the essential acids in a chemical laboratory, but it is also one of the more difficult to obtain for small scale chemical research. It is therefore of importance to look into low-cost synthesis of it. This paper describes a detailed procedure for synthesizing nitic acid from more common and easy to obtain compounds. Due to the difficulty of purchasing nitrate compounds, they will be avoided in the described process and substituted with more common ammonium compounds.
**Methods:** The process in question is the Ostwald process, usually used for large scale acid production. It is modified and therefore a customized apparatus has been developed to convert abundant and easy to get ammonium compounds into the desired nitric acid.
**Results:** The experimental analysis shows that although the developed setup manages to synthesize the nitic acid, the final efficiency will be determined by ammonia-air mixture and the gas flow rate.
**Conclusion:** Despite its solubility in water it is very difficult to collect and it escapes the apparatus even after being bubbled in water several times. A possible solution for this "smoke" would be a fine quartz wool air filter that could be used to collect the ammonium nitrate, but its generation should be avoided because of the inefficiency it brings. The catalyst and apparatus used in this paper allow for the Ostwald process to be used for small scale nitric acid production offer better control over its production speed than the Birkeland-Eyde process.

**Keywords:** Synthesis, nitric acid, oxidation

## Introduction
Nitric acid is one of three essential acids in a chemistry laboratory, but it is also one of the most difficult to obtain. Hydrochloric and sulfuric acid can be found in most places and need little to no purification and concentration to be used for basic reactions [1]. On the other hand, nitric acid and its subsequent compounds are much harder to find and usually undergo regulations in shipping and purchase. This makes the usual means of synthesis of nitric acid from its salts difficult and financially unviable for small scale acid production [2].
As this is an essential acid, an alternative method of nitric acid production is crucial to promote the application of chemistry in places where it would otherwise be impossible to obtain. The process in question is the Ostwald process, usually used for large scale acid production [3]. It can be modified and with the use of simple customized apparatus may convert abundant and easy to get ammonium compounds into the desired nitric acid. The preparation of the apparatus might pose a challenge and be more expensive than nitrate salt method, but long-term use will make the initial investment pay off.

## Materials and Methods
The materials used in this experiment are the following:
- Heat resistant borosilicate glass tubing for gas
- Fused quartz tube serving as reactor tube
- Quartz wool (9 μ m strands) for the catalyst structure
- Sodium silicate as high temperature glue for glass to glass joints
- Propane burner for reaction tube heating
- Adjustable air flow pump

## Chemicals
- 23% hydrochloric acid
- Sodium hydroxide (10% water content)

- Ammonium chloride
- Platinum metal (99,9%, <50 mg)

**Making appropriate catalyst**

The heart of the process of converting ammonia to nitric acid is the oxidation of ammonia. The efficiency of the process and the final quality of the produced acid relies on even combustion of ammonia in oxygen. To ensure this a catalyst is needed, but it must be made to the specifications required in this process.

For optimal results a platinum catalyst is used, however a simple wire or powder coated granules are not sufficient for this process. The catalyst must have a high surface area, but also must have the maximum contact or "flow through" area with the reactant gas mixture. This is achieved by having the catalyst in the form of a platinum covered quartz wool.

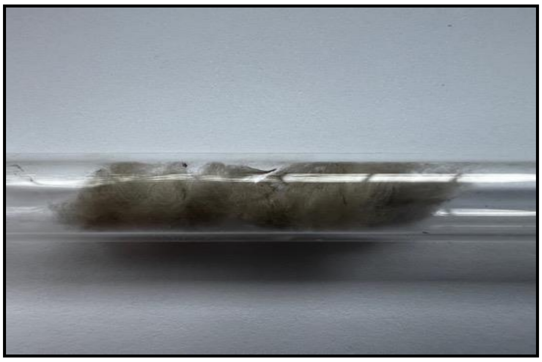

**Fig 1:** Platinum covered quartz wool.

The density of the wool provides resistance with the incoming mixture, giving it a greater chance of reacting upon contact with platinum particles.

A catalyst such as the one described cannot be found and must be prepared.

The catalyst is made by coating the quartz wool in a platinum compound that can be decomposed upon heating in order to leave a thin platinum particle film (Figure 1). The compound used for this is chloroplatinic acid which is obtained by a specialized apparatus.

Due to the main assumption of the paper that nitric acid cannot be obtained, it cannot be used to dissolve platinum.

A small quantity of platinum is dissolved by placing it in HCl and bubbling chlorine gas through it. Specialized setup is used to ensure safety and efficiency of the process.

**Setup for generating chloroplatinic acid**

In the next picture the setup for the production of the chloroplatinic acid is depicted (Figure 2).

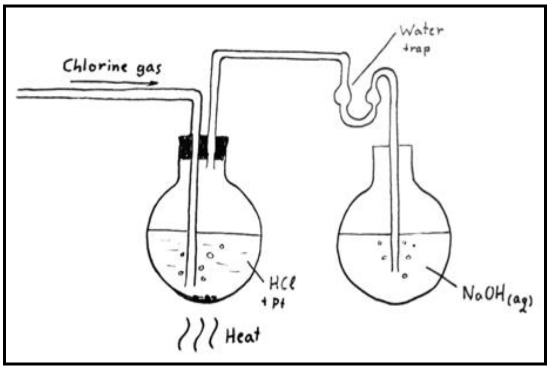


**Fig 2:** Experimental setup.

A flask is filled with a small amount of concentrated hydrochloric acid and a small amount of platinum powder is added. A adapter is added on the flask which allows the addition of chlorine gas into the solution inside. The flask is heated and chlorine is added. The chlorine rich atmosphere should dissolve the platinum in a matter of minutes. The escaping chlorine and HCl vapors are passed through a bubbler in a concentrated NaOH solution. A water trap must be used to prevent the NaOH solution from being sucked back in the platinum containing flask as it can destroy the product.

**Depositing platinum on quartz wool**

The obtained chloroplatinic acid solution is left to sit for several hours to a day to air out the majority of the dissolved chlorine. After that a piece of quartz wool is covered in the solution and dried at low heat. The dried quartz wool is put in a flame and heated to red hot. The chloroplatinic acid on the strands of the wool will decompose and cover them in fine platinum particles. The wool is left in the flame for some time in order for the platinum to bind to it properly. This can be observed through looking at the color of the wool. Wool that was heated for some time will have a light silvery color opposed to the darker appearance of wool that only went through the decomposition faze.

**Setup for oxidation of ammonia**

For the oxidation reaction vessel, a quartz tube is used with the catalytic quartz wool placed inside. On one end a glass nozzle is inser4ed (Figure 3). It serves to mix ammonia with air. The nozzle consists of a glass tube with an inlet for the ammonia on the side and a thin glass tube in line with the main tube to serve as an inlet for air. This configuration allows for proper mixing of the gases before reaching the catalyst. The nozzle is connected to a quartz tube filled with the platinum wool catalyst. The other end of the tube is connected to a glass bubbler placed in a meter long test tube with a diameter of 30mm (Figure 4).

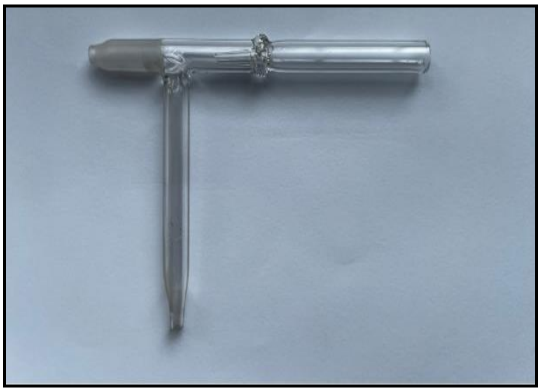

**Fig 3:** Glass nozzle.

This is in order to let the gas bubbles have as much surface area to intact with the water in the test tube and to interact for the longest period of time to ensure all nitrogen oxides are collected. The test tube is placed in a lager tube filled with ice water to maintain a low temperature.

**Fig 4:** Test tube glass bubbler un ice water.

The air is fed in by air pump and the ammonia is produced by the reaction between an ammonium-based salt with strongly basic NaOH solution.

The addition of aqueous NaOH to the ammonium must be controlled and monitored to ensure that the production of gas is stable. The air pump must be tuned to provide enough air to combust the ammonia on the catalytic wool.

To activate the catalyst, it is extremely heated until it produces enough heat to operate on its own.

**Activation of catalyst and product collection**

To generate the ammonia a separate flask is used. It is filled with a ammonium salt. For the experiment ammonium chloride was used. A addition funnel is added to the flask and it is filled with a concentrated NaOH solution.

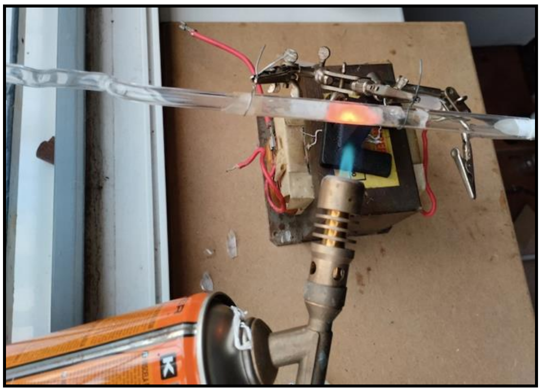

**Fig 4:** Activation of catalyst by outside flame.

An adapter is used in the flask in order to allow the generated ammonia to flow out of the flask and into the gas mixing nozzle. Before starting the generation of ammonia a flame is used to heat the catalyst to red hot and air flow through the system is started. Once the catalyst is at temperature the NaOH solution can be added to ammonium salt. Upon initial combustion a smoke should form on the catalyst and then disappear. The production of ammonia must be adjusted so that this smoke does not form and so the catalyst can maintain the combustion of ammonia without the need for an external heat source (Figure 5).

As for the collection of the nitrogen oxides produces there are two option that were analyzed. The first was to use a cold solution of hydrogen peroxide and water to collect the gases and produce dilute nitric acid. The second involved a basic solution that would react with the gasses and form nitrates.

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The addition of aqueous NaOH to the ammonium must be controlled and monitored to ensure that the production of gas is stable. The air pump must be tuned to provide enough air to combust the ammonia on the catalytic wool.

To activate the catalyst, it is extremely heated until it produces enough heat to operate on its own.

**Results**

The two methods of collecting the nitrogen oxides give different results. Using a hydrogen peroxide solution gives a dilute nitric acid with a concentration of less than 10%. This method of collecting the gases is temperature sensitive as it relies on the solubility of the gases in water. The gases can dissolve to a certain point but the fact that the gases are heated in the combustion reaction means that they will have more difficulty dissolving. Although it is not viable for collecting the product this method is great for calibrating the system as it shows immediately if residual ammonia is passing through the catalyst based on the ph.

The method of using a basic solution for nitrate collection proved to be much more efficient with a nitrate yield of over 40%. This was on a run where there was trouble with ammonia leakage and adjusting ammonia flow in the system but based on monitoring just the yield of the tuned system it can be expected to reach upwards of 50 to 60% with minor improvements. There is potential for even higher yields over 70% if a more controlled pressurized source of ammonia is used to prevent flow fluctuations. Adding multiple stages of gas collection and can increase yield to 80%.

**Discussion**

The final efficiency of the process is determined by two main factors. The first one is the ammonia-air mixture. A slight excess of air is required for all of the ammonia to react. This is dependent on the catalyst in the sense that it still must have enough ammonia to be able to maintain temperature without an external heat source.

The second factor that determines the efficiency is the gas flow rate. This is also catalyst dependent. A dense and long catalyst is better suitable for higher flow rates because the ammonia won't oversaturate the first part of the catalyst. It will be oxidized over the whole length of the catalyst. If a lower flow rate is used on a long catalyst that is heated, it is possible that the back part of the catalyst decomposes the nitrogen oxides produced, destroying the product.

For short catalysts it is important to use lower flowplates to ensure complete oxidation. Otherwise there will be residual ammonia mixed with the nitrogen oxides that forms a smoke consisting of ammonium nitrate.

A second approach for making nitric acid on small scale would be using the Birkeland-Eyde (B-E) process. The principle of operation of a B-E reactor would be much simpler than one operating on the Ostwald process, but it would have certain drawbacks. The main one would be that a carefully controlled plasma must be formed and maintained in order for atmospheric nitrogen to be oxidated. The second would be the limitation in effective flow rate of the reactor. A B-E reactor needs to be much longer than an Ostwald reactor to have equal output of nitric acid per unit of time.

## Conclusion

Despite its solubility in water it is very difficult to collect and it escapes the apparatus even after being bubbled in water several times. A possible solution for this “smoke” would be a fine quartz wool air filter that could be used to collect the ammonium nitrate, but its generation should be avoided because of the inefficiency it brings.

The catalyst and apparatus used in this paper allow for the Ostwald process to be used for small scale nitric acid production offer better control over its production speed than the Birkeland-Eyde process.

## Funding

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## Conflict of Interests

The author declares that there is no conflict of interest.

## References

1. Balarini CM, Cruz JC, Alves JLB, França-Silva MS, Braga VA. Developing New Organic Nitrates for Treating Hypertension. In: Seabra AB, editor. Nitric Oxide Donors. Academic Press; 2017. p. 243-262.
2. Ramanujam A. Purex and Thorex Processes (Aqueous Reprocessing). In: Buschow KHJ, Cahn RW, Flemings MC, Ilschner B, Kramer EJ, Mahajan S, Veyssière P, editors. Encyclopedia of Materials: Science and Technology. Elsevier; 2001. p. 7918-7924.
3. Gad SC. Nitric Oxide. In: Wexler P, editor. Encyclopedia of Toxicology. 3rd ed. Academic Press; 2014. p. 528-529.
4. Ruan C, Wang X, Wang C, Zheng L, Li L, Lin J, *et al*. Selective catalytic oxidation of ammonia to nitric oxide via chemical looping. Nature Communications. 2022 Feb 7;13(1):718.
5. Abdel Latef AA, Dawood MF, Hassanpour H, Rezayian M, Younes NA. Impact of the static magnetic field on growth, pigments, osmolytes, nitric oxide, hydrogen sulfide, phenylalanine ammonia-lyase activity, antioxidant defense system, and yield in lettuce. Biology. 2020 Jul 17;9(7):172.