CHEMICAL PREPARATION FOR PRISONERS

- 1. How to prepare pure hydrochloric acid
- 2. Ammonium Nitrate from Household Chemicals
- 3. How to Prepare Chlorine Gas
- 4. Potassium Nitrate Recipe
- 5. Sodium Carbonate from Baking Soda
- 6. How To Make Sulfuric Acid at Home
- 7. Sulfuric Acid Projects and More
- 8. How to Make Nitrous Oxide or Laughing Gas

1. How to prepare pure hydrochloric acid

Dense fumes when vapors of Hydrochloric acid and ammonia interact



Description of Hydrochloric acid

Hydrochloric acid is a solution of hydrogen chloride gas dissolved in water. It is a corrosive and toxic strong acid that emits choking fumes when concentrated. The impure technical grade is called muriatic acid which is available at home improvement stores as well as pool care centers. It is usually contaminated with iron, chlorine, and organics giving it a yellow tint.

To perform titrations or make pure chloride salts, a purer form of hydrochloric acid will be needed. In this hub, it will be discussed on how hydrochloric acid in pure form can be prepared in a home lab.

Traditional procedure of hydrochloric acid synthesis

The oldest method of preparation of hydrochloric acid is to create hydrogen chloride gas by adding concentrated sulfuric acid to a chloride salt such as sodium chloride. This hydrogen chloride gas is led into chilled water until an acid of the desired concentration is achieved. The sulfuric acid is a source of protons and the hydrogen chloride gas is volatile and is removed as it is formed. This is a useful method but requires alot of apparatus. Also, in the later stages of the reaction, external heating is needed to drive the reaction because the sodium bisulfate formed slows evolution of hydrogen chloride.

Another variation on this theme is to take muriatic acid purchased from a home center or pool supply place and add sulfuric acid to this at a controlled rate. The heat generated keeps the reaction going and the gas can be led into purified water. When hydrogen chloride evolution ceases, the sulfuric acid is diluted and it can be concentrated by heating it in pyrex beaker or flask until specific gravity of 1.84 is reached. This way no sulfuric acid is wasted.



Source: Calcium HCl Gen

calcium chloride method

If using sulfuric acid is not option, then you should consider the calcium chloride method. In this method concentrated muriatic acid can be dripped on anhydrous calcium chloride. This will evolve some hydrgen chloride. The best proceedure is to take an erlenmeyer flask and place the solid calcium chloride pellets in the flask. Then secure a pressure equalizing addition funnel filled with concentrated hydrochloric and add dropwise collecting the gas evolved. This is from the article in Journal Chemical Education circa 1995 concerning this preparation.

Again the dehydrating agent, calcium chloride, can be regenerated by heating the solid mass to 200 Celsius.

Pure hydrochloric acid from impure muriatic acid

On youtube, I found the most interest video regarding the preparation of pure hydrochloric acid from impure muriatic acid. It is so simple and requires no expensive glassware. It is based on taking two small plastic or glass containers and putting an equal volume of muriatic acid and distilled water in each one. Then carefully placing both open containers in a larger container and sealing it undistrubed for about a week or so. After that time, you will have two containers with hydrochloric acid of the same concentration! What happens is the fumes from the muriatic acid are trapped in a closed system and the pure distill water absorbs them. As this occurs, more vapors are evolved from the muriatic acid to continue the process until an equilibrium is reached. This produces dilute hydrochloric acid only but that is all that is needed for preparing most pure chlorides. More hydrogen chloride can be dissolved to concentrate the diluted acid.

Purify hydrochloric acid - Possibly another method

This by no means practical for large scale work, but may be a way to reclaim some HCl from chlorides. Some metal chlorides, especially the hydrated heavy metal ones, give off HCl fumes when heated leaving a mass of metal oxide or oxychloride behind. Several metal salts are capable such as magnesium chloride, iron(II)chloride, iron(III)chloride, aluminum chloride etc. It advisable to find a chloride that decomposes at a relatively low temperature to minimize difficulties.

An example is if you use muriatic acid to remove mill scale, after time, the acid will become green/yellow with iron(II)chloride and iron(III)chloride. In time it would lose its potency. To alleviate this, a glass distilation set up can be set up and unused hydrochloric acid can be collected. Then when the iron chloride mix is more concentrated, the heat can be increased to liberate free HCl to be collected. Depending on what iron chloride was present, black iron oxide or red iron oxide would be left behind.

2. Ammonium Nitrate Synthesis

Make Ammonium Nitrate from Household Chemicals

 $\begin{bmatrix} H \\ I \\ H \\ H \\ H \end{bmatrix}^{+} \begin{bmatrix} O \\ I \\ O \\ O \\ O \\ O \\ O \end{bmatrix}^{-}$

This is the two-dimensional chemical structure of ammonium nitrate.

Ben Mills

You can make ammonium nitrate from common household chemicals. The ammonium nitrate may be used to make pyrotechnics or cold packs or to perform other interesting demonstrations. Keep in mind you can buy ammonium nitrate as a pure chemical or you can collect it from instant cold packs or some fertilizers, probably more easily and inexpensively than making it yourself. The easiest way to make ammonium nitrate is simply by reacting nitric acid with ammonia, but if you don't have access to nitric acid (or don't want to mess with it), you can make ammonium nitrate from readily available home chemicals.

Ammonium Nitrate Chemicals

You will need:

- 138 g sodium bisulfate (found with pool chemicals, used to lower pH)
- 1 mole equivalent of a nitrate salt... any of the following
 - 85 g sodium nitrate (common food preservative)
 - 101 g potassium nitrate (which you can buy or make yourself)
 - 118 g calcium nitrate (tetrahydrate)
- ammonia (common household cleaner)
- methanol (optional, which may be found as HEET fuel treatment)

Prepare Ammonium Nitrate

- 1. Dissolve the sodium bisulfate in the mininum amount of water (about 300 ml).
- 2. Dissolve your nitrate salt in the minimum amount of water (amount depends on the salt).
- 3. Mix the two solutions.
- 4. Next you want to neutralize the solution, which is quite acidic. Stir in ammonia until the pH of the mixture is 7 or higher. Use a pH meter (or pH paper). Reacting ammonia, sodium bisulfate, and nitrates will give you sodium sulfate and ammonium nitrate.

- 5. Sodium sulfate and ammonium nitrate have different solubilities in water, so boil the solution to get the sodium sulfate to crystallize. Remove the liquid from heat when crystals of sodium sulfate form in the bottom of the pan.
- 6. Chill the solution in the freezer to get as much of the sodium sulfate as possible to drop out of the solution.
- 7. Run the solution through a filter (coffee filter or paper towels) to separate the solid sodium sulfate from the ammonium nitrate solution.
- 8. Allow the ammonium nitrate solution to evaporate, which will give you ammonium nitrate, with some sodium sulfate impurity. This is 'good enough' for most chemistry projects.
- 9. If you want to further purify the ammonium nitrate, dissolve it in about 500 ml of methanol. The ammonium nitrate is soluble in methanol, while the sodium sulfate is not.
- 10. Run the solution through a filter, which will give you sodium sulfate on the filter and a solution of ammonium nitrate.
- 11. Allow the methanol to evaporate from the solution to obtain crystalline ammonium nitrate.

Safety Information

The chemicals used in this project are smelly and corrosive, so this project should be performed under a fume hood or outdoors. As always, wear gloves, eye protection, and appropriate clothing. Some of the reagents and the final product are flammable or are oxidizers, so keep the chemicals away from open flames.

3. How to Prepare Chlorine Gas

Gas Preparation Instructions

These are instructions for preparing chlorine gas (Cl₂) from potassium permanganate and concentrated hydrochloric acid.

Reactants

potassium permanganate concentrated hydrochloric acid

Gas Preparation

Add concentrated hydrochloric acid dropwise onto a small quantity of potassium permanganate crystals (in a flask). Collect the chlorine gas by upward displacement of air in a hood. Use proper safety precautions. Concentrated hydrochloric acid is extremely caustic. Chlorine gas is toxic.

Reaction

6HCl + 2KMnO₄ + 2H⁺ --> 3Cl₂ + 2MnO₂ + 4H₂O + 2K⁺

Potassium Chlorate from Bleach and Salt Substitute

How to Make Potassium Chlorate from Household Chemicals

Potassium chlorate is an important potassium compound that can be used as an oxidizer, disinfectant, source of oxygen, and component in pyrotechnics and chemistry demonstrations. You can make potassium chlorate from common household bleach and salt substitute. The reaction is not particularly efficient, but it's something to keep in mind if you need potassium chlorate right away or just want to know how to make it.

Materials for Making Potassium Chlorate

- chlorine bleach
- potassium chloride (sold as a salt substitute)
- filter paper or coffee filter

Prepare Potassium Chlorate

 Boil a large volume (at least a half liter) of chlorine bleach, just until crystals start to form. Do this outdoors or under a fume hood, to avoid inhaling the vapor. Boiling bleach disproportionates sodium hypochlorite into sodium chloride and sodium chlorate.

 $3 \text{ NaClO} \rightarrow 2 \text{NaCl} + \text{NaClO}_3$

- 2. As soon as crystals start to form, remove the bleach from heat and allow it to cool.
- 3. In a separate container, prepare a saturated solution of potassium chloride by stirring potassium chloride into water until no more will dissolve.
- 4. Mix equal volumes of the boiled bleach solution and potassium chloride solution, taking care to keep solids from either solution out of the mixture. Potassium chlorate will precipitate out, leaving sodium chloride in solution.

 $\mathsf{KCI} + \mathsf{NaCIO}_3 \rightarrow \mathsf{NaCI} + \mathsf{KCIO}_3$

- 5. Cool the solution in the freezer to increase the potassium chlorate yield.
- 6. Filter the mixture through filter paper or a coffee filter. Keep the solid potassium chloride; discard the sodium chloride solution.
- 7. Allow the potassium chlorate to dry before storing or using it. NurdRage has a video of the process, if you'd prefer to see how it's done.

You can test the potassium chlorate in a simple chemistry demonstration:

- Purple Fire (shown) Mix potassium chlorate and half as much sugar. Ignite the mixture either by applying a flame or adding a few drops of sulfuric acid (instant chemical fire).
- Dancing Gummi Bear The candy is the source of the sugar in this demonstration. The vigorous reaction between the candy bear and the potassium chlorate makes the bear appear to dance in purple fire.

4. Potassium Nitrate Recipe

Make Potassium Nitrate from Lite Salt and a Cold Pack

Make potassium nitrate (saltpeter) from common household ingredients. Potassium chloride from lite salt and ammonium nitrate from a cold pack are reacted to yield potassium nitrate and ammonium chloride. This is an easy way to make your own potassium chloride if you can't find it in a store or just want to try a fun chemistry experiment.

Potassium Nitrate Ingredients

- 40 g ammonium nitrate (from an instant cold pack which has ammonium nitrate listed as its ingredient)
- 37 g potassium chloride (sold as a salt substitute, with potassium chloride listed as the ingredient)
- 100 ml water

The Chemical Reaction

Aqueous solutions of ammonium nitrate and potassium chloride are reacted to exchange the ions and form potassium nitrate and ammonium chloride. The ammonium chloride is much more soluble in water than the potassium nitrate, so you will get potassium nitrate crystals, which can be separated from the ammonium chloride solution.

 $NH_4NO_3 + KCI \rightarrow KNO_3 + NH_4CI$

Make Potassium Nitrate

- 1. Dissolve 40 g of ammonium nitrate into 100 ml of water.
- 2. Filter the solution through a coffee filter to remove any undissolved material.
- 3. Heat the solution with 37 g potassium chloride to dissolve the lite salt. Do not boil the solution.
- 4. Filter the solution and either set it in the freezer to chill or else put it in an ice bath so you can observe the crystallization of the potassium nitrate.
- 5. Pour off the ammonium chloride solution, leaving the potassium nitrate crystals. You can recover the ammonium chloride, too, if you like.
- 6. Once the potassium nitrate crystals are dry, you can use them for chemistry experiments. The resulting potassium nitrate does contain impurities, but it will work fine for pyrotechnics projects and other experiments described on this site.

5. Sodium Carbonate from Baking Soda

How to Make Sodium Carbonate from Sodium Bicarbonate

These are easy instructions for making sodium carbonate, also known as washing soda or soda ash, from baking soda or sodium bicarbonate.

Make Sodium Carbonate

Sodium bicarbonate is CHNaO₃ while sodium carbonate is Na₂CO₃. Simply heat baking soda or sodium bicarbonate in a 200°F oven for about an hour. Carbon dioxide and water will be given off. Dry sodium carbonate will remain.

6. How To Make Sulfuric Acid at Home

Concentrated Sulfuric Acid

Sulfuric acid is a useful acid to have on hand for a variety of home chemistry projects. However, it is not easy to obtain. Fortunately, you can make it yourself.

Homemade Sulfuric Acid Materials

Actually, this method starts with diluted sulfuric acid, which you boil to make concentrated sulfuric acid. This is the safest and easiest method of making sulfuric acid at home.

- car battery acid
- glass container
- outdoor source of heat, like a grill

Battery acid, which may be purchased at an automotive supply store, is approximately 35% sulfuric acid. In many cases, this will be strong enough for your activities, but if you need concentrated sulfuric acid, all you need to do is remove the water. The resulting acid will not be as pure as reagent-grade sulfuric acid, so keep this in mind.

Safest Method

If you aren't in a hurry, you can concentrate sulfuric acid by allowing the water to evaporate naturally. This takes several days.

- 1. Place an open container of sulfuric acid someplace with good circulation, safe from the possibility of a spill.
- 2. Loosely cover the container to minimize contamination with dust and other particulates.
- Wait. The water will evaporate out of the solution, eventually leaving you with concentrated sulfuric acid. Note that sulfuric acid is highly hygroscopic, so it will retain a certain amount of water. You would need to heat the liquid to drive off the remaining water.

Quickest Method

The fastest method to concentrate sulfuric acid is to boil the water out of the acid. This is fast, but requires extreme care. You'll want to do this outdoors so that you won't be exposed to acid fumes, using borosilicate glass (e.g., Pyrex or Kimax). There is always a risk of shattering a glass container, no matter what you are heating, so you need to be prepared for that possibility. Do not leave this project unattended!

- 1. Heat the battery acid in a borosilicate glass pan.
- 2. When the liquid level stops dropping, you will have concentrated the acid as much as you can. At this point, the steam will be replaced by white vapor, too. Be careful to avoid inhaling the fumes.
- 3. Allow the liquid to cool before transferring it to another container.
- 4. Seal the container to prevent water from the air from getting into the acid. If the container is left open for too long, the sulfuric acid will become diluted.

Safety Notes

- It's advisable to keep baking soda (sodium bicarbonate) or another base on hand. If you spill some acid, you can quickly neutralize it by reacting it with the baking soda. Simply sprinkle baking soda on the spill.
- Be careful to avoid contact with the sulfuric acid! Sulfuric acid is one of the strong acids. It is extremely corrosive and will react vigorously and unpleasantly with skin, mucous membranes, clothing and just about anything else it touches. Do not breathe the vapors, do not touch the acid and do not spill it. Tie long hair back, wear goggles and gloves and cover exposed skin.
- Don't use metal pans or utensils. Sulfuric acid reacts with metal. Also, it will attack some types of plastic. Glass is a good choice.
- Sulfuric acid reacts with water in an exothermic reaction, but dilution with water is the best
 way to deal with an acid spill. Be sure to have copious amounts of water available, just in
 case something goes wrong. You can flood a small amount of acid with water. Caution:
 Sulfuric acid will splash when mixed with water! If you are going to work with this acid, know
 and respect its properties.

7. Sulfuric Acid Projects and More

How To Prepare Sulfuric Acid Solutions/Dilutions Sulfuric Acid and Sugar Demo Mixing Sulfuric Acid and Water Sulfuric Acid Burn Accident What Is Battery Acid?

Notes About Battery Acid

Battery acid is about 35% sulfuric acid. You can purchase it at an automotive supply store. It may not be on the shelf, so ask for it. Battery acid may be sold in 5 gallon boxes, with the acid in a heavy duty plastic bag and a plastic tube to dispense the liquid. The box is heavy; it would be disastrous to drop it. Therefore, it's a good idea to know what to expect.

It's practical to dispense a working volume of acid, rather than try to deal with the entire container. Although the acid may come in a plastic container, it's best to store this acid in a glass bottle. Sulfuric acid reacts with some types of plastic and may corrode a plastic container. I used a glass wine bottle that had a plastic screw-top cap. Whatever container you use, be **certain** to label it as sulfuric acid and poison and store it somewhere that children and pets can't get to it. Also, don't store acid with ammonia because the two chemicals mix to release toxic fumes.

8. How to Make Nitrous Oxide or Laughing Gas



You can make nitrous oxide or laughing gas quite easily in the lab or at home. However, there are reasons why you might want to forego the preparation unless you have chem lab experience.

What Is Nitrous Oxide or Laughing Gas?

Nitrous oxide (N_2O) is also known as laughing gas. It is a colorless sweet-smelling and sweettasting gas that is used in dentistry and surgery because inhaling the gas produces analgesic and anesthetic effects. The gas is also used to produce the engine output of automotive vehicles and as an oxidizer in rocketry. Nitrous oxide gets the name "laughing gas" because inhaling it produces euphoria.

How to Make Nitrous Oxide or Laughing Gas

Joseph Priestley first synthesized nitrous oxide in 1772 by collecting the gas produced from sprinkling nitric acid over iron filings, however, nitrous oxide usually is produced using Humphry Davy's method of gently heating ammonium nitrate to decompose it into nitrous oxide and water vapor:

 NH_4NO_3 (s) \rightarrow 2 H_2O (g) + N_2O (g)

The key here is *gently* heating the ammonium nitrate to between 170°C and 240°C, because higher temperatures may cause the ammonium nitrate to detonate. People have been doing this without incident for over 150 years, so the procedure is safe as long as you take care.

Next, the hot gases are cooled to condense the water. The best way to do this is using a pneumatic trough, which involves a tube leading from the ammonium nitrate container that bubbles the gases up through water into a collection jar. You want the rate of gas production to be a bubble or two per second. The pneumatic trough removes the water from the reaction as well as smoke from impurities in the ammonium nitrate.

The gas in the collection jar is your nitrous oxide or laughing gas, plus lesser amounts of other nitrogen oxides, including nitric oxide or nitrogen monoxide. Nitric oxide eventually is oxidized to nitrous oxide upon exposure to oxygen, although acid and base treatments are used to remove impurities for commercial-scale production of nitrous oxide. When your container is full of gas, discontinue heating the ammonium nitrate and disconnect the tubing so that water will not flow up into your collection container. Cover the container so that you can turn it upright without losing the gas. If you don't have a lid for the container, a flat sheet of glass or plastic works fine.

Safety Precautions

- Higher purity ammonium nitrate is more stable than ammonium nitrate that contains impurities, so safety improves if you start with high quality starting material.
- Don't exceed 240°C or you'll risk explosive decomposition of the ammonium nitrate.
- If you are using a direct heat source, such as a thermostat controlled burner, don't decompose the last bit of ammonium nitrate since it is more likely to overheat.
- Nitrous oxide is a safe lab gas, but overexposure via inhalation may result in asphyxiation, in much the same way as overexposure to helium gas presents a risk.