

# HOMEMADE CYANIDE

Cyanide is the granddaddy of synthetic poisons. Easily made from common chemicals, cyanide is used as is for poisoning bullets and food, and is used in the making of Hydrogen Cyanide, Cyanogen Chloride, Tabun, and numerous other poison gases.

A dose as small as 50 milligrams (ingested) can kill an adult man. Of course, the more the better.

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To make cyanide, you must first make ferrocyanide. This can either be made, or preferably, bought.

## Ferrocyanide Preparation

Ferrocyanide is obtained by heating 10 parts (by weight) potassium (or sodium) carbonate; 10 parts coke, cinders, or coal; and 3 parts iron turnings, all in coarse powder. to a full red heat in an open crucible, stirring occasionally until small jets of purple flame arc no longer seen. When cool, the soluble matter is dissolved out of it, the solution filtered, evaporated, and crystallized. The crystals obtained are redissolved in hot water and cooled very slowly, forming large yellow crystals of the ferrocyanide.

In order to obtain a pure form, melt dried ferrocyanide in a glass vessel and let cool, dissolve the fused mass in water, neutralize any excess of alkali with acetic acid (vinegar), and precipitate the salt by adding strong alcohol to the solution. Wash the precipitate with a little weak alcohol, redissolve it in water, and crystallize.

Sodium Ferrocyanide ( $\text{Na}_4\text{Fe}(\text{CN})_6 \cdot 10\text{H}_2\text{O}$ , FW 484.07, mp 82C, CAS# 13601-19-9, AKA Yellow Prussiate of Soda) can be ordered quite cheaply from [www.sciencealliance.com](http://www.sciencealliance.com).

Catalog #s and prices are: C7435-100 100g \$4.12, C7435-500 500g \$8.96. And there's no hazardous shipping fees for it either. Cyanide costs about \$47 a pound plus \$15 hazardous.

## Cyanide Preparation

To convert the ferro to cyanide do this (please note that these instructions were for the potassium not sodium salt. I'm assuming that the ratios are similar for both); Mix thoroughly 8 parts of dry sodium ferrocyanide and 3 parts dry sodium carbonate (pool pH adjuster), heat them in a steel container (cleaned oil filter can works nicely), with constant stirring (use clothes hanger, straightened out), with a propane torch or other intense heat source till it melts into a clear liquid. Heating is continued until the mix no longer fizzes and the fluid portion is colorless. After a few minutes rest, to allow the contents to settle, the clear portion is poured from the heavy black sediment (iron) at the bottom at the bottom of the crucible and onto to a clean slab or steel bowl. It's then broken up while still warm and stored in airtight bottles. This will be

almost pure cyanide.

# RICIN

Ricin is just about the easiest, and at the same time, most toxic poison that a criminal can make. Less than a milligram (1/1,000 of a gram) injected or inhaled will kill a person several times over. For individual killings, it has the advantage of being undetectable in toxicology scans since the poison is a catalyst that starts a chain reaction in the body, and is destroyed before the symptoms begin to show.

With properly sized and dispersed dry particles, ricin is at least 10x more toxic than the most potent nerve gas. A 1% water solution atomized with a small explosive burster has the same effectiveness as sarin nerve gas. The only disadvantage ricin has is the time it takes for the victims to die is about 1 - 2 weeks. So you won't have the quick tactical effect of nerve gas. But this can also be good in that, using a covert dissemination, the criminal has time to escape before the attack is detected.

The information presented below is from a US Patent #3,060,165, assigned to the US Army.

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## Tips

Here's a few things you need to know to make your production go much easier.

1. The seeds are readily available through wholesale seed suppliers for about \$20 for a pound of seeds. Castor bean seeds are very tough to crack or peel. Soak them for an hour in a solution of 2 tablespoons lye in 1 cup water. Then use pliers to crack the shell. The shell will peel off the bean easily then.
2. Use a 1/2 cup of acetone to every ounce of bean pulp. Blend well. Let sit for several days with occasional shaking. Pour off the acetone and add an additional 1/2 cup of acetone and repeat. This will remove almost all the castor oil from the seeds.
3. The patent doesn't mention it, but you can use magnesium sulfate (Epsom salt) instead of sodium sulfate. Epsom salt is easily available in any drug store for just about a dollar a pound.
4. Use a plastic membrane filter if you can get them. The ricin forms a layer that is difficult to remove from a regular coffee paper filter without scraping off fibers as well.
5. Wear a gas mask and gloves. Try to keep the ricin wet at all times to avoid generating any dust (DEADLY!). And always shower and change clothes after handling.

## Preparation

Ricin is a protoplasmic poison prepared from castor beans after the extraction of castor oil therefrom. It is most effective as a poison when injected intravenously or inhaled, the latter requiring extreme commutation and small particle size to be effective, It is believed that the toxic action is catalytic rather than stoichiometric which probably accounts for the high toxicity

of the agent.

Because of its relative instability ricin must be handled with extreme care. In neutral aqueous solution it is stable only up to 60"-75" C., and in solid form up to 100"- 110" C., although for short exposures, temperatures up to 130" may be tolerated. It is sensitive to acids, alkalis and halogens and may also be inactivated by mechanical working such as grinding or pulverizing. These factors are of great importance in developing a satisfactory method for preparing the material.

Although ricin has been prepared in crystalline condition in the laboratory in small quantities, it becomes necessary, for purposes of toxicological warfare, to prepare relatively large quantities in a high state of purity. This necessitates that as much as possible of the non-toxic material present be removed in the process.

In preparing the protein material, the castor beans are first ground and pressed to remove most of the oil. The pressed cake still retains about 15% oil and this may be removed by means of solvents which will extract an additional 150 pounds of oil per ton of beans and reduce the oil retained in the cake to a little over 1%. In the event that the expressing step is supplemented by solvent extraction, it is important to prevent detoxification of the protein during the solvent removal step. If residual solvent is removed from the ground beans by blowing with steam, considerable detoxification results. Blowing with nitrogen effectively prevents detoxification but is expensive when carried out on a large scale.

After the oil has been removed, the pressed cake or pomace is extracted by agitating with water at a pH of 3.8+-0.1 at 25" C. which removes substantially all of the toxic protein. The extraction process is operative within a pH range of about 3 to 4.5 although the preferred range is about 3.5 to 4. The optimum operating point is a pH of 3.8+-0.1, as indicated above. A careful pH control is essential in order that as much non-toxic protein as possible may be eliminated and also that the filtration rate may be held at a satisfactory value. Either HCl or H<sub>2</sub>SO<sub>4</sub> may be used to get the desired pH for the extraction water, but H<sub>2</sub>SO<sub>4</sub> is preferred due to its lower corrosion rate and ease of handling in concentrated form. The acid should be used in reasonably dilute form to prevent undue local concentrations during its addition. A 5% concentration is satisfactory.

Following the extraction, the slurry is filtered using either a conventional recessed plate filter or a continuous string discharge vacuum filter. With the latter about 7% of filter aid, based on meal weight, was found necessary for satisfactory filtration.

The filtrate from the water extraction step, which contains the ricin, was treated with a 16.7% solution of Na<sub>2</sub>SO<sub>4</sub> to precipitate the protein. This solution is composed of 20 pounds of salt in 100 pounds of water and the amount used was such that the salt content equaled 20% of the filtrate weight. This amount and concentration of salt solution was about optimum considering the factors of cost and toxin recovery. Somewhat higher concentrations and larger amounts of solution can be used, however.

The precipitation process is not limited to the use of Na<sub>2</sub>SO<sub>4</sub> since a saturated solution of NaCl can be used successfully, but Na<sub>2</sub>SO<sub>4</sub> solution gives better nitrogen fractionation, more rapid precipitation, and can be operated under wider pH limits. It is desirable to raise the pH to about

7-8 before precipitation as this gives better recovery and greater non-toxic nitrogen removal. The pH was raised to this value by using NaOH or Na<sub>2</sub>CO<sub>3</sub> the latter being preferred. The base used was quite dilute in order to prevent detoxification due to high local concentrations in the solution. A 5% solution of NaOH was used, whereas with Na<sub>2</sub>CO<sub>3</sub> a 12% solution was preferred.

In general, this higher pH during precipitation gave a greater non-toxic nitrogen fractionation and at the same time maintained the toxin loss at less than 2%. After precipitation, the slurry was filtered using from 1 to 4% filter aid, based on slurry weight, for satisfactory filtration, the amount of filter aid needed being dependent on the type of press used. Washing the filter cake with Na<sub>2</sub>SO<sub>4</sub> solution removed additional non-toxic nitrogen which is desirable. In this washing step a 16.7% solution of Na<sub>2</sub>SO<sub>4</sub> was again used. This washing step removed an additional 15% of non-toxic nitrogen from the cake.

After filtration the filter cake which contains the ricin in combination with the Na<sub>2</sub>SO<sub>4</sub> may be dried and slurried with CCl<sub>4</sub> to separate the ricin by flotation. Separation of the ricin after a single precipitation and washing step is possible, but it is preferred to carry the process through an additional extraction and precipitation step. This is accomplished by slurrying the filter cake in three times its weight of water and the pH of the slurry is again brought to 3.8±.1 by means of 5% H<sub>2</sub>SO<sub>4</sub>. The slurry is filtered and a second precipitation is brought about by adding Na<sub>2</sub>SO<sub>4</sub> solution. Although pH control here is not wholly essential it is advantageous to bring the pH to approximate neutrality by adding 12% Na<sub>2</sub>CO<sub>3</sub>.

A precipitation time of 45 minutes was necessary to obtain complete removal of the toxin. In filtering out the precipitate, no filter aid was used and the filter cake was washed with Na<sub>2</sub>SO<sub>4</sub> solution on the filter whereby an additional amount of non-toxic nitrogen was removed from the cake. This washing was effective only the first time and repeated washings had little effect in removing further non-toxic nitrogen.

The ricin-Na<sub>2</sub>SO<sub>4</sub> precipitate was dried at about 50° to 60° C. on a hot air tray dryer. The dried product was ground to pass a 40 mesh screen and agitated with 5 times its weight of CCl<sub>4</sub> which served to separate the ricin from the Na<sub>2</sub>SO<sub>4</sub> by flotation. After settling, the ricin was skimmed off the top. This reduced the Na<sub>2</sub>SO<sub>4</sub> content of the mixture from a previous 40 to 50% down to 15 to 18%. About 1 to 2% of nitrogen remained in the Na<sub>2</sub>SO<sub>4</sub> salt which could then be used for subsequent precipitations.

The final precipitation produced a particle size of 1-2 μ. On drying the wet cake, however, the ricin cemented together forming larger particles. These could not be broken down to their original size by ordinary grinding methods and since a very fine particle size was necessary in order that the product might be used as a toxic weapon, it was thought desirable to seek some method to prevent the agglomeration or cementing process that took place on drying.

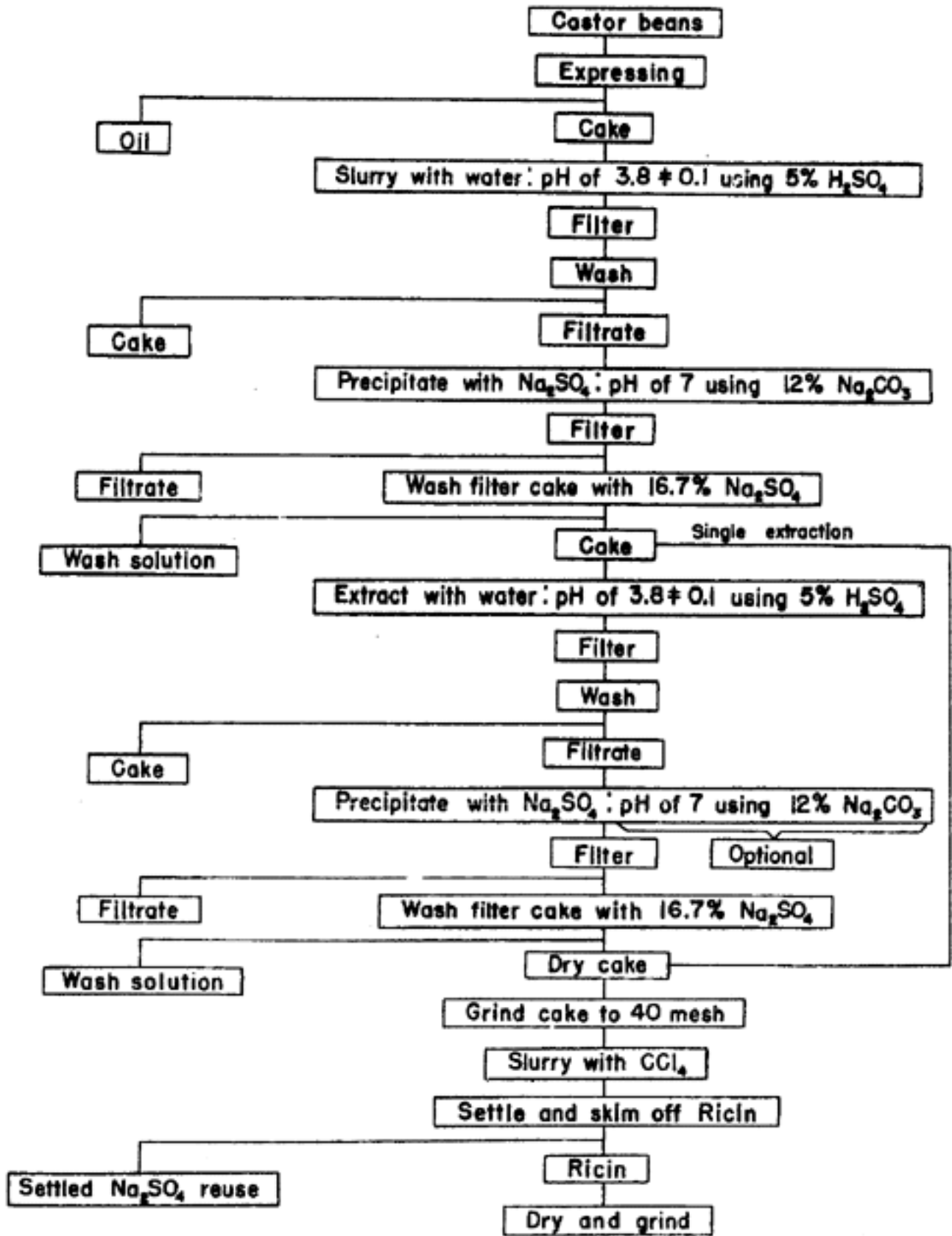
To attempt to affect this result, physical conditions prevailing under the precipitation process were changed. This included changing the temperature of precipitation and the rate of agitation. Other changes included precipitation with only partial saturation of Na<sub>2</sub>SO<sub>4</sub> and the use of wetting and seeding agents. None of these expedients produced any significant improvement in particle size. Ordinary dry ball and hammer milling of the dried ricin produced considerable detoxification perhaps due to the generation of excess heat. The use of CCl<sub>4</sub> slurry plus the

use of low temperature and low moisture content of the ricin reduced detoxification during ball milling.

Spray drying proved to be an even better method of securing a reasonably small particle size. Best results were achieved by using a solution having about 20% solids, an inlet temperature of 150° C. and an atomizing air pressure of 150 to 180 p.s.i. The particle size secured was 6 to 8  $\mu$ .

The best means of securing a small particle size was by air grinding. This was carried out in an apparatus having a chamber with conical top and bottom. The material to be ground has been fed into this chamber and is withdrawn from the bottom and forced back into the center of the chamber tangentially through a venturi. Compressed air of about 100 p.s.i. was fed to the venturi to provide the grinding force. The fines are drawn off the top and the large particles settle to the bottom to be recirculated and reground. This process produced particles having a mass median diameter of 2.5 to 3.5  $\mu$ .

Numerous variations are possible in the several steps of the process commencing with the water extraction and precipitation which may be a single or multiple step. Although a single extraction step can be used, as indicated before, some process modifications are necessary for its successful operation on a plant scale. Double extraction proved to be quite efficient but additional steps beyond the second extraction step were not found necessary. The drawing is self-descriptive and shows the various steps of the process described.



References Cited in this patent:

Kabat et al.: J. Biol. Chem., vol. 168, 1947, pages 629-39 .

Kunitz et al.: J. Gen. Physiol, vol. 32 (1948), pages 25-31.