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O. R. Sweeney
Iowa State College

L. K. Arnold
Iowa State College

R. K. Dix
Iowa State College

W. R. Stelzer Jr.
Iowa State College

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THE PRODUCTION OF CHLOROPICRIN AS NEEDED FROM DRY MIXTURES OF PICRIC ACID AND BLEACHING POWDER

O. R. SWEENEY, L. K. ARNOLD, R. K. DIX AND
W. R. STELZER, JR.

Chloropicrin or trichloromethane is a colorless liquid boiling at 112°C. and having a specific gravity of 1.69. It was first prepared by Stenhouse from picric acid and bleaching powder. Its first use was in chemical warfare by the Allied troops in 1918. It was prepared in large quantities at Edgewood Arsenal during the World War, by the method developed and patented by the senior author.¹ Picric acid, hydrated lime, and water were mixed to form a calcium picrate suspension. This suspension together with a suspension of bleaching powder and water was run into a still. The chloropicrin was steam-distilled off.

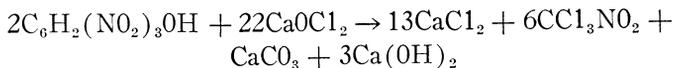
Chloropicrin is used extensively as an insecticide, mainly in warehouses and grain elevators. It has the advantages of high toxicity, practically complete freedom from fire and explosion hazards, relative non-reactivity with metals, and ability to penetrate bulk commodities. It is also toxic to rodents. In rodent control it is only necessary to apply at the mouth of the holes or burrows, the heavy gas sinking down and filling the openings.

Chloropicrin is available on the market in steel cylinders at a cost of about one dollar a pound. In this form it is not only expensive but has to be handled carefully to avoid accidental poisoning. In spite of the high cost increasing amounts of chloropicrin are being used every year by small gardeners, fruit growers, nursery men, and grain millers.

Since chloropicrin may be produced from bleaching powder and picric acid, it was thought that they might be mixed together and this mixture shipped in ordinary moisture proof containers to the consumer. The consumer could then prepare his own chloropicrin from the mixture by merely adding water or allowing the mixture to take up moisture from the air much as is done with calcium cyanide. An alternative method which might be more satisfactory, especially for large users, would be for the consumer to buy bleaching powder and powdered picric acid and mix them as needed.

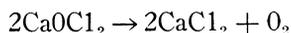
¹ Sweeney, O. R. *Process of Making Chloropicrin*. U. S. Pat. 1,413,198, April 18, 1922.

For soil sterilization it was thought that it would only be necessary to plow the mixture into the soil allowing the natural moisture of the soil to cause the reaction to proceed. The reaction would be as follows:



Chloropicrin produced in that manner should cost considerably less than in cylinders and would be easier and safer to handle.

Studies on the production and utilization of chloropicrin from dry mixtures of bleaching powder and picric acid were begun in the Chemical Engineering laboratory at Iowa State College. The two materials were ground together in small amounts in a porcelain mortar so as to secure a uniform and intimate mixture. One mixture ignited spontaneously. This was believed to have been the burning of the picric acid resulting from the elevated temperature caused by the friction of grinding and the presence of oxygen evolved from the bleaching powder. It is known that in the presence of iron bleaching powder may liberate oxygen according to the reaction



The bleaching powder which was being used, was known to contain iron. It was found that by mixing the picric acid into the bleaching powder a little at a time and grinding thoroughly between each addition that the mixing could be carried on without ignition. In this way the heat produced in grinding and mixing was not allowed to build up to the ignition temperature. In all of the work the picric acid used contained 10 per cent water.

Six parts by weight of bleaching powder to one of picric acid or approximately the theoretical proportions were used. It was found that chloropicrin was evolved slowly from an intimate mixture of the two solids without the addition of water. For example, when cockroaches were placed in a beaker above the dry mixture enough chloropicrin was produced to kill the cockroaches in less than one minute. Dry powder was scattered in the crack of a wooden platform in which cockroaches were very abundant. A day later all were gone except those that had died before they could get away. When water was added the chloropicrin was evolved more rapidly, the rate depending upon the temperature of the water. When warm water was added to a pile of the mixture, a small explosion occurred. When the mixture was spread out the addition of warm water merely produced a rapid evolution of chloropicrin. An office

was successfully rid of cocroaches by placing the dry mixture about in watchglasses and adding a small amount of cold water to each. The evolution of chloropicrin began rapidly.

To determine the effect of the dry mixture when applied to soil, studies were carried out in cooperation with Dr. I. E. Melhus, Head of the Botany Department. One half gram of the bleaching powder-picric acid mixture was added to each of two pots containing ten pounds of field soil. The mixture was distributed as evenly as possible in the upper one inch of depth of soil. Moisture was then added and the pot covered with a newspaper. Two days later 50 alfalfa seeds were planted in each pot. The same number of seeds were also planted in two pots of untreated soil and two of steam sterilized soil. The average germination in the untreated soil was 55 per cent; in the steam sterilized soil, 62 per cent; and in the chloropicrin treated soil 72 per cent. From observations made in connection with these studies it is believed that better results can be secured by placing all of the mixture in a single hole in dirt in each pot rather than to mix it as was done. It was also apparent that considerable chloropicrin escaped, the newspapers not forming a sufficiently tight cover.

The work to date indicates that chloropicrin may be produced and effectively used where needed from a dry mixture of bleaching powder and picric acid. The cost of the materials for producing chloropicrin in this manner is as follows:

1 pound picric acid	\$0.35
6 pounds bleaching powder	0.12
Total	<u>\$0.47</u>

This produces 2 pounds of chloropicrin, making the cost approximately 24 cents a pound as compared with the commercial price of one dollar a pound.

CHEMICAL ENGINEERING DEPARTMENT,
IOWA STATE COLLEGE,
AMES, IOWA.