

UNITED STATES PATENT OFFICE.

CHAUNCEY C. LOOMIS, OF SYRACUSE, NEW YORK, ASSIGNOR TO SEMET-SOLVAY COMPANY, OF SOLVAY, NEW YORK, A CORPORATION OF NEW YORK.

PROCESS OF CHLORINATING TOLUENE.

1,280,612.

Specification of Letters Patent.

Patented Oct. 1, 1918.

No Drawing.

Application filed October 18, 1917. Serial No. 197,205.

To all whom it may concern:

Be it known that I, CHAUNCEY C. LOOMIS, a citizen of the United States, residing at Syracuse, in the county of Onondaga and State of New York, have invented a new and Improved Process of Chlorinating Toluene, of which the following is a specification.

My invention relates to the chlorination of toluene with consequent production of benzyl chlorid and its related products, benzal chlorid and benzo trichlorid. This has heretofore been accomplished by passing chlorin gas through boiling toluene and also by admitting to a mixture of toluene and bleaching powder an acid gas to decompose the bleach with liberation of nascent chlorin.

Both these processes are subject to objections, the former because of the difficulties incident to handling the chlorin and the production of by-products of little value which are not readily separable from the product desired and the latter by reason of the inconvenience of generating and applying the acid gas and the gumming up caused by its reaction with the bleaching powder in the toluene. I have discovered, however, and my invention broadly stated consists in the application of the fact, that by heating to a high temperature an anhydrous mixture of toluene and bleaching powder, with thorough commingling, and without the use of any acid, the chlorination of the toluene is effected without any of the disadvantages incident to the processes above referred to. In the absence of any added water the chlorin from the bleaching powder seems to go directly to produce benzyl chlorid in the side chain without attacking the nucleus, even though the process is carried on in the presence of iron, as in an iron vessel.

In carrying my invention into effect, dry bleaching powder (calcium hypochlorite, or other equivalent hypochlorite,) and toluene are mixed in proportions varying according to the extent of chlorination desired. For example, 200 kilograms of toluene are heated, as in a steam jacketed iron mixing kettle to a high temperature, say 90° C. Dry bleaching powder is then slowly added to this, the temperature being gradually raised until it approximates the boiling point of

toluene, which, of course, cannot be exceeded. When 200 kilograms of bleaching powder have been added the mixture is held at the high temperature attained, (*i. e.*, to secure the best results, from 100° C. to 105° C.) for about one hour, or, until the reaction is effected, with continuous thorough mixing. The escape of toluene vapor during the operation may be prevented by means of a reflux condenser fitted to the mixing vessel.

The resulting mixture is then cooled down and allowed to settle and as much oil as possible is siphoned off from the top. The oil left adhering to the line residue is then removed by steam distillation and added to that siphoned off. In this way 200 to 210 kilos of oil are obtained, having a specific gravity of .940 to .960 at 20° C. and containing from 30 to 35 per cent. of benzyl chlorid and from 70 to 65 per cent. of toluene. These can then be separated by fractional distillation. A higher percentage of chlorinated product can be obtained by using a greater proportion of the bleaching powder. In this case, however, some benzal chlorid and benzo trichlorid will be formed. Or, if desired, sufficient bleaching powder may be added to convert all the toluene directly to benzal chlorid and benzo-trichlorid and the resulting mixture of lime and the chlorids can be converted into benzaldehyde and benzoic acid by boiling with water.

Having thus described my invention, what I claim as new and desire to secure by Letters Patent is:

1. The process of chlorinating toluene which consists in heating together an anhydrous mixture of toluene and bleaching powder without other admixture.

2. The process of chlorinating toluene which consists in heating together toluene and dry bleaching powder in the absence of acid.

3. The process of chlorinating toluene which consists in heating together toluene and dry bleaching powder in the absence of acid or other agent for decomposing the bleaching powder.

In testimony whereof I have affixed my signature, this 15 day of October, 1917.

CHAUNCEY C. LOOMIS.