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1,3 Diacetone Dicarboxylic Acid Detection Method

In a 250ml three-necked flask, add 50 grams (0.5 mol) of concentrated hydrochloric acid (chemically pure, content 36.5%), stir thoroughly, heat up to 30 °C, drop 42.9 grams (0.436 mol) of epichlorohydrin, add dropwise for about 45 minutes. After completion, the reaction is incubated for another 2h. After the reaction is completed, stand for separation and separate the organic phase into 1,3-dichloropropanol, which is dehydrated with anhydrous sodium carbonate at room temperature.

In a 250ml three-necked flask, add 138.8g of 30% sodium cyanide aqueous solution (0.85mol), warm up to reflux, slowly add the organic phase of the step reaction, about 1h to complete the dropwise addition, incubate the reaction for 2h, cool to room temperature, divide. The organic phase is 1,3-dinitrile propanol, and it is washed with 30ml*3 water. Chemicalbook.

In a 250ml three-necked flask, add the 1,3-dinitrile propanol obtained in the previous step, 111.1g of 75% sulfuric acid, and 50ml of xylene. Heat at 135-140°C and reflux for 24h, then cool to room temperature and add 50ml of water to precipitate crystals as 3-Hydroxyglutaric acid. In a 250ml three-necked flask, add 100ml of an aqueous solution containing 38.7g (0.39mol) of chromium trioxide, cool to 20°C, then add the 3-hydroxyglutaric acid collected in the previous step, stir well, and slowly add dropwise containing 77.4g of concentrated sulfuric acid. The aqueous solution of sulfuric acid was 105 grams, and the dripping was completed in about 5 hours, and the reaction was kept warm after stirring for 3 hours. It was extracted with ethyl acetate 100ml*5, and the white needle-like solid of acetone dicarboxylic acid was obtained by removing the solvent.