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A CONVENIENT METHOD OF N-METHYLPHTHALIMIDE SYNTHESIS

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Abstract: We suggest a convenient method to obtain N-methylphthalimide with a high yield in the reaction of phthalic anhydride with aqueous methylamine.

Nowadays N-methylphthalimide is produced in several ways, but two of them are the most widespread. First, potassium phthalimide or phthalimide with potassium carbonate is alkylated by methyl iodide (77% yield) [1] or by methyl ester of p-toluenesulphonic acid (90% yield) [2]. The second method does not require such expensive reagents, since it is based on the reaction between phthalic anhydride and gaseous [3] or aqueous [4,5] methylamine. This reaction encompasses two steps: the opening of phthalic anhydride ring by methylamine and then the closing of the phthalimide ring.

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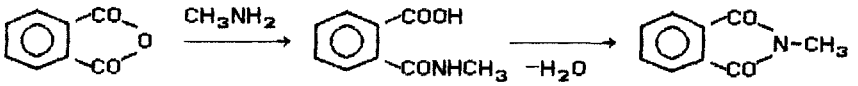

Since the latter step is reversible, the product yield depends on the degree of water removal from the reaction zone. Two ways are used for this perpose [3,4,5]. When phthalic anhydride interacts with aqueous solution of methylamine, is fist evaporated and then the residue is either distilled over at 286 °C (yield is not mentioned) [4] or sublimated (44% yield) [5]. For gaseous $MeNH_2$ the reaction is carried out in a flow installation at 300 °C (72% yield) [3].

In this paper we suggest a simple and convenient method to produce methylphthalimide, which is feasible for both laboratory and pilot plants.

Toluene (450 ml) and 33% aq $MeNH_2$ (146 ml, 1.32 mol) are added to 133.2 g phthalic anhydride [pure substance content is 97.8% (0.88 mol)]. The mixture is refluxed with a Din-Stark attachment untill the water contained in $MeNH_2$ solution is removed. Removal of water formed by cyclization reaction is not recommended, otherwise the salt of phthalic acid and methylamine resides, causing the vigorous effervescence of reaction mixture. Toluene-water azeotrop distilling should be carried out for three hours as minimum, otherwise the yield decreases.

After the reaction finished, organic and aqueous parts are separated, the former is cooled to room temperature and, in a 3 to 4 hours, the residue is filtrated. After drying one gets 131.9 g methylphthalimide with the pure substance content 100% (GLC). M. p. 133-134 °C (133.5-134 °C in ref. [6]). 93% yield.

The filtrate obtained from methylphthalimide distillation has been used 7 times without any yield decreasing and product quality deterioration.

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