

UNITED STATES PATENT OFFICE

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STABILIZATION OF TETRAHYDROFURAN

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This invention relates to the stabilization of heterocyclic organic compounds and in particular it relates to the stabilization of tetrahydrofuran.

Tetrahydrofuran is a colorless liquid which is a potentially important and valuable compound for the preparation of a wide variety of organic compounds. An obstacle to its extensive use is the fact that it forms peroxides during storage. Although freshly distilled tetrahydrofuran sometimes contains small concentrations of peroxide, in the order of a few hundredths of one per cent, which are relatively harmless, upon standing for a comparatively short period, the peroxide concentration rapidly increases to the point where the material becomes unsafe to handle. Peroxide formation in tetrahydrofuran not only introduces an undesirable impurity but also results in a hazardous condition since the peroxide formed may decompose with explosive violence when relatively high concentrations are present particularly when the material is distilled or heated. No method has heretofore been available for stabilizing tetrahydrofuran.

It is an object of this invention to provide a novel method for the stabilization of tetrahydrofuran. Another object is to inhibit peroxide formation in tetrahydrofuran. A further object is to provide a new stabilizer for tetrahydrofuran. An additional object is to provide a relatively stable tetrahydrofuran composition. These and other objects will be apparent from the following description of my invention.

The above objects are attained in accordance with my invention by incorporating into tetrahydrofuran p-cresol.

I have discovered that when small amounts of p-cresol are incorporated into tetrahydrofuran peroxide formation is substantially inhibited and a relatively stable composition results. Tetrahydrofuran stabilized in accordance with my invention may be stored over relatively long periods without the development of hazardous concentrations of peroxide.

Various concentrations of p-cresol may be utilized in the practice of my invention although I prefer to utilize concentrations within the range 0.05 to 1.0% by weight. Concentrations above 1.0% may be used successfully but in general amounts greater than 1.0% are unnecessary for satisfactory results. Less than 0.05% of p-cresol may be utilized but for consistently satisfactory results concentrations of more than 0.05% are desirable.

The following example illustrates my invention.

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Example

A glass-stoppered clear glass bottle was half filled with freshly distilled anhydrous tetrahydrofuran containing 0.1% p-cresol. The bottle containing the tetrahydrofuran and p-cresol was stored in diffused light and an analysis of the peroxide content was made from time to time. The analysis for peroxide content was made by pipetting a 10 cc. sample of tetrahydrofuran into an excess of potassium iodide in dilute sulfuric acid solution and allowing the sample to stand in the dark during 10 minutes. The liberated iodine was then determined by titration with a standard sodium thiosulfate solution. The results are shown in tabular form below:

Time in Days	Cc. ¹ 0.0893 N Thiosulfate/10 cc. Sample
Start.....	0.52
21.....	1.00
31.....	0.98
78.....	2.52
103.....	3.04
122.....	3.44

A sample of tetrahydrofuran containing no p-cresol was tested as a control with the following results:

Time in Days	Cc. ¹ 0.0895 N Thiosulfate/10 cc. Sample
Start.....	0.63
3.....	4.40
7.....	6.00
14.....	8.94

¹ One cc. of this solution/10 cc. sample of tetrahydrofuran is equivalent to approximately 0.05% peroxide when it is assumed that the peroxide contains one mole of tetrahydrofuran.

The foregoing data conclusively demonstrate the inhibiting effect of p-cresol on peroxide formation in tetrahydrofuran. Within 14 days, the unstabilized sample contained a higher concentration of peroxide than the stabilized sample developed in 122 days.

My invention provides a simple and economical means for stabilizing tetrahydrofuran. Not only may the stabilized material be stored over relatively long periods or shipped long distances without appreciable deterioration but also the hazard inherent in the presence of peroxides in the tetrahydrofuran is held to a minimum.

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I claim:

1. The method for stabilizing tetrahydrofuran which comprises incorporating therein 0.05 to 1.0% of p-cresol.

2. A new stable tetrahydrofuran composition comprising tetrahydrofuran having incorporated therein 0.05 to 1.0% of p-cresol.

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