

N,N'-dipropyl, *N,N'*-bis(4-methyl benzene sulfonyl) hydrazide: a new radical source for chain polymerization of vinyl monomers

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Abstract

A new radical initiator *N,N'*-dipropyl, *N,N'*-bis(4-methyl benzene sulfonyl) hydrazide (DBSH) has been prepared by condensation of *N*-chloro and sodium derivatives of *N*-propyl,4-methyl benzene sulfonamide at room temperature. As a radical initiator DBSH shows interesting properties that, although homolysis of the *N–N* band takes place around 52.5°C, only very low polymerization yields (~1.0 %) are obtained below 100°C. At 100°C, bulk polymerization of methyl methacrylate proceeds rapidly and the reaction is almost quantitative in 2.5 h. This can be attributed to stability of the sulfamidyl radicals to some extent. Radical initiation efficiency of DBSH is about 0.16 and gives high molecular weight of polymers at that temperature. Presence of sulfamidyl groups in polymer chain ends have been confirmed by NMR and elementary analysis.

In the study, for radical initiation ability of DBSH in bulk polymerization of methyl methacrylate and styrene monomers has been investigated in various time, temperature conditions. © 2001 Elsevier Science Ltd. All rights reserved.

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1. Introduction

Radical generation based on thermal splitting of relatively weak covalent bonds is one of the most common way of radical initiation in vinyl polymerizations. Although many diacyl or dialkyl peroxides and azo compounds have been studied, dibenzoyl peroxide and azo-isobutyronitrile have found common acceptance in practice. Search for new organic radical sources for controlled polymerizations is being current interest by polymer chemists. Disulfides [1] and nitroxides [2] have

found much interest as metal-free organic radical sources for controlling polymerizations. Although decomposition of aryl sulfonyl hydrazides yielding nitrogen evolution have been reported [3] there appears only one report dealing with homolytic cleavage of disulphonyl hydrazides yielding sulfamidyl radicals [4]. However, polymerization by sulfamidyl radicals has not been reported yet, presumably due to high bond breaking energy of *N–N* bonds [5].

We thought that *N*-alkylated sulfonyl hydrazides might be efficient radical sources in combination with suitable activators. Before doing so, in the present study *N,N'*-dipropyl, *N,N'*-bis(4-methyl benzene sulfonyl) hydrazide (DBSH) has been synthesized and its thermal initiation ability has been tested in bulk polymerization of methyl methacrylate and styrene.

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