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Short communication

# Photochromic properties of a vacuum-deposited film of (*E*)-dicyclopropylmethylene-(2,5-dimethyl-3-furylethyldiene)-succinic anhydride

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## Abstract

A yellow-orange film was prepared by vacuum deposition technique under a pressure of  $1 \times 10^{-4}$  Pa and a boat temperature of  $300^\circ\text{C}$ . Upon irradiation with UV light (366 nm), the yellow-orange film was turned violet, the later color was switched back to the original pail yellow color. When the film was irradiated with light posses a wavelength more than 500 nm. The coloration process was lost when the film was annealed at 80, 100 and  $120^\circ\text{C}$ . The rate constant of the coloration process was determined to be  $9.8 \times 10^{-4} \text{ s}^{-1}$ . © 2001 Elsevier Science B.V. All rights reserved.

**Keywords:** Fulgides; Photochromic; Photochromism; Thin film; Thermal evaporation

## 1. Introduction

Work on organic photochromic compounds has been animated because of their high potential as a data-storage media. Photochromic spirobenzopyran derivatives have been studied in depth [1–4]. Photochromic “fulgides” which are derivatives of 2,3-bis(methylene)succinic anhydride with at least one aryl group. Heller [5,6,8] and Heller and Langan [7] has promoted the study of photochromic fulgides since three decades. He showed that fulgides containing heterocyclic structure, such as furyl, thienyl and pyrrolyl, were excellent candidates for data storage media because of their efficient thermal stability. The application of photochromic compounds in optical data storage or any other application will certainly be provided as thin films. It is therefore very important to study the photochromic performance of fulgides thin films. We here report the photochromic properties of vacuum-deposited film of fulgide (**1-E**).

## 2. Experimental

Fulgide **1** was prepared according to general procedure previously reported [9]. The films were prepared on rectangular optically flat quartz substrates at temperature of

$300^\circ\text{C}$ . The pressure of vacuum during the entire evaporating process was above  $10^{-4}$  Pa.

## 3. Film annealing

Fig. 1 showed the absorption spectra of the film before and after annealing the film for 2 h at 80, 100 and  $120^\circ\text{C}$ . When a new film was annealed at temperature below the melting points (mp  $150^\circ\text{C}$ ), for instance 80, 100 and  $120^\circ\text{C}$  for 2 h the photochromic properties was lost, and the film was turned from pail orange to faint red. Comparing the IR spectrum of the thin film before annealing with that after the annealing showed that the fulgide thin film does not decompose. We believed that the fulgide **1-E** at the annealing temperature isomerizes to the *Z*-isomer **1-Z** and then the crystal packing prohibited the isomerization of the later to the *E*-form of **1-E** and hence to the colored form of **1-C**. Changing the annealing temperature does not show a great difference in the absorption spectra of annealed film except a slight decrease in the intensity.

## 4. Result and discussion

The selection of the title ‘fulgide’ was based on the fulfillment of the following properties: (a) photochemical

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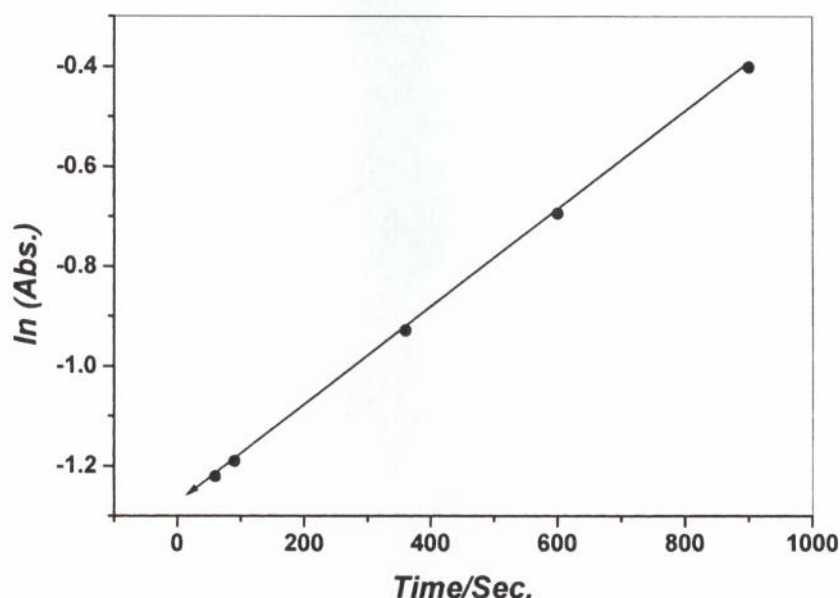


Fig. 3. First-order rate plot of the coloration reaction monitored at 500 nm.

stability allowing for repetitive cycles without the loss of optical properties, e.g. fatigue resistant, (b) its photoreaction must not be thermally reversible at the temperature of the experiment, (c) the photoreaction must proceed with reasonable photochemical yield, and (d) the two forms must absorb in different spectral regions, preferably where convenient light sources exist. The open form of fulgide **1** can exist in the two geometrical isomers **1-Z** and **1-E**. Upon irradiation of **1-Z** it isomerizes to **1-E** first and then cyclized to the colored form **1-C** (Scheme 1). Fig. 2 showed the spectrum of fulgide **1** before irradiation and after the irradiation of the film at intervals of time. The pure fulgide **1-E** possesses an absorption in the UV region and upon irradiation with mercury lamp (using a filter allowing only light with wavelength of 366 nm) the pail yellow film turned pink and a new absorption band started to develop as a result of the formation of the 7,7a-dihydro-7,7-dicyclopropyl-2,4,7a-trimethylbenzo[*b*]-furan-5,6-dicarboxylic anhydride **1-C**. This photochromic processes of fulgides have been studied and well documented [5–8]. In contrast to the same phenomenon observed for the same fulgide, which showed a near quantitative conversion of the open form **1-E** to the closed form **1-C** in toluene, the conversion in the film is only about 40%.

This was believed to be due to the crystal arrangement in the film, which hindered the UV light from reaching the inner part of the film. The coloring and bleaching of the film can be repeated for more than hundred times. The photocyclization process was found to obey a first order rate equation (Fig. 3) and the rate constant for the process was determined to be  $9.8 \times 10^{-4} \text{ s}^{-1}$ .

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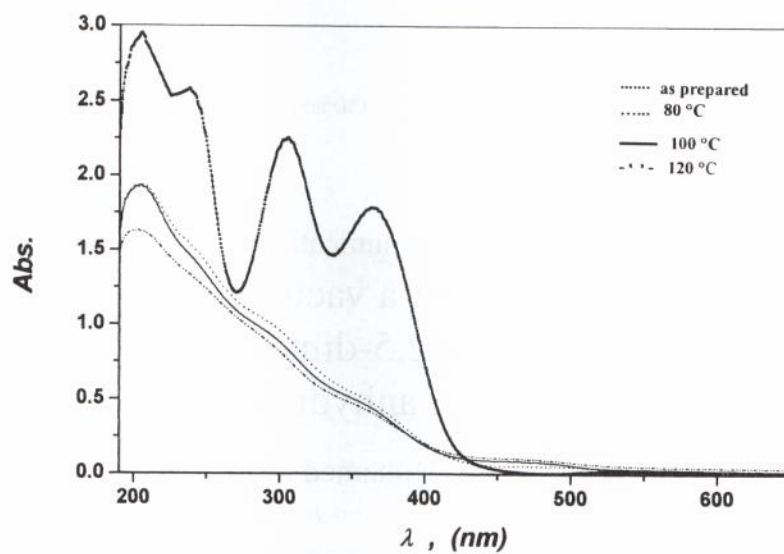
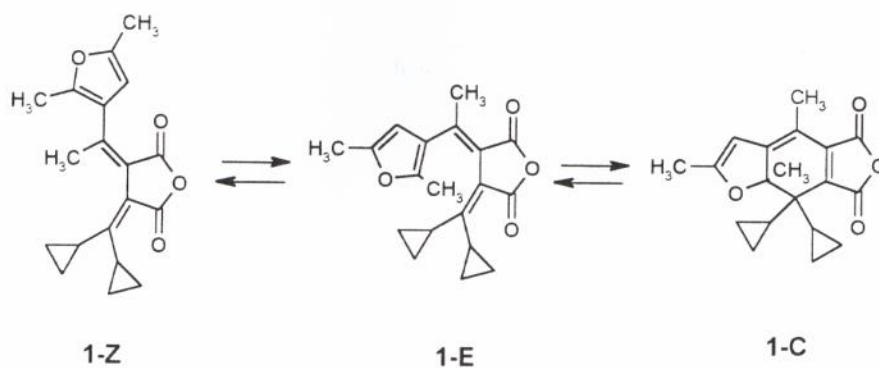


Fig. 1. The effect of annealing on thin film of fulgide 1-E.



Scheme 1.

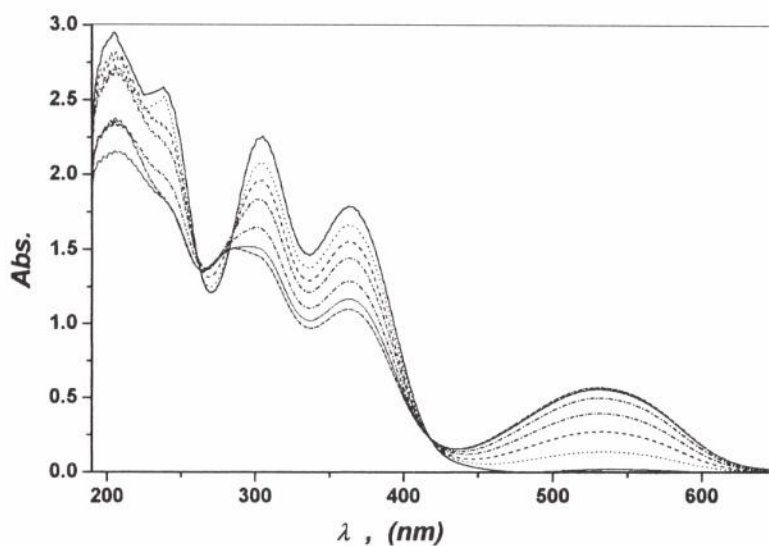


Fig. 2. The electronic absorption spectra of thin film of Fulgide 1-E before and after irradiation at 366 nm for intervals of 5 min.