

Synthesis and photographic properties of novel development-accelerator-releasing couplers

Yuting Liu^{a,b,*}, Feng Lv^a, Jing Zou^c, Dade Zhang^a, Zuguang Yao^a

^a*Institute of Fine Chemicals, East China University of Science and Technology, Shanghai 200237, PR China*

^b*College of Chemistry and Chemical Engineering, Shaanxi University and Technology, Xianyang 712081, PR China*

^c*China Luckyfilm Cooperation, Baoding 071054, PR China*

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Abstract

Several novel development-accelerator-releasing couplers (DAR couplers) were synthesized. The structure of DAR couplers was confirmed by MS, IR and ¹H NMR spectroscopy. At the same time, the effect of DAR couplers derived from different hydrazines on the photographic properties of color negative material were studied. It was found that the DAR couplers examined may be used together with cyan coupler or yellow coupler in a color negative material to increase the photosensitivity; DAR couplers 4c and 8c that contained 1-trifluoroacetyl-2-(4-aminophenyl)hydrazine increased photosensitivity the most.

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1. Introduction

Hydrazine derivatives can be used to increase the photosensitivity and contrast of a silver halide light-sensitive material during development [1–3]. A hydrazine derivative may be introduced, as a functional group, at the active position of a color coupler, and the resulting compound is termed a development-accelerator-releasing (DAR) color coupler [4–6]. The inclusion of a DAR color coupler in a color negative imaging layer has been suggested as a means of increasing photosensitivity and photo efficiency in the color development process. A detailed photomicrographic

study has revealed that the action of a DAR color coupler involves a localized fogging of unexposed grains in close proximity to a strongly developing grain, and hence increases the number of developed silver halide grains per unit area [7]. The DAR coupler can be represented by the general formal Cp–L–A in which “Cp” represents a coupler residue (cyan, magenta or yellow), “A” represents a development accelerating functional group and “L” represents a di-valent linking group. In the present paper, a class of novel DAR couplers—three cyan DAR couplers and three yellow DAR couplers were synthesized and their photographic properties on color negative material were studied. It was found that all DAR couplers could increase photosensitivity, but the 4c and 8c DAR couplers that contained 1-trifluoroacetyl-2-(4-aminophenyl)-hydrazine compounds (4c and 8c) as development accelerators were the best and increased photosensitivity the most.

* Corresponding author. Institute of Fine Chemicals, East China University of Science and Technology, Shanghai 200237, PR China. Fax: +86 21 64252840.

E-mail address: yutingliu318@sohu.com (Y. Liu).

2. Experimental

2.1. Synthesis of DAR couplers

2.1.1. General

Mass spectra have been obtained with a HITACHI M-80 spectrometer, IR spectra with a NICOLET FT-IR 20sx spectrometer and ^1H NMR spectra with a BRUKER ADVANCE 500 spectrometer, model WP-500SY; TMS was the initial standard used. All melting points reported are uncorrected.

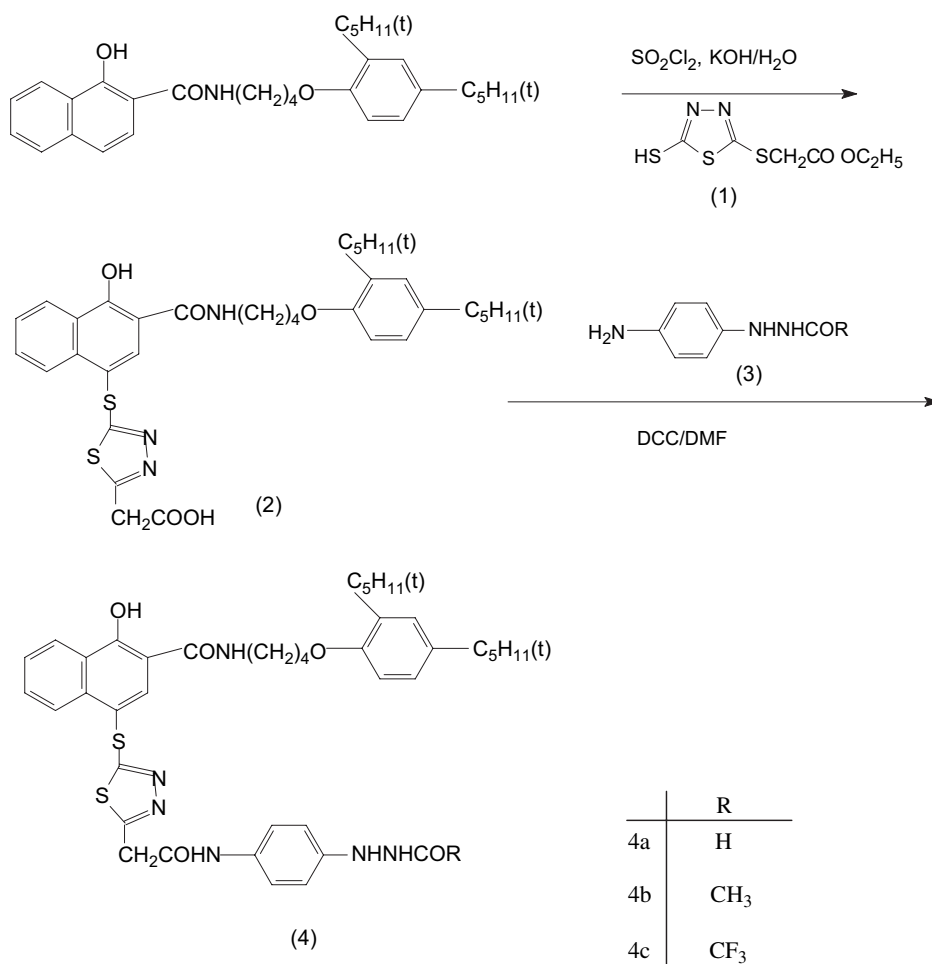
2,5-Dimercapto-1,3,4-thiadiazole [8], 2-ethoxycarbonylmethylthio-5-mercapto-1,3,4-thiadiazole [9], 2-(carboxymethylene)thio-5-mercapto-1,3,4-thiadiazole [9], 5%Pd–C catalyst [10], 1-formyl-2-(4-aminophenyl)hydrazine (3a) [5,11], 1-acetyl-2-(4-aminophenyl)hydrazine (3b) [5,12] were prepared according to the literature procedures.

The DAR couplers can be synthesized by various routes and typical syntheses are illustrated in Schemes 1 and 2.

2.1.2. Synthesis of 1-trifluoroacetyl-2-(4-aminophenyl)hydrazine (3c)

p-Nitrophenylhydrazine (0.3 mol) was dissolved in acetonitrile (160 ml). A solution of trifluoroacetic anhydride (0.6 mol) in acetonitrile (20 ml) was added dropwise over 20 min at room temperature, the mixture refluxed with stirring for 2 h, cooled to 5 °C, the solution was filtered and solids were washed with acetonitrile and recrystallized to give 1-trifluoroacetyl-2-(4-nitrophenyl)-hydrazine as yellow solids. Yield: 98.6%.

To 10 g 1-trifluoroacetyl-2-(4-nitrophenyl)-hydrazine was added 1 g of 5%Pd–C catalyst and 250 ml of ethanol and the mixture was subjected to catalytic hydrogenation in an autoclave. After removing the catalyst by filtration, the filtrate was cooled and the precipitated crystals were collected by filtration to obtain 3c as white solids. Yield, melting point and MS data are shown in Table 1.



Scheme 1. The route of cyan DAR couplers were synthesized.

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