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Novel Perylene Derivatives as Highly Photostable Fluorescent Dyes

Heinz Langhals*

Abstract. Perylene-3,4-dicarboxylic imides are prepared by a new decarboxylizing condensation with moderately sterically hindered primary amines. Perylene-3,4-dicarboxylic anhydride is prepared *via* a saponification reaction of the dicarboximide and is a starting material for a number of new types of chromophores. A second route to novel perylene derivatives is a partial hydrolysis of perylene dyes and a condensation with diamines. The dyes thus obtained are orange to red fluorescing in solution.

1. Introduction

Perylene-3,4:9,10-tetracarboxylic bisanhydride (**1**, C.I. No. 71127) is prepared in an analytically pure state on technical scale and it is the starting material for the preparation of perylene dyes (perylene-3,4:9,10-tetracarboxylic bisimides, **2**) [1]. The perylenes **2** are well-known as highly photostable pigments and fluorescent dyes; for a review see [2]. An essential point for the lightfastness and inertness of **2** is the

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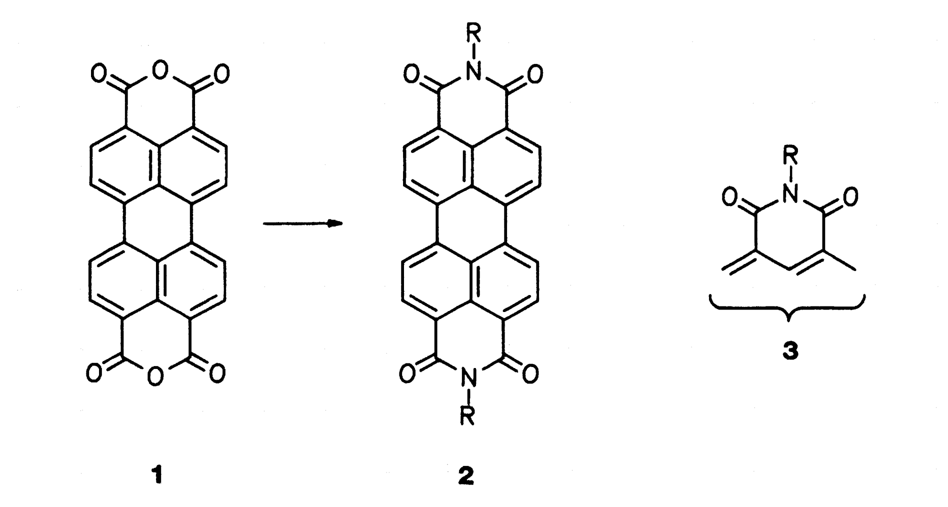
linkage of two units of the strongly resonance stabilized cyclic carboxylic bisimide structure element (**3**) to the perylene skeleton in *peri*-position.

The anhydride **1** is predominantly used for the preparation of **2**, but it is also a valuable starting material for other derivatives with a perylene structure element. This has been reported for the preparation of carboximides with five-membered rings [3], and will be demonstrated for a number novel fluorescent dyes.



1 2 3

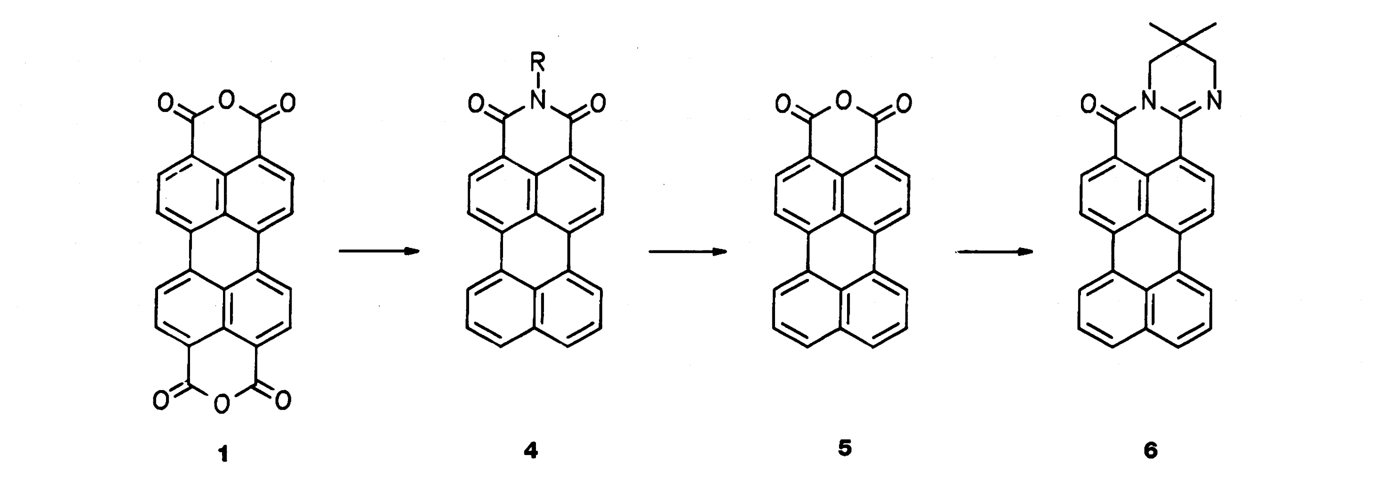
2. Results

2.1. *Perylene-3,4-dicarboxylic Imides* 4

Whereas **1** is a well established class of dyes there are only a few derivatives of the related **4**, because there is no preparation procedure for **5**. However, if moderately sterically hindered primary amines like 2,5-di(*tert*-butyl)aniline are condensed with **1** in presence of H_2O a decarboxylation proceeds with the condensation to the imide **4a** in 50% yield.

The imides **4** are hydrolysed with KOH in *tert*-butyl alcohol to **5**. The anhydride **5** is a general starting material for **4** and other perylene derivatives. The pure anhydride **5** is a red pigment with an intense solid-state fluorescence; the UV/VIS spectra are shown in the *Figure*.

The dyes **4** are extraordinarily photostable: in solution more even than a factor of 20 (!) compared to **2**. This may be a consequence of the stabilizing structure element **3**. The dyes are highly fluorescent. Some derivatives



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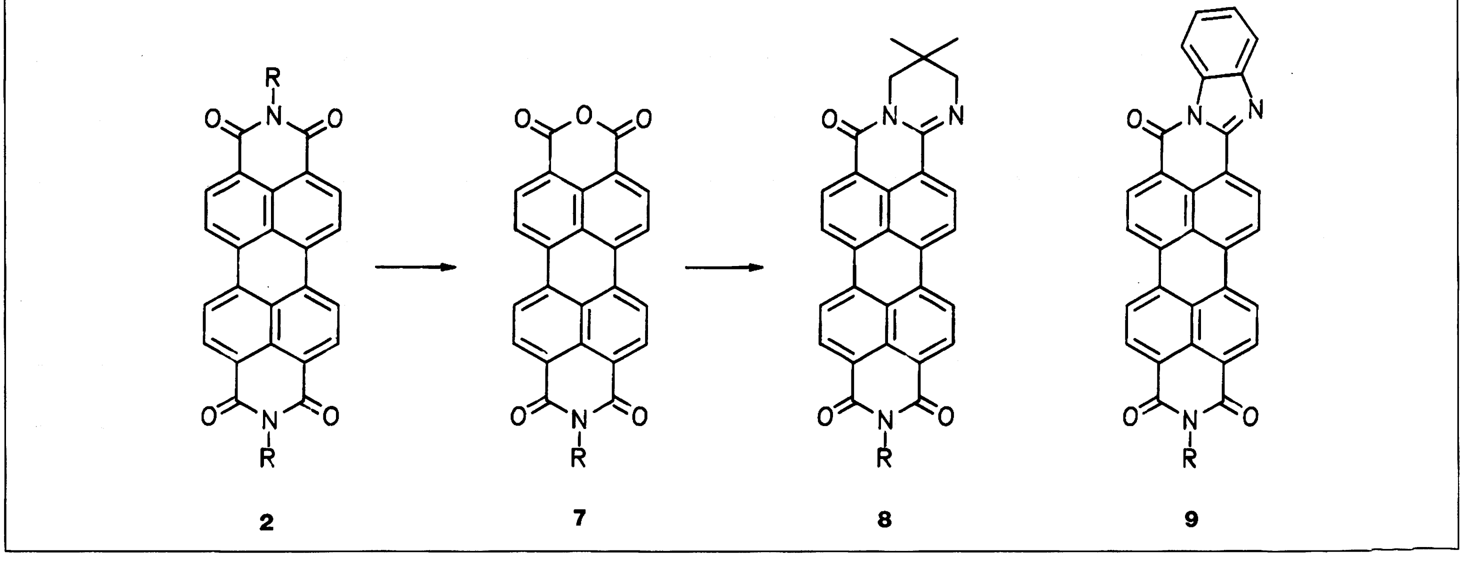

2 7 8 9

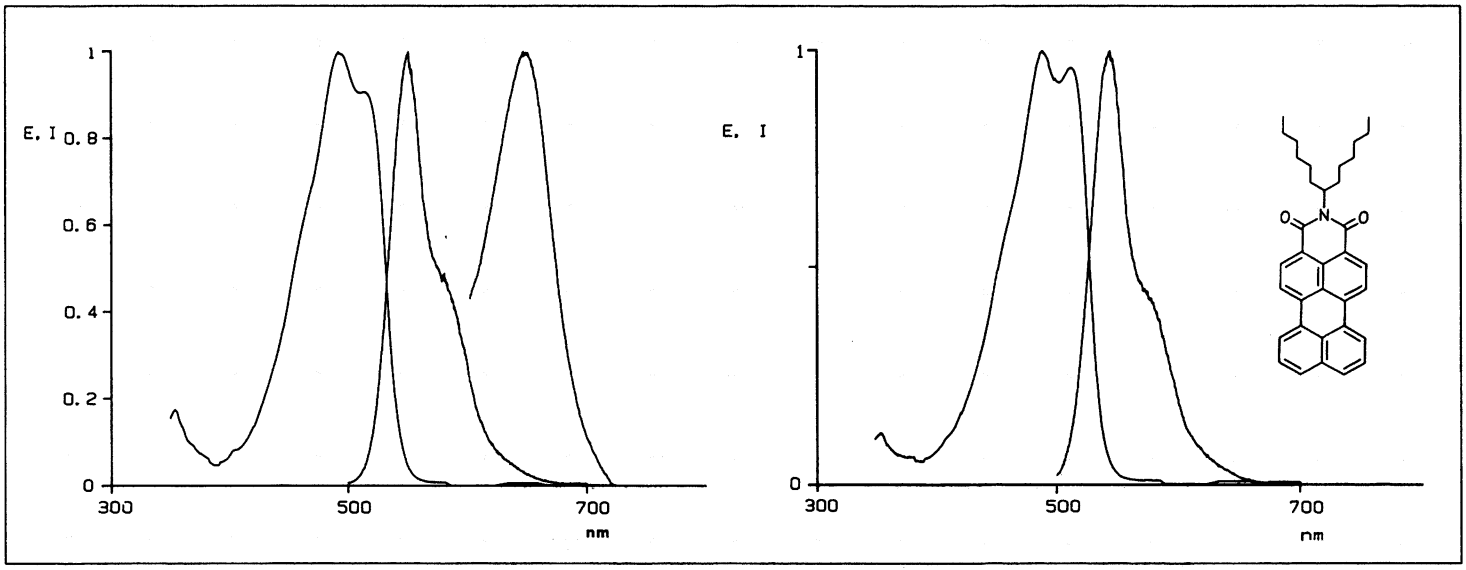

Figure. *UV/VIS Spectra in* $CHCl_3$. *Left*: **5** (absorption, fluorescence and solid-state fluorescence); *right* **4b** (absorption and fluorescence).

of **4** exhibit an intense solid-state fluorescence.

2.2. *Peryleneamidineimides* **8** *or* **9**

Many properties of **2** can be controlled by the substituents R, but not the UV/VIS spectra in solution [4] because of nodes in the orbitals HOMO and LUMO at the N-atoms [5]. On the other hand, little is achieved by altering the basic chromophore of **2**. A possibility of shifting the UV/VIS spectra is an exchange of carbonyl groups of the imide structure **3** by the related imino group. This has been done in a preceding work [6] for one carbonyl group of each imino ring. A bathochromic shift was obtained, but the fluorescence quantum yields went down to 60% or even lower.

A more moderate altering of the chromophore of **2** is the exchange of one single carbonyl group to an imino group [7]. This can be attained by a partial alkaline saponification of **2** to **7** and a condensation of **7** with diamines to amidineimides like **8** or **9**. These are intensely orange to red fluorescing photostabe dyes.

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