Photochemical and Photobiological Sciences

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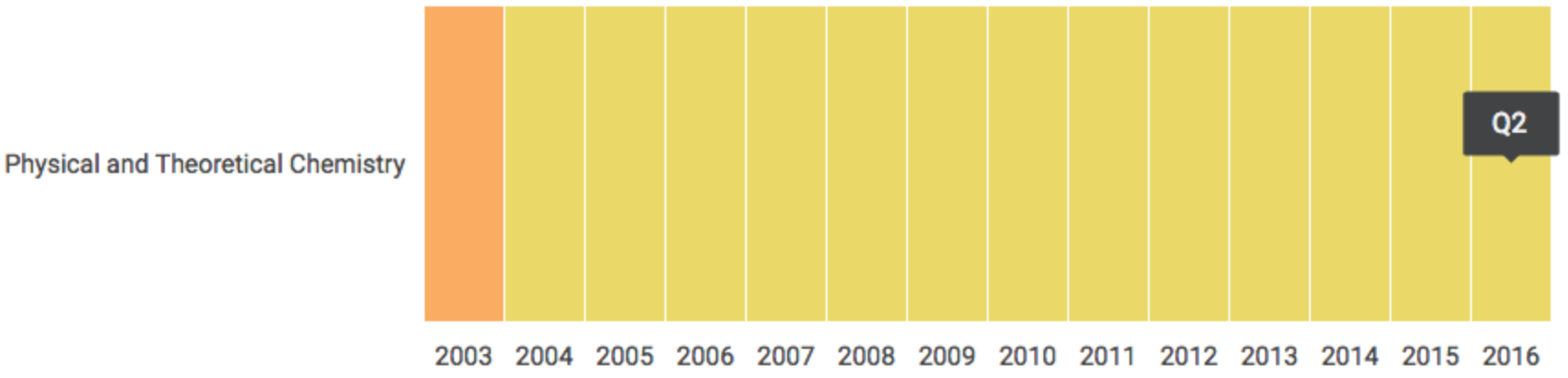

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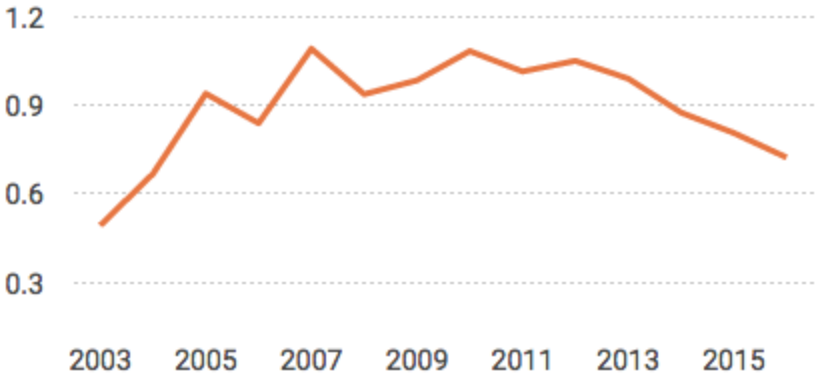

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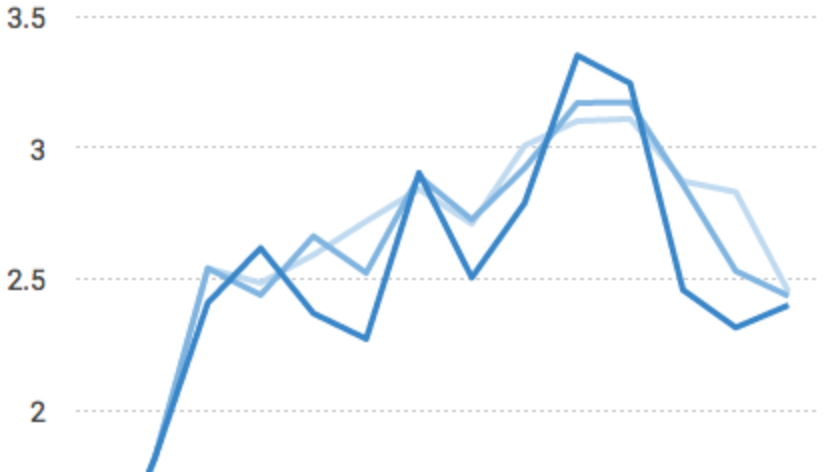

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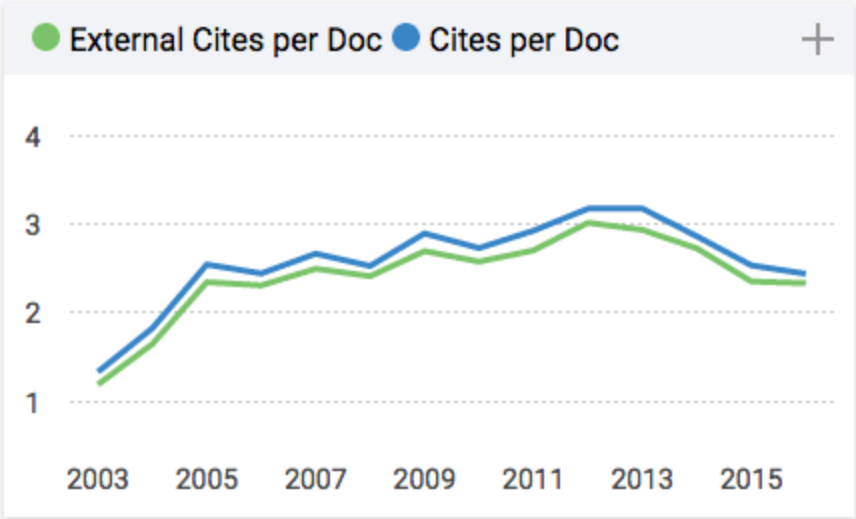
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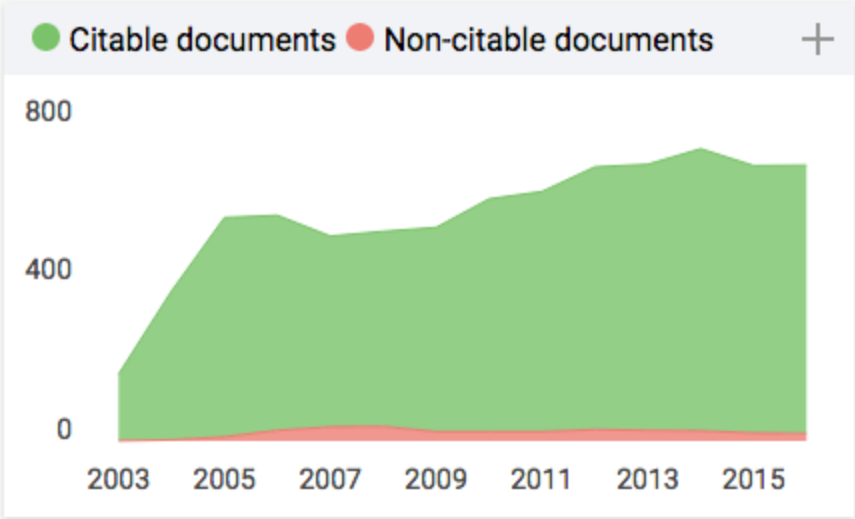

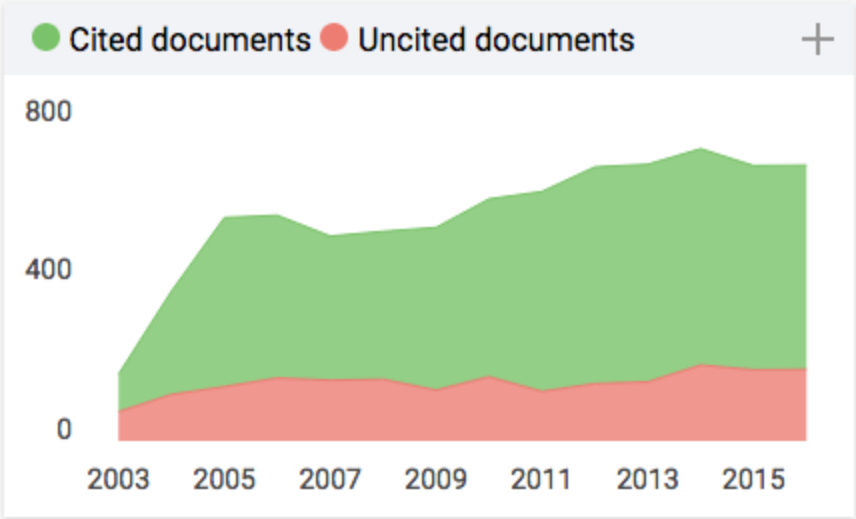

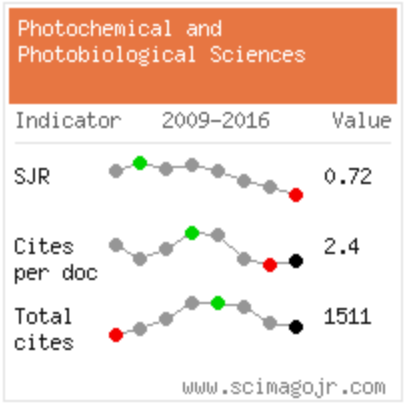

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The absorption spectrum of *cis*-azobenzene†

Ľubica Vetráková, ‡[a] Vít Ladányi, ‡[a] Jamaludin Al Anshori, §‡[a] Pavel Dvořák, [b] Jakob Wirz [c] and Dominik Heger *[a]

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Azobenzene is a prototypical photochromic molecule existing in two isomeric forms, which has numerous photochemical applications that rely on a precise knowledge of the molar absorption coefficients (ε). Careful analysis revealed that the previously reported absorption spectra of the "pure" isomers were in fact mutually contaminated by small amounts of the other isomer. Therefore, the absorption spectra of both *trans*- and *cis*-azobenzene in methanol were re-determined at temperatures of 5–45 °C. The thermodynamically more stable *trans*-azobenzene was prepared by warming the solution in the dark. To obtain the spectrum of *cis*-azobenzene three methods were used, which gave consistent results within the limits of error. The method based on the subtraction of derivative spectra coupled with a global analysis of the spectra recorded during thermal *cis*–*trans* isomerization is shown to give slightly more reliable results than the method using isomeric ratios determined by ^{1}H-NMR. The described methods are readily generalizable to other azobenzene derivatives and to other photochromic systems. The practical implication of the re-determined ε values is demonstrated by a very high precision of spectrophotometric species analysis in azobenzene isomeric mixtures. The new ε values imply that the previously reported quantum yields must be revised.

1 Introduction

The existence of the *cis*-isomer of azobenzene was first reported by Hartley in 1937 in a *Nature* paper entitled: "The *Cis*-form of Azobenzene".[1] The discovery was based on the increased solubility of *cis*-azobenzene in polar solvents compared to that of the *trans*-isomer and its high dipole moment. Although the photochemical and thermal isomerization of azobenzene and its derivatives became textbook examples of the process, the details of the reaction mechanism are still under dispute.[2–8] The reversible photoisomerization reaction (Fig. 1) of azobenzene derivatives has been exploited in numerous applications of photochemistry such as photoswitching to provide effective photocontrol of a large variety of biomolecules,[9,10] molecular receptors,[11] logic units,[12] smart materials[13] and devices,[14] holographic materials,[15] for molecular motion,[16] and last but not least, azobenzene is frequently used as a convenient actinometer to count incident photons. Azobenzene was proposed as an actinometer for monochromatic light sources at wavelengths up to 450 nm by Gauglitz.[17–20] Besides ferrioxalate, azobenzene is arguably the most common and frequently used actinometer,[20] however, sometimes with inconsistent results from different laboratories.[21–24] Accurate knowledge of both the quantum yields and the molar absorption coefficients is a prerequisite for spectrophotometric actinometry.

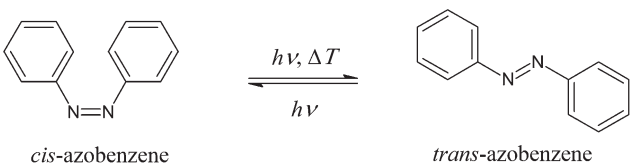

Fig. 1 The photoisomerization of azobenzene.

In an effort to determine photon fluxes by using both the ferrioxalate and azobenzene actinometers we obtained significant discrepancies in the results and we noted a few pitfalls in the recommended procedures of quantum yield determination about which we wish to warn potential users. In this paper, we address the first problem that needs to be resolved: the molar

[a]*Masaryk University, Faculty of Science, Department of Chemistry and RECETOX, Kamenice 5/A8, 625 00 Brno, Czech Republic. E-mail: hegerd@chemi.muni.cz; Tel: +420 54949 3322*

[b]*Masaryk University, Faculty of Science, Department of Physical Electronics, Kotlářská 267/2, 611 37 Brno, Czech Republic*

[c]*University of Basel, Department of Chemistry, Klingelbergstrasse 80, CH-4056 Basel, Switzerland. E-mail: J.Wirz@unibas.ch*

†Electronic supplementary information (ESI) available: The values of the ε_{trans}, ε_{cis}, Gaussian peaks analysis. Minimization procedure for obtaining the concentrations of *trans*- and *cis*-azobenzenes from the absorption spectrum. Fig. S1–S16. See DOI: 10.1039/C7PP00314E

‡These authors contributed equally.

§Present address: Department of Chemistry, Faculty of Mathematics and Natural Sciences, Padjadjaran University, Bandung-Sumedang km.21, West Java 45363, Indonesia.