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Photocontrol of Polar Aromatic Interactions by a bis-Hemithioindigo Based Helical Receptor

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Abstract

The first example of a bis-Hemithioindigo (bis-HTI) based molecular receptor was realized. Its folding and selective binding affinity for aromatic guest molecules can precisely be controlled by visible light and heat. The thermodynamically stable state of the bis-HTI is the s-shaped planar Z,Z-configuration. After irradiation with 420 nm light only the E,Z-configuration is formed in a highly selective photoisomerization. The E,Z-isomer adopts a helical conformation because of the conscious implementation of repulsive sterical interactions. The E,Z-configured helix is able to recognize electron poor aromatic guests exclusively via polar aromatic interactions and also distinguishes between regioisomers. After heating the Z,Z-configuration is completely restored and the aromatic guest molecule is efficiently released.

Main text

Spatial and temporal control of molecular interactions is a central topic for the generation of complex molecular functions similar to the ones found in natural systems. At present great efforts are made in supramolecular,^[1] medicinal,^[2] and biological^[2b, 3] chemistry as well as in the materials sciences^[4] to implement different control elements for the regulation of molecular recognition processes. Using light for this purpose is of special high interest and numerous systems employ photoswitches as external control unit^[1c, 1e, 5] or integral part of their molecular architecture.^[1d, 3g, 6] A fundamental problem of most photoswitches is their dependence on UV light, which has to be used for at least one switching step and leads to severe limitations for instance in biological or materials applications.^[7] Additionally, most molecular interactions that can be altered by light irradiation at the moment are relatively strong and directional in nature, e.g. hydrogen bonding,^[1c, 1e, 8] ionic interactions,^[6c, 9] or metal-ligand bonds.^[10] Weaker and less directed interactions are much harder to control with precision and have therefore not well been studied in the context of photoresponsive recognition.

In the following we present the first example of a molecular receptor that undergoes photoswitching in the visible region of the spectrum and binds guest molecules exclusively by polar aromatic interactions. Release of the guest is conveniently achieved by a simple heating step. To this end we have developed a completely new supramolecular system **1**, which is based on Hemithioindigo (HTI) photoswitches.^[3b, 11] Upon irradiation with visible light receptor **1** changes its conformation from a well defined planar geometry into a well defined folded helix. The helical form is able to bind electron-poor aromatic guest molecules efficiently while the affinity of the planar form is extremely low. Therefore, a reversible capture-release cycle can be realized by alternating irradiation and heating steps interconverting the high and low affinity configurations of **1** in solution.

Bis-HTI **1** was synthesized in a short, convergent, and highly modular fashion (Scheme 1). The central tricyclic bis-thiophenone ring **7** was obtained by twofold intramolecular Friedel-Crafts acylation starting from the already described bis-acid **6**.^[12] The light sensitive compound **7** was not isolated but directly subjected to the condensation reaction with two equivalents of biphenyl aldehyde **5** yielding bis-HTI **1**. Aldehyde **5** was synthesized in two steps via Suzuki cross-coupling from the known precursors **2**^[13] and **4**^[14] (see Supporting Information for more details).



Scheme 1. Synthesis of bis-HTI 1. The thermodynamically stable *Z*,*Z* isomer of 1 is shown.

Bis-HTI **1** can exist in four different isomeric states depending on the particular configuration of its two double bonds: *Z*,*Z*, *E*,*Z*, *Z*,*E*, or *E*,*E*. The *Z*,*Z* configuration is the thermodynamically stable form, which is also obtained in >95% after heating of an isomeric mixture. After irradiation of a toluene solution of *Z*,*Z*-**1** with 420 nm light the *E*,*Z* isomer is obtained in 94% yield (Figure 1). A quantum yield $\Phi_{Z,Z/E,Z}$ of 7.0% was determined for this process. Such an efficient and highly selective photoisomerization in electronically coupled switching systems is usually impossible^[15] and a distinct property of bis-HTI **1**.

The sole presence of the E,Z configuration after irradiation was directly proven by an extensive NMR analysis in solution (see Supporting Information for the complete signal assignment).



b)



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Figure 1. (a) Bis-HTI *Z*,*Z*-1 undergoes highly selective (94%) photoisomerization to the *E*,*Z* isomeric form after irradiation with 420 nm light. Heating restores the initial *Z*,*Z* isomer. *E*,*Z*-1 shows a highly enhanced binding affinity for electron poor aromatic substrates because of its helical preorganization. The binding of 27DDF (8) via polar aromatic interactions can thus effectively be controlled by irradiation and heat. (b) Aromatic region of the ¹H NMR spectra (400 MHz, 300 K, [D₈]toluene) of *Z*,*Z*-1 before irradiation (1), *E*,*Z*-1 after irradiation with 420 nm light (2), *E*,*Z*-1 after irradiation with 420 nm light and addition of 8 (3), and restored *Z*,*Z*-1 in the presence of 8 after heating (4). Signals marked with * are satellites of the solvent.

Especially unambiguous is the cross-peak signal between the proton of the remaining *Z*configured double bond and the closest carbonyl-carbon atom of the bis-thiophenone ring in the HMBC NMR spectrum of *E*,*Z*-1 (Figure 2b and c). The backisomerization to the *Z*,*Z* configuration was achieved by simple heating of the toluene solution. The thermal stability of the metastable *E*,*Z*-1 was quantified by a kinetic analysis of its thermal conversion to the *Z*,*Z* isomer at 60 °C (see Supporting Information, Figure S15). The free activation enthalpy of this process is 28.2 kcal/mol, which leads to a favorably high bistability (a half life of 1.7 years at 25 °C) of receptor 1. After irradiation of *E*,*Z*-1 with 530 nm light only 32% of *Z*,*Z*-1 are obtained in the photostationary state (pss). A quantum yield $\Phi_{E,Z|Z,Z}$ of 0.1% was determined for this process. The reversible switching between the *Z*,*Z* and *E*,*Z* configuration is shown in Figure 1.

Z,*Z*-1 and *E*,*Z*-1 can each adopt four different conformations in which the two biphenyl units are rotated by 180° around the C-C single bonds connecting them to the isomerizable double bonds. Introducing a *n*-pentyl substituent in one of the *ortho*-positions to this single bond leads to a strong preference of only one conformation. In this conformation the *n*-pentyl substituent is positioned at the less hindered side of the double bond to avoid repulsive steric interactions with the bis-thioindigo fragment. The geometry of the preferred conformer could directly be proven by strong cross-peak signals between the olefinic protons of the double bonds and the benzylic protons of the *n*-pentyl substituents in the NOESY solution spectra of *Z*,*Z*-1. NOE signals of the other rotameric conformers were barely visible (see Supporting Information, Figure S5). Because of this negative preorganization the spatial relation of the two terminal aniline groups is precisely defined and *Z*,*Z*-1 adopts a s-shaped geometry in solution. The same geometry is also present in the crystalline state, as is revealed by the crystal structural analysis of *Z*,*Z*-1 (Figure 2a).

The negative preorganization is also effective in E,Z-1, which was again proven by strong cross-peak signals between the olefinic protons of the double bonds and the benzylic protons of the *n*-pentyl substituents in the NOESY spectra of E,Z-1 (Figure 2d). Because of this preorganization E,Z-1 must adopt a helically folded structure to avoid steric clashes between the terminal aniline groups, which are now in close proximity of each other. Strong chemical shifts of the aniline proton signals in the ¹H NMR spectrum of the E,Z isomer are indicative for this close proximity.

In the helical conformation of *E*,*Z*-**1** the terminal electron rich aniline groups should be able to bind electron deficient aromatic guest molecules via polar aromatic interactions in a "sandwich"-like fashion. Addition of 9-dicyanomethylene-2,7-dinitrofluorene (27DDF, **8**) to a solution of *E*,*Z*-**1** in [D₈]toluene did indeed cause pronounced shifts of the aniline-proton signals in the ¹H NMR spectrum. A binding constant of $240\pm30 \text{ L-mol}^{-1}$ was determined by titration for a *E*,*Z*-**1** : **8** = 1 : 1 stoichiometry. To this end the NMR data were fitted using the program Bindfit,^[16] which is based on previously published non-linear regression binding programs.^[17] The established binding affinity is astonishingly high for a non-rigid receptor system, which solely interacts via polar aromatic interactions with its substrate. Since

addition of 27DDF affects only the proton signals of the terminal aniline-biphenyl groups of E,Z-1 and all signals are shifted upfield the guest molecule must bind between these groups in a planar fashion. This observation is a strong indication for the predicted chelate-like binding mode resulting from multiple polar aromatic interactions in the host-guest complex.

A more detailed analysis of the ¹H NMR spectra revealed that also the Z,Z isomeric form of **1** interacts weakly with **8**. This weak binding apparently affects only one aniline moiety of Z,Z-**1** (the exo-aniline group, i.e. the left side of the molecule shown in Figure 1).



Figure 2. (a) Crystal structure of *Z*,*Z*-1. (b) After irradiation of *Z*,*Z*-1 with 420 nm light the helical isomer *E*,*Z*-1 is obtained in 94%. (c) Aromatic part of the HMBC-NMR spectrum (600 MHz, 300 K, toluene- d_8) of *E*,*Z*-1. The selective photoisomerization resulting in formation of only *E*,*Z*-1 was proven by HMBC cross-peak signals between protons 1 and 9 with the carbonyl C atom 7; the chemical shift of proton 9 unambiguously shows a *Z* configured double bond. The signal of the *E* configured double-bond proton 9' does not give a cross-peak signal with the carbonyl C atom 7, therefore the *Z*,*E* configuration is excluded. (d) Section of the NOESY-NMR spectrum (400 MHz, 300 K, [D₈]toluene) of *E*,*Z*-1 proving one single preferred conformation in which the *n*-pentyl groups and the protons of the double bonds are seen. The NOE cross-peak signal between proton 21' and the aromatic proton 15' is barely visible. The corresponding cross-peak signal between protons 21 and 15 could not be resolved.

The corresponding binding constant was also determined via NMR titration and is very small (K = 0.13 ± 0.01 L mol⁻¹ for a *Z*,*Z*-**1** : **8** = 1 : 1 stoichiometry). Because of the very large difference between the binding affinities of *E*,*Z*-**1** and *Z*,*Z*-**1** the aromatic guest molecule **8** can effectively be released after thermal backisomerization of *E*,*Z*-**1** to *Z*,*Z*-**1**. Guest binding and release is completely reversible and can be repeated multiple times (see Figure S21 in the Supporting Information).

A binding study with 9-dicyanomethylene-2,5-dinitrofluorene (25DDF, 9) - a regioisomer of 8 - revealed no signal shifts for *E*,*Z*-1 upon addition of this guest (see Figure S20 in the Supporting Information). Apparently, bis-HTI receptor 1 is able to distinguish efficiently between two regioisomeric electron poor aromatic guests, thus showing a remarkable selectivity in its binding affinity.

In summary, we have developed a photoswitchable molecular receptor **1** whose affinity for electron poor aromatic guests can be controlled with visible light and heat with extraordinary high precision. Additionally, even regioisomeric guest molecules can efficiently be distinguished with the new receptor. The core structure of this responsive system is a completely new bis-HTI motive, which shows an unprecedented selectivity for its photoisomerization reaction. After irradiation of **1** with 420 nm light only one of four possible configurations - the E,Z isomer - is obtained in up to 94% yield. The conscious implementation of negative preorganization further allowed us to precisely control the conformations of receptor **1** and force it into only one of four possible geometries in each isomeric state. In this way the reversible transition from a defined low-affinity planar structure to a defined helical structure with high affinity could be realized. Bis-HTI **1** can therefore be regarded as basic unit of a new type of photoswitchable foldamer, which possesses a defined structure even in the unfolded state. The helicity based chiral properties of this new receptor system will be explored in future studies especially in the context of enantioselective recognition and chiroptical properties of responsive materials.

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Text for Table of Contents

A novel bis-Hemithioindigo photoswitch is reported, whose conformation and helical folding can precisely be controlled by visible light and heat. The helical form is obtained after a highly selective photoisomerization and is able to bind electron deficient aromatic guests in a regioselective manner. A simple heating step leads to effective release of the guest.



Keywords

Host-guest systems • Molecular recognition • Photochemistry • Photochromism • Receptors