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(54) Title: PHOTOCROMIC DIARYLETHENE-CONTAINING COORDINATION COMPOUNDS AND THE PRODUCTION THEREOF

(57) Abstract: Diarylethene-containing ligands and their coordination compounds are described. The ligands display photochromism with UV excitation, while the coordination compounds display photochromism with both excitation in the UV region and excitation into lower energy absorption bands characteristic of the coordination compounds, through which the excitation wavelengths for the photocyclization can be extended from $\lambda \leq 340$ nm to wavelengths beyond 470 nm. Switching of the luminescence properties of the compounds has also been achieved through photochromic reactions.

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PHOTOCHROMIC DIARYLETHENE-CONTAINING
COORDINATION COMPOUNDS AND THE
PRODUCTION THEREOF

5 FIELD OF THE INVENTION

This invention is related to the design and the photochromic behavior of novel photochromic ligands and their coordination compounds. The design of these photochromic ligands and their coordination compounds is based on the *cis*-diarylethene structure, which forms part of a mono- or poly-cyclic ring structure that contains one or more donor
10 heteroatom(s) for coordination to an acceptor atom to form photochromic coordination compounds.

BACKGROUND OF THE INVENTION

Photochromism is defined as "a reversible transformation of a single chemical species
15 being induced in one or both directions by absorption of electromagnetic radiation, with two states having different distinguishable absorption spectra." Thus, photochromic compounds are compounds that possess at least two isomeric forms which have different physical properties, such as absorption properties, refractivity, and the like, and can be transformed from one form to another by light excitations at prescribed wavelengths.

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Photochromism has been intensively studied due to its potential use for optical recording and other optical functioning devices. To be practically used as optical recording materials, both isomeric forms must be thermally stable and possess excellent durability for reversible photochromic reactivity. Diarylethene is one class of photochromic compounds which
25 possesses these properties, and therefore is a suitable class of compounds for the construction of optical functioning devices. The *cis*-configuration of both aryl groups in the diarylethenes studied is generally fixed by an upper cycloalkene structure, such as fluorinated alicyclic group, aromatic group, anhydride and maleimide group. Apart from the difference in absorption characteristics and the like between the two forms and their thermal stabilities, the
30 availability of desirable excitation wavelengths that can be tuned and selected for the photochromic reactions also represents an important aspect in the design of materials for optical functioning devices. It has been shown that with the more π -conjugated upper cycloalkene structures, such as maleimide derivatives, in the diarylethene compounds, the

photocyclization proceeded with lower energy excitation in the visible region.

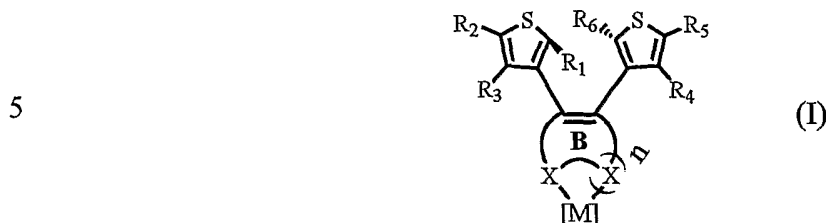
Further information can be found in U.S. patents 5,175,079, 5,183,726, 5,443,940, 5,622,812, and 6,359,150; Japanese patents JP 2-250877, JP 3-014538, JP 3-261762, JP 3-261781, JP 3-271286, JP 4-282378, JP 5-059025, JP 5-222035, JP 5-222036, JP 5-222037, JP 6-199846, JP 10-045732, JP 2000-072768, JP 2000-344693, JP 2001-048875, JP 2002-226477, JP 2002-265468 and JP 2002-293784; and in Irie et al., "Thermally Irreversible Photochromic Systems. Reversible Photocyclization of Diarylethene Derivatives", *Journal of Organic Chemistry*, 1988, 53, 803-808, Irie et al., "Thermally Irreversible Photochromic Systems. A Theoretical Study", *Journal of Organic Chemistry*, 1988, 53, 6136-6138, and Irie, "Diarylethenes for Memories and Switches", *Chemical Review*, 2000, 100, 1685-1716. The photochromic compounds of this invention can be used in the same way as described in these references.

The present invention relates to the use of coordination compounds to perturb the properties of the diarylethenes in photochromic compounds. Described below is a report of the design, synthesis and studies of *cis*-diarylethene-containing ligands, with the upper cycloalkene being part of a mono- or poly- cyclic ring structure that contains one or more donor heteroatom(s), such as phenanthrolines, pyridines, diazines, triazines, polypyridines, porphyrins and phthalocyanines and the like, for coordination compound formation.

SUMMARY OF THE INVENTION

The object of the present invention is to provide a new class of diarylethene-containing coordination compounds capable of displaying perturbed and sensitized photochromic properties. The invented photochromic compound is a coordination compound that contains a diarylethene with one or more donor atoms coordinated to an acceptor atom of the coordination compound. Any diarylethene in which the ethene group in a heterocyclic moiety, monocyclic or polycyclic, with any donor atom(s) capable of forming a coordination compound can be used in the present invention. There is no restriction on the nature of the aryl groups and they can be heteroaryl groups such as, for instance, thienyl groups. Likewise, any acceptor atoms which can be coordinated with the ethene-containing heterocyclic ligand moiety can be employed.

In a preferred form, the photochromic coordination compound is expressed by the following general formula (I):



where unit B represents a mono- or poly-cyclic ring structure, such as phenanthroline, pyridine, diazine, triazine, polypyridine, porphyrin and phthalocyanine and the like, that contains one to four donor heteroatom(s) X, such as nitrogen, oxygen, sulfur, phosphorus, selenium, i.e., n is integer from 0 to 3, [M] represents the coordination unit containing an acceptor atom M, such as rhenium(I), zinc(II), ruthenium(II), osmium(II), rhodium(III), iridium(III), gold(III), copper(I), copper(II), platinum(II), palladium(II), iron(II), cobalt(III), chromium(III), cadmium(II), boron(III) and the like, R₁ and R₆ individually represent alkyl groups and alkoxy groups, and R₂ to R₅ individually represent atoms or groups selected from the group of hydrogen atom, halogen atom, hydroxyl group, alkyl group, alkoxy group, cyano group, nitro group, alkylcarbonyl group, alkoxy carbonyl group, perfluoroalkyl group, aryl group, cycloalkyl group, arylcarbonyl group, aryloxy carbonyl group, mono- or dialkylaminocarbonyl group, alkylcarbonyloxy group, arylcarbonyloxy group, aryloxy group, alkoxy carbonyl group, aryloxy carbonyloxy group, and the like. In general, any alkyl or alkoxy group contains 1 to about 20 carbon atoms, any cycloalkyl group contains 3 to 8 carbon atoms, and any aryl group contains 6 to about 20 carbon atoms.

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A non-limiting list of examples of diarylethene compounds containing a heterocyclic ethene-containing ligand moiety includes 5,6-dithienyl-1,10-phenanthroline, 2,3,7,8,12,13,17,18-octathienyl-5,10,15,20-tetraphenyl porphyrin, 6,7-dithienyl-dipyrido[3,2-a:2',3'-c]phenazine and the like.

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A non-limiting list of coordination units includes chlorotricarbonylrhenium(I), dithiolatozinc(II), dihaloplatinium(II), bipyridylplatinum(II) bis[bipyridyl]-ruthenium(II), diphosphinocopper(I), bipyridylcopper(I) and the like.

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One of the advantages of the formation of coordination compounds from their pure organic counterparts (free ligands) in this invention is the extension of the excitation wavelength for the photocyclization of the diarylethene moiety from $\lambda \leq 340$ nm to lower

energy, so that the photochromic forward reaction can proceed with visible light excitation by utilization of the low-energy absorptions characteristic of coordination compounds. In addition, the photochromic reactions can be utilized to switch the photoluminescence properties characteristic of the coordination compounds.

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BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 shows a representative synthetic route for a diarylethene-containing ligand and its coordination compounds using 5,6-dithienyl-1,10-phenanthroline and its chlorotricarbonylrhenium(I) and dithiolatozinc(II) compounds as illustrative examples.

10 Fig. 2 shows the overlaid electronic absorption spectra of the open form (—) and the close form (— —) of a diarylethene-containing nitrogen donor ligand (L1).

Fig. 3 shows the overlaid electronic absorption spectra of the open form (—) and the close form (— —) of a diarylethene-containing coordination compound (1).

15 Fig. 4 shows the photochromic reactions of (a) a diarylethene-containing ligand and (b) its coordination compound using 5,6-dithienyl-1,10-phenanthroline and its chlorotricarbonylrhenium(I) compound as illustration.

Fig. 5 shows the overlaid corrected emission spectra of the open form (—) and the close form (— —) of (1) in benzene solution at 298 K.

20 Fig. 6 shows the overlaid corrected emission spectra of the open form (—) and the close form (— —) of (1) in EtOH-MeOH (4:1 v/v) glass at 77 K.

Fig. 7 shows the absorption spectral changes of complex (4) in benzene upon excitation at $\lambda = 300$ nm.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

25 Example 1

The ligand (L1) is synthesized by the Suzuki cross-coupling reactions of 2.5 equivalents of 2,5-dimethyl-3-thienylboronic acid and 5,6-dibromo-1,10-phenanthroline in the presence of the palladium catalyst, Pd(PPh₃)₄, and sodium carbonate in a heterogeneous mixture of water and THF according to the synthetic route depicted in Fig. 1. Excitation of (L1) with 313
30 nm light resulted in the formation of the close form, corresponding to the photocyclization product. The overlaid electronic absorption spectra of the open and close forms of (L1) in benzene solution are shown in Fig. 2.

Upon coordination to a chlorotricarbonylrhenium(I) complex, the open form of the corresponding complex (1) undergoes photocyclization with the excitation of both the intraligand absorptions at $\lambda \leq 340$ nm and the metal-to-ligand charge transfer (MLCT) absorption characteristic of this coordination compound up to $\lambda \leq 480$ nm. The electronic absorption data of (L1) and complex (1) are summarized in Table 1. The close forms of these compounds are found to undergo thermal backward reactions. The half-lives of the close forms have been determined and summarized in Table 2. The quantum yields for both photocyclization and photo-cycloreversion of (L1) and its rhenium complex (1) are summarized in Table 3.

TABLE 1

Compound	Configuration	Absorption (in benzene)
		$\lambda_{\text{abs}}/\text{nm}$ ($\epsilon / \text{dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$)
(L1)	Open form	304 (8670)
(L1)	Close form	366 (24340), 510 (4050), 540 (3860)
(1)	Open form	338 (4930), 396(4690)
(1)	Close form	390 (36670), 546 (5390), 580 (5050)

TABLE 2

Compound	Half-lives ($t_{1/2}$) at 20 °C	Half-lives ($t_{1/2}$) at 60 °C
(L1)	143 hours	222 mins
(1)	77.7 hours	79.3 mins

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TABLE 3

Compound	Photochemical Quantum Yield/ ϕ			
	Photocyclization ^a		Photo-cycloreversion	
	ϕ_{313}	ϕ_{440}	ϕ_{365}	ϕ_{510}
(L1)	0.486	0	0.123	0.029
(1)	0.552	0.648	0.028	0.009

^aValues reported are corrected to the ratio of the photochromic active conformation, i.e. with respect to the anti-parallel configuration

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The photoluminescence properties of both the open and close forms were measured. Fig. 5 and 6 display the overlaid emission spectra of the open form and the close form of complex (1) in benzene at 298 K and in EtOH-MeOH glass (4:1 v/v) at 77 K. The emission of complex (1) was found to change from metal-to-ligand charge transfer (MLCT) phosphorescence to ligand-centered (LC) phosphorescence upon photocyclization of the open form to the close form. These demonstrate the change of emission properties upon photochromic reactions. Table 4 summarized the emission data of ligand (L1) and complex (1).

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TABLE 4

Compound	Medium (T/K)	Emission	
		$\lambda_{em}^a/nm (\lambda_0/\mu s)$	
		Open form	Close form
(L1)	Benzene (298)	383 (<0.1)	644 (<0.1)
	Glass ^b (77)	-- ^c	577 (5.2)
(1)	Benzene (298)	595 (0.26)	626 (<0.1)
	Glass ^b (77)	535 (7.2)	620 (6.4)

^aExcitation wavelength at *ca.* 355 nm. Emission maxima are corrected values.

^bEtOH–MeOH (4:1, v/v)

^cNon-emissive

5

Example 2

Upon coordination of (L1) to a dithiolatozinc(II) complex, the open forms of the corresponding complexes (2), (3) and (4) undergo photocyclization with excitation at $\lambda \leq 340$ nm. Fig. 7 shows the absorption spectral changes of complex (4) upon excitation at $\lambda = 300$ nm. The electronic absorption maxima of both the open and the close forms of complexes (2), (3) and (4) are summarized in Table 5.

TABLE 5

Complex	Configuration	Absorption maximum (in benzene)
		λ_{abs}/nm
(2)	Open form	302, 326, 378
(2)	Close form	366, 382, 536, 576
(3)	Open form	302, 326, 382
(3)	Close form	366, 382, 538
(4)	Open form	302, 336, 396
(4)	Close form	366, 384, 542, 584

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Those skilled in the art will recognize that various changes and modifications can be made in the invention without departing from the spirit and scope thereof. The various embodiments described were for the purpose of further illustrating the invention and were not intended to limit it.

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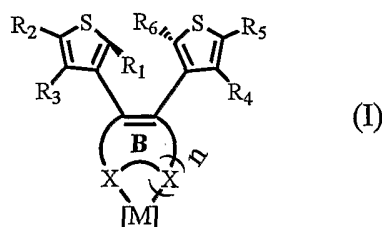
What is claimed is:

1. A photochromic coordination compound comprising a diarylethene with one or more donor atoms coordinated to an acceptor atom of the coordination unit.

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2. A photochromic compound which is a diarylethene-containing coordination compound in which the diarylethene contains part of a mono- or poly-cyclic ring structure with one or more donor heteroatom(s) coordinated to a coordination unit [M], which contains an acceptor atom, M, expressed by the general formula (I):

10



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where unit B represents a mono- or poly-cyclic ring structure that contains one to four heteroatom(s) X, n is integer from 0 to 3, [M] represents a coordination unit containing an acceptor atom M, R₁ and R₆ individually represent an alkyl group or an alkoxy group, and R₂ to R₅ individually represent atoms or groups selected from the group of hydrogen atom, halogen atom, hydroxyl group, alkyl group, alkoxy group, cyano group, nitro group, alkylcarbonyl group, alkoxy carbonyl group, perfluoroalkyl group, aryl group, cycloalkyl group, arylcarbonyl group, aryloxy carbonyl group, mono- or dialkylaminocarbonyl group, alkylcarbonyloxy group, arylcarbonyloxy group, aryloxy group, alkoxy carbonyl group, and aryloxy carbonyloxy group.

25

3. A photochromic compound in accordance with claim 2, wherein unit B is selected from the group consisting of phenanthroline, pyridine, diazine, triazine, polypyridine, porphyrin and phthalocyanine; and acceptor atom M is selected from the group consisting of rhenium(I), zinc(II), ruthenium(II), osmium(II), rhodium(III), iridium(III), gold(III), copper(I), copper(II), platinum(II), palladium(II), iron(II), cobalt(III), chromium(III), cadmium(II) and boron(III).

30

4. A photochromic compound in accordance with claim 2, wherein the heteroatom X is selected from the group consisting of nitrogen, oxygen, sulfur, phosphorus and selenium.

5. A photochromic compound in accordance with claim 2, wherein unit B is a phenanthroline.

5 6. A photochromic compound in accordance with claim 2, wherein unit B is a porphyrin.

7. A photochromic compound in accordance with claim 5 or claim 6, wherein R_1 and R_6 represent methyl groups, and R_3 and R_4 represent hydrogen atoms.

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8. A photochromic compound in accordance with claim 7, wherein R_2 and R_5 represent hydrogen atoms.

15 9. A photochromic compound in accordance with claim 7, wherein R_2 and R_5 represent methyl groups.

10. A photochromic compound in accordance with claim 7, wherein R_2 and R_5 represent bromine atoms.

20 11. A photochromic compound in accordance with claim 5, wherein M represents rhenium(I).

12. A photochromic compound in accordance with claim 7, wherein M represents zinc(II).

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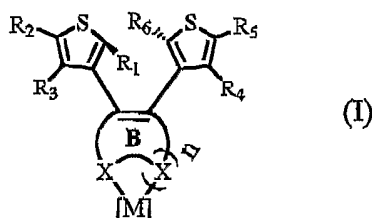
13. A photochromic compound in accordance with claim 7, wherein M represents platinum(II).

AMENDED CLAIMS

[Received by the International Bureau on 27 September 2004 (27.09.04)
original claim 1 amended; remaining claims unchanged (1 page).

1. A photochromic coordination compound comprising a diarylethene, in which the ethene moiety forms part of a mono- or poly-cyclic ring structure that contains or is connected to one or more donor atoms coordinated to an acceptor atom of the coordination unit.

2. A photochromic compound which is a diarylethene-containing coordination compound in which the diarylethene contains part of a mono- or poly-cyclic ring structure with one or more donor heteroatom(s) coordinated to a coordination unit [M], which contains an acceptor atom, M, expressed by the general formula (I):



where unit B represents a mono- or poly-cyclic ring structure that contains one to four heteroatom(s) X, n is integer from 0 to 3, [M] represents a coordination unit containing an acceptor atom M, R₁ and R₆ individually represent an alkyl group or an alkoxy group, and R₂ to R₅ individually represent atoms or groups selected from the group of hydrogen atom, halogen atom, hydroxyl group, alkyl group, alkoxy group, cyano group, nitro group, alkylcarbonyl group, alkoxy carbonyl group, perfluoroalkyl group, aryl group, cycloalkyl group, arylcarbonyl group, aryloxy carbonyl group, mono- or dialkylaminocarbonyl group, alkylcarbonyloxy group, aryloxy carbonyloxy group, aryloxy group, alkoxy carbonyl group, and aryloxy carbonyloxy group.

3. A photochromic compound in accordance with claim 2, wherein unit B is selected from the group consisting of phenanthroline, pyridine, diazine, triazine, polypyridine, porphyrin and phthalocyanine; and acceptor atom M is selected from the group consisting of rhenium(I), zinc(II), ruthenium(II), osmium(II), rhodium(III), iridium(III), gold(III), copper(I), copper(II), platinum(II), palladium(II), iron(II), cobalt(III), chromium(III), cadmium(II) and boron(III).

4. A photochromic compound in accordance with claim 2, wherein the heteroatom X is selected from the group consisting of nitrogen, oxygen, sulfur, phosphorus and selenium.

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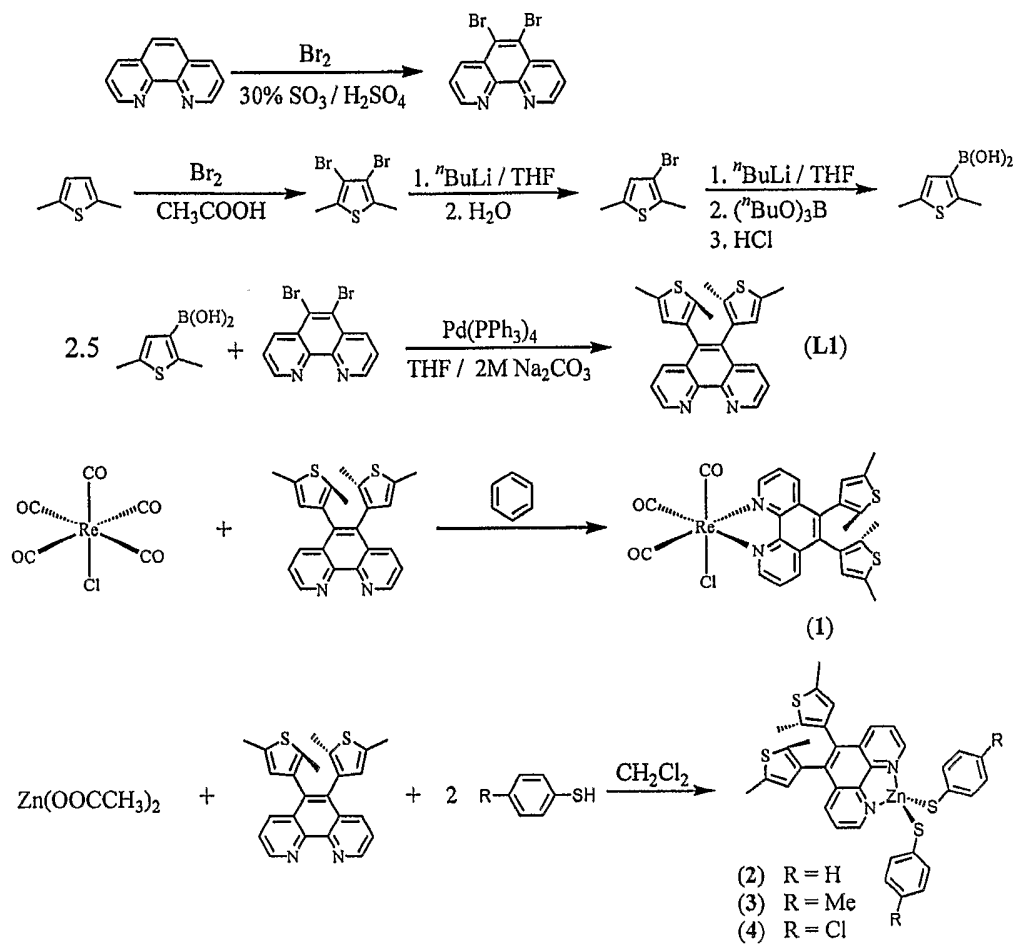


FIG. 1

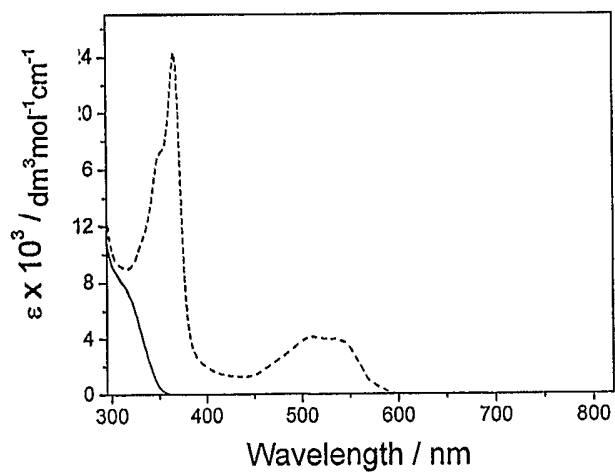


FIG. 2

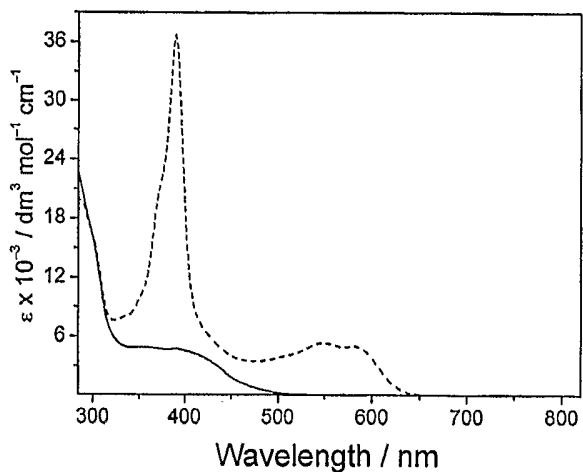


FIG. 3

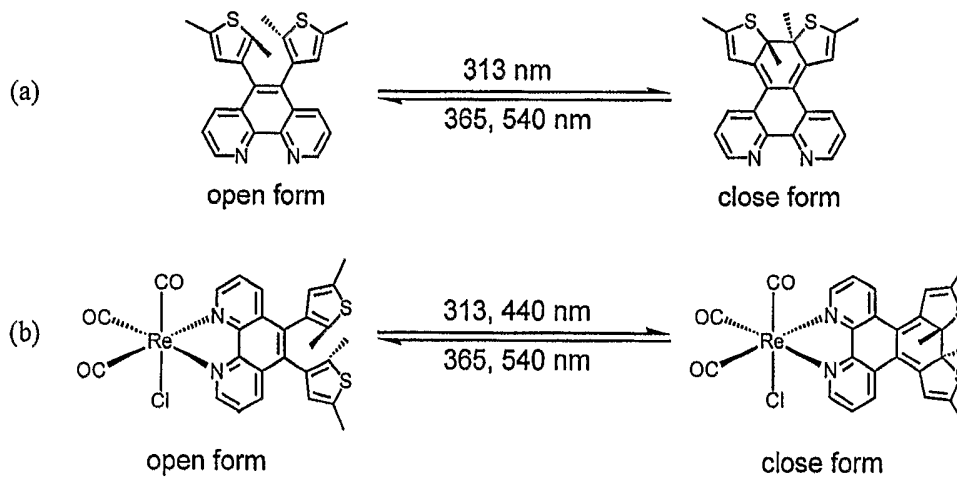


FIG. 4

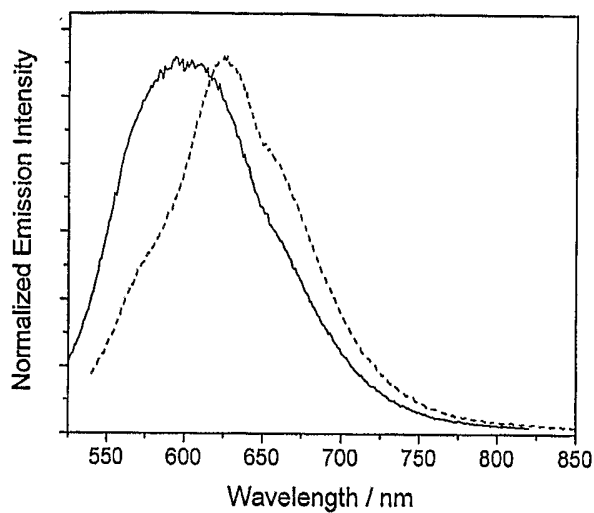


FIG. 5

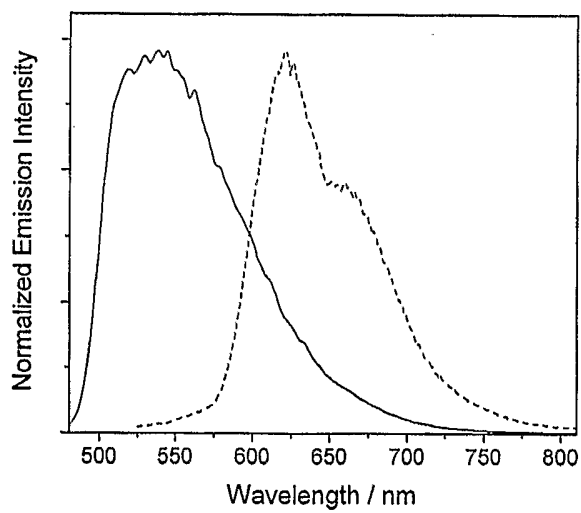


FIG. 6

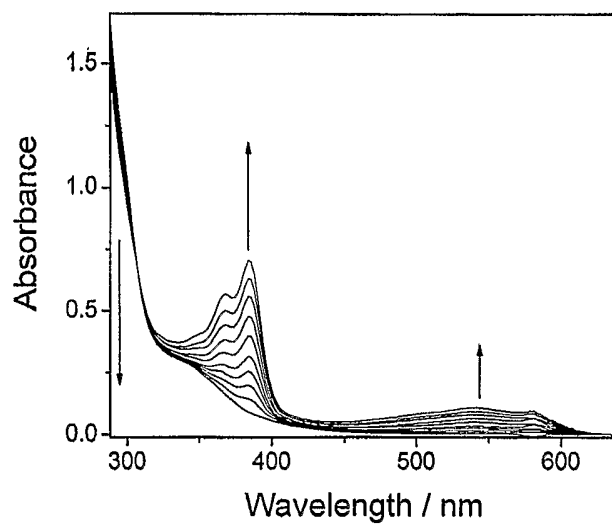



FIG. 7

INTERNATIONAL SEARCH REPORT

International application No.
PCT/CN2004/000755

A. CLASSIFICATION OF SUBJECT MATTER <p style="text-align: center;">C07D409/14, C09K09/02</p> <p>According to International Patent Classification (IPC) or to both national classification and IPC</p>		
B. FIELDS SEARCHED <p>Minimum documentation searched (classification system followed by classification symbols)</p> <p style="text-align: center;">IPC7 C07D409, C09K09, C07D333,</p> <p>Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched</p> <p>Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)</p> <p style="text-align: center;">EPODOC, WPI, PAJ, CPRS, PAJ ,CNKI, Chemical Abstract</p>		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	JP-A-8-245579 (IRIE M), 24. Sep. 1996(24. 09. 1996), whole document	1-13
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A	US-A-5183726 (Pioneer Electronic Corp.) 02. Feb. 1993(02. 02. 1993), whole document	1-13
<input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C. <input checked="" type="checkbox"/> See patent family annex.		
* Special categories of cited documents: "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier application or patent but published on or after the international filing date "L" document which may throw doubts on priority claim (S) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family	
Date of the actual completion of the international search	Date of mailing of the international search report	
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Name and mailing address of the ISA/CN	Authorized officer	
6 Xitucheng Road., Jimen Bridge, Haidian District, 100088, Beijing, China		
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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
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