

Samsung Display Co., Ltd.
v.
Pictiva Displays International, Ltd.

Case No. IPR2024-01094
U.S. Patent No. 8,558,223

Petitioner's Demonstratives

October 24, 2025 Oral Hearing

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SDC Ex. 1041

Instituted Grounds Of Unpatentability

Trial Instituted on the '223 Patent (Ex. 1001)

- Ground I: Claims 1–4, 6, and 8–14 are rendered obvious by Werner (Ex. 1006; Translation Ex. 1005)
- Ground II: Claims 1–2, 6, and 8–14 are rendered obvious by Ma (Ex. 1007)

Ground 1 - Werner

Ground 1 - Werner

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(26) Publication Language: German

(30) Priority Data: 10 2004 010 954.0 March 3, 2004 (03/03/2004) DE

(71) Applicant (for all designated states except the US): NOVALED GMBH [DE/DE]; Tatzberg 49, 01307 Dresden (DE).

(72) Inventors; and
(73) Inventors/Applicants (for the US only): WERNER, Angar [DE/DE]; Altenberger Strasse 20, 01277 Dresden (DE); KÜHL, Olaf [DE/DE]; Mittelstrasse 10, 04416 Markkleeberg (DE); GESSLER, Simon [DE/DE]; Hauptstrasse 79, 69117 Heidelberg (DE); HARADA, Keitaro [DE/DE]; Blumenstrasse 90, 01307 Dresden (DE); HARTMANN, Heide [DE/DE]; Wollnstrasse 4.

(74) Attorneys: SCHOLZ, Volker et al.; Boehmert & Boehmert, Holleralle 32, 28209 Bremen (DE).

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(84) Designated States (unless otherwise indicated, for every available regional protection type): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, ...)

(54) Title: USE OF A METAL COMPLEX AS N-DOPANT FOR AN ORGANIC SEMICONDUCTING MATRIX MATERIAL, ORGANIC SEMICONDUCTOR MATERIAL, AND ELECTRONIC COMPONENT, AS WELL AS DOPANT AND LIGAND AND METHOD FOR PRODUCING THE SAME

(57) Abstract: The invention relates to the use of a metal complex as n-dopant for doping an organic semiconductor matrix material for modifying the electrical properties of the same, wherein the compound represents an n-dopant with regard to the matrix material. The aim of the invention is to provide n-doped organic semiconductors for matrix materials that have little reduction potential while achieving high conductivities. According to the invention, a neutral electron-rich metal complex having a central atom as the preferably neutral or charged transition metal atom having a valence electron number of at least 16 is used as the dopant compound. The complex can especially be multinuclear and comprises at least one metal-metal bond. At least one ligand can form a pi-complex with the central atom, or can be a bridging ligand, especially hpp, a borate, carbamate or triazacycloalkane or comprise at least one carbonium carbon atom or a bivalent atom selected from the group including C (carbene), Si (silylene), Ge (germylene), Sn, Pb. The invention also relates to novel n-dopants and methods for producing them.

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- PCT Application Publication No. WO 2005/086251 (“Werner”) was published on September 15, 2005

- Werner is directed to “an electronic device with an organic semiconducting material doped with a metal complex as an n-dopant.”

Werner, 1:14-16

Werner

Ground 1 - Issues In Dispute

- Independent claim disputes:
 - Whether Werner discloses metal complexes and organic compounds in combination
 - Whether Werner discloses joint vaporization
- Dependent claim disputes:
 - Whether Werner discloses organic LED (OLED)
 - Whether $W_2(hpp)_4$ and BPhen coordinate
 - Whether a chain-like structure is formed by $W_2(hpp)_4$ and BPhen
 - Whether Werner discloses that the degree of cross-linking of the electron-conducting layer is controlled by the ratio between the metal complex and the organic compound during vaporization

Werner Discloses Metal Complexes And Organic Compounds In Combination

Claims 1, 10

Werner Satisfies Claims 1 And 10

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(54) Title: USE OF A METAL COMPLEX AS N-DOPANT FOR AN ORGANIC SEMICONDUCTING MATRIX MATERIAL, ORGANIC SEMICONDUCTOR MATERIAL, AND ELECTRONIC COMPONENT, AS WELL AS DOPANT AND LIGAND AND METHOD FOR PRODUCING THE SAME

(57) Abstract: The invention relates to the use of a metal complex as n-dopant for doping an organic semiconductor matrix material for modifying the electrical properties of the same, wherein the compound represents an n-dopant with regard to the matrix material. The aim of the invention is to provide n-doped organic semiconductors for matrix materials that have little reduction potential while achieving high conductivities. According to the invention, a neutral electron-rich metal complex having a central atom as the preferably neutral or charged transition metal atom having a valence electron number of at least 16 is used as the dopant compound. The complex can especially be multinuclear and comprises at least one metal-metal bond. At least one ligand can form a pi-complex with the central atom, or can be a bridging ligand, especially hpp, a borate, carbamate or triazacyclohexane or comprise at least one carbonium carbon atom or a bivalent atom selected from the group including C (carbene), Si (silylene), Ge (germylene), Sn, Pb. The invention also relates to novel n-dopants and methods for producing them.

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Werner

1. An organic electronic component, comprising:
a substrate,
a first electrode,
a second electrode,
an electron-conducting layer which is arranged in such a way that it is electrically conductively connected to at least one of the electrodes,
wherein the electron-conducting layer is obtained by joint vaporization of a metal complex, which comprises a central metal atom, with an organic compound, wherein the organic compound comprises a heteroaromatic compound which is conjugated with an aromatic compound via a C—C bond.

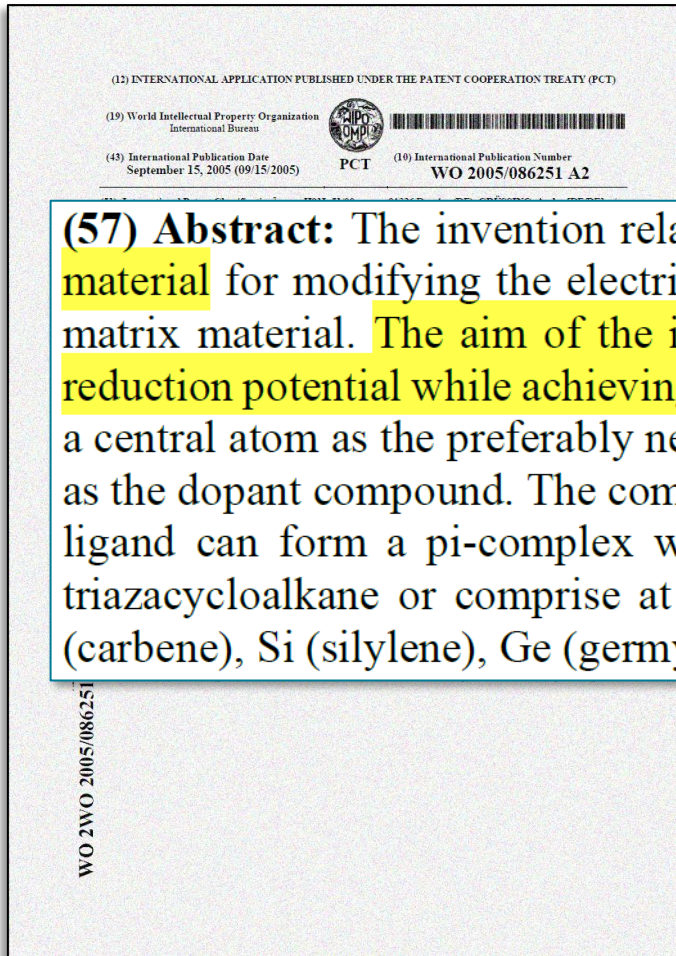
'223 Patent, claim 1

'223 Patent, claim 10

10. A method for manufacturing an organic electronic component, comprising the method steps of:

- A) providing a substrate,
 - B) applying a first electrode,
 - C) depositing an electron-conducting layer on the substrate,
 - D) applying a second electrode,
- wherein the electron-conducting layer is deposited by means of simultaneous vaporization of a metal complex and an organic compound, wherein the organic compound comprises a heteroaromatic compound which is conjugated with an aromatic compound via a C—C bond.

Werner Discloses Using A “Metal Complex” To Dope An Organic Matrix Material (i.e., Compound), As Required By Claims 1 And 10



(57) Abstract: The invention relates to the use of a metal complex as n-dopant for doping an organic semiconducting matrix material for modifying the electrical properties of the same, wherein the compound represents an n-dopant with regard to the matrix material. The aim of the invention is to provide n-doped organic semiconductors for matrix materials that have little reduction potential while achieving high conductivities. According to the invention, a neutral electron-rich metal complex having a central atom as the preferably neutral or charged transition metal atom having a valence electron number of at least 16 is used as the dopant compound. The complex can especially be multinuclear and comprises at least one metal-metal bond. At least one ligand can form a pi-complex with the central atom, or can be a bridging ligand, especially hpp, a borate, carborane or triazacycloalkane or comprise at least one carbanion carbon atom or a bivalent atom selected from the group including C (carbene), Si (silylene), Ge (germylene), Sn, Pb. The invention also relates to novel n-dopants and methods for producing them.

Werner, Abstract

Werner

Werner Discloses Using Substituted Heteroaromatics, Including BPhen, As The Organic Compound

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(72) Inventors; and (73) Inventors/Applicants (for the US): Angerer (DE/DE); Altenberger (DE); KÜHL, Olaf (DE/DE); Mackdeesberg (DE); GESS, Hauptstraße 79, 69117 Heidelberg (DE); Bhamann (DE); HARTMANN, Hans-Joachim (DE/DE); Werner (DE/DE); Tatzberg 49, 01307 Dresden (DE).

(74) Attorneys: SCHOLZ, Volker et al.; Boehmert & Boehmert, Hollerstraße 32, 28209 Bremen (DE).

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(54) Title: USE OF A METAL MATERIAL, ORGANIC SEMICONDUCTOR AND LIGAND AND METHOD FOR THE PRODUCTION THEREOF

(57) Abstract: The invention relates to a method for producing a matrix material. The aim of the invention is to provide a matrix material which has a high reduction potential while achieving a central atom as the preferred use as the dopant compound. The compound can form a pi-complex with a heterocyclic compound or comprise at least one heterocyclic compound (carbene), Si (silyle), Ge (germyl).

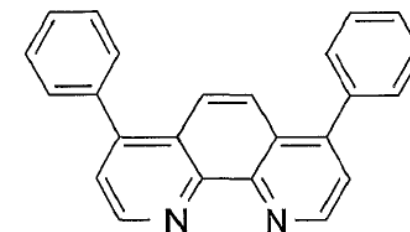
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Phenanthrolines, which can be substituted or unsubstituted, in particular aryl-substituted, such as phenyl or naphthyl-substituted, can also be used as matrix materials. In particular, Bphen can be used as a matrix material.

Werner, 55:3-6

Heteroaromatics, in particular triazoles, can also be used as matrix materials, possibly also pyrroles, imidazoles, triazoles, pyridines, pyrimidines, pyridazines, and the like. The heteroaromatics are preferably substituted, in particular aryl-substituted, such as phenyl or naphthyl-substituted. In

Werner, 55:8-12



BPhen = bathophenanthroline
(4,7-Diphenyl-1,10-phenanthroline)
C₂₄H₁₆N₂

Werner, 54:11-13

Werner

Werner Discloses Using $W_2(hpp)_4$ As The Metal Complex

(57) **Abstract:** The invention relates to the use of a metal complex as n-dopant for doping an organic semiconducting matrix material for modifying the electrical properties of the same, wherein the compound represents an n-dopant with regard to the matrix material. The aim of the invention is to provide n-doped organic semiconductors for matrix materials that have little reduction potential while achieving high conductivities. According to the invention, a neutral electron-rich metal complex having a central atom as the preferably neutral or charged transition metal atom having a valence electron number of at least 16 is used as the dopant compound. The complex can especially be multinuclear and comprises at least one metal-metal bond. At least one ligand can form a pi-complex with the central atom, or can be a bridging ligand, especially hpp, a borate, carborane or triazacycloalkane or comprise at least one carbanion carbon atom or a bivalent atom selected from the group including C (carbene), Si (silylene), Ge (germylene), Sn, Pb. The invention also relates to novel n-dopants and methods for producing them.

Werner, Abstract

The metal atoms of multinuclear metal complexes are preferably predominantly or entirely transition metal atoms, in particular from groups 6 or 7 to 10, for example selected from the group Cr, Mn, Fe, Co, Ni, Cu, Zn, Mo, W.

Werner, 11:21-24

Application examples

An electron-rich neutral metal complex according to the invention is provided, where the metal complex can be, for example:

M-bis-(terpyridine) with M equal to Cr, Mn, Fe, Co, Ni, Cu, Zn, Mo, or W; M_2hpp_4 with M equal to Cr, Mn, Fe, Co, Ni, Cu, Zn, Mo, or W; $M_2(AlkylCOO)_4$ F with M equal to Cr, Mn, Fe, Co, Ni, Cu, Zn,

Werner, 60:16-21

Werner

Case Law Does Not Require Separate Motivation To Combine Elements Disclosed For Use Together

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“[T]he Board’s approach would require a motivation to combine each element of the claim—even those present together in a reference. This analysis unduly dissects prior art references into collections of individual elements, requiring a party showing obviousness to re-do the work already done in the prior art reference.”

Gen. Elec. Co. v. Raytheon Tech. Corp., 983 F.3d 1334, 1352 (Fed. Cir. 2020)

Werner

Werner Discloses That Using Metal Complexes Like $W_2(hpp)_4$ For Doping Organic Compounds Like BPhen Will Be Successful

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(71) Applicant: NOVAL, Dresden

(72) Inventor: Angar [DE], KU, Mackdee, Hauptstr. 1, 01309 Dresden (DE), HA

(54) Title: MATERIAL AND LIGAND

(57) Abstract: material for an matrix materia reduction poten a central atom as the dopant c ligand can for triazacycloall (carbene), Si (c

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The invention has the object of specifying metal complexes as n-dopants for doping an organic semiconducting matrix material, in particular for producing electronic devices that in particular have sufficiently low oxidation potentials for producing electron transport materials for organic light-emitting diodes (OLEDs) without adverse effects on the matrix material, effectively increasing the number of charge carriers in the matrix material, and being relatively easy to handle. Another object of the

Werner, 2:21-29

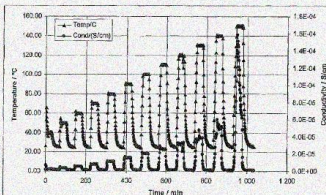
Werner

Electronic device

Using the organic compounds according to the invention to produce n-doped organic semiconducting materials, which can be arranged in particular in the form of layers or electrical conduction paths, a variety of electronic devices or devices containing such with an n-doped organic semiconducting layer can be produced, which are thus included in the invention. In the sense of the invention, the term "electronic devices" also includes optoelectronic devices. The invention's compounds can beneficially alter the electronic properties of an electronically functionally effective area of the device, such as its electrical conductivity, light-emitting properties, or the like. For example, using the metal complexes as n-dopants according to the invention can improve the conductivity of the doped layers and/or the charge carrier injection from contacts into the doped layer.

Werner, 58:21-35

Evidence Of Other $W_2(\text{hpp})_4$ -Doped Phenanthrolines Confirms That POSA Would Have Had An Expectation Of Success

		US000792541B2						
(12) United States Patent Werner et al.		(10) Patent No.: US 7,972,541 B2						
		(45) Date of Patent: Jul. 5, 2011						
(54) DOPED ORGANIC SEMICONDUCTOR MATERIAL.	(56) References Cited	U.S. PATENT DOCUMENTS						
(75) Inventors: Alexander Werner, Dresden (DE); Andrea Lutz, Dresden (DE); Josef Salbeck, Kaufungen (DE)	2,566,209 A 8/1991 Jochims 3,083,242 A 1/1963 Ramsden 3,228,450 A 12/1965 Blazynski 3,558,671 A 1/1971 Martin 3,563,751 A 2/1971 Cohen 4,003,048 A 1/1977 Fukunaga 4,006,559 A 1/1978 Lim 4,133,821 A 1/1979 West et al. 4,438,423 A 1/1986 Kato							
(73) Assignee: Novaled AG, Dresden (DE)								
(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 357 days.								
(21) Appl. No.: 12/093,167								
(22) PCT Filed: Nov. 10, 2006								
(86) PCT No.: PCT/EP2006/010816								
§ 371 (c)(1), (2), (4) Date: Jul. 21, 2008								
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H01L 29/08 (2006.01)								
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H01B 1/12 (2006.01)								
H32D 9/00 (2006.01)								
(52) U.S. CL.	252/519.2; 428/690; 257/40; 257/102; 257/103							
(58) Field of Classification Search	257/40; 257/102; 103; 252/519.2; 428/690							
See application file for complete search history.								
		(74) Attorney, Agent, or Firm Sutherland, Asbill & Brennan, LLP						
		(57) ABSTRACT						
The present invention relates to a doped organic semiconductor material comprising at least one organic matrix material, which is doped with at least one dopant; the matrix material being selected from a group consisting of certain phenanthroline derivatives; and also an organic light-emitting diode which comprises such a semiconductor material.								
29 Claims, 2 Drawing Sheets								
								

Pictiva EX2044
 Sampling v Pictiva
 IPR2024-01094

Ex. 2044 Werner '541

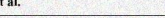
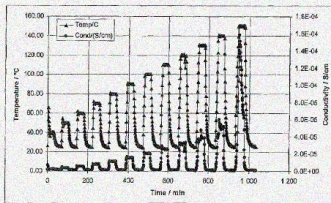
Selected phenanthroline derivatives, which are shown in Table 2 below, were used as matrix material for doping with $W_2(hpp)_4$ (structure IVa), and the conductivity was measured. The results are summarized in Table 2.

Ex. 2044 Werner '541, 9:18-21, 8:6-15

TABLE 2					
structure I					
R	T _g (° C.)	T _s (° C.)	LUMO (eV)	Conductivity (S/cm)*	
Ia R2, R4, R5, R7 = H R1, R3, R6, R8 = phenyl	106	260	2.47	3E-4	
Ib R2, R4, R5, R7, R8 = H R3, R6 = phenyl R1 = 1-naphthyl	104	265	2.42	5E-6	
Ic R2, R4, R5, R7 = H R3, R6 = phenyl R1, R8 = naphthyl	119	280	2.5	2E-6	
Id R2, R4, R5, R7 = H R3, R6 = phenyl R1, R8 = 1-(4,4'-biphenyl)	140	317	2.53	6E-5	
Ie R2, R4, R5, R7 = H R3, R6 = phenyl R1, R8 = 1-phenanthryl	165	317	2.51	2E-6	

Ex. 2044 Werner '541, 9:22-49

Contrary To Pictiva's Argument, Werner '541 Does Not Teach Away From Using $W_2(hpp)_4$ For Doping BPhen

		US007972541B2		TABLE 1	
(12) United States Patent Werner et al.		(10) Patent No.: US 7,972,541 (45) Date of Patent: Jul. 1, 2009		CV data, empirically determined LUMO energies and measured conductivities of various electron transport materials (BAL ₂ = bis(2-methyl-8-quinolinato)-4-(phenylphenolate) aluminium (III), BPhen = bathophenanthroline, Alq ₃ = tris (8-hydroxyquinoline) aluminium, Cl6-Alq = tris (5,7-dichloro-8-hydroxyquinoline) aluminium)	
(54) DOPED ORGANIC SEMICONDUCTOR MATERIAL		(56) References Cited U.S. PATENT DOCUMENTS 2,566,208 A 8/1951 Jordan 3,083,242 A 3/1963 Ramsden 3,226,850 A 12/1965 Blazegak 3,558,671 A 1/1971 Martin 3,563,751 A 2/1971 Cohen 4,003,882 A 1/1977 Fukunaga 4,066,509 A 1/1978 Lim 4,133,221 A 1/1979 West et al. 4,618,453 A 10/1980 Kim 4,960,616 A 10/1990 Park et al. 5,093,698 A 3/1993 Egan (Continued)		FOREIGN PATENT DOCUMENTS CA 2549369 9/2005 (Continued)	
(75) Inventors: Angar Werner, Dresden (DE); Andrea Lux, Dresden (DE); Josef Salbeck, Kufstein (DE)		OTHER PUBLICATIONS Akiba, Kin'ya et al., "Direct Synthesis of 2,2-diary-3-dihydrobenzothiazoles from 3-methyl-2,3-dihydrobenzothione and some mechanistic aspects" (Bulletin of the Society of Japan, vol. 52(1), pp. 156-158, (1979)). (Continued)		LUMO in eV (determined by CV	
(73) Assignee: Novaled AG, Dresden (DE)		(57) ABSTRACT The present invention relates to a doped organic semiconductor comprising at least one organic matrix which is doped with at least one dopant, the matrix being selected from a group consisting of certain p-line derivatives; and also an organic light-emitter which comprises such a semiconductor material.		σ (conductivity) in S/cm with a doping concentration of 5 mol %	
(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 357 days.		Primary Examiner — Mark Kopeck Assistant Examiner — Melissa P. Thomas (74) Attorney, Agent, or Firm — Sutherland, Brennan, LLP		Matrix with Fe/Fe ⁺ as internal standard)	
(21) Appl. No.: 12/093,167		Foreign Application Priority Data US 2009/0001327 A1 Jun. 1, 2009		σ (conductivity) in S/cm undoped	
(22) PCT Filed: Nov. 10, 2006		(51) Int. Cl. H01L 29/08 (2006.01) H01L 55/00 (2010.01) H01B 1/12 (2006.01) B52B 9/06 (2006.01)		Alq ₃ 2.4 <1E-10 9.2E-8	
(86) PCT No.: PCT/EP2006/011816 § 371 (c)(1), (2), (4) Date: Jul. 21, 2008		(52) U.S. CL. 252/519.2; 428/690; 257/40; 257/102; 257/103; 252/519.2; 428/690 See application file for complete search history.		BPhen 2.42 <1E-10 4E-9	
(87) PCT Pub. No.: WO2007/054345 PCT Pub. Date: May 18, 2007		(59) Field of Classification Search 257/102, 103; 252/519.2; 428/690 See application file for complete search history.		BAL ₂ 2.39 <1E-10 8e-8	
		29 Claims, 2 Drawing Sheets			

Ex. 2044 Werner '541

EXAMPLE 2

A glass substrate is provided with contacts made of indium tin oxide. A layer of **Ie** (Table 2) doped with a dopant of structure **IVa** is then produced on the substrate. The doping concentration of the dopant **IVa** is 4 mol %. The conductivity

Ex. 2044 Werner '541, 9:55-60

Comparative Example 2 was carried out in a manner analogous to the procedure according to Example 2, but BPhen was used instead of the matrix material Ie. The resulting conductivity of the layer is 4×10^{-7} S/cm. As can be seen from FIG. 2,

Ex. 2044 Werner '541, 10:9-12

The data in Werner '541 shows a 40x - 4000x conductivity improvement of the BPhen:W₂(hpp)₄ film over undoped BPhen

Pet. Reply, 9

Ma Shows That A 2-3 Fold Increase In Conductivity Was Considered Significant

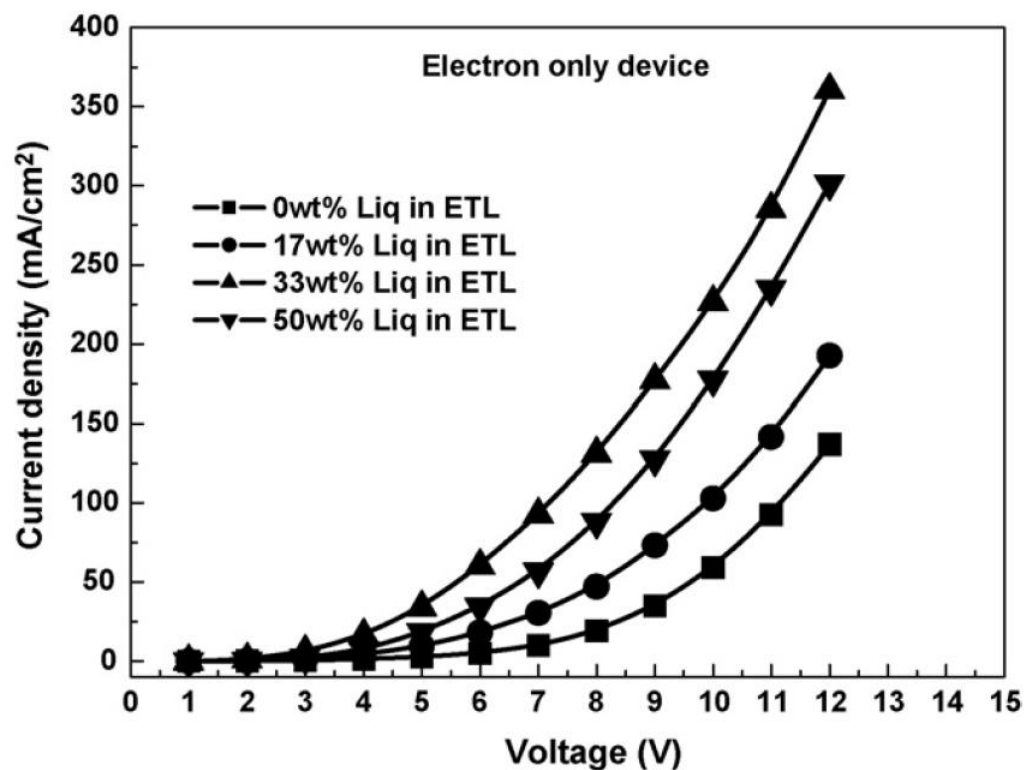


Fig. 5. Current density vs voltage characteristics of electron-only devices of 0 wt.% 17 wt.%, 33 wt.% and 50 wt.% Liq doped into BPhen.

Ma, Fig. 5

In this paper, we have demonstrated a device with an new n-doping (33 wt.% Liq:BPhen) layer as ETL and p-type (*m*-MTDATA:*x* wt.% F₄-TCNQ) layer as HTL. Here, 33 wt.% Liq is chosen due to the fact that the device with the doping ratio of 33 wt.% Liq demonstrates the best current density vs voltage (*J*-*V*) curve among all the electron only devices. Using this method, we obtained a significant improvement in the device performance as compared with the referenced device.

Ma, 810-811

compared with the referenced device.” *Id.*, 810–811. In other words, researchers would consider about a two to three-fold increase in conductivity as a significant improvement. Compared to this result, a POSA would consider that the 4000-fold increase in conductivity obtained in the ’541 patent by W₂(hpp)₄-doped BPhen was a significant improvement. Further, the 4000-fold increase was obtained by merely

Ex. 1020 Rand Reply Decl., ¶ 12

Pictiva's Reliance On One Statement In Werner '541 About HOMO-LUMO Differences Is Contradicted By Other Disclosures In The Same Reference



US007972541B2

(12) **United States Patent**
Werner et al.

(10) Patent No.: **US 7,972,541 B2**
(45) Date of Patent: **Jul. 5, 2011**

(54) **DOPED ORGANIC SEMICONDUCTOR MATERIAL**

(75) Inventors: **Amgar Werner, Dresden (DE); Andrea Lux, Dresden (DE); Josef Salbeck, Rastatt (DE)**

(73) Assignee: **Novaled AG, Dresden (DE)**

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 357 days.

(21) Appl. No.: **12/093,167**

(22) PCT Filed: **Nov. 10, 2006**

(86) PCT No.: **PCT/EP2006/010816**
§ 371 (c)(1), (2), (4) Date: **Jul. 21, 2008**

(87) PCT Pub. No.: **WO2007/054345**
PCT Pub. Date: **May 18, 2007**

(65) **Prior Publication Data**
US 2009/0001327 A1 Jan. 1, 2009

(30) **Foreign Application Priority Data**
Nov. 10, 2005 (EP) 05024515

(51) Int. Cl. **H01L 29/08** (2006.01)
H01L 51/00 (2010.01)
H01L 1/12 (2006.01)
H22B 9/06 (2006.01)
(52) U.S. Cl. **252/519.2; 428/690; 257/40; 257/102; 257/103**
(58) **Field of Classification Search** 257/102, 103, 252/519.2, 428/690
See application file for complete search history.

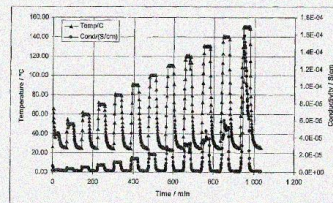
(56) **References Cited**
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3,083,242 A 3/1963 Ramsden
3,226,450 A 12/1965 Hasegawa
3,558,671 A 1/1971 Martin
3,563,751 A 2/1971 Cohen
4,003,943 A 1/1977 Fukunaga
4,006,569 A 1/1978 Lim
4,133,821 A 1/1979 West et al.
4,618,475 A 10/1986 Kim
4,960,916 A 10/1990 Park et al.
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CA 2549369 9/2005
(Continued)

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(Continued)

ABSTRACT
The present invention relates to a doped organic semiconductor material comprising at least one organic matrix material, which is doped with at least one dopant, the matrix material being selected from a group consisting of certain phenanthroline derivatives; and also an organic light-emitting diode which comprises such a semiconductor material.

29 Claims, 2 Drawing Sheets



Pictiva EX2044
Samsung v Pictiva
IPR2024-01094

Ex. 2044 Werner '541

It is also proposed that the matrix material has an energy level for the lowest unoccupied molecular orbital (LUMO), which differs by 0-0.5 V from the ionization potential (HOMO) of the dopant, preferably by 0-0.3 V, particularly preferably by 0-0.15V.

Ex. 2044 Werner '541, 8:19-23;
Ex. 2041 Schanze
Opening Decl., ¶ 69

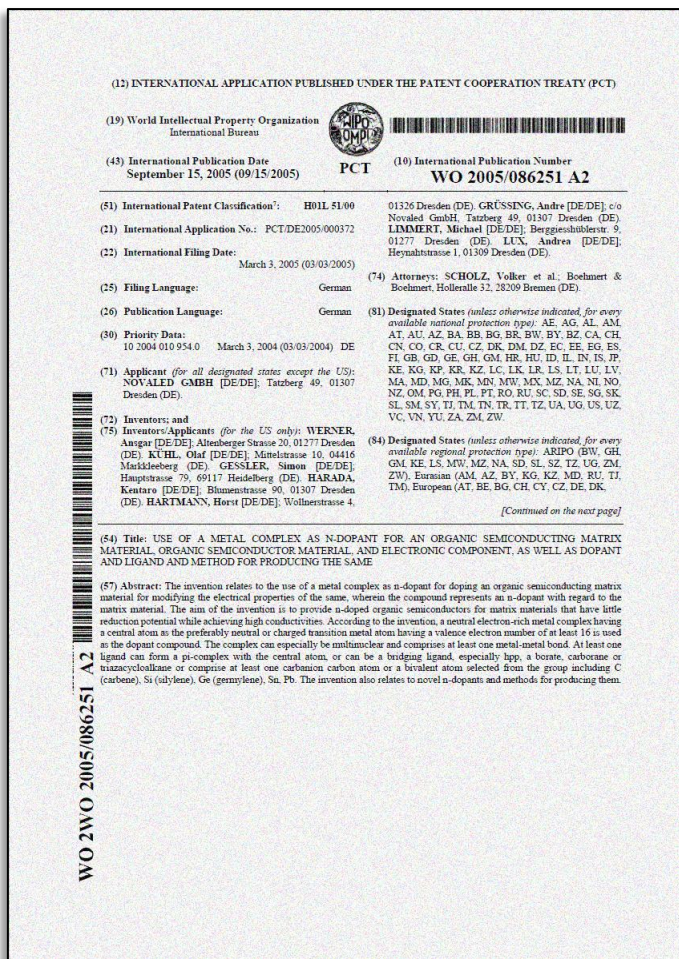
The difference between HOMO and LUMO			
Matrix material	LUMO value	if W ₂ (hpp) ₄ HOMO is 3.6 eV	if W ₂ (hpp) ₄ HOMO is 2.5 eV
BPhen	2.42	1.18	0.08
Ia	2.47	1.13	0.03
Ib	2.42	1.18	0.08
Ic	2.5	1.1	0
Id	2.53	1.07	0.03
Ie	2.51	1.09	0.01

Ex. 1020 Rand Reply Decl., ¶¶ 20-28 (citing Ex.2044 Werner '541, 8:46-53, 4:15-19)

Werner Discloses Joint/Simultaneous Vaporization

Claims 1, 10

Werner Discloses Joint Vaporization For Combining Any Of The Disclosed Metal Complexes And Organic Compounds



Werner

Implementation of doping

The doping of the respective **matrix material** with the **n-dopants** according to the invention can be carried out by one or a combination of the following methods:

a) **Co-evaporation** in vacuum with a source for the matrix material and one for the dopant

Werner, 56:12-18

The provided **metal complex** is evaporated simultaneously with the **matrix material**. According to the example, the matrix material is

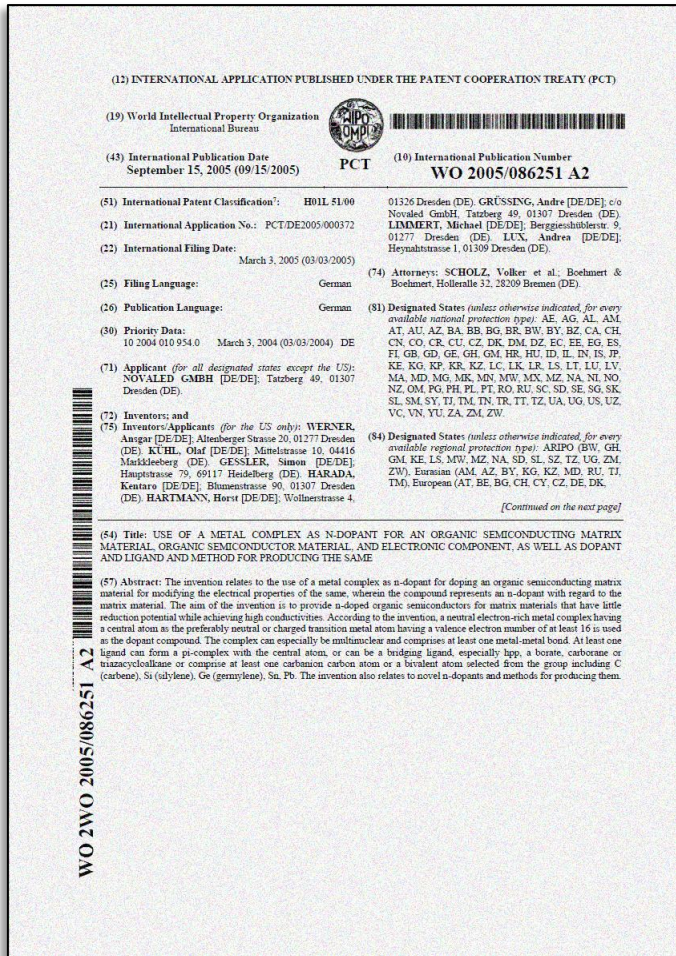
Werner, 61:10-11

Example 2:

A layer of zinc-phthalocyanine (ZnPc) was doped with the metal complex [Cr₂(hpp)₄]. The doped layer was produced by **co-evaporation** of the ZnPc matrix and the dopant Cr₂(hpp)₄ in high vacuum. The

Werner, 62:13-16

Werner Distinguishes Its “Co-Evaporation” From “Sequential Deposition”



Werner

Implementation of doping

The doping of the respective matrix material with the n-dopants according to the invention can be carried out by one or a combination of the following methods:

- Co-evaporation in vacuum with a source for the matrix material and one for the dopant
- Sequential deposition of the matrix material and the n-dopant on a substrate with subsequent diffusion of the dopant, in particular through thermal treatment

Werner, 56:12-22

Prior Art Demonstrates That $W_2(hpp)_4$ Was Vaporized

US007972541B2

(12) **United States Patent**
Werner et al.

(10) **Patent No.:** US 7,972,541 B2
(45) **Date of Patent:** Jul. 5, 2011

(54) **DOPED ORGANIC SEMICONDUCTOR MATERIAL**

(75) Inventors: **Werner, Dresden (DE); Andrea Law, Dresden (DE); Josef Salbeck, Kaufungen (DE)**

(73) Assignee: **Novaled AG, Dresden (DE)**

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 357 days.

(21) Appl. No.: **12/093,167**

(22) PCT Filed: **Nov. 10, 2006**

(86) PCT No.: **PCT/EP2006/010816**

§ 371 (c)(1), (2), (4) Date: **Jul. 21, 2008**

(87) PCT Pub. No.: **WO2007/054345**
PCT Pub. Date: **May 18, 2007**

(65) **Prior Publication Data**
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(30) **Foreign Application Priority Data**
Nov. 10, 2005 (EP) 05024515

(51) **Int. Cl.**
H01L 29/08 (2006.01)
H01L 31/00 (2006.01)
H01L 1/12 (2006.01)
B32B 9/06 (2006.01)

(52) **U.S. Cl.** 252/519.2; 428/690; 257/40; 257/102; 257/103

(58) **Field of Classification Search** 257/40; 257/102, 103, 252/519.2; 428/690
See application file for complete search history.

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3,226,450 A 12/1965 Blazek
3,558,671 A 1/1971 Martin
3,563,751 A 2/1971 Cohen
4,003,943 A 1/1977 Fukunaga
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4,618,455 A 10/1990 Kim
4,960,916 A 10/1990 Paulk et al.
5,093,698 A 3/1992 Egan

(Continued)

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CA 2549969 9/2005 (Continued)

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(Continued)

ABSTRACT

(57) The present invention relates to a doped organic semiconductor material comprising at least one organic matrix material, which is doped with at least one dopant, the matrix material being selected from a group consisting of certain phenanthroline derivatives; and also an organic light-emitting diode which comprises such a semiconductor material.

29 Claims, 2 Drawing Sheets

Pictiva EX2044
Samsung v Pictiva
IPR2024-01094

See application file for complete search history.

www.sciencemag.org SCIENCE VOL 298 6 DECEMBER 2002

Pictiva EX2009
Samsung v Pictiva
IPR2024-01094

1971

“Samples were handled and spectra were obtained using rigorous air-sensitive techniques . . . $W_2(hpp)_4$ sublimed at 250° to 280°C.”

Ex.2009 Cotton et al. (2002), Ref. 16; Ex.1020 Rand Reply Decl., ¶ 35

Using $W_2(hpp)_4$ with matrix materials having evaporation temperatures over 200°C suggests its use in the gas phase

Ex.2044 Werner '541, 8:39-40; Ex.1020 Rand Reply Decl., ¶ 35

Werner Discloses Use Of Its Doped Matrix Materials In OLEDs

Claim 9

Werner Satisfies Claim 9

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(19) World Intellectual Property Organization
International Bureau

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(71) Applicant (for all designated states except the US): NOVALED GMBH [DE/DE]; Tatzberg 49, 01307 Dresden (DE).

(72) Inventors; and
(75) Inventors/Applicants (for the US only): WERNER, Angar [DE/DE]; Altenberger Strasse 20, 01277 Dresden (DE); KÜHL, Olaf [DE/DE]; Mittelstrasse 10, 04416 Markkleeberg (DE); GESSLER, Simon [DE/DE]; Hauptstrasse 79, 69117 Heidelberg (DE); HARADA, Keitaro [DE/DE]; Blumenstrasse 90, 01307 Dresden (DE); HARTMANN, Heiko [DE/DE]; Wollenstrasse 4.

(74) Attorneys: SCHOLZ, Volker et al.; Boehmert & Boehmert, Hollernalle 32, 28209 Bremen (DE).

(81) Designated States (unless otherwise indicated, for every available national protection type): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MY, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every available regional protection type): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LT, LU, LV, MA, MD, ME, NL, NO, PL, PT, RO, RU, SE, SI, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW).

(54) Title: USE OF A METAL COMPLEX AS N-DOPANT FOR AN ORGANIC SEMICONDUCTING MATRIX MATERIAL, ORGANIC SEMICONDUCTOR MATERIAL, AND ELECTRONIC COMPONENT, AS WELL AS DOPANT AND LIGAND AND METHOD FOR PRODUCING THE SAME

(57) Abstract: The invention relates to the use of a metal complex as n-dopant for doping an organic semiconducting matrix material for modifying the electrical properties of the same, wherein the compound represents an n-dopant with regard to the matrix material. The aim of the invention is to provide n-doped organic semiconductors for matrix materials that have little reduction potential while achieving high conductivities. According to the invention, a neutral electron-rich metal complex having a central atom as the preferably neutral or charged transition metal atom having a valence electron number of at least 16 is used as the dopant compound. The complex can especially be multinuclear and comprises at least one metal-metal bond. At least one ligand can form a pi-complex with the central atom, or can be a bridging ligand, especially hpp, a borate, carbamate or triazacycloalkane or comprise at least one carbonium carbon atom or a bivalent atom selected from the group including C (carbene), Si (silylene), Ge (germylene), Sn, Pb. The invention also relates to novel n-dopants and methods for producing them.

WO 2005/086251 A2

9. The organic electronic component according to claim 1, which is formed as an organic LED (OLED) which additionally comprises an organic emitting layer.

'223 Patent, claim 9

Werner

Pictiva does not dispute that Werner



See Pet., 49; Pet. Reply, 16

POR, 24

- Werner, 58:21-60:14

Pet., 49; Pet. Reply, 16

BPhen Coordinates To $W_2(hpp)_4$

Claims 2, 11

Werner Satisfies Claims 2 And 11

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
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(72) Inventors; and
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(84) Designated States (unless otherwise indicated, for every available regional protection type): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, NL, PL, PT, RO, RU, SE, SI, SK, SM, TR, UA, UK, US), African and Asian (AR, AU, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MY, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, VZ, VC, VN, YU, ZA, ZM, ZW).

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(57) Abstract: The invention relates to the use of a metal complex as n-dopant for doping an organic semiconducting matrix material for modifying the electrical properties of the same, wherein the compound represents an n-dopant with regard to the matrix material. The aim of the invention is to provide n-doped organic semiconductors for matrix materials that have little reduction potential while achieving high conductivities. According to the invention, a neutral electron-rich metal complex having a central atom as the preferably neutral or charged transition metal atom having a valence electron number of at least 16 is used as the dopant compound. The complex can especially be multinuclear and comprises at least one metal-metal bond. At least one ligand can form a pi-complex with the central atom, or can be a bridging ligand, especially hpp, a borate, carbamate or naphthalenylcarbazone or comprise at least one carbonium carbon atom or a bivalent atom selected from the group including C (carbene), Si (silylene), Ge (germylene), Sn, Pb. The invention also relates to novel n-dopants and methods for producing them.

WO 2005/086251 A2

2. The organic electronic component according to claim 1, wherein the organic compound coordinates to the metal complex due to the joint vaporization.

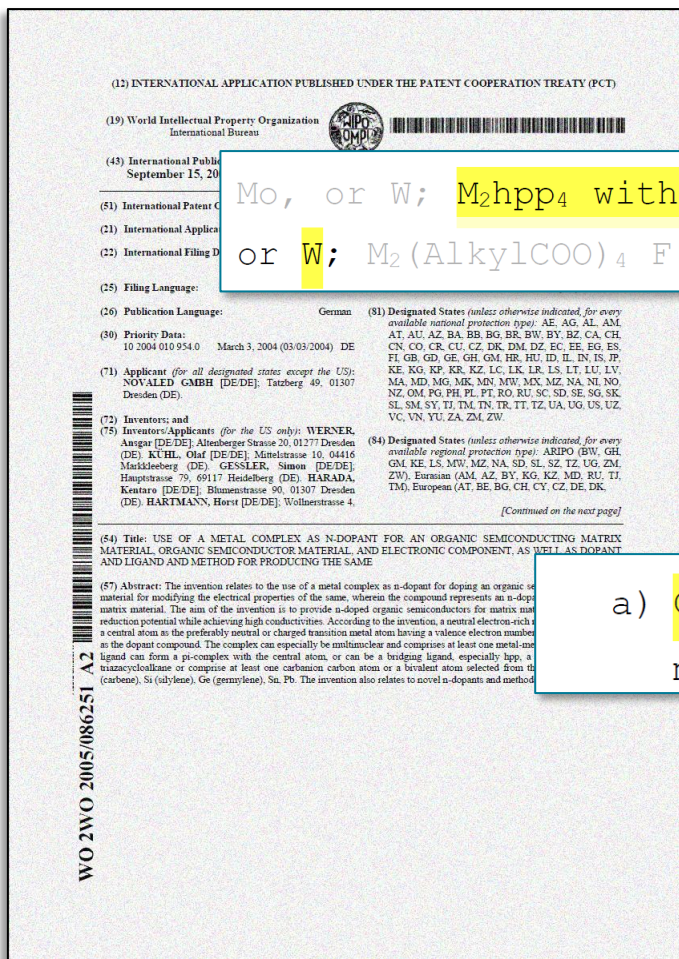
'223 Patent, claim 2

11. The method according to claim 10, wherein during the simultaneous vaporization the organic compound coordinates in the gas phase to the metal complex.

'223 Patent, claim 11

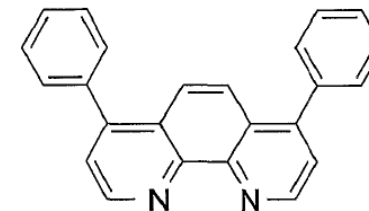
Werner

Werner Demonstrates “Coordination” Through Its Disclosure Of Joint Vaporization Of $W_2(hpp)_4$ And BPhen



Mo, or W; M_2hpp_4 with M equal to Cr, Mn, Fe, Co, Ni, Cu, Zn, Mo, or W; $M_2(AlkylCOO)_4$ F with M equal to Cr, Mn, Fe, Co, Ni, Cu, Zn,

Werner, 60:20-21



BPhen = bathophenanthroline
(4,7-Diphenyl-1,10-phenanthroline)
 $C_{24}H_{16}N_2$

Werner, 54:11-13

a) Co-evaporation in vacuum with a source for the matrix material and one for the dopant

Werner, 56:17-18

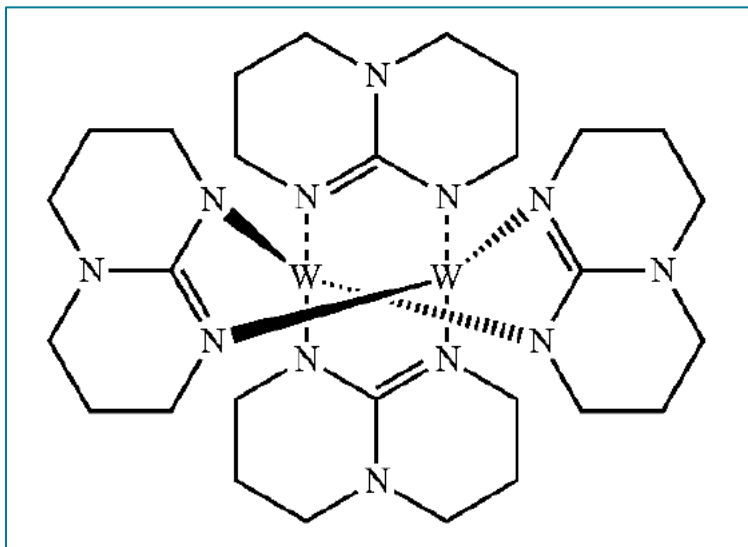
Werner

Pictiva Asserts No Further Evidence Is Required To Demonstrate “Coordination” Beyond Materials And Processing Conditions

are structural features that enable coordination. Co-evaporation results in maximum proximity between the two molecules and allows orbital interaction, enabling the formation of coordinative bonds between the orbitals of the Liq complex and those of compound $^{742}\text{C}_{52}\text{H}_{34}\text{N}_6$. As shown in the examples of Patent-in-Suit 2 and described in para. [0034], no additional technical measure is needed beyond co-evaporation of the compounds – provided that the metal complex and the organic compound are suitable for forming coordination bonds. As shown above, this is true for lithium complexes. As also

Ex. 1025 Complaint in German Proceeding, ¶ 108

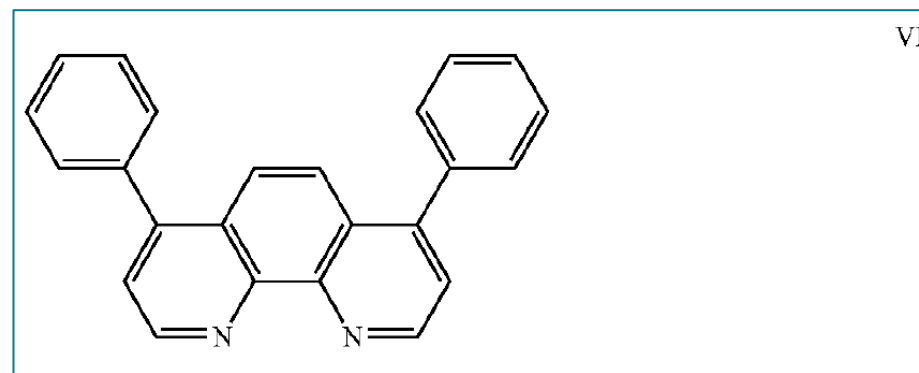
'223 Patent Discloses That Joint Vaporization Of $W_2(hpp)_4$ And BPhen Results In Coordination



'223 Patent, 3:25-37

By virtue of the joint vaporization of the metal complex with an organic compound, these two compounds interact with one another in the gas phase or when they meet one another on the substrate surface. This interaction causes the compounds to be mutually coordinated. The compounds are

'223 Patent, 1:60-63



'223 Patent, Col. 6

By suitable selection of the metal complexes, for example binuclear metal complexes, and suitable selection of the organic compounds such as for example the compounds I to XXIII, the joint vaporization of the metal complex with the organic compound in the gas phase or on the deposited surface results in interactions, so that the compounds coordinate to each other.

'223 Patent, 11:17-23

Werner's Prior Disclosure Renders Claims 2 And 11 Obvious Even Absent Express Discussion Of "Coordination"

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(75) Inventors/Applicants (for the US only): WERNER, Angar [DE/DE]; Altenberger Strasse 20, 01277 Dresden (DE); KÜHL, Olaf [DE/DE]; Maitelstrasse 10, 04146 Markkleeberg (DE); GESSLER, Simon [DE/DE]; Hauptstrasse 79, 69117 Heidelberg (DE); HARADA, Keitaro [DE/DE]; Blumenstrasse 90, 01307 Dresden (DE); HARTMANN, Heide [DE/DE]; Wollnstrasse 4.

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(54) Title: USE OF A METAL COMPLEX AS N-DOPANT FOR AN ORGANIC SEMICONDUCTING MATRIX MATERIAL, ORGANIC SEMICONDUCTOR MATERIAL, AND ELECTRONIC COMPONENT, AS WELL AS DOPANT AND LIGAND AND METHOD FOR PRODUCING THE SAME

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WO 2005/086251 A2



"It is not invention to perceive that the product which others had discovered had qualities they failed to detect."

In re Kubin, 561 F.3d 1351, 1357 (Fed. Cir. 2009)

Werner

BPhen Coordinates To $W_2(hpp)_4$, Thereby Forming Chain-Like Structures

Claims 8, 12

Werner Satisfies Claims 8 And 12

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Novaled GmbH, Tatzberg 49, 01307 Dresden (DE);
LIMMERT, Michael [DE/DE]; Berggasse 9,
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Heynstrasse 1, 01309 Dresden (DE).

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[Continued on the next page]

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ligand can form a pi-complex with the central atom, or can be a bridging ligand, especially hpp, a borate, carbamate or
nitracycloalkane or comprise at least one carbonium carbon atom or a bivalent atom selected from the group including C
(carbene), Si (silylene), Ge (germylene), Sn, Pb. The invention also relates to novel n-dopants and methods for producing them.

WO 2005/086251 A2

8. The organic electronic component according to claim 1, wherein at least parts of the organic compounds bridge two metal complexes together, so that a chain-like structure of the metal complexes is formed and/or at least parts of the organic compounds bridge more than two metal complexes together, so that a network-like structure of the metal complexes is formed.

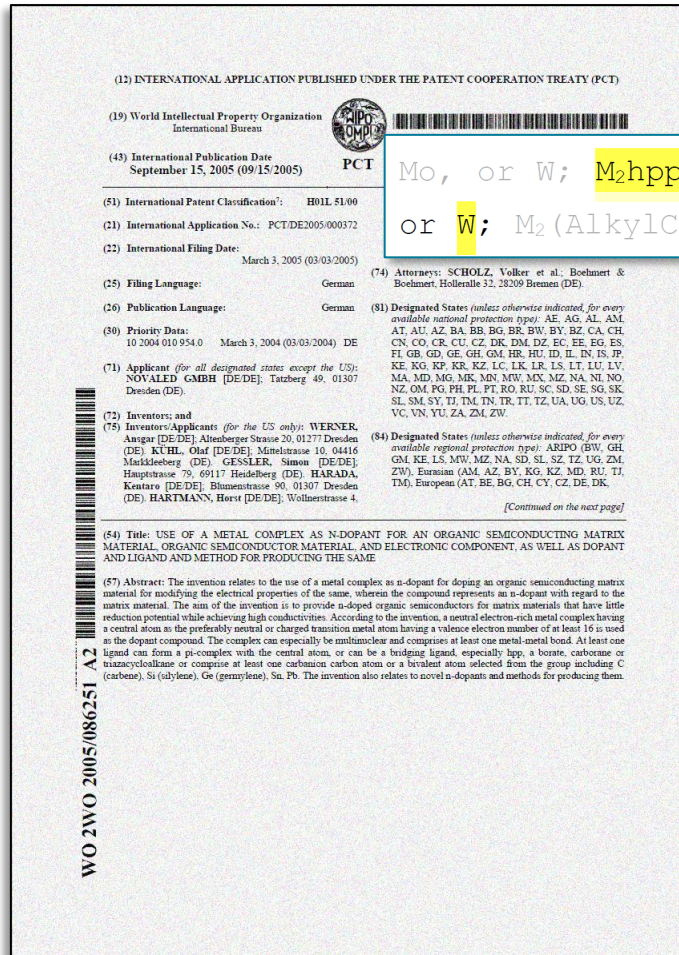
'223 Patent, claim 8

12. The method according to claim 10, wherein the electron-conducting layer is deposited as a chain-like or network-like structure.

'223 Patent, claim 12

Werner

Werner Demonstrates “Chain-Like Structures” Through Its Disclosure Of Joint Vaporization Of $W_2(hpp)_4$ And BPhen

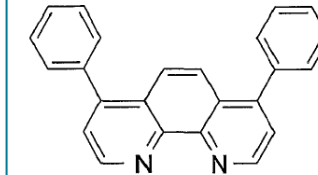


Werner

See Pet., 32-48; Pet. Reply, 15

Mo, or W; M_2hpp_4 with M equal to Cr, Mn, Fe, Co, Ni, Cu, Zn, Mo, or W; $M_2(AlkylCOO)_4$ F with M equal to Cr, Mn, Fe, Co, Ni, Cu, Zn,

Werner, 60:20-21



BPhen = bathophenanthroline
(4,7-Diphenyl-1,10-phenanthroline)
 $C_{24}H_{16}N_2$

Werner, 54:11-13

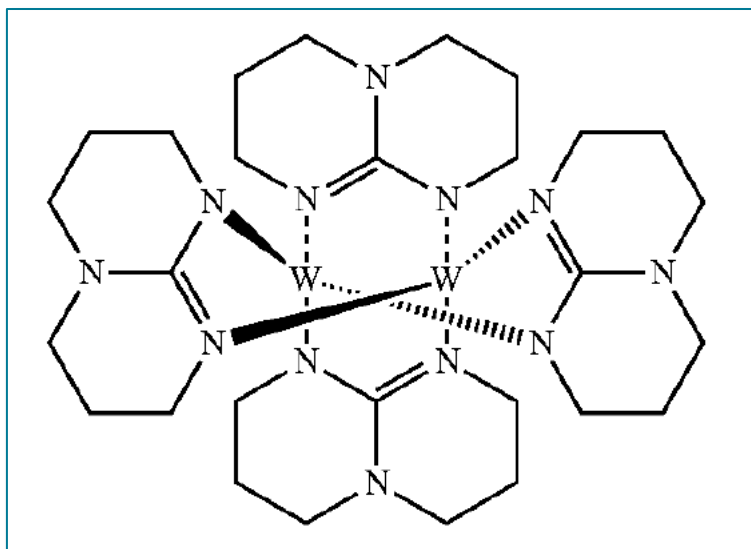
a) Co-evaporation in vacuum with a source for the matrix material and one for the dopant

Werner, 56:17-18

more) metal complexes together. In my opinion, using the same metal complex ($W_2(hpp)_4$), organic compound (BPhen) and vaporization process as in Werner, the '223 patent describes such a combination as forming chain-like or network like structures, wherein at least parts of the organic compounds bridge two (or more) metal complexes together. See, e.g., '223 patent, 1:60-62, 7:48-67, 11:17-26. I address each

Ex. 1003 Rand
Opening Decl., ¶ 86

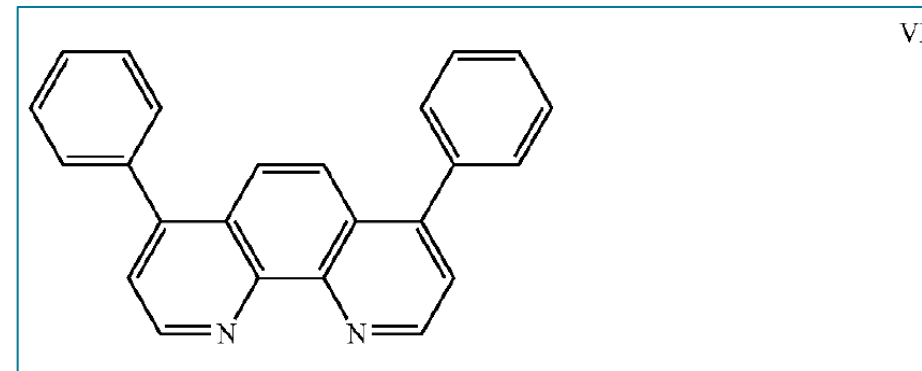
'223 Patent Discloses Joint Vaporization Of $W_2(hpp)_4$ And BPhen, Which Results In Chain-Like Structure



'223 Patent, 3:25-37

another. Metal complexes having two central atoms are particularly well suited for the formation of chains. In this regard,

'223 Patent, 2:49-50



'223 Patent, Col. 6

tion sites for metal complexes. These compounds I to XIII allow not only for the formation of two-dimensional chains, but, by virtue of their multiplicity of coordination sites, they also allow for the formation of three-dimensional networks.

'223 Patent, 7:48-51

Werner's Prior Disclosure Renders Claims 8 And 12 Obvious Even Absent Express Discussion Of "Chain-Like Structures"

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WO 2005/086251 A2



"It is not invention to perceive that the product which others had discovered had qualities they failed to detect."

In re Kubin, 561 F.3d 1351, 1357 (Fed. Cir. 2009)

Werner

Werner Discloses Cross-Linking Controlled
By The Ratio Between The Metal Complex
And Organic Compound

Claim 13

Werner Satisfies Claim 13

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[Continued on the next page]

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13. The method according to claim 12, wherein the degree of cross-linking of the electron-conducting layer is controlled by the ratio between the metal complex and the organic compound during vaporization.

'223 Patent, claim 13

Werner

Werner Discloses Using Different Ratios Of Its Metal Complexes

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(Continued on the next page)

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Preferably, the mentioned metal complexes are used according to the invention as n-dopants, e.g., in a ratio of $\leq 1:1$ or $\leq 1:2$

Werner, 58:4-5

129. It was well-known to a POSA as of the priority date that the electron conductivity increases as the doping ratio of the metal complex to the organic compound increases up to a certain level, but that the electron conductivity decreases when the doping ratio is too high. *See, e.g.,* Ma (Ex.1007) at 812 (illustrating state of the art), Fig. 5. *See infra* Section VIII.B.9.

Ex. 1003 Rand Opening Decl., ¶ 129

Werner

Ground 2 - Ma

Ground 2 - Ma



- J.W. Ma et al., *Organic light-emitting diodes based on new n-doped electron transport layer*, *Synthetic Metals* 158, 810–814 (2008) (“Ma”) is an article published in *Synthetic Metals* and available online as of August 29, 2008

- Ma is directed to “[o]rganic light-emitting diodes [OLEDs] with 8-hydroxy-quinolinato lithium [Liq] doped 4’7-diphenyl-1, 10-phenanthroline [BPhen] as electron transport layer (ETL).”

Ma, 810

Ma

Ground 2 - Issues In Dispute

- Independent Claims
 - Whether Ma is prior art
 - Whether Ma discloses joint vaporization
 - Whether Liq is a metal complex comprising a central metal atom

- Dependent Claims
 - Whether Liq and BPhen coordinate
 - Whether a chain-like structure is formed by Liq and BPhen
 - Whether Ma discloses that the degree of cross-linking of the electron-conducting layer is controlled by the ratio between the metal complex and the organic compound during vaporization

Ma Is Prior Art

Ma Was Available Online As Of August 29, 2008

Synthetic Metals 158 (2008) 810–814

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Synthetic Metals

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Organic light-emitting diodes based on new n-doped electron transport layer

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ABSTRACT

Organic light-emitting diodes with 8-hydroxy-quinolophenanthroline as electron transport layer (ETL), and 4,4',4''-tris(3-methylphenylphenylamino)triphenylamine. The conductivity of carrier transport layers with different and electron-only devices. Compared with the referenced and power efficiency of the p-i-n device are enhanced by improvement is attributed to the improved conductivity balance in the emission zone.

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Ma, 810

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Organic light-emitting electron transport layer

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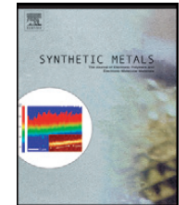


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- As the Patent Owner's expert and Board recognize, Elsevier is an established publisher of scientific articles

citations to relevant caselaw). Elsevier is an established publisher, as demonstrated by Dr. Schanze's testimony. Ex. 2001 ¶ 116. Furthermore, Ma itself indicates the website on which Ma abstract would have been available: "www.elsevier.com/locate/synmet." Ex. 1007, 810. Based on the evidence presented at this juncture, therefore, Petitioner shows sufficiently that an ordinarily skilled artisan would have been able to locate Ma by exercising reasonable diligence. Pet. 16–17; Reply 1–3.

Paper 11 Institution Decision, 19

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Ex. 1018 Garland Decl., ¶ 6

10. The article and abstract from Science Direct shows the date of publication for the first online version. Science Direct populates this information using persistent publication metadata that is embedded in the articles metadata. Once this publication metadata has been first calculated it is not changed.

Ex. 1018 Garland Decl., ¶ 10

11. J. Ma, et. Al., "Organic light-emitting diodes based on new n-doped electron transport layer" was published in Synthetic Metals, Vol. 158, Issues 21-24 and was available online on August 29, 2008. The article is currently available for public download from the Elsevier digital library, Science Direct.

Ex. 1018 Garland Decl., ¶ 11

Ex. 1007 Is the Version Available In August 2008

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■ The Board noted:

Indeed, Ms. Garland testifies that:

The article and abstract from Science Direct shows the date of publication for the first online version. Science Direct populates this information using persistent publication metadata that is embedded in the articles metadata. *Once this publication metadata has been first calculated it is not changed.*

Id. ¶ 10 (emphasis added).

Paper 11 Institution Decision, 17

■ Master of Library and Information Science, June Ann Munford confirmed:

10. In my opinion, Exhibit 1007, with its present substantive content, was made available online as of the date provided on the front of the article: August 29, 2008, via the Elsevier ScienceDirect website.

Ex. 1037 Munford Decl., ¶ 10

Ma Discloses Joint Vaporization

Claims 1 and 10

A POSA Would Interpret “Co-Deposition” In Ma As “Joint Vaporization”

The structure of the complete devices fabricated in this work is as follows:

ITO/*m*-MTDATA:*x* wt.% F₄-TCNQ (40 nm)/NPB (10 nm)/Alq₃ (20 nm)/ BPhen:*y* wt.% Liq (50 nm)/LiF (1 nm)/ Al (130 nm).
Device A (referenced device): *x* = 0, *y* = 0; Device B: *x* = 0, *y* = 33; Device C: *x* = 0.3, *y* = 33; Device D: *x* = 2, *y* = 33; Device E: *x* = 4, *y* = 33.

Then, a 50 nm-thick BPhen:Liq ETL was co-deposited on the EML. In the co-deposition process, organic sources were independently controlled. The deposition rates for the co-deposition layer were monitored using two independent quartz sensors. Finally, a shadow mask was used to define the cathode consisting of 130 nm-thick Al. All the layers (organic layers, and Al electrode) of the devices were fabricated by conventional thermal evaporation in a high vacuum chamber with a base pressure of 1×10^{-5} Torr. The deposition rate was maintained at $0.5 \pm 0.2 \text{ \AA/s}$ for the organic materials, $0.1 \text{ \AA/s}$ for LiF and $5 \pm 2 \text{ \AA/s}$ for Al.

Ma, 811



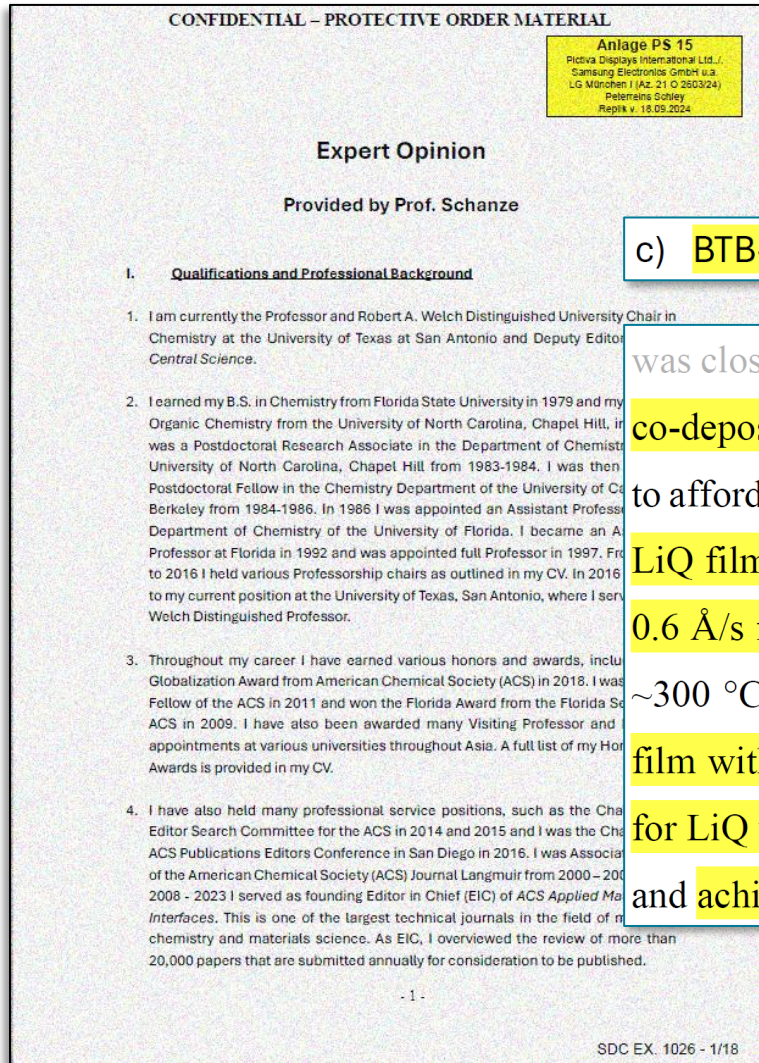
Ma

See Pet. Reply, 21-22

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Demonstrative Exhibit – Not Evidence 48

Dr. Schanze Used The Terms “Co-Deposition” And “Co-Evaporate” To Describe “Joint Vaporization”



c) BTB-LiQ co-deposition

was closed and evacuated to a pressure of 10⁻⁹ mbar. For the BTB-LiQ co-deposited films, the heater power was adjusted in the range 70 – 95% to afford the desired evaporation rate of the two materials. For the BTB-LiQ film with a ratio of 70:30, a rate of 1.4 Å/s for BTB and a rate of 0.6 Å/s for LiQ was used to co-evaporate BTB at ~290 °C and LiQ at ~300 °C and achieve a film thickness of ~200 nm. For the BTB-LiQ film with a ratio of 50:50, a rate of 1 Å/s for BTB and a rate of 1 Å/s for LiQ was used to co-evaporate BTB at ~280 °C and LiQ at ~310 °C and achieve a film thickness of ~200 nm.

12 Q. And you, as the person who oversaw this
13 experiment, did you jointly -- jointly vaporize the
14 B2B and the LiQ?
15 A. To the best of my understanding, yes.

Ex. 1021 Schanze
Tr., 38:12-15

Ex. 1026 Schanze Expert Opinion, ¶ 50;
Ex. 1020, ¶ 52

Ex. 1026

See Pet. Reply, 21-22

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Demonstrative Exhibit – Not Evidence 49

Joint Vaporization Vs. Sequential Vaporization: Two Different Vaporization Methods

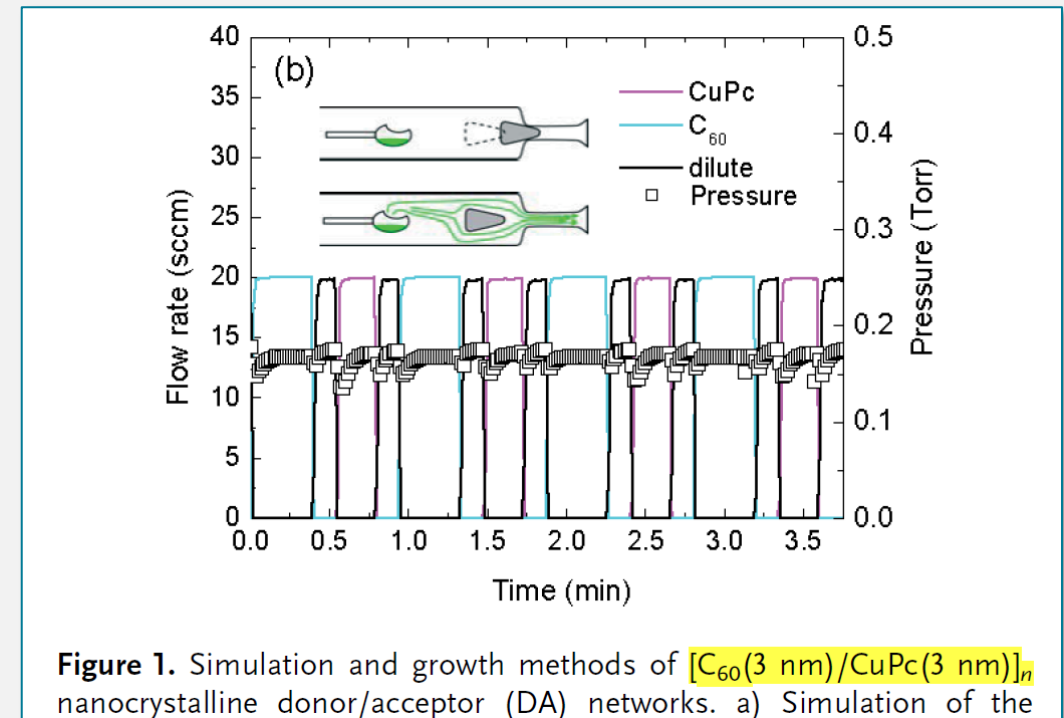
**Joint vaporization (claim 1) /
simultaneous vaporization
(claim 10)**

ZnPc:C₆₀(1:1, 12 nm)

Ex. 1029 Hong, 203505-2, Table 1, Structure B

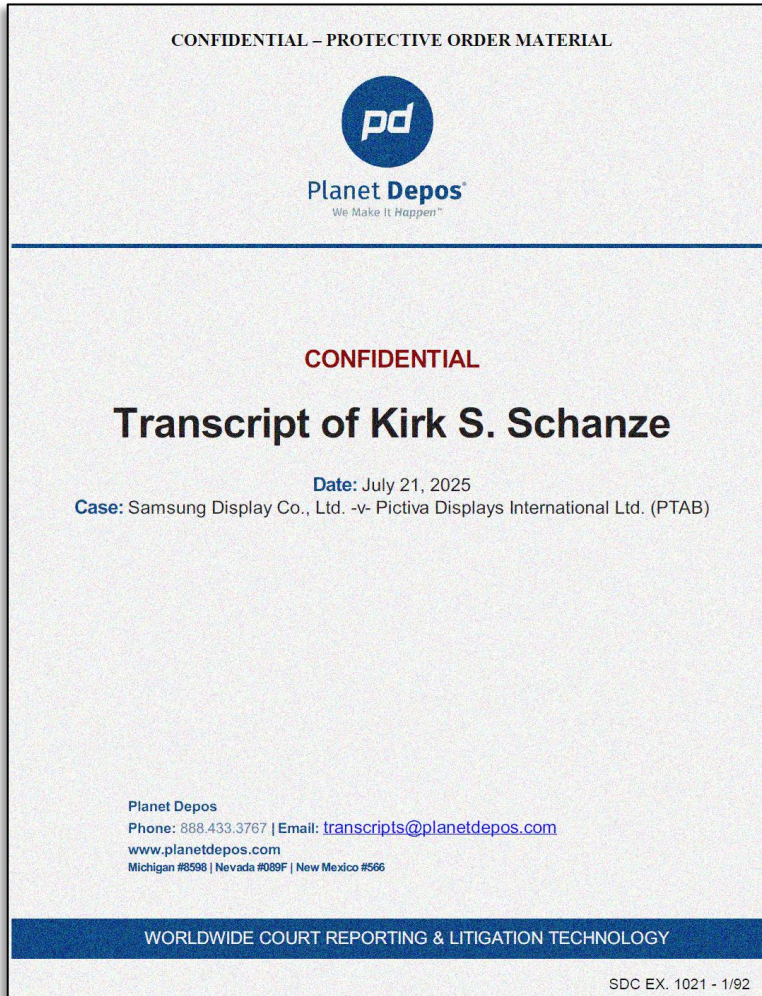
Vs.

Sequential vaporization



Ex. 1028 Yang, 4167, Figure 1

Sequential Vaporization Requires Specific Description



Ex. 1021

See Pet. Reply, 21-22

47. For example, Yang 2007 [Ex.1028] deposited C₆₀ and CuPc by sequential deposition. As Figure 1 and its figure description show, the thickness of each alternating layer is specified as “[C₆₀(3 nm)/CuPc(3 nm)]_n,” meaning 3 nm of C₆₀ and 3 nm CuPc were alternately deposited *n* times. Ex.1028, 4167, Figure 1. For example, Yang 2007 used descriptions like, “[CuPc(6.1 nm)/C₆₀(6.1 nm)]₁₀” and “[CuPc(3.1 nm)/C₆₀(3.1 nm)]₁₇” to clearly indicate how thick each layer was and how many alternating layers were deposited. *Id.* Figure 1(b) shows how long each material was

20 And if one did such a, as you describe it,
21 sequential deposition, one would expect that it
22 would affect the structure of the material and the
23 device, if it was used in a device. And so, you
24 would expect it to be stated.

Ex. 1020 Rand Reply
Declaration, ¶ 47

Ex. 1021 Schanze Tr., 39:20-24; Pet. Reply, 22

Liq Is A Metal Complex Comprising A Central Metal Atom

Claim 1

Liq Is A Metal Complex Comprising A Central Metal Atom Under Pictiva's Infringement Theory

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Organic light-emitting diodes based on electron transport layer
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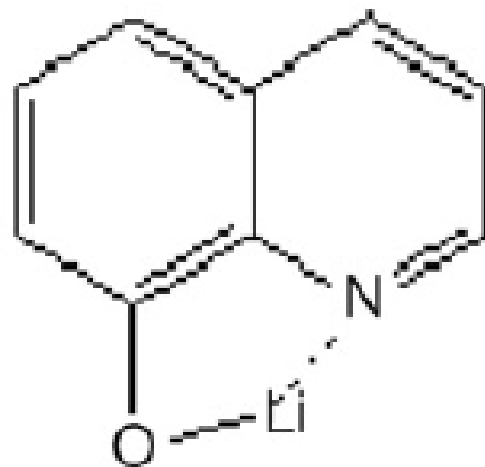
ABSTRACT
Organic light-emitting diodes (OLEDs) based on 4,4',4''-tris(3-methylphenyl)-2,2',2''-tris(methylphenyl)amine (Liq) as electron transport layer (ETL) and p-type (m-MTDATA:x wt.% F₄-TCNQ) layer as HTL. Here, 33 wt.% Liq is chosen due to the fact that the device with the doping ratio of 33 wt.% Liq demonstrates the best current density vs voltage (J–V) curve among all the electron only devices. Using this method, we obtained a significant improvement in the power efficiency of the device.

1. Introduction
Since the discovery of organic light-emitting devices (OLEDs), there has been a considerable interest in developing OLEDs with high efficiency for display applications [1]. In order to lower driving voltage and enhance power efficiency, it is critical to enhance the carrier injection from the electrode to the transporting layer and to increase the transport conductivity [2,3]. The hole injection can be improved by increasing the work function of ITO with different surface treatments such as O₂ plasma or UV-ozone treatment of ITO surface [4]. It can also be enhanced by introducing hole injection layer (HIL), such as copper phthalocyanine (CuPc) [5], starburst polyamines [6], polymeric PEDT:PSS [7] and 4,4',4''-tris(N-(2-naphthyl)-N-phenylamino)triphenylamine (2-TNATA) between the ITO/hole transport layer (HTL) interface. Recently, the p-doping of HTL has drawn a lot of attentions because of its ability to enhance hole injection and lower driving voltages in OLEDs. The p-doping HTL is typically made by co-evaporating the hole transporting materials with a strong electron acceptor like tetrafluoro-tetracyano-quinodimethane (F₄-TCNQ) [8], or oxidants like SbCl₅ [9], FeCl₃ [10] and iodine [11]. p-Doping can

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SDC EX. 1007 - 1/5



Ma, Fig. 1

First, Ma discloses Liq as the metal complex used in the ETL. Ma, 811.

Although the '223 patent is silent as to the meaning of the term “central” in this claim, Patent Owner has specifically identified Liq as a “metal complex” comprising Li as a “central metal atom.” Ex.1003, ¶139. That is, under Patent Owner’s interpretation, which Petitioner adopts for purposes of this specific proceeding,⁸ Ma discloses a metal complex which comprises a central metal atom. Liq comprises the metal atom of lithium (Li), as shown below. Ex.1003, ¶139.

Pet., 58

not be understood as a metal oxide. Second, “central metal atom” just refers to the metal center of a metal complex, and the metal center need not be centrally located or have the properties discussed in preferred embodiments. See '223, 2:21-27, 3:4-

POR, 57

Ma

Pet., 58-59; Pet. Reply, 23

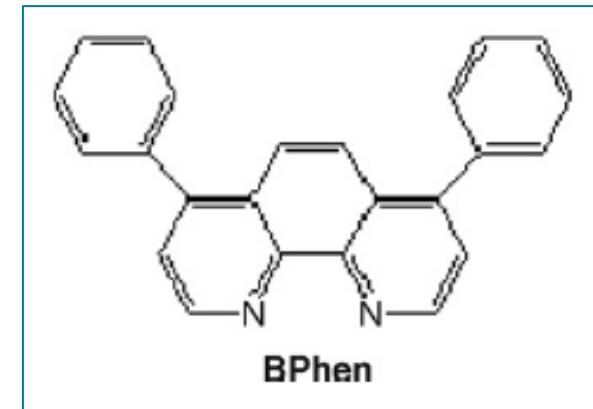
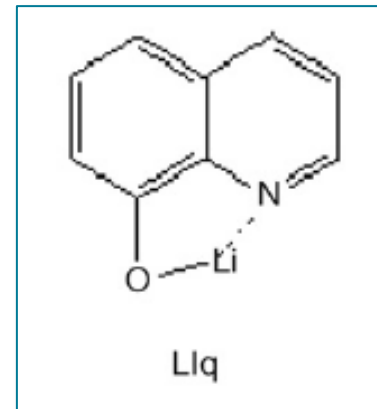
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Demonstrative Exhibit – Not Evidence 53

BPhen Coordinates To Liq

Claims 2 and 11

Ma Discloses Joint Vaporization For Combining Liq And BPhen



Then, a 50 nm-thick BPhen:Liq ETL was co-deposited on the EML.

Ma, 811, Fig. 1

Pictiva Asserts No Evidence Of Coordination Is Needed Beyond Materials And Processing Conditions

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Contains **color** illustrations

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for court costs based on a preliminary estima

21 O 13746/23
Hearing date: December 18, 2024

Munich, March 7, 2024
Our reference: 51112-0002LL1

Statement of Extension of Claim

by **Pictiva Displays International Ltd.**, The Glasshouses GH2, 92
Georges Street Lower, Dun Laoghaire, Dublin,
Ireland, A96 VR66, represented by the
Managing Director Angela Quinlan, *same*
address

– Plaintiff –

Legal counsel: The attorneys of Peterreins Schley Patent- und
Rechtsanwälte PartG mbB, lead counsel Dr.
Thomas Adam, with the involvement of the
patent attorneys of the firm, in particular Patent
Attorney Dipl.-Ing. Felix Glöckler and Patent
Attorney Dipl.-Chem. Markus Coeln,
Hermann-Sack-Str. 3, 80331 Munich

vs.

Dr. Marc Grünwald ^{1,4}
Dr. Simon Reuter ^{1,*}
Carsten Niepelt ^{2,3,*}
Dr. Claudia Feller ^{1,*}
Constanze Wedding ^{1,*}
Stefan Kohl ^{2,3,*}
Martin Obster ^{2,3,*}
Dr. Christoph Horst ^{2,3,*}

¹ Attorney
² Patent Attorney
³ European Patent Attorney
⁴ Specialist Attorney
ⁱⁿ Intellectual Property Law
^{*} Not a partner of the firm

SDC EX. 1025 - 2/47

⁷⁴²C₅₂H₃₄N₆. As shown in the examples of Patent-in-Suit 2 and described in para. [0034], no additional technical measure is needed beyond co-evaporation of the compounds – provided that the metal complex and the organic compound are suitable for forming coordination bonds. As shown above, this is true for lithium complexes. As also

Ex. 1025 Complaint in German Proceeding, ¶ 108

Pictiva Argued BPhen Coordinates To Liq When Jointly Vaporized

CONFIDENTIAL – PROTECTIVE ORDER MATERIAL

Anlage PS 15
Pictiva Displays International Ltd.
Samsung Electronics GmbH
LG München I (Az: 21 O 2603)
Peterehns-Götley
Replik v. 18.09.2024

Expert Opinion

Provided by Prof. Schanze

1. Qualifications and Professional Background

1. I am currently the Professor and Robert A. Welch Distinguished University Chair in Chemistry at the University of Texas at San Antonio and Deputy Editor of *ACS Central Science*.
2. I earned my B.S. in Chemistry from Florida State University in 1979 and my Ph.D. in Organic Chemistry from the University of North Carolina, Chapel Hill, in 1983. I was a Postdoctoral Research Associate in the Department of Chemistry at the University of North Carolina, Chapel Hill from 1983-1984. I was then a Miller Postdoctoral Fellow in the Chemistry Department of the University of California, Berkeley from 1984-1986. In 1986 I was appointed an Assistant Professor in the Department of Chemistry of the University of Florida. I became an Associate Professor at Florida in 1992 and was appointed full Professor in 1997. From 1997 to 2016 I held various Professorship chairs as outlined in my CV. In 2016 I moved to my current position at the University of Texas, San Antonio, where I serve as the Welch Distinguished Professor.
3. Throughout my career I have earned various honors and awards, including the Globalization Award from American Chemical Society (ACS) in 2018. I was made a Fellow of the ACS in 2011 and won the Florida Award from the Florida Section of ACS in 2009. I have also been awarded many Visiting Professor and Lecturer appointments at various universities throughout Asia. A full list of my Honors and Awards is provided in my CV.
4. I have also held many professional service positions, such as the Chair of the Editor Search Committee for the ACS in 2014 and 2015 and I was the Chair of the ACS Publications Editors Conference in San Diego in 2016. I was Associate Editor of the American Chemical Society (ACS) Journal *Langmuir* from 2000–2008. From 2008–2023 I served as founding Editor in Chief (EIC) of *ACS Applied Materials & Interfaces*. This is one of the largest technical journals in the field of materials chemistry and materials science. As EIC, I overviewed the review of more than 20,000 papers that are submitted annually for consideration to be published.

- 1 -

SDC EX. 1026 - 1/18

39. Kang *et al.* report in exhibit 11 on page 140207, right col:

“the complexation of ETL (BPhen) and Liq was proved by analyses of the Gibbs – DiMarzio prediction and X-ray photoelectron spectroscopy[16].”

40. As a side remark, for the benefit of the Court, the reference to complexation in the above quote means the same as coordination in the sense of the patent-in-suit, i.e. reference is made to a coordination of LiQ with the pyridinic nitrogen atoms of BPhen. For further information, BPhen has the following structure:

Ex. 1026 Schanze Expert Opinion, ¶¶ 39-40

*Reference [16] is to Ki (Ex. 2050)

Ex. 1026

See Pet. Reply, 23-25

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Demonstrative Exhibit – Not Evidence 57

Ma And Ki Used The Same Processing Conditions To Jointly Vaporize Liq And BPhen

Ki

Materials and sample preparation

The silicon wafer substrates were blown dry with nitrogen gas and then treated by UV–ozone for 15 min prior to loading into a thermal evaporation system. Each film of ≈ 200 nm was deposited by a thermal co-evaporation at a base pressure of $\approx 10^{-7}$ torr. The mixing percentage was controlled from the deposition rates of two source materials, which were individually monitored by two quartz crystal microbalances. The sum of deposition rates of the two materials was kept at $\approx 2 \text{ \AA s}^{-1}$. The mixing percentage presented in the manuscript is volume percentage unless specified otherwise. Both encapsulated and as-deposited samples were produced, and the encapsulation process was conducted under vacuum.

Ex. 1031 Ki, Supporting Information, 1

Ma

Then, a 50 nm-thick BPhen:Liq ETL was co-deposited on the EML. In the co-deposition process, organic sources were independently controlled. The deposition rates for the co-deposition layer were monitored using two independent quartz sensors. Finally, a shadow mask was used to define the cathode consisting of 130 nm-thick Al. All the layers (organic layers, and Al electrode) of the devices were fabricated by conventional thermal evaporation in a high vacuum chamber with a base pressure of 1×10^{-5} Torr. The deposition rate was maintained at $0.5 \pm 0.2 \text{ \AA/s}$ for the organic materials, $0.1 \text{ \AA/s}$ for LiF and $5 \pm 2 \text{ \AA/s}$ for Al.

Ma, 811

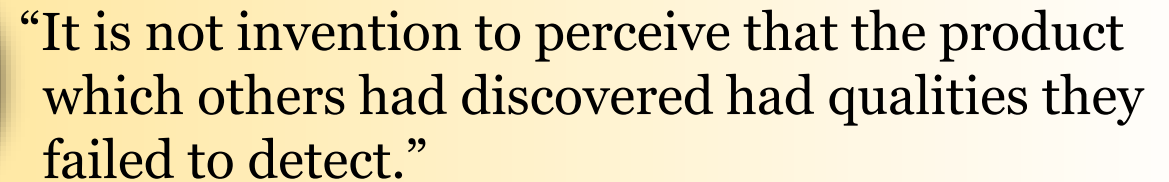
Ma

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E-mail address: majunwei250802944@163.com (J.W. Ma).

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SDC EX. 1007 - 1/5



In re Kubin, 561 F.3d 1351, 1357 (Fed. Cir. 2009)

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ABSTRACT

Organic light-emitting diodes with 8-hydroxy-quinolinato lithium dopant 4,4'-diphenyl-1, 10-phenanthroline as electron transport layer (ETL), and tetrafluoro-tetracyano-quinodimethane dopant 4,4',4''-tris (3-methylphenyl)phenylamino triphenylamine as hole transport layer (HTL) are demonstrated. The conductivity of carrier transport layers with different dopant concentration is examined by hole-only and electron-only devices. Compared with the reference device (without dopant), the current efficiency and power efficiency of the p-i-n device are enhanced by approximately 51% and 89%, respectively. This improvement is attributed to the improved conductivity of the transport layers and the efficient charge balance in the emission zone.

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1. Introduction

Since the discovery of organic light-emitting devices (OLEDs), there has been a considerable interest in developing OLEDs with high efficiency for display applications [1]. In order to lower driving voltage and enhance power efficiency, it is critical to enhance the carrier injection from the electrode to the transporting layer and to increase the transport conductivity [2,3]. The hole injection can be improved by increasing the work function of ITO with different surface treatments such as O_2 plasma or UV-ozone treatment of ITO surface [4]. It can also be enhanced by introducing hole injection layer (HIL), such as copper phthalocyanine (CuPC), Si_3 starburst polymynes [6], polymeric PEDT-PSS [7] and 4,4'-bis-(*N*-(2-naphthyl)-*N*-phenylamino)triphenylamine (2-TNATA) between the ITO/hole transport layer (HTL) interface.

Recently, the p-doping of HTL has drawn a lot of attentions because of its ability to enhance hole injection and lower driving voltages in OLEDs. The p-doping HTL is typically made by co-evaporating the hole transporting materials with a strong electron acceptor like tetrafluoro-tetracyano-quinodimethane (F_4-TCNQ) [8], or oxidants like $SbCl_5$ [9], $FeCl_3$ [10] and iodine [11]. p-Doping can

also achieve ohmic conductivity to reduce driving voltage of device and the proper control of doping levels can also lead to efficient carrier injection by tunnelling [12].

However, organic molecules are known to be poor in electron injection and transport compared with that of hole. Many attempts have been made to improve the electron injection and transport, including using low work function metals [113–115] and organic molecules [116–118]. In particular, the use of electron transporting layer [16–20] to enhance the electron injection. Great efforts have been made to enhance the electron conductivity [21–24]. Kido and Matsumoto [22] reported the improved electron transport by doping Li into tri(8-hydroxyquinoline) aluminum (Alq₃). Feng et al. [21] also demonstrated that the device performance can be improved by using a lithium salt of 10-phenanthroline (Bphen) into Alq₃ as a cohost electron transport layer (c-ETL). However, few studies have been reported about using lithium quinolinol complex such as 8-hydroxy-quinolinato lithium (LiQ) as an electron injection layer [25], and no work has yet been reported about using Liq-doped Bphen as electron transport layer (EPET) [26].

In this paper, we have demonstrated a device with an n-type n-doping (33 wt.% Liq:Bphen) layer as ETL and p-type (M-TDAta:x wt.% F₃C-TCNQ) layer as HTL. Here, 33 wt.% Liq is chosen due to the fact that the device with the doping ratio of 33 wt.% Liq demonstrates the best current density vs voltage (*J*-*V*) curve among all the electron only devices. Using this method, we obtained a sig-

Pictiva Presents Inconsistent Positions

42. Thus, there is direct experimental evidence of the pyridinic nitrogen atoms of BPhen coordinating to the LiQ metal complex.

Ex. 1026 Schanze Expert Opinion, ¶ 42

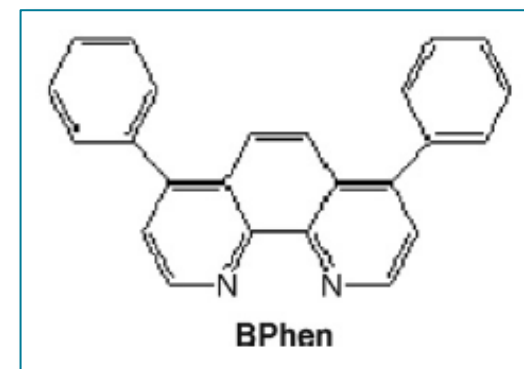
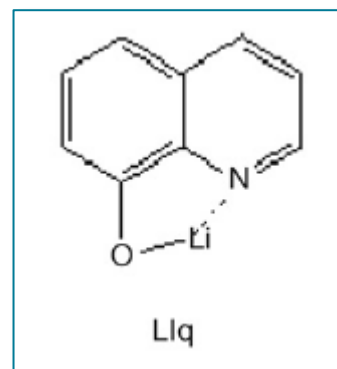
request), ¶26 (“unclaimed compound BPhen”). As such, Petitioner’s reliance on statements in the German proceeding is misplaced and those statements do not support Petitioner’s argument that in Ma, BPhen coordinates to Liq. EX1032, ¶¶26-

PO’s Sur-Reply, 23

Liq And BPhen Form A Chain-Like Structure

Claims 8 and 12

Ma Discloses Joint Vaporization Of Liq And BPhen



Then, a 50 nm-thick BPhen:Liq ETL was co-deposited on the EML.

Ma, 811, Fig. 1

154. Moreover, Ma discloses the same vaporization process that, according to the '223 patent, ('223 patent, 1:60–62; *see also id.*, 11:17–29), allows for formation of chain-like or network like structures, wherein *at least parts of the organic compounds bridge two (or more) metal complexes together*. Ma discloses

Ex. 1003 Rand Opening Decl., ¶ 154

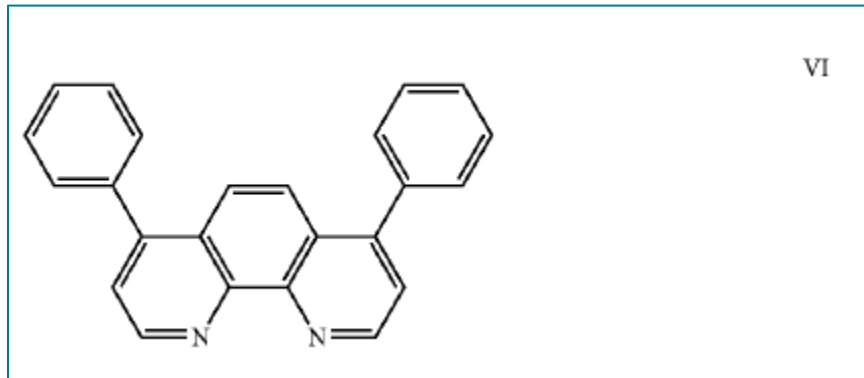
Ma

See Pet., 65–66

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Demonstrative Exhibit – Not Evidence 62

'223 Patent Discloses BPhen Has Coordination Sites, Which Results In Chain-Like Structure



BPhen

'223 Patent, Col. 6

The compounds illustrated in the structures I to XIII are particularly well suited as an organic compound for the electron-conducting layer. Each of these compounds comprises both an aromatic compound and also a nitrogen heterocycle. Each of these compounds thus comprises several coordination sites for metal complexes. These compounds I to XIII allow not only for the formation of two-dimensional chains, but, by virtue of their multiplicity of coordination sites, they also allow for the formation of three-dimensional networks.

'223 Patent, 7:43-51

component. The formation of chain-like or network-like structures enables additional electrons to be able to be transferred from the metal complexes to the organic compounds, said electrons are then available for charge transport in the electron-conducting layer. In this regard, reference is made to

'223 Patent, 11:29-33

Ma's Prior Disclosure Renders Claims 8 And 12 Obvious



“It is not invention to perceive that the product which others had discovered had qualities they failed to detect.”

In re Kubin, 561 F.3d 1351, 1357 (Fed. Cir. 2009)

Ma

See Pet., 65–66; Pet. Reply, 25

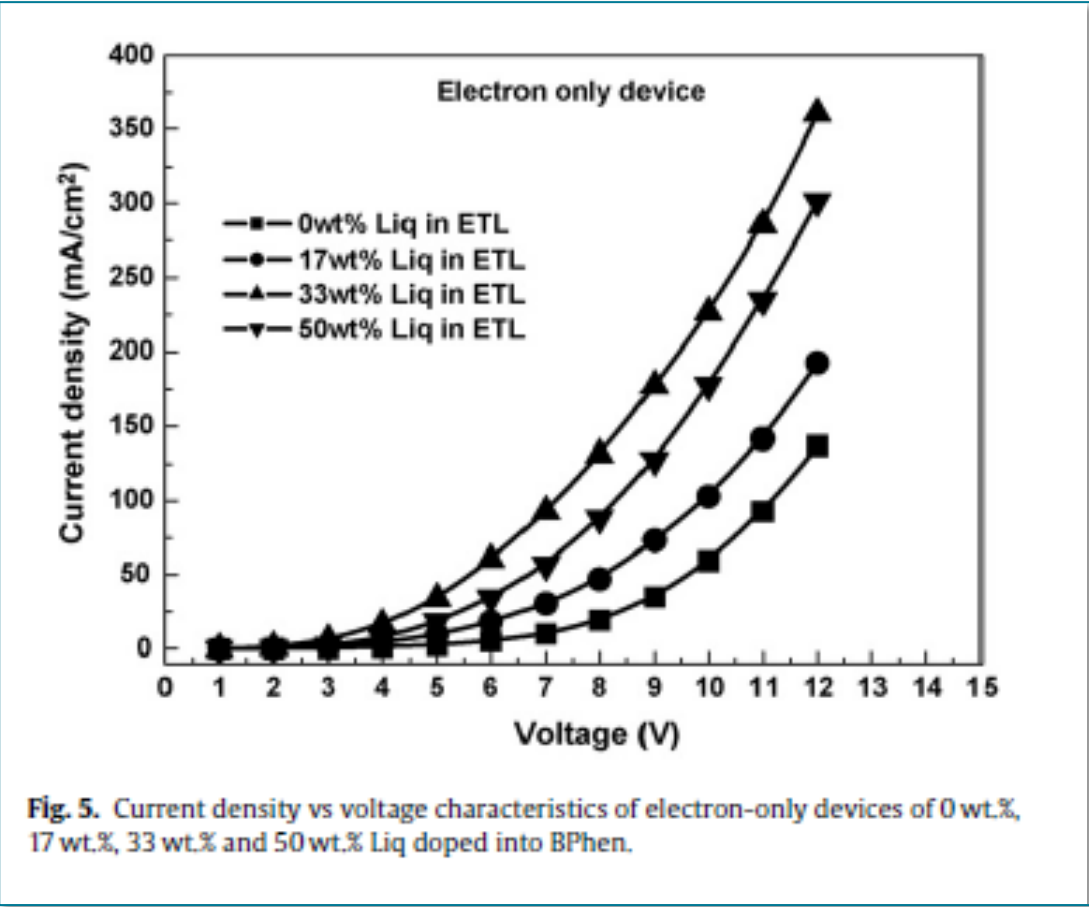
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Demonstrative Exhibit – Not Evidence 64

Ma Discloses Cross-Linking Controlled By
The Ratio Between The Metal Complex
And Organic Compound

Claim 13

Electron Conductivity Is Controlled By The Ratio Between The Metal Complex And The Organic Compound



Ma, 812, Fig. 5

Ma

See Pet., 72-73

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Demonstrative Exhibit – Not Evidence 66

Werner Back-up Slides

It Is Not Disputed That Werner Satisfies Claims 3, 4, 6, And 14

3. The organic electronic component according to claim 1, wherein the metal complex comprises more than one central metal atom.

Application examples

An electron-rich neutral metal complex according to the invention is provided, where the metal complex can be, for example:

M-bis-(terpyridine) with M equal to Cr, Mn, Fe, Co, Ni, Cu, Zn, Mo, or W; M_2hpp_4 with M equal to Cr, Mn, Fe, Co, Ni, Cu, Zn, Mo, or W; $M_2(AlkylCOO)_4$ F with M equal to Cr, Mn, Fe, Co, Ni, Cu, Zn,

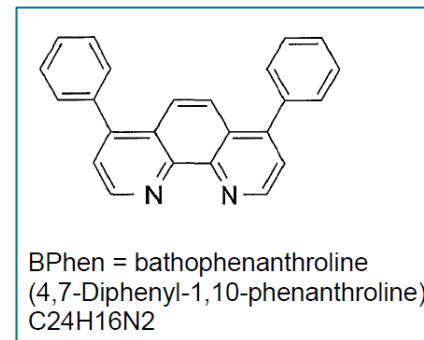
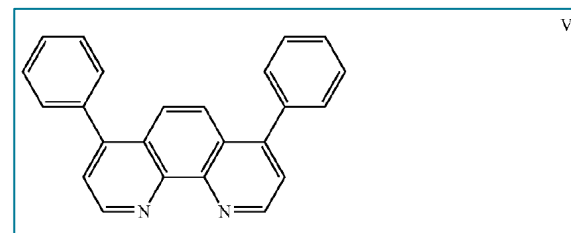
Werner, 60:16-21

4. The organic electronic component according to claim 1, wherein the central metal atom or central metal atoms is/are selected from Cr, Mo or W.

Mo, or W; M_2hpp_4 with M equal to Cr, Mn, Fe, Co, Ni, Cu, Zn, Mo, or W; $M_2(AlkylCOO)_4$ F with M equal to Cr, Mn, Fe, Co, Ni, Cu, Zn,

Werner, 60:20-21

6. The organic electronic component according to claim 1, wherein the organic compound is selected from:



Werner, 54:11-13

14. The method according to claim 10, wherein the organic compound does not coordinate to the metal complex prior to vaporization.

The doping of the respective matrix material with the n-dopants according to the invention can be carried out by one or a combination of the following methods:

- a) Co-evaporation in vacuum with a source for the matrix material and one for the dopant

Werner, 56:13-18

Additional Prior Art Specifically Combines $W_2(hpp)_4$ And BPhen

US007972541B2

(12) **United States Patent**
Werner et al.

(10) **Patent No.:** US 7,972,541 B2
(45) **Date of Patent:** Jul. 5, 2011

(54) **DOPED ORGANIC SEMICONDUCTOR MATERIAL**

(75) **Inventors:** Aungar Werner, Dresden (DE); Andrea Lux, Dresden (DE); Josef Salbeck, Kaufungen (DE)

(73) **Assignee:** Novaled AG, Dresden (DE)

(*) **Notice:** Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 357 days.

(21) **Appl. No.:** 12/093,167

(22) **PCT Filed:** Nov. 10, 2006

(86) **PCT No.:** PCT/EP2006/010816
§ 371 (c)(1), (2), (4) **Date:** Jul. 21, 2008

(87) **PCT Pub. No.:** WO2007/054345
PCT Pub. Date: May 18, 2007

(65) **Prior Publication Data**
US 2009/0001327 A1 Jan. 1, 2009

(30) **Foreign Application Priority Data**
Nov. 10, 2005 (EP) 05024515

(51) **Int. Cl.**
H01L 29/08 (2006.01)
H01L 53/00 (2010.01)
H01B 1/12 (2006.01)
B32B 9/06 (2006.01)

(52) **U.S. Cl.:** 252/519.2; 428/690; 257/40; 257/102; 257/103

(58) **Field of Classification Search**
257/102, 103; 252/519.2; 428/690
See application file for complete search history.

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(Continued)

Primary Examiner — Mark Kopec
Assistant Examiner — Jason P Thomas
(74) **Attorney, Agent, or Firm** — Sutherland, Asbill & Brennan, LLP

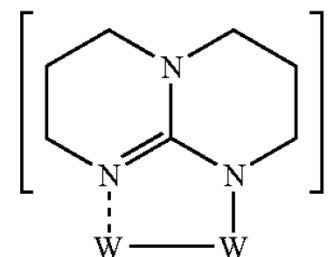
(57) **ABSTRACT**
The present invention relates to a doped organic semiconductor material comprising at least one organic matrix material, which is doped with at least one dopant, the matrix material being selected from a group consisting of certain phenanthroline derivatives; and also an organic light-emitting diode which comprises such a semiconductor material.

29 Claims, 2 Drawing Sheets

Pictiva EX2044
Samsung v Pictiva
IPR2024-01094

Ex. 2044

fusion problem. For this reason, a dopant having the structure IVa



structure IVa = $W(hpp)_4$

was tested as dopant for conventional electron transport materials, such as Alq_3 (tris(8-hydroxyquinoline) aluminium) or BPhen (4,7-diphenyl-1,10-phenanthroline).

Ex. 2044 Werner '541, 3:64-4:14

BPhen Coordinates To $W_2(hpp)_4$ In The Gas Phase

Because of the simultaneous vaporization of metal complexes and organic compounds, both compounds are simultaneously present next to each other in the gas phase. This enables the organic compound to coordinate to the metal complex in the gas phase. The coordination can be effected

'223 Patent, 13:36-40; Pet., 39

"It is not invention to perceive that the product which others had discovered had qualities they failed to detect."

In re Kubin, 561 F.3d 1351, 1357 (Fed. Cir. 2009)



(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
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(43) International Publication Date
September 15, 2005 (09/15/2005)

(10) International Publication Number
WO 2005/086251 A2

(51) International Patent Classification: H01L 51/00

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(22) International Filing Date: March 3, 2005 (03/03/2005)

(25) Filing Language: German

(26) Publication Language: German

(30) Priority Data: 10 2004 010 954 0 March 3, 2004 (03/03/2004) DE

(71) Applicant (for all designated states except the US): NOVALED GMBH [DE/DE]; Tatzberg 49, 01307 Dresden (DE).

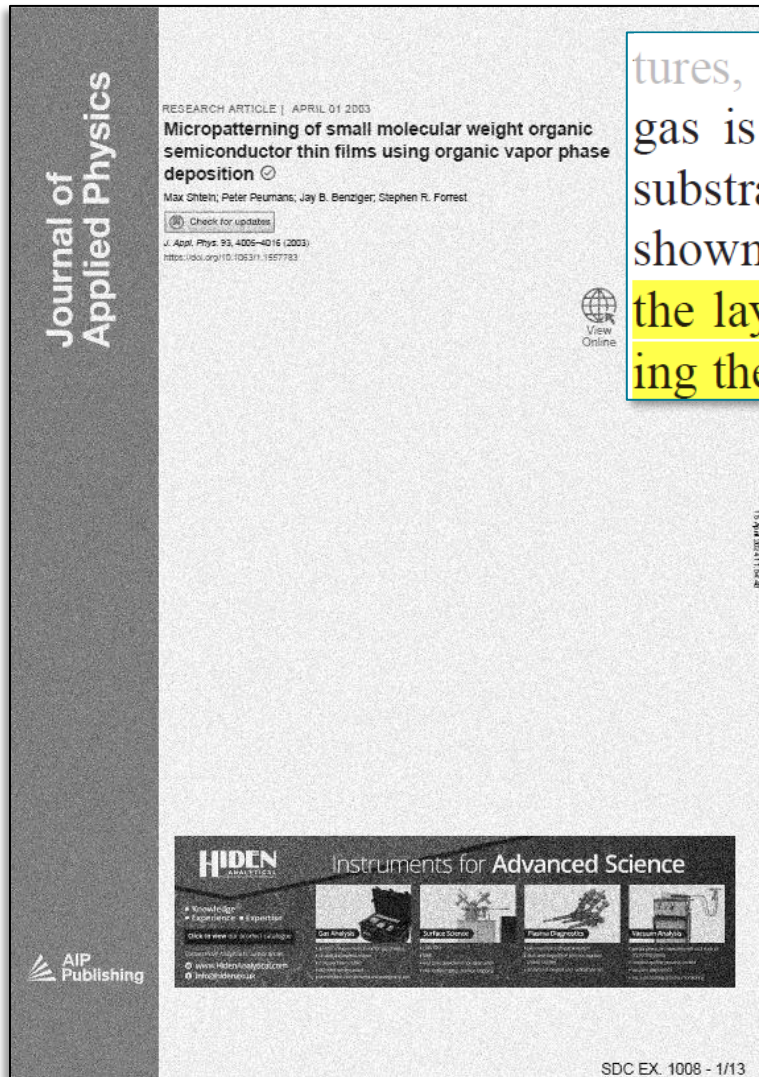
(72) Inventors; and
(75) Inventors/Applicants (for the US only): WERNER, Ansgar [DE/DE]; Altenberger Strasse 20, 01277 Dresden (DE); KÜHL, Olaf [DE/DE]; Mittelstrasse 10, 04416 Markkleeberg (DE); GESSLER, Simon [DE/DE]; Hauptstrasse 79, 69117 Heidelberg (DE); HARADA, Keitaro [DE/DE]; Blumenstrasse 90, 01307 Dresden (DE); HARTMANN, Horst [DE/DE]; Wollnerstrasse 4, [Continued on the next page]

(54) Title: USE OF A METAL COMPLEX AS N-DOPANT FOR AN ORGANIC SEMICONDUCTING MATRIX MATERIAL, ORGANIC SEMICONDUCTOR MATERIAL, AND ELECTRONIC COMPONENT, AS WELL AS DOPANT AND LIGAND AND METHOD FOR PRODUCING THE SAME

(57) Abstract: The invention relates to the use of a metal complex as n-dopant for doping an organic semiconducting matrix material for modifying the electrical properties of the same, wherein the compound represents an n-dopant with regard to the matrix material. The aim of the invention is to provide n-doped organic semiconductors for matrix materials that have little reduction potential while achieving high conductivities. According to the invention, a neutral electron-rich metal complex having a central atom as the preferably neutral or charged transition metal atom having a valence electron number of at least 16 is used as the dopant compound. The complex can especially be multinuclear and comprises at least one metal-metal bond. At least one ligand can form a pi-complex with the central atom, or can be a bridging ligand, especially hpp, a borate, carborane or triazacycloborane or comprise at least one carbanion carbon atom or a bivalent atom selected from the group including C (carbene), Si (silylene), Ge (germylene), Sn, Pb. The invention also relates to novel n-dopants and methods for producing them.

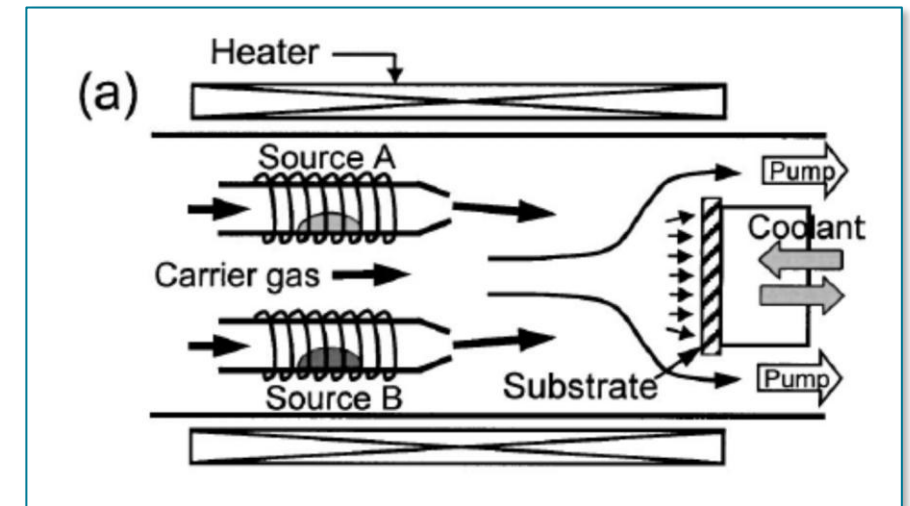
WO 2005/086251 A2

Organic Vapor Phase Deposition (OVPD) Method



tures, and devices.^{20–24} In OVPD, a hot, nonreactive carrier gas is used to transport an organic vapor toward a cooled substrate onto which the organic materials condense, as shown in Fig. 1(a). The method affords precise control over the layer thickness and dopant concentration, while increasing the materials use efficiency over that obtained by VTE.²³

Ex. 1008 Shtein, 4006

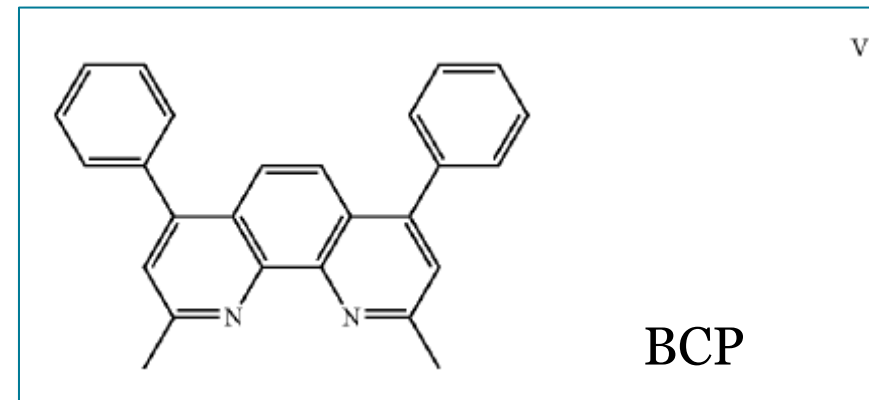


Ex. 1008 Shtein, Fig. 1(a)

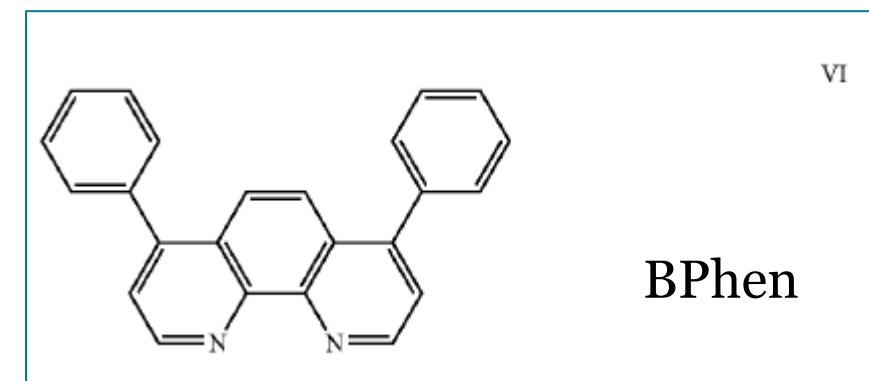
Evidence Of Other $W_2(hpp)_4$ –Doped Phenanthrolines Confirms That POSA Would Have Had An Expectation of Success

the organic compound of the structure V, was used for the electron-conducting layers 3 of the three components in all three measurements. In case of the measurement curve 21, a proportion of 10 mol % of $W_2(TBD-A)_4$ is present as metal complex in the organic compound V. In case of the measurement curve 22, the proportion of $W_2(TBD-A)_4$ as metal complex is only 1 mol %. The measurement curve 23 was produced with the pure organic compound V, which means without a metal complex. The graph illustrated in FIG. 5 shows that, even with a proportion of 1 mol % (measurement curve 22) of the metal complex, very good current densities can be achieved at higher voltages. Even at a low voltage, the current densities achieved with 1 mol % of the metal complex (measurement curve 22) are already considerably better than the current densities which are achieved with the pure organic compound (measurement curve 23). The current densities

'223 Patent, 17:1-16



'223 Patent, Col. 5



'223 Patent, Col. 6

Improving Conductivity By Two Magnitudes Does Not Teach Away But Rather Demonstrates Success

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)	
(19) World Intellectual Property Organization International Bureau	
(43) International Publication Date September 15, 2005 (09/15/2005)	(10) International Publication Number WO 2005/086251 A2
(51) International Patent Classification: H01L 51/00	(74) Attorneys: SCHOLZ, Volker et al.; Boehmer & Boehmer, Hollerland 32, 28209 Bremen (DE)
(21) International Application No.: PCT/DE2005/000372	(8) Designated States (unless otherwise indicated, for every available national protection type): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MY, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW.
(22) International Filing Date: March 3, 2005 (03/03/2005)	(84) Designated States (unless otherwise indicated, for every available regional protection type): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LI, LU, NL, PT, SE, SI, SK, TR, UK, US), OAPI (BF, BJ, GM, GN, GU, KE, ML, MR, NE, NG, SN, TD, TG).
(25) Filing Language: German	
(26) Publication Language: German	
(30) Priority Data: 10 2004 010 954.0 March 3, 2004 (03/03/2004) DE	
(71) Applicant (for all designated states except the US): NOVALED GMBH (DE/DE); Tatzberg 49, 01307 Dresden (DE).	
(72) Inventors; and (73) Inventors/Applicants (for the US only): WERNER, Angar (DE/DE); Altenberger Strasse 20, 01277 Dresden (DE); KÜHL, Olaf (DE/DE); Mittelstrasse 10, 04416 Markkleeberg (DE); GESSLER, Simon (DE/DE); Hauptstrasse 79, 69117 Heidelberg (DE); HARADA, Keitaro (DE/DE); Blumenstrasse 90, 01307 Dresden (DE); HARTMANN, Heide (DE/DE); Wollnstrasse 4.	
(54) Title: USE OF A METAL COMPLEX AS N-DOPANT FOR AN ORGANIC SEMICONDUCTING MATRIX MATERIAL, ORGANIC SEMICONDUCTOR MATERIAL, AND ELECTRONIC COMPONENT, AS WELL AS DOPANT AND LIGAND AND METHOD FOR PRODUCING THE SAME	
(57) Abstract: The invention relates to the use of a metal complex as n-dopant for doping an organic semiconducting matrix material for modifying the electrical properties of the same, wherein the compound represents an n-dopant with regard to the matrix material. The aim of the invention is to provide n-doped organic semiconductors for matrix materials that have little reduction potential while achieving high conductivities. According to the invention, a neutral electron-rich metal complex having a central atom as the preferably neutral or charged transition metal atom having a valence electron number of at least 16 is used as the dopant compound. The complex can especially be multinuclear and comprises at least one metal-metal bond. At least one ligand can form a pi-complex with the central atom, or can be a bridging ligand, especially hpp, a borate, carbene or diazacycloalkane or comprise at least one carbonyl carbon atom or a bivalent atom selected from the group including C (carbene), Si (silylene), Ge (germylene), Sn, Pb. The invention also relates to novel n-dopants and methods for producing them.	

Preferably, the mentioned metal complexes are used according to the invention as n-dopants, e.g., in a ratio of $\leq 1:1$ or $\leq 1:2$, or as detailed above. Using the metal complexes as n-dopants according to the invention, semiconducting layers with conductivities at room temperature in the range of 10^{-5} S/cm or higher can be achieved when using fullerene C₆₀ as the matrix, for example, 10^{-3} S/cm or higher, or 10^{-1} S/cm. When using phthalocyanine-zinc as the matrix, a conductivity of higher than 10^{-8} S/cm, for example 10^{-6} S/cm, was achieved. It was previously not possible to dope this matrix with organic donors due to the low reduction potential of the matrix. The conductivity of undoped phthalocyanine zinc, however, is at most 10^{-10} S/cm.

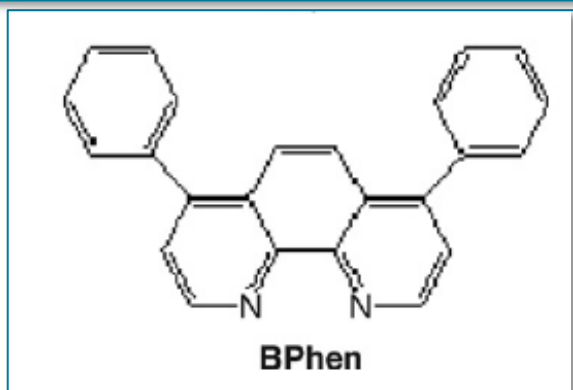
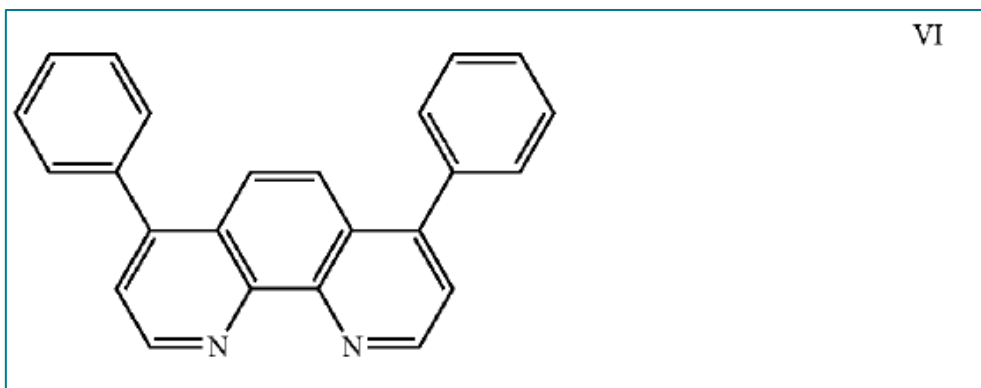
Werner, 58:4-15

Werner

Ma Back-up Slides

It Is Not Disputed That Ma Satisfies Claims 6, 9, And 14

6. The organic electronic component according to claim 1, wherein the organic compound is selected from:



Ma, 811, Fig. 1

9. The organic electronic component according to claim 1, which is formed as an organic LED (OLED) which additionally comprises an organic emitting layer.

Organic light-emitting diodes based on new n-doped electron transport layer

J.W. Ma^{a,*}, Wei Xu^a, X.Y. Jiang^a, Z.L. Zhang^{a,b}

Ma, 810

14. The method according to claim 10, wherein the organic compound does not coordinate to the metal complex prior to vaporization.

Then, a 50 nm-thick BPhen:Liq ETL was co-deposited on the EML. In the co-deposition process, organic sources were independently controlled. The deposition rates for the co-deposition layer were monitored using two independent quartz sensors. Finally, a shadow

Ma, 811

Dr. Kymissis' Testimony On Joint Vaporization

453. Under Pictiva's view that the accused products contain an electron-conducting layer that is obtained by "joint vaporization" (which I disagree with), then Ma discloses this limitation. I do not agree that this process constitutes "joint vaporization." I intend to offer more detailed opinions on this topic in my rebuttal report.

Ex. 2051 Kymissis Rpt., ¶ 453

range is disclosed by Ma. Moreover, in the district court, when discussing Ma, the RPI's expert stated unambiguously that he "do[es] not agree that this [Ma's] process constitutes 'joint vaporization'." EX2051, ¶¶ 452-453. Thus, Petitioner has not shown that this limitation is met.

POR, 56

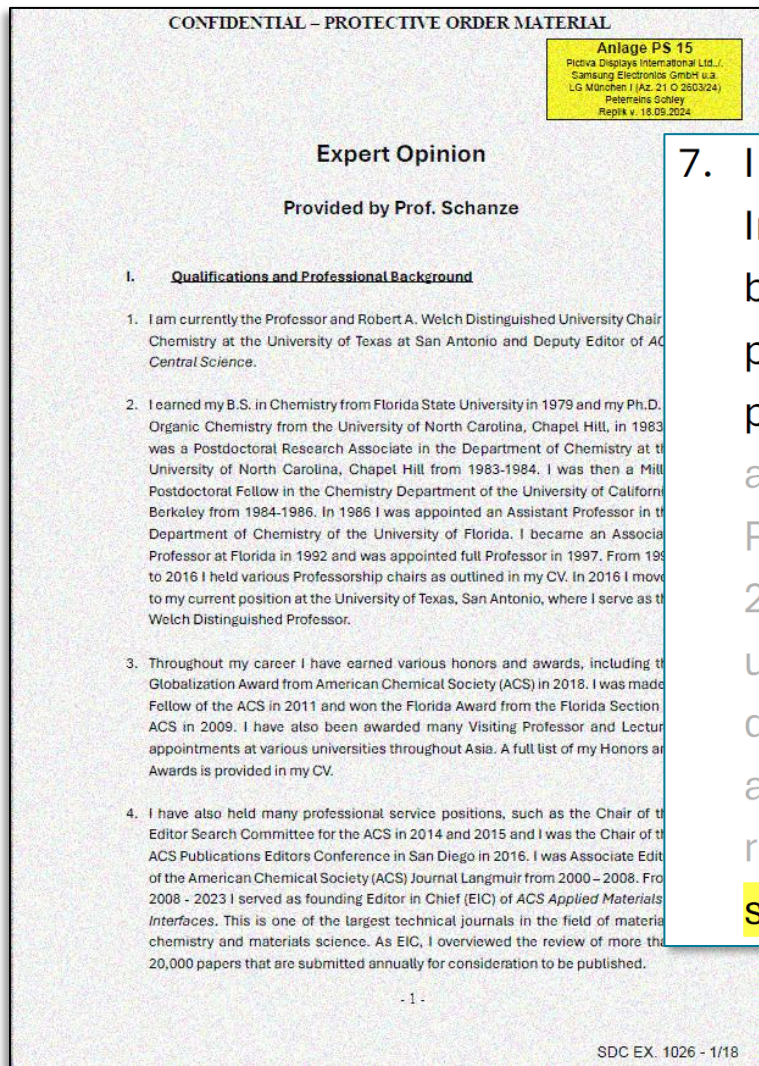
Ma

POR, 56

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Demonstrative Exhibit – Not Evidence 76

German Proceeding: Dr. Schanze Provided His Opinion Based On The '223 Patent



7. I have been made aware of the pending lawsuit between Pictiva Displays International Ltd. (“Pictiva”) and Samsung Electronics GmbH (“Samsung”) on the basis of the German patent DE 10 2008 056 391 B4; in this context I have been provided with the technical documents in these proceedings, in particular the patent in suit, English translations of Pictiva’s writ of complaint, and Samsung’s answer to the complaint, including an English translation of the expert opinion of Prof. Straßner. I am also aware that the filing date of the patent in suit is Sept. 26, 2008, and that I have to read the patent-in-suit from the viewpoint of the common understanding in the art at that point in time, excluding for that purpose any further developments that may have occurred afterwards. I have also been provided with an English translation of the request that Pictiva wants to assert in the lawsuit. The request is annexed as exhibit 1 to this opinion. My understanding of the patent-in-suit is based on the parallel US patent 8,558,223 B2 which is annexed as exhibit 2.

Ex. 1026 Schanze Expert Opinion, ¶ 7

Ex. 1026

See Pet. Reply, 22

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Demonstrative Exhibit – Not Evidence 77

BPhen Coordinates To Liq In The Gas Phase

Because of the simultaneous vaporization of metal complexes and organic compounds, both compounds are simultaneously present next to each other in the gas phase. This enables the organic compound to coordinate to the metal complex in the gas phase. The coordination can be effected

'223 Patent, 13:36-40; Pet., 69

"It is not invention to perceive that the product which others had discovered had qualities they failed to detect."

In re Kubin, 561 F.3d 1351, 1357 (Fed. Cir. 2009)



SDC EX. 1007 - 1/5

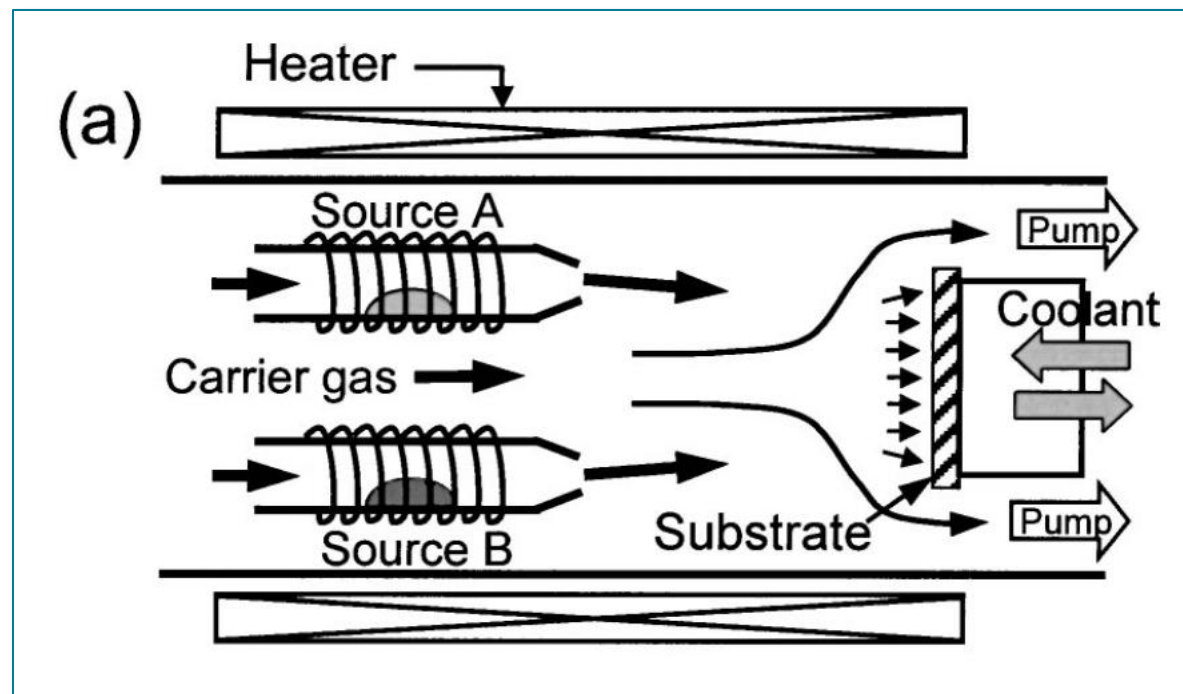
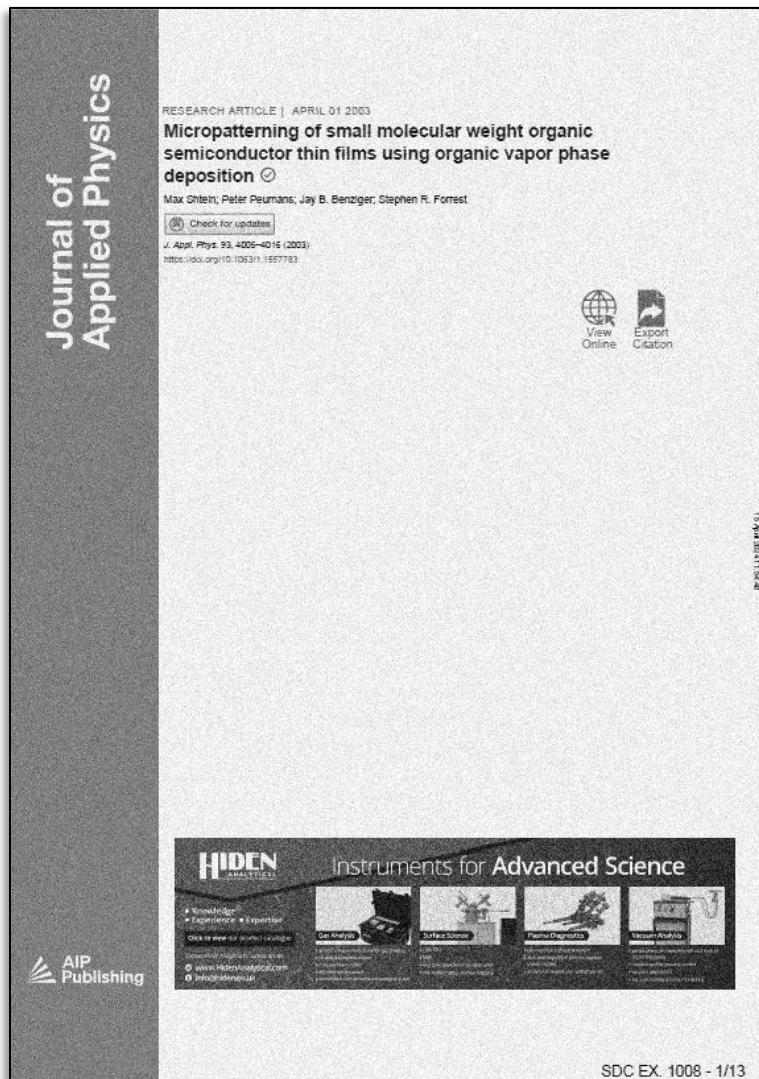
Ma

See Pet., 69-71; Pet. Reply, 26-27

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Demonstrative Exhibit – Not Evidence 78

Organic Vapor Phase Deposition (OVPD) Method



Ex. 1008 Shtein, 4006, Fig. 1(a)

Formation Of Chain-Like Structures Enables Electron Transfer



component. The formation of chain-like or network-like structures enables additional electrons to be able to be transferred from the metal complexes to the organic compounds, said electrons are then available for charge transport in the electron-conducting layer. In this regard, reference is made to

'223 Patent, 11:29-33



"It is not invention to perceive that the product which others had discovered had qualities they failed to detect."

In re Kubin, 561 F.3d 1351, 1357 (Fed. Cir. 2009)

Ma

See Pet., 72- 74

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Demonstrative Exhibit – Not Evidence 80