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Munich, 29 September 2011

Application Number: 00 932 308.0-2111
1 449 238

THE TRUSTEES OF PRINCETON UNIVERSITY et al.

Our Ref.: P 8317 O001/ KK

In preparation for the oral proceedings, which will take place on
3 November 2011, the following submission is made on behalf of the
Patentee:

I. Requests

It is requested to maintain the patent on the basis of the claims as granted
(Main Request). The Patentee refrains from repeating the arguments
presented with respect to the Main Request but refers to the previous
submission.

Additional claim sets are attached herewith as Auxiliary Requests in case the
Opposition Division is of the opinion that the attacked patent cannot be
maintained on the basis of the Main Request. The order of the Auxiliary
Requests is indicated on the respective claim set (First to Fourth Auxiliary
Request).

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EP 1 449 238 will be referred to as “the patent” in the following.

II. The invention

The invention underlying the patent is directed to an electroluminescent layer, which comprises a phosphorescent organometallic iridium compound or a phosphorescent organometallic osmium compound as the emitter, and OLEDs including such a layer.

The utilization of the specific class of phosphorescent organometallic iridium compounds as the emissive material in OLEDs allows for the production of OLEDs with very high and unexpected external efficiencies.

It was a key finding of the inventors, that the class of phosphorescent organometallic iridium compounds - due to their physical and chemical structure - possesses an extraordinarily strong spin-orbit coupling which provides intersystem crossing (ISC) between excited singlet and triplet states.

The class of phosphorescent organometallic iridium compounds shows an exceptional combination of firstly an extraordinarily strong spin orbit coupling and secondly a very small energy gap between the excited singlet and triplet states – both due to the physical and chemical structure of phosphorescent organometallic iridium compounds, i.e., due to a central iridium atom being bonded to a carbon atom of an organic ligand.

In other words, this class of phosphorescent organometallic iridium compounds has an exceptional balance of short exciton lifetimes together with a highly efficient intersystem crossing allowing for the harvesting of up to 100 % of the excitation energy as phosphorescence radiation.

The present invention, for the first time, makes it possible to produce highly efficient devices, dramatically outpacing the efficiencies of existent fluorescent and phosphorescent devices. This was nothing other than a

dramatic breakthrough, opening a new field of OLED technology for commercially relevant devices.

In the following pages, the invention is explained in more detail.

1. OLEDs – General technology

An OLED has one or more layers of organic materials sandwiched between an anode and a cathode. At least one of the organic layers, namely the electroluminescent layer, produces the electrically generated light emission. This emissive layer may consist of an emissive compound or may contain a host material in which the emissive compound is incorporated as a dopant.

When an electric field is applied to such a device, holes and electrons (represented by (+) and (-), respectively) migrate through the organic materials in response to the applied electric field, as depicted in Fig. 1 below.

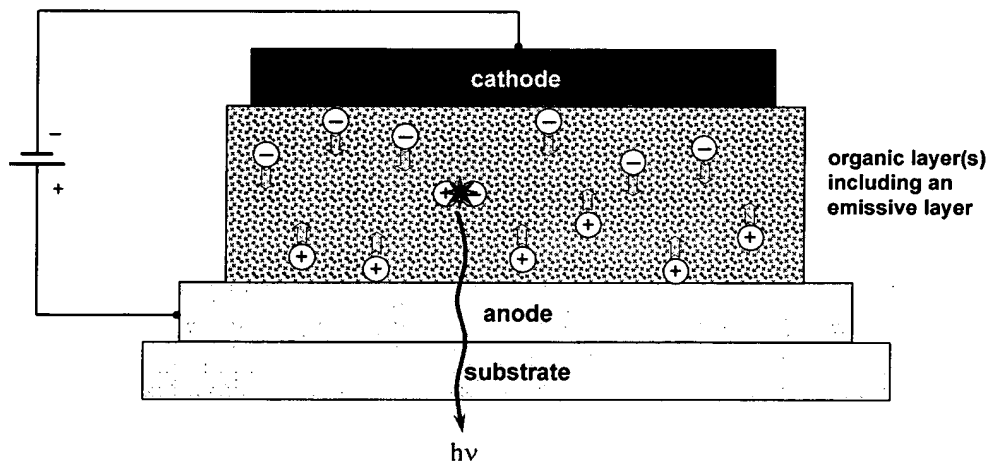


Fig. 1

When a hole and an electron migrate to positions such that they are residing on closely spaced or adjacent molecules, electron-hole recombination can occur, which results in an excited state, called an “exciton”. In contrast to photo-generated excited states, the application of an electric current induces

the direct formation of both excited singlet states (singlet excitons) and excited triplet states (triplet excitons). Under certain conditions this short-lived excited state can relax to its ground state via a photoemissive mechanism, i.e., by emitting light via fluorescence or phosphorescence.

2. State-of-the-art at the priority date of the patent

At the time of the priority date of the patent, OLEDs mainly emitted light by fluorescence, i.e., emission that results from the radiative decay from the singlet excited state to the singlet ground state ($S_1 \rightarrow S_0$) of the organic material. In 1997, OLEDs utilizing fluorescence were commercialized for the first time in front panels of in-vehicle radios. At that time, the light-emission efficiency of fluorescent OLEDs was approaching 5% external quantum efficiency, and there were already known many light emitting materials useful in OLEDs exhibiting strong fluorescence at room temperature.

However, fluorescent OLEDs suffer from a fundamental disadvantage:

As explained above, the excited state (exciton) formed by application of an electrical current and a recombination of an electron and a hole may be either a singlet excited state or a triplet excited state. Based on the spin statistics, the ratio of singlet excited states to triplet excited states formed by recombination is 1:3, respectively. However, the radiative decay from the triplet excited state to the singlet ground state ($T_1 \rightarrow S_0$), also known as phosphorescence, is a forbidden transition according to the selection rules of quantum mechanics, and thus was considered to be very inefficient and as a rule was not present at room temperature.

As a result, with fluorescent emission, only 25% of the electronically generated excitons, namely the singlet excitons, are available to emit light, whereas the triplet excitons that are formed relax to the ground state by undesired non-emissive mechanisms, generating heat. This 1:3 ratio between

singlet excitons and triplet excitons and the corresponding radiative decay is illustrated in Fig. 2 below (see left hand figure).

As will be explained, the present invention, on the other hand, allows for utilization of all of the electronically generated excited states, which increases the theoretical efficiency limit up to 100%. Furthermore, all emission then originates from the long lived triplet state (see Fig. 2, right hand figure).

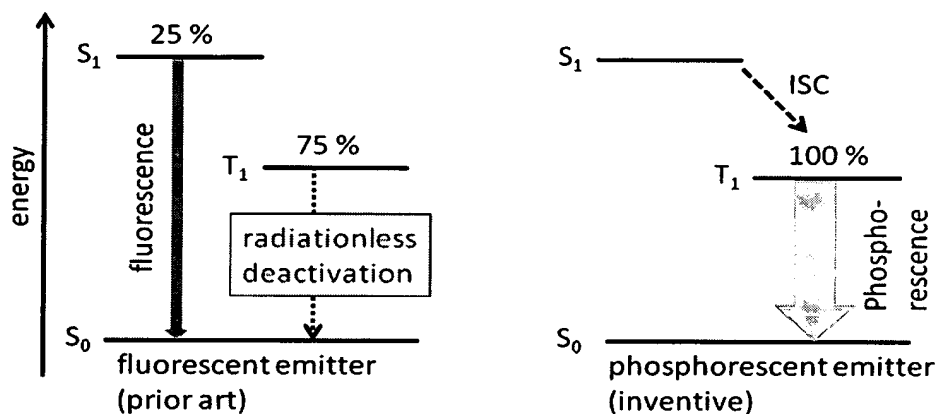


Fig. 2

At the time of the priority date of the patent, it was generally considered that it is extremely difficult to make use of phosphorescence emission in practical OLEDs. Document D8, for example, which was published in 1996 and is concerned with electroluminescence from triplet excited states in OLEDs, shows that light emission at room temperature was at best “hardly observable” (see D8, page 225, left column, third paragraph). Document D1, which was published two years later, reports an OLED with phosphorescent Os(II) complexes doped into a matrix material having an electroluminescence efficiency of only “below 0.1%” (see D1, page 247, right column). Similar observations were made by other research teams. Similarly, Document D3 reports OLEDs with phosphorescent rhenium complexes doped into a matrix

material having very low efficiency of 0.8 lm/W (approximately 0.1% external quantum efficiency, or less).

Then, in 1998, some of the inventors of the present patent reported an OLED using a platinum porphyrin derivative (PtOEP), wherein the device achieved an external quantum efficiency of about 4% with the use of phosphorescence (see D2, page 153, left column, second paragraph). This was the first and only time before the priority date of the patent that a phosphorescent OLED was reported that reached an industrially applicable level.

However, it turned out that the OLEDs with PtOEP have several limitations. For example, it is noted by the authors of D2 that the phosphorescent efficiency decreases rapidly at high current densities, and thus, it is difficult to achieve a high brightness of the OLED by increasing the current density (see D2, page 153, left column, first paragraph). Furthermore, it is suspected that the long lifetime of PtOEP may cause saturation of the emission at low dopant concentrations (see D2, page 53, left column, third paragraph).

3. The phosphorescent organometallic iridium compounds

The class of phosphorescent organometallic iridium compounds - due to their chemical structure - possesses an extraordinarily strong spin-orbit coupling which provides a complete intersystem crossing (ISC) between excited singlet and triplet states. The inventors recognized this from the surprisingly short exciton lifetimes that they measured as described in the patent (see patent, paragraph 28).

It was recognized by the present inventors that the phosphorescent organometallic iridium compounds possess a unique combination of firstly an extraordinary strong spin orbit coupling and secondly a very small energy gap between said excited singlet and triplet states – both due to a central iridium atom being bonded to a carbon atom of an organic ligand.

3.1. Strong spin orbit coupling and intersystem crossing

Before the invention described in the patent, it was not predictable for the skilled person that among all of the metals, only iridium, in combination with organometallic ligands, has these unique and unexpected spin-orbit coupling properties displayed by this class of phosphorescent organometallic compounds.

Opponent III alleges that iridium was an obvious choice for the skilled person, since it was known that Re, Os, and Pt may be used in phosphorescent OLEDs. Furthermore, by referring to D24, it is argued that the spin-orbit coupling required for triplet emission increases with increasing atomic number Z to the fourth power, which in turn leads to increased phosphorescence quantum efficiency. However, these statements largely ignore the scientific reality at the priority date of the patent, and overlook that there are several physical and chemical properties that must be fulfilled by a suitable emitter to provide efficient phosphorescent emission in an electroluminescent layer.

The search for an emitter that would facilitate the production of phosphorescent OLEDs with very high external quantum efficiencies was extremely difficult and it was not at all clear in which direction one should go.

If necessary, Prof. Dr. Mark Thompson, one of the inventors and a leading OLED expert, **is offered to be heard as a witness** and may provide further evidence for the skilled person's general knowledge at the priority date of the patent. He will attend the oral proceedings and will be available for being heard.

Although it was suspected in D2 that it may be unfavorable if the lifetime of a phosphorescent emitter is too long, it was not clear how such a lifetime

reduction could be achieved, and if it would necessarily lead to a more efficient device. This problem was made even more complicated by the fact that the emitter must also have a high phosphorescent quantum yield. Moreover, as can be readily seen from D1, a short phosphorescent lifetime (1.45 μs for the Os complexes of D1) and a high photoluminescence (PL) efficiency (0.33 for the Os complexes of D1) could not be directly correlated to a high OLED efficiency, since the OLED prepared with the phosphorescent Os complexes in D1 gave device efficiencies of less than 0.1% (see D1, page 247, left column, second paragraph, to right column).

Opponent III's conclusion that an increased spin-orbit coupling constant necessarily leads to increased quantum efficiency is certainly wrong. This becomes clear if one looks at lead and bismuth complexes, which possess a much higher spin-orbit coupling constant compared to iridium complexes, but show only little or no phosphorescence emission. Moreover, the Opponent's statement completely ignores the role of the ligand and the metal-ligand bonding.

Nothing different is stated in D24 which states that "*a heavy atom (with strong spin-orbit coupling) will have an impact on the phosphorescence quantum yield*" (see D24, page 485, third paragraph). In other words, a high spin-orbit coupling constant is important, but is not by itself sufficient to increase the quantum efficiency.

Furthermore, the atomic number of iridium is lower than that of platinum. Consequently, if one would follow Opponent III's line of argumentation, the skilled person would not have considered iridium compounds as a possibly more efficient emitter at all, but would have searched in the opposite direction of the third row of the transition meta series of the periodic table.

3.2. Utilization of up to 100% of the excitation energy

The strong spin orbit coupling of the iridium organometallic bond theoretically results in utilization of all the excitation energy produced in a device, from both singlet and triplet excitons, thus, realizing an enormous 100% upper limit in efficiency as compared with only a theoretical upper limit in efficiency of 25% as observed in fluorescent devices in the prior art. This is a four-fold increase in theoretical efficiency.

This is indicated in paragraph 28, lines 36-40, of the patent:

“These lifetimes are short and indicative of strong spin-orbit coupling, and together with the absence of Ir(ppy)₃ fluorescence in the transient response, we expect that Ir(ppy)₃ possesses strong intersystem crossing from the singlet to the triplet state. Thus all emission originates from the long lived triplet state.” (emphasis added).

This observation further distinguishes the patent’s teaching from D1 by Ma et al., who stated that the

“EL yield from the triplet excited state will be tripled if both singlet and triplet states have the same photoluminescent yield.”(emphasis added, see D1, page 245, sentence spanning left and right column).

Thus, there is no teaching or suggestion in D1 that all the emission can come from the triplet excited state. Quite to the contrary, this sentence asserts unambiguously that the D1 compounds were only expected to produce at most a three-fold increase in the triplet state emission, as distinct from the four-fold increase disclosed by the patent. This means there was no expectation that the D1 compounds would undergo intersystem crossing at

all, let alone being capable of the 100% intersystem crossing efficiency described in the patent.

3.3. What makes organometallic iridium compounds special?

The central iridium atom in combination with the organometallic bonding of the ligand causes an extraordinary strong spin orbit coupling which promotes a very efficient intersystem crossing (ISC) between the excited singlet and triplet states of the compound and promotes an efficient phosphorescent emission. Many later publications have acknowledged that the organometallic iridium complexes are the most effective emissive materials discovered to date for use in OLEDs, even compared to other heavy metal compounds.

The excited electronic states of the organometallic iridium complexes, which play an important role in the present case, are the ligand centered (LC) states, and the metal-ligand charge transfer (MLCT) states. LC states result from an electronic transition from a bonding π molecular orbital to an antibonding π^* molecular orbital on the ligand. MLCT states involve transition of an electron from a metal-centered orbital to a ligand-localized orbital, which is, in the present case, a transition from a d-orbital of the metal to an antibonding π^* molecular orbital of the ligand. Taking the spins and spin flips of such transitions into account, one obtains singlet and triplet LC and MLCT states.

The relative energy differences between the singlet MLCT state ($^1\text{MLCT}$), the triplet MLCT state ($^3\text{MLCT}$) and the triplet LC state (^3LC) are determined by the chemical structure of the metal containing compound. Phosphorescent organometallic iridium compounds generally emit from an energy state which is a ligand-centered triplet state (^3LC) with singlet metal-to-ligand charge transfer ($^1\text{MLCT}$) and triplet metal-to ligand charge transfer ($^3\text{MLCT}$)

character mixed in through spin-orbit coupling (see D29, page 147, last paragraph).

Due to the alignment of these three energy states in phosphorescent organometallic iridium compounds, the mixing of $^1\text{MLCT}$ character into the ^3LC state has dramatic effects on the photophysical properties of those compounds, and leads to a large decrease in the luminescence lifetime and an increase in phosphorescence efficiency.

The organometallic ligands of the iridium compounds stabilize the $^1\text{MLCT}$ state by strong σ -donation from the formally anionic C^- in the ligand, which further increases the efficiency of the $^1\text{MLCT}$ and ^3LC mixing. The improved mixing efficiency of these states in the iridium organometallic compounds additionally improves the emission properties of these compounds.

Furthermore, the energy gap between the $^3\text{MLCT}$ states and the $^1\text{MLCT}$ states is small so that the $^3\text{MLCT}$ states can experience efficient mixing with the close lying $^1\text{MLCT}$ states. As a consequence, the radiative decay rate is further increased, which is another reason for the very short phosphorescence lifetime of iridium complexes.

3.4. Response of the scientific community

The scientific community was very surprised when the inventors published that phosphorescent organometallic iridium compounds have such extraordinary and unexpected properties. This is, for example, evidenced by the fact that Dr. Tetsuo Tsutsui of Kyushu University, who had been widely known as one of the leading experts in the field of OLEDs, reported in a paper published only five months after the release of D6 (which presents some of the experimental data shown in the patent by the inventors) that he conducted a reproduction experiment to verify the results (see D28, page L1502, left column, second paragraph). Obviously, Dr. Tsutsui believed that

the results published in D6 were surprising and significant, and he immediately wanted to confirm that the high quantum efficiency values of organometallic iridium complexes were indeed correct. Stated differently, the results were so surprising, that he did not believe that they were correct and repeated the experiments himself.

If not even a worldwide-known expert in the field of OLED technology had expected that the class of organometallic iridium compounds would be a promising candidate for building highly efficient OLEDs, how would an ordinarily skilled person working in the OLED field have come to such a conclusion?

In fact, numerous researchers worldwide in the field of OLEDs have attributed the advance in the OLED field of the use of phosphorescent organometallic iridium compounds to the present inventors (often citing to the D6 Baldo *et al.* 1999 publication), and have praised the invention, characterizing it as “pioneering work”¹ and a “major breakthrough”² to name only two of the numerous post-published laudatory characterizations. The attention and praise that this invention has received from the scientific community provides clear, strong and objective evidence of the inventiveness of the present invention.

It would be preposterous if an invention acknowledged congruently by the scientific community as a pioneering breakthrough in the field should be regarded as being obviously derivable from the earlier papers of the same scientific community.

Consequently, for the ordinarily skilled person, it was not predictable from the prior art, without knowledge of the invention, that the class of

¹ Lo, S. C., et al., *Advanced Materials* 2005, 17, 1945-48 (D35); Zhang et al., *International Journal of Quantum Chemistry* 2009, 109, 1167-1176.

² Riel, H., et al., *Applied Physics Letters* 2003, 83 (3), 466-468.

organometallic iridium compounds has such extraordinary properties and would facilitate the production of phosphorescent OLEDs with very high external quantum efficiencies.

III. Article 100 (c) / Article 123 EPC

1. First Auxiliary Request

The First Auxiliary Request corresponds to the granted claim set, i.e. the Main Request, with the exception that the alternative feature “*a phosphorescent organometallic osmium compound*” has been deleted in claim 1.

Claims 2 to 4 are identical to granted claims 2, 4 and 5, and claims 5 to 12 correspond to granted claims 8 to 15.

Independent claim 13 is based on granted claim 16, wherein the alternative feature “*a phosphorescent organometallic osmium compound*” has been deleted.

Claims 14 to 16 correspond to granted claims 17, 19 and 20, and claims 17 to 24 correspond to granted claims 23 to 30.

Consequently, all claims of the First Auxiliary Request fulfil the requirements of Article 123(2) EPC.

2. Second Auxiliary Request

The Second Auxiliary Request claims 1 to 12 correspond to the First Auxiliary Request claims 13 to 24.

Therefore, all claims of the Second Auxiliary Request fulfil the requirements of Article 123(2) EPC.

3. Third Auxiliary Request

The Third Auxiliary Request is identical to the Second Auxiliary Request with the exception that in claim 1 it is defined that the organometallic iridium compound is “*cyclometallated*”. This amendment is supported by the description of the application documents as filed (see WO 00/70655, e.g., page 10, lines 18 to 20).

Therefore, all claims of the Third Auxiliary Request fulfil the requirements of Article 123(2) EPC.

4. Fourth Auxiliary Request

The Fourth Auxiliary Request is identical to the Third Auxiliary Request, wherein in claim 1 the cyclometallated organometallic iridium compounds were limited to compounds *with aromatic ligands*. This amendment is supported by the description of the application documents as filed (see WO 00/70655, e.g., page 14, lines 7 to 8).

Consequently, all claims of the Fourth Auxiliary Requests fulfil the requirements of Article 123(2) EPC.

IV. Article 100(a) EPC

1. Priority

1.1. Main Request and First Auxiliary Request

A priority can be validly claimed for “the same invention” in the sense of Article 87 EPC.

The invention of the priority document and the invention of claim 1 of the Main Request or the First Auxiliary Request, respectively, are the same. The

teaching of the present invention is that a specific class of unique compounds, namely the phosphorescent organometallic iridium compounds, provide for unique and unexpected advantages for the use as emitters in OLEDs (see section II. above). This class of compounds is disclosed on page 5, lines 30 to 31, as well as in independent claim 13 of the priority document.

Furthermore, the presence of an electroluminescent layer, as claimed in claim 1 of the Main Request or the Firsts Auxiliary Request, respectively, in OLEDs is an implicit part of every OLED for a person skilled in the art. Moreover, this feature is literally described on page 3, lines 5 to 8, of the priority document. This disclosure of the priority document shows that the presence of an electroluminescent layer in an OLED is part of the knowledge of a skilled person. Consequently, the skilled person directly and unambiguously derives the subject matter of claim 1 from the disclosure of the priority document, namely to use phosphorescent organometallic iridium compounds, in an electroluminescent layer.

A skilled person being provided with the insights of the present invention about the unique phosphorescent organometallic iridium compounds instantly understands that the present invention is not linked to an inessential feature like a heterostructure. The skilled person directly and unambiguously derives from the priority document that it is the “same invention” to use said specific compounds in an “emissive layer” (as already claimed by claims 1 to 8 of the priority document), or in an “electroluminescent layer” comprising such an emissive layer (as claimed by claims 1 to 15 of the patent), or in an “OLED” (as claimed by both the priority document and the patent).

Opponent I is undoubtedly wrong in asking, whether OLEDs, which are constructed from such electroluminescent layers of claim 1, extend beyond the OLEDs described in the priority document.

In line with the principles of G 2/98, the question to be answered for assessing the validity of the priority claim is whether the electroluminescent layer of claim 1 is directly and unambiguously derivable from the priority document by a skilled person using his/her general knowledge. As shown above this question has to be affirmed.

Consequently, claim 1 of the Main Request or the First Auxiliary Request, respectively, relating to an electroluminescent layer comprising phosphorescent organometallic iridium compounds validly claims the priority.

1.2. Second to Fourth Auxiliary Request

The Second to Fourth Auxiliary Requests lack granted claims 1 to 15.

The Patentee respectfully submits that the priority is valid for the subject-matter of the Second to Fourth Auxiliary Request.

2. Novelty

In the Preliminary Opinion the Opposition Division regarded the osmium complex disclosed in D1 as an organometallic complex, and therefore concluded that the subject-matter of granted independent claims 1 and 16 would be anticipated. Furthermore, it was argued that for granted claims 1 to 15 the priority claim is not valid, and thus, documents D6, D17 and D25 should be considered as prior art.

However, the subject-matter of the present invention is not anticipated by any of the cited references, as shown in the analysis below.

2.1. The term “organometallic”

The Patentee respectfully disagrees with the Opposition Division’s Preliminary Opinion that the osmium complex disclosed in D1 should be considered organometallic.

2.1.1. The definition of the patent is decisive

The Os(II) complexes disclosed in Fig. 1 of D1 are not organometallic compounds in the meaning of the patent. The term “organometallic” is clearly defined in the description of the patent (see patent, paragraph 22, lines 23 to 25), stating that

“The term “organometallic” is as generally understood by one of ordinary skill, as given, for example, in “Inorganic Chemistry” (2nd edition) by Gary L. Miessler and Donald A. Tarr, Prentice-Hall (1998).”

As already explained in our last submission, according to D4, which is an excerpt from Miessler et al., organometallic compounds are defined as compounds containing direct metal-carbon bonds (see D4, page 1, first paragraph). Furthermore, it is noted in D4 that cyanide is usually considered a classical, nonorganic ligand (see D4, page 424).

The Patentee respectfully submits that this definition is the only relevant one, since the patent is its own dictionary (see, e.g., T311//93, T1321/04).

2.1.2. The definition complies with the skilled person’s general knowledge

The definition of the term “organometallic” used in the patent is also in line with the understanding of the experts in this field. This is confirmed by the Expert Declaration of Prof. Dr. Dr. h.c. mult. Wolfgang A. Herrmann, the

current president of the Technical University of Munich, undisputedly an expert in the field of Organometallic Chemistry with high reputation. The Expert Opinion of Prof. Herrmann is attached herewith as

- document **D40** -.

In the attached Expert Opinion D40, Prof. Herrmann points out that the prototypical property of an organometallic compound is:

“Covalent bonds between the metal and at least one carbon of the organic groupings.” (see D40, page 2, third paragraph).

In other words, the essential characteristic of organometallic compounds is a direct covalent metal-carbon bond involving an organic ligand. Thus, ligands such as bipyridine or triphenylphosphine are not organometallic ligands because they do not have a direct metal-carbon bond.

Prof. Herrmann notes that the CO complexes are often grouped with the organometallics because the metal-CO bonding has analogous features to standard type organic ligands (see D40, page 3, first paragraph). According to Prof. Herrmann

“This originates from the simple reasoning that such ligands form covalent bonds with the respective metal(s) in a σ -donor/ π -acceptor bonding scheme.” (see D40, page 3, first paragraph).

However, Prof. Herrmann clarifies that these prerequisites are not fulfilled by the ligand CN^- , and points out that

“It is generally accepted among organometallic chemists that pronouncedly electronegative groups are outside the definition of OMCs [organometallic compounds]. This typically accounts for the halides X (e.g., $X = F, Cl, Br, I$) and their congeners such as cyanide (CN^-) [...]” (see D40, page 3, second paragraph).

Furthermore, Prof. Herrmann points out that the *Journal of Organometallic Chemistry* does not accept papers for publication, which report metal-cyano complexes without exhibiting other ligands typical of organometallic compounds (see D40, page 3, second paragraph).

Document D10, which is concerned with luminescence properties of organometallic complexes, also explicitly excludes cyanide ligands by stating that *“this review is concerned only with transition-metal organometallic complexes and does not include metal cyanides”* (emphasis added, see D10, page 712, left column, first paragraph).

The same is true for many internationally well-known textbooks on organometallic chemistry (see D40, page 5, last paragraph, to page 7, first paragraph).

This common understanding of the term “organometallic” is also in line with the recommendations for the “Nomenclature of Organometallic Compounds of the Transition Metals”, the so-called IUPAC Recommendations, published in 1999, i.e. close to the priority date of the patent. In this 28 page-document, it is stated in the introduction that an organometallic compound is defined as any chemical species containing at least one bond between a carbon atom in an organic molecule, ion, or radical and a metal. In the German translation of these recommendations “organic molecules”, in turn, are clearly defined, and the cyanide (CN) group definitely does not belong to them (see D40, page 4, middle paragraph).

Furthermore, the fact that cyanide is not considered as an organometallic ligand is not just an arbitrary classification, but it is due to the very different chemical and physical properties of this ligand:

“In the chemical literature the cyano (cyanide) ligand (CN) is stringently considered a “pseudo halide” like fluoride and its

congeners. This has its good reasons in that this particular ligand is electronegative and thus behaves like an inorganic ligand, and its metal complexes fall within the scope of the so-called “Inorganic Coordination Compounds”. ” (see D40, page 3-4, bridging paragraph).

Prof. Herrmann also confirms that for a skilled person the compounds disclosed in Fig. 1 of D1 are “*typical “metal coordination compounds” but surely not “organometallic compounds” because of the lack of organic ligands bound to the metal via a carbon atom.*” (see D40, page 8, second paragraph).

In consideration of the above, the Patentee is of the opinion that the term “organometallic” can only be understood properly as referring to compounds having a covalent direct metal-carbon bond with a carbon of an organic ligand, and definitely excludes the ligand cyanide.

If the Opposition Division intends to deviate from the understanding of the term “organometallic” as set out in D40, we **additionally request to hear Prof. Herrmann as a witness** in order to confirm the facts set out in D40 with respect to the definition of the term “organometallic” and the skilled person’s knowledge with regard to what is an organometallic compound. He can be summoned via the Patentee.

Additionally, the special properties of the organometallic iridium-carbon bond strongly affect a key finding of the patent’s invention. That is, the strong spin orbit coupling of the organometallic iridium complexes, facilitated by the iridium-carbon bond with a carbon of an organic ligand, allows for the utilization of all of the excitation energy produced in an OLED device, from both singlets and triplets, thereby realizing an enormous 100% upper limit in efficiency. In comparison to the 25% upper limit in efficiency

of prior art fluorescent devices, this represents a four-fold increase in theoretical efficiency.

2.2. First Auxiliary Request

The novelty arguments presented in the previous submission with respect to the Main Request are also valid for the First Auxiliary Request, and therefore the Patentee refrains from repeating all arguments presented in the previous submission.

Furthermore, it is to be noted that the priority claim is valid as shown in section IV.1. above. Consequently, documents D6, D17, and D25, which are published after the priority date of the patent, are not prior art.

In addition, the Patentee would like to note the following:

2.2.1. Ma et al. (D1)

D1 does not disclose a phosphorescent organometallic compound, and specifically not an iridium compound of this type.

The molecules disclosed in Fig. 1 of D1 are not “organometallic” because they do not contain an organometallic carbon-metal bond (i.e., a covalent bond between the metal atom and a carbon atom of an organic ligand). As explained in section IV.2.1. above, cyanide (CN⁻) is not an organometallic ligand. Also, the bipyridine and anthroline ligands of D1 are not organometallic ligands, as these ligands are complexed to the metal, not through carbon atoms, but only through the nitrogen atoms of the ligands.

The osmium complexes of D1 are metal complexes characterized as “coordination compounds”. This is in line with the fact that throughout D1 the osmium complexes are termed “transition metal complexes”.

Moreover, Prof. Herrmann confirms that the compounds disclosed in Fig. 1 of D1 are “*typical “metal coordination compounds” but surely not “organometallic compounds” because of the lack of organic ligands bound to the metal via a carbon atom.*” (see D40, page 8, second paragraph).

Consequently, the subject-matter of independent claims 1 and 13 is novel over D1.

2.2.2. Vander Donckt et al. (D7)

D7 does not disclose an electroluminescent layer, and it also fails to disclose an emission by phosphorescence.

D7 discloses Ir(ppy)₃ being immobilized in Amberlite powder which is taped to a support (see D7, paragraph bridging pages 207 and 208). Furthermore, the photoluminescence spectra of Ir(ppy)₃ embedded in Amberlite are presented in Fig. 2 of D7. The Amberlite powder containing Ir(ppy)₃ is irradiated under different concentrations of oxygen and the re-emitted light is measured (see D7, paragraph bridging pages 207 and 208).

However, D7 does not directly and unambiguously disclose that Ir(ppy)₃ emits by phosphorescence.

There is also no discussion in D7 of the electroluminescent characteristics of Ir(ppy)₃. The experimental set-up described in D7 does not contain electrodes and thus there is no means disclosed in D7 to even produce electroluminescence. Furthermore, the Amberlite is in the form of a powder, and it is not electrically conductive. Consequently, it is not possible to apply a voltage across the Amberlite layer with the aim of producing electroluminescence. Therefore, D7 does not disclose an electroluminescent layer.

Consequently, the subject-matter of independent claims 1 and 13 is novel over D7.

2.2.3. Di Marco et al. (D9)

D9 does not disclose an electroluminescent layer, and it also fails to disclose an emission by phosphorescence.

D9 discloses photoluminescence from a cyclometallated iridium(III) compound doped into a solid pPEGMA matrix for use as oxygen sensors. There is no discussion in D9 of electroluminescent characteristics of Ir(ppy)₃. The experimental set-up of D9 does not contain electrodes and thus there is no means disclosed in D9 to even produce electroluminescence. Therefore, D9 does not disclose an electroluminescent layer.

Furthermore, D9 does not directly and unambiguously disclose that the iridium(III) compound emits by phosphorescence.

Consequently, the subject-matter of independent claims 1 and 13 is novel over D9.

2.2.4. Colombo et al. (D38)

D38, newly introduced recently by Opponent III, does not disclose an electroluminescent layer.

D38 discloses Ir(ppy)₃ and Ir(thpy)₃ embedded in PMMA (see D38, page 545, right column, penultimate paragraph, to page 546, left column, third paragraph), and photoluminescence spectra thereof (see D38, Fig. 5).

However, D38 does not disclose that the Ir(ppy)₃ embedded in PMMA layer shows electroluminescence when a voltage is applied across the PMMA layer. There is no discussion in D38 of electroluminescent characteristics of

Ir(ppy)₃ or of the other disclosed complexes. The experimental set-up of D38 does not contain electrodes and thus there is no means disclosed in D38 to even produce electroluminescence. Therefore, D38 does not disclose an electroluminescent layer.

Consequently, the subject-matter of independent claims 1 and 13 is novel over D38.

2.3. Second to Fourth Auxiliary Requests

The Patentee respectfully submits that the novelty arguments put forward for the subject-matter of the First Auxiliary Request apply *mutatis mutandis* to the subject-matter of the Second to Fourth Auxiliary Requests.

Furthermore, it is to be noted that none of D7, D9, and D38 discloses an OLED comprising a heterostructure. Also, D1 does not disclose a cyclometallated iridium compound (Third Auxiliary Request), nor does D1 disclose a cyclometallated iridium compound with aromatic ligands (Fourth Auxiliary Request).

3. Inventive step

The subject-matter of the present invention also involves an inventive step, as indicated above and as will be shown in the following.

As a preliminary remark to inventive step, D1, which appears to be the closest prior art, has a fundamental deficiency: The only phosphorescent OLED reported in D1 performs significantly worse than the fluorescent prior art devices, and, therefore would have no commercial value. All the speculation and wishful thinking expressed in D1 about MLCT states and the alleged, but unsubstantiated, high (>50%) prior art photoluminescence efficiencies (see D1, page 245, left column) cannot overcome this

fundamental deficiency. None of the references [4-7] cited by D1 in this context provide evidence of such a quantum yield. Moreover, none of the non-OLED prior art cited by the Opponents, e.g., D7, D9, D38, can overcome this fundamental deficiency of D1, because they are totally silent on this OLED-based deficiency.

In other words, combining a prior art teaching which is completely void of any information on electroluminescent efficiency with D1's teaching of an OLED which has very low efficiency, and is in fact orders-of-magnitude less efficient than known devices, is at best wishful thinking, but in essence is purely ex-post-facto analysis.

3.1. First Auxiliary Request

The inventive step arguments presented in the previous submission with respect to the Main Request are also valid for the First Auxiliary Request, and therefore the Patentee refrains from repeating all arguments presented in the previous submission.

In addition, the Patentee would like to note the following:

3.1.1. D1 in combination with any one of D7, D9, D10, or D12

Closest art:

D1 discloses electrophosphorescence from an emissive layer comprising osmium complexes in an OLED. It belongs to the same technical field as the invention claimed in the patent.

Difference over D1:

The subject-matter of claim 1 differs from D1 in that the emissive molecule is an iridium compound, and is organometallic.

Effect of the difference:

The effect of the two distinguishing features is that the central iridium atom, in combination with the organometallic iridium-carbon bonding to the ligand, causes a strong-spin orbit coupling, as explained in paragraph 28 of the patent, which *inter alia* promotes intersystem crossing and efficient phosphorescence. As a result, the combination of both distinguishing features leads to a highly efficient phosphorescent emitter having a short phosphorescence lifetime as well as a very high phosphorescence quantum yield. Furthermore, the emission originates from the long lived triplet state which increases the theoretical efficiency limit up to 100%, as no fluorescence is observed. In other words, the functional interaction between the distinguishing features achieves a combined technical effect which exceeds the sum of the technical effects of the individual features.

The objective technical problem:

The technical problem to be solved may be regarded as finding a class of compounds which are highly efficient emitters when used in electroluminescent layers and OLEDs.

The claimed solution:

The solution to the foregoing problem is given in claim 1, namely, phosphorescent organometallic iridium compounds.

The claimed solution is not obvious:

The authors of D1 presume that complexes which exhibit stronger triplet state emission provide the possibility to design high efficiency OLEDs by using these triplet excited-state materials (see D1, page 245, left column, first paragraph). However, the results obtained in D1 using Os(II) coordination compounds doped into a matrix material as the emitting layer in an OLED

show that the electroluminescence (EL) efficiency of these systems is very low, namely below 0.1% (see D1, page 247, right column, lines 22 to 23). The very low EL efficiency of the D1 devices is a fundamental deficiency in that disclosure.

As described above (see section II.2.) at the priority date of the patent, fluorescent OLEDs having an external quantum efficiency approaching 5% were state of the art. Furthermore, a phosphorescent OLED having an external quantum efficiency of about 4% was published one year before the priority date of the patent (see D2, page 153, left column, second paragraph).

Therefore, when reading D1, the skilled person would come to the conclusion that emissive materials having high photoluminescence (PL) efficiency of the triplet state will not improve the EL efficiency of an OLED.

For that reason alone, the skilled person is discouraged from combining D1 with any other document disclosing emissive molecules with high PL efficiency. These technical facts disclosed in D1 teach away from such a combination.

The speculation of the authors of D1 that the results suggest that materials with high triplet state PL efficiency can be used as the emitting layer of OLEDs is in clear contradiction with their own data, and consequently, is nothing more than wishful thinking. A person of ordinary skill in the art would not look to unsubstantiated puffery when evaluating the disclosure of D1, but rather would look to the reported data. This is especially true in the event that the experimental data contradict the author's conclusion, as is the case for D1.

It was also not recognized by D1 that a strong intersystem crossing from a singlet to a triplet state may result in utilization of 100% of the excitation energy produced in an OLED, i.e., a four-fold increase in efficiency

compared to a fluorescent OLED. Rather, D1 states that *"the EL yield from a triplet excited state will be tripled if both singlet and triplet states have the same photoluminescence yield"* (emphasis added, see D1, page 245, sentence bridging left and right column). In other words, the authors of D1 did not realize that a strong intersystem crossing between the singlet and triplet state would lead to a situation where all singlet excitation energy is converted into triplet excitation energy, and thus all excitation energy is emitted by phosphorescence.

Furthermore, D1 does not contain any information that would prompt the skilled person to replace the Os(II) complex by another emissive molecule.

On one hand, it is stated in D1 that the optimization of the cell structure should bring about higher intensity (see D1, page 247, right column, lines 23 to 24). On the other hand, it is noted in D1 that the ligand structure of the doped complex and its concentration may be adapted to obtain a higher EL efficiency (see D1, page 247, right column, line 24 ff.). However, these statements are very general and the only information given in D1 with respect to the structural amendments of the doped complex is on page 246, right column, second paragraph: *"The electronic structure and emission properties of the Os(II) complexes can be controlled by changing the ligand structures"*. Examples for suitable ligands are given in Fig. 1 of D1. None of these ligand modifications leads to an organometallic metal-carbon bond. Only substitution patterns are changed. Furthermore, osmium is kept as the metal center of the complex. Therefore, when reading D1 the skilled person at best would come to the conclusion that more efficient OLEDs may be obtained by changing the substitution patterns of the non-organometallic ligands of the Os(II) complexes.

It is clear that these very general statements in D1 do not give the skilled person a clear starting point for improving the OLED efficiency. Rather,

these statements are as helpful as saying “*if you want to make your car faster, you have to tune the motor*” without explaining which parts of the motor should be amended and how and why. However, these very general statements actually confirm that at the priority date of the patent it was simply not clear how the efficiency of phosphorescent OLEDs could be improved.

It is established case law of the Boards of Appeal that the question is not whether the skilled person could have arrived at the invention by modifying the prior art, but rather whether, in expectation of the advantages actually achieved (i.e. in the light of the technical problem addressed), he would have done so because of promptings in the prior art (T 219/87, T 455/94, T 414/98).

It is clear from the above observations that based on the information given in D1, the skilled person would not have replaced the molecules listed in Fig. 1 of D1 by a molecule disclosed in any of D7, D9, D10, or D12, with a reasonable expectation of success. Any such combination of references constructed by the Opponents is artificial and results from an inadmissible ex-post-facto analysis having the present invention in mind.

The same arguments apply *mutatis mutandis* to the OLED of independent claim 13.

For the sake of completeness, it is to be noted that none of D7, D9, D10, or D12 is concerned with OLEDs, electroluminescence or emitters that are suitable for OLEDs. D7 and D9 relate to a different technical field, namely oxygen sensors. D10 is a review article that summarizes the photoluminescence properties of a vast number of organometallic complexes, and D12 is concerned with the photocatalytic activity of Ir(III) complexes.

As shown in detail above, there was no objective reason to combine the teachings of these documents with that of D1, other than hindsight knowledge of the solution as claimed by the patent. Consequently, the skilled person would not have combined any of these documents with D1.

Consequently, the subject-matter of claim 1 and claim 13 involves an inventive step over D1 in combination with any one of D7, D9, D10, or D12.

3.1.2. D2 in combination with any one of D7, D9, D10, D12 or D20

D2 differs from the subject-matter of claims 1 and 13 by the same features as D1, namely that the emissive molecule is an iridium compound, and is further organometallic. Thus, it may equally be regarded as the closest prior art in an inventive step analysis.

The effect of the differences and the objective technical problem are the same as for D1, as well as the solution provided by the patent.

It is noted in D2 that the addition of platinum to the porphine ring reduces the phosphorescence lifetime by increasing spin-orbit coupling, which also enhances the efficiency of intersystem crossing from the singlet to the triplet state (see D2, page 151, right column, second paragraph).

According to D2, a maximum external quantum efficiency of 4% was obtained at a PtOEP concentration of 6% in an Alq₃ host material. The phosphorescence lifetime matched the quantum efficiency results: the shorter lifetimes observed at high current densities correspond to lower quantum efficiencies (see D2, page 153, left column, second paragraph).

Although D2 mentions that the long exciton lifetime of PtOEP may lead to saturation of emissive sites at low dopant concentrations, there is no clear suggestion or hint in D2 that the lifetime of the excited triplet state plays a crucial role in increasing the efficiency of the electrophosphorescence.

Rather, the decreased quantum efficiency is partially attributed to a decreased lifetime (see D2, page 153, bridging sentence between left and right column), which would lead the skilled person to the conclusion that a shorter lifetime may be unfavorable since it lowers the quantum efficiency.

Furthermore, D2 does not motivate the skilled person to replace PtOEP by another emitter. Rather, the authors of D2 conclude that other host materials should be used with PtOEP to increase the efficiency beyond the values reported in D2 (see D2, page 153, right column, last paragraph).

Moreover, a person skilled in the art would have known from the other references cited by the Opponents that the lifetime of an emitter cannot be directly correlated to device efficiency. D1 reports an emission lifetime of the osmium complexes of 1.45 μs or less (see D1, page 247, second paragraph) and reports a very poor device efficiency of less than 0.1%. Notably, the reported lifetime for the osmium complexes of D1 is even shorter than the reported emission lifetime of Ir(ppy)₃ (2.0 μs) reported in D12 (see D12, page 1431, right column).

The text passage in D2 “*Given the performance of PtOEP, other phosphorescent dyes emitting in the green or blue regions of the spectrum also present an attractive area of study for display application*” (see D2, page 153, right column, last paragraph), certainly does not suggest to search for more efficient complexes, as argued by the Opponents. Rather, this text passage unspecifically suggests searching for different complexes to provide different colors.

To summarize, D2 neither contains any information that would motivate the skilled person to replace the PtOEP complex by another emissive molecule, nor gives the skilled person a clear starting point for further improving the OLED efficiency.

Therefore, based on the information given in D2 the skilled person would not have replaced PtOEP by a molecule disclosed in D7, D9, D10, D12, or D20. Any such combination of references constructed by the Opponents is artificial and results from an inadmissible ex-post-facto analysis having the present invention in mind.

Similar arguments apply *mutatis mutandis* to the subject-matter of independent claim 13.

Consequently, the subject-matter of claim 1 and claim 13 involves an inventive step over D2 in combination with any one of D7, D9, D10, D12 or D20.

3.1.3. D19 in combination with any one of D7, D9, or D12

D19 relates to the field of phosphorescent OLEDs and is concerned with improved energy transfer in OLEDs. More precisely, D19 studies the external quantum efficiencies of OLEDs, wherein PtOEP is doped at various concentrations into different matrix materials (see D19, page 442, right column, last paragraph).

The subject-matter of claim 1 differs from D19 in the same way as D2, and the disclosure of D19 is very similar to that of D2, which is not surprising considering that all authors of D19 are also authors of D2.

Consequently, the arguments provided above for non-obviousness over D2 in combination with any of D7, D9 and D12 similarly apply here.

In addition, D19 is concerned with the problem of how the efficiency of an OLED comprising PtOEP as the emissive molecule can be improved. More precisely, D19 aims at reducing non-radiative losses and improving the external quantum efficiency by altering the host material and the device structure (see D19, page 442, left column, first paragraph).

It is shown in D19 that the quantum efficiency of an OLED comprising PtOEP as the emitter may be improved by incorporating an exciton-blocking layer (see D19, page 444, left column, last paragraph). Based on their results, the authors of D19 propose that a general technique for obtaining high efficiency from phosphorescent emitters is to employ a host chosen such that the phosphorescence lifetime is as long as possible and to use a blocking layer to keep the triplets within the luminescent region (see D19, page 444, right column, penultimate paragraph).

Consequently, D19 teaches the skilled person directly away from searching for other emitters but instead suggests optimizing the host materials of the OLED.

Therefore, based on the information given in D19 the skilled person would not have replaced PtOEP by a molecule disclosed in D7, D9, or D12.

3.1.4. D1 in combination with D38

In its latest submission, it is argued by Opponent III that D38 would be a relevant document for inventive step of granted claims 1 to 16.

This is clearly not the case, as it does not provide any new aspects over the other prior art documents already on file.

Above all, D38 relates to a passive experimental set-up, not at all suited for electroluminescence. In particular, D38 neither discloses nor suggests that the iridium complexes embedded in a PMMA layer disclosed therein show electroluminescence if a voltage is applied (see section IV.2.2.4. above). Consequently, D38 does not disclose the electroluminescent layer of claim 1.

Rather, D38 is a study on spectroscopic properties using photoluminescence (see D38, page 545, left column). D38 presents photoluminescence spectra of iridium and rhodium complexes, analyzes the absorption and emission bands

of these complexes, and tries to assign the observed bands to specific excited energy states.

D38 neither relates to the same technical field as the patent, namely the field of OLEDs, nor does it relate to a similar purpose, namely the preparation of highly efficient OLEDs. Thus, D38 clearly does not constitute the most promising starting point for an obvious development leading to the invention.

As already discussed in detail in section IV.3.1.1, D1 neither contains any information that would prompt the skilled person to replace the Os(II) complex by another emissive molecule, nor gives the skilled person a clear starting point for improving the OLED efficiency.

By referring to two text passages from D1, Opponent III alleges that D1 would give the skilled person a clear guidance that organometallic iridium complexes containing excited MLCT states would be suitable as light emitting materials in OLEDs.

The first text passage is from the introductory part of D1 on page 245, left column, first paragraph, and reads: *“As we know, due to strong interaction between metal center and the ligands, the transition metal complexes (such as Ru, Os, Ir) exhibit a metal-ligand charge-transfer (MLCT) excited state which shows the triplet nature due to long excited-state lifetime [...]”*. The authors of D1 expect that electroluminescence may be observed from such a state (see D1, page 245, sentence bridging left and right column). However, this is pure speculation, but certainly not a clear guidance that organometallic iridium complexes that contain an excited MLCT state would be suitable as light emitting materials in OLEDs. Also, the authors of D1 expressly expect long excited-state lifetime, which is exactly the opposite of the finding of the inventors of the patent.

Furthermore, it is to be noted that most phosphorescent heavy metal complexes exhibit some MLCT character in the excited states. However, the mere fact that these complexes share this common characteristic does not somehow convey that all of these complexes are suitable as light emitting materials in OLEDs.

The second text passage on which Opponent III's conclusion is based seems to be on page 248, left column, last paragraph, which reads "*The EL emission from the triplet MLCT state of Os(II) complexes was observed by using ITO/Os complex: PVK/Al and ITO/Os(II) complex PVK/PBD/Al cells.*" Based on these results, the authors of D1 speculate that materials with high triplet state photoluminescence efficiency can be used as the emitting layer in OLEDs (see D1, page 248, right column, first paragraph).

However, as already discussed in detail in section IV.3.1.1, the data presented in D1 contradict the authors' statement and would lead the skilled person to the opposite conclusion, namely that emissive materials having high photoluminescence (PL) efficiency and emitting from the triplet MLCT state will not improve the electroluminescence (EL) efficiency.

Therefore, based on the information given in D1, the skilled person would not have replaced the molecules listed in Fig. 1 of D1 by a molecule disclosed in D38. The combination of D1 and D38 constructed by Opponent III is purely artificial and results from an inadmissible ex-post-facto analysis having the present invention in mind.

Consequently, the subject-matter of claim 1 and claim 13 involves an inventive step over D1 in combination with D38.


3.2. Second to Fourth Auxiliary Requests

The Patentee respectfully submits that the inventive step arguments put forward for the subject-matter of the First Auxiliary Request apply *mutatis mutandis* to the subject-matter of the Second, Third, and Fourth Auxiliary Request.

V. Conclusion

Considering the arguments and relevant background information as provided above, and in view of the congruent praise by the scientific community of the present invention as a pioneering breakthrough in the field, it would be preposterous if such an invention should be regarded as being obviously derivable from the earlier papers of the same scientific community.

Consequently, the request to maintain the patent on the basis of the Main Request or Auxiliary Requests and to reject the opposition is fully justified.


Maiwald Patentanwalts GmbH
(Norbert Hansen)

Encl.:

D40 (Expert declaration)
First to Fourth Auxiliary Request
(clean and marked-up)