Perfluorinated Porphyrazines. 5. Perfluoro-α,β–dicyanostyrbene: Molecular Structure and Derived (Sub)Porphyrazine Complexes with IIIa Group Elements

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Dedicated to Prof. Dieter Wöhrle on the occasion of his Birthday

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Template cyclomerization of perfluoro-α,β-dicyanostyrbene in the presence of trichlorides of IIIa group elements results in formation of pentafluorophenyl substituted boron(III) subporphyrinate ([Cl]BsP AF30) and aluminum(III) and gallium(III) porphyrinates ([Cl]MPAF â‚¬, M=Al, Ga). Their spectral-luminescence properties are discussed, revealing blue shift of the Q-band and brighter fluorescence comparatively to non-fluorinated analogues. In addition, the molecular structure of trans-perfluoro-α,β-dicyanostyrbene was determined by single crystal X-ray diffraction.

Keywords: Perfluoro-α,β-dicyanostyrbene, subporphyrinate, porphyrinate, complexes, IIIa group elements, X-ray, synthesis.

Perфторированные порфиразины. 5. Перфтор–α,β–дицианостильбен: Молекулярная структура и комплексы (суб)порфиразинов с элементами IIIa группы на его основе

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При темплатной цикломеризации пентафтор-α,β-дицианостильбена в присутствии хлоридов элементов IIIa группы образуются пентафторфенилзамещенные субпорфиразинаты бора ([Cl]BsP AF30) и порфиразинаты алюминия и галлия ([Cl]MPAF â‚¬, M=Al, Ga). Приводится сравнительная характеристика спектрально-люминесцентных свойств полученных комплексов и их нефторированных аналогов. Структура транс-изомера пентафтор-α,β-дицианостильбена определена методом рентгеноструктурного анализа.

Ключевые слова: Перфтор-α,β-дицианостильбен, субпорфиразины, порфиразины, комплексы, элементы группы IIIa, рентгено-структурный анализ, синтез.
Introduction

Fluorinated porphyrinoids reveal a wide range of outstanding features that differ from nonfluorinated analogues due to electron-donating character of fluorine atoms. Their peculiarities and application in various fields highlighted by H. Hanack[1] (non-linear optics, D. Wöhrle[2] (organic electronics), T. Goslingk[3] (biochemical applications) and other authors (see review chapter[4]) inspired us for studying the perfluorinated porphyrazines. In a series of our works we have prepared octa(pentafluorophenyl)porphyrazine [H,F,PF,] and its complexes with Mg2+, Zn2+ and In3+ and demonstrated the crucial role of perfluorination in regulation of acid-base properties of meso-and inner nitrogen atoms and in photophysical properties such as quantum yield of fluorescence and singlet oxygen generation.[5,6] In addition, the presence of fluorinated phenyl residues widens the opportunities for modification of periphery of the macrocycle by nucleophilic substitution of fluoride atom(s). Thus, S,Ar reaction was employed for preparation of butoxy and water soluble galactosyl derivatives from [ZnPAF40], in which up to eight F-atoms in p-positions of phenyl rings can be substituted.[7] It was also demonstrated by Nemykin and coauthors[8] that μ-oxodiiiron(III) complex, [μ-O(FePAF40)]2, is an effective catalyst of oxidation reactions.

Introduction of electronegative fluoride atoms in macrocycle allows controlling the properties of tripyrrolic macrocycles as well. Thus, it was observed that fluorinated subphthalocyanines [(Cl)BsPcF3] are taken much stronger by breast tumor cells as compared with non-fluorinated species [(Cl)BsPc] and exhibit significant intracellular fluorescence.[9] Perfluorinated subphthalocyanine [(Cl)BsPcF60] was shown to be efficient as an acceptor in organic photovoltaic devices for solar energy conversion.[10] A number of low-symmetry subporphyrazines containing fused tetrafluorobenzene moieties were reported (see [4] and refs. therein, [11]). It was also found that fluorination of polyphenylenediquinoxalinesubporphyrazines gives rise to superior singlet oxygen quantum yields.[12] But still, there are only few examples of subporphyrazines bearing fluorinated residues.[13,14] Here we report on the pentafluorophenyl substituted macrocyclic complexes of IIIa group elements which are formed in the course of template cyclomerization of perfluoro-α,β-dicyanostylylene in the presence of corresponding trichlorides. In addition, the molecular structure of trans-perfluoro-α,β-dicyanostylylene was determined by single crystal X-ray diffraction.

Experimental

General

UV-vis spectra were recorded using Cary-60 spectrophotometer. Mass-spectrometric measurements were carried out on a MALDI-TOF Bruker Ultraflex (the Center of the Collective Usage of ISUCT). NMR spectra were recorded in CDC11 solutions on a Bruker AV-400 or AV-500 spectrometers operating at 376.31 MHz for 19F (CF3COOH). IR spectra were recorded on Cary 630 FTIR (KBr). Fluorescence was measured on Shimadzu RF-6000 spectrofluorophotometer. Fluorescence quantum yields were determined by comparative method using ZnPc as a reference (Φ0 = 0.32 in THF) as described in [22]. Commercially available solvents were dried and distilled prior use.

Synthesis

Perfluoro-α,β-dicyanostylylene (1) was obtained following the previously described procedure[13] as a mixture of cis- and trans-isomers which was used for template cyclomerization reactions without separation. The single crystals of the trans-isomer, trans-1, were obtained occasionally by slow evaporation of the first fraction collected in the course of purification of 2 by column chromatography (see below).

Hexakis(pentafluorophenyl)subporphyrazinatoboron(III) chloride, [(Cl)BsPAF40] (2). Perfluoro-α,β-dicyanostylylene 1 (mixture of cis- and trans-isomers, 300 mg, 0.73 mmol) was placed into two-necked flask and purged with argon for 10 minutes. Then BCl3 (0.75 mL of 1 M solution in p-xylene, Aldrich) was added by syringe. The mixture was refluxed at 150 °C under argon for two hours. At the beginning of heating the color of mixture changed from orange to green and then to dark-blue. The color got a “burgundy” tone after the cooled reaction mixture was allowed to contact with air. The obtained solution was passed through a column with silica (eluent: CH2Cl2) to remove unreacted dirinile. Slow evaporation of the 1st fraction which was slightly colored due to admixture of the cyclotrimerization product afforded crystals of the trans-isomer of the dirinile-recursor, trans-1, which were suitable for X-ray diffraction analyses. The second intensified colored pink-purple fraction contained the target subporphyrazine 2 which was isolated as burgundy solid (70 mg, yield 30 %). Rf = 0.61 (CH2Cl2-hexane 1:1). MS (LDI-TOF) negative mode: m/z = 1277.3 (100 %) [M-H]+; positive mode: m/z = 1241.3 (100 %) [M-Cl]. IR (KBr); 1525, 1466, 1426, 1404, 1369, 1298, 1258, 1219, 1260, 1310, 1390, 1400, 1498, 1523, 1560, UV-Vis (CH2Cl2), λmax nm: 528.

Octakis(pentafluorophenyl)porphyrazinatoaluminuun(III) chloride, [(Cl)AlPAF40] (3). Mixture of cis- and trans-perfluoro-α,β-dicyanostylenes 1 (1.87 g, 4.56 mmol) was melted with anhydrous AlCl3 (670 mg, 5 mmol) at 260 °C until solidification (ca. 30 min.). The residue was dissolved in CH2Cl2, filtered and chromatographed on neutral alumina (II grade Brockmann, eluent CH2Cl2, with 1–3 % methanol). After evaporation of the solvent the product was obtained as green solid (760 mg, 40 %). MS (LDI-TOF), m/z: 1667.7 (100 %) [M]+ (calc. for C64H36F40N4AlCl3 1701.91, 1686.7 (48 %) [M+Cl]+), 1702.7 (6 %) [M-Cl] (calc. for C64H36F40N4AlCl1701.91, 1719.8 (50 %) [M]+, IR (KBr), v cm–1: 819m, 874m, 994s, 1124w, 1157m, 1413m, 1501s. UV-Vis (CH2Cl2), λmax nm (log e): 361 (4.41), 571 (3.54), 620 (4.12).

Octakis(pentafluorophenyl)porphyrazinatogallium(III) chloride, [(Cl)GaPAF40] (4), was obtained analogously to 3 from dirinile 1 (0.6 g, 1.46 mmol) and GaCl3 (1.47 mmol). The product was obtained as emerald green solid (270 mg, 42 %). MS (LDI-TOF), m/z: 1710.3 (100 %) [M]+, 1729.0 (11 %) [M+Cl]+, 1746.0 (17 %) [M]+ (calc. for C64H36F40N4GaCl1745.91, IR (KBr), v cm–1: 818m, 862m, 996s, 1124w, 1157m, 1413m, 1501s. UV-Vis (CH2Cl2), λmax nm (log e): 372 (4.49), 571 (3.89), 624 (4.59).

Crystal Structure Determination

The diffraction data for 1 were collected on a XTALab Pro P200K MM003 diffractometer with MoKa radiation (λ = 0.71073, MicroMax 003) by doing o scan at 100 K temperature. Absorption correction was done empirically using spherical harmonics in CrysalisPro software.[15] Crystallographic data and refinement details for 1 are given in Table 1 (see Supplementary information).
Perfluorinated (Sub)Porphyrazines

The primary structure was solved by direct methods using SHELXL-2018/1 and WinGX. The positions of other atoms were determined by the difference syntheses of electron density and were refined against \( |F|^2 \) by the least squares method.

The crystallographic data have been deposed in the Cambridge Crystallographic Data Centre under the deposition codes CCDC 1967396.

Results and Discussion

Synthesis

The dinitrile precursor 1 for perfluorinated complexes 2–4 was prepared as a mixture of cis- and trans-isomers following the previously described procedure. Earlier it was shown that tedious chromatographic separation of cis- and trans-isomers of 1 is a dispensable procedure and their mixture can be successfully used in template cyclotetramerization leading to formation of porphyrazine complexes \([\text{MPA}_4\text{F}_4]\) \((\text{M} = \text{Mg}^{II}, \text{Zn}^{II}\) and \(\text{X}^\text{III}\).\)[5,6] Non-fluorinated compounds – complexes of octaphenylporphyrazine, e.g. \([\text{Al}^\text{III} \text{PA}_4\text{H}_4]\) and \([\text{Ga}^\text{III} \text{PA}_4\text{H}_4]\) can be easily obtained directly from trans-dicyanostylylene (i.e. fumaric acid derivative) by melting with corresponding metal halides. So in the present work a mixture of cis-1 and trans-1 was also used in the template cyclotetramerization in a melt with anhydrous Al\(^{III}\) and Ga\(^{III}\) salts affording the corresponding porphyrazine complexes 3 and 4 \([\text{Cl}\text{MPA}_4\text{F}_4]\) \((\text{M} = \text{Al}^{III}\) and Ga\(^{III}\).\)[18] It is known that the boron atom is too small to assist the cyclohexamerization of phthalodinitriles with formation of phthalocyanines and B\(^{III}\) halides can serve as templates only for cyclopentamerization affording boron(III) subphthalocyanines.\[19,20,21]\] Similar cyclotrimerization with formation of subporphyrazine macrocycle was also observed for heterocyclic vicinal dinitriles (e.g. with pyrazine\[22\]) or 1,2,5-chalcogenadiazole rings\[11b,23\]) and for alkyl and alkylsulfanyl substituted maleodinitriles.\[24,25\] It was reported that alkyl or aryl substituted fumarodinitriles having trans-configuration are inactive in cyclotrimimerization with formation of subporphyrazines.\[25\] Since diaryl substituted maleodinitriles were not available, hexaarylated subporphyrazines were obtained\[14\] only by peripheral arylation of ethylsulfanyl substituted porphyrazines using Pd-catalyzed Cu-mediated cross-coupling reaction with arylboronic acids with overall yield from \(\text{di(ethylsulfanyl)maleonitrile}\) not exceeding 6 %. We have observed that reaction of cis/trans-1 mixture with BCl\(_3\) in \(p\)-xylene under reflux leads to cyclotrimerization product – perfluorophenyl substituted subporphyrazine 2, which was isolated with ca. 30 % yield as chloroboron(III) complex, \([\text{Cl}\text{B}^\text{P}3\text{PAF}_3]\). During column chromatography (silica, CH\(_2\)Cl\(_2\) it was eluted as a second fraction, while the first fraction contained the unreacted fumaric isomer trans-1, which was obtained as single crystals suitable for X-ray diffraction study. Evidently cis-configuration of two cyano groups is not strictly necessary requirement for cyclotetramerazation, but is essential for successful cyclotrimerization.

X-Ray Crystal Structure of trans-1

Unreacted bis(pentafluorophenyl)fumarodinitrile, trans-1, isolated at purification of subporphyrazine complex 2 could be crystallized and its molecular structure was determined using single crystal X-ray diffraction (Figure 1). In a centrosymmetric molecule the carbon atom of the ethylene fragment is disordered between two positions (C7A – 76.7 % and C7B – 23.3 %). The pentafluorophenyl fragments form the dihedral angle 61.1° with the central dicyanoethylene moiety, thus excluding the conjugation. The ethylene fragment contains C=C double bond (1.346 Å) and the C7A-C1 distance to aryl group is typical for C-C single bond (1.509 Å). In the case of non-fluorinated precursor (diphenylmaleodinitrile) the dihedral angle 42.5° allows some conjugation leading to elongated C…C bond (1.455 Å).\[26\]

Characterization of Macrocyclic Complexes

Mass-spectra. Formation of the macrocyclic complexes in the course of template cyclomerization

Scheme 1. Template cyclomerization of 1 in the presence of IIIa group chlorides.
of the dinitrile 1 was confirmed using MALDI-TOF mass-spectrometry. Perfluorophenyl groups strongly enhance the electron affinity of the macrocycles and facilitate formation of the negative ions, while ionization with appearance of cations becomes difficult. For subporphyrazine complex 2 the positive molecular ion peak \([M]^+\) observed at 1276 Da is accompanied by more intense peak \([M-Cl]^+\) at 1241 Da. For porphyrazine complexes 3 and 4 no positive molecular ion peaks could be seen. The negative molecular ion peaks with characteristic isotope distribution are present in the LDI-TOF mass-spectra of all compounds (Figure 2). For subporphyrazine 2 only negative molecular ion \([M]^–\) is present. For porphyrazines 3 and 4 the molecular ion \([M]^+\) is much less intense than fragmentation anion \([M-Cl]^–\) which formally corresponds to the complex between \(M^{III}\) and doubly reduced porphyrazine macrocycle. Formation of the secondary anion \([M-Cl+\text{F}]^–\) due to exchange of axial chlorine by fluorine is also observed.

**Electronic absorption spectra** of perfluorinated complexes with IIIa group elements and their non-fluorinated analogues are shown in Figure 3. The spectra of the \(Al^{III}\) and \(Ga^{III}\) complexes 3 and 4 are typical for porphyrazines and contain intense \(Q\)-band in the visible region (620 and 624 nm) and Soret band in the UV-region (361 and 372 nm). For the subporphyrazine complex 2 the \(Q\)-band is much broader and its maximum is shifted by ca. 100 nm to the shorter wavelength (to 528 nm) due to contraction of the \(\pi\) chromophoric system as compared to porphyrines. The maxima of the \(Q\)- and Soret bands for macrocyclic complexes 2, 3 and 4 containing perfluorinated phenyl rings are shifted hypsochronically as compared to non-fluorinated species. A similar effect of perfluorination was observed in the case of \(In^{III}\) and \(Zn^{II}\) complexes\(^{[5,6]}\) and was explained by stronger stabilization of HOMO than LUMO. The effect of perfluorination on the \(Q\)-band position correlates with electronegativity of the central coordinating atom and for the IIIa group elements is decreased in the order: \(B^{III}\) (387 cm\(^–1\)) > \(Al^{III}\) (381 cm\(^–1\)) > \(Ga^{III}\) (302 cm\(^–1\)) > \(In^{III}\) (222 cm\(^–1\)). In the spectra of non-fluorinated phenyl substituted macrocyclic complexes the intense charge transfer (CT) band from peripheral aryl groups to the central macrocycle is observed: \(Ar\rightarrow\text{subporphyrazine}\) at ca. 400 nm\(^{[14,22]}\) or \(Ph\rightarrow\text{porphyrazine}\) at 450–480 nm\(^{[5,6]}\)). This CT band disappears when peripheral phenyl groups are perfluorinated and become strong electron-acceptors. In addition perfluorination increases the dihedral angles of aryl groups with pyrrole rings of macrocycle,\(^{[6]}\) thus diminishing the \(\pi\)–interaction.

**Fluorescence spectra.** Fluorescence emission and excitation spectra of perfluorinated macrocyclic complexes with IIIa group elements and their non-fluorinated analogues are shown in Figure 4. For porphyrazine complexes \([X]M^{III}\text{P}_{40}\) the values of the Stock’s shifts increase in the order \(Al^{III}\) (4 nm, 140 cm\(^–1\)) < \(Ga^{III}\) (8 nm, 240 cm\(^–1\)) < \(In^{III}\) (15 nm, 300 cm\(^–1\)).

**Figure 1.** Molecular structure of bis(pentafluorophenyl) fumarodinitrile trans-1 with atom numbering and bond length. Thermal ellipsoids are shown with 30% probability.

**Figure 2.** LDI-TOF mass-spectra of macrocyclic complexes 2, 3 and 4 obtained by template cyclomerization of perfluoro-\(\alpha,\beta\)-dicyanostyldiene 1 in the presence of \(MCl_3\) (\(M = B, Al, Ga\)).
The perfluorinated BIII subporphyrazine complex has very low fluorescence ($\Phi_F < 0.01$) and its fluorescence emission spectrum is characterized by very large Stock's shift (51 nm, 1600 cm$^{-1}$). For BIII complex of non-fluorinated hexaphenylsubporphyrazine and other aryl substituted subporphyrazines the very low fluorescence was also observed ($\Phi_F < 0.001–0.03$). At the same time boron(III) complexes of more rigid 1,2,5-thiadiazole and pyrazine fused subporphyrazines exhibit stronger fluorescence ($\Phi_F = 0.11–0.15$), and phenyl substitution on pyrazine rings even increases it ($\Phi_F = 0.31$). Therefore we can make a general conclusion that aryl substituted porphyrazines and subporphyrazines are more sensitive to the factors causing deformation of the macrocycle and promoting radiationless deactivation of the excited states than more rigid (sub)porphyrazines with fused heteroarenes.

The fluorescence quantum yields ($\Phi_F$) of perfluorinated porphyrazines [(Cl)M$\text{PAF}_4$] determined in THF decrease due to heavy atom effect in the order Al$^{III}$ (0.13) $>$ Ga$^{III}$ (0.085) $>$ In$^{III}$ (0.019). Complexes of non-fluorinated octaphenylporphyrazines [(Cl)MP$A_4$] which we have studied for comparison are less fluorescent ($\Phi_F = 0.031$ and 0.012 for $M = \text{Ga}^{III}$ and In$^{III}$, respectively). The effect of brighter fluorescence of perfluorinated complexes, also observed for the Zn$^{II}$ complexes ($\Phi_F = 0.19$ for [ZnPAF$_4$] and 0.12 for [ZnPA]$_8$), was explained by lesser involvement of more orthogonal pentafluorophenyl groups than phenyl groups in non-radiative deactivation of the excited state. Interestingly that complexes of porphyrazines with more rigid macrocycle are more fluorescent and have higher $\Phi_F$ values (e.g. for the Al$^{III}$, Ga$^{III}$ and Zn$^{II}$ complexes of tetakis(1,2,5-thiadiazolo)porphyrazine $\Phi_F = 0.53$, 0.21 and 0.24, respectively). Evidently non-radiative pathways of deactivation become more significant in more flexible aryl substituted porphyrazines allowing stronger deformation of the macrocyclic skeleton in the excited state than in more rigid 1,2,5-thiadiazole fused species resulting in a considerable decrease of fluorescence.

The perfluorinated B$^{III}$ subporphyrazine complex 2 has very low fluorescence ($\Phi_F < 0.01$) and its fluorescence emission spectrum is characterized by very large Stock's shift (51 nm, 1600 cm$^{-1}$). For B$^{III}$ complex of non-fluorinated hexaphenylsubporphyrazine and other aryl substituted subporphyrazines the very low fluorescence was also observed ($\Phi_F < 0.001–0.03$). At the same time boron(III) complexes of more rigid 1,2,5-thiadiazole and pyrazine fused subporphyrazines exhibit stronger fluorescence ($\Phi_F = 0.11–0.15$), and phenyl substitution on pyrazine rings even increases it ($\Phi_F = 0.31$). Therefore we can make a general conclusion that aryl substituted porphyrazines and subporphyrazines are more sensitive to the factors causing deformation of the macrocycle and promoting radiationless deactivation of the excited states than more rigid (sub)porphyrazines with fused heteroarenes.
Conclusions

The interaction of perfluoro-α,β-dicyanostylylene 1 with salts of IIIa group elements allows the corresponding products of its tri- (2) and tetramerization (3, 4). In the case of subporphyrazine 2, [(Cl)BsPF$_6$], the role of cis-trans-isomerization of 1 is more crucial than for formation of porphyrazines 3, 4, [(Cl)MPF$_6$] (M = Al, Ga), for synthesis of which cis-configuration of two cyano groups is not strictly necessary requirement. According to the data of single crystal X-ray diffraction of trans-isomer of 1, the conjugation of dicyanoethylene moiety with pentfluorophenyl fragments is excluded in contrast to non-fluorinated precursor (diphenylfumarodinitrile). Perfluorination of phenyl rings in 2-4 leads to the hypsochromic shift of the Q-band and the disappearance of the CT-band in their UV-Vis spectra. The subporphyrazine 2 is practically non-fluorescent, while porphyrazines 3, 4 acquire slightly more pronounced fluorescent properties at perfluorination.

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