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Organic dyes based on fluorene and its derivatives

I V Kurdyukova, A A Ishchenko

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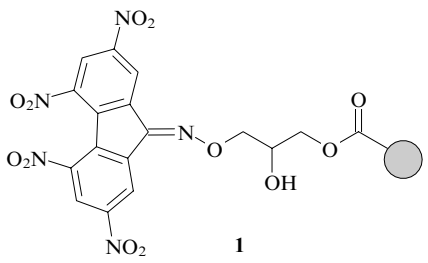
Abstract. Data on various types of organic dyes based on fluorene and its derivatives, including polymethine, styryl, triphenylmethane, spiran, merocyanine, porphyrin and polymeric dyes, as well as azo dyes and donor – acceptor polyenes, are described systematically. The key methods for their synthesis are considered. The properties of the dyes are analyzed and summarized. The principles of development of modern functional materials based on these dyes are outlined. The use of these materials in advanced fields of science and technology such as photovoltaics, electroluminescence, nonlinear optics, holography, sensing photodynamic therapy are considered. The bibliography includes 476 references.

I. Introduction

Organic dyes based on fluorene and its derivatives are widely used in science and technology, in particular, in modern nanotechnology. They are parts of polymer compositions of organic light-emitting diodes (OLED),[1,2] molecular superconducting materials, holographic media and optoelectronic devices,[3–5] and they are also used as molecular and ionic sensors.[6] Antiviral agents[7] and effective antimalarial drugs[8] were found among fluorene derivatives. Using these compounds, new organometallic Ziegler – Natta catalysts were developed,[9–12] and fluorescent platinum complexes[13] that effectively self-assemble on the gold surface[14,15] were obtained. Molecular wires made of fluorene rings with donor and acceptor groups form antenna systems.[16]

At the first glance, fluorene may seem poorly suitable for the design of dyes, because it does not contain traditional auxochrome ionized heteroatomic groups needed to facilitate the displacement of the electron density upon absorption of light quanta.[17] However, the fluorene molecule has an extensive π-system, has high electron symmetry and contains a methylene group, which is potentially active in condensation reactions owing to conjugation with benzene rings. The acidity of the protons of this group, and, hence, its reactivity, vary over a broad range depending on the electronic nature of the substituents in the aromatic rings. For example, the following acidity constants pK_a were reported:[18–22] 24.2 for 2-dimethylaminofluorene, 14.6 for 2,7-dicyanofluorene, and ~1.2 for 2,4,5,7-tetranitrofluorene. This attests to a great potential of synthetic methods involving the methylene group providing practically important dyes with gradually changing donor-acceptor properties.

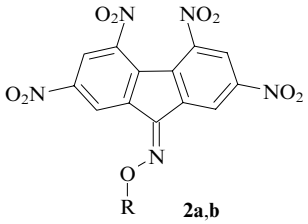
A special niche among fluorene derivatives is occupied by polynitro compounds. They are distinguished not only by their reactivity but also by specific physicochemical behaviour. Owing to the strong electron-withdrawing action of the nitro groups, these derivatives are readily condensed at position 9.[23–25] Polynitrofluorenes can form charge transfer complexes with various π-donors.[26–37] This feature was employed to develop a desulfurization process for aromatic fractions of oil[27] and later to design sorbents (for example, derivatives **1** are modified polyglycidyl methacrylate grains) for effective removal of aromatic nitrogen compounds from oil fractions.[36,37] The synthesis was reported and the complexation ability towards aromatic compounds was studied for [(±)-2-(2,4,5,7-tetranitrofluoren-9-ylideneaminooxy)]propionic acid (TAPA, **2a**).[38] Its complexes have been used for partitioning of polycyclic aromatic compounds.



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2a,**b**

R = CHMeCO$_2$H (**a**), CH(CO$_2$(CH$_2$)$_2$OH)CH$_2$CO$_2$(CH$_2$)$_2$OH (**b**).

Optically active *O*-alkyl derivative **2b** of 2,4,5,7-tetranitrofluorenone oxime, which has two ester groups in the alkyl fragment, was used to separate racemic aromatic compounds *via* diastereomeric π-complexes.[39] Chlorophyll *a* complexes with trinitro fluorene derivatives were studied by absorption and fluorescence spectroscopy.[40] It was shown that complexation reduces the fluorescence intensity of chlorophyll. Complexes with polymers form the basis of new organic materials for photovoltaics owing to their capability for effective charge photogeneration.[41–43] The complexation with chiral derivatives of nitrofluorene can change the chirality of other molecules: thus racemic heterohelicene was converted to a single enantiomer.[44, 45] The ability to form complexes and to effectively transfer charge was used to synthesize numerous fluorene-based dyes.

All of the benzene hydrogen atoms of fluorene derivatives can be involved in substitution reactions under certain conditions. The atoms in positions 2, 7 and 4, 5 are most reactive. Functionalization of the benzene rings opens up the way to the synthesis of unique star-like or dendrimer dyes with several chromophores.

The lack of auxochrome heteroatomic groups in the fluorene chromophore may in some cases be a weighty advantage over the traditional heterocyclic chromophores rather than a drawback. The point is that the absence of heteroatoms in the dye molecules rules out the possibility of the forbidden n–π transitions, which often form a powerful channel for the photodestruction of organic compounds.[46] They also induce non-radiateve deactivation of electronically excited states, resulting in a substantial decrease in the fluorescence ability of the dyes.[47–49]

The introduction of the fluorene nucleus into a chromophore system is beneficial for the increase in the fluorescence quantum yields (Φ) not only due to the absence of heteroatoms but also due to its planarity, structural rigidity and the presence of extensive π-systems. Fluorene-based dyes with fluorescence quantum yields varying over a broad range, 0.36–1.02, were reported (9,10-diphenylanthracene with $\Phi = 0.86$ was used as the standard).[50, 51] All of these compounds are readily soluble and highly photostable. They are widely used in the production of light-emitting materials,[52–66] fluorescent indicators and sensors.[67]

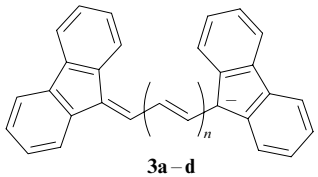
These structural and behavioural features of fluorene and its derivatives gave a strong impetus to the development of various classes of dyes with a broad range of physicochemical, nonlinear optical, electrical and magnetic properties. Fundamentally new types of dyes were developed. First of all, mention should be made of polymeric dyes, which are swiftly introduced into optoelectronic and photovoltaic devices. Numerous papers and patents devoted to various aspects of the fluorene dye chemistry have been published to date. However, no reviews on this topic are available.

This review is the first attempt to give a systematic account of various types of fluorene-based organic dyes known to date and to analyze and summarize the results of their studies. The methods for preparation of these dyes, their properties and applications in various areas are considered. The review discusses the achievements in the field of fluorene dyes and functional materials based on them made mainly in the last decade. Some pioneering works of the second half of the 20th century that are the starting points for the understanding of the subject were also included.

II. Various classes of dyes based on fluorene and its derivatives

1. Polymethine dyes

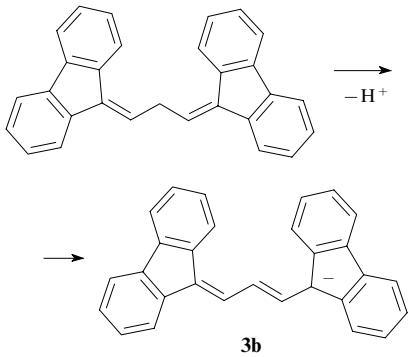
The polymethine dyes with anions **3a**–**d** based on unsubstituted fluorene were first described in a series of publications of Kuhn and Fischer[68–72] devoted to new polyene hydrocarbons.



3a–**d**

n = 0 (**a**), 1 (**b**), 2 (**c**), 3 (**d**).

They were prepared by deprotonation of the corresponding unsaturated hydrocarbons induced by strong bases, for example:



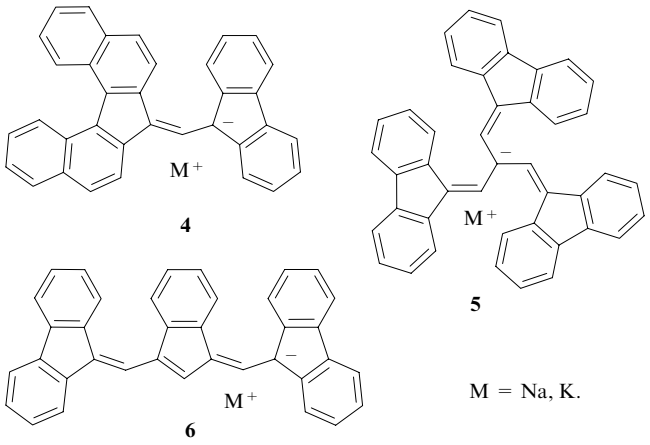
3b

The energies of their electronic transitions were calculated by the Hückel method.[73] Dyes **3** demonstrated the possibility to produce a deep colour, although they do not have auxochrome groups in the chromophore. The absorption maximum of pentamethine **3c** is located at 740 nm.[71, 74] However, dyes **3** were unstable in air.

Aza analogues of compounds **3** in which one methine group was replaced by a nitrogen atom were reported.[70] The acidity of polyenes **3a**,**b** was determined under various conditions; the average pK_a values were 14.0 and 12.3, respectively.[75, 76] The same researchers[71] prepared coloured free radicals of a similar structure stable in solution and recorded the electronic absorption spectra for them. These radicals were found to dimerize and to react slowly with air oxygen to give peroxides; the peroxide formation was confirmed by positive potassium iodide test reaction and by characteristic signals in the IR spectra.

Cationic dyes with the same carbon skeleton were obtained by treating alcohols that are formed as intermediates in the synthesis of unsaturated hydrocarbons with perchloric acid. However, the cations were much less stable than the anions or the radicals. Only colours of their solutions were described.[71, 73]

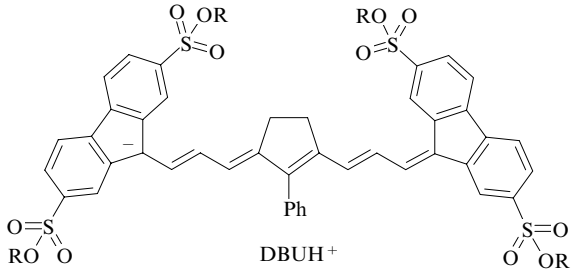
Kuhn's group synthesized the fluorene-based polyenes that were deprotonated to afford unsymmetrical anionic dyes, for example, compounds **4**, tris-dyes **5** and dyes in which the polymethine chain comprises a ring, for example, salts **6**.[77]



Later, dyes **3a**,**b** and **6** were mentioned in a study[78] dealing with acidity of weak CH acids. Anions **3** and **6** were found to form different types of ion pairs with various metal cations, which affects their spectral properties and pK_a values. Solvent-separated ion pairs with the lithium cation absorb at longer wavelengths than contact ion pairs with the caesium cation. The pK_a values of anions **3** and **6** incorporated in solvent-separated ion pairs are lower than these values for contact ion pairs.

Quantum chemical analysis of the electron density distribution in dyes **3** showed that of all possible fluorene positions suitable for substitution, only for positions 2 and 7, the susceptibility for substitution increases upon excitation.[79] According to the perturbation MO theory, electron-withdrawing substituents present in these positions are expected to induce a long-wavelength shift of the absorption bands. In addition, substituents in positions 2 and 7 of the fluorene moiety are *meta*-directing groups relative to substituents in position 9 but are not directly conjugated with them. However, owing to the presence of the five-membered ring, they can become conjugated with these substituents *via* a longer route involving all bonds of this ring and the benzene ring. Therefore, 2- and 7-substitution produces the longest chain of conjugation compared with any other substitution (see structure **7**). This also makes an additional contribution to deepening of the colour of 2,7-disubstituted fluorene compounds (DBUH$^+$ is protonated 1,8-diazabicyclo[5.4.0]undec-7-ene) (Scheme 1).

Therefore, the absorption maximum of anionic polymethine dye **7** based on bis(2,2,3,3,4,4,5,5-octafluoropenyl)-9*H*-fluorene-2,7-disulfonate[79] occurs in the near-IR region (λ_{max} = 790 nm). It is noteworthy that anionic dyes based on widely used diethylbarbituric acid acceptor moiety, which markedly deepens the colour, absorb light at 130 nm shorter wavelength than pentamethine **7**, the length of the polymethine chain being the same. The absorption bands of cationic pentamethine dyes derived from classical indolenine, benzothiazole and benzoxazole heterocycles are shifted to shorter wavelengths by approximately 120 – 150 nm with respect to the band of compound **7**.[80, 81] Note that nonamethine derivative **8** with a five-membered ring in the δ,δ′-positions of the chain



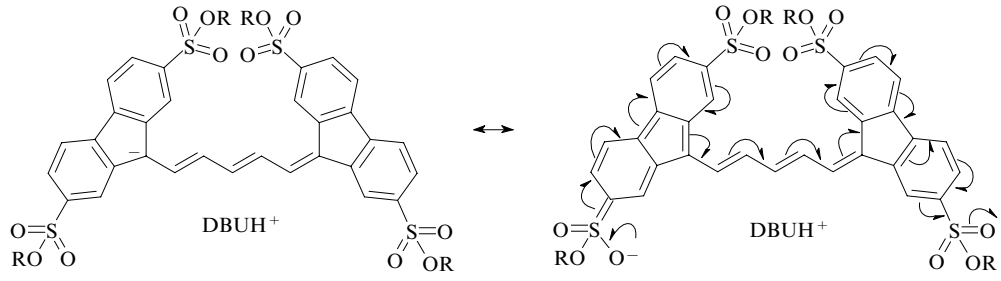
8

absorbs light at the longest wavelength (λ_{max} = 1086 nm) among the known anionic polymethine dyes.[79]

2. Styryl dyes

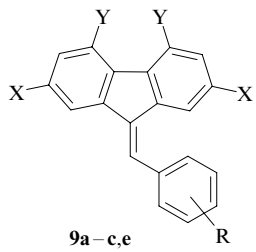
Fluorene-based styryl dyes have been synthesized using a number of approaches. Fluorenes with an active methylene group, for example, polynitrofluorenes, readily condense with aromatic aldehydes. Derivatives **9a** were obtained in this way.[24] Styryl dyes derived from less reactive fluorenes or deactivated aldehydes were prepared by the Wittig (ylide) reaction. Phosphonium ylides were either prepared on the basis of the fluorene nucleus and then made to react with aromatic aldehydes,[82] or on the basis of the aryl moiety.[83] In this case, a substituted fluorenone functioned as the second component. Series of styryl dyes **9b** and **9c** were synthesized from the corresponding fluorenones and various phosphonium ylides, which formed from phosphonium salts *in situ*.[83]

Scheme 1



7

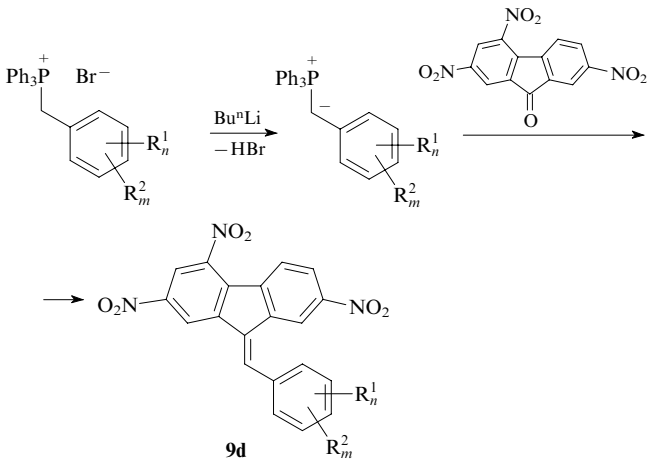
R = $CH_2(CF_2)_3CF_2H$.



9a–c,e

X = Y = NO_2: R = H, 4-NO_2, 3-NO_2, 4-Br, 4-Cl, 4-OMe, 4-NMe_2, *etc*. (**a**);
X = NO_2, Y = H: R = H, 2-NO_2, 3-NO_2, 4-Cl, 4-Br, 4-Me, 4-OMe, *etc*. (**b**);
X = Br, Y = H: R = H, 2-NO_2, 3-NO_2, 4-Cl, 4-Br, 4-Me, *etc*. (**c**);
X = Y = R = H (**e**).

This synthetic method was described also in Japanese patents[84,85] devoted to unsymmetrically substituted fluorene derivatives **9d**.

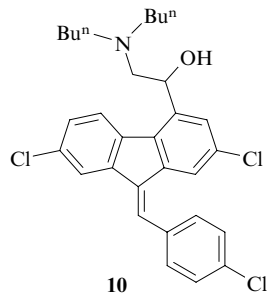


9d

R^1 = Alk, OAlk, Hal; R^2 = NO_2, haloalkyl;
m = 1–3, n = 0–4, $m+n$ = 1–5.

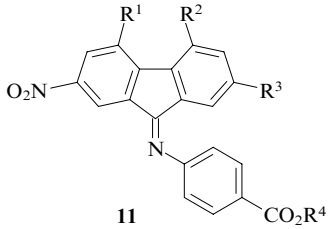
Compounds **9a** have found use as photosensitizers and active components of photoreceptors.[86]

Unsubstituted styrylfluorene (**9e**) was taken as an example to study the effect of the counter-ion and the solvent on the product yield in the reaction of fluorene alkali metal salts with benzaldehyde. On going from lithium to caesium or tetrabutylammonium cations, the product yield increases but the reaction rate decreases. Upon replacement of dimethoxyethane by diethyl ether or benzene, the yield of the styryl decreases, while in hexamethylphosphorotriamide (hexametapol), the product yield, conversely, increases.[87] Beletskaya *et al*.[88] studied the phase transfer-catalyzed synthesis of styryls. The activity of various catalysts and the ratio of the reaction products depending on the concentration of the base and the catalyst used were studied. Styryl dye **10** (lumefantrine, or benflumetol)[8,89–91] is currently one of most widely used drugs against malaria. 2-Dibutylamino-1-(fluoren-4-yl)ethanols, which are precursors of this compound, were tested as antimalarial agents.[92]



10

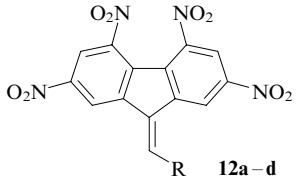
Nitrofluorenone Schiff's bases **11**, aza analogues of styryl dyes, were reported.[93] They were synthesized by condensation of *p*-aminobenzoic acid or its esters with appropriate nitrofluorenones. It was shown that the unsymmetrical Schiff's bases exist as *syn*- and *anti*-isomers, which exist in equilibrium at elevated temperature.



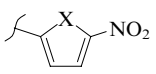
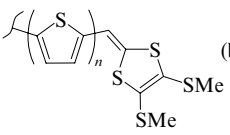
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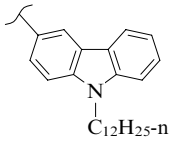
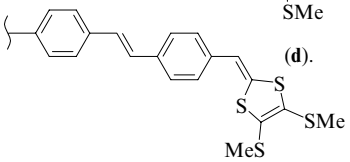
R^1, R^2, R^3 = H, NO_2; R^4 = H, Et, Na.

Compounds **12a–d** prepared by coupling of the electron-withdrawing moiety of tetranitrofluorene with diverse electron-donating heterocycles are hetero-analogues of styryl dyes.[4,94–96] They have noticeable intramolecular charge transfer (IMCT) and are promising for the design of optoelectronic materials.[4]

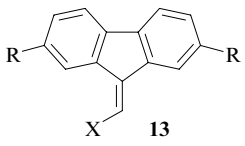


12a–d

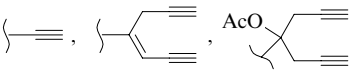
R =  (X = O, S) (**a**),  (**b**),

 (**c**),  (**d**).

Nitro fluorenone derivatives were converted, *via* the Wittig reaction, to interesting styryl-like enynes, enediynes and dienediynes of general formula **13**. They were assayed for cytotoxicity with respect to human carcinoma cells but the activity was too low.[97]

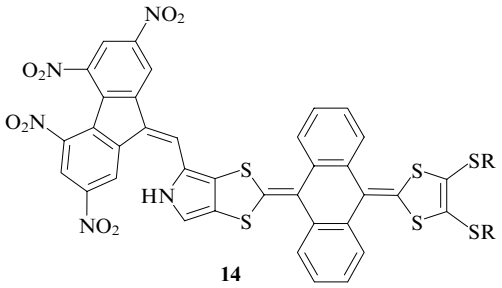


13

X = ; R = H, NO_2.

Heterostyryls **14** can adopt two different conformations caused by the chair-like shape of 9,10-bis(1,3-dithiol-2-ylidene)-9,10-dihydroanthracene moiety, which was confirmed by the ^{1}H NMR spectra. The absorption spectra of

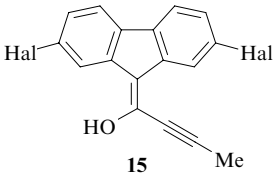
these compounds exhibit a distinct charge transfer band, which is considered to be responsible for their nonlinear optical properties.[98]



14

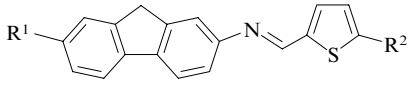
R = Me, Bu^n.

Dihalofluorene derivatives **15** are able to form inclusion complexes with various alcohols and thus facilitate their isolation from aqueous solutions.[99]



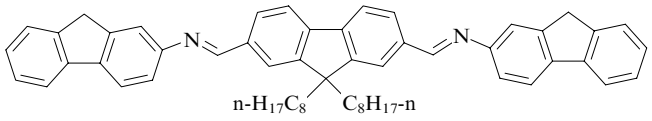
15

Dufresne *et al.*[100–102] described fluorene-, fluorenone- and thiophene-based azastyryls (azomethines) (in particular, compounds **16**, **17**) substituted at fluorene position 2 rather than position 9 as usual. These compounds are prone to deactivation of the excited state through phosphorescence, and they are promising for the development of new light-emitting materials.



16

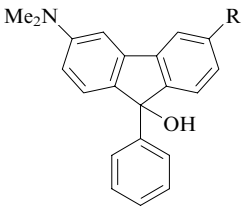
$R^1 = R^2 = H$; $R^1 = NH_2$: $R^2 = H, NO_2$; $R^1 = H$, $R^2 = NO_2$.



17

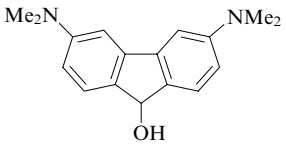
3. Analogues of triphenylmethane dyes

Fluorene analogues malachite green and crystal violet were prepared by elimination of the hydroxy group from compounds **18a**,**b** upon acidification of solutions. The cations of fluorene derivatives showed substantial bathochromic shifts compared with triphenylmethane dyes. However, the analogue of the Michler's hydrol blue **19** was colourless. This is caused by the fact that no hydroxy group elimination occurs for compound **19**. Instead, one dimethylamino group is protonated, and compound remains colourless.[103]



18a,**b**

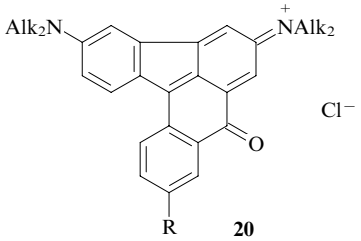
R = H (**a**), NMe_2 (**b**).



19

Brown and Dewar[104] carried out LCAO-MO calculations of the spectral characteristics of these dyes. Ground and excited state parameters were calculated with fairly high accuracy. The results of these calculations suggested that the deeper colour of the fluorene analogues of malachite green and crystal violet is caused by the presence of additional C–C bonds. The authors also calculated, by the perturbation method, the differences between the spectral characteristics of analogous diphenylmethane and fluorene derivatives brought about by the new C–C bond. These calculations predicted the correct trend but much smaller spectral shifts (compared to the experimental values). It was concluded that the substantial bathochromic effect is not a direct result of the formation of the new bond but is caused by transition from the alternant system of the triphenylmethane dye to the non-alternant fluorene system.

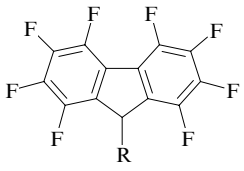
Deeply coloured fluorene-based triarylmethane dyes **20**, structural analogues of rhodamine, were described. Their electronic absorption spectra exhibit two bands at 770 and 835 nm. Compounds **20** are promising as light filters for the near-IR region.[105]



20

R = H: Alk = Me, Et; R = SO_3H, Alk = Me.

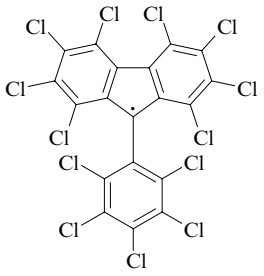
A number of works[106–115] report methods for the synthesis of octafluorofluorene and its derivatives **21**, which are structural analogues of triphenylmethane dyes. For polyfluorinated compounds, the spectral characteristics,[112] acidity[113] and crystal structures[114] were determined. Polyfluorofluorene derivatives of this type are used as components of catalysts for olefin polymerization.[115–117]



21

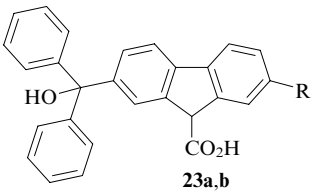
R = $C_6H_3(CF_3)_2$-3,5, C_6F_5.

Perchloro-9-phenylfluorenyl radical (**22**)[118, 119] is one of the first stable free radicals for which mass spectra were recorded. These spectra proved to be almost identical to the spectra of the corresponding neutral compounds. Radical **22** and neutral compounds of similar structure were proposed as mass-spectrometry standards for m/z range of 300–700.[120]



22

Mono- and disubstituted fluorenecarboxylic acids **23a**,**b** were prepared.[121] In concentrated sulfuric acid, they form coloured cations; in the case of compound **23b**, this cation can be considered as a bis(triphenylmethane) dye.



23a,**b**

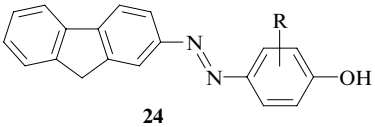
R = H (**a**), CPh_2OH (**b**).

4. Azo dyes

Azo dyes are known as components of high-performance light-sensitive materials used in modern electrophotographic matrices and other data recording means. They are used in printing and copying industry as components of photoreceptors of laser printers. These materials have very good electrophotographic characteristics (such as the ability to retain the surface electric charge, minimum dark discharge, sensitivity, low residual potential, *etc.*); they have sufficient sensitivity in the long-wavelength region (up to 780 nm); on repeated use, they almost do not lose their characteristics.[122]

Monoazo dyes are usually prepared by introduction of a substituent into fluorene position 2 or, more rarely, position 9. Bisazo dyes are based on 2,7-substituted fluorene nucleus. Polyazo dyes are obtained by various combinations of substituents in positions 2, 4, 5, 7 and 9 of fluorene.

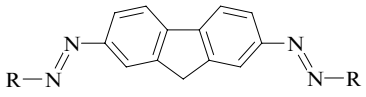
Structures **24** were first mentioned back in the 1940s.[123] Later, these compounds were studied by Krasovitskii [124–126] and Ray and Peters.[127]



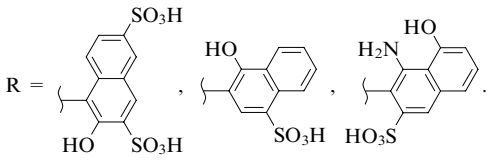
24

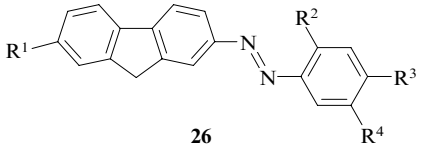
R = H, OH, $(OH)_2$, Me, OMe, *etc.*

Krasovitskii and coworkers focused attention on the effect of planarity of the biphenyl core on the spectral properties of the dyes. It was shown that compound **25** absorbs light at longer wavelength than the corresponding biphenyls.[124, 125] The dependence of spectral properties of monoazomethines on the nature of the substituents in the aldehyde moiety was also investigated.[126] Ray and Peters [127] synthesized a number of diazo dyes **26** and studied their carcinogenic properties. The scientists were especially interested in the ability of these compounds to be detected in the gastric juice after intraperitoneal injection and to be cleaved under these conditions to an active carcinogen, 2-aminofluorene. Monoazamethines formed by substitution at other positions of the fluorene nucleus were reported in another publication.[128]



25

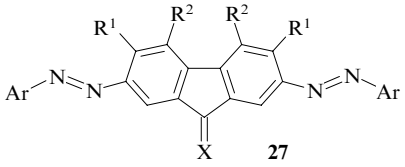




26

R^1 = H, NHAc; R^2 = NH_2, NMe_2, OH, Me;
R^3 = NH_2, NMe_2, OH; R^4 = H, Me.

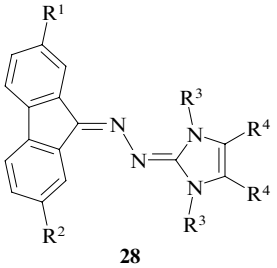
Fluorene-based azo dyes received much attention of Japanese researchers in the 1980s. This work resulted in a series of patents,[129–149] which describe diverse azo dyes corresponding to general formula **27**. These are mainly 2,7-diazo dyes but a number of 2,4,5,7-derivatives are also included. These compounds are used as components of photoreceptors for copying devices and printers.



27

X = 2 H, O, $C(CN)_2$, CHAr, CHHet, $CH(CH{=}CH)_n$Het;
R^1, R^2 = H, Me, N=NAr.

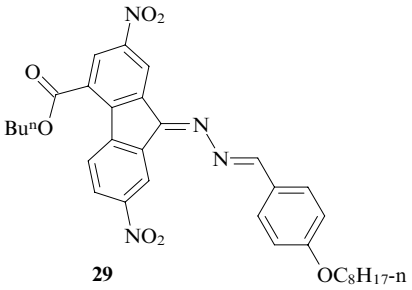
Kleinpeter and Stamboliyska [150] calculated the hyperpolarizability of fluorofluoreneazines **28**, in particular, 2,7-dichloro-, 2,7-dibromo- and 2-fluoro-substituted derivatives. These calculations led to the conclusion that compounds of this structure may be promising as nonlinear optical materials.



28

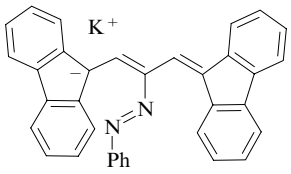
R^1	R^2	R^3	R^4
H	H	Mes	H
Br	Br	Mes	H
H	F	Mes	H
Cl	Cl	Mes	H
H	H	Pr^i	Me

Diazostyryls based on nitrofluorenes, *e.g.*, derivative **29**, were prepared and studied. The IMCT inherent in them makes them useful for holography, optical data media, photovoltaics and light-emitting diodes.[151]



29

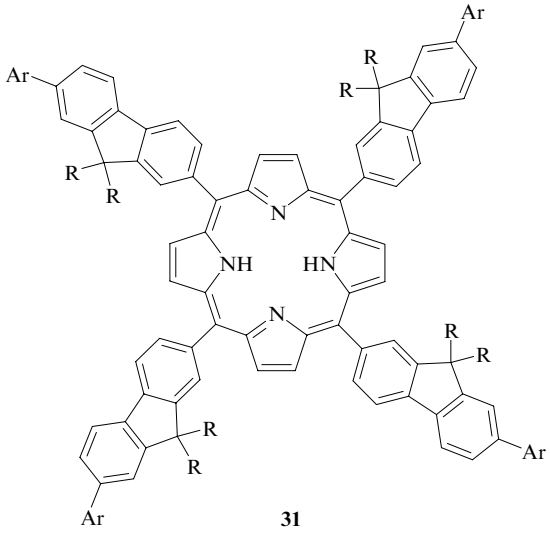
Azo dye **30** in which the azo group is a substituent in the polymethine chain of the symmetrical fluorene-based anionic dye was reported. Upon acidification of the solution of this compound, the azo group is protonated. The solution colour changes from dark blue to red-orange and neutral phenylhydrazone precipitates.[152]



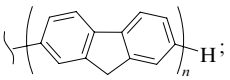
30

5. Porphyrin derivatives

Compounds **31** based on porphyrin modified with fluorene residues were reported. Formerly, porphyrin derivatives were used as dopants to fluorene polymers to prepare red light-emitting materials for organic light-emitting diodes. The creation of a common π-conjugated system from moieties used previously as a host (fluorene) and a guest (porphyrin) could solve a lot of problems of mixed light-emitting materials. The combination of these moieties into a single molecule resulted in higher solubility and higher Förster resonance energy transfer (FRET) rate, prevented the aggregation of porphyrin nuclei, and, hence, self-quenching of fluorescence and facilitated the preparation of homogeneous amorphous films optimal for the design of OLED. The fluorescence quantum yield of dyes **31** is higher than that of traditionally used porphyrin derivatives. These dyes are highly efficient in transfering the light energy from the fluorene substituents to the porphyrin core (the transfer efficiency in solution is 99% for the dye with pyrene moieties).[153, 154]



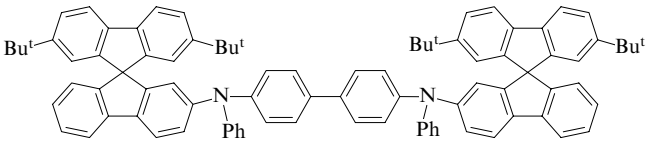
31

R = n-C_8H_{17}, Ar = ;

R = $CH_2CHEtBu^n$: Ar = C_6H_4CN-4, C_6H_4Ac-4, pyren-2-yl.

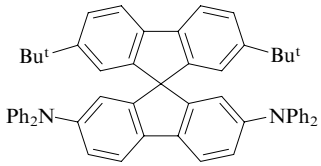
6. Spirans

Spiro compounds with the fluorene C(9) spiro atom demonstrate high fluorescence quantum yields, thermal and photo stability, and, hence, they are promising for the creation of light-emitting materials.[155–158] For example, for dye **32** with fluorescence quantum yield of 82%, the glass transition temperature is 205 °C.[155]



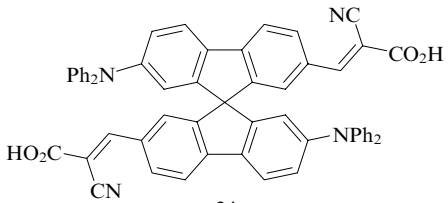
32

For light-emitting devices in which spirocyclic dye **33** serves as the hole-conducting layer, more than 4000 h of service life at a brightness of 300 cd m^{-2} was predicted.[156]



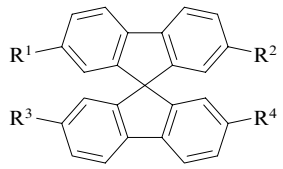
33

Spirocyclic dye **34** demonstrated good performance characteristics when used in TiO_2-based solar cells. Due to the spiro structure, it fails to aggregate but strongly binds to the TiO_2 surface and demonstrates an efficiency of light energy conversion to electric energy of up to nearly 4%.[159]



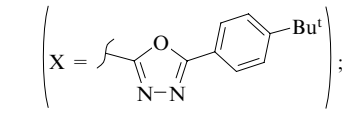
34

The unsymmetrically substituted spirofluorenes **35a–d** show fast photoinduced electron transfer with its rate being dependent on the ratio of the donor to acceptor substituents in the compound.[160] The absorption spectra of these systems depend little on the solvent polarity, but they exhibit considerable positive solvatofluorochromism. Therefore, as the solvent polarity increases, the Stokes shift grows but the fluorescence intensity decreases.[161] The electroluminescence of spirocyclic fluorenes **35** is related to the radical ion annihilation upon electrochemical oxidation or reduction. Presumably, the triplet–triplet annihilation mechanism is involved. On average, the electroluminescence maximum is shifted by 30 nm to longer wavelengths as compared with the photoluminescence maximum.[162]



35a–d, **36**

35: $R^1 = H$, $R^2 = NPh_2$: $R^3 = H$, $R^4 = X$ (**a**); $R^3 = R^4 = X$ (**b**);
$R^1 = R^2 = NPh_2$: $R^3 = H$, $R^4 = X$ (**c**); $R^3 = R^4 = X$ (**d**)

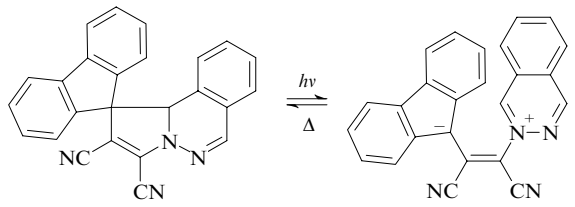


36: $R^1 = R^2 = H$, $R^3 = R^4 = C_6H_4Bu^t$-4.

Fluorenes **35** proved to be highly efficient photodetectors for type A ultraviolet radiation, which are simultaneously insensitive to the visible light. This makes theses compounds promising for the use in biochemistry, medicine, environmental monitoring, astronomy, *etc.*[163]

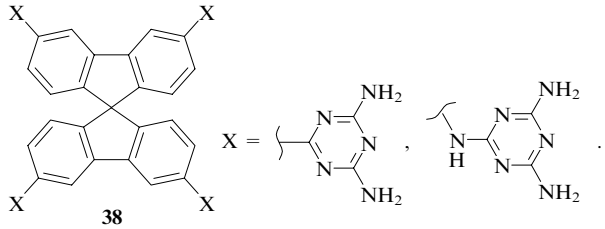
Ambipolar amorphous materials based on one chromophore are rarely encountered. Spirocyclic fluorenes are most promising for the design of economically rational ambipolar semiconductor materials. They are subject to fine tuning of the electron and hole conductivity balance upon modification by various substituents. Taiwan scientists[164] synthesized spirofluorene **36**, which possesses nearly identical conductivities of both types.

A number of photochromic spirocyclic compounds based on fluorene, for example, derivative **37** was reported.[165,166] The spiro fragment of these compounds tends to open on exposure to light. As this takes place, they are converted to coloured compounds similar to cyanine dyes. On heating, the reverse process takes place.



37

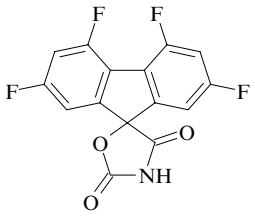
Crystalline spirocyclic fluorene derivatives **38** form framework structures with high porosity (up to 75%), which is the highest porosity for small-size molecules.[167] These structures can be used subsequently for separation (chromatography), catalysis and in other areas, as they are able to form host–guest type complexes.



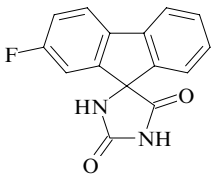
38

Spiro compounds **39** and **40** with various heterocyclic moieties were tested as drugs preventing diabetes and nervous system and vascular disorders.[168] Hydantoin fluorene derivatives **40** are also effective for preventing the development of complications that accompany diabetes mellitus, in particular, cataract and neuropathy.[169]

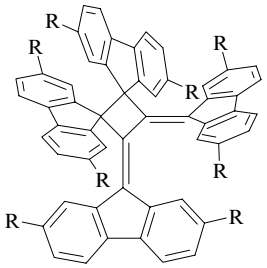
Spiro compounds **41** of unusual structure can form host–guest clathrates with low-polarity molecules.[170]



39



40



41

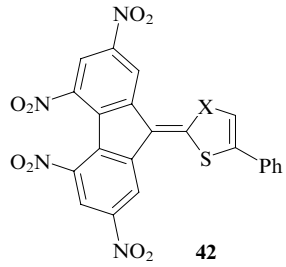
R = H, Me, Bu^t.

7. Donor–acceptor polyenes

This group of dyes includes donor–acceptor polyenes in which the fluorene moiety is conjugated to moieties of other π-systems either through position 9 or through the benzene ring.

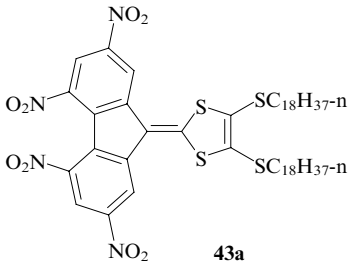
Mysyk and Perepichka[171] described for the first time dithiolylidene and thioselenolylidene derivatives of polynitrofluorenes **42**, **43a**. Subsequently these compounds were studied in detail and vinylogues of compounds **43a** (**43b**,**c**) and related polyheterocycles **44–47** were prepared. In donor–acceptor polyenes **42–47**, the electron-withdrawing polynitrofluorene nucleus is conjugated through position 9 with the electron-donating chalcogenofulvalene nuclei. These compounds refer to the merocyanine-type dyes. Owing to the IMCT, they were effective holographic sensitizers of carbazole-based photothermoplastic polymers.[172–179] Being introduced in the polymer film, merocyanine **43b** considerably increases the photoresponse in the far-visible and near-IR region without reducing the diffraction efficiency of holograms.[175] The publications cited[172–176] gave impetus to the search for new types of merocyanines with strong IMCT.

Compounds **48a**,**b**, which demonstrate the IMCT, differ slightly in the chemical structure but have different symmetry and form different supramolecular crystal structures, because these structures are caused by the dipole–dipole interactions. Asymmetric compound **48a** forms aggregates resembling flower buds, while symmetric compound **48b** forms tubular aggregates. The shape of supramolecular structures is also affected by the conditions of cooling of the solutions. Suspensions of these nanostructures differ by spectral characteristics.[180]

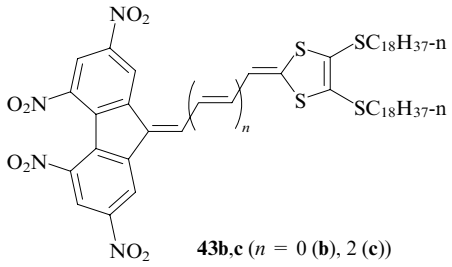


42

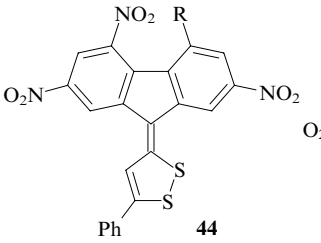
X = S, Se.



43a

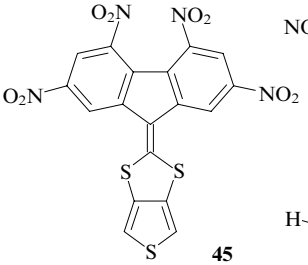


43b,c (n = 0 (**b**), 2 (**c**))

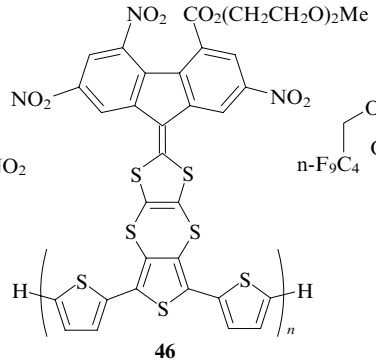


44

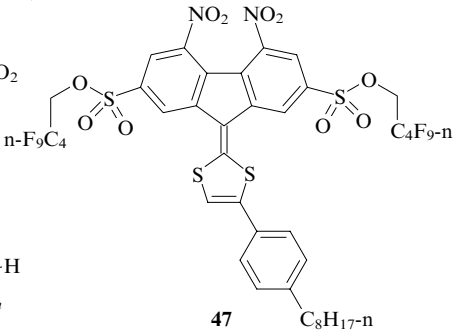
R = H, CO_2H, CO_2Me, NO_2.



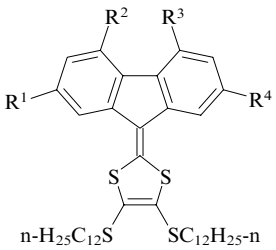
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46

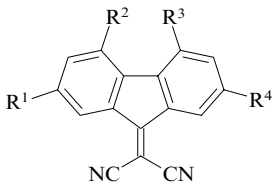


47



48a,b

$R^1 = R^3 = R^4 = NO_2$, R^2 = H (**a**);
$R^1 = R^2 = R^3 = R^4 = NO_2$ (**b**).



49a–c, 50a–c, 51, 52

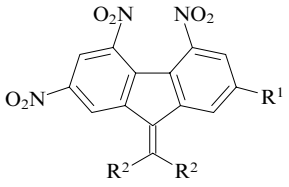
Compound	R^1	R^2	R^3	R^4
49a	NO_2	NO_2	CN	NO_2
49b	NO_2	NO_2	NO_2	CN
49c	NO_2	NO_2	CO_2H	NO_2
50a	SO_2Bu^n	NO_2	NO_2	NO_2
50b	SO_2Bu^n	NO_2	NO_2	SO_2Bu^n
50c	SO_2Bu^n	NO_2	SO_2Bu^n	NO_2
51	SO_2NH_2	NO_2	NO_2	SO_2NH_2
52	H	H	H	$SO_2C_6H_2Pr^i_3$-2,4,6

Dicyanomethylidene fluorene derivatives with IMCT have been repeatedly investigated, and the investigations concerned the complexation of these compounds, sensitization of polymers and the electrochemical behaviour. These compounds were actively studied by Perepichka's group, first independently and then in cooperation with the Bryce group.[26,181–183] Polynitrofluorene derivatives were the first to be described; then the range of electron-acceptor substituents in 2-(9*H*-fluoren-9-ylidene)malononitriles was extended by cyano, sulfo and sulfoester groups.[184–186] For compounds **49a–c**, the effect of the nature of substituents on the electron-withdrawing properties (electron affinity) and complex-forming properties was studied; the redox potentials were estimated.

The complexation of compounds **49** with aromatic donor compounds was investigated. The formation of the charge transfer complex with poly(*N*-epoxypropylcarbazole) in dioxane solutions, resulting in more efficient sensitization of the photoconductivity of this polymer, was investigated.

Cyclic voltammetric studies of compound **50a–c** demonstrated the formation of electrochemically stable tetraanions.[186] For some of the compounds, for example, for compounds **51**, the crystal structures were described.[187] The crystals of the 2,7-dicyano-9-dicyanomethylidene-4,5-dinitrofluorene complex with benzonitrile were obtained.[188] Dicyanomethylidene fluorene derivatives such as **52** were patented as electron transport agents in electrophotographic elements.[189,190]

Pronounced IMCT was observed upon the introduction of the amino group into molecules with a nitrofluorene moiety (compounds **53a,b**).[191,192] This results in very easy rotation about the C(9)=C(α) double bond of molecule **53a**; this rotation was detected by ^{1}H NMR spectroscopy.

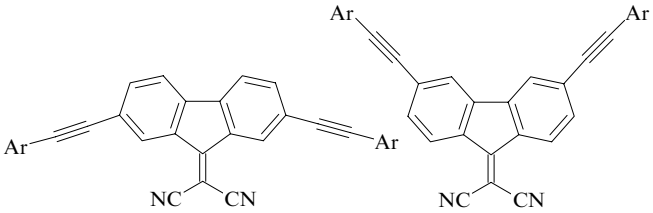


53a,**b**

$R^1 = NO_2$, $R^2 = NMe_2$ (**a**); $R^1 = NMe_2$, $R^2 = CN$ (**b**).

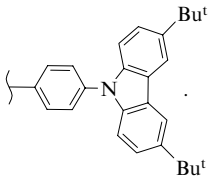
Tetranitrofluorenones and other nitro fluorene derivatives are often used as photosensitizers for carbazole-based polymers.[193–196]

Ambipolar malonodinitrile fluorene derivatives **54a**,**b** were reported.[197]



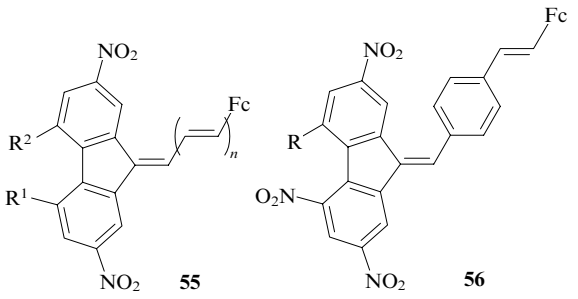
54a **54b**

Ar = Ph,



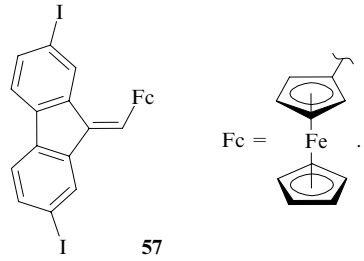
The dependence of the photophysical properties of compounds **54a**,**b** on the substituent position in the ring was investigated. The substituents in positions 2 and 7 mainly delocalize the electron density and substituents in less typical positions 3 and 6 effectively interact through the fluorene skeleton with substituents in position 9. If electron-donating groups are introduced in positions 3 and 6 and electron-withdrawing groups are introduced in position 9, this may give an antenna system with effective electron transfer. This transfer is one of the fluorescence quenching paths for compounds **54b** at room temperature, although a deactivation mechanism through the formation of rotation conformers cannot be ruled out either.

Merocyanine ferrocene derivatives **55** and **56** were reported.[95,198] They tend to exhibit pronounced IMCT; hence, they are of interest as materials for nonlinear optics,[95,198] photothermoplastic holography and magneto-active media.[199] Diiodo derivative **57** was used as the monomer unit in the synthesis of the copolymer with 1,4-bis(decyloxy)-2,5-diethynylbenzene.[200]



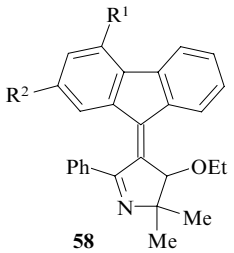
55 **56**

R^1, R^2 = H, NO_2, CN, CO_2Me. R = NO_2, $CO_2(CH_2CH_2O)_3Me$.



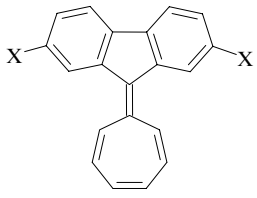
57

Under the action of visible light, structures of type **58** were found to undergo photoisomerization. These compounds are biomimetic photochemical switches (they are similar to the retinal chromophores). The fact that rotation in these molecules is induced by visible light opens up prospects for their use for monitoring ionic complexation and catalysis and as molecular motors.[201–210]



58

R^1 = H, R^2 = CO_2Me;
R^1 = CO_2Me, R^2 = H.

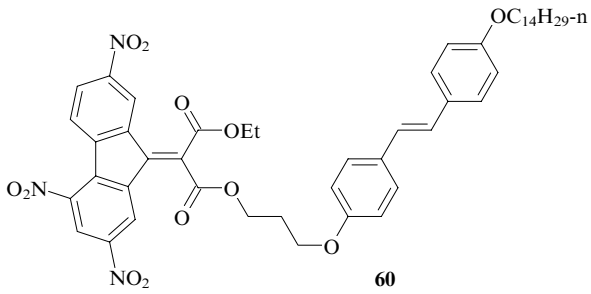


59a–d

X = H (**a**), C≡CNaph (**b**) (Naph is 1-naphthyl), C≡CPh (**c**), $C_6H_4NMe_2$-4 (**d**).

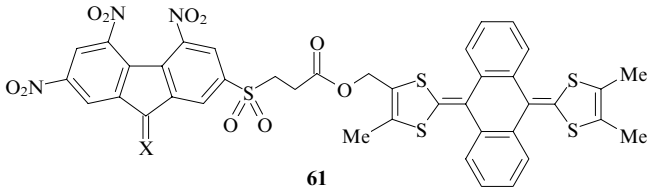
The spectral luminescence properties of fluorene derivatives **59a–d** containing a 9-cycloheptatriene moiety[211–213] appreciably depend on the pH.[214–216] Dye **59d** was proposed as a highly efficient pH sensor in the pH range of 0.97–7.06. The so broad range of sensitivity is caused by stepwise protonation of the molecule, first, of the dimethylamino groups of the substituents and next of the fluorene nucleus with positive charge redistribution to the tropylium moiety.[216]

Numerous fluorene-based compounds characterized by through-space charge transfer, unlike dyes **42–58** with the IMCT along the π-system, have been synthesized. Polynitrofluorene moieties serve as the terminal electron-withdrawing groups. Thus compound **60** forms a Langmuir–Blodgett monolayer at the water–air interface; fluorescence quenching in this monolayer is due to the energy transfer from the donor stilbene moiety to the acceptor trinitrofluorene moiety.[217]



60

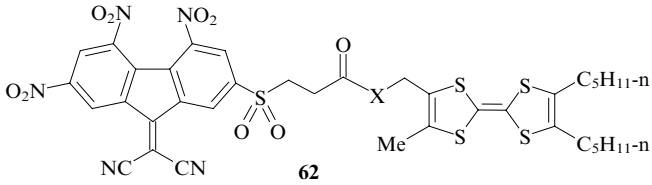
For the covalent D–A conjugates (D is donor, A is acceptor) of fluorene with tetrathiafulvalene **61**, the unusual covalent redox state $D^{2+}/A^{-\bullet}$ was observed for the first time.[218, 219] This feature of systems of this type stimulated investigations of compounds containing strong donor (tetrathiafulvalene, thioxanthene) and acceptor (polynitrofluorene) moieties.[220–223]



61

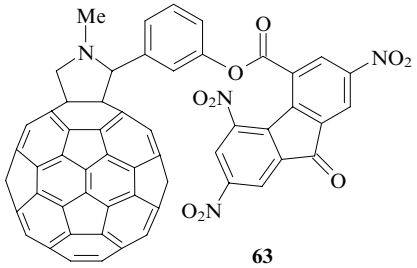
X = O, $C(CN)_2$.

The preparation of polycyclic compounds **62**–**65** has been reported. Under certain conditions, the charge transfer in this type of molecules may be very efficient. Thus compound **63** has a long-lived state with separated charges (23 ms in benzonitrile at 298 K).[224] The mechanical mixtures of tetranitrofluorenone with tetrathiafulvalenes containing cationic polymethine and merocyanine dyes as the photoactive centres were used to gain a photovoltaic effect in the near-IR spectral range.[225]



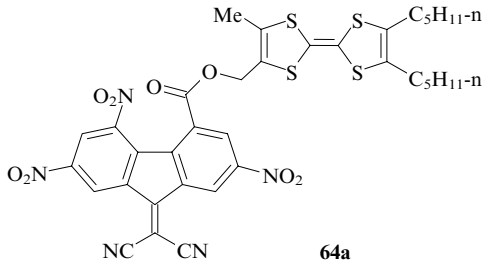
62

X = O, NBn.

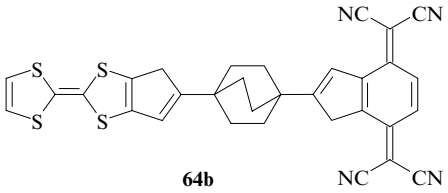


63

Compound **64a** functioned as a molecular rectifier, which is the closest well studied analogue of the Aviram–Ratner model **64b**. Compound **64a** tends to have exceptionally small gap between the highest occupied (HOMO) and lowest unoccupied (LUMO) molecular orbitals (0.3 eV) and the direction of current rectification being dependent on the molecule orientation in the Langmuir–Blodgett monolayer.[221]

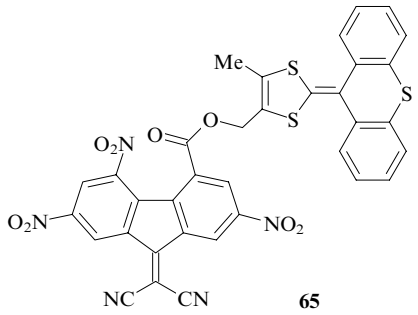


64a



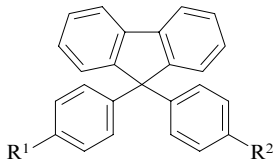
64b

The optical properties, electrochemical behaviour, spectroelectrochemistry of all donor–acceptor diads were studied; X-ray diffraction analysis and quantum chemical calculations were performed. The attention was focused on the redox states. In the case of compound **65** with the thioxanthene donor moiety, owing to the aromaticity, the dication was stabilized to a greater extent than the radical cation localized in the thioxanthene moiety.[223]

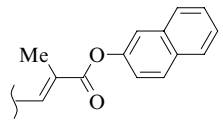


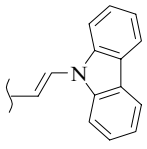
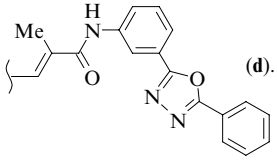
65

A sky-blue phosphorescent OLED with a quantum yield of ~15% was created based on silane derivative **66a**.[226] 9,9-Disubstituted fluorenes **66b**–**d** have fluorescence quantum yields of 19%–83% relative to the quinine sulfate as the standard; hence, they are potential luminophores.[227, 228]

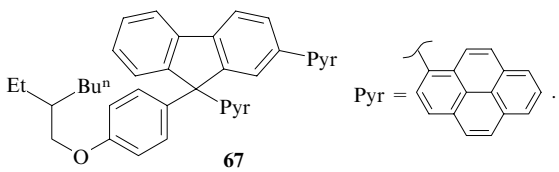


66a–d

R^1 = $SiPh_3$, R^2 = H (**a**); R^1 = R^2 =  (**b**),

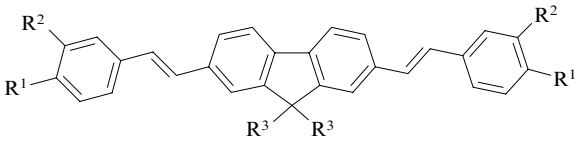
 (**c**),  (**d**).

Fluorene **67** with pyrenyl substituents behaved as simultaneously a hole injection and light-emitting layer for electroluminescence devices. A blue light-emitting diode based on this compound showed brightness of 11 620 cd m^{-2} (Refs 52 and 53).



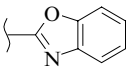
67

Numerous donor–acceptor systems attached to fluorene positions 2 and 7 have also been developed. Compounds of type **68a–c** proved to be fairly sensitive to change in the pH owing to the presence of hydroxy and amino groups, which are capable of being protonated and deprotonated. Dyes **68a**,**b** responded to an increase (upon the addition of a sodium hydroxide solution) and to a decrease (upon the addition of trifluoroacetic acid) in the pH, respectively, by changing the colour of fluorescence from blue to bright green and from cyan to blue.[229] Apart from the sensitivity to the increase in the pH (bathochromic shifts in the absorption and fluorescence spectra and increase in the fluorescence quantum yield from 9% to 32%), compound **68c** shows high sensitivity to zinc ions in solutions under physiological conditions. Zinc plays an important role in living organisms (in particular, it stimulates the insulin synthesis); therefore, the design of such sensors arouses interest in researchers.[230] The formation of chelate-type complexes with zinc ions induces colour deepening and increase in the fluorescence quantum yield from 9% to 46%.[231] Dyes **68a–c** show high sensitivity to the Zn^{2+} ions upon the two-photon absorption.[232]



68a–d

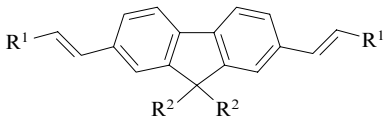
R^2 = H, R^3 = n-C_9H_{19}: R^1 = OH (**a**), NH_2 (**b**);

R^1 = OH, R^2 = , R^3 = n-C_6H_{13} (**c**);

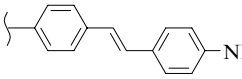
R^1 = , R^2 = H, R^3 = n-$C_{10}H_{21}$ (**d**).

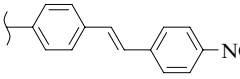
Recently, fluorene-based donor – acceptor systems have been more and more often studied for two- and three-photon absorption.[233–239] The studies deal with the effects of the conjugation chain length and the nature of the donor and acceptor moieties on the two-photon absorption cross-section, the efficiency of the fluorescence caused by this absorption[235] and the photoexcitation mechanisms in centrosymmetrical and unsymmetrical systems.[236] For the fluorene derivatives **69a**,**b** with terminal donor and acceptor moieties (push-push and pull-pull fluorophores, respectively), characteristics of the absorption, fluorescence and two-photon absorption spectra were determined. The push-push systems were found to have greater two-photon absorption cross-sections than the pull-pull systems and the elongation of the conjugated system or replacement of the phenylene group by the thienyl group does not always improve characteristics of the two-photon absorption.[237,238] For compound **69a** in toluene, a considerable two-photon absorption cross-section (584 GM†) was detected.[240] Chinese researchers[241] studied the absorption in the excited state initiated by two-photon absorption for dye **69b**.

† 1 GM = 10^{-50} cm^4 s $photon^{-1}$ $molecule^{-1}$; the unit is called after M Goeppert-Mayer.

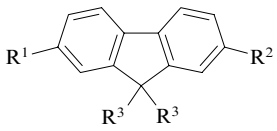


69a,**b**

R^1 = , R^2 = Bu^n (**a**);

R^1 = , R^2 = n-C_8H_{17} (**b**).

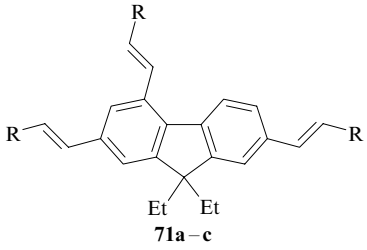
Much attention to two- and three-photon absorption of fluorene derivatives was paid by Belfield's research group.[242–254] In particular, these compounds (for example, fluorenes **70**)[255] have been studied as singlet oxygen photosensitizers.[256,257] Compounds that generate singlet oxygen[258] are of interest for the photodynamic therapy of tumours.[259–261]



70

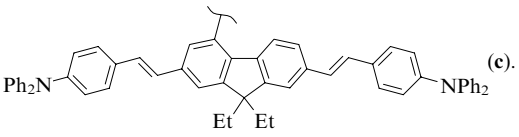
R^1 = NPh_2, R^2 is π-acceptor, R^3 = Alk; R^1 = NO_2, R^2 = I:
R^3 = $(CH_2)_2CO_2H$, $(CH_2CH_2O)_2Me$; R^1 = NO_2,
R^2 is 1,3-benzothiazol-2-yl: R^3 = $(CH_2)_2CO_2H$, $(CH_2CH_2O)_2Me$.

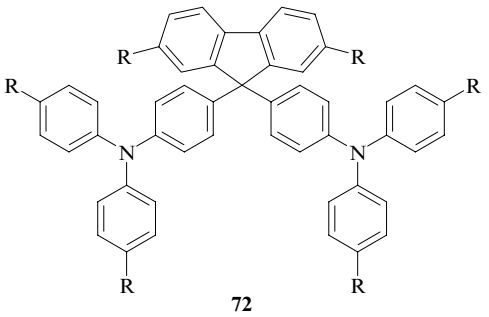
Very large two-photon absorption cross-sections were found for some fluorene derivatives. For example upon pumping at 570 nm, this value is 3115 GM for dye **71a**, 5765 GM for **71b**, 4194 GM for branched compound **71c**,[245] and for compound **68d** this value reaches 5300 GM in hexane.[251]



71a–c

R = $C_6H_4NPh_2$-4 (**a**), $C_6H_4NO_2$-4 (**b**),

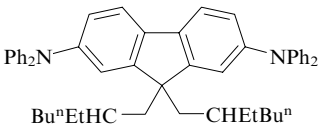
 (**c**).



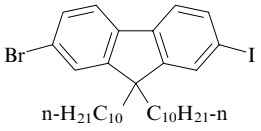
72

R = CH=CHC$_6$H$_4$Bu^t-4.

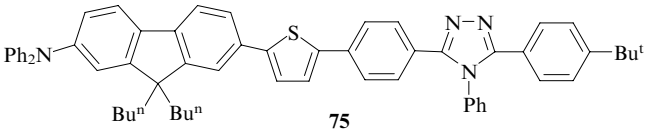
The characteristics of three-photon absorption of fluorene derivatives are not less interesting than one- and two-photon absorption. The effect of the solvent on the three-photon absorption cross-section of dye **68d** was studied.[263] It was found that the lower the solvent polarity, the greater this cross-section. Three-photon absorption was also studied for compound **73**[264] and dihalo-substituted fluorenes (for example, derivative **74**). These dihalofluorenes can be potential photosensitizers for photodynamic therapy.[265] Pumping of oligomer **75** by infrared femtosecond laser induced effective two- and three-photon-excited photoluminescence and laser radiation with a maximum at 482 nm.[266, 267]



73

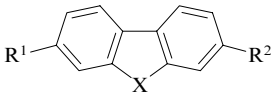


74



75

Compounds **76a**,**b** as parts of liquid-crystal compositions were described in patents [268, 269] devoted to the development of ferroelectric liquid-crystal displays.



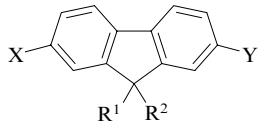
76a,**b**

X = CH$_2$, R^1 = n-C$_5$H$_{11}$, R^2 = OC(O)–C$_6$H$_4$–Pr^n (**a**);

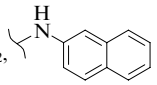
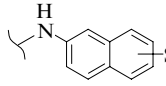
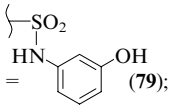
X = C=O, R^1 = NHC$_6$H$_4$Bu^t-4, R^2 = NHC$_6$H$_4$CF$_3$-3 (**b**).

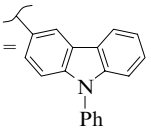
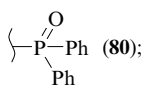
The fairly simple 9,9-dibutyl-7-(dimethylamino)fluorene-2-sulfonic acid molecule (**77**) proved to be very promising for the investigation of proteins and their binding to various drugs. This fluorene derivative was shown to effectively bind to the human serum albumin hydrophobic regions and to change fluorescence intensity in the presence of medical drugs.[270]

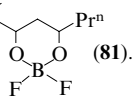
The ^{35}S-labelled fluorenesulfonamide derivatives **78** were used to study the distribution of radioactive dyes in tumours and organs of test mice. It was supposed that the dye will be accumulated in the tumour cells but it was mainly concentrated in the liver, kidneys and blood, while its content in the tumour was much lower. The best ratio was found for fluorene-2,7-bis[*N*-(2-naphthyl)sulfonamide]-^{35}S.[271]



77–**81**

R^1 = R^2 = Bu^n, X = NMe$_2$, Y = SO$_3$H (**77**); R^1 = R^2 = H, X = Y = SO$_2$R^3 (R^3 = NH$_2$,  , ) (**78**); R^1 + R^2 = O, X = Y =  (**79**);

R^1 = Ph, R^2 =  , X = Y =  (**80**);

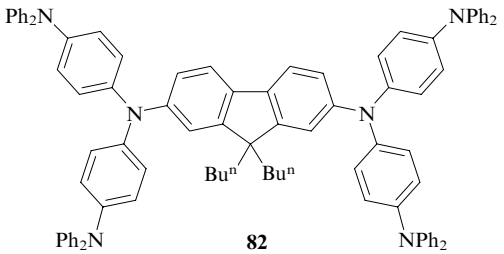
R^1 = H, R^2 = CHMePr^n, X = Y =  (**81**).

A patent [272] describes sulfonamide derivatives of fluorenone, in particular, compound **79**. Compounds of this type act as P2X3 and P2X2/3 purine receptor antagonists. They can be used as anaesthetic and anti-urinary incontinence drugs.

Fluorene **80** with two phosphine oxide groups was used as the host material for the design of blue electroluminescent devices. To this end, various amounts of the iridium complex that emits light from a triplet level in the blue spectral region were added to it.[273] The material of this type is simultaneously a source of electrons and holes and effectively transports them. In addition, the presence of bulky and sterically hindered molecules of fluorene derivatives precludes the development of fluorescence self-quenching of the iridium luminophore. Test samples of this material demonstrated an efficiency of 14.8% and the brightness of more than 1000 cd m^{-2}. The introduction of phosphine oxide groups had almost no influence on the fluorene ring geometry but had a pronounced effect on the ionization potential and electron affinity.[274]

Compound **81** with dioxaborinane moieties obtained by US scientists [275] demonstrated effective electron transport behaviour. This compound was used to develop an OLED for the near-IR spectral region.

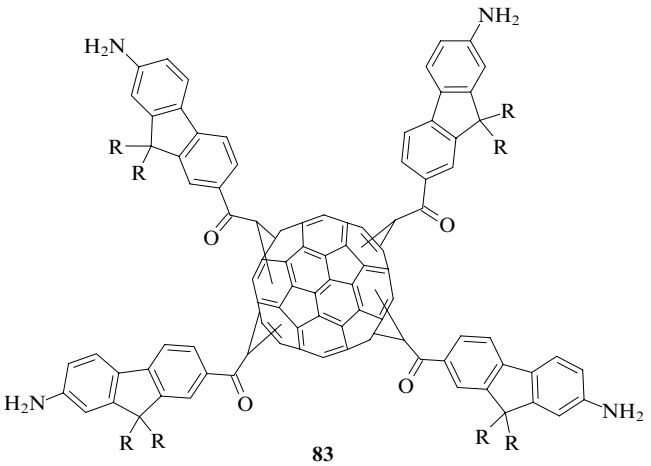
The current efficiency of the OLED based on compound **82** reached 5.2 cd A^{-1} (Ref. 276).



82

Quite a lot of compounds having extended conjugation systems in the molecules based on two or more fluorene nuclei have been prepared. Some molecules have effective

auxochrome groups. The spectral properties, characteristics of two- and three-photon absorption, electroluminescence and photovoltaic characteristics of these compounds were investigated. Thus new nonlinear optical materials were developed based on the fullerene core, which is a strong electron acceptor, with one, two or four fluorene substituents. For tetrasubstituted star-like adduct **83**, the two-photon absorption cross-section reaches 6820 GM.[277]



83

R is 3,5,5-trimethylhexyl.

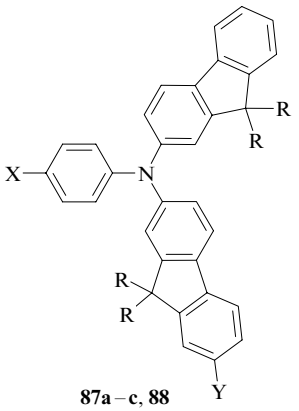
The electron transition energies for a number of aromatic fluorophores were found by semiempirical ZINDO/S quantum chemical calculations with preliminary AM1 geometry optimization. The absorption maxima of 15 compounds and the fluorescence maxima of 34 compounds were predicted rather accurately.[278]

An OLED sample with a maximum brightness of 4600 cd m^{-2} and quantum efficiency of 3.3% was developed on the basis of dye **84** with anthracene and fluorene moieties.[63] Red-coloured OLED were designed using iridium complexes with fluorene-containing ligands, *e.g.*, compound **85**.[279]

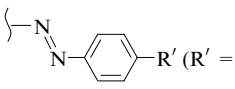
Dendrimer **86** displayed itself as a chemical fluorescent sensor for 1,4-dinitrobenzene vapour; the latter is a model compound used instead of explosive 2,4,6-trinitrotoluene. The detection is due to the formation of π-complexes between the electron-donating dendrimer and the electron-withdrawing 1,4-dinitrobenzene molecule, which is accompanied by fluorescence quenching.[67]

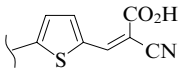
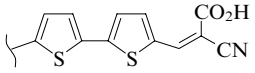
In some cases, the fluorene moiety is not located in the main chromophore of the dye but is a substituent functioning as an electron density donor, as in compounds **87a**–**c**.[280,281] Dyes **87b**,**c** exhibited high efficiency of the light-to-electric energy conversion. When they are used as sensitizers of TiO_2 films in solar cells, the efficiency of conversion is 8.01%.[281]

New organoboron compound **88** is fairly sensitive to fluoride ions owing to the strong interaction of boron with these ions.[282] As tetrabutylammonium fluoride is added to a solution of derivative **88**, the increase in the salt concentration entails a decrease in the charge transfer band and growth of the short-wavelength band. Dyes with such

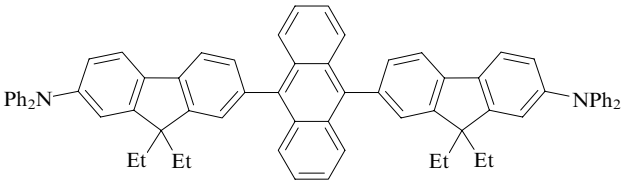


87a–**c**, **88**

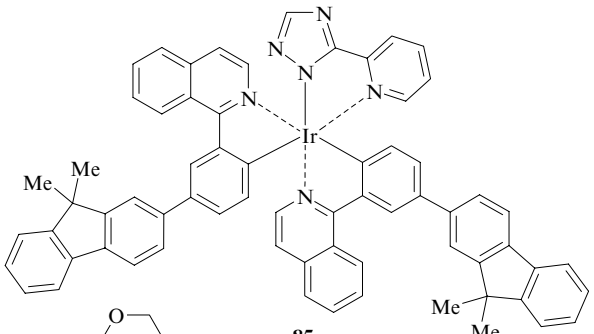
R = Me, Y = H: X =  (**87a**),

 (**87b**),  (**87c**);

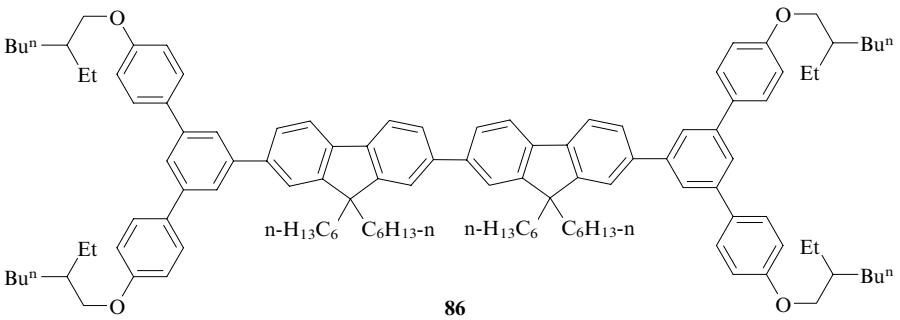
R = Et, X = H, Y = $BMes_2$ (**88**).



84



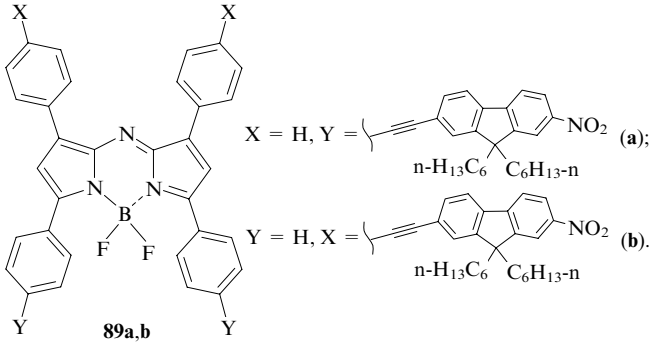
85



86

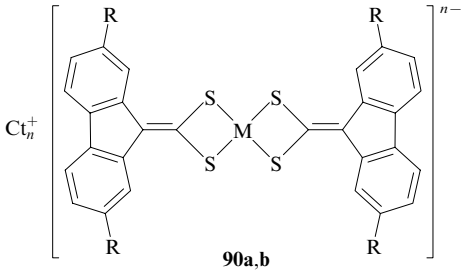
changes in the absorption spectrum may be good chemosensors for fluoride ions.[282]

So-called Bodipy (from the words boron-dipyrromethene) dyes **89a**,**b** were reported; modifying the substituents in these dyes by nitrofluorene residues shifted the fluorescence maximum to the near-IR region of the spectrum.[283]



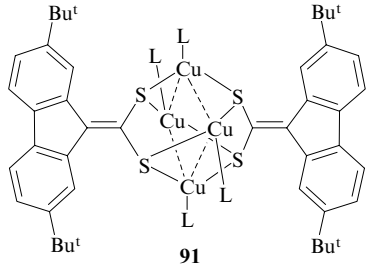
89a,**b**

Gold complexes **90a** with fluorenylidenemethanedithiolate ligands were synthesized and studied.[284] Their absorption spectra exhibit a well-defined nπ transition band and a low-energy charge transfer band from the dithiolate to the gold(III) atom. The same group of researchers prepared platinum complexes **90b** of a similar structure,[285] demonstrating charge transfer in the absorption spectra and fluorescence both in the solid states and in solutions at room temperature.



90a,**b**

M = Au, n = 1 (**a**); M = Pt, n = 2 (**b**);
$Ct^+ = (Ph_3P)_2N^+$, $Bu^n_4N^+$, $Pr^n_4N^+$; R = H, Bu^t, OC_8H_{17}-n.



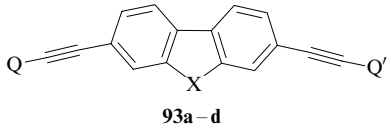
91

L = PPh_3, $P(C_6H_4OMe\text{-}4)_3$, PPr^i_3, *etc.*

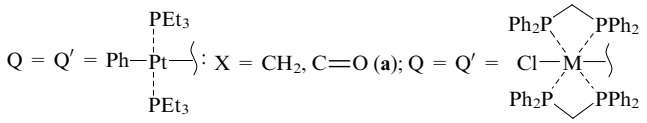
The next stage of the studies of complexation of fluorenedithiolate ligands concerned copper(I) and copper(II) complexes.[286] Complexes **91** can serve as models for the study of structures and functions of the active sites of copper–sulfur proteins (including enzymes), which serve for molecular oxygen transport in redox processes, electron transfer and the transport and storage of copper ions. These compounds can find industrial use as antioxidants for lubricating oils.

The fluorene moiety is often used as a π-conjugated linker or the base for new conjugated systems.[287] An example is provided by structure **92** in which effective energy transfer between 'red' (on the left) and 'blue' (on the right) Bodipy dyes occurs.[288]

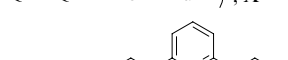
Platinum, ruthenium and osmium acetylenide complexes with phosphine ligands **93a**,**b** based on a fluorene or fluorene type central moiety were described.[289] Gold(I) and mercury(II) bis(alkynyl) complexes (*e.g.*, complex **93c**) were also synthesized.[290] By modifying the substituent in position 9 of the fluorene ring, it is possible to finely tune the optical properties of these complexes.



93a–d

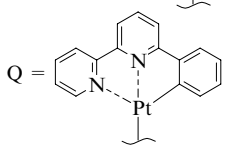


(M = Ru, Os), X = C=O (**b**);



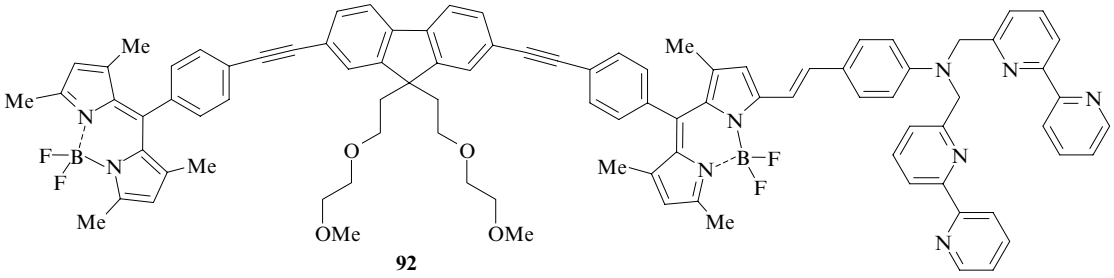
, X = C=O (**c**);

, X = CH_2 (**d**);



, Q′ = H or C≡CH, X = CH_2 (**d′**).

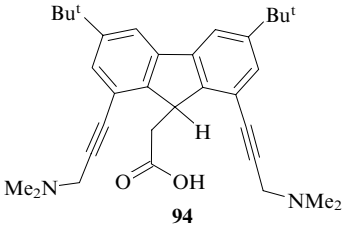
Quantum chemical calculations of mono- (**93d′**) and dinuclear (**93d**) platinum fluorene–acetylenide complexes were carried out by TDDFT method at B3LYP level of theory using the ECP basis set with additional f-orbital polarization function (f = 0.18) for the Pt atom and the 6-31G(d) basis set for other atoms.[291] A study of the charge transfer mechanism and more detailed description of linear and non-linear optical properties provided the conclusion that these complexes may become a new type of nonlinear optical materials.



92

Gold complex **93c** showed much higher antitumour activity than the parent metal-containing antitumour drug cisplatin.[292] The authors suggest that due to the presence of a keto group, complex **93c** can stimulate the generation of active oxygen species, which destroy the tumour.[292] They also consider it possible that the cytotoxicity of complex **93c** towards human carcinoma cells is caused by the tendency of gold atoms to be coordinated to DNA.

In the development of enzyme mimetics containing two iron atoms, it was necessary to obtain a compound mimicking the enzyme active site, *i.e.*, that would be able to interact simultaneously with two metal atoms. The synthesized dye **94** comprising two N-donor moieties and a carboxy group between them is a potential ligand for two metal atoms simultaneously.[293]



94

The same publication reported an interesting approach to the synthesis of 1,8-disubstituted fluorenes, which are exceptionally difficult to prepare under usual conditions due to deactivation of positions 1, 8 and 3, 6 of the fluorene moiety toward to the electrophilic attack.

8. Polymeric dyes

In 2000, Alan J Heeger (USA), Alan G MacDiarmid (USA) and Hideki Shirakawa (Japan) won the Nobel Prize for the discovery and development of conducting polymers.[294–296] The essence of the discovery was in the fact that doping of π-conjugated polymers, in particular, polyacetylene, with low-molecular-mass admixtures gives rise to charge carriers, *viz.*, holes and free electrons, which results in sharp increase in their electrical conductivity, which becomes commensurable with the conductivity of metals such as copper and silver.[294–296]

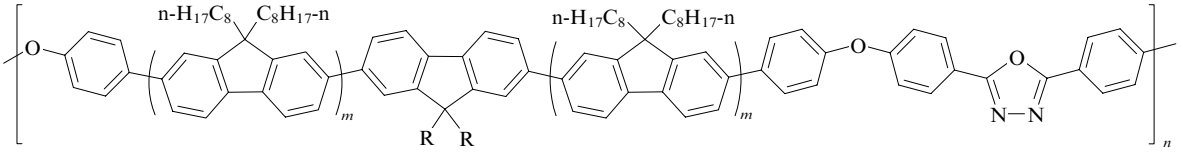
Charge carriers may arise in conjugated polymers also without doping upon the reaction with electromagnetic waves. For example, the oscillating electric-field vector of light causes alternating electron density shift from one end of the chromophore to the other end and back.[295] In other words, instantaneous dipole (exciton) arises, which is positively charged in the electron density depletion area and negatively charged in electron density accumulation area. If this polymer is placed between electrodes to which a voltage is applied, exciton will split into opposite charges, which would migrate from one electrode to the other, *i.e.*, photogenerated current will arise (photovoltaic effect). This effect underlies operation of a solar cell converting light energy to electric energy.[297–299] Since in conjugated polymers the charge generation and transport are induced by light, they are called photoconductive.

If a photoconductive polymer is placed between two electrodes and electric current is passed through it instead of illumination, charge injection from the electrodes will take place, as opposed to the photovoltaic effect, and this will finally also give rise to holes and electrons. They will move to oppositely charged electrodes, *i.e.*, towards one another. As the holes and the electrons meet, they recombine, giving rise to light quantum emission.[300, 301] This electroluminescence phenomenon is widely used in organic light emitting diodes,[65, 302–307] which are more economic light source than filament lamps.

All photoconductive polymers have a chromophore system. In most of them, it is quite extended; hence, they are coloured even in the absence of typical auxochrome groups. In terms of this feature, photoconductive polymers can be assigned to organic polymer dyes (OPD). These substances are investigated for many subjects, in particular, for the effect of the preparation method on their properties.[308–311] Considerable attention is paid to investigation of deactivation pathways of the electronically excited states of OPD, the mechanisms of transfer of the absorbed light energy, photogeneration and transport of charge carriers and intermolecular interactions.[312–319]

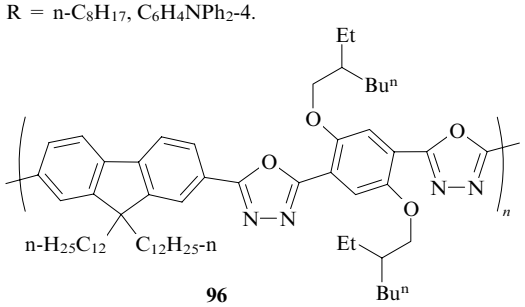
The organic polymer dyes are widely used as active components of the OLED, solar cells, sensors, probes, optoelectron and nonlinear optical devices. The polymeric dyes used in the OLED ensure three key colours of the radiation: blue, green and red, which are combined to produce all intermediate colours.

The most numerous group of fluorene-based OPD are substances emitting blue light, for example, derivatives **95**–**98**.[320–328] For compound **95**, the glass transition tem-

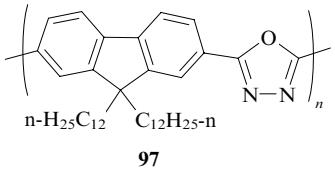


95

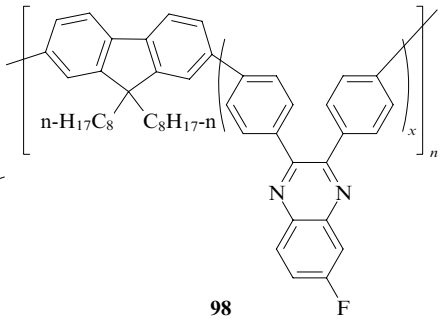
R = n-C$_8$H$_{17}$, C$_6$H$_4$NPh$_2$-4.



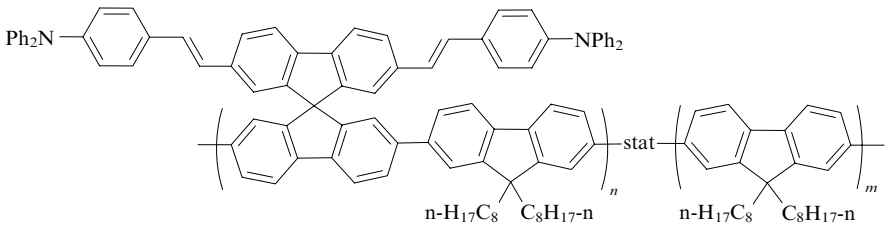
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97



98



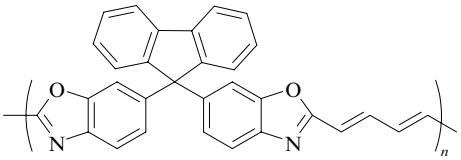
99

perature is 156 °C, which is much higher than that of well-studied homopolyfluorenes, and the photoluminescence quantum yield of this OPD is 91%.[320] Compounds with oxadiazole moieties, for example OPD **96** and **97**, are most promising among blue emitters due to high quantum yields,[321] ease of preparation and thermal stability.[328]

Polymeric dye **98** with quinoxaline moieties emits blue light very close to the NTSC (National Television System Committee) standard for blue. When a cathode buffer was used as a hole emission layer, OLED based on OPD **98** demonstrated a brightness of 2776 cd m^{-2} (Ref. 323).

Polymeric dye **99**,‡ which combines donor and acceptor moieties emits pure blue light due to the cascade energy transfer from the former to the latter.[329]

Polymers in which the fluorene ring is connected to other moieties *via* position 9 rather than positions 2 and 7, for example dye **100**, emit blue or green light.[330]



100

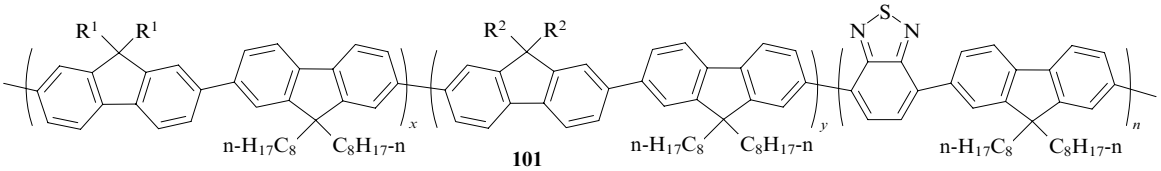
‡ Hereinafter, stat in the polymer formula implies statistic distribution of monomer units in the chain.

The group of green emitters is less numerous. For example, OLED with a very bright green emission can be produced by using OPD **101** with benzothiadiazole moieties in the backbone and oxadiazole substituents.[331]

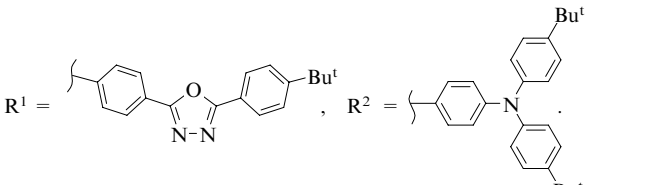
In addition, green light is emitted by OPD **102** with benzothiadiazole[332] and OPD **103** with adamantane[333] units in the backbone and OPD **104** with benzimidazobenzoisoquinolone moieties at the end of the chain.[334] Quite promising are polymers with fluorescent coumarin moieties:[335] thus OPD **105** in the film demonstrates orange photoluminescence at λ_{max} = 585 nm and electroluminescence at λ_{max} = 597 nm.

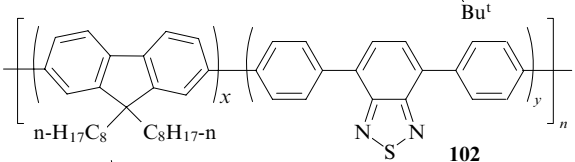
For attaining deeper (having greater wavelength) colour of absorption and luminescence, the polymer chains are modified. There are several approaches such as altering the binding position of the monomer units; introduction of electron-withdrawing groups into the conjugation chain; construction of polymers from deeply coloured moieties. For example, depending on the binding position of the thiophene rings in OPD **106a**, blue, orange or red radiation can be produced;[336] the introduction of cyano groups (polymer **106b**) into the chromophore gives rise to red luminescence.[337]

Phosphorescent polymeric light emitting diodes based on OPD with iridium complexes in the side chains (*e.g.*, polymer **107**) emit orange-red light.[338, 339]

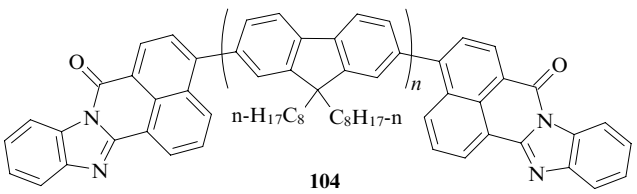


101

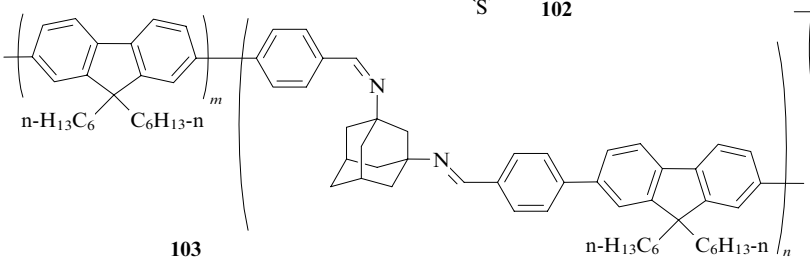




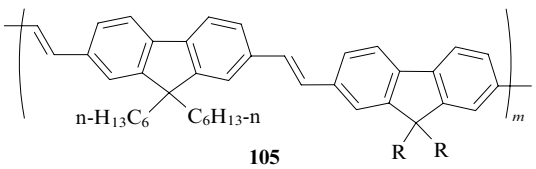
102



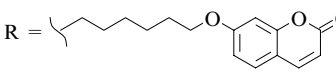
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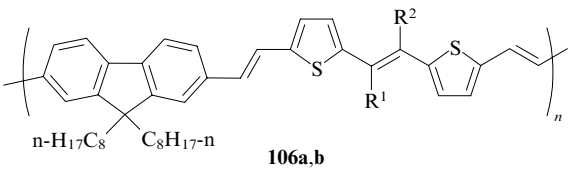


103



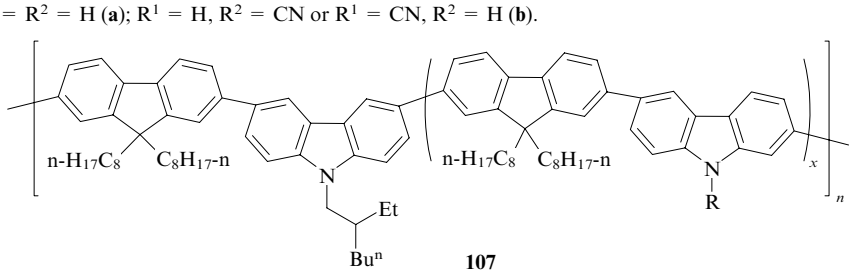
105





106a,**b**

$R^1 = R^2 = H$ (**a**); $R^1 = H$, $R^2 = CN$ or $R^1 = CN$, $R^2 = H$ (**b**).

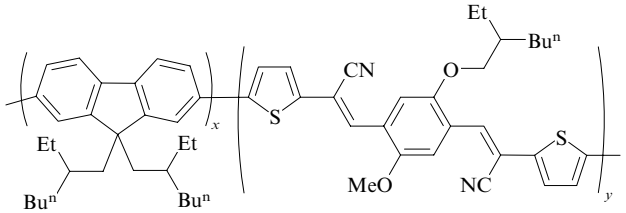


107

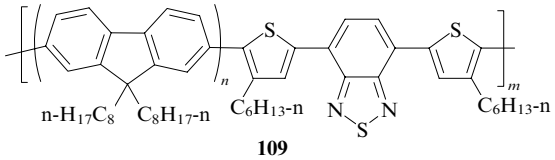
Red emitters **108**–**110** were reported.[340–342] Polymeric dye **108** showed almost complete identity with the NTSC red standard,[340] and OPD **109** was used as a dopant to the green emitter for the design of high brightness (1105 cd m^{-2}) OLED.[342] The efficiency of photoluminescence of OPD **110** reaches 42%, its maximum being shifted bathochromically relative to the electroluminescence maximum.[341]

A white light emitting diode can be manufactured in several ways. The first way is to design a multilayer diode each of layers of which emits a particular colour. Yet another frequently used method is the addition to the polymer of emitters with one of the main emission colours (blue, green, red), depending on the polymer emission colour.[343–348]

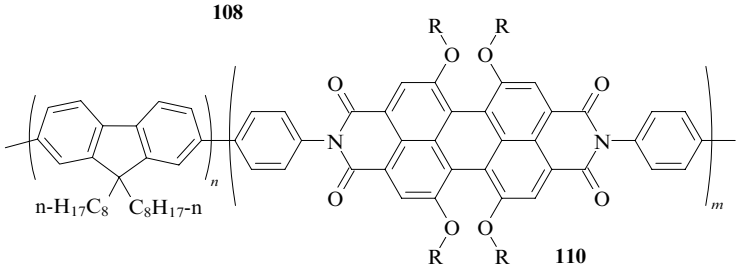
A new trend in the development of white OLED is the production of copolymers from fragments emitting the main



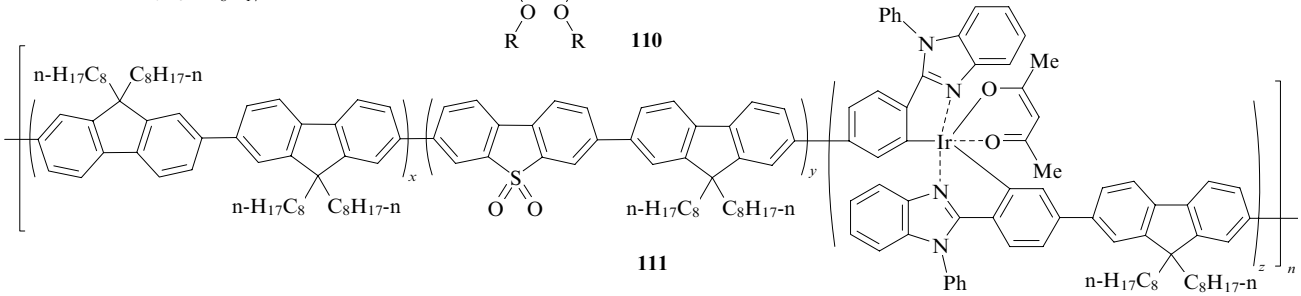
108



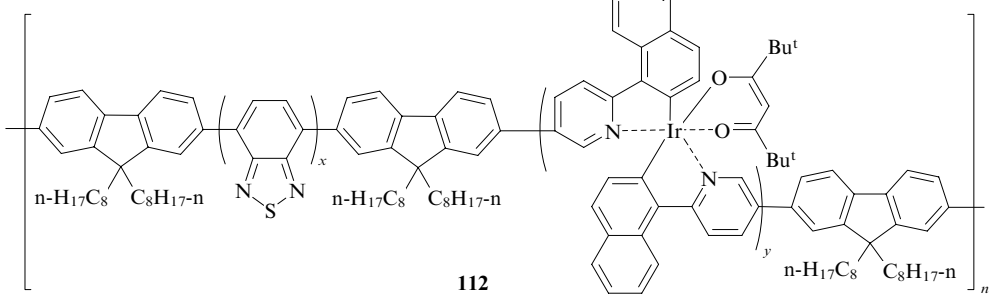
109



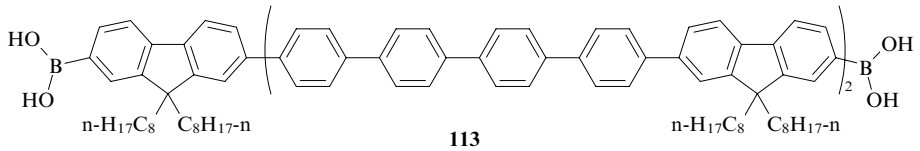
110



111



112



113

colours of the spectrum. The overlap of the emission bands of particular fragments in these polymers (*e,g.*, OPD **111** and **112**) gives rise to white homogeneous single-layer devices.[1,2]

Along with the colour, other important characteristics of the light emitting diodes include emission brightness and efficiency, the voltage applied, durability and so on. Therefore, a large number of publications are devoted to these characteristics of polymers.

Oligomeric dye **113** with alternating *p*-quaterphenyl and fluorene moieties was synthesized to obtain an emitter combining the best characteristics of the mother homopolymers: thermal stability, solubility, high quantum yields and durability. The expectations were justified, and OPD **113** showed a high fluorescence quantum yield (85.6% with quinine sulfate as the standard), almost complete inertness to fluorescence quenching by oxygen and thermal stability.[349]

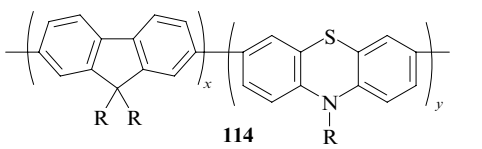
Diodes based on copolymer **114** with phenothiazine moieties showed rather high efficiency and brightness (2.831 cd A^{-1} and 8610 cd m^{-2}, respectively).[350]

The p–n dual-channel copolymer **115** is promising for the design of OLED. The spiro moiety with oxadiazole substituents serves as the electron transport channel and the backbone is a hole transport channel. They are rigidly connected owing to the sp^3-hybridized spiro carbon atom but conjugation is missing.[351] Spirofluorene OPD **116**–**118** can also be used in light-emitting diodes.[352–355]

Organic polymer dyes that combine electron-donating and electron-withdrawing moieties demonstrate good electroluminescent properties. The best way to avoid the fluorescence quenching upon IMCT is to synthesize polymers composed of a p-type backbone and n-type side groups. For polymer **119** ($m = 95$, $n = 5$), the quantum efficiency reaches 1.74%, and the maximum brightness equals 3926 cd m^{-2}, which markedly exceeds similar characteristics of the homopolymer based on 9,9-dioctylfluorene and of fluorene polymers containing only triphenylamine moieties.[356]

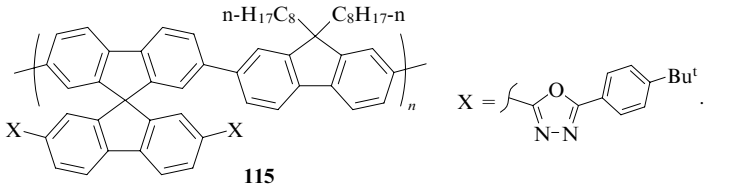
In the absorption spectra of OPD **120** with perylene moieties, three bands can be clearly distinguished, the longest-wavelength band corresponding to charge transfer from the donor to acceptor moiety. The fluorescence maximum of this OPD (red emission) is shifted bathochromically relative to the fluorescence maximum of OPD composed of only perylenediimide units (yellow emission).[357]

The optical properties are affected by not only the type of monomer units but also the configuration of polymer chains; therefore, apart from linear polymers, dendrimer

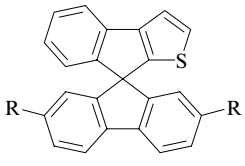


114

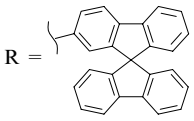
R = $C_6H_4O(C_8H_{17}$-n)-4.

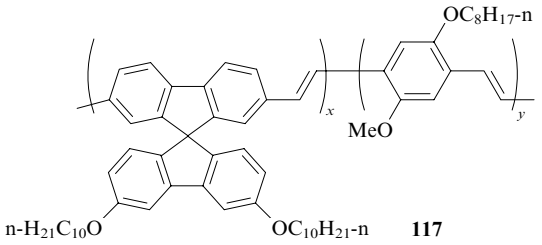


115

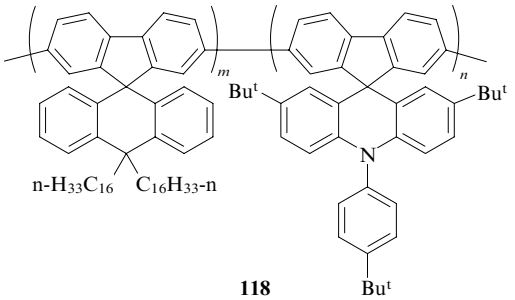


116

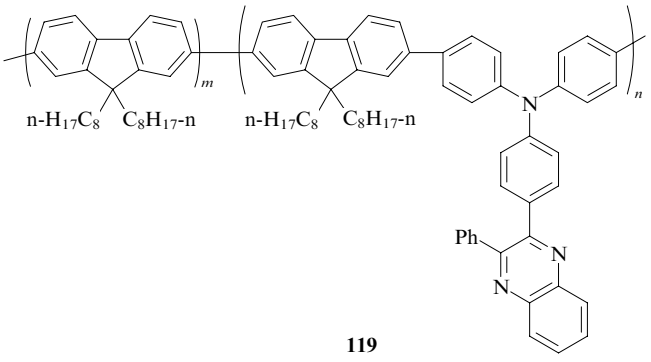




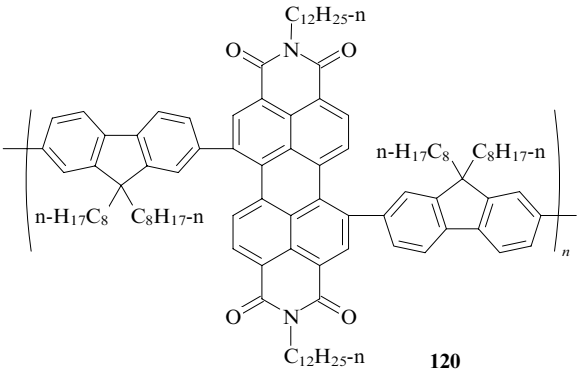
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118

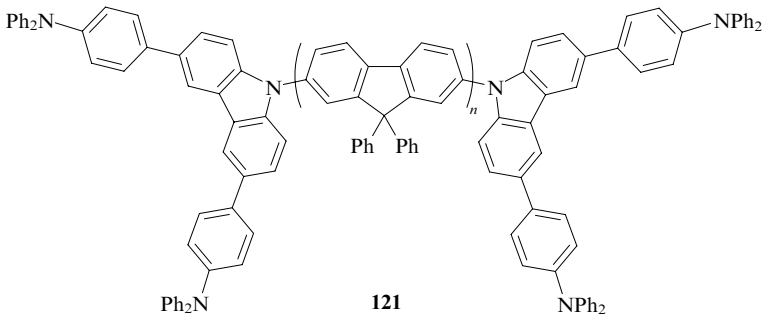


119

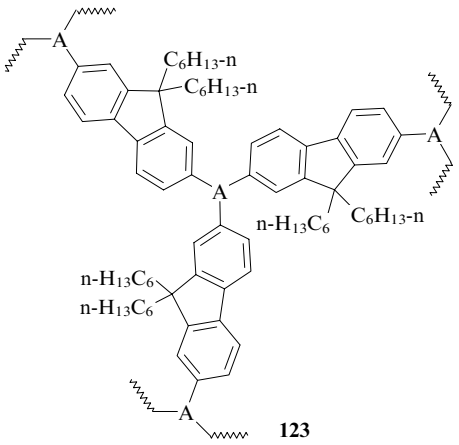


120

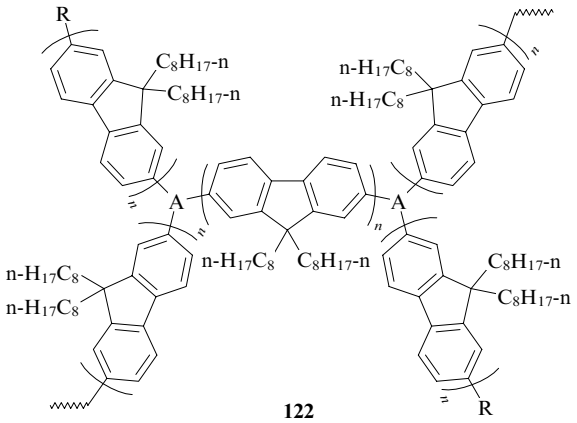
(branched) OPD were also tested for the use in OLED.[358–368] In some cases, branching points are present only at the ends of polymer chains as in the case of OPD **121**.[369] Star-like OPD in which branching starts from the midpoint (*e.g.*, compounds **122**–**126**) are more often encountered. The central unit may be composed of a triarylamine, fluorene, carbazole, triaryl-1,3,5-triazine or truxene (OPD **125**) moiety.



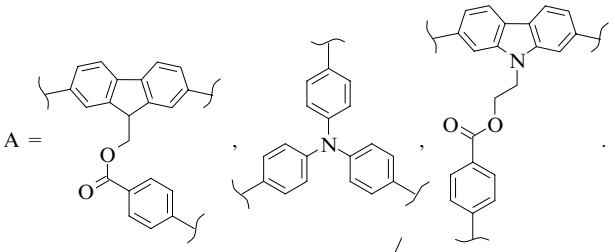
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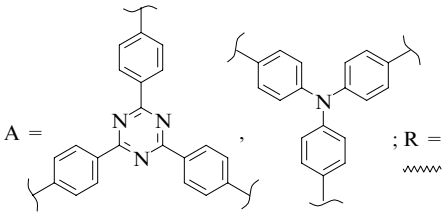


123

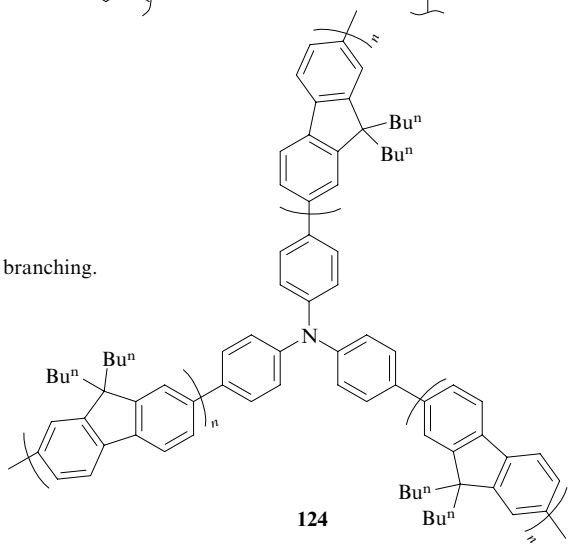


122

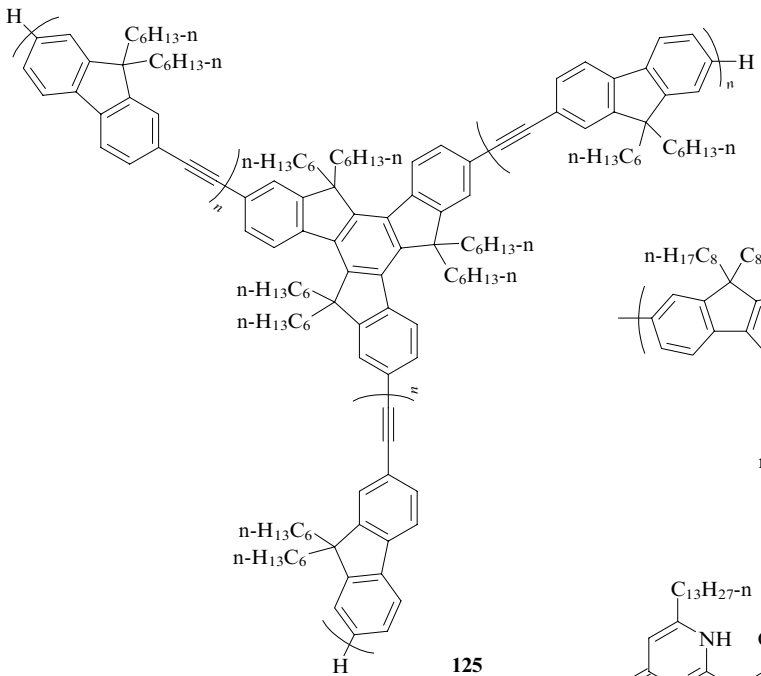




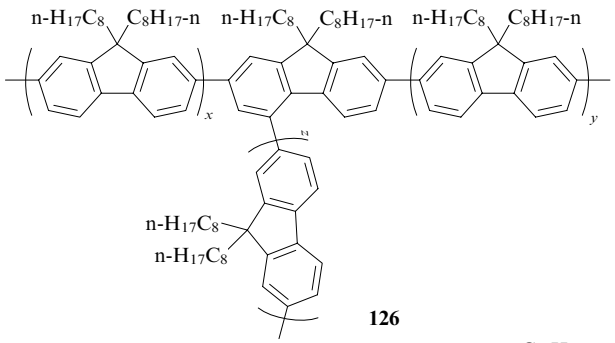
; R = $C_6H_4NPh_2$-4, $C_6H_4SiPh_3$-4;
is continuation of the chain with branching.



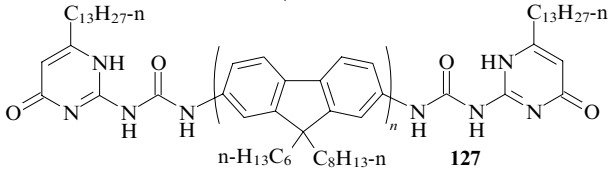
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125



126

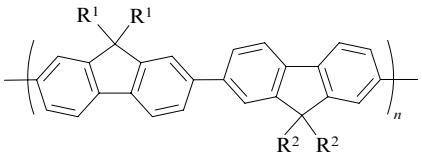


127

Relatively small-size oligomers **127** (n = 1, 3, 5, 7) are promising for light-emitting diodes. They can form supramolecular chain OPD owing to the four hydrogen bonds and they are also effective energy donors and can transfer energy when hydrogen-bonded to the acceptor.[370]

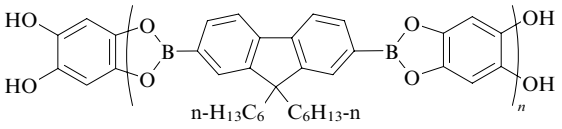
Some OPD are suitable for the use in both optical and electrochemical devices. Test specimens of a light-emitting diode and a galvanic cell based on OPD **128a** were fabricated.[371] These cells are quite promising for the manufacture of inexpensive devices for common use.

A new type of organoelement OPD was mentioned in the literature.[372, 373] The boron atoms in these OPD have a trigonal planar geometry, as was confirmed by ^{11}B NMR spectroscopy. Polymers **129** based on fluorene-2,7-diboronic esters demonstrated stability, the ability to form films and electroluminescence.[373] The semiempirical calculations for OPD **129** showed a degree of electron delocalization of almost 70%; this value is close to the corresponding values for traditional conducting polymers such as polyacetylenes or polythiophenes. These OPD also displayed self-recovery after hydrolytic degradation.[372]



128a,**b**

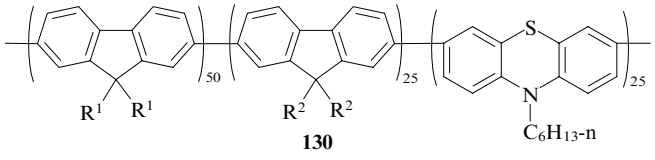
$R^1 = (CH_2)_6N^+Me_3Br^-$, $R^2 = (CH_2CH_2O)_2Me$ (**a**);
$R^1 = (CH_2)_3NMe_2$, $R^2 = C_8H_{17}$-n (**b**).



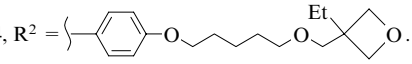
129

The use of OPD **128b** as the electron transport layer in the OLED with aluminium cathode and a poly[2-methoxy-5-(2-ethylhexyloxy)-1,4-phenylenevinylene] (MEH-PPV) light-emitting layer gave rise to a brightness of 3004 cd m^{-2} at a quantum efficiency of 2.29%.[374] The photoluminescence quantum yield for this polymer is 37.3%, being decreased to 4.7% after quaternization of nitrogen atoms in the substituents. The electroluminescence maximum for the quaternized polymer is shifted hypsochromically with respect to the maximum for the neutral polymer. This type of conjugated polyelectrolytes is readily soluble in polar solvents such as methanol, DMF or DMSO.[374]

After ultraviolet irradiation in the presence of the photoacid generator, the polymer chains of OPD **130** are cross-linked, and the polymer becomes insoluble; however, the absorption and fluorescence maxima almost do not shift, while the electroluminescence brightness and efficiency increase almost twofold. These characteristics make OPD **130** a promising candidate for the development of novel wear-resistant materials for the OLED.[375]

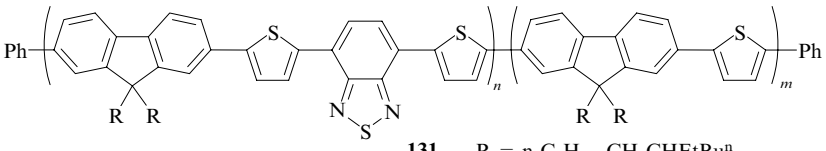


130

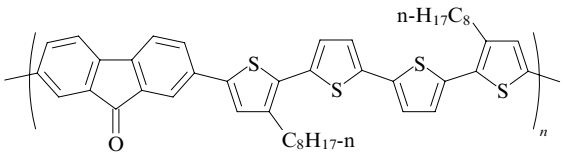
$R^1 = C_6H_4O(C_8H_{17}\text{-n})\text{-4}$, $R^2 =$ 

Organic solar cells are developed using a lot of different polymers, in particular, fluorene derivatives. The APFO Green series of OPD based on fluorene, thiophene and thienodiazine or thiadiazolequinoxaline moieties (*e.g.*, polymer **131**) was described in a review.[376]

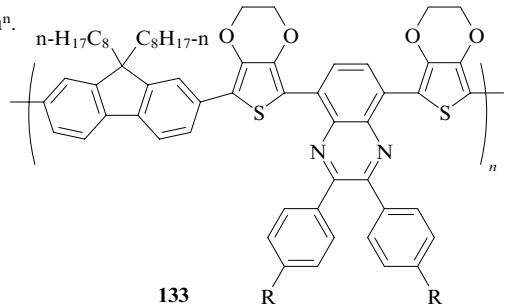
The applicability of OPD for the use in photovoltaic cells depends on a number of characteristics such as the energy conversion efficiency, the fill-factor, the electric current density, *etc*. Therefore, these characteristics are studied as fully as possible for every new specimen.[377–379] The energy conversion efficiency was 1.5% for the fluore-



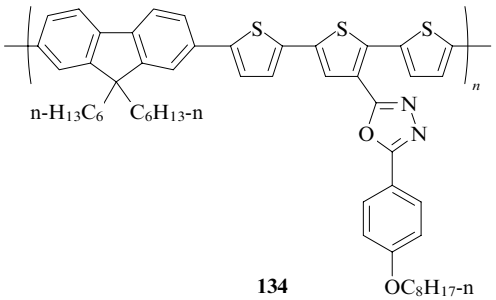
131 R = n-C_6H_{13}, $CH_2CHEtBu^n$.



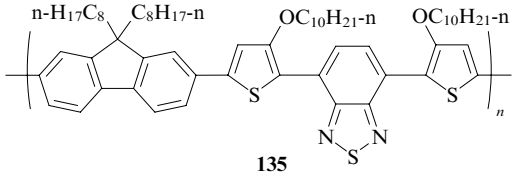
132



133 R = $OCH_2CHEtBu^n$.



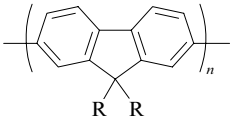
134



135

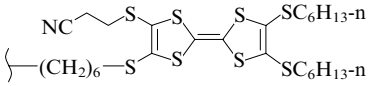
none–thiophene OPD **132** and 1.1% for OPD **133** (APFO-14). The fluorene–thiophene OPD **134** with oxadiazole substituents demonstrated an efficiency of 1.49%.[380–382] In the case of OPD **135**, an energy conversion efficiency of 1.6% and a fill-factor of 48.6% were observed upon white light irradiation of a sample of photovoltaic panel cell with the ratio polymer : PCMB = 1 : 4 [PCMB is (6,6)-$C_{60}CPh(CH_2)_3CO_2Me$, methanofullerene derivative of methyl butyrate].[383]

Some OPD developed for solar cells proved also suitable for light-emitting materials. For the OLED based on OPD **136a**, the breakover voltage was one of the lowest in magnitude among the polymeric light-emitting devices with phosphorescent dopants as emitters.[384] The use of OPD **128a** as a layer between the active layer of a solar cell and a metal cathode increased the energy conversion from 2.95% to 3.77%.[3]



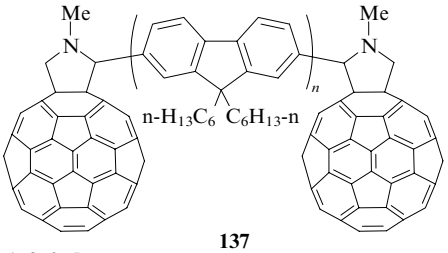
136a,**b**

R = $(CH_2CH_2O)_2Me$ (**a**),



(**b**).

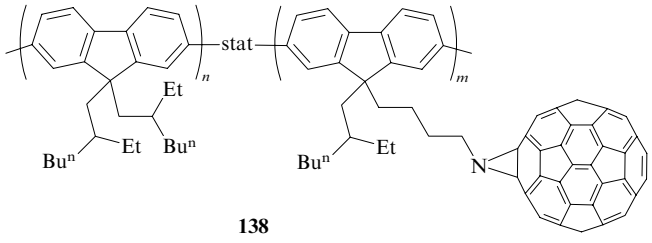
The energy transfer from the fluorene moiety to the electron-withdrawing fullerene moiety was observed for oligomers **137** in the singlet excited state; therefore, these oligomers are of interest for the design of photovoltaic media.[385, 386]



137

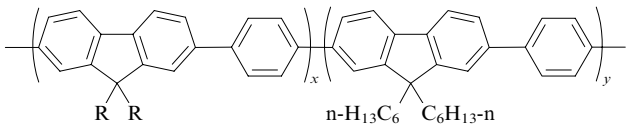
n = 1, 2, 3, 5.

However, OPD **138** in which the fullerene moieties are linked by the alkyl chains to the C(9) atoms of the fluorene units proved unsuitable due to the too low band gap and inefficient electron transport.[387]



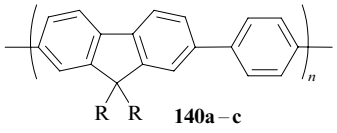
138

As noted above, fluorene-based polymers show bright fluorescence. Hence, fluorescence quenching or enhancement may serve an effective sensor. For example, OPD **139** responds to the presence of Hg^{2+} ions in the solution by fluorescence enhancement.[6] The reason is that the metal ions coordinate nitrogen atoms of the aniline moieties and thus preclude the IMCT, which is the main cause of fluorescence self-quenching in the polymer dye. The considerable decrease in the IMCT in OPD **139** increases the fluorescence quantum yield from 25.3% to 56.4% upon the addition of 25 equiv. of Hg^{2+}. Meanwhile, OPD **140a** responds to the presence of mercury ions by almost complete fluorescence quenching.[388] This OPD demonstrates the quenching of fluorescence by 5% at the Hg^{2+} concentration of 10 nmol litre^{-1}. [The 10 nmol litre^{-1} concentration of Hg^{2+} is the EPA (US Environmental Protection Agency) limit for mercury content in drinking water.]



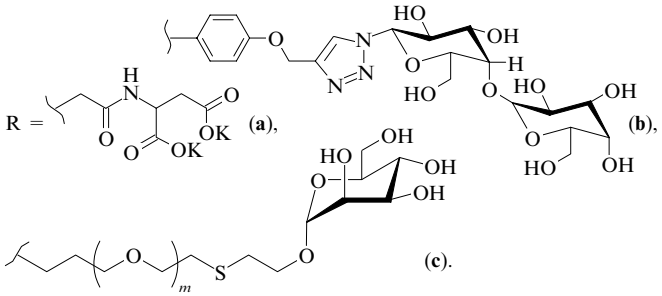
139

R = $C_6H_4NEt_2$-4.

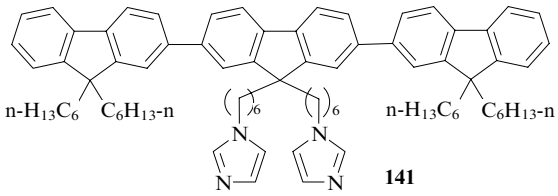


140a–c

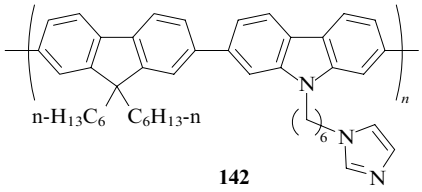
R =



(**a**), (**b**), (**c**).



141

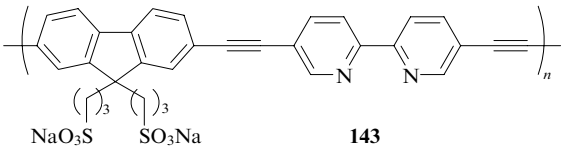


142

Oligomer **141** demonstrated a selective response to the presence of Cu^{2+} ions in solution: its fluorescence was completely quenched under these conditions.[389] The OPD fluorescence quenching by copper ions was used to design a sensor for α-amino acids. If copper(II) ions are added to a solution of OPD **142**, the fluorescence is completely quenched and the addition of an amino acid (*e.g.*, histidine) to this complex restores the fluorescence.[390]

Polymeric dye **143** is an effective sensor for Ni^{2+} ions in aqueous solutions. In the presence of transition metal ions, the absorption spectrum of this OPD shows a bathochromic shift; the stronger chelation, the more pronounced the shift. The fluorescence of OPD **143** is quenched upon the addition of transition metal ions. This process is induced most efficiently by nickel ions: The Stern–Volmer quenching

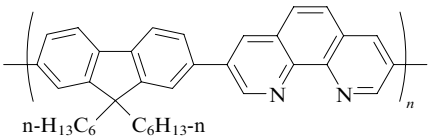
constant in the presence of Ni^{2+} ions is 8.5×10^8 litre mol^{-1}.[391, 392]



143

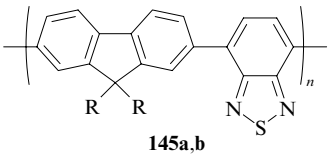
When the solution contains calcium ions, the fluorescence intensity of OPD **140b** decreases, because the polymer backbones aggregate with one another as a result of complex formation with calcium cations.[393]

Polymer **144** with alternating fluorene and phenanthroline moieties can form complexes with lanthanide ions. The resulting complexes demonstrate considerable red shift of the luminescence maximum with respect to non-coordinated polymers.[394]



144

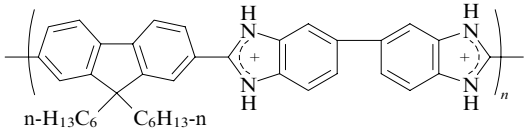
The spectral characteristics of the cationic OPD depend on the nature of the anion. For example, the fluorescence quantum yield of OPD **145a** in water increases from 3% to 22% as the bromide anion is replaced by a tetrakis(3,5-trifluoromethylphenyl)borate.[395] In films, it increased even more, from 5% to 41%.



145a,**b**

R = $(CH_2)_6N^+Me_3X^-$ (X = Br, BF_4, OSO_2CF_3, PF_6, BPh_4, $B[C_6H_3(CF_3)_2\text{-}3,5]_4$ (**a**); R = $(CH_2CH_2O)_2(CH_2)_2N^+Me_3Br^-$ (**b**).

In a formic acid solution, OPD **146** serves as a sensor of iodide ions: upon increase in their concentration, the fluorescence intensity of the polymer decreases. The process follows an electrostatic mechanism in which the acid protonates the benzimidazole polymer moieties thus converting the polymer to a peculiar polycationic molecular wire, which attracts iodide ions. This causes fluorescence quenching.[396]

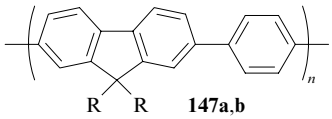


146

Fluorene-based polymeric dye **136b** with tetrathiafulvalene substituents is promising as a conducting material (conductor) or a fluorescent switch. It has a slight fluorescence in the neutral state but it is considerably enhanced after chemical oxidation.[397]

Cationic OPD **145b** of a rather simple structure demonstrated the ability to detect heparin in solutions upon effective complexation with it. Note the selectivity of this OPD: it almost does not react with hyaluronic acid, which is a close analogue of heparin. As the heparin concentration increases, the polymer fluorescence increases; however, an increase in the water content in the solution decreases the fluorescence intensity.[398]

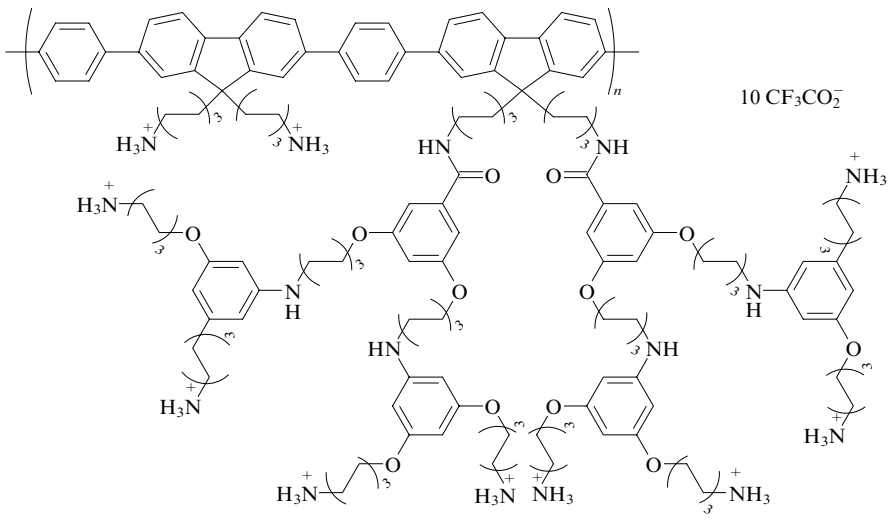
The cationic water-soluble OPD **147a** is suitable for detecting single-stranded DNA in solutions upon their modification with fluorescein: the Förster energy transfer from the polymer to the dye causes intense luminescence.[399] Other OPD were also investigated as sensors for DNA,[400–403] for example, water-soluble dendronized polymers **148**; the more branched the polymer, the more efficient the operation.[404]



147a,**b**

R = $(CH_2)_6N^+Me_3X^-$ (X = I (**a**), Br (**b**)).

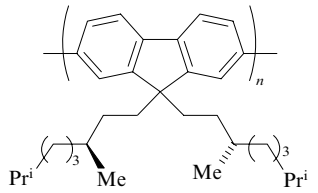
Fluorescent OPD **147b** was used for the optical determination of the protein streptavidin.[405] When this specific



148

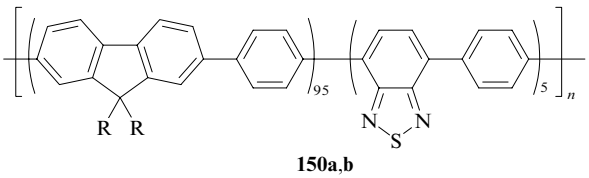
protein is present in the solution, no OPD fluorescence quenching by the biotinylated fluorescein probe is observed, as the latter is bound by the protein. In the absence of streptavidin, FRET results in fluorescence quenching.

The optical properties of chiral OPD **149** were studied.[406] In particular, pronounced photoinduced circular dichroism was detected.



149

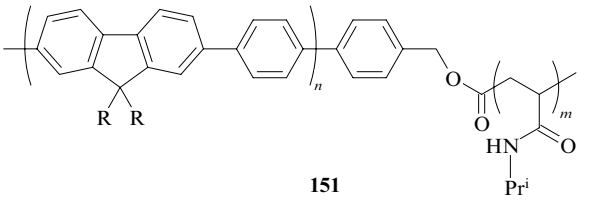
Fluorene-based polymeric dyes **150a**,**b** were used as effective probes for monitoring the action of two enzymes — alkaline phosphatase and trypsin.[407] The polymer interaction with oppositely charged substrates causes a bathochromic shift of the fluorescence maximum: its colour changes from blue to green. When the substrate is cleaved by the enzyme, the electrostatic interaction becomes weaker and blue colour of the fluorescence is restored.



150a,**b**

R = $(CH_2)_6N^+Me_3Br^-$ (**a**); $C_6H_4O(CH_2)_4SO_3Na$-4 (**b**).

An important aspect of using OPD as sensors for biological objects is the solubility of these polymers in aqueous media. Methods for ther synthesis of water-soluble OPD are being developed.[408] A new water-soluble diblock copolymer **151** can be a promising bio- or chemosensor. Also, it has a temperature-dependent susceptibility for aggregation.[409]



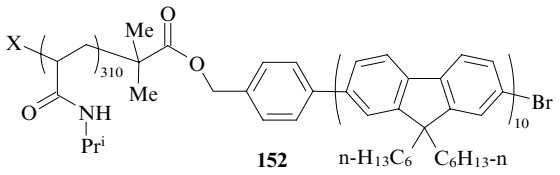
151

R = $(CH_2)_3N^+Me_2EtBr^-$.

Polymeric dyes modified by sugar residues can serve as probes for the presence of lectins in solutions. The reaction is accompanied by fluorescence quenching.[410]

Water-soluble OPD **140c** proved to be suitable for detecting *Escherichia coli*.[411] The polymeric dye forms fluorescent cage clusters owing to the interaction of the sugar residues with the pili of bacteria. These clusters show bathochromic shift of the absorption and fluorescence maxima.

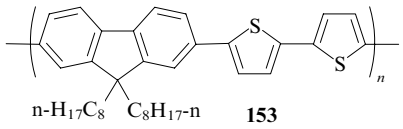
Micelles of the amphiphilic copolymer **152** in aqueous solutions ensure highly effective generation of singlet oxygen,[412] and, hence, this OPD is of interest for photodynamic therapy.



152

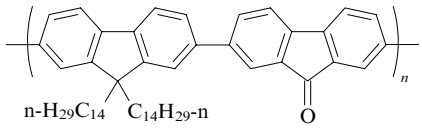
X = Cl, Br.

By addition of fullerene derivatives to OPD **153**, trial models of organic photodetectors were produced.[413]



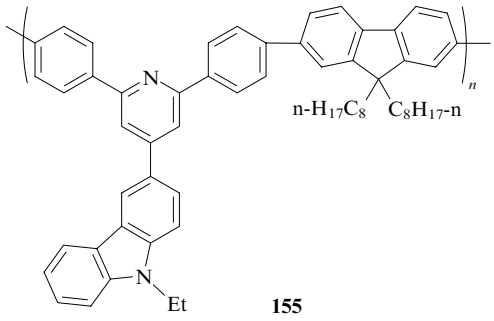
153

The nanoparticles of OPD **154** adsorbed on cellulose fibres were used to obtain paper producing green luminescence.[414]



154

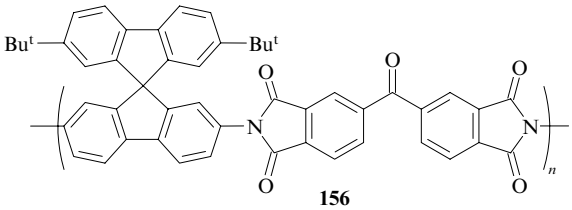
New OPD **155** demonstrated good electrochromic and chemochromic properties. On acid protonation in tetrahydrofuran, the colour of its fluorescence changes from light-blue to yellow. In films, upon increase in the electric voltage applied, OPD **155** changes colour from grey to yellow. The fluorescence quantum yield is 88% in a solution and 6% in a film (relative to 9,10-diphenylanthracene in tetrahydrofuran).[415]



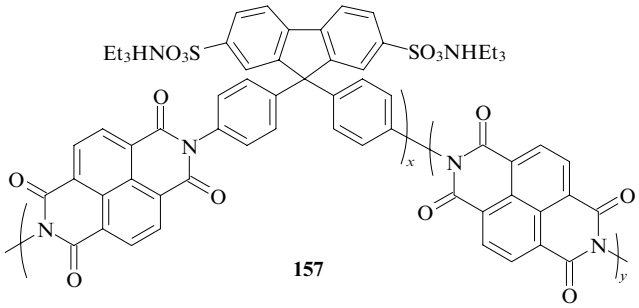
155

The fluorene OPD were used to obtain highly selective membranes with a broad scope of applicability. In particular, they are stable in harsh environment.[416] The polyimide membranes based on polymer **156** with spirofluorene units demonstrated very high degree of separation of gaseous O_2 and N_2 at a very high oxygen permeability.[417] Membranes based on sulfonated fluorene derivatives **157** can be used as electrolytes in fuel cells.[418] Polyacetylenes with fluorene substituents, for example, polymer **158**, are suitable for producing membranes with high gas permeability.[419]

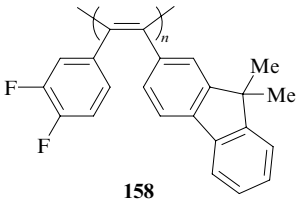
Hybrid fluorene — heteroaromatic OPD **159**–**163** were synthesized and studied.[420–425] Their applications are quite diverse. Thus OPD based on fluorene and thiophene moieties are used as unipolar transistors.[426]



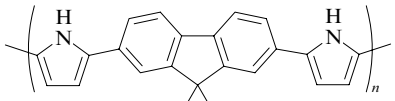
156



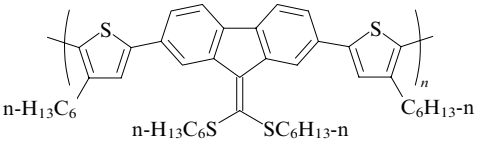
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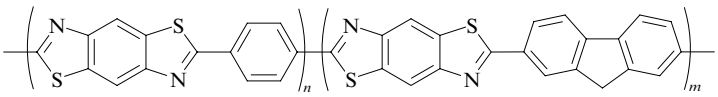
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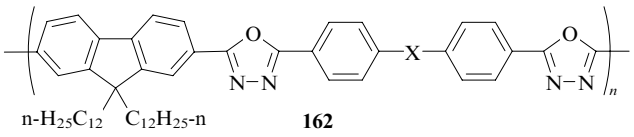
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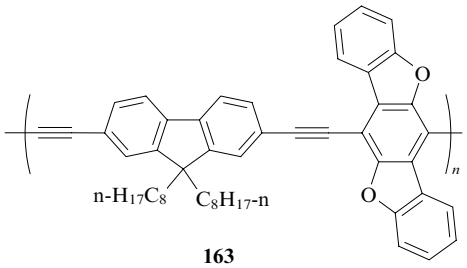
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161



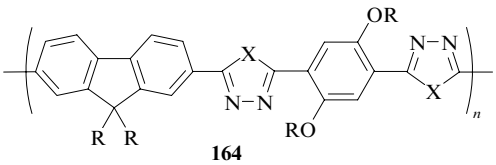
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163

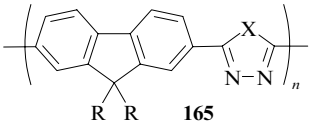
X = $C(CF_3)_2$, O, $O(CH_2)_6O$.

Polymeric dyes **164** and **165** were also efficient as electron transport materials.[427]



164

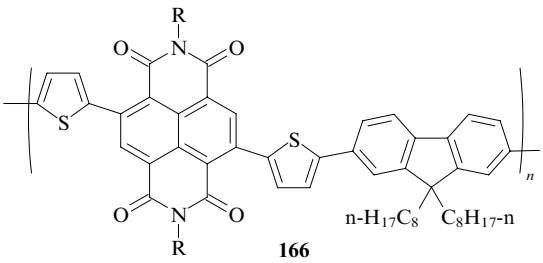
R = $CH_2CHEtBu^n$; X = O, S.



165

R = $CH_2CHEtBu^n$; X = O, S, NPh.

Hybrid OPD **166** based on naphthalenetetracarboxylic acid bisimide, fluorene and thiophene show only weak red luminescence, but they are promising materials for the semiconductor layers of unipolar transistors and photogalvanic cells, as they are thermally stable and have a narrow band gap.[428]



166

R = n-C_6H_{13}, C_6H_4Me-4.

Fluorene OPD are used to fabricate multilayer polymer photodiodes[429] and thin-layer organic transistors.[430] They are used also to produce fibre optics materials.[5]

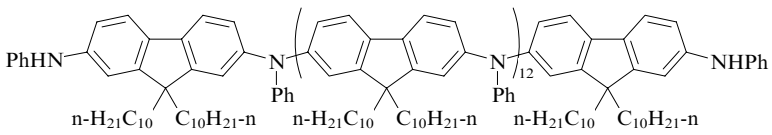
The low-molecular-mass OPD **167** with 14 fluorene units demonstrates a very high two-photon absorption cross-section (17 200 GM),[431] and, hence, it is promising as an optical limiter for light fluxes.[432]

Polymeric dye **168** has not only a great two-photon absorption cross-section (13 260 GM in 1,2-dichloroethane at a 940 nm laser wavelength upon femtosecond excitation) but also considerable solvatochromism and solvatofluorochromism.[433]

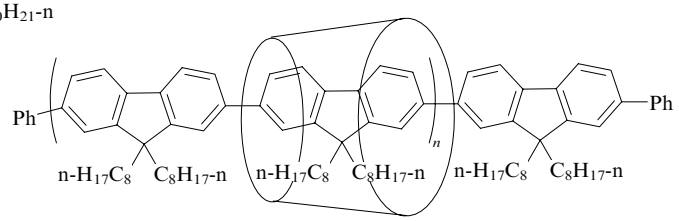
A two-photon solid-state laser was developed based on poly[2,7-(9,9-dioctyl)fluorene].[434]

A study of rotaxane-type OPD **169** comprising β-cyclodextrin moieties (shown by a truncated cone) revealed that the presence of these moieties affects only the physicochemical properties of polymers but does not affect the optical properties.[435] This can be used to adapt OPD **169** to various operation conditions at specified optical characteristics.

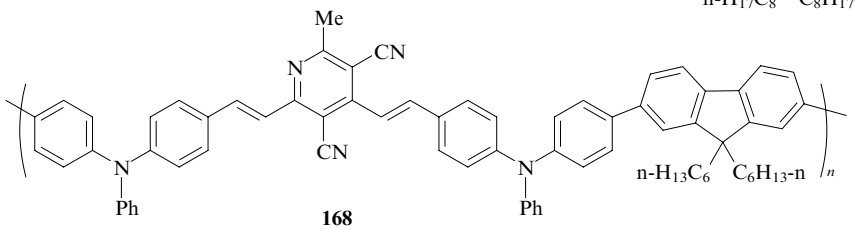
Molecular wires, for example, polymer **170**, with lengths of up to 10 nm were prepared based on the fluorene moiety.[436, 437] They were synthesized by a method differing from traditional approaches that are used to prepare molecular wires. Compound **170** was obtained by the Sonogashira condensation with simultaneous regioselective removal of protecting groups. The spectral properties and crystal structures of these wires were investigated. Systems of this type can self-assemble to nanostructures and bind to metal surfaces and, hence, they could find use in the molecular electronics.[436, 437]



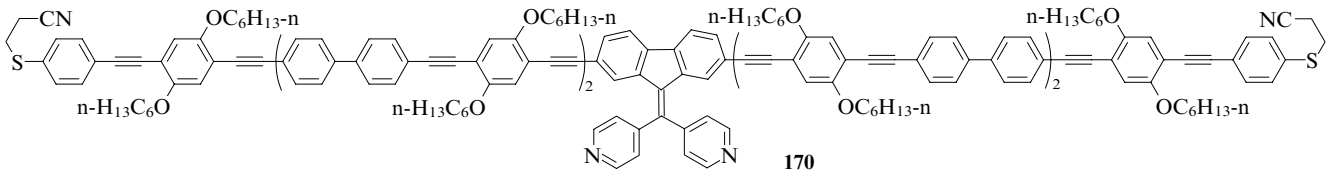
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169

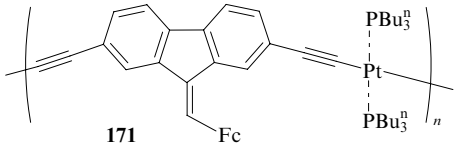


168



170

Merocyanine-type organometallic OPD based on acetylenide platinum complexes **171** represent an alternative to the classical macromolecular chemistry.[438,439] The common chromophore system of these molecules is composed of the conjugated merocyanine and photoconductive polymer. Hence, specific dye and polymer properties can be combined in one molecule by targeted change of its chemical structure for various applications.



171

III. Conclusion

Fluorene and its derivatives are multipurpose synthons for a wide range of functional materials with a unique set of physicochemical properties. Despite the diversity of dyes based on them, further search for new types of dyes is still topical. Of special interest are polymethine dyes, as they offer the greatest diversity of spectral luminescence, electrophysical and magnetic properties among organic dyes.[42,43,440,441] To date, only a few fluorene-based polymethine compounds have been prepared.[79] It is especially important to develop such dyes for the practically significant near-IR region (> 1000 nm). Actually, it was found[79] that fluorene-based polymethines demonstrate uniquely high absorption selectivity and intensity in highly polar solvating media, which is hardly practicable for polymethine dyes based on heterocycles. The reason is that the shift of the absorption band to the above-mentioned region is achieved by elongating the polymethine chain. The chain elongation considerably violates the uniformity of the charge distribution in the dye chromophore.[80] This in turn enhances the nucleophilic and electrophilic solvation of the charged centres of the dye by molecules of the medium and, as a consequence, absorption selectivity and intensity sharply decrease.[80]

The interest in polymethines has now considerably grown due to their enhanced tendency, as compared with other organic dyes, to form self-assembling nanostructures, in particular, so-called J-aggregates.[442–447] The high absorption intensity, weak electron–phonon interactions, short excited state lifetimes, the capacity for effective exciton energy transfer along the aggregate chain, and the possibility of packing of a large number of J-aggregate structures in unit surface area account for their use as light energy converters in various fields of science and technology.[445–450]

No fluorene-based zwitter-ionic squarylium dyes have been described as yet. Squaraines are subjects of enhanced interest owing to their high thermal and photo stability and record-breaking intensity of absorption bands.[451,452] The easy intra- and intermolecular charge transfer[453,454] accounts for their effective use as photothermoplastic holographic media.[42,43,455] The aggregation behaviour of squaraines was used in the development of fluorescent probes for the study of biological objects.[456] Among them, highly efficient singlet oxygen sensitizers were found.[457,458] Also, they can control the radical polymerization of acrylic monomers.[459]

The fluorene zwitter-ions (croconates and higher polyions) are also unknown as yet. The interest in such compounds is first of all due to the exceptionally high two-photon absorption cross-section of polyions.[460–462]

Unlike zwitter-ion dyes, fluorene-based merocyanines are subjects of quite a few publications.[170–174,182,183] However, there are almost no data on merocyanine series with smoothly changing electron asymmetry in which a fluorene derivative serves as one terminal group and some heterocycle serves as the other terminal group. By changing the donor–acceptor properties of these terminal groups, one

could control the polyene – polymethine electron relaxation dynamics of merocyanines[463] in the ground and excited states and thus create compounds needed for effective solution of particular applied problems.[464]

The electronic structure of fluorene OPD, like that of photoconductive polymers of other classes, varies within the neutral polyene – aromatic hydrocarbon range. This is due to the fact that owing to the absence of ionized atoms in key positions of the chromophore, a charge alternation on atoms comparable with that of typical organic dyes cannot be attained in this case. Therefore, photoconductive polymers have much lower extinction coefficients than dyes.[465] Hence, it is necessary both to develop the synthetic routes to OPD with ionized heteroatoms in the chromophore and to continue the search for new photoconductive polymer – dye compositions.

Fluorene derivatives are readily compatible with carbon nanomaterials based on fullerene and nanotubes both through covalent binding and mechanical blending. This line of research is still gaining momentum. As the prospects of its development, one can expect the design of coloured composite materials,[466–470] especially those based on the new carbon polymorph (graphene)[471, 472] and fluorene dyes. Broad opportunities are also opened up in the design of hybrid organic-inorganic materials[473–476] containing dyes based on fluorene and its derivatives.

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