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COORDINATION COMPOUNDS

Effect of Alkyl, Aryl, and *meso*-Aza Substitution on the Thermal Stability of BODIPY

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Abstract—The effect of peripheral alkyl, aryl, and *meso*-aza substitution on the thermal stability of BODIPYs in an argon or oxygen atmosphere has been analyzed using thermogravimetric study results. It has been shown that an increase in the length of 2,6-alkyl substituents to seven carbon atoms is accompanied by the growth of BODIPY thermal stability by 80°C. The greatest increase in the destruction temperature of BODIPY (by 100°C) is attained via the introduction of phenyl groups in the 1,3,5,7-positions of its dipyrromethenmethene framework. *meso*-Aza substitution does not almost produce any effect on the thermal stability of BODIPY dyes. The BODIPY destruction beginning temperature decreases by 60–90°C in the presence of air oxygen. The thermal stability of BODIPY tends to decrease with reducing degree and symmetry of alkyl substitution in the dipyrromethene framework. A lower thermal stability of BODIPY in comparison with zinc(II) dipyrromethenates is due to the participation of fluorine atoms in intramolecular redox processes.

Keywords: dipyrromethenmethene, aza-dipyrromethene, alkyl, phenyl substituents, BODIPY, thermal stability, structural

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One of the urgent problems of contemporary chemistry is the synthesis of new fluorescent dyes suitable for application in analytical chemistry, medicine, biology, laser engineering, etc. An important indicator of the applicability of luminophor dyes as components of optical materials, temperature sensors, etc. is the temperature range within which these compounds are not subject to destruction and do not change their properties under certain ambient conditions.

Our analysis of the literature data showed that most information on the thermal stability of acyclic oligopyrrol compounds is found in the publications of our scientific team. By now, we have collected a database on the thermal stability of some acyclic olygopyrrols, such as ligands of 2,2'-dipyrromethenmethenes, their furyl and thiehyl derivatives, 2,2'-, 2,3'-, and 3,3'-bis(dipyrromethenmethenes), their salts with hydrobromic acid, and corresponding coordination compounds with *d* metals [1–11]. However, there are almost no data on the thermal properties of representatives of the most promising class of luminophors, i.e., 4,4-difluoro-4-boro-3a,4a-diaza-S-indacene (BODIPY) derivatives and their aza analogues (aza-BODIPY).

In two recent decades, BODIPYs have attained the status of the most promising luminophors with a broad spectrum of their practical application as markers, chemosensors, laser dyes, photodynamic therapy agents, components of photosensitive organic materials for photogalvanic devices, etc. [12–16]. The variety of application fields for BODIPYs dyes is undoubtedly associated with the unique set of such practically important characteristics as intense fluorescence in the visible and near-infrared spectral regions, high photostability, and low chemical activity. A number of directions in the practical application of BODIPYs are associated with the use of increased temperatures. For this reason, the study of their thermal stability is one of the main problems for estimating the efficiency of the practical application of BODIPY luminophors.

It is obvious that the distinctions in the thermal stability of BODIPYs will be determined in the first instance by the specific features of the molecular structure of dipyrromethenmethene ligands, i.e., by the nature of a *meso*-spacer and substituents at the periphery of pyrrol rings [6, 9, 17]. The first results of studying the thermal stability of a series of alkyl-substituted BODIPY were obtained in an air oxygen atmosphere in the earlier work [18]. The effect of aryl and *meso*-aza substitution and ambient conditions (oxidative or inert medium) on the thermal stability of BODIPY has not been studied before.

To study the listed effects, we performed the thermogravimetric analysis of crystalline BODIPY samples **1–8** (Fig. 1) in an inert argon atmosphere. Molecules of BODIPYs **1–6** contain methyl groups in the 1,3,5,7-positions of pyrrol rings. In contrast to

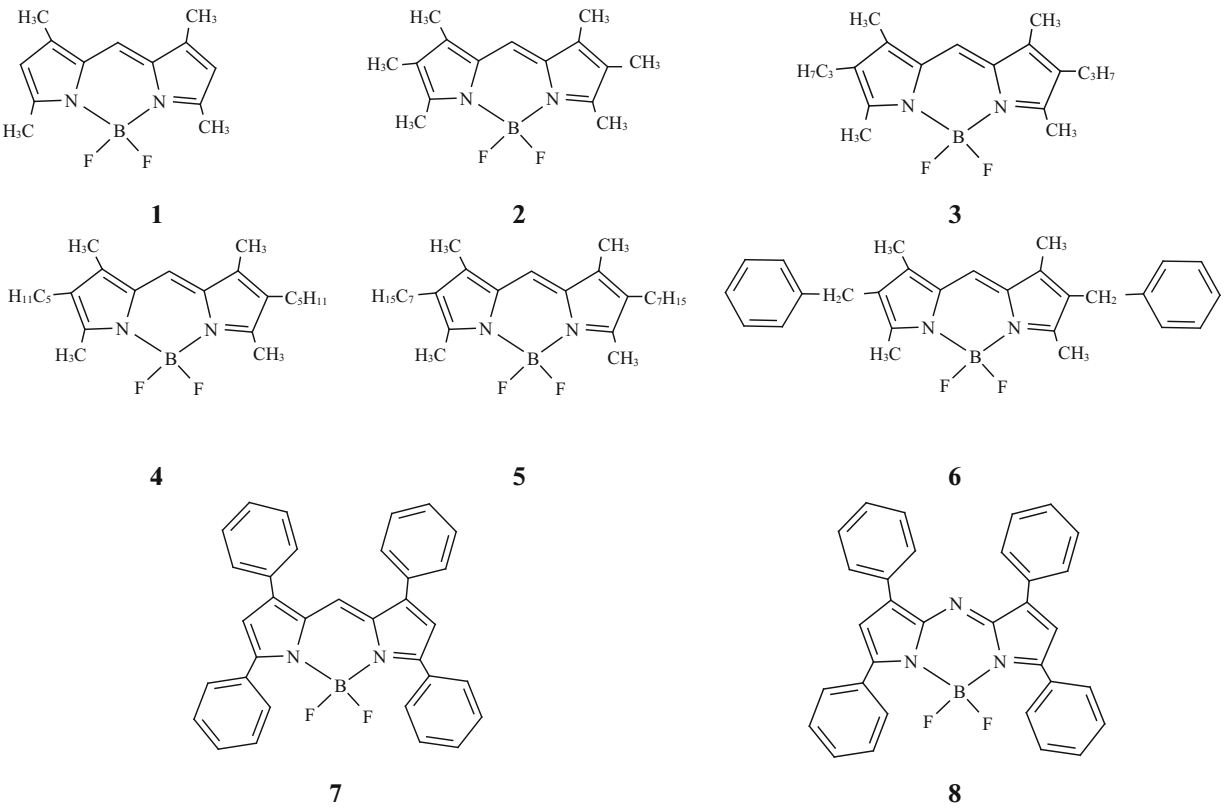


Fig. 1. Structures of BODIPYs **1–8.**

tetramethyl-substituted BODIPY **1**, the hydrogen atoms in the 2,6-positions of molecules of BODIPYs **2–5** are replaced by alkyl substituents with a sequentially increasing number of carbon atoms (C_1, C_3, C_5, and C_7, respectively) in their hydrocarbon chain. The 2,6-positions in a molecule of BODIPY **6** are occupied by seven-membered benzyl groups, in which the aromatic system of their phenyl ring does not participate in the effective conjugation with the dipyrromethenmethene aromatic system. In molecules of BODIPY **7** and aza-BODIPY **8**, four phenyl groups are introduced immediately in the 1,3,5,7-positions of pyrrol rings. A small angle of the turn of phenyl group planes with respect to the dipyrromethenmethene framework plane provides the partial conjugation of their aromatic systems, thus appreciably improving the characteristics of the practically important spectral-luminescent properties of these dyes [19].

EXPERIMENTAL

The synthesis, mass spectrometry, and elemental analysis of complexes **1–8** and the NMR and UV spectroscopy data for them are described in the works [18, 19].

BODIPYs **1–8** were synthesized by the traditional method using the reaction of a corresponding dipyrromethenmethene or 8-*meso*-aza-dipyrromethen with boron trifluoride etherate in methylene dichloride at room temperature with a high yield (from 79.5 to 97%) (Fig. 2). The mixture was stirred for several hours and then washed with water three times. The organic layer was separated out and evaporated to dryness on a rotary evaporator at a decreased pressure. The solid precipitate was dissolved in methylene dichloride and subjected to chromatographic analysis on a silicagel. The effluent was evaporated, and the complex was precipitated with methanol, filtered out, and dried in air at room temperature.

1,3,5,7-Tetramethyldipyrromethenmethene difluoroborate (1). Yield, 0.263 g (1.06 mmol, 92.9%). ^{1}H NMR spectrum (δ, ppm): 7.07 s (1H, *ms*-H), 6.07 s (2H, 2,6-H), 2.55 s (6H, CH_3), 2.27 s (6H, CH_3).

For $C_{13}H_{15}BF_2N_2$, anal. calcd. (%): C, 62.94; H, 6.09; N, 11.29.

Found (%): C, 62.83; H, 6.0; N, 11.18.

1,2,3,5,6,7-Hexamethyldipyrromethenmethene difluoroborate (2). Yield, 0.436 g (1.58 mmol, 97%). ^{1}H NMR spectrum (δ, ppm): 6.96 s (1H, *ms*-CH), 2.49 s (6H, 3,5-CH_3), 2.16 s (6H, 1,7-CH_3), 1.94 s (6H, 2,6-CH_3).

For $C_{15}H_{19}BF_2N_2$, anal. calcd. (%): C, 65.25; H, 6.94; N, 10.14.

Found (%): C, 65.18; H, 6.85; N, 10.04.

1,3,5,7-Tetramethyl-2,6-dipropyldipyrromethenmethene difluoroborate (3). Yield, 0.303 g (0.914 mmol, 95%). ^{1}H NMR spectrum (δ, ppm): 6.97 s (1H, *ms*-H), 2.51 s (6H, 3,5-CH_3), 2.35 t (4H, J = 7.4 Hz, 2,6-CH_2–propyl), 2.17 s (6H, 1,7-CH_3), 1.49 quartet (4H, J = 7.4 Hz, CH_2–propyl), 0.95 t (6H, J = 7.4 Hz, CH_3–propyl).

For $C_{19}H_{27}BF_2N_2$, anal. calcd. (%): C, 68.69; H, 8.19; N, 8.43.

Found (%): C, 68.29; H, 8.03; N, 8.13.

1,3,5,7-Tetramethyl-2,6-dipentyldipyrromethenmethene difluoroborate (4). Yield, 0.411 g (1.05 mmol, 87%). ^{1}H NMR spectrum (δ, ppm): 6.96 s (1H, *ms*-H), 2.50 s (6H, 3,5-CH_3), 2.36 t (4H, J = 7.3 Hz, 2,6-CH_2–pentyl), 2.18 s (6H, 1,7-CH_3), 1.45 quartet (4H, J = 7.3 Hz, CH_2–pentyl), 1.33 quartet (8H, J = 7.3 Hz, CH_2–pentyl), 0.92 t (6H, CH_3–pentyl).

For $C_{23}H_{35}BF_2N_2$, anal. calcd. (%): C, 71.13; H, 9.08; N, 7.21.

Found (%): C, 1.01; H, 8.95; N, 7.14.

1,3,5,7-Tetrametyl-2,6-diheptyldipyrromethenmethene difluoroborate (5). Yield, 0.480 g (1.07 mmol, 97%). ^{1}H NMR spectrum (δ, ppm): 6.96 s (1H, *ms*-H), 2.50 s (6H, 3,5-CH_3), 2.36 t (4H, J = 7.5 Hz, 2,6-CH_2–heptyl), 2.17 s (6H, 1,7-CH_3), 1.44 quartet (4H, J = 7.5 Hz, CH_2–heptyl), 1.32 m (16H, CH_2–heptyl), 0.91 t (6H, J = 7.5 Hz, CH_3–heptyl).

For $C_{27}H_{43}BF_2N_2$, anal. calcd. (%): C, 72.96; H, 9.75; N, 6.30.

Found (%): C, 72.85; H, 9.63; N, 6.21.

1,3,5,7-Tetramethyl-2,6-dibenzyldipyrromethenmethene (6). Yield, 81.2%. ^{1}H NMR spectrum (δ, ppm): 7.29 t (4H, J = 7.3 Hz, 3',5'-H–phenyl benzyl moiety), 7.21 t (2H, J = 7.3 Hz, 4'-H–phenyl benzyl moiety), 7.15 d (4H, J = 7.3 Hz, 2',6'-H–phenyl benzyl moiety), 7.07 s (1H, *ms*-H), 3.80 s (4H, CH_2–benzyl), 2.47 s (6H, 3,5-CH_3), 2.18 s (6H, 1,7-CH_3).

For $C_{27}H_{27}BF_2N_2$, anal. calcd. (%): C, 75.71; H, 6.35; N, 6.54.

Found (%): C, 75.67; H, 6.29; N, 6.42.

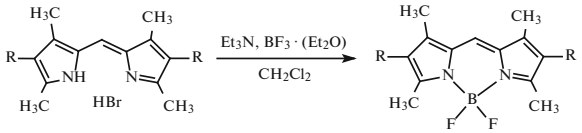
1,3,5,7-Tetraphenyldipyrrolylmethenmethene difluoroborate (7). Yield, 79.5%. ^{1}H NMR spectrum (δ, ppm): 7.43–7.58 (m, 8H, *o*-H–Ph), 7.17–7.40 (m, 12H, *m*,*p*-H–Ph), 6.76 (s, 2H, 2,6-H), 5.40 (s, 1H, *ms*-H).

For $C_{33}H_{23}BF_2N_2$, anal. calcd. (%): C, 79.85; H, 4.67; N, 5.64.

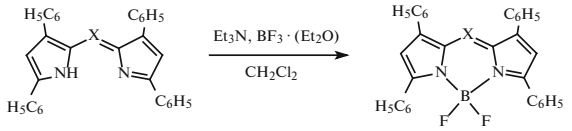
Found (%): C, 79.07; H, 4.48; N, 5.32.

1,3,5,7-Tetraphenyl-8-aza-dipyrrolylmethenmethene difluoroborate (8). Yield, 85.6%. ^{1}H NMR spectrum (δ, ppm): 8.04–8.15 (m, 8H, *o*-H–Ph), 7.44–7.58 (m, 12H, *m*,*p*-H–Ph), 7.07 (s, 2H, 2,6-H).

For $C_{32}H_{22}BF_2N_3$, anal. calcd. (%): C, 77.28; H, 4.46; N, 8.45.



R = H (**1**), CH_3 (**2**), C_3H_7 (**3**), C_5H_{11} (**4**), C_7H_{11} (**5**), CH_2–C_6H_5 (**6**).



X = CH (**7**), N (**8**)

Fig. 2. Scheme for the synthesis of BODIPYs **1–8**.

Found (%): C, 76.85; H, 4.22; N, 8.32.

^{1}H NMR spectra (500 MHz, $CDCl_3$) of the complexes were recorded on a Bruker 500 spectrometer (Germany) in the Shared Facilities Center Upper Volga Regional Center of Physicochemical Studies of the Krestov Institute of Solution Chemistry of the Russian Academy of Sciences. Elemental analysis was performed on a FLASH EA1112 analyzer (Italy).

Thermal analysis (TG/DTG) of the complexes was performed in argon using a Netzsch TG 209 F1 microthermobalance (Shared Facilities Center Upper Volga Regional Center of Physicochemical Studies. Krestov Institute of Solution Chemistry, Russian Academy of Sciences). The sample heating rate was 10 K/min, and the mass of portions was 3–9 mg. The temperature range was 25–700°C. The microthermobalance resolution was 1×10^{-4} mg. The reproducibility of thermograms was controlled via the three- or five-fold repetition of experiment for every complex. Before measurements, the samples were ground and dried in a vacuum pistol under slight heating to a constant weight.

Mass spectra were taken on a Shimadzu MALDI-TOF AXIMA Confidence mass spectrometer (Shared Facilities Center, Ivanovo State University of Chemical Technology).

RESULTS AND DISCUSSION

The destruction of complexes **1–8**, as well as the earlier studied bis(dipyrromethenates) of *d*-metals [20], in an inert argon atmosphere occurs at the expense of intramolecular redox processes. The thermograms of complexes **1–8** in an argon atmosphere are shown in Fig. 3. The TG and DTG curves of complexes **1–6** have similar shapes. No mass loss is almost observed for the samples below the destruction beginning temperature in the TG and DTG curves of the thermograms of complexes **1–6**, thus indicating the absence of residual solvent traces in their composition.

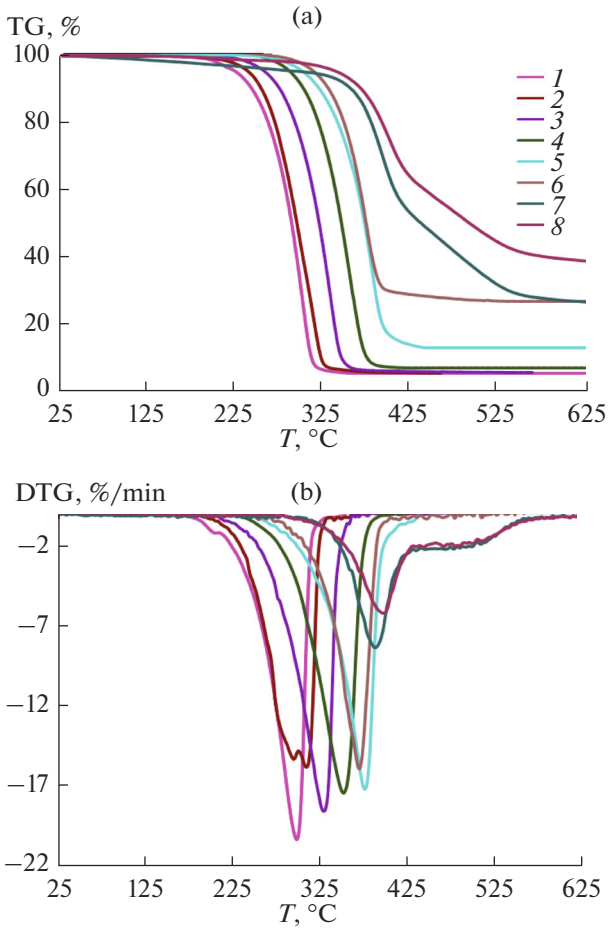


Fig. 3. (a) TG and (b) DTG curves for BODIPYs **1–8**.

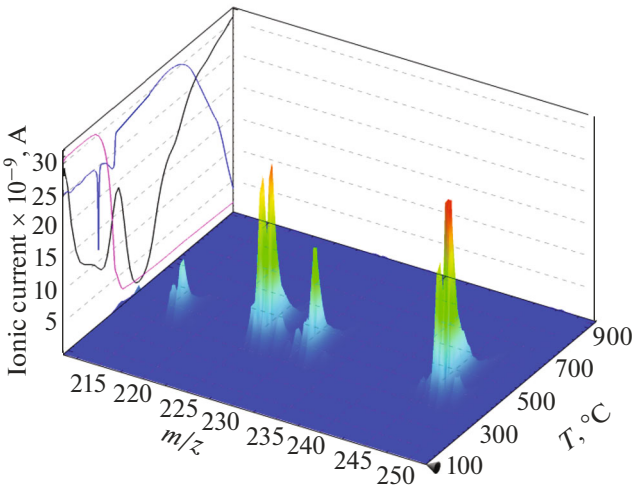


Fig. 4. Mass spectrum fragment for the gas phase of complex **1** depending on temperature.

The destruction of complexes **1–6** begins at temperatures above 260°C and incorporates one sample mass loss stage extending in the TG and DTF curves throughout the interval from 100 to 150°C (Fig 3).

The mass analysis of the composition of the gas phase over solid BODIPY sample **1** (Fig. 4) under heating have shown the presence of the most intense peak corresponding to the molecular ion with m/z = 248 in the mass spectrum. The other peaks with masses 232, 227, 217, 114, 107, and lower have low intensity and correspond to fragmentary ions.

In contrast to **1–6**, the destruction of tetraphenyl-substituted BODIPY **7** and aza-BODIPY **8** occurs in two stages. The first stage (Fig. 3b) begins at ~360°C and is accompanied by a mass loss of 42 and 56% (for BODIPY **7** and BODIPY **8**, respectively). The second stage of mass loss within a range of 410–540°C is accompanied by a much smaller mass loss in a sample (19.4%).

The quantitative characteristics of the thermal destruction processes of BODIPYs **1–8** (destruction process beginning, maximum effect, and end temperatures) are given in Table 1. All dyes **1–8** in an inert argon atmosphere have demonstrated high thermal stability. The destruction beginning temperature (T_1) of BODIPY **1–8** in an argon atmosphere grows from 258.5 to 361.3°C in the sequential series of complexes as **1** < **2** < **3** < **4** < **5** ~ **6** < **7** ~ **8** (Table 1).

Alkyl substitution. Methylation at the 2,6-positions of the dipyrromethenmethene framework does not almost produce any effect on the thermal stability of boron(III) hexamethyl-substituted dipyrromethenate **2** in comparison with tetramethyl-substituted BODIPY **1**. A more appreciable effect is observed upon an increase in the size of 2,6-alkyl substituents. An increase in the length of a hydrocarbon chain by every two carbon atoms is accompanied by a growth in the thermal stability of BODIPY by 22–30°C. The introduction of heptyl and benzyl 2,6-substituents gives the same effect of increase in the thermal stability of BODIPY **5** and BODIPY **6**. As a result, the destruction beginning temperature differs by 80.2°C for the outermost members of the series of complexes **1, 2, 3, 4, 5,** and **6**.

The comparison with the earlier obtained data [18] shows that the thermal stability of BODIPYs **1–6** in an inert argon atmosphere increases by 62–94°C in comparison with an oxidative air oxygen atmosphere.

It should be noted that, in contrast to an inert atmosphere, the thermal destruction of alkyl-substituted BODIPYs **1–6** in an air oxygen atmosphere includes, according to [18], three mass loss stages accompanied by the broadened exotherms in their DTA curve. In an oxidative atmosphere, the regularity of growth in the thermal stability of BODIPYs **1–6** with increasing alkylation degree and length of 2,6-alkyl substituents also persists in the series of complexes **1, 2, 3, 4, 5,** and **6**, and the destruction beginning temperature T_1 maximally increases by 66°C [18].

Table 1. Thermal decomposition of BODIPYs **1–8** in the atmosphere of air (O_2) [18] and argon (Ar)*

BODIPY	$T_1(O_2)$ [18]	Ar				
		T_1, °C	T_{1min}, °C	T_2, °C	T_{2min}, °C	T_3, °C
1	188	258.5	300.5	–	–	311.0
2	199	260.7	297.5	–	–	323.4
3	206	290.6	331.6	–	–	344.4
4	235	312.8	354.3	–	–	369.8
5	245	338.7	378.5	–	–	391.4
6	254	338.0	372.0	–	–	386.3
7	–	361.0	390.5	407.9	482.4	525.7
8	–	361.3	399.3	415.6	480.0	537.2

*T_1 and T_2 are the beginning temperatures of the first and second sample destruction stages, respectively; T_{min} is the minimum temperature on a DTG curve; and T_3 is the destruction end temperature.

The comparison of data on the thermal stability of complexes **1–6** and earlier studied dyes **9–12** [18] (Fig. 5) allows us to observe the tendency to a decrease in the thermal stability of BODIPY dyes upon the violation of symmetry in the arrangement of alkyl substituents in the dipyrromethenmethene framework and a decrease in their number and length [18].

Taking into account the earlier obtained data [21], it is possible to conclude that the thermal stability of BODIPY is 58–81°C lower than for the $[Zn(dpm)_2]$ complexes homotypic by their dipyrromethenmethene ligand in both an oxidative air oxygen atmosphere and argon. For example, the destruction beginning temperature of hexamethyl-substituted BODIPY **2** in an air oxygen atmosphere is ~80°C lower than for the zinc(II) complex with the corresponding $[Zn(dpm)_2]$ ligand [5, 9]. In argon, the temperature T_1 for BODIPY **1** is 35°C lower than for $[Zn(dpm)_2]$ with the corresponding tetramethyl-substituted ligand [21].

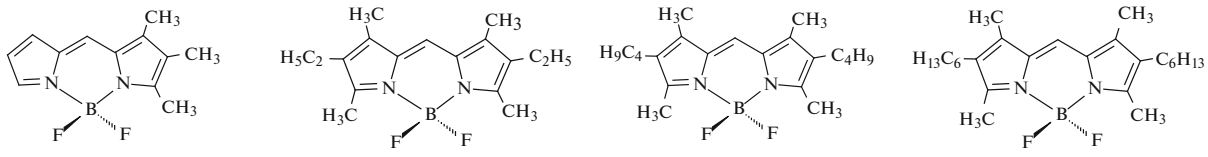
However, the alkyl-substituted zinc(II) dipyrromethenates as compared with the studied BODIPY exhibit the opposite tendency in the effect of the length of 2,6-alkyl substituents: the thermal stability of the $[Zn(dpm)_2]$ complexes slightly decreases upon the sequential replacement of methyl substituents by ethyl and butyl ones (280, 275, and 279°C, respectively) [5, 9].

The thermal stability of binuclear zinc(II) 3,3'-bis(dipyrromethenmethene) helicates $[Zn_2(bisdpm)_2]$ is comparable with the thermal stability of studied BODIPY **1–6** in an atmospheric oxygen medium, but the destruction beginning temperature of $[Zn_2L_2]$ in an argon medium is much higher (by ~150–200°C) [20].

A lower thermal stability of BODIPY in comparison with dipyrromethenates of transition metals may be due to a great oxidation potential of fluorine atoms and a high covalence of B–F bonds, which initiate the processes of intramolecular oxidation in $[BF_2(dpm)]$ at lower temperatures.

Aryl and *meso*-aza substitution. The replacement of methyl substituents in the 1,3,5,7-positions of the BODIPY framework by phenyl groups gives an appreciable increase (100°C) in the thermal stability of complex **7** in comparison with BODIPY **1**.

Aza substitution in the *meso*-spacer of the dipyrromethenmethene ligand does not almost have any effect



9, T_1 = 185°C **10**, T_1 = 203°C **11**, T_1 = 212°C **12**, T_1 = 236°C

Fig. 5. Structures of BODIPYs **9–12** and their destruction beginning temperatures.

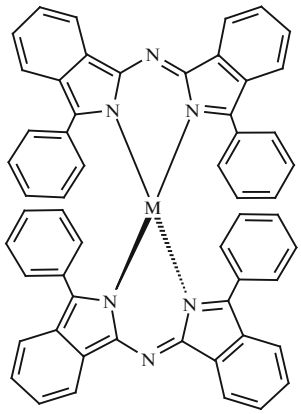


Fig. 6. Bis(phenyl)diisoindol-aza-methenmethene chelate complexes (M = Co, Ni, Cu, Zn, Hg).

on the thermal stability of tetraphenyl-aza-BODIPY **8** in comparison with tetraphenyl-BODIPY **7**.

It should be noted that the same regularity is also observed for the zinc(II) complexes [$Zn(dpm)_2$] with similar tetramethyl-, tetraphenyl-, and tetraphenyl-aza-dipyrromethenmethenes [21]. Thus, the zinc(II) chelate with tetramethyldipyrromethenmethene in an inert argon atmosphere is less stable (T_1 = 293°C) than zinc(II) tetraphenyl dipyrromethenate and zinc(II) *meso*-aza-tetraphenyl dipyrromethenate, for which T_1 = 481 and 458°C, respectively. In this case, tetraphenyl-BODIPY **7** and tetraphenyl-aza-BODIPY **8** are also less thermally stable (by 100–120°C), than the zinc(II) complexes with similar ligands and a structurally similar dye (Fig. 6) [22].

On the other hand, the thermal stability of phenyl-substituted BODIPY **7** and aza-BODIPY **8** is comparable or 15–75°C higher than for Ni(II)), Cu(II), and Hg(II) chelate complexes with *meso*-aza-dipyrromethenmethene due to higher negative oxidation potentials of complex forming agents [22].

It is important to note that complexes **1–8** in thermogravimetric experiments at a heating rate of their samples of ≥(5–10) K/min sustain sublimation with retention of their molecular structure at atmospheric pressure and temperatures, which are much lower than their destruction temperatures. The capability of complexes **1–8** for sublimation under relatively "mild" conditions is of considerable interest for the synthesis of optical materials via the deposition of thin films of luminophors onto multilayered substrates of hybrid matrices by the vacuum sublimation method and requires further studies.

CONCLUSIONS

Hence, it has been established on the basis of the obtained data that alkyl-substituted BODIPYs **1–6**, 1,3,5,7-tetraphenyl-BODIPY **7,** and 1,3,5,7-tetraphenyl-*meso*-aza-BODIPY **8** are thermally stable dyes up to T = 258.5–361.3°C. The greatest increase (by 100°C) in thermal stability is attained by replacing the methyl groups of BODIPY **1** in the 1,3,5,7-positions of its dipyrromethenmethene framework by the phenyl groups of BODIPY **7**. An increase in the length of 2,6-alkyl substituents to C_7 leads to a growth in the destruction beginning temperature of BODIPYs **2–6** by 80°C in comparison with 2,6-nonsubstituted analogue BODIPY **1**. The replacement of the methine *meso*-spacer by a nitrogen atom does not almost produce any effect on the thermal stability of aza-BODIPY **8** in comparison with nonsubstituted analogue BODIPY **7**. The replacement of argon by oxygen decreases the thermal stability of BODIPYs **1–8** by 62–94°C. The common tendency to a reduction in the thermal stability of BODIPYs with a decrease in the degree and symmetry of alkyl substitution in the chromophore framework of a ligand and in the size of the hydrocarbon chains of alkyl substituents is observed. It has been established that the thermal stability of BODIPYs is lower than for the zinc(II) complexes with the same ligands probably due to the participation of fluorine atoms in intramolecular redox processes.

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