Macrocycles, derivatives of nitrogen-containing heterocycles
3.* Synthesis of di(imidazo[1,5-*a*]quinoxalina)-2(1,3)-benzadithiacycloalkaphanes

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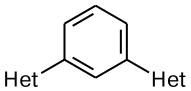
A three-step method for the construction of di(imidazo[1,5-*a*]quinoxalina)-2(1,3)-benzadithiacycloalkaphanes has been developed, which includes introduction of a haloalkyl group of various length and nature into 1,3-bis(4(5*H*)-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl)-benzene at the carbamoyl fragment, transformation of the bishalo-substituted compound thus obtained to bisthiol, and coupling of the latter to a disulfide.

Key words: 1,3-bis(4(5*H*)-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl)benzene, alkylation, isothioureides, hydrolysis, mercaptanes, molecular iodine, oxidation, disulfides, quinoxalinacycloalkaphanes, IR, NMR, and mass spectra.

For assembling systems of molecular recognition and efficient catalytic systems, design and development of methods for the synthesis of macrocyclic ligands containing fragments of nitrogen heterocycles are of great importance.[2] These structures are of interest including a possibility of preparation of mono- and binuclear metal complexes, analogs of nongemic metalloproteines, based on such macrocycles and related 1,3-dihetarylbenzenes **1**.[3—5] There are several examples of macrocycles synthesized specially for binuclear complexation.[6—9] Some of them included biologically actual imidazole rings.



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Het stands for thienyl,[10,11] pyrazolyl,[12] imidazolyl,[12,13] thiazolyl,[13,14] 1,3,4-oxadiazolyl,[15] 1,3,4-thiadiazolyl,[13] 1,2,4-triazolyl,[16—18] benzoxazolyl,[19,20] indolyl[21], benzimidazolyl,[22—24] 1,2,4-triazolo[3,2-*a*]isoindolyl,[25] 1,2,4-triazolo[3,2-*c*]quinazoline[18] (**1** are 10,10′-arylenebis(7*H*-benzo[*de*]-1,2,4-triazollo[5,1-*a*]isoquinolin-7-ones[25]).

The presence in *meta*-positions of the benzene ring of two heterocyclic fragments rigidly oriented with respect to each other makes 1,3-dihetarylbenzenes **1** very interesting as both the bidentate chelating agents and the macrocycle precursors. When appropriate and easily transformable functional groups are present on the heterocyclic rings, compounds **1** can be used for construction of various types of heteromacrocyclic systems including those containing a disulfide fragment, which is of significant interest for organic and bioorganic chemistry.[26]

It is also known that macrocyclic compounds containing a disulfide fragment are under study as redox-active systems and switches,[27,28] in combination with amide functional groups as selective complexation agents with respect to various doubly-charged metal cations,[29] and are of interest as compounds with a wide range of pharmaceutical activity[30—32] including antitumor and inhibiting human immune deficiency virus.

Earlier, we have found that the reaction of 3-(α-chlorobenzyl)quinoxalin-2-one and 3-benzoylquinoxalin-2-one (**2**) with benzylamine in DMSO gives imidazo[1,5-*a*]quinoxalin-4(5*H*)-one.[33] Proliferation of this new approach of assembling the imidazo[1,5-*a*]quinoxalin-4(5*H*)-one system on *m*-di(aminomethyl)benzene allowed us to obtain 1,3-bis(4(5*H*)-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl)benzene (**3**), a valuable precursor for the synthesis of various functionalized macroheterocycles including those containing a disulfide fragment (Scheme 1). The use of a carbamoyl function in molecule **3** for the introduction of various structural fragments opens wide possibilities for performing a macrocycle closing. The presence in the imidazo-[1,5-*a*]quinoxalin-4-one system of various types of nitrogen atoms gives us a reason to consider them as promising chelating agents.

* For Part 2, see Ref. 1.

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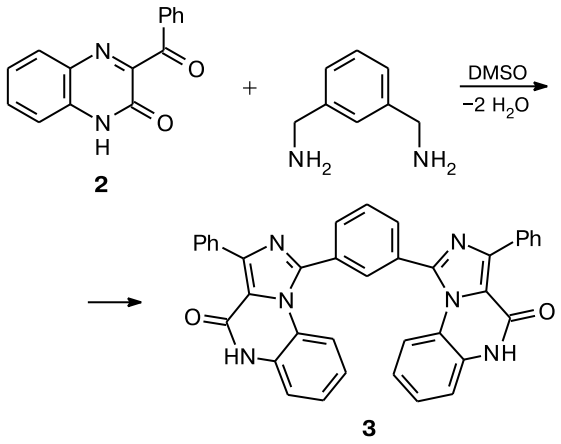
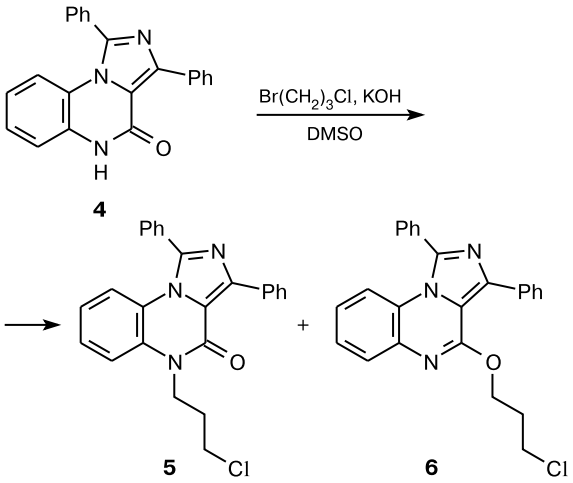
Scheme 1



Scheme 2



Results and Discussion

The purpose of the present work is to reveal possibilities of using compound **3** as a building block for development of macrocyclic systems containing various chains with disulfide groups. To solve the problem set, we implemented a three-step process consisting of (1) introduction of a haloalkyl fragment into 1,3-bis(4(5*H*)-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl)benzene (**3**), (2) transformation of the haloalkyl fragments to mercaptoalkyl ones, and (3) oxidation of 1,3-bis{5-(3-mercaptoalkyl-1)-4(5*H*)-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl}benzene with molecular iodine.

Introduction of a haloalkyl spacer into *m*-bis(4(5*H*)-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl)benzene (3). The presence in the 1,3-bis(4(5*H*)-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl)benzene molecule (**3**) of two carbamoyl fragments, capable of being alkylated at the nitrogen and oxygen atoms in the reactions with dihaloalkanes, suggests formation of products of the N,N′-, O,O′-, and N,O-alkylation, as well as their oligomeric derivatives. Therefore, we started from the development of alkylation procedure and isolation of products based on the model compound, which formally was a half of the molecule of compound **3**, *viz.*, 1,3-diphenylimidazo[1,5-*a*]-quinoxalin-4(1*H*)-one (**4**) synthesized by the reaction of 3-benzoylquinoxalin-2(1*H*)-one **1** with benzylamine proceeding through the oxidative imidazoannulation.[33]

Two ways were used to introduce a haloalkyl fragment into compound **4**. The first of them is a one-step and includes direct alkylation of imidazo[1,5-*a*]quinoxalin-4(1*H*)-one **4** with dihaloalkanes in the presence of KOH in dioxane, which yields two regioisomeric products of N- (**5**) and O-alkylation (**6**) easily separated by column chromatography. The overall yield of the alkylation products was 48%, the ratio was 47 : 1 in favor of the N-alkylation product (Scheme 2).

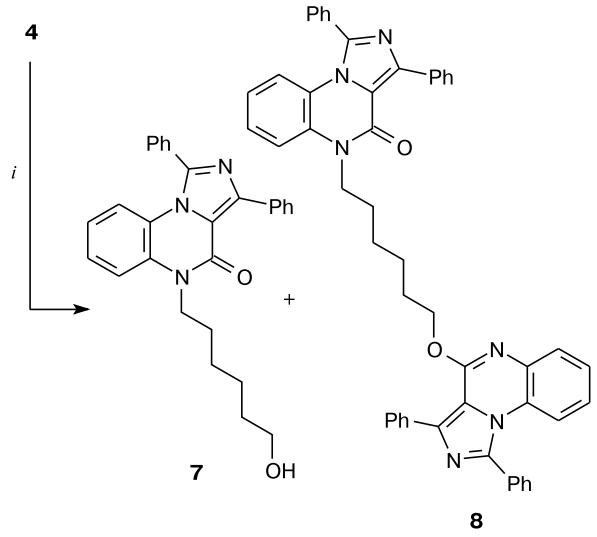
In the ^{1}H NMR spectra of compounds **5** and **6**, in addition to the signals for the aromatic protons, there are signals for the haloalkyl fragment: two triplet signals for the CH_2Cl and NCH_2 groups and a multiplet signal for the third CH_2 group (see Experiment). In this case, the signals for the CH_2Cl and OCH_2 groups of the newly introduced fragment in the O-alkylated isomer **6** are displaced, respectively, upfield and downfield approximately by 0.3—0.4 ppm as compared to the signals for the CH_2Cl and NCH_2 groups in compound **5**.

The second way is a two-step reaction, which is based on the alkylation of 1,3-diphenylimidazo[1,5-*a*]quinoxalin-4(5*H*)-one (**4**) with haloalcohols with subsequent reaction of the hydroxyalkyl derivative of compound **7** with thionyl chloride. The reaction of imidazo[1,5-*a*]-quinoxalin-4(5*H*)-one **4** with 6-bromohexan-1-ol in the presence of KOH in DMSO for 30 h at 40 °C gives two products (Scheme 3). The first, minor product, precipitates from the solution in analytically pure form. In its ^{1}H NMR spectrum, there are two triplet signals with relative intensities 1 : 1 in the region 4.0—4.5 ppm, one of which, resonating more high-field, is broadened like the triplet signal for the NCH_2 group in compound **5**, the other is not broadened like the signal for the OCH_2 group in compound **6**, which confirms the presence of the N- and O-alkylation fragments in the compound formed. In the region 6.5—7.0 ppm, there are two signals with δ 6.81 and 6.92 ppm having, respectively, constants J = 8.3, 7.3, 1.5 and 8.6, 7.4, 1.5 Hz for the H(8) protons of various quinoxaline systems in the proportion 1 : 1 with intensities half as large as compared to the triplet signal in the region 4.0—4.5 ppm.

These data indicate formation of the product containing N- and O-alkylated imidazo[1,5-*a*]quinoxaline systems bound with the hexamethylene chain. Position, splitting, and intensity of other signals do not conflict with the structure suggested and the MALDI mass spectra, in which

the molecular ion peak MH^+ 757 is present, and elemental analysis data definitively confirm the structure and composition of the minor product **8**.

Scheme 3



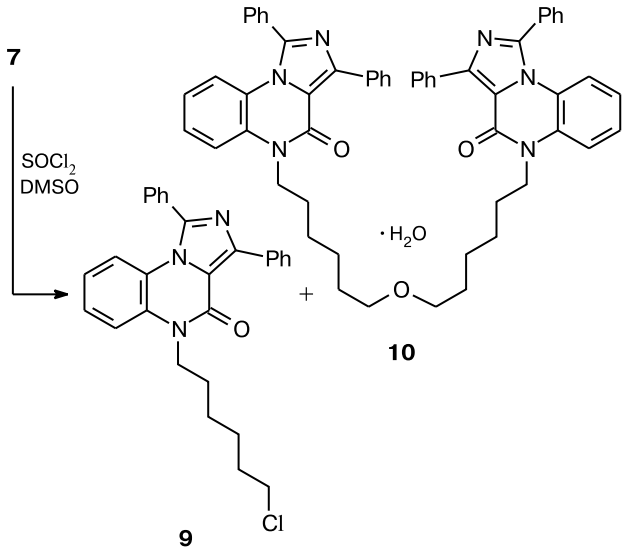
i. $Br(CH_2)_6Cl$, KOH, DMSO.

As it was expected, *N*-hydroxyhexyl derivative **7** was the major product of this reaction, it was isolated from the mother liquor in 40% yield and purified by column chromatography mainly from the unreacted starting tricycle **4**. In the ^1H NMR spectrum of the product, in addition to the aromatic protons there are present five signals for the hydroxyhexyl fragment including the triplet signal for the hydroxy group at δ 4.38 (J = 5.2 Hz) and triplet-of-doublet signal for the methylene group at δ 3.40 ppm (J = 5.9, 5.2 Hz) bonded to the hydroxy group (see Experimental).

The reaction of compound **7** with thionyl chloride upon reflux in excess thionyl chloride for 8 h leads to the substitution of the hydroxy group for the chlorine atom and preparation of imidazo[1,5-*a*]quinoxaline **9** with the *N*-chlorohexyl substituent in 79% yield. It should be noted that the MALDI mass spectrum of the reaction mixture, in addition to the peak of compound **9** with MH^+ 456, exhibits the peak with MH^+ 876 corresponding to the bisimidazo[1,5-*a*]quinoxalinone monohydrate **10** (Scheme 4). However, we failed in isolation and characterization of this compound in the individual state due to its trace amount in the reaction mixture.

The presence of two carbamoyl groups in 1,3-bis(4(5*H*)-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl)benzene **3** should increase the probability of formation of O-alkylated compounds (observed as the side products in the alkylation of imidazo[1,5-*a*]quinoxalin-4(1*H*)-one **4**) in the reactions with 1,n-dihaloalkanes, as is increased

Scheme 4

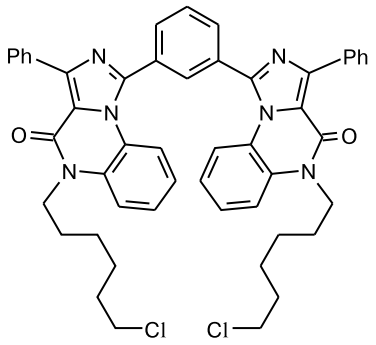


a possibility of formation of unsymmetric product of the N,O-alkylation. A poor solubility of 1,3-bis(4(5*H*)-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl)benzene (**3**) even in warm DMSO or DMF makes it difficult to involve it into the reaction and purify desired products. Nevertheless, we succeeded in the introduction of two haloalkyl fragments into compound **3**.

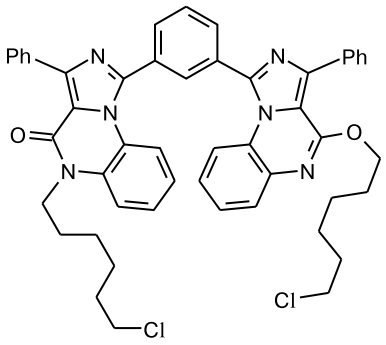
In fact, alkylation of compound **3** with various 1,n-dihaloalkanes (1-bromo-3-chloropropane (*a*), 1,4-dichlorobutane (*b*), 1,5-dichloro-3-oxapentane (*c*), 1,6-dibromohexane (*d*), 1,6-dichlorohexane (*e*)) sometimes leads to the formation of a mixture of isomers insoluble in organic solvents in ~30% yield together with oligomeric products.

Only in the case of 1-bromo-3-chloropropane (*a*), exclusive formation of the N,N′-alkylation product, compound **11a**, occurs (Scheme 5). In other cases, formation of a difficult to isolate mixture of isomers of the N,N′- and N,O-alkylation is observed, which leads to the reduction in the yield of the desired product to 15—20%. The maximum yield of O-alkylated derivatives was reached in the reaction in DMF when 1,6-dichlorohexane was used as the alkylating reactant. This reaction results in the formation of all three isomeric products **11e′**, **11e″**, and **11e‴** in the ratio 39.2 : 60 : 0.8 (the HPLC data).

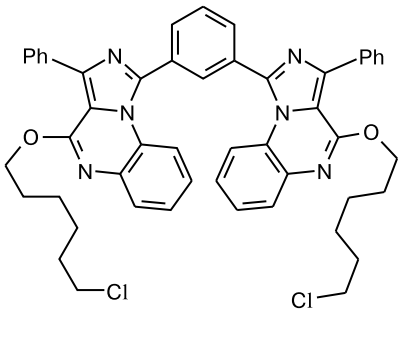
To sum up, compound **11e″**, the product of the N,O-alkylation, is the major product in the reaction of 1,3-bis(4(5*H*)-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl)benzene **3** with dichlorohexane. However, the use of DMSO or dioxane as the solvent instead of DMF allowed us to increase the yields of the desired *N*,*N*′-regioisomer to 70—80%, whereas alkylation in dioxane, unlike in DMSO, allowed us to use dibromoalkanes (1,4-dibromobutane and 1,6-dibromohexane) as the alkylating agents



11e´



11e″

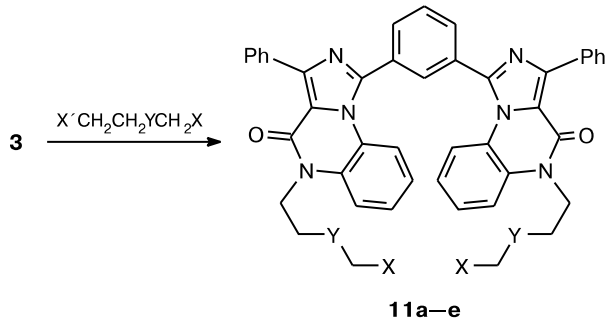


11e´´´

and obtain compounds with more reactive terminal bromoalkyl fragments.

Position and splitting of the signals for the *m*-phenylene protons in the ^{1}H NMR spectra of compounds **11**, independent of the haloalkyl fragment in them, always remain virtually the same. These protons resonate in form of three rather than four signals, which suggests symmetric structure of compounds under study. In addition to the signals for other aromatic protons, in high fields there are additional three signals in compound **11a**, four in compounds **11b,c**, six in compound **11d**, which also confirms symmetry of the structures. The presence in the IR spectra of the absorption band for C=O in the region 1649—1655 cm^{-1} confirms that the N,N´- rather than O,O´-alkylated products are formed.

Scheme 5



11a—e

X´CH$_2$CH$_2$YCH$_2$X				
X	X´	Y	Solvent	**11**
Cl	Br	—	DMSO	**a**
Br	Br	CH_2	Dioxane	**b**
Cl	Cl	OCH_2	DMSO	**c**
Br	Br	$(CH_2)_3$	Dioxane	**d**
Cl	Cl	$(CH_2)_3$	DMF	**e**

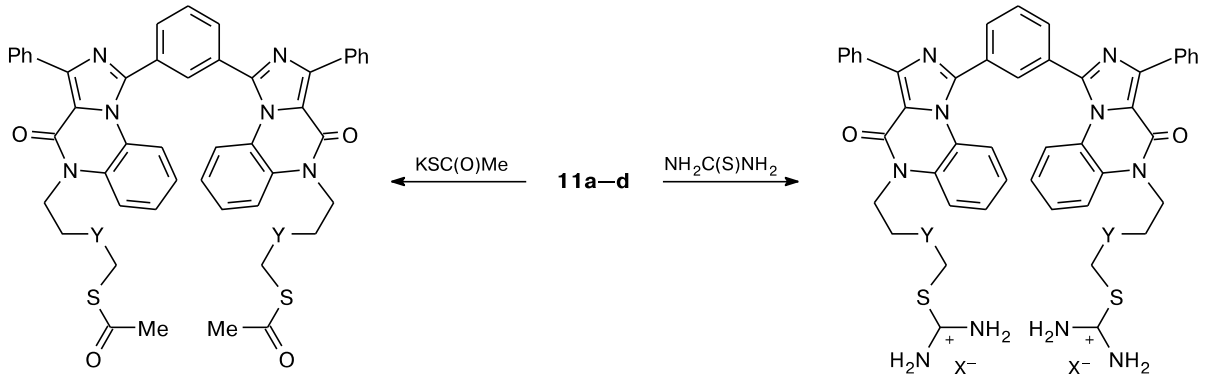
Introduction of a mercapto group into N,N´-alkylated derivatives of *m*-bis(4(5*H*)-oxo-3-phenylimidazo[1,5-*a*]-quinoxalin-1-yl)benzene (11). Transformation of the haloalkyl substituent to mercaptoalkyl one in 1,3-bis(4(5*H*)-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl)benzene derivatives was accomplished by two known ways: the reaction of compounds **11** with thiourea and subsequent alkaline hydrolysis of isothiuronium salts **12** (see Ref. 34) and the reaction of compounds **11** with potassium thioacetate and subsequent acid hydrolysis of thioacetates **13** (see Ref. 35) (Schemes 6 and 7). The reaction of bishalides **11** with thiourea was performed upon heating in dioxane. Bisisothiuronium salts **12** precipitated from the solution in the analytically pure form. The reaction time depended not only on the nature of the halogen, but also on the nature of the substituent. For instance, if dibromides **11b,d** were converted to the corresponding isothioureides in almost quantitative yields over 20 h and dichloride **11a** over 70 h, then the reaction of dichloride **11c** with thiourea even over 120 h led only to 5% yield of isothiuronium salt **12c**.

The ^{1}H NMR spectra of compounds **12** and **13** exhibit an upfield displacement of the triplet signals for the terminal CH_2 group from 3.5—3.7 ppm in compounds **11** to 3.2—3.4 ppm in compounds **12** and to 3.0 ppm in compounds **13**. In addition, in the ^{1}H NMR spectra together with the signals for the aromatic protons there are present the singlet signal for the protons of the methylene groups of the thioacetyl fragments at δ 2.3 ppm in compound **13** and broadened singlet signals for the isothioureide fragments in compound **12**.

Alkaline hydrolysis of isothiuronium salts **12** and acid hydrolysis of thioacetates **13** leads to bisthiols **14** (Scheme 7). The formation of the latter is confirmed by the presence of the peaks for the MH^+ ions in the ESI mass spectra: 745 for compound **14a**, 773 for compound **14b**, and 805 for compound **14c**. The presence in the ^{1}H NMR spectra of the triplet signal for the SH group in the region 1.32—1.59 (t, J = 7.7—8.1 Hz) and the doublet of triplet signals for the CH_2 group bound with the SH group in the region 2.52—2.63 (td, J = 7.5—8.1, 6.2—7.0 Hz), as well as the presence in the IR spectra of the absorption band for the SH group in the region 2542—2566 cm^{-1} confirm the structure of compounds **14**. The yields of bisthione in these reactions is approximately the same and is about 40%.

Oxidation of 1,3-bis[5-(3-mercaptoalkyl-1)-4-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl]benzenes with molecular iodine. Oxidation reaction yielding mainly disulfide is

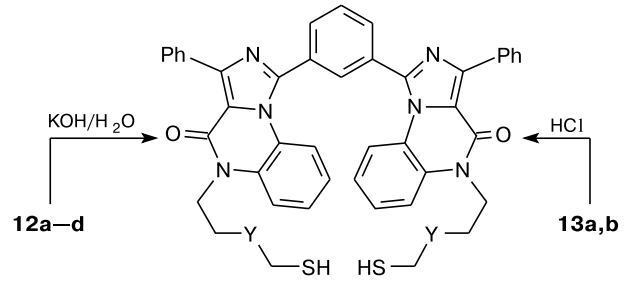
Scheme 6



	Y
13a	—
13b	OCH_2

	X	Y		X	Y
12a	Cl	—	**12c**	Cl	OCH_2
12b	Br	CH_2	**12d**	Br	$(CH_2)_3$

Scheme 7



14	X	Y	**14**	X	Y
a	Cl	—	**c**	Cl	OCH_2
b	Br	CH_2	**d**	Br	$(CH_2)_3$

of importance in chemistry of thiols. First of all, this is due to the fact that some compounds with the mercapto group, for example, glutathione and cysteine play important part in living organisms. Various oxidation procedures have been developed[36] with application of such oxidants as molecular oxygen in the presence of strong bases,[37] salts of high-atomic-weight metals including peroxocomplexes of molybdenum, tungsten, vanadium,[38,39] chromium,[40—42] stannum compounds,[43] Fe^{III},[44] hydrogen peroxide,[45] halogens,[46,47] disulfide dicationic salts.[48]

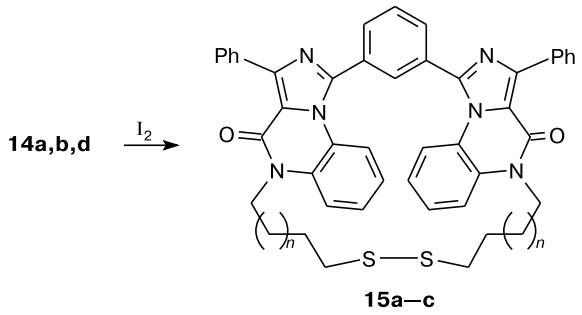
We have chosen molecular iodine successfully used in oxidation of indolisines to biindolisines.[49] The use of molecular iodine as the oxidant for mercaptanes, as a rule, requires presence of a base to combine with the liberated hydrogen iodide, which is a strong reducing agent. However, the presence in bisthiols **14** of the "pyridine" nitrogen atom allowed us to carry out this reaction without using the base. 1,3-Bis[5-(3-mercaptoalkyl-1)-4-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl]benzenes **14** are easily oxidized with molecular iodine in chloroform at room temperature with the formation of dithiacyclophanes **15** (Scheme 8). It was shown that the longer the substituents with the mercapto group in 1,3-bis[5-(3-mercaptoalkyl-1)-4-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl]benzenes **14** the higher the yields of macrocyclic disulfides, which are 8% for **15a**, 30% for **15b**, and 45% for **15c**.

In the ^{1}H NMR spectra, the signals for the geminal methylene protons of the spacer of macrocycles **15** are found separately from the corresponding protons for the starting bismercaptanes **15**, one is more downfield, another is more upfield, the $\Delta\delta$ value varies in the range 1.0—1.5 ppm for signals of the NCH_2 group, which unambiguously indicates the nonequivalence of the protons confirming the formation of macrocyclic systems. Transformation of bismercaptanes **14** to imidazoquinoxalinadithiocycloalkaphanes **15** as well significantly changes position of the singlet signal for the H(2) of the 1,3-phenylene ring, which is displaced upfield from 8.2 ppm in compounds **14** to 7.2 ppm in macrocycle **15a**, 7.4 ppm in macrocycle **15b**, 7.6 ppm in macrocycle **15c**.

It should be also noted that in the IR and ^{1}H NMR spectra of the oxidation products of bismercaptanes **15**, no signs of the SH group are found.

The formation of macrocycles **15** is additionally confirmed by the presence of characteristic peaks of ions in the ESI and EI mass spectra. For example, for the reaction products of compound **14a** with I_2, there are observed peaks of ions with m/z 743 and $[2M+H]^+$ 1485 (compound **15a**), whereas for the reaction product of **14c** with I_2, the EI mass spectrum exhibits the peak of the molecular ion M^+ 826 corresponding to compound **15c**.

Scheme 8



15: n = 0 (**a**), 1 (**b**), 3 (**c**)

In conclusion, a three-step method was developed for the synthesis of diimidazo[1,5-*a*]quinoxalina-2(1,3)-benzadithiacycloalkaphanes, which includes (1) introduction of a haloalkyl fragment of various length and nature into 1,3-bis(4(5*H*)-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl)benzene at the carbamoyl group, (2) transformation of the haloalkyl fragments to the mercaptoalkyl through isothioureide and thioacetate derivatives, and (3) oxidation of 1,3-bis{5-mercaptoalkyl-4-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl}benzene with molecular iodine.

Experimental

Melting points were determined on a Boetius heating stage. IR spectra were recorded on a Avatar-360 spectrometer in KBr pellets for compounds **5—9**, **11a—c**, **12a—c**, **13a,b**, and **14a—c** and on a Bruker Vector-22 spectrometer for compounds **11d**, **12d**, **14d**, and **15a—c**. ^{1}H NMR spectra were recorded on a Bruker Avance-300 Fourier-spectrometer (300.13 (^{1}H)) in $CDCl_3$ for compounds **6**, **8**, **11a,b**, **13a**, and **14a—c,** in DMSO-d_6 for compounds **5**, **7**, **9**, and **12a—c**; on a Bruker Avance-600 spectrometer (600.00 MHz (^{1}H)) in $CDCl_3$ for compound **15a**; on a Bruker Avance-400 spectrometer (400.00 MHz (^{1}H)) in $CDCl_3$ for compounds **14d** and **15a—c** and in DMSO-d_6 for compounds **11d** and **12d**. ^{1}H NMR spectrum for compound **11c** and ^{13}C NMR spectra for all compounds were recorded on a Bruker DRX-500 (500.13 MHz (^{1}H) and 125.75 (^{13}C)) in DMSO-d_6 for compounds **12a—c** and in $CDCl_3$ for compounds **11a—c**. Residual signal of the corresponding solvent was used as an internal standard. Mass spectra of electron ionization (EI) were obtained on a ThermoQuest TRACE MS quadrupole mass spectrometer with direct injection of the sample using water cooling (DIP). MALDI mass spectra were obtained on a Bruker Daltonic GmbH ULTRAFLEX III mass spectrometer, *p*-nitroaniline was used as a matrix. Mass spectra obtained by electrospray method (ESI-MS) were recorded on a Bruker Esquire mass spectrometer with detector of ions.

The IUPAC nomenclature was used in naming of macrocycles.[50,51]

Reaction of 1,3-diphenylimidazo[1,5-*a*]quinoxalin-4(5*H*)-one (4) with 3-bromochloropropane. A mixture of compound **4** (1.0 g, 3 mmol), KOH (0.26 g, 4.6 mmol), and DMSO (15 mL) was stirred for 5 min, then 1-bromo-3-chloropropane (0.35 mL, 3.5 mmol) was added, the mixture was stirred for 25 h at room temperature and 5 h at 40 °C. The reaction mixture was cooled, the crystals formed were filtered off, washed with Pr^{i}OH and water to obtain analytically pure compound **5**. The filtrate was poured into water, the crystals formed were filtered off, washed with water, dried, products **5** and **6** were separated by column chromatography (eluent: $CHCl_3$: EtOAc = 9 : 1) on silica gel (Merck).

5-(3-Chloroprop-1-yl)-1,3-diphenylimidazo[1,5-*a*]quinoxalin-4-one (5). The yield of compound **5** was 0.58 g (47%), white powder, m.p. 210—212 °C. IR, ν/cm^{-1}: 1326, 1394, 1443, 1483, 1591, 1611, 1651. ^{1}H NMR, δ: 2.10—2.25 (m, 2 H, $CH_2C\underline{H}_2CH_2$); 3.83 (t, 2 H, CH_2Cl, J = 6.6 Hz); 4.37 (m, 2 H, CH_2N, J = 7.2 Hz); 7.00 (dd, 1 H, H(8), J = 7.9 Hz, J = 7.5 Hz); 7.20 (d, 1 H, H(9), J = 8.6 Hz); 7.35—7.68 (m, 8 H, H(6), H(7), *m*-, *p*-H_{Ph}); 7.73 (dd, 2 H, *o*-H, C_{Ph}(1), J = 7.6 Hz, J = 1.6 Hz); 8.15 (dd, 2 H, *o*-H, C_{Ph}(3), J = 8.6 Hz, J = 1.7 Hz). MS MALDI TOF, *m/z*: $[M+H]^+$ 414. Found (%): C, 72.37; H, 4.97; N, 10.05; Cl, 8.51. $C_{25}H_{20}N_3OCl$. Calculated (%): C, 72.55; H, 4.87; N, 10.15; Cl, 8.57.

4-(3-Chloroprop-1-yloxy)-1,3-diphenylimidazo[1,5-*a*]quinoxaline (6). The yield of compound **6** was 12 mg (1%), white powder, m.p. 139—141 °C. IR, ν/cm^{-1}: 563, 594, 651, 686, 698, 753, 731, 773, 914, 928, 971, 1024, 1094, 1128, 1157, 1226, 1302, 1322, 1350, 1365, 1425, 1473, 1539, 1559, 1615, 2904, 2971, 3058. ^{1}H NMR, δ: 2.15—2.25 (m, 2 H, $CH_2C\underline{H}_2CH_2$); 3.44 (m, 2 H, CH_2Cl, J = 6.5 Hz); 4.67 (m, 2 H, CH_2O, J = 6.0 Hz); 7.00—7.10 (m, 1 H, H(8)); 7.30—7.90 (m, 13 H, H_{Ph}(6), H_{Ph}(7), H_{Ph}(9)). MS MALDI TOF, *m/z*: $[M+H]^+$ 414. Found (%): C, 72.42; H, 4.99; N, 10.22; Cl, 8.43. $C_{25}H_{20}N_3OCl$. Calculated (%): C, 72.55; H, 4.87; N, 10.15; Cl, 8.57.

Reaction of 1,3-diphenylimidazo[1,5-*a*]quinoxalin-4(5*H*)-one (4) with 6-bromohexanol. A mixture of compound **4** (0.95 g, 2.8 mmol), KOH (0.24 g, 4.3 mmol), and DMSO (25 mL) was stirred for 5 min, 6-bromohexanol (0.44 mL, 3.4 mmol)l was added, and the mixture was stirred for 30 h at 40 °C. The reaction mixture was cooled, the crystals formed were filtered off, washed with Pr^{i}OH and water to obtain analytically pure compound **8** (characteristics are given below). The filtrate was poured into water, the crystals formed were filtered off, washed with water, dried, and purified by column chromatography (eluent: $CHCl_3$: MeOH = 100 : 1) on silica gel (60A, 0.060—0.200 mm) to obtain compound **7**.

5-(6-Hydroxyhex-1-yl)-1,3-diphenylimidazo[1,5-*a*]quinoxalin-4-one (7). The yield of compound **7** was 0.47 g (38%), white powder, m.p. 109—112 °C. IR, ν/cm^{-1}: 556, 591, 667, 697, 745, 768, 779, 924, 1012, 1056, 1115, 1249, 1298, 1394, 1460, 1485, 1592, 1612, 1659, 2853, 2927, 3000—3500. ^{1}H NMR, δ: 1.35—1.45 (m, 6 H, $OCH_2(C\underline{H}_2)_3$); 1.55—1.65 (m, 2 H, $NCH_2C\underline{H}_2$); 3.40 (td, 2 H, OCH_2, J = 5.9 Hz, J = 5.2 Hz); 4.22 (t, 2 H, CH_2N, J = 7.4 Hz); 4.38 (t, 1 H, OH, J = 5.2 Hz); 7.00 (dd, 1 H, H(8), J = 7.6 Hz, J = 7.5 Hz); 7.20 (d, 1 H, H(9), J = 8.6 Hz); 7.38—7.68 (m, 8 H, H(6), H(7), *m*-, *p*-H_{Ph}); 7.72—7.78 (m, 2 H, *o*-H, C_{Ph}(1)); 8.14 (dd, 2 H, *o*-H, C_{Ph}(3), J = 8.2 Hz, J = 1.3 Hz). MS MALDI TOF, *m/z*: $[MH]^+$ 438. Found (%): C, 76.79; H, 6.30; N, 9.57. $C_{28}H_{27}N_3O_2$. Calculated (%): C, 76.86; H, 6.22; N, 9.60.

1-(1,3-Diphenylimidazo[1,5-*a*]quinoxalin-4-yloxy)-6-(4-oxo-1,3-diphenylimidazo[1,5-*a*]quinoxalin-5-yl)hexane (8). The yield of compound **8** was 50 mg (2.3%), white powder, m.p. 138—140 °C. IR, ν/cm^{-1}: 558, 594, 669, 693, 744, 770, 917, 1026, 1116, 1228, 1300, 1322, 1392, 1463, 1486, 1540,

1612, 1653, 2854, 2929, 3052. ^{1}H NMR, δ: 1.20—1.35 (m, 4 H, $O(CH_2)_2C\underline{H}_2C\underline{H}_2(CH_2)_2N$); 1.60—1.80 (m, 4 H, $OCH_2C\underline{H}_2(CH_2)_2C\underline{H}_2CH_2N$); 4.09 (t, 2 H, CH_2N, J = 7.7 Hz); 4.44 (t, 2 H, OCH_2, J = 6.2 Hz); 6.81 (ddd, 1 H, H(8), quinoxaline, J = 8.6 Hz, J = 7.4 Hz, J = 1.5 Hz); 6.92 (ddd, 1 H, H(8), quinoxaline, J = 8.3 Hz, J = 7.3 Hz, J = 1.5 Hz); 7.13—7.62 (m, 18 H, H(5)—H(7), quinoxaline, *m*-, *p*-H_{Ph}); 7.77 (dd, 2 H, *o*-H, $C_{Ph}(1)$, J = 7.5 Hz, J = 1.3 Hz); 8.14 (dd, 2 H, *o*-H, $C_{Ph}(3)$, J = 8.4 Hz, J = 1.4 Hz). MS MALDI-TOF, *m/z*: $[MH]^+$ 757. Found (%): C, 79.37; H, 5.37; N, 11.02. $C_{50}H_{40}N_6O_2$. Calculated (%): C, 79.34; H, 5.33; N, 11.10.

5-(6-Chlorohex-1-yl)-1,3-diphenylimidazo[1,5-*a*]quinoxalin-4-one (9). A solution of compound **7** (0.73 g, 1.7 mmol) in $SOCl_2$ (5 mL) was refluxed for 8 h, the solvent was evaporated, the residue was treated with aqueous $NaHCO_3$, the crystals formed were filtered off, washed with water, dried, and purified by column chromatography (eluent: $CHCl_3$: EtOAc = 9 : 1). The yield of compound **9** was 0.60 g (79%), white powder, m.p. 137—139 °C. IR, ν/cm^{-1}: 555, 585, 646, 668, 691, 744, 780, 881, 969, 1027, 1072, 1121, 1180, 1252, 1299, 1392, 1462, 1484, 1590, 1610, 1648, 2851, 2925, 2951, 3055. ^{1}H NMR, δ: 1.40—1.60 (m, 4 H, $Cl(CH_2)_2C\underline{H}_2C\underline{H}_2(CH_2)_2N$); 1.65—1.85 (m, 4 H, $ClCH_2C\underline{H}_2(CH_2)_2C\underline{H}_2CH_2N$); 3.65 (t, 2 H, $ClCH_2$, J = 6.4 Hz); 4.23 (t, 2 H, CH_2N, J = 7.4 Hz); 7.00 (dd, 1 H, H(8), J = 7.9 Hz, J = 7.5 Hz); 7.20 (d, 1 H, H(9), J = 7.9 Hz); 7.38—7.68 (m, 8 H, H(6), H(7), *m*-, *p*-H_{Ph}); 7.72—7.78 (m, 2 H, *o*-H, $C_{Ph}(1)$); 8.14 (d, 2 H, *o*-H, $C_{Ph}(3)$, J = 6.9 Hz). MS MALDI TOF, *m/z*: $[MH]^+$ 456. Found (%): C, 73.70; H, 5.71; N, 9.30; Cl, 7.71. $C_{28}H_{26}N_3OCl$. Calculated (%): C, 73.75; H, 5.75; N, 9.22; Cl, 7.78.

Reaction of 1,3-bis(4(5*H*)-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl)benzene (3) with dihaloalkanes. ***A.*** *Reaction of compound* **3** *with dichloroalkanes.* A mixture of compound **3** (3.0 g, 5.03 mmol), KOH (0.85 g, 15.2 mmol), and DMSO (150 mL) was heated to 100 °C, stirred for 15 min, and cooled to 50 °C, followed by addition of the corresponding dichloroalkane (12.0 mmol). The reaction mixture was stirred for 72 h at 50 °C, poured into water, the crystals formed were filtered off, washed with water, dried, and purified by column chromatography on silica gel (eluent CH_2Cl_2).

B. *Reaction of compound* **3** *with dibromoalkanes*. A mixture of compound **3** (2.5 g, 4.2 mmol), KOH (0.70 g, 12.5 mmol), 1,*n*-dibromoalkane (12 mmol), and dioxane (200 mL) was stirred for 48 h at 100 °C, cooled, the crystals were filtered off and washed with CH_2Cl_2 (3×80 mL). Dioxane and CH_2Cl_2 were evaporated *in vacuo* of a water-jet pump. The residual tar-like mass was dried *in vacuo* at 50 °C for 6 h, then purified by column chromatography (eluent CH_2Cl_2) on silica gel (60A, 0.060—0.200 mm).

1,3-Bis[5-(3-chloroprop-1-yl)-4-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl]benzene (11a). The yield of compound **11a** was 1.13 g (30%), white powder, m.p. 279—281 °C. IR, ν/cm^{-1}: 571, 648, 668, 692, 745, 754, 763, 781, 966, 1129, 1175, 1250, 1276, 1299, 1374, 1396, 1443, 1484, 1592, 1609, 1655. ^{1}H NMR, δ: 2.20—2.35 (m, 4 H, $CH_2C\underline{H}_2CH_2$); 3.72 (t, 4 H, CH_2Cl, J = 6.1 Hz); 4.41 (t, 4 H, CH_2N, J = 7.6 Hz); 6.99 (ddd, 2 H, H(8), quinoxaline, J = 8.4 Hz, J = 7.7 Hz, J = 1.3 Hz); 7.30—7.50 (m, 10 H, H(7), H(9), quinoxaline, *m*-, *p*-H_{Ph}); 7.54 (dd, 2 H, H(6), quinoxaline, J = 8.4 Hz, J = 1.0 Hz); 7.75 (dd, 1 H, H(5), J = 7.7 Hz, J = 7.6 Hz); 7.93 (dd, 2 H, H(4), H(6), J = 7.7 Hz, J = 1.7 Hz); 8.10—8.15 (m, 5 H, H(2), *o*-H_{Ph}). ^{13}C NMR, δ: 29.93, 39.72, 42.64, 115.56, 118.11, 118.46, 122.49, 122.67, 127.49, 127.88, 128.62, 129.53, 129.81, 129.86, 130.08, 130.20, 130.86, 131.01, 131.17, 132.63, 132.93, 143.51, 149.90, 155.48. MS MALDI TOF, *m/z*: $[MH]^+$: 749. Found (%): C, 70.37; H, 4.67; N, 11.28; Cl, 9.54. $C_{44}H_{34}N_6Cl_2O_2$. Calculated (%): C, 70.49; H, 4.57; N, 11.21, Cl, 9.46.

1,3-Bis[5-(4-bromobut-1-yl)-4-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl]benzene (11b). The yield of compound **11b** was 0.62 g (17%), white powder, m.p. 244—246 °C. IR, ν/cm^{-1}: 606, 667, 692, 745, 756, 783, 803, 920, 967, 988, 1126, 1234, 1250, 1300, 1360, 1396, 1445, 1486, 1609, 1649, 2856, 2926, 3027, 3061. ^{1}H NMR, δ: 1.90—2.07 (m, 8 H, $CH_2(C\underline{H}_2)_2CH_2$); 3.48 (t, 4 H, CH_2Br, J = 6.2 Hz); 4.30 (t, 4 H, CH_2N, J = 7.1 Hz); 6.98 (ddd, 2 H, H(8), quinoxaline, J = 8.4 Hz, J = 6.1 Hz, J = 2.4 Hz); 7.25—7.50 (m, 10 H, H(7), H(9), quinoxaline, *m*-, *p*-H_{Ph}); 7.53 (d, 2 H, H(6), quinoxaline, J = 7.9 Hz); 7.75 (dd, 1 H, H(5), J = 7.9 Hz, J = 7.6 Hz); 7.94 (dd, 2 H, H(4), H(6), J = 7.6 Hz, J = 1.7 Hz); 8.00—8.10 (m, 5 H, H(2), *o*-H_{Ph}). ^{13}C NMR, δ: 25.85, 29.85, 33.09, 40.66, 115.71, 118.15, 118.45, 122.38, 122.71, 127.36, 128.58, 129.81, 129.89, 130.17, 130.86, 131.16, 132.71, 132.96, 143.43, 145.86, 155.38. MS ESI, *m/z*: $[MH]^+$: 865.2, 867.2, 869.2, [2M+H] 1729.2, 1731.4, 1732.3, 1735.3, 1736.3. Found (%): C, 63.67; H, 4.37; N, 9.60; Br, 18.39. $C_{46}H_{38}N_6Br_2O_2$. Calculated (%): C, 63.75; H, 4.42; N, 9.70, Br, 18.44.

1,3-Bis[5-(5-chloro-3-oxapent-1-yl)-4-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl]benzene (11c). The yield of compound **11c** was 0.73 g (18%), white powder, m.p. 195—197 °C. IR, ν/cm^{-1}: 464, 695, 750, 921, 968, 1006, 1057, 1123, 1259, 1301, 1334, 1398, 1484, 1593, 1611, 1655. ^{1}H NMR, δ: 3.57 (t, 4 H, CH_2Cl, J = 5.4 Hz); 3.73 (t, 4 H, $OC\underline{H}_2CH_2Cl$, J = 5.4 Hz); 3.90 (t, 4 H, $OC\underline{H}_2CH_2N$, J = 5.4 Hz); 4.46 (t, 4 H, CH_2N, J = 5.4 Hz); 6.97 (dd, 2 H, H(8), quinoxaline, J = 8.3 Hz, J = 8.0 Hz); 7.33 (dd, 2 H, H(7), quinoxaline, J = 5.9 Hz, J = 7.6 Hz); 7.35 (dd, 2 H, *p*-H_{Ph}, J = 6.9 Hz, J = 6.9 Hz); 7.46 (dd, 4 H, *m*-H_{Ph}, J = 7.6 Hz, J = 7.3 Hz); 7.51 (d, 2 H, H(9), quinoxaline, J = 8.2 Hz); 7.58 (d, 2 H, H(6), quinoxaline, J = 8.2 Hz); 7.74 (dd, 1 H, H(5), J = 7.9 Hz, J = 7.6 Hz); 7.93 (d, 2 H, H(4), H(6), J = 7.6 Hz); 8.13 (s, 1 H, H(2)); 8.14 (d, 4 H, *o*-H_{Ph}, J = 7.3 Hz). ^{13}C NMR, δ: 41.99, 42.81, 68.51, 71.16, 116.80, 118.09, 118.18, 122.45, 122.48, 127.13, 127.83, 128.54, 129.74, 129.82, 130.82, 131.10, 132.65, 132.88, 143.46, 145.82, 155.65. MS MALDI TOF, *m/z*: $[MH]^+$: 809. Found (%): C, 68.07; H 4.70; N, 10.50; Cl, 8.90. $C_{46}H_{38}N_6Cl_2O_4$. Calculated (%): C, 68.23; H, 4.73; N, 10.38, Cl, 8.76.

1,3-Bis[5-(6-bromohexyl)-4-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl]benzene (11d). The yield of compound **11d** was 0.58 g (15%), white powder, m.p. 180—182 °C. IR, ν/cm^{-1}: 696, 728, 748, 782, 804, 845, 881, 920, 968, 1073, 1121, 1178, 1260, 1301, 1396, 1483, 1593, 1610, 1657. ^{1}H NMR, δ: 1.38—1.50 (m, 8 H, $Br(CH_2)_2C\underline{H}_2$); 1.65—1.75 (m, 4 H, $BrCH_2C\underline{H}_2$); 1.75—1.85 (m, 4 H, $NCH_2C\underline{H}_2$); 3.53 (t, 4 H, CH_2Br, J = 6.7 Hz); 4.22 (t, 4 H, CH_2N, J = 7.1 Hz); 7.07 (ddd, 2 H, H(8), quinoxaline, J = 7.9 Hz, J = 7.6 Hz); 7.37—7.50 (m, 10 H, H(7), H(9), quinoxaline, *m*-, *p*-H_{Ph}); 7.58 (d, 2 H, H(6), quinoxaline, J = 8.6 Hz); 7.88 (dd, 1 H, H(5), J = 7.6 Hz, J = 7.6 Hz); 8.02 (d, 2 H, H(4), H(6), J = 8.0 Hz); 8.10 (s, 1 H, H(2)); 8.15 (dd, 4 H, *o*-H_{Ph}, J = 7.3 Hz). Found (%): C, 65.07; H, 5.17; N, 9.20; Br, 17.20. $C_{50}H_{46}N_6Br_2O_2$. Calculated (%): C, 65.08; H, 5.02; N, 9.11, Br, 17.32.

1,3-Bis[5-(3-isothioureidoalkyl)-4-oxo-3-phenylimidazo-[1,5-*a*]quinoxalin-1-yl]benzene hydrohalides 12. A solution of

compound **11** (0.5 mmol) and thiourea (1.2 mmol) in dioxane (50 mL) was stirred for 20—120 h (20 h for **11b,d**, 70 h for **11a**, 120 h for **11c**) at 100 °C. The crystals formed were filtered off, washed with dioxane, and dried.

1,3-Bis[5-(3-isothioureidopropyl)-4-oxo-3-phenylimidazo-[1,5-*a*]quinoxalin-1-yl]benzene hydrochloride (12a). The yield of compound **12a** was 0.4 g (90%), white powder, m.p. 244—245 °C. IR, ν/cm^{-1}: 669, 693, 746, 783, 804, 1129, 1175, 1260, 1392, 1442, 1483, 1592, 1611, 1646, 2600—3500. ^{1}H NMR, δ: 1.95—2.41 (m, 4 H, $CH_2C\underline{H}_2CH_2$); 3.35 (t, 4 H, CH_2S, J = = 7.29 Hz); 4.36 (br.s, 4 H, CH_2N); 7.09 (dd, 2 H, H(8), quinoxaline, J = 7.9 Hz, J = 7.8 Hz); 7.30—7.50 (m, 10 H, H(7), H(9), quinoxaline, *m*-, *p*-H_{Ph}); 7.70 (d, 2 H, H(6), quinoxaline, J = 8.6 Hz); 7.78 (dd, 1 H, H(5), J = 7.7 Hz, J = 7.3 Hz); 8.01 (d, 2 H, H(4), H(6), J = 8.0 Hz, J = 1.3 Hz); 8.06 (s, 1 H, H(2)); 8.14 (dd, 4 H, *o*-H_{Ph}, J = 7.5 Hz, J = 1.5 Hz); 9.30 (br.s, 8 H, $SC(NH)NH_2 \cdot HCl$). ^{13}C{^{1}H} NMR, δ: 27.03, 27.41, 116.46, 117.69, 118.00, 122.29, 127.35, 127.61, 128.18, 129.63, 129.82, 130.55, 131.10, 132.70, 132.81, 143.08, 143.73, 154.86, 169.77. MS ESI, *m/z*: $[M - 2\,HCl + H]^+$ 829, $[M - 2\,HCl + Na]^+$ 851. Found (%): C, 61.01; H, 4.80; N, 15.34; S, 7.15; Cl, 7.71. $C_{46}H_{44}N_{10}O_2S_2Cl_2$. Calculated (%): C, 61.12; H, 4.91; N, 15.50; S, 7.09; Cl, 7.84.

1,3-Bis[5-(4-isothioureidobutyl)-4-oxo-3-phenylimidazo-[1,5-*a*]quinoxalin-1-yl]benzene hydrobromide (12b). The yield of compound **12b** was 0.34 g (66%), white powder, m.p. 260—262 °C. IR, ν/cm^{-1}: 678, 693, 701, 746, 757, 784, 803, 1127, 1260, 1301, 1322, 1399, 1443, 1484, 1591, 1611, 1639, 2700—3500. ^{1}H NMR, δ: 1.77 (br.s, 8 H, $CH_2(C\underline{H}_2)_2CH_2$); 3.23 (br.s, 4 H, CH_2S); 4.26 (br.s, 4 H, CH_2N); 7.10 (dd, 2 H, H(8), quinoxaline, J = 8.0 Hz, J = 7.9 Hz); 7.30—7.50 (m, 10 H, H(7), H(9), quinoxaline, *m*-, *p*-H_{Ph}); 7.63 (d, 2 H, H(6), quinoxaline, J = 8.6 Hz); 7.88 (dd, 1 H, H(5), J = 7.7 Hz, J = 7.2 Hz); 8.03 (dd, 2 H, H(4), H(6), J = 7.4 Hz, J = 1.5 Hz); 8.11 (s, 1 H, H(2)); 8.14 (dd, 4 H, *o*-H_{Ph}, J = 8.1 Hz, J = 1.5 Hz); 9.00 (br.s, 8 H, $SC(NH)NH_2 \cdot HCl$). ^{13}C{^{1}H} NMR, δ: 25.78, 26.03, 29.83, 116.61, 117.78, 118.01, 122.34, 127.47, 127.73, 128.30, 129.73, 129.88, 130.12, 130.71, 131.24, 132.80, 132.92, 143.22, 143.79, 154.79, 169.79. MS ESI, *m/z*: $[M - 2\,HBr + H]^+$ 857.4. Found (%): C, 56.38; H, 4.70; N, 13.64; S, 6.35; Cl, 15.72. $C_{48}H_{48}N_{10}O_2S_2Br_2$. Calculated (%): C, 56.47; H, 4.74; N, 13.72; S, 6.28; Br, 15.65.

1,3-Bis[5-(5-isothioureido-3-oxapentyl)-4-oxo-3-phenyl-imidazo[1,5-*a*]quinoxalin-1-yl]benzene hydrochloride (12c). The yield of compound **12c** was 24 mg (5%), white powder, m.p. 203—205 (decomp.) °C. IR, ν/cm^{-1}: 669, 690, 747, 814, 966, 1055, 1110, 1178, 1300, 1396, 1444, 1484, 1499, 1609, 1648, 2660—3500. ^{1}H NMR, δ: 3.36 (t, 4 H, CH_2S, J = 5.6 Hz); 3.73 (t, 4 H, $OC\underline{H}_2CH_2S$, J = 5.6 Hz); 3.81 (t, 4 H, $OC\underline{H}_2CH_2N$, J = 5.9 Hz); 4.44 (t, 4 H, CH_2N, J = 5.9 Hz); 7.11 (ddd, 2 H, H(8), quinoxaline, J = 8.3 Hz, J = 8.0 Hz); 7.35—7.50 (m, 10 H, H(7), H(9), quinoxaline, *m*-, *p*-H_{Ph}); 7.67 (d, 2 H, H(6), quinoxaline, J = 8.7 Hz); 7.89 (dd, 1 H, H(5), J = 7.7 Hz, J = 7.3 Hz); 8.03 (dd, 2 H, H(4), H(6), J = 7.6 Hz, J = 1.5 Hz); 8.11 (s, 1 H, H(2)); 8.15 (dd, 4 H, *o*-H_{Ph}, J = 8.3 Hz, J = 1.5 Hz); 9.12 (br.s, 8 H, $SC(NH)NH_2 \cdot HCl$). ^{13}C{^{1}H} NMR, δ: 30.62, 67.38, 68.48, 116.95, 117.66, 117.98, 122.21, 122.45, 127.42, 127.74, 128.33, 129.72, 130.27, 131.26, 132.75, 132.86, 143.29, 143.98, 154.95, 170.19. MS ESI, *m/z*: $[M-2HCl+H]^+$ 889.2. Found (%): C, 59.68; H, 5.20; N, 14.64; S, 6.55; Cl, 7.32. $C_{48}H_{48}N_{10}O_4S_2Cl_2$. Calculated (%): C, 59.81; H, 5.02; N, 14.53; S, 6.65; Cl, 7.36.

1,3-Bis[5-(6-isothioureidohexyl)-4-oxo-3-phenylimidazo-[1,5-*a*]quinoxalin-1-yl]benzene hydrobromide (12d). The yield of compound **12d** was 0.46 g (86%), white powder, m.p. 241—243 °C. IR, ν/cm^{-1}: 696, 727, 758, 920, 968, 1070, 1126, 1178, 1255, 1301, 1335, 1397, 1551, 1611, 1649, 2700—3500. ^{1}H NMR, δ: 1.46 (br.s, 8 H, $(CH_2)_2(C\underline{H}_2)_2(CH_2)_2$); 1.60—1.80 (m, 4 H, $CH_2C\underline{H}_2(CH_2)_2C\underline{H}_2CH_2$); 3.16 (t, 4 H, CH_2S, J = 7.1 Hz); 4.23 (t, 4 H, CH_2N, J = 6.8 Hz); 7.10 (dd, 2 H, H(8), quinoxaline, J = 8.2 Hz, J = 7.6 Hz); 7.35—7.52 (m, 10 H, H(7), H(9), quinoxaline, *m*-, *p*-H_{Ph}); 7.60 (d, 2 H, H(6), quinoxaline, J = 8.6 Hz); 7.89 (dd, 1 H, H(5), J = 7.9 Hz, J = 7.60 Hz); 8.03 (dd, 2 H, H(4), H(6), J = 7.9 Hz, J = 1.6 Hz); 8.11 (s, 1 H, H(2)); 8.15 (dd, 4 H, *o*-H_{Ph}, J = 8.0 Hz, J = 1.0 Hz); 8.96 (br.s, 8 H, $SC(NH)NH_2 \cdot HBr$). Found (%): C, 57.88; H, 5.21; N, 13.14; S, 5.88; Br, 14.82. $C_{52}H_{56}N_{10}O_2S_2Br_2$. Calculated (%): C, 57.99; H, 5.24; N, 13.01; S, 5.95; Br, 14.84.

1,3-Bis[5-(3-acetylthioalkyl)-4-oxo-3-phenylimidazo[1,5-*a*]-quinoxalin-1-yl]benzenes 13. A mixture of compound **11** (30 mmol) and KSC(O)Me (96 mmol) in dioxane (30 mL) was stirred for 40 h at 100 °C. Dioxane was partially evaporated *in vacuo* of a water-jet pump, CH_2Cl_2 (30 mL) was added to the residue, the mixture was washed with water (3×10 mL). The solvent was evaporated. Compound **13** thus obtained was used in further reactions without purification.

1,3-Bis[5-(3-acetylthiopropyl)-4-oxo-3-phenylimidazo[1,5-*a*]-quinoxalin-1-yl]benzene (13a). The yield of compound **13a** was 0.21 g (85%), yellow powder, m.p. 202—204 °C. IR, ν/cm^{-1}: 629, 692, 698, 746, 754, 782, 965, 1048, 1116, 1246, 1299, 1334, 1356, 1396, 1442, 1483, 1591, 1608, 1654, 1686. ^{1}H NMR, δ: 2.00—2.15 (m, 4 H, $CH_2C\underline{H}_2CH_2$); 2.34 (s, 6 H, CH_3); 3.01 (t, 4 H, CH_2S, J = 7.2 Hz); 4.29 (t, 4 H, CH_2N, J = 7.6 Hz); 6.78 (ddd, 2 H, H(8), quinoxaline, J = 8.3 Hz, J = 6.3 Hz, J = 1.9 Hz); 7.25—7.50 (m, 10 H, H(7), H(9), quinoxaline, *m*-, *p*-H_{Ph}); 7.53 (d, 2 H, H(6), quinoxaline, J = 8.1 Hz); 7.74 (dd, 1 H, H(5), J = 7.9 Hz, J = 7.6 Hz); 7.93 (dd, 2 H, H(4), H(6), J = 7.6 Hz, J = 1.68 Hz); 8.10—8.15 (m, 5 H, H(2), *o*-H_{Ph}).

1,3-Bis[5-(5-acetylthio-3-oxapentyl)-4-oxo-3-phenylimid-azo[1,5-*a*]quinoxalin-1-yl]benzene (13b). The yield of compound **13b** was 0.19 g (78%), yellow powder, m.p. 88—90 °C. IR, ν/cm^{-1}: 623, 747, 781, 966, 1099, 1254, 1297, 1361, 1386, 1442, 1484, 1590, 1610, 1653, 1687. ^{1}H NMR, δ: 2.02 (s, 6 H, CH_3); 3.03 (t, 4 H, CH_2S, J = 6.2 Hz); 3.60 (t, 4 H, $OC\underline{H}_2CH_2S$, J = 6.2 Hz); 3.86 (t, 4 H, $OC\underline{H}_2CH_2N$, J = 5.9 Hz); 4.43 (t, 4 H, CH_2N, J = 5.9 Hz); 6.97 (ddd, 2 H, H(8), quinoxaline, J = 7.9 Hz, J = 7.4 Hz, J = 0.8 Hz); 7.30—7.52 (m, 10 H, H(7), H(9), quinoxaline, *m*-, *p*-H_{Ph}); 7.56 (dd, 2 H, H(6), quinoxaline, J = 8.2 Hz, J = 0.6 Hz); 7.74 (dd, 1 H, H(5), J = 8.0 Hz, J = 7.5 Hz); 7.93 (dd, 2 H, H(4), H(6), J = 7.7 Hz, J = 1.5 Hz); 8.10—8.15 (m, 5 H, H(2), *o*-H_{Ph}).

1,3-Bis[5-(3-mercaptoalkyl)-4-oxo-3-phenylimidazo[1,5-*a*]-quinoxalin-1-yl]benzenes 14. ***A.*** A mixture of compound **12** (0.62 mmol), KOH (1.5 mmol), and water (50 mL) was stirred for 3 h at 100 °C and cooled. Crystals were filtered off, washed with water, dried, and purified by column chromatography on silica gel (eluent CH_2Cl_2 : MeOH = 500 : 1).

B. A solution of compound **13** (0.14 mmol) in THF (10 mL) and conc. HCl (0.5 mL) was stirred for 20 h at 55 °C, cooled, neutralized with aqueous KOH, extracted with CH_2Cl_2 (3×10 mL), and washed with water. The solvent was evaporated *in vacuo* of a water-jet pump, the residue was purified by column

chromatography (eluent: CH_2Cl_2 : MeOH = 500 : 1) on silica gel (60A, 0.060—0.200 mm).

1,3-Bis[5-(3-mercaptopropyl)-4-oxo-3-phenylimidazo[1,5-*a*]-quinoxalin-1-yl]benzene (14a). The yield of compound **14a** was 0.18 g (40%) (procedure *A*), 50 mg (48%) (procedure *B*), white powder, m.p. 263—265 °C. IR, ν/cm^{-1}: 604, 668, 693, 711, 747, 763, 783, 804, 921, 966, 1128, 1175, 1260, 1299, 1396, 1442, 1483, 1592, 1609, 1649, 2542. ^{1}H NMR, δ: 1.59 (t, 2 H, SH, J = 8.1 Hz); 2.00—2.15 (m, 4 H, $CH_2C\underline{H}_2CH_2$); 2.62 (dt, 4 H, CH_2S, J = 8.1 Hz, J = 6.9 Hz); 4.37 (t, 4 H, CH_2N, J = 7.5 Hz); 6.98 (ddd, 2 H, H(8), quinoxaline, J = 8.4 Hz, J = 7.5 Hz, J = 1.4 Hz); 7.32—7.49 (m, 10 H, H(7), H(9), quinoxaline, *m*-, *p*-H_{Ph}); 7.54 (dd, 2 H, H(6), quinoxaline, J = 8.3 Hz, J = 1.1 Hz); 7.75 (dd, 1 H, H(5), J = 7.9 Hz, J = 7.6 Hz); 7.93 (dd, 2 H, H(4), H(6), J = 7.7 Hz, J = 1.7 Hz); 8.10—8.15 (m, 5 H, H(2), *o*-H_{Ph}). MS ESI, *m/z*: $[MH]^+$ 745.2. Found (%): C, 70.88; H, 4.71; N, 11.30; S, 8.65. $C_{44}H_{36}N_6O_2S_2$. Calculated (%): C, 70.94; H, 4.87; N, 11.28; S, 8.61.

1,3-Bis[5-(4-mercaptobut-1-yl)-4-oxo-3-phenylimidazo-[1,5-*a*]quinoxalin-1-yl]benzene (14b). Procedure *A* yielded 0.19 g (39%) of compound **14b** as white powder, m.p. 245—247 °C. IR, ν/cm^{-1}: 603, 668, 692, 745, 755, 782, 803, 967, 990, 1129, 1176, 1259, 1300, 1359, 1396, 1443, 1484, 1591, 1609, 1649, 2561, 2849, 2919, 3065. ^{1}H NMR, δ: 1.38 (t, 2 H, SH, J = 7.9 Hz); 1.72—1.97 (m, 8 H, $CH_2(C\underline{H}_2)_2CH_2$); 2.61 (dt, 4 H, CH_2S, J = 7.8 Hz, J = 6.9 Hz); 4.26 (t, 4 H, CH_2N, J = 7.5 Hz); 6.98 (ddd, 2 H, H(8), quinoxaline, J = 8.5 Hz, J = 6.6 Hz, J = 2.2 Hz); 7.25—7.50 (m, 10 H, H(7), H(9), quinoxaline, *m*-, *p*-H_{Ph}); 7.55 (dd, 2 H, H(6), quinoxaline, J = 8.6 Hz, J = 1.0 Hz); 7.68 (dd, 1 H, H(5), J = 8.0 Hz, J = 7.5 Hz); 7.86 (dd, 2 H, H(4), H(6), J = 7.6 Hz, J = 1.7 Hz); 8.00—8.10 (m, 5 H, H(2), *o*-H_{Ph}). MS ESI, *m/z*: $[MH]^+$ 773.3, $[M+Na]^+$ 795.3, $[M+K]^+$ 811.3. Found (%): C, 71.71; H, 5.11; N, 10.70; S, 8.35. $C_{46}H_{40}N_6O_2S_2$. Calculated (%): C, 71.48; H, 5.22; N, 10.87; S, 8.30.

1,3-Bis[5-(5-mercapto-3-oxapentyl)-4-oxo-3-phenylimidazo[1,5-*a*]quinoxalin-1-yl]benzene (14c). Procedure *B* yielded 55 mg (49%) of compound **14c** as white powder, m.p. 103—105 °C. IR, ν/cm^{-1}: 667, 692, 745, 781, 863, 967, 1054, 1100, 1178, 1254, 1297, 1362, 1385, 1442, 1484, 1590, 1609, 1652, 2566, 2855, 2922. ^{1}H NMR, δ: 1.45 (t, 2 H, SH, J = 8.1 Hz); 2.63 (dt, 4 H, CH_2S, J = 8.1 Hz, J = 6.2 Hz); 3.67 (t, 4 H, $OC\underline{H}_2CH_2S$, J = = 6.2 Hz); 3.86 (t, 4 H, $OC\underline{H}_2CH_2N$, J = 5.7 Hz); 4.46 (t, 4 H, CH_2N, J = 5.7 Hz); 6.98 (dd, 2 H, H(8), quinoxaline, J = 7.9 Hz, J = 7.4 Hz); 7.32—7.58 (m, 10 H, H(7), H(9), quinoxaline, *m*-, *p*-H_{Ph}); 7.59 (d, 2 H, H(6), quinoxaline, J = 8.5 Hz); 7.75 (dd, 1 H, H(5), J = 7.9 Hz, J = 7.4 Hz); 7.93 (dd, 2 H, H(4), H(6), J = 7.7 Hz, J = 0.9 Hz); 8.10—8.17 (m, 5 H, H(2), *o*-H_{Ph}). MS ESI, *m/z*: $[MH]^+$ 805.3. Found (%): C, 68.78; H, 5.99; N, 10.33; S, 7.87. $C_{46}H_{40}N_6O_4S_2$. Calculated (%): C, 68.64; H, 5.01; N, 10.44; S, 7.97.

1,3-Bis[5-(6-mercaptohexyl)-4-oxo-3-phenylimidazo[1,5-*a*]-quinoxalin-1-yl]benzene (14d). Procedure *A* yielded 0.16 g (32%) of compound **14d** as white powder, m.p. 108—110 °C. IR, ν/cm^{-1}: 667, 693, 712, 749, 1073, 1122, 1174, 1246, 1296, 1385, 1441, 1484, 1609, 1656, 2852, 2924. ^{1}H NMR, δ: 1.32 (t, 2 H, SH, J = 7.7 Hz); 1.45—1.50 (m, 8 H, $(CH_2)_2(C\underline{H}_2)_2(CH_2)_2$); 1.60—1.68 (m, 4 H, $SCH_2C\underline{H}_2CH_2(CH_2)_3$); 1.75—1.83 (m, 4 H, $NCH_2C\underline{H}_2CH_2(CH_2)_3$); 2.52 (dt, 4 H, CH_2S, J = 7.7 Hz, J = 7.0 Hz); 4.23 (t, 4 H, CH_2N, J = 7.9 Hz); 6.98 (ddd, 2 H, H(8), quinoxaline, J = 8.1 Hz, J = 7.1 Hz, J = 1.1 Hz); 7.27—7.49 (m, 10 H, H(7), H(9), quinoxaline, *m*-, *p*-H_{Ph}); 7.55 (dd, 2 H, H(6), quinoxaline, J = 8.4 Hz, J = 1.1 Hz); 7.76 (dd, 1 H, H(5), J = 7.7 Hz, J = 7.7 Hz); 7.94 (dd, 2 H, H(4), H(6), J = 7.7 Hz, J = 1.8 Hz); 8.11—8.18 (m, 5 H, H(2), *o*-H_{Ph}). Found (%): C, 72.34; H, 5.80; N, 10.10; S, 7.65. $C_{50}H_{48}N_6O_2S_2$. Calculated (%): C, 72.43; H, 5.84; N, 10.14; S, 7.73.

1^3,3^3-Diphenyl-1,3(1,5)-diimidazo[1,5-*a*]quinoxalina-2(1,3)-benzadithiacycloalkaphane-1^4,3^4-diones 15. A solution of bis-mercaptane **14** (0.12 mmol) and a solution of iodine (15 mg, 0.06 mmol) in dichloromethane (50 mL) were added over 1 h from two dropping funnels into a flask with dichloromethane (100 mL) with stirring, the mixture was stirred for 4 h, followed by addition of 5% aq. sodium thiosulfate (50 mL) and 5% aq. $NaHCO_3$ (50 mL). The reaction mixture was stirred, the organic layer was separated, washed with water, dried with sodium sulfate, and filtered. The solvent was evaporated and the residue was purified by column chromatography (eluent CH_2Cl_2 : EtOH = 300 : 1) on silica gel (60A, 0.060—0.200 mm).

1^3,3^3-Diphenyl-1,3(1,5)-diimidazo[1,5-*a*]quinoxalina-2(1,3)-benza-7,8-dithiacycloundecaphane-1^4,3^4-dione (15a). The yield of compound **15a** was 7 mg (8%), white powder, m.p. 312—314 °C. ^{1}H NMR, δ: 2.20—2.45 (m, 8 H, SCH_2CH_2); 3.78 (ddd, 2 H (1 H, NCH_2), J = 15.0 Hz, J = 4.1 Hz, J = 3.5 Hz); 5.21 (ddd, 2 H (1 H, NCH_2), J = 15.0 Hz, J = 10.8 Hz, J = 3.7 Hz); 6.93 (dd, 2 H, H(8), quinoxaline, J = 7.9 Hz, J = 7.4 Hz, J = 1.0 Hz); 7.07 (d, 2 H, H(9), quinoxaline, J = 8.0 Hz); 7.20 (s, 1 H, H_{benz}(2)); 7.26—7.30 (m, 4 H, H(6), H(7), quinoxaline); 7.43 (dd, 2 H, *p*-H_{Ph}, J = 7.6 Hz, J = 7.3 Hz); 7.50 (dd, 2 H, *m*-H_{Ph}, J = 7.6 Hz, J = 7.3 Hz); 8.00 (dd, 1 H, H_{benz}(5), J = 7.9 Hz, J = 7.6 Hz); 8.19 (d, 4 H, *o*-H_{Ph}, J = 7.3 Hz); 8.34 (d, 2 H, H_{benz}(4), H_{benz}(6), J = 7.6 Hz). MS ESI, *m/z*: $[MH]^+$ 743.6, $[2M+H]^+$ 1485.4. MS EI, *m/z* (I_{rel}): 744(17), 743(38), 742(69) M^+, 711(23), 710(45), 709(28), 678(18), 677(41), 676(27), 650(15), 649(11), 637(13), 636(11), 635(13), 623 (19), 622(13), 621(15), 596(19), 436(21), 403(15), 389(11), 377(10), 376(22), 375(23), 363(13), 362(14), 361(10), 335(20), 324(12), 318(13), 292(10), 291(23), 252(16), 235(28), 233(29), 219(78), 218(26), 206(23), 205(88), 170(16), 128(14), 118(10), 104(44), 103(15), 90(49), 89(100), 76(18), 72(42). No peaks for masses of fragment ions with relative intensities less than 10% are given.

1^3,3^3-Diphenyl-1,3(1,5)-diimidazo[1,5-*a*]quinoxalina-2(1,3)-benza-8,9-dithiacyclotridecaphane-1^4,3^4-dione (15b). The yield of compound **15b** was 28 mg (30%), white powder, m.p. 302—304 °C (DMSO). IR, ν/cm^{-1}: 694, 750, 993, 1051, 1074, 1122, 1172, 1239, 1296, 1335, 1395, 1444, 1485, 1600, 1657, 2854, 2926. ^{1}H NMR, δ: 1.35—1.48 (m, 2 H); 1.50—1.75 (m, 4 H); 1.85—2.00 (m, 2 H); 2.45—2.53 (m, 2 H (1 H, SCH_2); 2.35—2.44 (m, 2 H (1 H, SCH_2); 3.70 (ddd, 2 H (1 H, NCH_2), J = 14.4 Hz, J = 4.4 Hz, J = 4.4 Hz); 5.17 (ddd, 2 H (1 H, NCH_2), J = 14.4 Hz, J = 10.4 Hz, J = 4.0 Hz); 7.02 (dd, 2 H, H(8), quinoxaline, J = 8.6 Hz, J = 5.1 Hz, J = 3.3 Hz); 7.23 (d, 2 H, H(9), quinoxaline, J = 8.04 Hz); 7.31—7.35 (m, 4 H, H(6), H(7), quinoxaline); 7.39 (s, 1 H, H_{benz}(2)); 7.44 (dd, 2 H, *p*-H_{Ph}, J = 7.3 Hz, J = 7.3 Hz); 7.51 (dd, 2 H, *m*-H_{Ph}, J = 7.7 Hz, J = 7.0 Hz); 8.03 (dd, 1 H, H_{benz}(5), J = 7.7 Hz, J = 7.7 Hz); 8.20 (dd, 4 H, *o*-H_{Ph}, J = 7.7 Hz, J = 1.3 Hz); 8.27 (dd, 2 H, H_{benz}(4), H_{benz}(6), J = 7.9 Hz, J = 1.3 Hz). MS EI, *m/z* (I_{rel}): 771(4) 770(7) M^+, 202(28), 198(16), 188(17), 184(17), 174(16), 170(34), 156(22), 146(30), 132(27), 131(39), 118(33), 117(63), 104(39), 103(24), 91(64), 90(51), 89(79), 86(100), 78(39). No peaks for masses of fragment ions with relative intensities less than 10% are given. Found (%): C, 71.64; H, 4.80; N, 10.81; S, 8.25. $C_{46}H_{38}N_6O_2S_2$. Calculated (%): C, 71.66; H, 4.97; N, 10.90; S, 8.32.

1^3,3^3-Diphenyl-1,3(1,5)-diimidazo[1,5-*a*]quinoxalina-2(1,3)-benza-10,11-dithiacycloheptadecaphane-1^4,3^4-dione (15c). The yield of compound **15c** was 45 mg (45%), white powder, m.p. 287—289 °C. IR, ν/cm^{-1}: 753, 782, 996, 1035, 1076, 1097, 1123, 1167, 1244, 1298, 1337, 1360, 1403, 1442, 1464, 1486, 1551, 1607, 1665, 2853, 2925. ^{1}H NMR, δ: 1.20—1.40 (m, 8 H); 1.40—1.60 (m, 4 H); 1.60—1.80 (m, 4 H); 2.40—2.60 (m, 4 H); 3.45—3.65 (m, 2 H (1 H, NCH_2); 4.95—5.15 (m, 2 H (1 H, NCH_2); 6.80—7.00 (m, 2 H, H(8), quinoxaline); 7.26—7.30 (m, 4 H, H(7), H(9), quinoxaline); 7.32 (d, 2 H, H(6), quinoxaline, J = 7.7 Hz); 7.40 (dd, 2 H, *p*-H_{Ph}, J = 7.3 Hz, J = 7.0 Hz); 7.47 (dd, 2 H, *m*-H_{Ph}, J = 7.7 Hz, J = 7.00 Hz); 7.64 (s, 1 H, H_{benz}(2)); 7.94 (dd, 1 H, H_{benz}(5), J = 8.1 Hz, J = 7.7 Hz); 8.11 (d, 4 H, *o*-H_{Ph}, J = 7.3 Hz); 8.15 (d, 2 H, H_{benz}(4), H_{benz}(6), J = 7.7 Hz). MS EI, m/z (I_{rel}): 827(3) 826(5) M^+, 794(12), 793(19), 362(19), 230(30), 216(25), 202(78), 188(43), 170(13), 132(14), 130(43), 104(37), 103(35), 90(35), 89(54), 86(22), 78(15), 77(100), 76(28), 65(27), 64(26), 63(25). No peaks for masses of fragment ions with relative intensities less than 10% are given. Found (%): C, 72.65; H, 5.55; N, 10.11; S, 7.66. $C_{50}H_{46}N_6O_2S_2$. Calculated (%): C, 72.61; H, 5.61; N, 10.16; S, 7.75.

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