# Synthesis of Aryl(hetero)methylene-1,3-indandione Based Molecular Glasses

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**Summary.** Novel glass-forming 2-(1-phenyl-1,2,3,4-tetrahydroquinoline-6-ylmethylene)-1,3-indandione derivatives were synthesized and their thermal properties were studied. The results of a preliminary investigation of the photoelectric properties of amorphous films of the title compounds are briefly reported. The ionization potential of these molecular glasses is *ca.* 5.6 eV and the hole drift mobility exceeds $10^{-8} \, \mathrm{cm^2 \, V^{-1} \, s^{-1}}$ at strong electric fields.

**Keywords.** 1,3-Indandione; 1,2,3,4-Tetrahydroquinoline; Molecular glasses; Ionization potential; Charge mobility.

## Introduction

Organic molecules containing conjugated charge-acceptor and charge-donor molecular fragments belong to a new class of polar organic crystals, which due to their significant photoconductivity and enhanced nonlinear optical properties are interesting for their possible applications in molecular optics and molecular electronics [1–3]. Amongst them, the aryl(hetero)methylene-1,3-indandiones constitute an interesting class of dipolar compounds exhibiting high hyperpolarizability, intramolecular charge transfer ability, and excellent photoconductivity, which are important for possible applications of these compounds in an active medium of optoelectronic devices (OED) [2, 4–6].

Low molecular aryl(hetero)methylene-1,3-indandiones are usually crystalline materials and may form uniform, transparent thin films by vapor deposition. They are not capable of forming thin neat homogenous layers by spin-coating methods, and must be used in combination with polymeric binders. Even in such compositions, the possibility of aryl(hetero)methylene-1,3-indandiones crystallization remains and causes problems during OED preparation and long application, because low-molecular-weight compounds possessing 1,3-indandione moieties generally tend to form crystals. It is of interest and significance to develop photo- and electroactive low-molecular-weight amorphous materials that form stable amorphous glasses having glass-transition temperatures above room temperature [7]. These molecular glasses will form stable films without polymeric host materials, and are expected to manifest properties and functions more effectively relative to molecularly doped polymer compositions. Molecular glasses are already used in technical applications and can be regarded as attractive candidates for some newly developing fields. They are used as charge-transporting and/or emitting materials in light-emitting diodes, electrophotographic materials, and photovoltaic devices [7–9]. As photoconductive or multifunctional materials they can be applied in photorefractive systems [10]. Molecular glasses are also candidates for the application as electrically conducting materials [11], pho-

tochromic materials [7], and resist materials for electron-beam lithography [12].

In previous contributions we have reported on the synthesis of a novel class of low-molar-mass compounds capable of existing in amorphous state. We prepare such compounds by the reactions of oxiranes containing electroactive groups with different difunctional compounds, such as aromatic diols, dimercapto compounds, and derivatives of anilines [13–15]. In the light of these findings we put our efforts to the synthesis of indandione moiety containing molecular glasses. To the best our knowledge, the synthesis route to the aryl(hetero)methylene-1,3-indandione based molecular glasses has never been reported.

In the present work, we describe the synthesis and characterization of 2-(1-phenyl-1,2,3,4-tetrahydroquinoline-6-ylmethylene)-1,3-indandione based molecular glasses. Increase of the molar mass usually leads to increasing stability of molecular glasses [16]. In addition, the molecules of these compounds consist of two 2-(1-phenyl-1,2,3,4-tetrahydroquinoline-6-ylmethylene)-1,3-indandione branches linked by a central flexible bridge. This circumstance should also enhance the inclination of the designed molecules to form glasses.

## Results and Discussion

The synthesis route to aryl(hetero)methylene-1,3-indandione derivatives **7a** and **7b** possessing 1-phenyl-1,2,3,4-tetrahydroquinoline moieties and two hydroxyl groups in the flexible bridge is shown in Scheme 1.

In general, heteroaromatic aldehydes react with 1,3-indandione to yield *Knoevenagel* or *Michael* type adducts [17]. We found that **1** and 1,3-indandione in ethanol afforded the condensation product 3-acetyl-6-(1,3-dioxoindan-2-ylmethylene)-1-phenyl-1,2,3,4-tetrahydroquinoline (**2**) at room temperature even in the absence of a catalyst. Then, the hydroxyl group was deprotected to give **3**. Unfortunately, the alkylation of **3** with epichlorohydrin was unsuccessful: here the formation of the considered epoxy compound **6** could never be observed, even if the reaction conditions (molar ratio, catalyst, temperature, and solvent) were systematically varied. Consequently, we had to reject the above-described synthesis route to 2-(1-phenyl-1,2,3,4-tetrahydroquinoline-6-ylmethylene)-1,3-indandione based molecular glasses and made our efforts to develop an alternative synthesis for the epoxy compound **6**. It
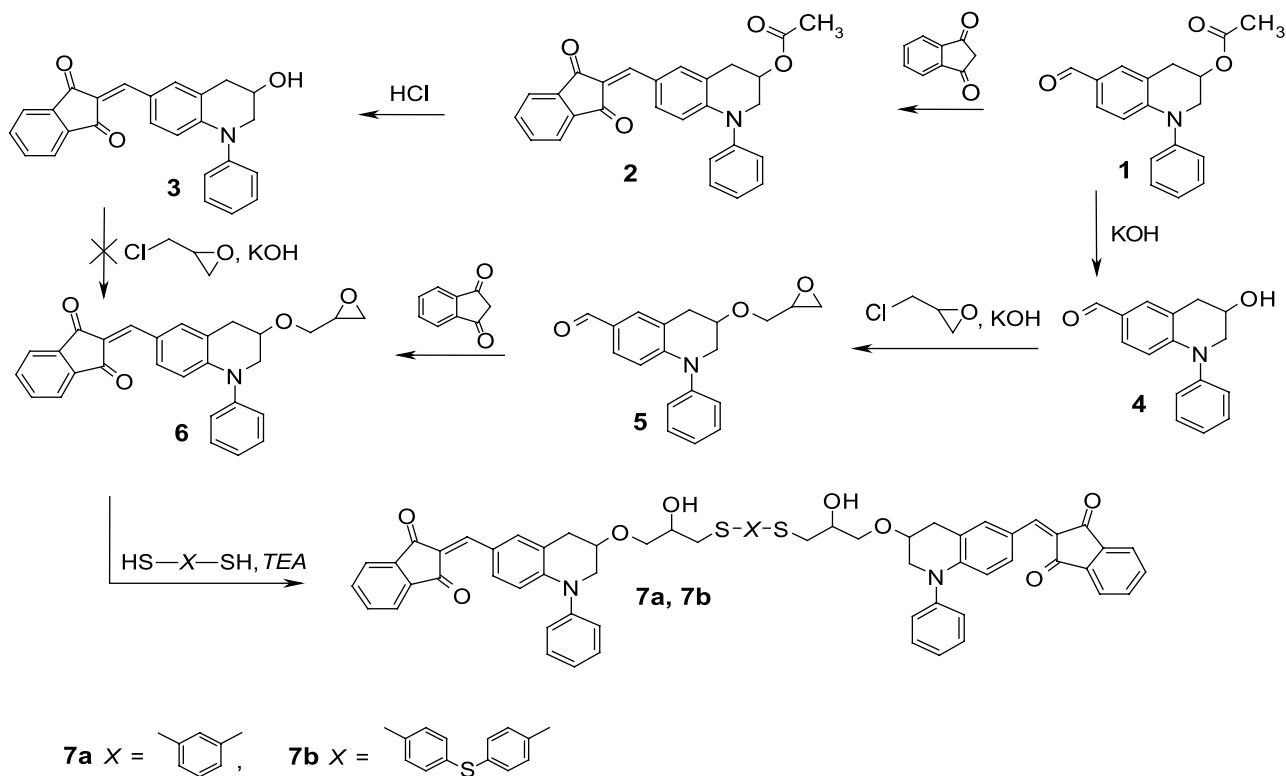


**Scheme 1**

proved to be successful when **4** containing deprotected hydroxyl was alkylated with epichlorohydrin to obtain 3-oxiranylmethoxy-1-phenyl-1,2,3,4-tetrahydroquinoline-6-carboxaldehyde (**5**), which in turn was transformed to the aryl(hetero)methylene-1,3-indandione epoxypropyl derivative **6**. Finally, **7a** and **7b** were prepared by the reaction of 1,3-benzenedithiol and 4,4′-thiobisbenzenethiol with two equivalents of the epoxypropyl derivative **6** in the presence of a catalytic amount of triethylamine (*TEA*).

The chemical structures of **7a** and **7b** were confirmed by IR, $^1$H NMR, UV, and APCI-MS spectroscopy. Thus, the mass spectrum and elemental analysis are consistent with the both desirable structures. In the $^1$H NMR spectra, the peaks of aromatic protons of both **7a** and **7b** were observed at $\delta = 8.48$–7.04 ppm, while aliphatic protons appear in the region of $\delta = 4.10$–2.86 ppm. Some individual downfield peaks were assigned to characteristic protons of heterocyclic or aromatic moieties: a singlet of a proton of the alkene group appeared in the region of $\delta = 8.5$ ppm, while the H-7 and H-8 protons of the heterocycle gave two doublets at $\delta = 8.0$ and 6.5 ppm with $J = 8.8$ Hz. A splitting of these signals indicates that isolated compounds **7a** and **7b**, containing several stereogenic centers, were obtained as mixtures of diastereomers, which could not be separated by purification procedures. The IR spectra of **7a** and **7b** are characterized by the occurrence of two carbonyl absorptions at 1712 (1714) cm$^{-1}$ and

1670 cm$^{-1}$. This is in accordance with reports in Ref. [17] assigning the two different signals to the symmetrical and asymmetrical stretching mode of the carbonyl groups. Moreover, hydroxyl groups participating in hydrogen bonding give rise to a broad oscillation at 3620–3200 cm$^{-1}$. Electron transitions to the higher energy states in **7a** and **7b** give three main absorption maximum at *ca.* 208, 248, and 486 nm (Fig. 1). Since **7a** and **7b** contain several isolated π-electron systems, the total light absorption spectrum is a sum of the spectra of the separate molecule fragments. The comparison of the UV/Vis spectra of compound **7a** with **7b** showed that **7b** gave an extra maximum at *ca.* 278 nm due to the presence of the central linking fragment, i.e. 4,4′-thiobisbenzenethiol.

The final compounds **7a** and **7b** were isolated by column chromatography, dissolved in toluene and precipitated with *n*-hexane. Obtained by such a procedure **7a** and **7b** were amorphous compounds. All our attempts to crystallize them were unsuccessful. X-Ray diffraction patterns of **7b** show only broad halos (Fig. 2). Such high morphological stability of the glasses **7a** and **7b** can apparently be explained by existence of several diastereomers, which could neither be separated nor characterized individually, the possibility of intermolecular hydrogen bonding and the flexibility of aliphatic linking chains.

All synthesized molecules are thermally stable; they decompose above 300°C. The formation of the glassy state in **7a** and **7b** was confirmed by differential scanning calorimetry (DSC). Table 1 lists
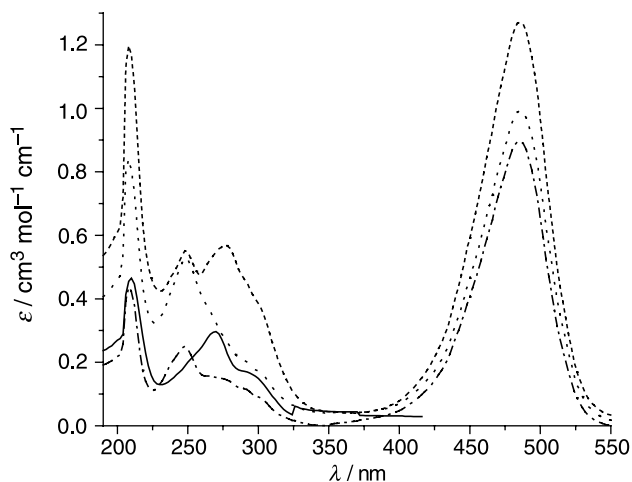


**Fig. 1.** Absorption spectra of dilute *THF* solutions ($10^{-4}\,M$) of *u/l*-**6** (— · —), *u/l*-**7a** ( · · · · · ), *u/l*-**7b** (– –), and 4,4′-thiobisbenzenethiol (——)
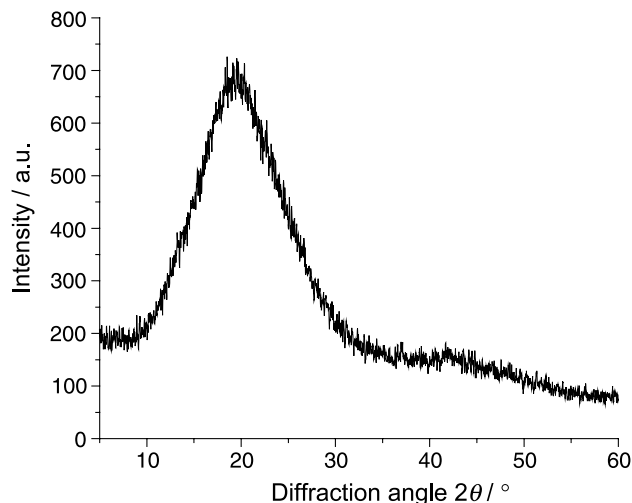


**Fig. 2.** X-Ray diffraction patterns of *u/l*-**7b**

**Table 1.** Characteristics of $u/l$-**7a** and $u/l$-**7b**

| Compound | $T_g/°C$ | $T_{dec-5\%}/°C$ | $I_p/eV$ |
|---|---|---|---|
| **7a** | 94 | 305 | 5.6 |
| **7b** | 113 | 300 | 5.62 |

glass transition temperatures ($T_g$) and 5% weight loss temperatures ($T_{dec-5\%}$) for the synthesized compounds **7a** and **7b**. These investigations revealed that both investigated 2-(1-phenyl-1,2,3,4-tetrahydroquinoline-6-ylmethylene)-1,3-indandione based molecular glasses were found only in the amorphous phase. No crystallization takes place at first heating of **7a**, only $T_g$ is revealed at 94°C during the second heating (Fig. 3). This is a common feature for both **7a** and **7b**. $T_g$ of **7b** is higher (113°C) than
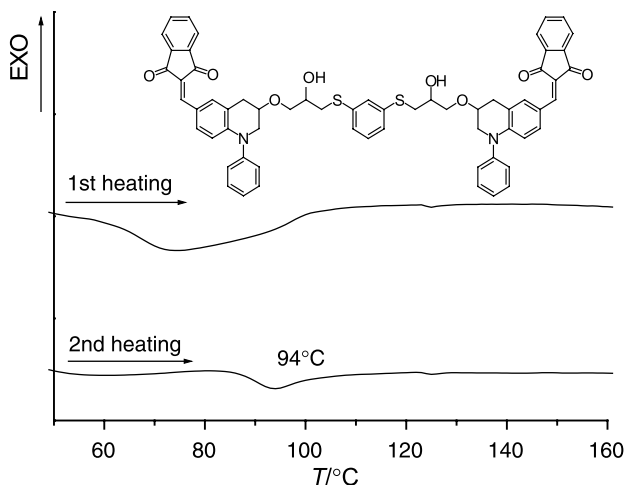


**Fig. 3.** DSC curves for $u/l$-**7a** (heating rate to 10 K/min)
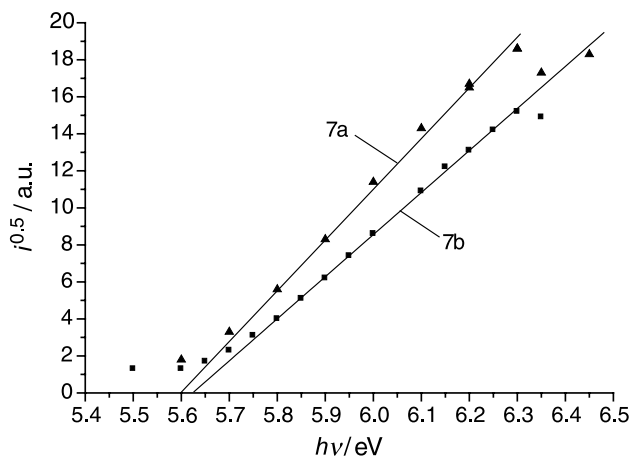


**Fig. 4.** Photoemission (in air) spectra of $u/l$-**7a** and $u/l$-**7b**

of **7a** due to the thiobisbenzene unit at the linking fragment.

The glass-forming aryl(hetero)methylene-1,3-indandiones **7a** and **7b** reported here are soluble in common organic solvents, such as acetone, chloroform, *THF*, *etc*. Transparent films on substrates were prepared by the casting technique. The photoelectron emissions in air spectra for the investigated molecular glasses are given in Fig. 4, and ionization potencial ($I_p$) values are presented in Table 1. Due to the similar $\pi$-electron conjugation, as mentioned earlier, there is a small difference of the ionization potential values between **7a** and **7b**.

In conclusion, two novel glass-forming aryl(hetero)methylene-1,3-indandione derivatives $u/l$-**7a** and $u/l$-**7b** were synthesized by the reaction of 1,3-benzenedithiol and 4,4′-thiobisbenzenethiol with 3-oxiranylmethoxy-6-(1,3-dioxoindan-2-ylmethylene)-1-phenyl-1,2,3,4-tetrahydroquinoline in the presence of triethylamine. The molecular structure of these 2-(1-phenyl-1,2,3,4-tetrahydroquinoline-6-ylmethylene)-1,3-indandione based molecular glasses prevents crystallization in the layer and allows stable films to be prepared even without polymeric binder. Preliminary charge mobility was measured by the time-of-flight technique. The hole drift mobility in these compounds exceeds $10^{-8} \, cm^2 \, V^{-1} \, s^{-1}$ at strong electric fields. The details of the photoelectric properties of these compounds will be reported elsewhere.

## Experimental

The $^1$H NMR spectra were taken on Varian Unity Inova (300 MHz) spectrometer in CDCl$_3$ or *DMSO*. The IR spectra were taken for samples in KBr pellets on a Perkin Elmer Spectrum BX II FT-IR System spectrometer. The UV spectra were recorded on a Spectronic Genesys 8 spectrometer in *THF* ($10^{-4} M$) in a microcell with an internal width of 1 mm. The course of the reaction products was monitored by TLC on Silufol UV-254 plates (eluent: acetone/$n$-hexane = 7/18) and development with I$_2$ or UV light. Silica gel (grade 62, 60–200 mesh, 150 Å, Aldrich) was used for column chromatography. Elemental analyses (C, H, and N) were conducted using the Elemental Analyser CE-44, their results were found to be in good agreement ($\pm$0.2%) with the calculated values. Melting points were determined in capillary tubes on capillary melting point apparatus MEL-TEMP. The ionization potential ($I_p$) was measured by the photoemission in air method, similar to the one used in Ref. [15] and described in Ref. [14]. The charge carrier mobilities were measured by the time of flight technique [16]. Thermal properties for **7a** and **7b** were examined by using a Netzsch STA 409 PC Luxx apparatus at a heating

rate of 10 K/min under nitrogen atmosphere. The glass transition temperatures ($T_g$) were determined from the second heating. The XRD analysis was performed using diffractometer DRON-UM2. Diffraction patterns were recorded at 30 kV and 20 mA in $1°\text{sec}^{-1}$ detector's movement speed, intensity was measured every $2\theta = 0.02°$. A flat diffracted beam pyrolitic graphite monochromator was used to remove fluorescent X-rays. Compound **1** was made according to Ref. [17].

### rac-3-Acetyl-6-(1,3-dioxoindan-2-ylmethylene)-1-phenyl-1,2,3,4-tetrahydroquinoline (2, C$_{27}$H$_{21}$NO$_4$)

Compound *rac*-**1** (20 g, 0.06 mol) and 9.5 g 1,3-indandione (0.06 mol) were stirred in 170 cm$^3$ ethanol at room temperature overnight. The crystalline product that had been formed was filtered off, washed with diethyl ether:*n*-hexane = 1:2 and recrystallized from toluene. Yield 20.6 g (75%); mp 237–239°C; IR (KBr): $\bar{\nu} = 3060$ (CH$_{\text{arom}}$), 2995, 2754 (CH$_{\text{aliph}}$), 1727 (OC=O, C=O), 1667 (C=O), 1613, 1594, 1567, 1464 (C=C, C–N), 937, 834, 779, 736, 704, 677 (CH=CH of mono-, 1,2-di- and 1,2,4-trisubstituted benzenes) cm$^{-1}$; $^1$H NMR (CDCl$_3$, 300 MHz): $\delta = 8.49$ (s, CH of methine), 8.09 (dd, $J_{7,8} = 8.8$, $J_{7,5} = 1.6$ Hz, H-7 of Ht), 7.94–7.21 (m, 10H, Ar), 6.57 (d, $J_{7,8} = 8.8$ Hz, H-8 of Ht), 5.45–5.37 (m, CH), 3.96–3.73 (m, NCH$_2$), 3.26 (dd, 1H, $J_{AB} = 16.5$, $J_{AX} = 4.3$ Hz, H$_A$ of C*H*$_2$CH), 3.18 (dd, 1H, $J_{BX} = 4.7$ Hz, H$_B$ of C*H*$_2$CH), 2.05 (s, CH$_3$) ppm; APCI-MS: $m/z$ (%) = 424 [M + H]$^+$ (100), 364 [M + H–COOCH$_3$]$^+$ (20).

### rac-3-Hydroxy-6-(1,3-dioxoindan-2-ylmethylene)-1-phenyl-1,2,3,4-tetrahydroquinoline (3, C$_{25}$H$_{19}$NO$_3$)

Compound *rac*-**2** (20.6 g, 0.05 mol) was dissolved in 40 cm$^3$ dioxane by heating. The solution was cooled to room temperature, and 40 cm$^3$ 37% hydrochloric acid were added. The reaction mixture was refluxed for 3.5 h. After termination the mixture was stored at room temperature overnight. The crystalline product was filtered off, washed with water until neutral, and and then with 2-propanol. The product was recrystallized from toluene. Yield 13.8 g (74%); mp 198–200°C; IR (KBr): $\bar{\nu} = 3412$ (OH), 3051 (CH$_{\text{arom}}$), 2891, 2641 (CH$_{\text{aliph}}$), 1714, 1668 (C=O), 1614, 1540, 1565, 1439 (C=C, C–N), 934, 828, 734, 698, 677 (CH=CH of mono-, 1,2- and 1,2,4-trisubstituted benzenes) cm$^{-1}$; $^1$H NMR (CDCl$_3$, a few drops of *DMSO*, 300 MHz): $\delta = 8.38$ (s, CH of methine), 8.06 (dd, $J_{7,8} = 8.9$, $J_{7,5} = 1.7$ Hz, H-7 of Ht), 7.87–7.25 (m, 10H, Ar), 6.47 (d, $J_{7,8} = 8.9$ Hz, H-8 of Ht), 5.10 (d, $J = 3.7$ Hz, OH), 4.37–4.27 (m, CH), 3.80 (dd, 1H, $J_{AB} = 12.2$ Hz, $J_{AX} = 2.1$ Hz, H$_A$ of NCH$_2$), 3.62 (dd, 1H, $J_{BX} = 5.6$ Hz, H$_B$ of NCH$_2$), 3.14 (dd, 1H, $J_{AB} = 15.9$, $J_{AX} = 4.0$ Hz, H$_A$ of C*H*$_2$CH), 2.94 (dd, 1H, $J_{BX} = 6.4$ Hz, H$_B$ of C*H*$_2$CH) ppm; APCI-MS: $m/z$ (%) = 382 [M + H]$^+$ (100).

### rac-3-Hydroxy-1-phenyl-1,2,3,4-tetrahydroquinoline-6-carbaldehyde (4, C$_{16}$H$_{15}$NO$_2$)

Compound *rac*-**1** (22 g, 0.07 mol) was dissolved in 40 cm$^3$ methanol by heating. A solution of 4.7 g 85% KOH (0.07 mol) in 7 cm$^3$ H$_2$O was added to the mixture. The reaction mixture was refluxed for 35 min. When the reaction was over, the mixture was extracted with ethyl acetate. The organic

layer was dried over anhydrous MgSO$_4$, filtered, and ethyl acetate was removed. The residue was dissolved in 40 cm$^3$ methanol and, after keeping at 5°C, crystals formed. The crystalline product was filtered off, washed with 2-propanol, and recrystallized from methanol. Yield 6 g (45%); mp 198–200°C; IR (KBr): $\bar{\nu} = 3371$ (OH), 3036 (CH$_{\text{arom}}$), 2896, 2666 (CH$_{\text{aliph}}$), 1653 (CHO), 1604, 1555, 1491 (C=C, C–N), 943, 822, 791, 768, 702 (CH=CH of mono- and 1,2,4-trisubstituted benzenes) cm$^{-1}$; $^1$H NMR (CDCl$_3$, 300 MHz): $\delta = 9.61$ (s, CHO), 7.57–7.22 (m, 7H, Ar), 6.52 (d, $J_{7,8} = 8.6$ Hz, H-8 of Ht), 4.43–4.33 (m, CH), 3.83–3.56 (m, NCH$_2$), 3.16 (dd, 1H, $J_{AB} = 16.2$, $J_{AX} = 4.1$ Hz, H$_A$ of C*H*$_2$CH), 2.93 (dd, 1H, $J_{AB} = 16.2$, $J_{BX} = 5.3$ Hz, H$_B$ of C*H*$_2$CH), 2.87–2.78 (m, OH) ppm; APCI-MS: $m/z$ (%) = 254 [M + H]$^+$ (100).

### u/l-3-Oxiranylmethoxy-1-phenyl-1,2,3,4-tetrahydro-quinoline-6-carboxaldehyde (5)

To 27 cm$^3$ epichlorohydrin (0.34 mol) 6 g *rac*-**4** (0.02 mol) were added and stirred vigorously at 35–40°C. During the reaction 7.6 g 85% KOH (0.12 mol) and 2.7 g Na$_2$SO$_4$ (0.02 mol) were added in 5 equal portions every hour with prior cooling to 30°C. The reaction mixture was stirred at 35–40°C overnight. After termination of the reaction (control with TLC, diethyl ether:*n*-hexane = 2:1), the mixture was extracted with ethyl acetate. The organic layer was dried over anhydrous MgSO$_4$, filtered, and ethyl acetate was removed to give 6 g of crude product *u/l*-**5**, which could be used in the further synthesis of *u/l*-**6** without purification.

### u/l-3-Oxiranylmethoxy-6-(1,3-dioxoindan-2-ylmethylene)-1-phenyl-1,2,3,4-tetrahydroquinoline (6, C$_{28}$H$_{23}$NO$_4$)

Compound *u/l*-**5** (6 g, 0.02 mol) was dissolved in 15 cm$^3$ methanol by heating. The mixture was cooled to room temperature, and 2.9 g 1,3-indandione (0.02 mol) were added. The reaction mixture was stirred at room temperature for 3 days. At the end of the reaction (control with TLC, diethyl ether:*n*-hexane = 2:1) the crystalline product was filtered off, washed with 2-propanol, and recrystallized from 2-propanol:ethyl acetate = 2:1. Yield 7 g (82%); mp 164–166°C; IR (KBr): $\bar{\nu} = 3362$ (CH$_{\text{arom}}$), 2999, 2894 (CH$_{\text{aliph}}$), 1714, 1670 (C=O), 1617, 1539, 1509, 1440 (C=C, C–N), 903, 824, 732, 703, 697, 676 (CH=CH of mono-, 1,2-di-, and 1,2,4-trisubstituted benzenes) cm$^{-1}$; $^1$H NMR (CDCl$_3$, 300 MHz): $\delta = 8.54$ (s, 0.5H CH of methine of one diastereomer), 8.52 (s, 0.5H CH of methine of other diastereomer) 8.09–8.00 (m, H-7 of Ht), 7.94–7.25 (m, 10H, Ar), 6.56 (d, 0.5H, $J_{7,8} = 8.8$ Hz, H-8 of one diastereomer), 6.53 (d, 0.5H, $J_{7,8} = 8.8$ Hz, H-8 of other diastereomer), 4.18–4.08 (m, 1H, NCH$_2$C*H*O), 3.99–3.40 (m, 4H, NCH$_2$, OCH$_2$), 3.29–2.99 (m, 3H, CH$_2$CHO of epoxy gr.), 2.82–2.75 (m, 1H, one H of PhC*H*$_2$CH), 2.64–2.56 (m, 1H, another H of PhC*H*$_2$CH) ppm; APCI-MS: $m/z$ (%) = 438 [M + H]$^+$ (100), 364 [M + H − OCH$_2$CHCH$_2$O]$^+$ (16).

### u/l-1,3-Bis-{3-hydroxy-4-[6-(1,3-dioxoindan-2-ylmethylene)-1-phenyl-1,2,3,4-tetrahydroquinolin-3-yloxy]-1-thiabuthyl}-benzene (7a, C$_{62}$H$_{52}$N$_2$O$_8$S$_2$)

Compound *u/l*-**6** (3 g, 7 mmol) and 25 cm$^3$ *MEK* were refluxed for 10 min. The mixture was cooled to room temper-

ature, and 0.4 g 1,3-benzenedithiol (3 mmol) and 0.4 cm$^3$ *TEA* were added. The reaction mixture was refluxed for 1 h under argon atmosphere. After termination of the reaction *MEK* and *TEA* were removed and the residue was purified by column chromatography (eluent: acetone/*n*-hexane = 1/4). The obtained compound was dissolved in 15 cm$^3$ toluene and poured with intensive stirring into 220 cm$^3$ *n*-hexane to obtain amorphous product **7a**. Yield 1.7 g (49%); IR (KBr): $\bar{\nu}$ = 3422 (OH), 3062 (CH$_{arom}$), 2898, 2868, 2641 (CH$_{aliph}$), 1714, 1670 (C=O), 1615, 1595, 1552, 1432 (C=C, C–N), 936, 870, 813, 774, 735, 698, 681 (CH=CH of mono-, 1,2-di-, 1,3-di-, and 1,2,4-trisubstituted benzenes) cm$^{-1}$; $^1$H NMR (*DMSO*-d$_6$, 300 MHz): $\delta$ = 8.46 (splitted s, 2H, CH of methine), 8.04–7.21 (m, 2H, H-7 of Ht), 7.91–6.96 (m, 24H, *Ar*), 6.41 (m, 2H, H-8 of Ht), 5.21–5.12 (m, 2H, OH), 4.11–3.98 (m, 2H, OCH$_2$C*H*), 3.91–3.41 (m, 10H, NCH$_2$CHOCH$_2$), 3.16–2.80 (m, 8H, SCH$_2$, NCH$_2$CHC*H*$_2$) ppm; APCI-MS: *m/z* (%) = 1016 [M − H]$^-$ (15).

*u/l*-4,4′-Bis-{{3-hydroxy-4-[6-(1,3-dioxoindan-2-ylmethylene)-1-phenyl-1,2,3,4-tetrahydroquinolin-3-yloxy]-1-thiabuthyl}phenyl}sulfide (**7b**, C$_{68}$H$_{56}$N$_2$O$_8$S$_3$)

Synthesis of compound *u/l*-**7b** was carried out according to the procedure described for *u/l*-**7a** except that 3 g *u/l*-**6** (7 mmol), 15 cm$^3$ *MEK*, 0.8 g 4,4′-thiobisbenzenethiol (3 mmol), and 2 cm$^3$ *TEA* were used. Yield 2 g (52%); IR (KBr): $\bar{\nu}$ = 3420 (OH), 3062 (CH$_{arom}$), 2867, 2672 (CH$_{aliph}$), 1712, 1670 (C=O), 1615, 1594, 1552, 1505, 1432 (C=C, C–N), 935, 810, 762, 734, 698, 677 (CH=CH of mono-, 1,2-di-, 1,3-di-, and 1,2,4-trisubstituted benzenes) cm$^{-1}$; $^1$H NMR (*DMSO*-d$_6$, 300 MHz): $\delta$ = 8.47 (splitted s, 2H, CH of methine), 8.14–8.03 (m, 2H, H-7 of Ht), 7.91–6.93 (m, 38H, *Ar*), 6.33 (d, 0.5H, $J_{7,8}$ = 8.8 Hz, H-8 of one diastereomer), 6.32 (d, 0.5H, $J_{7,8}$ = 8.8 Hz, H-8 of another diastereomer), 5.23–5.09 (m, 2H, OH), 4.12–3.40 (m, 2H, OCH$_2$C*H*), 3.91–3.43 (m, 10H, NCH$_2$CH, OCH$_2$), 3.36–2.76 (m, 8H, SCH$_2$, NCH$_2$CHC*H*$_2$) ppm; APCI-MS: *m/z* (%) = 1124 [M − H]$^-$ (16).

## Acknowledgements

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