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Hydrazones Possessing a Phenyl-1,2,3,4-tetrahydroquinoline Moiety as Hole Transporting Materials

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Summary. Hydrazones containing 1-phenyl-1,2,3,4-tetrahydroquinoline units were synthesized starting from diphenylamine. These compounds were found to constitute novel hole transporting materials and were characterized by the time of flight method. The hole drift mobility in these compounds exceeds $10^{-6}\,cm^2\,V^{-1}\,s^{-1}$ at an electric field of $10^6\,V\,cm^{-1}$.

Keywords. Hydrazone; Epoxide; 1-Phenyl-1,2,3,4-tetrahydroquinoline; Charge transport.

Introduction

During recent years rapid developments in the chemistry of 1,2,3,4-tetrahydroquinolines were observed. One of the main interests in them is due to their biological activities. Thus, many relatively simple synthetic 1,2,3,4-tetrahydroquinolines are already used or have been tested as potential drugs [1–3]. Besides pharmaceutical applications, tetrahydroquinoline derivatives are useful as pesticides [4], antioxidants [5], corrosion inhibitors [6], and active components of various dyes [7, 8]. They are also widely used in modern recording technologies: as intermediates for photographic couplers [9], as high sensitivity photosensitizers in photography [10], in the preparation of optical information recording media [11], and as charge transporting materials in electrophotographic photoconductors [12].

Compounds possessing hydrazone moieties are also well known for their photoresponsible properties [13]. High photosensitivity, simple synthesis, and low price are the advantages of the hydrazones against other hole transporting materials. Low molecular weight transporting materials (TMs) containing hydrazone moieties are usually

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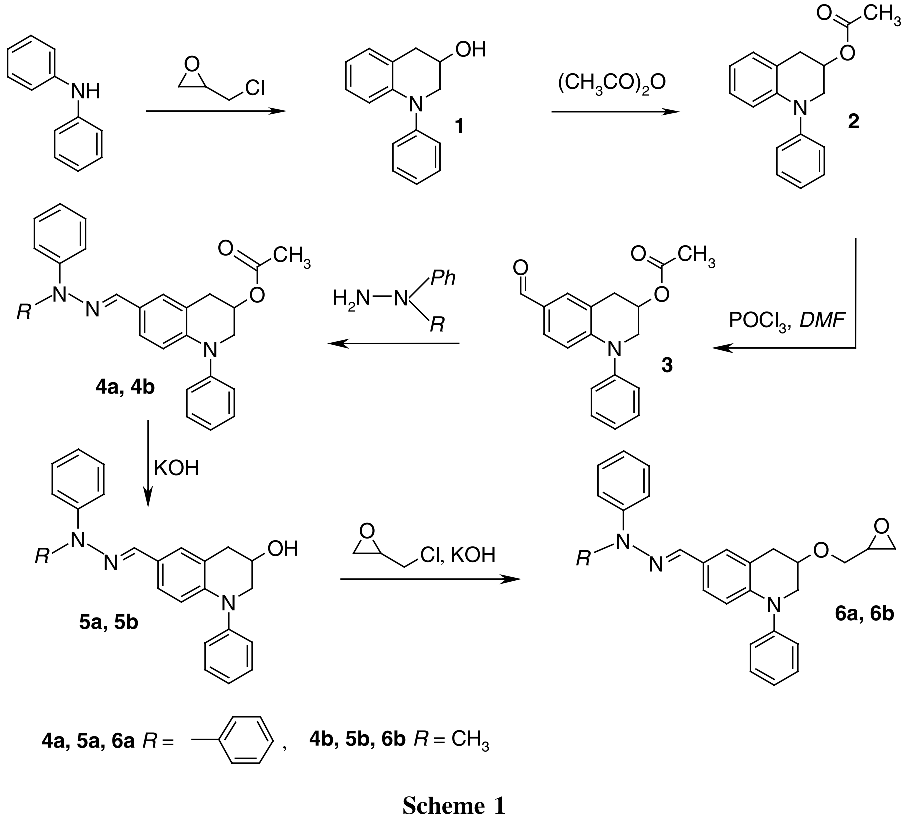
crystalline materials, not capable of forming thin neat homogenous layers, and must be used in combination with polymeric hosts, usually polycarbonate (*PC*). Aromatic hydrazone molecules dispersed in a binder polymer are used as the main constituent of electrophotographic devices due to their excellent hole-transporting property [14]. Recently, because of their rapid charge transporting ability, more attention has been paid to organic photoreceptors with hydrazones possessing 1-phenyl-1,2,3,4-tetrahydroquinoline moieties as hole transporting materials [15–17]. Unfortunately, the multi-step synthesis of the starting 1-phenyl-1,2,3,4-tetrahydroquinoline limits the development of organic photoreceptors with this promising moiety.

In the present article we report the synthesis, characterization, and photoconductive properties of the hydrazones **6a** and **6b** possessing the 1-phenyl-1,2,3,4-tetrahydroquinoline moiety as hole transporting materials. The molecules of these TMs consist of a large π-electron system conjugated through nitrogen atoms and a flexible oxiranylalkyl group attached to it. The molecular structure of these TMs makes crystallisation in solid state difficult and improves adhesion and compatibility with *PC*. Another peculiarity of these functionalized hydrazones is the possibility to use **6a** and **6b** as precursors for the synthesis of a novel class of hole TMs possessing a 1-phenyl-1,2,3,4-tetrahydroquinoline moiety [18–20].

Results and Discussion

Contrary to the multi-step synthesis of the widely used 1-phenyl-1,2,3,4-tetrahydroquinoline, the starting 3-hydroxy-1-phenyl-1,2,3,4-tetrahydroquinoline (**1**) was



Scheme 1

obtained by a one-pot reaction of diphenylamine with epichlorohydrin (Scheme 1). At first, in order to protect hydroxyl group, **1** was converted to **2**. The next step was a *Vilsmeier* formylation followed by condensation of the resulting **3** with hydrazine derivatives to get hydrazones **4a** and **4b** with different substituents at the hydrazone N-atom. Then, the hydroxyl group was deprotected to give the 1-phenyl-1,2,3,4-tetrahydroquinoline hydrazones **5a** and **5b**. Finally, by interaction of **5a** and **5b** with epichlorohydrin in presence of KOH, 3-oxiranylmethoxy-1-phenyl-1,2,3,4-tetrahydroquinoline-6-carboxaldehyde *N*,*N*-diphenylhydrazone (**6a**) and 3-oxiranylmethoxy-1-phenyl-1,2,3,4-tetrahydroquinoline-6-carboxaldehyde *N*-phenyl-*N*-methylhydrazone (**6b**) were isolated.

The chemical structures of **6a** and **6b** were confirmed by IR, UV, and ^{1}H NMR spectrometry. In the ^{1}H NMR spectra of **6a** and **6b** the epoxy group gave a double set of signals of the AB part of an ABX system at 2.99–2.50 ppm, which indicated that due to the presence of two stereogenic centers, the isolated hydrazones were mixtures of diastereomers.

Since π-electrons are very important for the charge transporting process in the TM structures, the absorption spectra of **6a** and **6b** were recorded. Electron transitions to the higher energy states in the investigated derivatives give two main absorption maxima (Fig. 1) at *ca.* 210 and 370 nm. The comparison of the UV/Vis Vis spectra of **6a** and **6b** with the starting **1** showed that the second absorption band is bathochromically shifted by 72 nm and is hyperchromically intensified due to the π-electron conjugation through the lone electron pair at the nitrogen atom. On the other hand, only a negligible bathochromic shift was observed for the diphenyl substituted hydrazone **6a** in comparison with the methylphenyl substituted **6b**. The former acts somewhat unusually – introduction of an additional aromatic moiety does not lead to a marked bathochromic shift.

The results of the electron photoemission in air are presented in Fig. 2. The ionization potential (I_p) value for **6a** is 5.37 eV. Replacement of the phenyl group by the methyl group leads to the decrease of I_p to 5.29 eV for **6b** (Table 1).

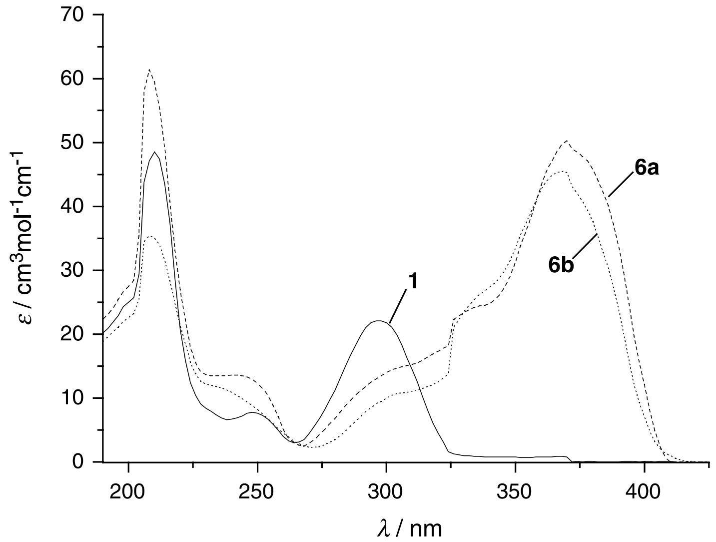


Fig. 1. Absorption spectra (CH_3CN, $c = 10^{-4}$ *M*) of **6a** and **6b** and starting compound **1**

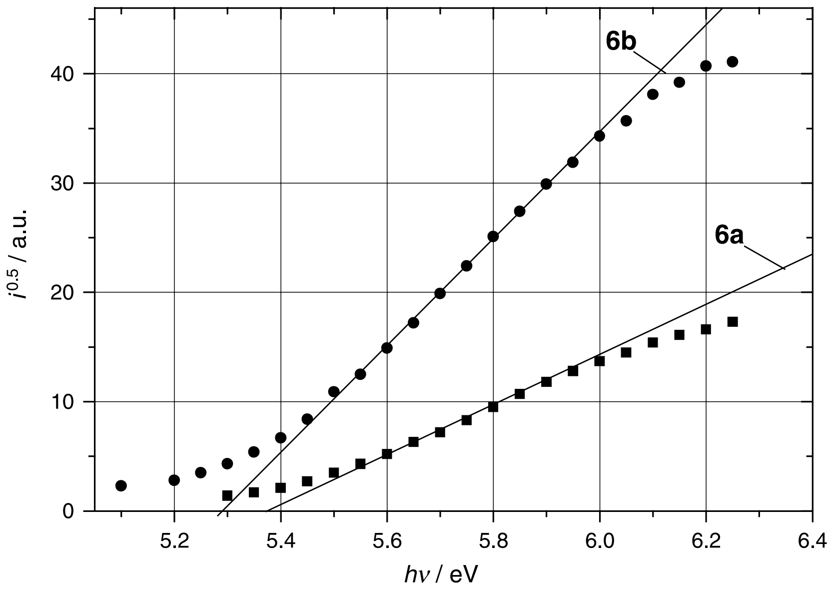


Fig. 2. Photoemission (in air) spectra of the TMs

Table 1. Characteristics of **6a** and **6b**

Compound	$\mu_0/\mathrm{cm}^2\,\mathrm{V}^{-1}\,\mathrm{s}^{-1}$	$\mu/\mathrm{cm}^2\,\mathrm{V}^{-1}\,\mathrm{s}^{-1}$	I_p/eV
6a	10^{-7}	$4.4 \cdot 10^{-6}$	5.37
6b	$6 \cdot 10^{-8}$	$4.6 \cdot 10^{-6}$	5.29

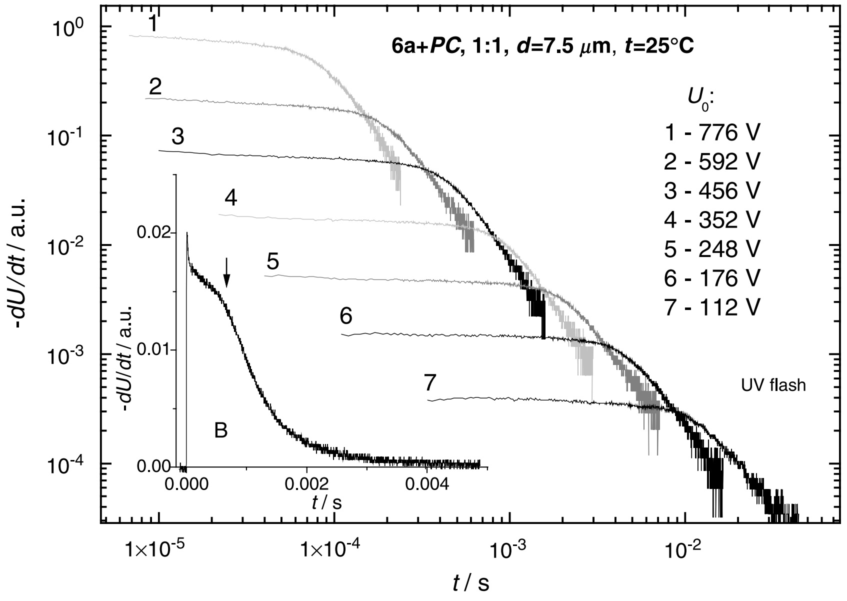


Fig. 3. XTOF transients for **6a**; insert shows one transient curve in linear plot

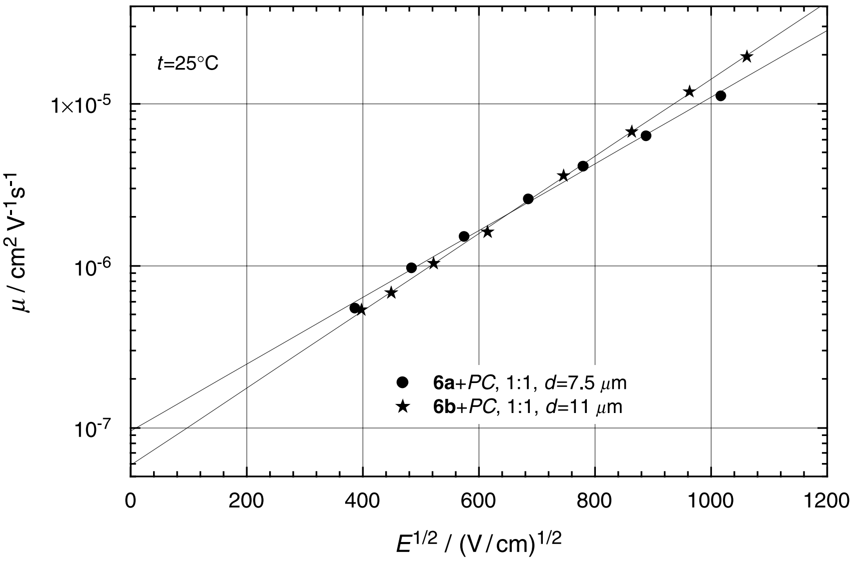


Fig. 4. Field dependencies of the hole drift mobilities of **6a** and **6b**

The hydrazones **6a** and **6b** are soluble in common organic solvents such as acetone, chloroform, *THF*, dioxane, *etc*. Clear, transparent, and homogeneous films were obtained using the casting technique. The hole drift mobility for **6a** and **6b** was measured by the xerographic time of flight method (XTOF).

XTOF measurements revealed that small charge transport transients are *Gaussian* with well-defined transit times on linear plots in all cases (Fig. 3). Figure 4 shows the room temperature dependencies of the hole-drift mobility on the electric field in the composition of **6a** and **6b** with *PC*. The mobility defining parameters μ_0 and the mobility value at the 6.7×10^5 V/cm field strength are given in Table 1. As seen from these results, the mobility values in amorphous films of compositions **6a** and **6b** with *PC* exceed $10^{-6}\,\mathrm{cm}^2\,\mathrm{V}^{-1}\,\mathrm{s}^{-1}$ at an electric field of 10^6 V/cm. Due to the same π-electron conjugation as mentioned earlier there is a small difference of mobility between the diphenyl substituted hydrazone **6a** and the methylphenylhydrazone **6b**. It should be noted that the measured mobility of the TM compositions with a binder is high enough for many practical applications and it remains approximately in the same range as for the recently reported 1-phenyl-1,2,3,4-tetrahydroquinoline-6-carboxaldehyde-1,1′-diphenylhydrazone [17].

In conclusion, a novel family of hole transporting materials containing the 1-phenyl-1,2,3,4-tetrahydroquinoline hydrazone moiety was synthesized and investigated as potential new materials for electrophotography. These TMs exhibit high hole drift mobilities and good compatibility with *PC*, and they possess the chemically active epoxy group, which enables them to be used as potential starting materials in the synthesis of various TM materials for optoelectronic devices.

Experimental

The ^{1}H NMR spectra were taken on a Varian Unity Inova (300 MHz) spectrometer in $CDCl_3$. 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 reactions 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 were performed with an Exeter Analytical CE-440 Elemental Analyzer; their results agreed satisfactorily with the calculated values. The ionization potential was measured by the photoemission in air method, similar to the one used in Ref. [21] and described in Ref. [18]. The samples for mobility measurements were prepared from a 1:1 mass proportion compositions of **6a** and **6b** with polycarbonate-Z (*PC*) (Iupilon Z-200 from Mitsubishi Gas Chemical Co.). The sample substrate was a polyester film with conductive Al layer. The layer thickness was in the range of 7–11 μm. The hole drift mobility was measured by xerographic time of flight technique [22–24]. Positive corona charging created electric field inside the TM layer. Charge carriers were generated at the layer surface by illumination with pulses of a N_2 laser (pulse duration 2 ns, wavelength 337 nm). The layer surface potential decrease as a result of pulse illumination was up to 1–5% of the initial potential before illumination. The capacitance probe that was connected to the wide frequency band electrometer measured the rate of the surface potential decrease, dU/dt. The transit time t_t was determined by the kink on the curve of the dU/dt transient in linear scale. The drift mobility was calculated by the formula $\mu = d^2/U_0 t_t$, where d is the layer thickness and U_0 is the surface potential at the moment of illumination.

3-Hydroxy-1-phenyl-1,2,3,4-tetrahydroquinoline (**1**)

To 169 g diphenylamine (1 mol) 172 cm^3 epichlorohydrin (2.2 mol) were added in a 500 cm^3 round bottom flask with reflux condenser. The mixture was heated at 155–160°C for 36–40 h. After termination of the reaction unreacted epichlorohydrin was removed by distillation and obtained residue was dissolved in 100 cm^3 ethanol. The crystals formed upon standing at room temperature were filtered off, washed with diethyl ether, and recrystallized from ethanol. Yield 140 g (62%); mp 78.0–79.0°C (Ref. [25] 78.5–79.0°C); ^{1}H NMR ($CDCl_3$, 300 MHz): $\delta = 7.41$–6.66 (m, 9H, Ar), 4.34–4.25 (m, CH), 3.77–3.46 (m, NCH_2), 3.13 (dd, 1H, $J_{AB} = 16.5$ Hz, $J_{AX} = 4.4$ Hz, H_A of $C\underline{H}_2CH$), 2.86 (dd, 1H, $J_{AB} = 16.5$ Hz, $J_{BX} = 4.6$ Hz, H_B of $C\underline{H}_2CH$), 2.44–2.23 (m, OH) ppm.

3-Acetyl-1-phenyl-1,2,3,4-tetrahydroquinoline (**2**)

Compound **1** (35 g, 0.2 mol) was dissolved in 150 cm^3 acetic anhydride and the mixture was heated at 100°C for 3 h. At the end of the reaction acetic anhydride was removed. The residue was dissolved in methanol by heating and crystals formed upon standing at room temperature. The crystalline product was filtered off, washed with methanol, and recrystallized from methanol. Yield 40 g (97%); mp 64–65°C (Ref. [25] 64.1–64.7°C); IR (KBr): $\bar{\nu} = 3053$, 3029, 3007 (CH_{arom}), 2949, 2936, 2913, 2875, 2825 (CH_{aliph}), 1723 (C=O), 1604, 1594, 1574, 1493 (C=C, C–N), 772, 756, 704 (CH=CH of mono- and disubstituted benzenes) cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): $\delta = 7.37$–6.71 (m, 9H, Ar), 5.35–5.25 (m, CH), 3.81–3.66 (m, NCH_2), 3.18 (dd, 1H, $J_{AB} = 16.6$ Hz, $J_{AX} = 4.4$ Hz, H_A of $C\underline{H}_2CH$), 2.94 (dd, 1H, $J_{AB} = 16.6$ Hz, $J_{BX} = 5.8$ Hz, H_B of $C\underline{H}_2CH$), 1.95 (s, CH_3) ppm.

3-Acetyl-1-phenyl-1,2,3,4-tetrahydroquinoline-6-carbaldehyde (**3**, $C_{18}H_{17}NO_3$)

To a 250 cm^3 3-neck round bottom flask equipped with thermometer, magnetical stirrer, and addition funnel 44 cm^3 *DMF* (0.6 mol) were added. The content was cooled in a salt/ice bath. 21 cm^3 $POCl_3$ (0.2 mol) were slowly added to the mixture. During the addition of $POCl_3$, the temperature of the mixture was not allowed to rise above 5°C. After the addition was completed, the reaction mixture was allowed to warm to room temperature. A solution of 30 g **2** (0.1 mol) in 50 cm^3 *DMF* was added to the mixture and the mixture was heated to 90°C for 1 h. The hot reaction mixture was poured into a 500 cm^3 beaker containing 200 g ice. The mixture was neutralized by adding 40% NaOH solution and allowed to stand at 5°C for 24 h. The precipitate was isolated by decanting, dissolved in ethyl acetate, and washed with H_2O until neutral, dried ($MgSO_4$), and the solvent was removed. The residue was dissolved in methanol and crystals formed upon standing at room temperature. The crystalline product was filtered off, washed with methanol, cooled to −5°C, and recrystallized from methanol. Yield 20 g (59%); mp 89–90.5°C; IR (KBr): $\bar{\nu} = 3065$, 3051 (CH_{arom}), 2982, 2915, 2839, 2748 (CH_{aliph}), 1728 (C=O), 1664 (CHO) cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): $\delta = 9.71$ (s, CHO), 7.63–7.20 (m, 7H, Ar), 6.59 (d, 1H, $J = 8.6$ Hz, 8-H of Ht), 5.43–5.35 (m, CH), 3.89–3.71 (m, NCH_2), 3.23 (dd, 1H, $J_{AB} = 16.6$ Hz, $J_{AX} = 4.3$ Hz, H_A of $C\underline{H}_2CH$), 3.05 (dd, 1H, $J_{AB} = 16.6$ Hz, $J_{BX} = 4.9$ Hz, H_B of $C\underline{H}_2CH$), 2.04 (s, CH_3) ppm.

3-Acetyl-1-phenyl-1,2,3,4-tetrahydroquinoline-6-carbaldehyde N,N-diphenylhydrazone

(**4a**, $C_{30}H_{27}N_3O_2$)

To a solution of 5 g **3** (17 mmol) in 9 cm^3 *THF* 6 g *N*,*N*-diphenylhydrazine hydrochloride (25 mmol) dissolved in 33 cm^3 methanol were added. The mixture was stirred at room temperature for 2 h. The crystalline product formed during the reaction was filtered off, washed with 2-propanol, and recrystallized from ethyl acetate:methanol = 1:1. Yield 6 g (77%); mp 162–164.5°C; IR (KBr): $\bar{\nu} = 3032$ (CH_{arom}), 2961, 2937, 2866 (CH_{aliph}), 1732 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): $\delta = 7.44$–7.06 (m, 18H, Ar, CH=N), 6.73 (d, 1H, $J = 8.6$ Hz, 8-H of Ht), 5.35–5.26 (m, CH), 3.83–3.68 (m, NCH_2), 3.18 (dd, 1H, $J_{AB} = 16.7$ Hz, $J_{AX} = 4.8$ Hz, H_A of $C\underline{H}_2CH$), 2.96 (dd, 1H, $J_{AB} = 16.7$ Hz, $J_{BX} = 5.4$ Hz, H_B of $C\underline{H}_2CH$), 1.96 (s, CH_3) ppm.

3-Acetyl-1-phenyl-1,2,3,4-tetrahydroquinoline-6-carbaldehyde N-phenyl-N-methylhydrazone

(**4b**, $C_{25}H_{25}N_3O_2$)

Compound **4b** was prepared according to the procedure described above for **4a** except that 20 g **3** (0.07 mol), 10 cm^3 *N*-phenyl-*N*-methylhydrazine (0.08 mol) and 100 cm^3 methanol instead of *THF* were used. The reaction mixture was stored over night at room temperature. Yield 24 g (89%); mp 158–160°C; ^1H NMR ($CDCl_3$, 300 MHz): $\delta = 7.47$–6.86 (m, 13H, Ar, CH=N), 6.78 (d, 1H, $J = 8.5$ Hz, 8-H of Ht), 5.33–5.29 (m, CH), 3.85–3.69 (m, NCH_2), 3.38 (s, NCH_3), 3.22 (dd, 1H, $J_{AB} = 16.4$ Hz, $J_{AX} = 5.0$ Hz, H_A of $C\underline{H}_2CH$), 3.01 (dd, 1H, $J_{AB} = 16.4$ Hz, $J_{BX} = 5.4$ Hz, H_B of $C\underline{H}_2CH$), 1.97 (s, CH_3) ppm.

3-Hydroxy-1-phenyl-1,2,3,4-tetrahydroquinoline-6-carbaldehyde N,N-diphenylhydrazone

(**5a**, $C_{28}H_{25}N_3O$).

To a solution of 24 g **4a** (0.05 mol) in 30 cm^3 dioxane 3.4 g KOH (0.05 mol) dissolved in 10 cm^3 H_2O were added. The mixture was refluxed for 3 h. Dioxane was removed and the residue was extracted with ethyl acetate and washed with H_2O until neutral. The organic layer was dried ($MgSO_4$), filtered off, and ethyl acetate was removed. The residue was purified by column chromatography (eluent: acetone:*n*-hexane = 1:7). Yield 16 g (73%); IR (KBr): $\bar{\nu} = 3374$ (OH), 3057, 3033 (CH_{arom}), 2920, 2850 (CH_{aliph}), 1590, 1493 (C=C, C–N) cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): $\delta = 7.44$–7.06 (m, 18H, Ar, CH=N), 6.67 (d, 1H, $J = 8.6$ Hz, 8-H of Ht), 4.38–4.26 (m, CH), 3.77–3.52 (m, NCH_2), 3.14 (dd, 1H, $J_{AB} = 16.3$ Hz, $J_{AX} = 4.3$ Hz, H_A of $C\underline{H}_2CH$), 2.96 (dd, 1H, $J_{AB} = 16.3$ Hz, $J_{BX} = 5.8$ Hz, H_B of $C\underline{H}_2CH$), 2.79 (d, $J = 5.5$ Hz, OH) ppm.

3-Hydroxy-1-phenyl-1,2,3,4-tetrahydroquinoline-6-carbaldehyde N-phenyl-N-methylhydrazone

(**5b**, $C_{23}H_{23}N_3O$)

To a solution of 24 g **4b** (0.06 mol) in 100 cm^3 *THF* 4 g KOH (0.06 mol) dissolved in 15 cm^3 H_2O were added and heated to reflux. The same amount of KOH/H_2O was added after 1, 4, and 8 h. After these additions the mixture was refluxed for another 11 h. Then the reaction mixture was diluted with ethyl acetate and crystals formed upon standing at room temperature. The crystalline product was filtered off, washed with 2-propanol, and recrystallized from ethyl acetate. Yield 14 g (65%); mp 149–151°C; ^1H NMR ($CDCl_3$, 300 MHz): $\delta = 7.49$–6.83 (m, 13H, Ar, CH=N), 6.71 (d, 1H, $J = 8.8$ Hz, 8-H of Ht), 4.42–4.29 (m, CH), 3.77–3.55 (m, NCH_2), 3.37 (s, CH_3), 3.19 (dd, 1H, $J_{AB} = 16.4$ Hz, $J_{AX} = 4.5$ Hz, H_A of $C\underline{H}_2CH$), 2.93 (dd, 1H, $J_{AB} = 16.4$ Hz, $J_{BX} = 4.9$ Hz, H_B of $C\underline{H}_2CH$), 2.11 (d, $J = 7.0$ Hz, OH) ppm.

3-Oxiranylmethoxy-1-phenyl-1,2,3,4-tetrahydroquinoline-6-carboxaldehyde N,N-diphenylhydrazone (**6a**, $C_{31}H_{29}N_3O_2$)

A mixture of 13 g **5a** (0.03 mol) and 36 cm^3 epichlorohydrin (0.47 mol) was stirred vigorously for 3 h at 35–40°C. Then 6.1 g 85% powdered KOH (0.09 mol) and 1.6 g anhydrous Na_2SO_4 (0.01 mol) were added in 6 equal portions every h with prior cooling of the reaction mixture to 30°C. Then the reaction mixture was extracted with ethyl acetate. The organic layer was dried ($MgSO_4$) and filtered. Ethyl acetate and excess of epichlorohydrin were removed and the residue was purified by column chromatography (eluent: ethyl acetate:*n*-hexane = 1:7) to obtain **6a**. Two months later the product crystallized. Yield 4 g (30%); mp 126–128°C; ^1H NMR ($CDCl_3$, 300 MHz): $\delta = 7.45–7.05$ (m, 18H, Ar, CH=N), 6.69, 6.70 (2d, 1H, $J = 2.4$ Hz, diasteromeric 8-H of Ht), 4.08–3.97 (m, $NCH_2C\underline{H}$), 3.88–3.40 (m, 4H, NCH_2, OCH_2), 3.20–3.07 (m, $C\underline{H_2}CHO$), 2.98–2.84 (m, CH from epoxy gr), 2.80–2.73 (m, H_A from $C\underline{H_2}CH$ of epoxy gr), 2.59 (dd, 0.5H, $J_{AB} = 5.0$ Hz, $J_{BX} = 2.8$ Hz, H_B of one diasteromer of $C\underline{H_2}CH$ from epoxy gr), 2.54 (dd, 0.5H, $J_{AB'} = 5.2$ Hz, $J_{B'X} = 2.6$ Hz, $H_{B'}$ of other diasteromer of $C\underline{H_2}CH$ from epoxy gr) ppm.

3-Oxiranylmethoxy-1-phenyl-1,2,3,4-tetrahydroquinoline-6-carboxaldehyde N-phenyl-N-methylhydrazone (**6b**, $C_{26}H_{27}N_3O_2$)

Compound **6b** was prepared according to the procedure described for **6a** except that 11 g **5b** (0.03 mol), 36 cm^3 epichlorohydrin (0.05 mol), 6.1 g powdered 85% KOH (0.09 mol), and 1.6 g Na_2SO_4 (0.01 mol) were used. The reaction time was 26 h. The product was crystallized from ethyl acetate:methanol = 2:1. The crystals were filtered off, washed with 2-propanol, and recrystallized from ethyl acetate: methanol = 2:1. Yield 9 g (71%); mp 92–94°C; ^1H NMR ($CDCl_3$, 300 MHz): $\delta = 7.48–6.85$ (m, 13H, Ar, CH=N), 6.77, 6.76 (2d, 1H, $J = 2.3$ Hz, diasteromeric 8-H of Ht), 4.10–3.96 (m, $NCH_2C\underline{H}$), 3.86–3.41 (m, 4H, NCH_2, $OC\underline{H_2}$), 3.36 (s, CH_3), 3.26–2.87 (m, $OCHC\underline{H_2}$), 2.99–2.89 (m, 1H, CH of epoxy gr), 2.79–2.73 (dd, 1H, H_A of $C\underline{H_2}CH$ from epoxy gr), 2.60 (dd, 0.5H, $J_{AB} = 4.9$ Hz, $J_{BX} = 3.0$ Hz, H_B of one diastereomer of $C\underline{H_2}CH$ from epoxy gr), 2.54 (dd, 0.5H, $J_{AB} = 4.9$ Hz, $J_{B'X} = 2.9$ Hz, $H_{B'}$ of other diastereomer of $C\underline{H_2}CH$ from epoxy gr) ppm.

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