

Design of Postmetallocene Schiff Base-Like Catalytic Systems for Polymerization of Olefins: XII.* Synthesis of Tetradentate Bis-salicylaldehyde Imine Ligands

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Abstract—Reactions of salicylaldehyde, 3-*tert*-butylsalicylaldehyde, and 3,5-di-*tert*-butylsalicylaldehyde with 1,4-diaminobutane, 1,6-diaminohexane, 4,4'-diaminodiphenylmethane, 4,4'-diamino-3,3',5,5'-tetramethyldiphenylmethane, 4,4'-diamino-5,5'-dicyclopentyl-3,3'-dimethyldiphenylmethane, 4,4'-diamino-5,5'-dicyclohexyl-3,3'-dimethyldiphenylmethane, bis(4-aminophenyl) sulfone, *o,o'*- and *p,p'*-diaminodiphenyl ethers, 1,4-bis(4-aminophenoxy)benzene, 2,2-bis[4-(4-aminophenoxy)phenyl]propane, and 4,4''-diamino-*p*-terphenyl gave a series of the corresponding Schiff bases which can be used as tetradentate ligands for the synthesis of titanium and zirconium complexes.

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Despite striking advances in the field of studying postmetallocene catalytic systems for polymerization of olefins, achievement of maximal control over the structure and properties of the resulting polymers remains an important problem [2]. Phenoxy–imine complexes of Group IV transition metals were found [3] to catalyze living polymerization which makes it possible to obtain polymers with required molecular weight and block copolymer structure. Therefore, design of new phenoxy–imine catalysts seems to be quite promising.

The main disadvantages of such catalysts include loss of activity and “living” character of the process and formation of low-molecular weight oligomers at elevated temperature [4]. The existing views on the mechanism of olefin polymerization over metallocene and postmetallocene catalysts imply that the reasons are increased contribution of chain transfer and β -hydride transfer processes and stronger deactivation of the catalyst as the temperature rises.

Processes inducing deactivation of catalysts are ligand transfer to aluminum cocatalyst present in a very

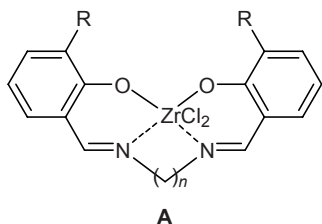
large excess in the catalytic system [5] and insertion of alkyl ligand into the C=N bond via 1,2-migration [6]. Moreover, cationic centers tend to undergo dimerization through formation of μ -alkyl bridges, which is referred to as *bimolecular deactivation* [7]; as a result, inactive species become to predominate in the catalytic system. Insofar as the above processes are sensitive to the ligand environment, the structure of available transition metal complexes which catalyze olefin polymerization at elevated temperature suggests that their modification should give rise to new promising catalysts.

We believe that a challenging method for modification of α -diimine, diiminopyridine, and bis(salicylaldehyde imine) complexes may be bonding of the imino groups through various bridges. In other words, modified ligands can be synthesized using diamines instead of primary alkyl- or arylamines. We also anticipated that complexes with linked imino groups would be more stable toward deactivation. These views are supported by the data of [8], according to which 1,2-bis(*p*-phenylene)ethane bridge connecting the arylimino substituents in [1,2-bis(arylimino)acenaphthene]-nickel(II) dibromide makes the modified complex

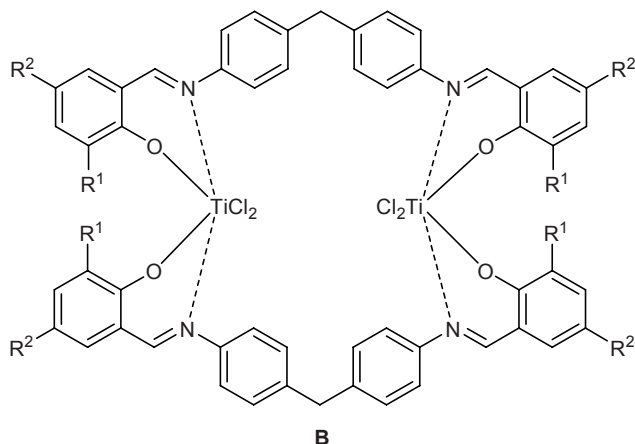
* For communication XI, see [1].

stable and highly active, as well as capable of promoting living polymerization at a considerably higher temperature (75°C) as compared to acyclic analog (0°C).

Clarkson et al. [9] showed that deactivation of complexes with bridged arylimine substituents and those having no such bridges follow different mechanisms. Presumably, structural variation of the bridging fragment could displace the maximal catalytic activity toward higher temperature by analogy with bridged metallocenes [10]. Ishii et al. [11] found that the catalytic activity of [6,6'-polymethylenebis(iminomethyl)-bis(2-R-phenoxy)]zirconium(IV) dichloride **A** in the polymerization of ethylene increases in parallel with the number of methylene units in the bridge. The catalytic activity also increases as the temperature rises, though the molecular weight of the polymerization product decreases.



While performing systematic studies on the effect of structural modification of metal complexes and their catalytic activity [12, 13] we found that bis{4,4'-methylenebis(*p*-phenylene)iminomethyl}bis(2-R¹-4-R²-phenoxy)}dititanium(IV) tetrachloride (**B**) even at 70°C ensures very effective preparation of linear polyethylene with high and ultrahigh molecular weight and increased melting point [13]. In the present work we examined the synthesis of tetradentate ligands containing various diimine bridges with a view to make them accessible for the preparation of bridged phen-

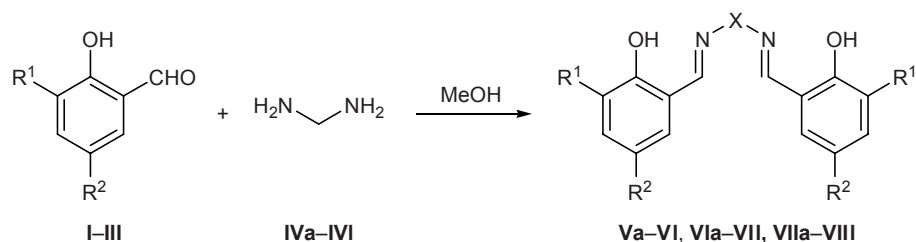


oxy-imine complexes. The synthetic procedure was based on the reaction of substituted salicylaldehydes with primary diamines, some examples of which have already been reported [6, 9, 11, 14]. We have extended this procedure to the synthesis of new bis-salicylaldehyde imine ligands **V–VII** and optimized conditions of their synthesis.

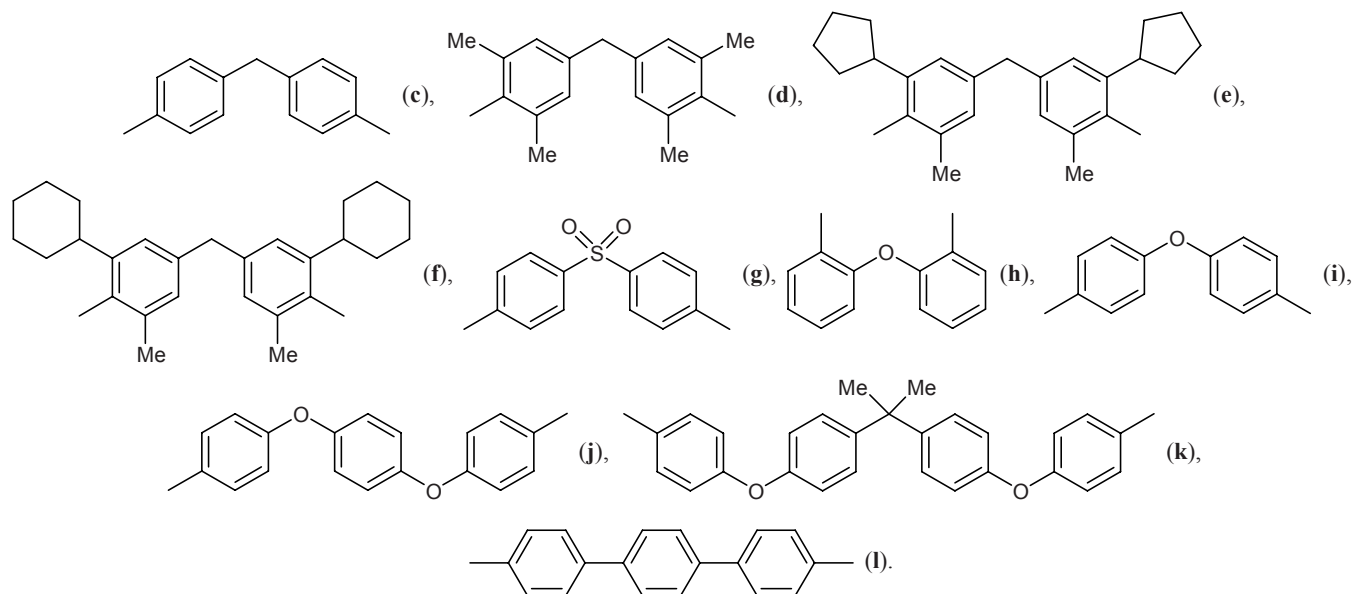
Aldehydes **I–III** reacted with 1,4-diaminobutane (**IVa**) and 1,6-diaminohexane (**IVb**) in methanol at room temperature in the absence of a catalyst, and the corresponding diimines **V–VII** (**a**, **b**) were formed in 5–6 min in quantitative yield (Scheme 1). The reactions with diaminodiphenylmethanes **IVc–IVf**, 4,4'-diaminodiphenyl sulfone (**IVg**), and *o,o'*-diaminodiphenyl ether (**IVh**) were carried out by heating in boiling methanol in the presence of a catalytic amount of formic acid, the reaction time ranged from 1 to 4 h, and the yields of diimines **Vc–Vh**, **VIc–VIh**, and **VIIc–VIIh** were 91–99%. The condensation of aldehydes **I–III** at both amino groups of *p,p'*-diaminodiphenyl ether (**IVi**) and diamines **IVj**, **IVk**, and **IVl** was possible only on prolonged heating of the reactants in methanol–methylene chloride (1:1) in the presence of a catalytic amount of formic acid. The yields of bis-salicylaldehyde imines **Vi–Vi**, **Vii–Vii**, and **Viii–Viii** ranged from 81 to 94%. When the reactions with diamines **IVi–IVl** were carried out in methanol, the process stopped at the stage of formation of the corresponding poorly soluble monoimines.

The structure of compounds **V–VII** was confirmed by analytical and spectral data. According to the ¹H NMR spectra, the isolated Schiff bases were pure *E* isomers. Compounds **V–VII** displayed in the ¹H NMR spectra singlets from the N=CH protons at δ 8.02–9.03 ppm and broadened singlets from the hydroxy protons in the region δ 12.33–14.15 ppm; the downfield position of the latter indicates formation of intramolecular hydrogen bond between the OH proton and imino nitrogen atom. In the ¹H NMR spectra of **VIa–VII**, protons in the *tert*-butyl groups resonated as singlets at δ 1.40–1.47 ppm, while the spectra of diimines **VIIa–VIII** contained two singlets from the *tert*-butyl groups, at δ 1.24–1.34 and 1.35–1.49 ppm. Methylene protons in the cycloalkyl substituents in Schiff bases **Ve**, **Vf**, **VIe**, **VIe**, **VIIe**, and **VIIe** gave multiplets in the region δ 1.11–2.05 ppm, and the cycloalkyl CH protons appeared as multiplets at δ 2.50–3.18 ppm. Diimines **Vd–Vf**, **VId–VIe**, and **VIIe–VIIe** showed singlets from methyl protons at δ 2.12–2.20 ppm. Aromatic protons resonated in the spectra of **V–VII** in the region δ 6.44–8.51 ppm. Singlets from the bridging

Scheme 1.



I, V, $R^1 = R^2 = \text{H}$; **II, VI**, $R^1 = t\text{-Bu}$, $R^2 = \text{H}$; **III, VII**, $R^1 = R^2 = t\text{-Bu}$; $X = (\text{CH}_2)_4$ (**a**), $(\text{CH}_2)_6$ (**b**),



CH_2 groups in diimines **Vc-Vf**, **Vlc-Vlf**, and **Vllc-Vllf** derived from diaminodiphenylmethanes were located at δ 3.77–4.01 ppm, while protons in the bridging methylene groups in compounds **Va**, **Vb**, **Vla**, **Vlb**, **Vlla**, and **Vllb** derived from diaminoalkanes **IVa** and **IVb** resonated as multiplets at δ 1.39–1.92 ppm and triplets (NCH_2) at δ 3.61–3.70 ppm.

The IR spectra of Schiff bases **Vc-VI**, **Vlc-VII**, and **Vllc-VIII** contained a strong absorption band in the region 1614–1623 cm^{-1} due to stretching vibrations of the $\text{C}=\text{N}$ bonds. The corresponding absorption band in the spectra of polymethylene-bridged derivatives **Va**, **Vb**, **Vla**, **Vlb**, **Vlla**, and **Vllb** was displaced to higher frequencies (1632–1635 cm^{-1}). The mass spectra of all ligands **V-VII** were characterized by the presence of strong molecular ion peaks.

EXPERIMENTAL

The ^1H NMR spectra were recorded on a Bruker WP-200 SY instrument at 200.13 MHz using hexamethyldisiloxane as internal reference. The IR spectra

were measured on a Vector 22 spectrometer from samples prepared as KBr pellets. The progress of reactions was monitored, and the purity of products was checked, by TLC on Silufol UV-254 plates using chloroform as eluent. The elemental compositions were determined on a Carlo Erba 1106 CHN analyzer, and their formulas were calculated from the high-resolution mass spectra which were recorded on a Finnigan MAT 8200 instrument. The melting points were determined by heating samples placed between glass plates at a rate of 1 deg/min.

Initial 2-cyclopentyl- and 2-cyclohexyl-6-methyl-anilines were synthesized according to the procedure described in [15].

4,4'-Methylenebis(2,6-dimethylaniline) (IVd). A mixture of 3.8 g (31.35 mmol) of 2,6-dimethylaniline and 6.2 ml of water was heated to 95°C, 2.6 ml of concentrated hydrochloric acid was added, the mixture was stirred for 10 min at 95°C, 0.45 g (15 mmol) of paraformaldehyde was added, and the mixture was stirred for 6 h at 100°C. The mixture was cooled, diluted with 10 ml of water, neutralized with a 20%

aqueous solution of potassium hydroxide, and extracted with diethyl ether (3×50 ml). The extract was washed with water and dried over MgSO₄, the solvent was distilled off on a rotary evaporator, and the solid residue was recrystallized from methanol. Yield 3.31 g (87%), mp 114–115°C [16].

4,4'-Methylenebis(2-cyclopentyl-6-methylaniline) (IVe) was synthesized in a similar way from 2-cyclopentyl-6-methylaniline. Yield 80%, mp 94–95°C. ¹H NMR spectrum, δ, ppm: 1.50–2.02 m (16H, CH₂), 2.06 s (6H, Me), 2.39 m (2H, CH), 3.35 br.s (4H, NH₂), 3.59 s (2H, CH₂), 6.54 s (2H, H_{arom}), 6.66 s (2H, H_{arom}). Found: [M]⁺ 362.28544. C₂₅H₃₄N₂. Calculated: M 362.28476.

4,4'-Methylenebis(2-cyclohexyl-6-methylaniline) (IVf) was synthesized in a similar way from 6-cyclohexyl-2-methylaniline. Yield 82%, mp 139–140°C. ¹H NMR spectrum, δ, ppm: 1.20–1.49 m (12H, CH₂), 1.60–1.95 m (8H, CH₂), 2.09 s (6H, Me), 2.88 m (2H, CH), 3.34 br.s (4H, NH₂), 3.65 s (2H, CH₂), 6.60 s (2H, H_{arom}), 6.72 s (2H, H_{arom}). Found: [M]⁺ 390.30323. C₂₇H₃₈N₂. Calculated: M 390.30348.

Schiff bases Va, Vb, VIa, VIb, VIIa, and VIIb (general procedure). Diamine IVa or IVb, 1 mmol, was added to a solution of 2 mmol of aldehyde I–III in 20 ml of methanol, and the mixture was stirred for 10 min at room temperature (until the initial compounds disappeared according to the TLC data). The light yellow precipitate was filtered off and washed with 2 ml of cold methanol.

2,2'-[Tetramethylenebis(iminomethyl)]diphenol (Va). Yield 98%, mp 87–89°C; published data [17]: mp 90°C. IR spectrum: ν 1632 cm⁻¹ (N=C). ¹H NMR spectrum (acetone-*d*₆), δ, ppm: 1.72–1.83 m (4H, CH₂), 3.66 t (4H, NCH₂, *J* = 7.5 Hz), 6.80–6.89 m (4H, H_{arom}), 7.22–7.38 m (4H, H_{arom}), 8.50 s (2H, N=CH), 13.39 br.s (2H, OH). Found: [M]⁺ 296.16264. C₁₈H₂₀N₂O₂. Calculated: M 296.16370.

2,2'-[Hexamethylenebis(iminomethyl)]diphenol (Vb). Yield 97%, mp 74–75°C; published data [17]: mp 73°C. IR spectrum: ν 1633 cm⁻¹ (N=C). ¹H NMR spectrum (acetone-*d*₆), δ, ppm: 1.39–1.53 m (4H, CH₂), 1.62–1.79 m (4H, CH₂), 3.61 t (4H, NCH₂, *J* = 7.6 Hz), 6.79–6.90 m (4H, H_{arom}), 7.23–7.37 m (4H, H_{arom}), 8.48 s (2H, N=C), 13.42 br.s (2H, OH). Found: [M]⁺ 324.18342. C₂₀H₂₄N₂O₂. Calculated: M 324.18377.

6,6'-[Tetramethylenebis(iminomethyl)]bis(2-*tert*-butylphenol) (VIa). Yield 95%, mp 146–147°C. IR spectrum: ν 1632 cm⁻¹ (N=C). ¹H NMR spectrum

(CCl₄), δ, ppm: 1.40 s (18H, *t*-Bu), 1.81–1.92 m (4H, CH₂), 3.65 t (4H, NCH₂, *J* = 7.6 Hz), 6.68 t (2H, H_{arom}, *J* = 7.5 Hz), 6.95 d.d (2H, H_{arom}, *J* = 7.5, 1.5 Hz), 7.20 d.d (2H, H_{arom}, *J* = 7.5, 1.5 Hz), 8.30 s (2H, N=CH), 13.55 br.s (2H, OH). Found: [M]⁺ 408.27780. C₂₆H₃₆N₂O₂. Calculated: M 408.27776.

6,6'-[Hexamethylenebis(iminomethyl)]bis(2-*tert*-butylphenol) (VIb). Yield 97%, mp 73–74°C. IR spectrum: ν 1633 cm⁻¹ (N=C). ¹H NMR spectrum (CDCl₃), δ, ppm: 1.40 s (18H, *t*-Bu), 1.42–1.53 m (4H, CH₂), 1.65–1.82 m (4H, CH₂), 3.65 t (4H, NCH₂, *J* = 7.5 Hz), 6.71 t (2H, H_{arom}, *J* = 7.8 Hz), 6.99 d.d (2H, H_{arom}, *J* = 7.8, 1.8 Hz), 7.22 d.d (2H, H_{arom}, *J* = 7.8, 1.8 Hz), 8.29 s (2H, N=CH), 13.84 br.s (2H, OH). Found: [M]⁺ 436.30935. C₂₈H₄₀N₂O₂. Calculated: M 436.30896.

6,6'-[Tetramethylenebis(iminomethyl)]bis(2,4-*di-tert*-butylphenol) (VIIa). Yield 94%, mp 143–144°C; published data [18]: mp 144–145°C. IR spectrum: ν 1635 cm⁻¹ (N=C). ¹H NMR spectrum (acetone-*d*₆), δ, ppm: 1.28 s (18H, *t*-Bu), 1.41 s (18H, *t*-Bu), 1.79–1.88 m (4H, CH₂), 3.70 t (4H, NCH₂, *J* = 7.0 Hz), 7.23 d (2H, H_{arom}, *J* = 2.0 Hz), 7.39 d (2H, H_{arom}, *J* = 2.0 Hz), 8.54 s (2H, N=CH), 13.99 br.s (2H, OH). Found: [M]⁺ 520.40348. C₃₄H₅₂N₂O₂. Calculated: M 520.40286.

6,6'-[Hexamethylenebis(iminomethyl)]bis(2,4-*di-tert*-butylphenol) (VIIb). Yield 95%, mp 125–126°C; published data [18]: mp 120–122°C. IR spectrum: ν 1634 cm⁻¹ (N=C). ¹H NMR spectrum (acetone-*d*₆), δ, ppm: 1.26 s (18H, *t*-Bu), 1.39 s (18H, *t*-Bu), 1.45–1.53 m (4H, CH₂), 1.60–1.79 m (4H, CH₂), 3.61 t (4H, NCH₂, *J* = 7.0 Hz), 7.21 d (2H, H_{arom}, *J* = 2.0 Hz), 7.37 d (2H, H_{arom}, *J* = 2.0 Hz), 8.49 s (2H, N=CH), 14.02 br.s (2H, OH). Found: [M]⁺ 548.434615. C₃₆H₅₆N₂O₂. Calculated: M 548.43415.

Schiff bases Vc–Vh, VIc–Vh, and VIIc–VIIh (general procedure). A mixture of 2 mmol of aldehyde I–III, 1 mmol of diamine IVc–IVh, 20 ml of methanol, and 10 mg of 99% formic acid was heated under reflux with stirring for 1–4 h (until the initial compounds disappeared according to the TLC data). The mixture was cooled, and the light yellow precipitate was filtered off and washed with 2 ml of cold methanol.

2,2'-[Methylenebis(*p*-phenylene)iminomethyl]diphenol (Vc). Yield 93%, mp 216–217°C. IR spectrum: ν 1619 (N=C). ¹H NMR spectrum (DMSO-*d*₆), δ, ppm: 4.01 s (2H, CH₂), 6.85–7.62 m (16H, H_{arom}),

8.90 s (2H, N=CH), 13.02 br.s (2H, OH). Found: $[M]^+$ 406.16812. $C_{27}H_{22}N_2O_2$. Calculated: M 406.16812.

2,2'-{Methylenebis[(3,5-dimethyl-1,4-phenylene)iminomethyl]}diphenol (Vd). Yield 91%, mp 132–133°C. IR spectrum: ν 1622 cm^{-1} (N=C). 1H NMR spectrum (acetone- d_6), δ , ppm: 2.15 s (12H, Me), 3.79 s (2H, CH₂), 6.84–6.95 m (8H, H_{arom}), 7.29–7.47 m (4H, H_{arom}), 8.44 s (2H, N=CH), 12.89 br.s (2H, OH). Found: $[M]^+$ 462.23093. $C_{31}H_{30}N_2O_2$. Calculated: M 462.23071.

2,2'-{Methylenebis[(3-cyclopentyl-5-methyl-1,4-phenylene)iminomethyl]}diphenol (Ve). Yield 92%, mp 77–78°C. IR spectrum: ν 1623 cm^{-1} (N=C). 1H NMR spectrum (acetone- d_6), δ , ppm: 1.49–1.99 m (16H, CH₂), 2.13 s (6H, Me), 2.95–3.12 m (2H, CH), 3.84 s (2H, CH₂), 6.83–7.09 m (8H, H_{arom}), 7.28–7.45 m (4H, H_{arom}), 8.38 s (2H, N=CH), 12.85 br.s (2H, OH). Found: $[M]^+$ 570.32464. $C_{39}H_{42}N_2O_2$. Calculated: M 570.32461.

2,2'-{Methylenebis[(3-cyclohexyl-5-methyl-1,4-phenylene)iminomethyl]}diphenol (Vf). Yield 98%, mp 154–155°C. IR spectrum: ν 1621 cm^{-1} (N=C). 1H NMR spectrum (acetone- d_6), δ , ppm: 1.15–1.85 m (20H, CH₂), 2.12 s (6H, Me), 2.57–2.75 m (2H, CH), 3.89 s (2H, CH₂), 6.90–7.18 m (8H, H_{arom}), 7.36–7.57 m (4H, H_{arom}), 8.49 s (2H, N=CH), 12.98 s (2H, OH). Found: $[M]^+$ 598.35551. $C_{41}H_{46}N_2O_2$. Calculated: M 598.35591.

2,2'-{Sulfonylbis[(*p*-phenylene)iminomethyl]}diphenol (Vg). Yield 96%, mp 268–269°C. IR spectrum: ν 1618 cm^{-1} (N=C). 1H NMR spectrum (DMSO- d_6), δ , ppm: 6.97 m (4H, H_{arom}), 7.43 t.d (2H, H_{arom}, J = 7.8, 2.0 Hz), 7.56 d (4H, H_{arom}, J = 7.8 Hz), 7.67 d.d (2H, H_{arom}, J = 7.8, 2.0 Hz), 8.02 d (4H, H_{arom}, J = 7.8 Hz), 8.92 s (2H, N=CH), 12.33 br.s (2H, OH). Found: $[M]^+$ 456.11544. $C_{26}H_{20}N_2O_4S$. Calculated: M 456.11437.

2,2'-{Oxybis[(*o*-phenylene)iminomethyl]}diphenol (Vh). Yield 99%, mp 181–182°C. IR spectrum: ν 1619 cm^{-1} (N=C). 1H NMR spectrum (acetone- d_6), δ , ppm: 6.79–6.98 m (6H, H_{arom}), 7.19–7.51 m (10H, H_{arom}), 8.90 s (2H, N=CH), 13.09 br.s (2H, OH). Found: $[M]^+$ 408.14845. $C_{26}H_{20}N_2O_3$. Calculated: M 408.14738.

6,6'-{Methylenebis[(*p*-phenylene)iminomethyl]}bis(2-*tert*-butylphenol) (VIc). Yield 94%, mp 96–97°C. IR spectrum: ν 1616 cm^{-1} (N=C). 1H NMR spectrum (CDCl₃), δ , ppm: 1.44 s (18H, *t*-Bu), 4.00 s (2H, CH₂), 6.79 t (2H, H_{arom}, J = 7.7 Hz), 7.08–7.37 m (12H, H_{arom}), 8.57 s (2H, N=CH), 13.73 br.s (2H, OH).

Found: $[M]^+$ 518.29359. $C_{35}H_{38}N_2O_2$. Calculated: M 518.29331.

6,6'-{Methylenebis[(3,5-dimethyl-1,4-phenylene)iminomethyl]}bis(2-*tert*-butylphenol) (VIId). Yield 93%, mp 170–171°C. IR spectrum: ν 1617 cm^{-1} (N=C). 1H NMR spectrum (CDCl₃), δ , ppm: 1.45 s (18H, *t*-Bu), 2.22 s (12H, Me), 3.79 s (2H, CH₂), 6.72–6.93 m (6H, H_{arom}), 7.09 d.d (2H, H_{arom}, J = 7.8, 1.8 Hz), 7.34 d.d (2H, H_{arom}, J = 7.8, 1.8 Hz), 8.31 s (2H, N=CH), 13.55 br.s (2H, OH). Found: $[M]^+$ 574.35694. $C_{39}H_{46}N_2O_2$. Calculated: M 574.35591.

6,6'-{Methylenebis[(3-cyclopentyl-5-methyl-1,4-phenylene)iminomethyl]}bis(2-*tert*-butylphenol) (VIe). Yield 91%, mp 212–213°C. IR spectrum: ν 1616 cm^{-1} (N=C). 1H NMR spectrum (CDCl₃), δ , ppm: 1.46 s (18H, *t*-Bu), 1.51–2.05 m (16H, CH₂), 2.17 s (6H, Me), 2.95–3.13 m (2H, CH), 3.84 s (2H, CH₂), 6.70–7.02 m (6H, H_{arom}), 7.10 d (2H, H_{arom}, J = 7.8 Hz), 7.34 d (2H, H_{arom}, J = 7.8 Hz), 8.29 s (2H, N=CH), 13.55 br.s (2H, OH). Found: $[M]^+$ 682.44931. $C_{47}H_{58}N_2O_2$. Calculated: M 682.44980.

6,6'-{Methylenebis[(3-cyclohexyl-3-methyl-1,4-phenylene)iminomethyl]}bis(2-*tert*-butylphenol) (VIIf). Yield 91%, mp 239–240°C. IR spectrum: ν 1616 cm^{-1} (N=H). 1H NMR spectrum (CDCl₃), δ , ppm: 1.11–1.39 m (8H, CH₂), 1.46 s (18H, *t*-Bu), 1.55–1.85 m (12H, CH₂), 2.15 s (6H, Me), 2.50–2.71 m (2H, CH), 3.87 s (2H, CH₂), 6.75–7.03 m (6H, H_{arom}), 7.14 d.d (2H, H_{arom}, J = 7.6, 1.8 Hz), 7.38 d.d (2H, H_{arom}, J = 7.6, 1.8 Hz), 8.29 s (2H, N=CH), 13.70 br.s (2H, OH). Found: $[M]^+$ 710.48320. $C_{49}H_{62}N_2O_2$. Calculated: M 710.48110.

6,6'-{Sulfonylbis[(*p*-phenylene)iminomethyl]}bis(2-*tert*-butylphenol) (VIg). Yield 91%, mp 178–179°C. IR spectrum: ν 1614 cm^{-1} (N=C). 1H NMR spectrum (CDCl₃), δ , ppm: 1.43 s (18H, *t*-Bu), 6.83 t (2H, H_{arom}, J = 7.8 Hz), 7.19 d.d (2H, H_{arom}, J = 7.8, 2.0 Hz), 7.29–7.42 m (6H, H_{arom}), 7.97 d (4H, H_{arom}, J = 8.0 Hz), 8.55 s (2H, N=CH), 13.16 br.s (2H, OH). Found: $[M]^+$ 568.23930. $C_{34}H_{36}N_2O_4S$. Calculated: M 568.23956.

6,6'-{Oxybis[(*o*-phenylene)iminomethyl]}bis(2-*tert*-butylphenol) (VIh). Yield 93%, mp 127–128°C. IR spectrum: ν 1614 cm^{-1} (N=CH). 1H NMR spectrum (CDCl₃), δ , ppm: 1.40 s (18H, *t*-Bu), 6.69 t (2H, H_{arom}, J = 8.0 Hz), 6.92–7.28 m (12H, H_{arom}), 8.61 s (2H, N=CH), 13.50 br.s (2H, OH). Found: $[M]^+$ 520.27307. $C_{34}H_{36}N_2O_3$. Calculated: M 520.27258.

6,6'-{Methylenebis[(*p*-phenylene)iminomethyl]}bis(2,4-di-*tert*-butylphenol) (VIIc). Yield 98%,

mp 196–197°C. IR spectrum: ν 1620 cm^{-1} (N=C). ^1H NMR spectrum (CDCl_3), δ , ppm: 1.30 s (18H, *t*-Bu), 1.44 s (18H, *t*-Bu), 4.01 s (2H, CH_2), 7.13 d (2H, H_{arom} , $J = 2.0$ Hz), 7.16–7.23 m (8H, H_{arom}), 7.37 d (2H, H_{arom} , $J = 2.0$ Hz), 8.59 s (2H, N=CH), 13.54 br.s (2H, OH). Found: $[M]^+$ 630.41786. $\text{C}_{43}\text{H}_{54}\text{N}_2\text{O}_2$. Calculated: M 630.41850.

6,6'-{Methylenebis[(3,5-dimethyl-1,4-phenylene)iminomethyl]}bis(2,4-di-*tert*-butylphenol) (VIId). Yield 93%, mp 147–148°C. IR spectrum: ν 1622 cm^{-1} (N=C). ^1H NMR spectrum (CDCl_3), δ , ppm: 1.30 s (18H, *t*-Bu), 1.45 s (18H, *t*-Bu), 2.27 s (12H, Me), 3.77 s (2H, CH_2), 6.61–6.92 m (4H, H_{arom}), 7.01 d (2H, H_{arom} , $J = 1.5$ Hz), 7.35 d (2H, H_{arom} , $J = 1.5$ Hz), 8.31 s (2H, N=CH), 13.29 br.s (2H, OH). Found, %: C 82.32; H 8.97; N 4.10. $[M]^+$ 686.5. $\text{C}_{47}\text{H}_{62}\text{N}_2\text{O}_2$. Calculated, %: C 82.23; H 9.04; N 4.08. M 686.5.

6,6'-{Methylenebis[(3-cyclopentyl-3-methyl-1,4-phenylene)iminomethyl]}bis(2,4-di-*tert*-butylphenol) (VIIe). Yield 94%, mp 176–177°C. IR spectrum: ν 1621 cm^{-1} (N=C). ^1H NMR spectrum (CDCl_3), δ , ppm: 1.30 s (18H, *t*-Bu), 1.45 s (18H, *t*-Bu), 1.51–2.05 m (16H, CH_2), 2.17 s (6H, Me), 2.95–3.18 m (2H, CH), 3.83 s (2H, CH_2), 6.79 s (2H, H_{arom}), 6.95 s (2H, H_{arom}), 7.03 s (2H, H_{arom}), 7.38 s (2H, H_{arom}), 8.29 s (2H, N=CH), 13.35 br.s (2H, OH). Found, %: C 83.09; H 9.26; N 3.59. $[M]^+$ 794. $\text{C}_{55}\text{H}_{74}\text{N}_2\text{O}_2$. Calculated, %: C 83.12; H 9.32; N 3.53. M 794.

6,6'-{Methylenebis[(3-cyclohexyl-5-methyl-1,4-phenylene)iminomethyl]}bis(2,4-di-*tert*-butylphenol) (VIIIf). Yield 92%, mp 110–112°C. IR spectrum: ν 1621 cm^{-1} (N=C). ^1H NMR spectrum (CDCl_3), δ , ppm: 1.31 s (18H, *t*-Bu), 1.32–1.43 m (8H, CH_2), 1.46 s (18H, *t*-Bu), 1.58–1.96 m (12H, CH_2), 2.20 s (6H, Me), 2.50–2.73 m (2H, CH), 3.82 s (2H, CH_2), 6.76 s (2H, H_{arom}), 6.88 s (2H, H_{arom}), 7.00 d (2H, H_{arom} , $J = 1.5$ Hz), 7.35 d (2H, H_{arom} , $J = 1.5$ Hz), 8.29 s (2H, N=CH), 13.35 br.s (2H, OH). Found, %: C 83.10; H 9.49; N 3.46. $\text{C}_{57}\text{H}_{78}\text{N}_2\text{O}_2$. Calculated, %: C 83.21; H 9.49; N 3.41.

6,6'-{Sulfonylbis[(*p*-phenylene)iminomethyl]}bis(2,4-di-*tert*-butylphenol) (VIIg). Yield 91%, mp 172–173°C. IR spectrum: ν 1618 cm^{-1} (N=C). ^1H NMR spectrum (acetone- d_6), δ , ppm: 1.31 s (18H, *t*-Bu), 1.45 s (18H, *t*-Bu), 7.47–7.64 m (8H, H_{arom}), 8.08–8.12 m (4H, H_{arom}), 8.95 s (2H, N=CH), 13.36 br.s (2H, OH). Found, %: C 83.10; H 9.35; N 3.47. $[M]^+$ 680. $\text{C}_{34}\text{H}_{36}\text{N}_2\text{O}_4\text{S}$. Calculated, %: C 83.21; H 9.49; N 3.41. M 680.

6,6'-{Oxybis[(*o*-phenylene)iminomethyl]}bis(2,4-di-*tert*-butylphenol) (VIIh). Yield 98%, mp 155–156°C. IR spectrum: ν 1618 cm^{-1} (N=C). ^1H NMR spectrum (acetone- d_6), δ , ppm: 1.24 s (18H, *t*-Bu), 1.35 s (18H, *t*-Bu), 6.96–7.54 m (12H, H_{arom}), 8.93 s (2H, N=CH), 13.72 br.s (2H, OH). Found: $[M]^+$ 632.39695. $\text{C}_{42}\text{H}_{52}\text{N}_2\text{O}_3$. Calculated: M 632.39777.

Schiff bases Vi–VI, VII–VII, and VIII–VIII (general procedure). A mixture of 2 mmol of aldehyde I–III, 1 mmol of diamine IVi–IVi, 10 ml of methanol, 10 ml of methylene chloride, and 10 mg of 99% formic acid was heated for 8–12 h under reflux with stirring (until the initial compounds disappeared according to the TLC data). The solvent was distilled off on a rotary evaporator, the residue was ground with 5 ml of methanol, and the light yellow precipitate was filtered off and washed with 2 ml of cold methanol.

2,2'-{Oxybis[(*p*-phenylene)iminomethyl]}diphenol (Vi). Yield 84%, mp 213–214°C; published data [19]: mp 210–211°C. IR spectrum: ν 1621 cm^{-1} (N=C). ^1H NMR spectrum (CDCl_3 –DMSO- d_6), δ , ppm: 6.44–6.68 m (8H, H_{arom}), 6.85–7.04 m (8H, H_{arom}), 8.27 s (2H, N=CH), 12.74 br.s (2H, OH). Found: $[M]^+$ 408.14740. $\text{C}_{26}\text{H}_{20}\text{N}_2\text{O}_3$. Calculated: M 408.14738.

2,2'-{*p*-Phenylenedioxybis[(*p*-phenylene)iminomethyl]}diphenol (Vj). Yield 83%, mp 231–232°C. IR spectrum: ν 1620 cm^{-1} (N=C). ^1H NMR spectrum (DMSO- d_6), δ , ppm: 6.58–7.15 m (12H, H_{arom}), 7.36–7.68 m (8H, H_{arom}), 8.94 s (2H, N=CH), 14.04 br.s (2H, OH). Found: $[M]^+$ 500.17299. $\text{C}_{32}\text{H}_{24}\text{N}_2\text{O}_4$. Calculated: M 500.17359.

2,2'-{Propane-2,2-diylbis[(*p*-phenylene)oxy-(*p*-phenylene)iminomethyl]}diphenol (Vk). Yield 81%, mp 122–123°C. IR spectrum: ν 1618 cm^{-1} (N=C). ^1H NMR spectrum (CDCl_3), δ , ppm: 1.69 s (6H, Me), 6.91–7.06 (12H, H_{arom}), 7.20–7.38 m (12H, H_{arom}), 8.60 s (2H, N=CH), 13.21 br.s (2H, OH). Found: $[M]^+$ 618.25140. $\text{C}_{41}\text{H}_{34}\text{N}_2\text{O}_4$. Calculated: M 618.25184.

2,2'-[(4,4''-*p*-Terphenylene)bis(iminomethyl)]-diphenol (VI). Yield 84%, mp 309–310°C. IR spectrum: ν 1622 cm^{-1} (N=C). ^1H NMR spectrum (DMSO- d_6), δ , ppm: 6.66 t (2H, H_{arom} , $J = 7.7$ Hz), 6.93–7.02 m (4H, H_{arom}), 7.34–7.81 m (14H, H_{arom}), 9.03 s (2H, N=CH), 13.10 br.s (2H, OH). Found: $[M]^+$ 468.18320. $\text{C}_{32}\text{H}_{24}\text{N}_2\text{O}_2$. Calculated: M 468.18377.

6,6'-{Oxybis[(*p*-phenylene)iminomethyl]}bis(2-*tert*-butylphenol) (VIi). Yield 90%, mp 146–147°C. IR spectrum: ν 1613 cm^{-1} (N=C). ^1H NMR

spectrum (CDCl₃), δ , ppm: 1.47 s (18H, *t*-Bu), 6.87 t (2H, H_{arom} , $J = 7.6$ Hz), 7.07 d (4H, H_{arom} , $J = 7.6$ Hz), 7.25 d (2H, H_{arom} , $J = 7.6$ Hz), 7.30 d (4H, H_{arom} , $J = 7.6$ Hz), 7.39 d (2H, H_{arom} , $J = 7.6$ Hz), 8.62 s (2H, N=CH), 13.83 s (2H, OH). Found: $[M]^+$ 520.27260. C₃₄H₃₆N₂O₃. Calculated: M 520.27258.

6,6'-{*p*-Phenylenedioxybis(*p*-phenylene)iminomethyl}bis(2-*tert*-butylphenol) (VIj). Yield 94%, mp 198–199°C. IR spectrum: ν 1614 cm⁻¹ (N=C). ¹H NMR spectrum (CDCl₃), δ , ppm: 1.47 s (18H, *t*-Bu), 6.87 t (2H, H_{arom} , $J = 7.7$ Hz), 7.01–7.33 m (14H, H_{arom}), 7.38 d (2H, H_{arom} , $J = 7.7$ Hz), 8.62 s (2H, N=CH), 13.89 br.s (2H, OH). Found: $[M]^+$ 612.29940. C₄₀H₄₀N₂O₄. Calculated: M 612.29879.

6,6'-{Propane-2,2-diylbis(*p*-phenyleneoxy(*p*-phenylene)iminomethyl}bis(2-*tert*-butylphenol) (VIk). Yield 85%, mp 83–84°C. IR spectrum: ν 1612 cm⁻¹ (N=C). ¹H NMR spectrum (CDCl₃), δ , ppm: 1.46 s (18H, *t*-Bu), 1.69 s (6H, Me), 6.86 t (2H, H_{arom} , $J = 7.8$ Hz), 6.93 d (4H, H_{arom} , $J = 8.0$), 7.04 d (4H, H_{arom} , $J = 8.0$), 7.19–7.29 m (10H, H_{arom}), 7.37 d.d (2H, H_{arom} , $J = 8.0$, 2.0 Hz), 8.61 s (2H, N=CH), 13.89 br.s (2H, OH). Found: $[M]^+$ 730.37740. C₄₉H₅₀N₂O₄. Calculated: M 730.37703.

6,6'-[(4,4''-*p*-Terphenylene)bis(iminomethyl)]bis(2-*tert*-butylphenol) (VII). Yield 91%, mp 280–281°C. IR spectrum: ν 1614 cm⁻¹ (N=C). ¹H NMR spectrum (DMSO-*d*₆), δ , ppm: 1.43 s (18H, *t*-Bu), 6.64–6.70 m (2H, H_{arom}), 6.88–7.04 m (2H, H_{arom}), 7.32–7.81 m (14H, H_{arom}), 8.25 s (2H, N=CH), 14.15 br.s (2H, OH). Found: $[M]^+$ 580.30910. C₄₀H₄₀N₂O₂. Calculated: M 580.30896.

6,6'-{Oxybis(*p*-phenylene)iminomethyl}bis(2,4-di-*tert*-butylphenol) (VIIi). Yield 87%, mp 215–216°C; published data [20]: mp 210–212°C. IR spectrum: ν 1619 cm⁻¹ (N=C). ¹H NMR spectrum (CDCl₃), δ , ppm: 1.32 s (18H, *t*-Bu), 1.47 s (18H, *t*-Bu), 7.07 d (4H, H_{arom} , $J = 8.0$ Hz), 7.21 d (2H, H_{arom} , $J = 2.0$ Hz), 7.29 d (4H, H_{arom} , $J = 8.0$ Hz), 7.44 d (2H, H_{arom} , $J = 2.0$ Hz), 8.64 s (2H, N=CH), 13.67 s (2H, OH). Found: $[M]^+$ 632.39790. C₄₂H₅₂N₂O₃. Calculated: M 632.39777.

6,6'-{*p*-Phenylenedioxybis(*p*-phenylene)iminomethyl}bis(2,4-di-*tert*-butylphenol) (VIIj). Yield 87%, mp 240–241°C. IR spectrum: ν 1618 cm⁻¹ (N=C). ¹H NMR spectrum (CDCl₃), δ , ppm: 1.32 s (18H, *t*-Bu), 1.47 s (18H, *t*-Bu), 7.01–7.46 m (16H, H_{arom}), 8.63 s (2H, N=CH), 13.68 s (2H, OH). Found, %: C 79.61; H 7.84; N 3.87. $[M]^+$ 724. C₄₈H₅₆N₂O₄. Calculated, %: C 79.52; H 7.79; N 3.86. M 724.

6,6'-{Propane-2,2-diylbis(*p*-phenylene)oxy(*p*-phenylene)iminomethyl}bis(2,4-di-*tert*-butylphenol) (VIIk). Yield 88%, mp 91–92°C. IR spectrum: ν 1618 cm⁻¹ (N=C). ¹H NMR spectrum (CDCl₃), δ , ppm: 1.31 s (18H, *t*-Bu), 1.46 s (18H, *t*-Bu), 1.68 s (6H, Me), 6.93 d (4H, H_{arom} , $J = 7.8$ Hz), 7.04 d (4H, H_{arom} , $J = 7.8$), 7.18–7.28 m (10H, H_{arom}), 7.43 d (2H, H_{arom} , $J = 1.8$ Hz), 8.61 s (2H, N=CH), 13.63 br.s (2H, OH). Found, %: C 81.15; H 7.93; N 3.27. C₅₇H₆₆N₂O₄. Calculated, %: C 81.20; H 7.89; N 3.32.

6,6'-{(4,4''-*p*-Terphenylene)bis(iminomethyl)}bis(2,4-di-*tert*-butylphenol) (VIII). Yield 93%, mp 290–291°C. IR spectrum: ν 1618 cm⁻¹ (N=C). ¹H NMR spectrum (CDCl₃), δ , ppm: 1.34 s (18H, *t*-Bu), 1.49 s (18H, *t*-Bu), 7.25–7.29 m (6H, H_{arom}), 7.38 d (4H, H_{arom} , $J = 8.0$ Hz), 7.47 d (2H, H_{arom} , $J = 2.0$ Hz), 7.69–7.73 m (4H, H_{arom}), 8.70 s (2H, N=CH), 13.68 br.s (2H, OH). Found: $[M]^+$ 692.43390. C₄₈H₅₆N₂O₂. Calculated: M 692.43415.

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