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Molecules with potential applications for non-linear optics: the combination of ferrocene and azulene

Rudolf Herrmann [a,*], Bjørn Pedersen [a], Gabriele Wagner [1,a], Joo-Hack Youn [b]

[a] *Institut für Organische Chemie und Biochemie der Technischen Universität München, Lichtenbergstrasse 4, D-85747 Garching, Germany*
[b] *Department of Chemical Engineering, Sun Moon University, # 100 Kalsanri Tangjeong Myon, Asansi, Choong Nam 336-840, South Korea*

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Abstract

The synthesis of ferrocene derivatives connected by an imino bridge to the azulene moiety is described, and the properties of the compounds were studied (NMR, UV–vis, redox potentials). The first hyperpolarizability of one of the compounds was determined by hyper-Rayleigh scattering (HRS). © 1998 Elsevier Science S.A. All rights reserved.

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1. Introduction

The development of materials with non-linear optical (NLO) properties is an active topic of research with industrial applications for optical devices [1–4]. Connecting a donor and an acceptor moiety with a conjugated ('conducting') bridge is a well-established strategy to obtain materials with high values of the hyperpolarizability [5–8]. We reasoned that the combination of an organometallic fragment with well-known donor properties like ferrocene with an aromatic compound with an unusual electron distribution like azulene (which is a donor–acceptor combination in itself) might lead to molecules with unique properties and potentially enhanced NLO coefficients. We reported already on some new compounds of this type [9] and now wish to complement this study by an investigation of the optical and redox properties, including several new compounds.

2. Synthesis and properties of azulene derivatives of ferrocene

In our previous study [9], the high efficiency of ferrocene to stabilize carbocations in the α-position to the cyclopentadienyl ring was exploited to prepare the amine **3** with no conjugation between azulene and ferrocene. The acidity of the methyl protons in 4,6,8-trimethylazulene, on the other hand, gave access to the alcohols **1** and, by dehydration, to the alkene **2** with a conjugated bridge between the electron-deficient seven-membered ring of the azulene and the ferrocene donor. For compounds of the donor–acceptor type, the NLO properties depend strongly on the strength and orientation of the dipole moments. We therefore prepared new compounds by replacing the C=C double bond as a bridge by C=N double bonds (imines), and attaching it to the electron-rich five-membered ring of azulene (Fig. 1). Here, one could expect that the permanent dipole moment is enhanced in the compound **5** (derived from 1-aminoazulene), as the dipole of the C=N bond points in roughly the same direction as the dipole in the azolene–ferrocene backbone, while it should be lower in the compounds **6** and **7** (derived from aminoferrocene) where the C=N dipole points in the opposite

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* Corresponding author. Tel.: +49 89 28913325; fax: +49 89 289 12727.

[1] Present address: Centro de Química Estrutural, Instituto Superior Técnico, Av. Rovisco Pais, P-1096 Lisboa, Portugal.

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direction. The synthesis of the imines by the reaction of the amines with the corresponding aldehydes is straightforward, however, it turned out that the imine **5** (with the higher dipole moment) is of very limited stability and decomposes at room temperature within a few hours. For comparison, we prepared also the non-conjugated imine **8** from 1-ferrocenyl-2-methylpropylamine. Attempts to obtain isomers of the imine **5** by reacting 4- or 6-aminoazulene [10] with ferrocencarbaldehyde failed because the nitrogen lone pair of these aminoazulenes seems to be delocalized in the electron-accepting seven-membered ring, and hence the nitrogen atom does not have any nucleophilic properties which are necessary for the formation of imines from amines.

The imines **5–8** show the NMR patterns expected for ferrocene and azulene derivatives and allow for a complete assignment of the signals. As these compounds were designed for optical applications, their UV–vis spectra were studied in several solvents, and are collected, together with the alkene **2**, in Table 1.

The electronic spectra generally show weak absorptions in the visible range, but much stronger ones in the ultraviolet. An interesting feature is the solvatochromy of the bands in the visible range. The origin of these bands is assumed to be a charge transfer between the donor and the acceptor moiety, and they are considered to be mainly responsible for the hyperpolarizability, within the framework of the two-level model (or the more refined sum-overstates model (SOS) [11]. In many cases (but not always), a high solvatochromy of these bands is indicative of high values of the hyperpolarizability [12]. There is indeed a shift of 20 nm (482 → 502) in the alkene **2** when going from hexane to acetone as solvent, which is much higher than in azulene itself (6 nm difference from pentane to acetonitrile) [13]. Although one might expect a more pronounced effect in the imines due to the higher dipole moment of the C=N group, this is not the case; the shift is always lower. The solvatochromy is found also in the non-conjugated imine **8** which suggests that for the imines **5–7** there is almost no effect of the conjugated bridge connecting ferrocene and azulene on the charge transfer bands. This surprising result leads to the conclusion that in these special cases the traditional strategy for enhancing NLO effects by extending the conjugated bridge between donor and acceptor will probably not be successful.

3. Electrochemistry

To get more insight in the electronic properties of the compounds, we studied their redox properties by cyclovoltammetry. The results are collected in Table 2. For solubility reasons, THF was used as solvent for the imines, although acetonitrile allows to measure a wider

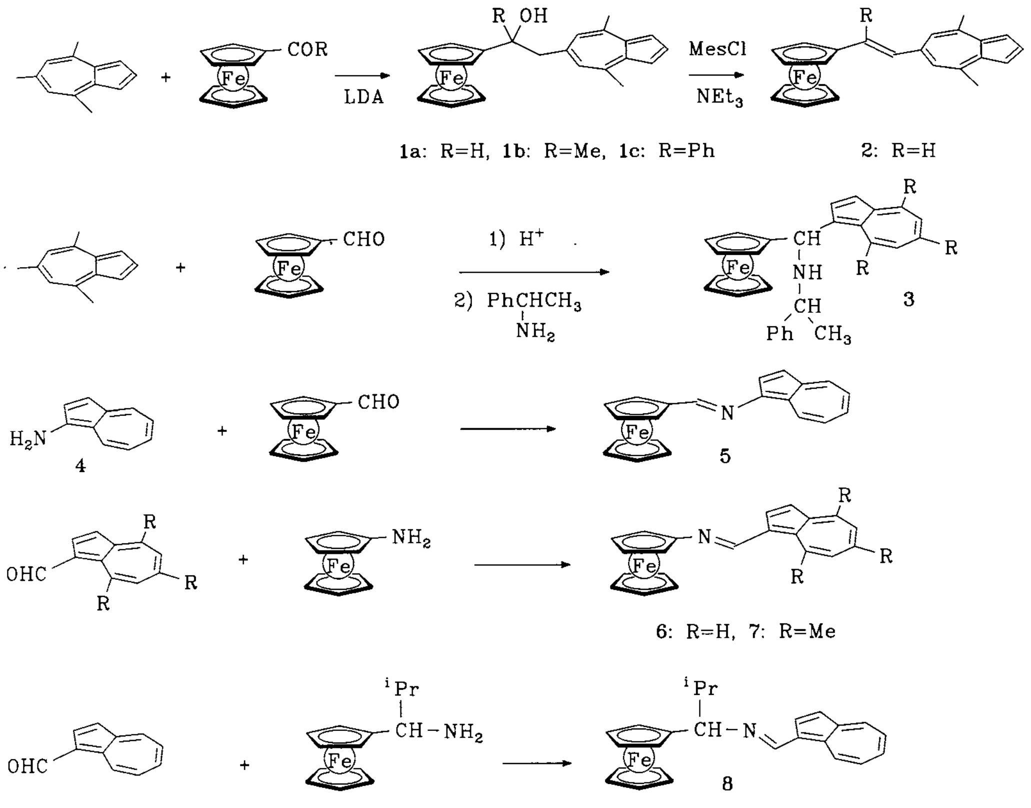

Fig. 1. Syntheses of ferrocene derivatives containing the azulene moiety.

Table 1
UV–vis spectra of ferrocene–azulene compounds, concentration 10^{-4} mol l^{-1}; λ_{max} in nm; ϵ in brackets

Compound	Solvent	λ_{max}	λ_{max}	λ_{max}	λ_{max}	λ_{max}
2	Hexane		482 (2090)	395 (6300)	332 (14000)	
	Acetone		502 (2870)	396 (8720)	334 (16000)	332 (12000)
5	Ether	740 (250)	646 (3030)	400 (11600)	381 (11600)	310 (11900)
	Acetone		639 (728)	399 (26100)	381 (24900)	
7	Dichloromethane		475 (4600)	399 (25600)	338 (35100)	
	acetone		480 (4118)	396 (23700)	334 (33330)	
	Formamide	639 (1460)		395 (10800)	323 (17900)	
8	Pentane	714 (52)	641 (155)	588 (200)	378 (6730)	309 (16000)
	Ether	710 (61)	635 (260)	584 (288)	378 (9940)	299 (31900)
	Acetone	700 (63)	630 (210)	581 (263)	377 (9650)	

range in the oxidations. For comparison, the starting materials azulene and 4,6,8-trimethylazulene were also included. The potentials are quoted relative to the ferrocene/ferrocenium redox couple.

The first anodic wave observed in the compounds containing ferrocene is reversible in all cases and found at a potential similar to that of ferrocene. No such wave is observed in azulene and 4, 6, 8-trimethylazulene. It is therefore reasonable to assign it to the ferrocene moiety. In acetonitrile, all compounds studied show an oxidation at more positive values (0.47–0.56 V) which is irreversible for the parent compound azulene and the amine **3**, but at least partially reversible in the other cases. In THF, this anodic wave is not observed as it falls in the range of electrolyte or solvent oxidation. For azulene and 4,6,8-trimethylazulene, very similar potentials for this oxidation were reported also by others [14] (0.51 and 0.46 V, respectively). Reductions are observed in all cases at values below -2.0 V which are reversible or at least partially reversible with the exception of the alkene **2**. The formation of reasonably stable monoanions by electrochemical reduction of aromatics (including azulene) is known for amine solvents [15], but due to different conditions, the redox potentials cannot be compared with the values obtained here. It seems, however, clear that the oxidation around 0.5 V and the reduction below -2.0 V occur in the azulene part of the molecules. The differences in the potentials reflect the electron withdrawing or donating properties of the substituents. Thus, azulene derivatives with donor substituents in the seven-membered ring show the lowest reduction potentials (≤ -2.18 V), while azulene itself and the imines **6** and **7** appear between -2.04 and -2.09 V. The potential of the trimethyl-substituted imine **7** is only slightly lower than that of its unsubstituted counterpart **6** indicating the dominating influence of the C=N double bond on the redox properties. In some cases, additional waves are observed which are either due to adsorption or to secondary reactions of the reduction or oxidation products. Without further data, no assignment to definite intermediates is possible.

Electrochemically, no obvious distinction between molecules having a conjugated bridge between ferrocene and azulene and those lacking it can be made. This means that both HOMO and LUMO of all the compounds are located mainly in the ferrocene or the azulene part, respectively, having only small contributions from the corresponding other moiety. This is in accord with the conclusion drawn from the electronic spectra on the lack of influence of the double bonds on the charge transfer bands.

4. Non-linear optics

The coefficient for the first hyperpolarizability, β, was determined for compound **2** in a chloroform solution by hyper Raleigh scattering (MRS) using a wavelength of 1500 nm for the incident light. As expected for ferrocene derivatives, the compound does not show fluorescence. The occurence of fluorescence makes exact measurements of HRS impossible [16]. The apparatus employed a Nd:YAG laser giving pulses of 3 ns at 1064 nm with a repetition rate of 20 Hz. The frequency was trippled by means of barium borate crystals and used for pumping an optical parametric power oscillator (OPPO) whose output (pulses of 3 mJ at 1500 nm) was focused into the HRS cell (for details of the experimental setup, see [17]). An average of over 100 pulses was sampled at the doubled frequency (750 nm) using a photomultiplier. The intensity was measured as a function of the half-wave plate angle and analyzed by the external reference method (ERM) [18] using 4-(*N*,*N*-dimethylamino)cinnamic aldehyde in chloroform as external reference compound whose first hyperpolarizability has been determined by EFISH measurements as $\beta_{333}^{1500} = 13.0 \times 10^{-50}$ C m^3 V^{-2} [18]. The measurements at concentrations in the range of $0.33-2.66 \times 10^{-5}$ mol l^{-1} showed that the dependence of the intensity on the concentration was not linear due to (albeit low) absorption at 750 nm. Using the slope at the lower concentration (up to 0.67×10^{-5} mol l^{-1}), the ratio of the orientational average of the square of the hyperpolarizability tensor between compound **2** and the reference compound was determined as $\langle\beta^2\rangle_2/\langle\beta^2\rangle_{\mathrm{ref}} = 0.54$. Assuming rod-like shape for the compound under investigation (which is approximately the case for alkene **2**) one can calculate the diagonal component of this tensor by comparison with the reference compound. The value of $\beta_{333}^{1500} = 9.6 \times 10^{-50}$ C m^3 V^{-2} is in the range of the reference compound 4-(*N*,*N*-dimethylamino) cinnamic aldehyde. As alkene **2** does not absorb at 1500 nm and only weakly at 750 nm, no degradation of the compound occurred during measurement (checked by repetition and NMR measurement afterwards). Due to somewhat higher absorption in the range of the frequency doubled light, it was not possible to determine the hyperpolarizabilities of the imines **5**–**7**.

Thus, the combination of ferrocene and azulene leads to compounds which show appreciable first-order hyperpolarizabilities. For applications in devices, there are, however, many other prerequisites; a main point is the chemical and optical stability. When left in solution in the daylight (with appreciable light intensity until ca. 380 nm) for about 1 week, the original azulene-like colour of the solution faints and the orange colour of ferrocene derivatives appears. This indicates the photochemical degradation of the compounds by destruction of the azulene part. Definite reaction products could not be found. Further development of such materials will therefore concentrate on improvements of the optical stability. The versatile chemistry of both ferrocene and azulene allows for many modifications by the introduction of donor and/or acceptor substituents. This makes it possible to fine-tune dipole moments and hyperpolarizability as well as the frequency of the maxima of the absorption bands, leading to enhanced photochemical stability. There is much less freedom in the design of the molecules in benzene-derived parent compounds, e.g. the reference standard 4-(*N*,*N*-dimethylamino) cinnamic aldehyde.

5. Experimental

^{1}H- and ^{13}C-NMR spectra were recorded with a Bruker AM 360 spectrometer (360.134 MHz for ^{1}H, 90.556 MHz for ^{13}C). UV–vis spectra were recorded with a Perkin-Elmer Lambda 2 spectrometer. Electrochemical studies were performed with an EG&G PAR 173 potentiostat and an EG&G PARC 175 universal programmer, using a 0.2 molar solution of NBu_4BF_4 in THF, a platinum working electrode and a silver reference electrode. Potentials were determined relative to the internal cyanoferrocene/cyanoferrocinium redox couple ($E_{1/2}^{\mathrm{ox}} = 0.89$ V vs SCE) [19]. They are quoted relative to ferrocene/ferrocinium ($E_{1/2}^{\mathrm{ox}} = 0$ V) . HRS measurements were performed with the equipment described in [17,18].

Solvents were purified by standard methods. 1-Nitroazulene [20], aminoferrocene [21], 1-ferrocenyl-2-methylpropylamine [22], 1-azulenecarbaldehyde, and 4,6,8-trimethyl-1-azulenecarbaldehyde [23] were prepared according to literature procedures. The synthesis of the compounds **1**, **2** and **3** has already been described [9].

5.1. *1-Aminoazulene* ***4***

1-Nitroazulene (l00 mg, 0.55 mmol) was hydrogenated in 25 ml of ethanol using Pd (10% on charcoal) as the catalyst (75 mg). After hydrogenation for 2 h the colour turned from red to green. The solution was filtered over a path of 1 cm of Al_2O_3 (neutral, activity I), and the solvent was removed under reduced pressure at -10°C to yield **4** (50 mg, 63%).

Table 2
Cyclovoltammetry of ferrocene–azulene compounds

Compound	Solvent	$E_{1/2}^{ox}$	E^{ox} (Az)	E^{red} (Az)	others
Azulene	MeCN	—	0.50 (i)	-2.08 (i)	$E_{\mathrm{p}}^{\mathrm{abs}}$ 0 18
4,6,8-Trimethylazulene	MeCN	—	0.45 (part. r)	-2.35 (r)	
1b	MeCN	-0.07	0.49 (part. r)	-2.29 (r)	
1c	MeCN	0.00	0.56 (r)	-2.28 (part. r)	$E_{\mathrm{p}}^{\mathrm{abs}}$ 0.24
3	MeCN	-0.12	0.47 (i)	-2.20 (part. r)	$E_{\mathrm{p}}^{\mathrm{ox}}$ 0.03, 0.23
2	THF	-0.04	—	-2.18 (i)	
6	THF	-0.07	—	-2.04 (r)	$E_{\mathrm{p}}^{\mathrm{ox}}$ -0.76 (after reduction) $E_{\mathrm{p}}^{\mathrm{red}}$ -1 43 (after oxidation)
7	THF	-0.04	—	-2.04 (r)	$E_{\mathrm{p}}^{\mathrm{ox}}$ -0.46 (after reduction) $E_{\mathrm{p}}^{\mathrm{red}}$ -1.48 (after oxidation)

Potentials in V relative to FcH/FcH$^+$. Peaks due to adsorption at the electrode are indicated by the abs superscript. Further abbreviations: r, reversible; part. R, partially reversible; i, = irreversible.

To avoid decomposition, the product was immediately redissolved in 5 ml of hexane and stored at −18°C. NMR spectra could not be obtained due to rapid decomposition in common NMR solvents.

An alternative route by reduction of 1-nitroazulene with Zn + HCl in acetone has been published [20].

5.2. *N-(Ferrocenylmethylidene)-1-azulenylamine* **5**

To a solution of 1-aminoazulene **4** (50 mg, 0.3 mmol) and ferrocenecarbaldehyde (90 mg, 0.45 mmol) in 10 ml of hexane, molecular sieves were added (4 Å) (3.5 g). The solution was stirred for 20 min and then decanted. After removal of the solvent under reduced pressure and chromatography on silicagel (ether/hexane 1:2), two fractions were obtained. The first brown band yielded 55 mg (41%, m.p. 121°C) of **5**, while the second contained 30 mg of ferrocenecarbaldehyde.

^{1}H-NMR: 8.80 (d, $J = 9.7$ Hz, 1H, Az-4); 8.68 (s, 1H, CH=N); 8.18 (d, $J = 9.2$ Hz, 1H, Az-8); 7.96 (d, $J = 4.1$ Hz, 1H, Az-2); 7.53 (t, $J = 9.7$ Hz, 1H, Az-6); 7.35 (d, $J = 4.1$ Hz, 1H; Az-3); 7.11 (t, $J = 9.7$ Hz, 1H); 7.05 (t, $J = 9.7$ Hz, 1H, Az-5,7); 4.89 (s, 2H, cp (subst.)); 4.48 (s, 2H, cp (subst.)); 4.24 (s, 5H, cp (unsubst.)).

^{13}C-NMR: 155.0 (CH=N); 140.4, 139.9 (Az-3a,8a); 138.8 (Az-6); 137.5 (Az-8); 135.0 (Az-4); 132.6 (Az-1); 123.6 (Az-2); 122.6; 122.3 (Az-5,7); 116.9 (Az-3); 82.1 (Cq, cp (subst.)); 70.7, 68.4 (cp (subst.)); 69.2 (cp (unsubst.)).

5.3. *N-(1-Azulenylmethylidene)-ferrocenylamine* **6**

To a solution of aminoferrocene (110 mg, 0.5 mmol) and 1-azulenecarbaldehyde (100 ma, 0.6 mmol) in 30 ml of chloroform, molecular sieves (4 Å) (4.5 g) were added. After 1 h of reflux, the solution was decanted and the solvent removed under reduced pressure. The residue was chromatographed on Al_2O_3 (neutral, activity I, ether/hexane 1:1). The second coloured band was subjected to a second chromatography ($A1_2O_3$, neutral, activity I, toluene:acetone 10:1). The main band yielded 30 mg (20%, m.p. 133–135°C) of **6**.

^{1}H-NMR: 9.19 (s, 1H, CH=N); 8.55 (d, $J = 9.9$ Hz, 1H, Az-4); 8.30 (t, $J = 9.9$ Hz, 2H, Az-5,7); 7.6 (d, $J = 4$ Hz, Az-2); 7.4-7.2 (m, 2H, Az-3,6); 6.81 (d, $J = 9.9$ Hz, 1H, Az-8); 4.63 (s, 2H, cp (subst.)); 4.25 (s, 2H, cp (subst.)); 4.21 (s, 5H, cp (unsubst.)).

^{13}C-NMR: 153.1 (CH=N); 142.1 (Az-1); 138.8 (Az–CH); 138.2 (Az-3a); 138.0 (Az–CH); 137.7 (Az–CH); 135.9 (Az–CH); 125.7 (Az–CH); 126.1 (Az-8a); 125.5 (Az–CH); 119.0 (Az-3); 77.1 (C_q, cp (subst.)); 69.4 (cp (unsubst.)); 66.7 (cp (subst.)); 62.4 (cp (subst.)).

5.4. *N-(4,6,8-Trimethyl-1-azulenylmethylidene)-ferrocenylamine* **7**

To a solution of aminoferrocene (156 mg, 0.78 mmol) and 4,6,8-trimethyl-1azulenecarbaldehyde (200 ma, 1 mmol) in 30 ml of chloroform, molecular sieves (4 Å) (4.5 g) were added. After 1 h of reflux, the solution was decanted and the solvent removed under reduced pressure. Chromatography on Al_2O_3 (neutral, activity I, toluene:acetone 10:1) yielded 90 mg (30%, m.p. 131–133°C) of **7**.

^{1}H-NMR: 9.51 (s, 1H, CH=N); 8.36 (d, 1H, $J = 4.4$ Hz, Az-2); 7.36 (d, 1H, $J = 4.4$ Hz, Az-3); 7.19, 7.12 (2 s, Az-5,7); 4.60 (t, 2H, $J = 1.8$Hz, cp (subst.)); 4.23 (s, 5H, cp (unsubst.)); 4.23 (t, 2H, $J = 1.8$Hz, cp (subst.)); 3.10, 2.89, 2.62 (3s, 9 H, Az–CH_3).

^{13}C-NMR: 156.4 (CH=N); 146.8, 146.6, 146.4 (Az-4, 6, 8); 140.4 (Az-1); 135.0 (Az–CH); 134.4 (Az-3a); 130.8 (Az–CH); 129.1 (Az–CH); 123.1 (Az-8a); 117.3 (Az-3); 77.1 (C_q, cp (subst.)); 69.3 (cp (unsubst.)); 66.6, 62.5 (cp(subst.)); 30.2, 28.3, 25.7 (Az–CH$_3$).

5.5. *N-(1-Azulenylmethyliden)-1-ferrocenyl-2-methylpropylamine* **8**

To a solution of 1-ferrocenyl-2-methylpropylamine (405 mg, 1.6 mmol) and 1-azolenecarbaldehyde (389 mg, 2.5 mmol) in 30 ml of dry chloroform molecular sieves were added (4 Å) (4.5 g). After heating to reflux for 3 days, the solution was decanted. Removal of the solvent under reduced pressure followed by chromatography of the residue (Al_2O_3, neutral, activity I, ether:hexane 1:1) yielded **8** (100 mg, 23%, oil) in the first coloured band.

^{1}H-NMR: 9.79 (d, $J = 9.9$ Hz, 1H, Az-4); 8.95 (s, 1H, CH=N); 8.41 (d, $J = 9.7$ Hz, 1H, Az-8); 8.26 (d, $J = 4$ Hz, 1H, Az-2); 7.73 (t, $J = 9.9$ Hz, 1H, Az-5); 7.46 (t, $J = 9.9$ Hz, 1H, Az-6); 7.41 (d, $J = 4.0$ Hz, 1H, Az-3); 7.31 (t, $J = 9.7$ Hz, 1H, Az-7); 4.23 (m, 2H, cp (subst.)); 4.13 (m, 2H, cp (subst.)); 3.99 (s, 5H, cp (unsubst.)); 3.68 (s, 1H, Fc–CH–N); 1.37 (m, 1H, CHMe$_2$); 0.93 (d, 6H, CH(CH_3)$_2$)

^{13}C-NMR: 154.8 (C=N); 143.9 (Az–CH); 139.4 (Az-1); 138.5 (Az–CH); 137.7 (Az-3a); 137.6 (Az–CH); 137.2 (Az–CH); 125.8 (Az–CH); 125.3 (Az-8a); 124.9 (Az–CH); 118.0 (Az-3); 81.7 (CH–N); 69.8 (C_q, cp (subst.)); 68.3 (cp (unsubst.)); 66.9 (cp (subst.)); 65.9 (cp (subst.)); 35.8 (CHMe$_2$); 27 2 (CH(CH$_3$)$_2$).

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