Rational design of an efficient one-pot synthesis of 6H-pyrrolo[2,3,4-gh]perimidine in polyphosphoric acid†

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Several highly efficient one-pot synthetic protocols were developed, enabling polyphosphoric acid-activated nitroalkanes to act as electrophiles in reactions with aminonaphthalenes. The featured methods allow for the single step assembly of polyheterocyclic aromatic derivatives of 6H-pyrrolo[2,3,4-gh]perimidine scaffold in high yields.

Introduction

1H-Cyclopenta(cd)phenalenes 2 are interesting polycyclic aromatic architectures that can be viewed as derivatives of the acenaphthene system peri-annealed by a benzene ring. These compounds attract the attention of chemists and material scientists due to being the smallest structural units modeling the orbital and physico-chemical properties of fullerenes and defected carbon nanotubes.† Along with pyrene (1) derivatives, such compounds could be used as fluorescent probes and lightsensing units in various biochemical assays.‡ However, due to a very low solubility in water and significant carcinogenicity, the potential of these structural fragments for cytological and in vivo applications could be limited. We pondered if this issue can be addressed by partial replacement of carbon with nitrogen atoms§ to afford aromatic polyheterocyclic scaffolds with improved solubility, which also are expected to show much lower metabolic stability. We previously demonstrated several synthetic approaches towards 1,3-diazopyrenes 3, which we intended as a convenient substitute of the pyrene unit for material science applications.¶ We also invested efforts in the development of synthetic approaches towards 6H-pyrrolo[2,3,4-gh]perimidine scaffold 4, primarily capitalizing on peri-annulations of a pyrrole ring to 1H-perimidines (Fig. 1).¶ Furthermore, we recently reported on umpolung-activation of nitroalkanes 6 toward the addition of carbon- and nitrogen-based nucleophiles with polyphosphoric acids (Scheme 1). This chemistry was employed for the installation of amino and carboxamide groups via direct C–H functionalization of arenes 5 (Scheme 1, eqn (1))∥ and for efficient preparation of benzoxazoles and benz-imidazoles 11 (Scheme 1, eqn (2)).‡ Herein we wish to report our synthetic studies towards 6H-pyrrolo[2,3,4-gh]perimidine 4 employing this unusual chemistry.
Results and discussion

Polyphosphoric acid (PPA) possesses an array of unique physico-chemical properties, which makes it a very attractive ionic liquid-like reaction medium to carry out cascade and one-pot transformations involving acid-catalyzed steps, such as electrophilic aromatic substitution, Schmidt and Beckmann rearrangements, Vilsmeier-type reactions, nucleophilic substitutions, as well as various acid-promoted elimination and condensation processes. Adjusting the content of P$_2$O$_5$ in PPA affects both effective acidity and anhydridic activity of this medium, allowing for fine-tuning of the reaction conditions and intelligent design of multi-step processes. Furthermore, an in situ phosphorylation of various functional groups can be used to act as a temporary protection or to modify reactivity, for example, to equip good leaving group or totally alter the function’s normal reactivity pattern. Thus, we previously demonstrated\textsuperscript{a,c} that generation of phosphorylated species 13 would trigger an electrophilic attack at C-7 to afford (azanediyl)bis(phosphonic acid) 14, which after elimination of H$_3$PO$_4$ at low pH would provide the protonated form of phosphorylated oxime 15 (Scheme 2). Intramolecular 5-exo-trig nucleophilic attack of the aniline function and subsequent elimination of phosphorylated hydroxylamine would provide species 17, which after deprotonation and rearomatization would yield the desired 6H-pyrrolo[2,3,4-gh]perimidine 18 (Scheme 3). To test this idea we subjected 6-amino-1H-perimidine 13a to the reaction with an excess of nitromethane (6a). First, the mixture was stirred in 86% PPA at 80–90 °C, then the reaction was forced to completion at 130–140 °C. The expected 6H-pyrrolo[2,3,4-gh]perimidine 18aa was formed as the sole product in good preparative yield (Scheme 3). Similarly, nitroethane (6b), 2-nitrotoluene (6c, f–h), 2-phenylnitroethane (6d), and 1-nitrohexane (6e), were also employed in the reaction with 6-amino-1H-perimides 13b–c substituted at C-2 to afford the corresponding 6H-pyrrolo[2,3,4-gh]perimides with different substituents C-2 and C-7 (Scheme 2). Remarkably, these compounds were obtained as sole products and isolated in pure form after aqueous work up followed by recrystallization. Purification by column chromatography was not required.

Next, we decided to investigate the possibility of generating 6-amino-1H-perimides species 13 in situ. It was anticipated that generation of phosphorylated aci-form 12 in the presence of naphthalene-1,4,5-triamine (19) would induce nucleophilic attack by one of the amino-groups affording phosphorylated iminamide 20 (shown in protonated from in Scheme 3). The later would undergo a 6-exo-trig cyclization involving a second amino group in the peri-position and provide cyclic methane-triamine intermediate 21. Subsequent acid-mediated elimination of phosphorylated hydroxylamine is expected to provide 6-amino-1H-perimidine 13 (Scheme 3), which should react further according to the mechanism, depicted in Scheme 2, ultimately yielding the target 6H-pyrrolo[2,3,4-gh]perimides 18. Gratifyingly, this multistep cascade transformation proceeded as planned. Reactions of 19 with excess (3.1 equiv.) nitromethane (6a) or nitroethane (6b) afforded products 18aa and 18bb, respectively, in good yields (Scheme 3).

We also envisioned an alternative method for the in situ generation of key intermediate 13 employing a PPA-assisted Schmidt reaction of perimides 25 in the presence of sodium azide. It is believed, that hydrazoic acid generated under these reaction conditions, reacts with PPA to form phosphazadic acid 22, which after protonation provides phosphonotriazynium ion 24, that exists in equilibrium with tautomer 23
Species 24 can be viewed as the stabilized form of N-amino-diazonium, and its reactivity reminds that of diazonium salts. In particular, it can participate in efficient S$_{Ar}$-reaction with electron-rich aromatic compounds, such as perimidines to afford the corresponding (aryltriaz-1-en-1-yl)phosphonic acids (existing in equilibrium with tautomeric form).

Acid-mediated decomposition of these species, accompanied with a evolution of nitrogen gas would afford intermediate 6-aminopiperidine, that can be consumed in a subsequent one-pot reaction with an appropriate nitroalkane. We were pleased to discover, that this process design was viable, and the corresponding 6H-pyrrolo[2,3,4-$gh$]perimidines were produced in very good yield (Scheme 4).

Next, we decided to elaborate on the one-pot preparation of target heteroaromatic tetracycles starting from readily available 6-acylperimidines. It was expected, that initial nucleophilic addition of the azide across C=O bond would provide phosphorylated 1-azidoalcohol, which should undergo facile Schmidt rearrangement to provide acetamide. The subsequent addition of nitroethane and in situ generation of electrophilic $ac$ form would invoke alkylation of at C-7 to yield intermediate, which, after elimination of $H_3$PO$_4$ would provide phosphorylated oxime (shown in Scheme 5 in protonated form). We reasoned, that the acetamide group in 32 could be further involved in nucleophilic 5-exo-trig cyclization affording tetracyclic intermediate, which after elimination of O-phosphoryl hydroxylamine would yield N-acetylpyrroloperimidine. Aqueous work up accompanied with hydrolytic cleavage of N-acetyl moiety would finally afford the target material.

Finally, we investigated the possibility to develop a multi-step cascade transformation involving an even more affordable starting material, 1,8-naphthalenediamine. It was anticipated that in the presence of $ac$-form, generated upon phosphorylation of nitroethane, two amino groups would be used to assemble a heterocyclic ring of perimidine (Scheme 6). This process would involve a two-fold nucleophilic attack by nitrogen atoms and its mechanism would be identical.
to the one previously described in Scheme 3. We also expected that the excess of \textit{aci}-form would serve as an electrophile in the subsequent acietamidation reaction, that should be directed to C-6 of perimidine.\textsuperscript{7} This reaction involves an initial alkylation of the activated heteroaromatic compound to form phosphorylated ketoxim 29, which upon heating in PPA should undergo Beckmann rearrangement to afford acetalanlide \textit{30b} (Scheme 6). The following transformation would proceed according to the mechanism outlined in Scheme 5 to afford the pyrroloperimidine core. We tested this transformation on a single example, and were thrilled to find that compound \textit{18bb} was formed as sole isolaable product, albeit in somewhat marginal yield (Scheme 5).

Conclusions

We have designed a series one-pot and cascade transformations involving an unusual electrophilic behavior of polyphosphoric acid-activated nitroalkanes. These processes involved PPA-mediated Schmidt and Beckmann rearrangements, reactions of direct electrophilic amination with azides and acietamidation of direct electrophilic amination with azides and acietamidation of direct electrophilic amination with azides and acietamidation of direct electrophilic amination with azides and acietamidation of direct electrophilic amination with azides and acietamidation of direct electrophilic amination with azides and acietamidation of direct electrophilic amination with azides and acietamidation of direct electrophilic amination with azides and acietamidation of direct electrophilic amination with azides and acietamidation of direct electrophilic amination with azides and acietamidation of direct electrophilic amination with azides and acietamidation of direct electrophilic amination with azides and acietamidation of direct electrophilic amination with azides and acietamidation of direct electrophilic amination with azides and acietamidation of direct electrophilic amination with azides.

Experimental part

\textsuperscript{1}H and \textsuperscript{13}C NMR spectra were recorded on a Bruker Avance-III spectrometer (400 or 100 MHz, respectively) equipped with BBO probe in DMSO-\textit{d}_6, using TMS as internal standard. High-resolution mass spectra were registered with a Bruker Maxis spectrometer (electrospray ionization, in MeCN solution, using HCO\textsubscript{2}Na–HCO\textsubscript{2}H for calibration). Melting points were measured with a Stuart smp30 apparatus. All reactions were performed in oven-dried 5 mL Erlenmeyer flasks open to the atmosphere, employing overhead stirring. Reaction progress and purity of isolated compounds were controlled by TLC on Silufol UV-254 plates. Compounds \textit{6f–h, 13a–c, 7a–c} and \textit{28a–c, 12} were prepared according to the literature procedures. All other reagents and solvents were purchased from commercial vendors and used as received.

Preparation of 2-(4-chlorophenyl)-6-(7)-acetylperimidine (28d)

Anhydrous aluminum chloride (1.33 g, 10.0 mmol) and acetyl chloride (85 μL, 94 mg, 1.2 mmol) were vigorously stirred in dry dichloromethane (20 mL) for 30 min at room temperature. 2-(4-Chlorophenyl)perimidine (279 mg, 1.0 mmol) was added by portions over 15 min, and the stirring was continued for 30 min. The formed solution was poured into cold water (20 mL) and dichloromethane was removed in vacuum. Solid precipitate was filtered off and re-crystallized from ethanol to afford the titled compound as carrot-orange crystals. Yield 295 mg (0.92 mmol, 92%), mp 236–238 °C (EtOH); \textsuperscript{1}H NMR (400 MHz, DMSO-\textit{d}_6): \(\delta 2.57\) (3H, s, CH\textsubscript{3}), 6.75 (1H, d, \textit{J} = 8.1 Hz, H-4(9)), 6.97 (1H, d, \textit{J} = 7.2 Hz, H-9(4)), 7.47 (1H, dd, \textit{J} = 8.7, 7.2 Hz, H-8(5)), 7.67 (2H, d, \textit{J} = 8.6 Hz, Ar (H-3,5)), 8.09 (3H, m, H-5(8), Ar (H-2,6)), 8.56 (1H, d, \textit{J} = 8.7 Hz, H-7(6)), 11.39 (1H, br. s, NH); \textsuperscript{13}C NMR (100 MHz, DMSO-\textit{d}_6): \(\delta 28.6\), 106.2, 111.4, 118.8, 121.2, 123.6, 127.7, 128.6 (2C), 129.1 (2C), 130.9, 132.6, 136.0, 136.5, 140.6, 145.4, 151.8, 197.5; FT-IR (NaCl, cm\textsuperscript{-1}): 3320 (NH), 2390, 1650 (CO).

General procedure for preparation of pyrrolo[2,3,4-\textit{gh}] perimides from 6-aminoperimidines (method A)

A mixture of 6-aminoperimidine 13 (1.00 mmol), nitroalkane 6 (2.10 mmol), and 86% PPA (3–4 g) was vigorously stirred for 1 h at 80–90 °C, then heated to 130–140 °C, and stirred for additional 3 h. The reaction mixture was poured out into stirred cold water (30 mL), and the mixture was basified with aqueous ammonia to pH 8. Formed precipitate was filtered off, the supernatant solution was extracted with toluene (3 × 50 mL). The precipitate was also extracted for 5 h with toluene (100 mL) in Soxhlet apparatus. Combined organic extracts were evaporated and the residual solid was re-crystallized.

General procedure for preparation of pyrrolo[2,3,4-\textit{gh}] perimides from 1,4,8-naphthalenetrimine (method B)

A mixture of 1,4,8-triaminonaphthalene 19 (0.173 g, 1.00 mmol), nitroalkane 6 (3.10 mmol), and 86% PPA (3–4 g) was vigorously stirred for 3 h at 80–90 °C, then heated to 130–140 °C, and stirred for additional 3 h. Aqueous work up, isolation and purification of products is carried out in the manner described for the method A.

General procedure for preparation of pyrrolo[2,3,4-\textit{gh}] perimides from perimidines (method C)

A mixture of perimidine 25 (1.00 mmol), sodium azide (0.195 g, 3.00 mmol), 86% PPA (3–4 g) was vigorously stirred for 4 h; the reaction progress was monitored by TLC. Then nitroalkane 6...
A mixture of 6-acetylperimidine (28) (1.00 mmol), sodium azide (0.070 g, 1.07 mmol) in 86% PPA (2–3 g) was vigorously stirred for 1 h at 55–60 °C. Then nitroalkane 6 was added (2.00 mmol), and the temperature was increased to 110–120 °C (90–100 °C for reactions with nitromethane) and the stirring was continued for additional 3 h. Aqueous work up, isolation and purification of products is carried out in the manner described for the method A.

Preparation of 2,7-dimethylpyrrolo[2,3,4-gh]perimidine from 1,8-diaminonaphthalene (method E) A mixture of 1,8-diaminonaphthalene (35) (0.158 g, 1.00 mmol), nitroalkane 6 (2.10 mmol), and 86% PPA (2–3 g) was vigorously stirred for 3 h at 90–100; the reaction progress was monitored by TLC, eluting with EtOAc/EtOH mixture, 1:1. Then, the temperature was increased to 140–150 °C and the stirring was continued for additional 3 h. Aqueous work up, isolation and purification of products is carried out in the manner described for the method A.

2,7-Dimethyl-6H-pyrrolo[2,3,4-gh]perimidine (18bb).e Yellowish orange crystals, mp 271–272 °C (toluene); 1H NMR (400 MHz, DMSO-d6): δ 2.86 (3H, s, 7-Me), 2.92 (3H, s, 2-Me), 7.46 (1H, d, J = 9.0 Hz, H-3), 7.64 (1H, d, J = 8.7 Hz, H-9), 8.22 (1H, d, J = 8.7 Hz, H-8), 8.38 (1H, d, J = 9.0 Hz, H-4), 13.1 (1H, br. s, NH); 13C NMR (100 MHz, DMSO-d6): δ 12.4, 27.0, 112.0, 112.8, 116.2, 120.0, 120.4, 120.5, 124.6, 130.7, 136.1, 147.7, 154.9, 162.9.

7-Methyl-2-phenyl-6H-pyrrolo[2,3,4-gh]perimidine (18bc).e Yellowish orange crystals, mp 245–246 °C (toluene/petroleum ether); 1H NMR (400 MHz, DMSO-d6): δ 2.93 (s, 3H, Me), 7.48–7.58 (3H, m, Ph (H-3,4,5)), 7.62 (1H, d, J = 9.0 Hz, H-3), 7.82 (1H, d, J = 8.7 Hz, H-8), 8.29 (1H, d, J = 9.0 Hz, H-4), 8.43 (1H, d, J = 9.0 Hz, H-4), 8.71 (2H, d, J = 8.4 Hz, Ph (H-2,6)), 13.1 (1H, br. s, NH); 13C NMR (100 MHz, DMSO-d6): δ 12.4, 112.7, 112.9, 117.1, 120.5, 120.6, 120.7, 124.9, 128.2 (2C), 129.4, 130.9, 136.2, 140.1, 147.9, 153.1, 159.6.

2-(4-Chlorophenyl)-7-methyl-6H-pyrrolo[2,3,4-gh]perimidine (18bd).e Yellowish orange crystals, mp 231–237 °C (toluene); 1H NMR (400 MHz, DMSO-d6): δ 7.54 (3H, m, Ph (H-3,4,5)), 7.66 (1H, d, J = 9.0 Hz, H-3), 7.79 (1H, d, J = 8.7 Hz, H-9), 8.18 (2H, d, J = 7.7 Hz, Ph (H-2,6)), 8.33 (1H, d, J = 8.7 Hz, H-8), 8.69 (1H, d, J = 9.0 Hz, H-4), 9.33 (1H, s, H-6), 13.3 (1H, br. s, NH); 13C NMR (100 MHz, DMSO-d6): δ 112.0, 112.2, 117.9, 120.9, 121.2, 122.3, 126.2, 127.2 (2C), 128.5, 129.3 (2C), 129.4, 131.0, 132.2, 148.4, 154.6, 163.5.

2-Benzyl-6H-pyrrolo[2,3,4-gh]perimidine (18da). Yellowish orange crystals, mp 251–252 °C (toluene); 1H NMR (400 MHz, DMSO-d6): δ 4.72 (2H, s, CH2), 7.24 (1H, t, J = 7.4 Hz, Ph (H-4)), 7.34 (2H, dd, J = 7.8, 7.4 Hz, Ph (H-3,5)), 7.42 (2H, d, J = 7.8 Hz, Ph (H-2,6)), 7.58 (1H, d, J = 9.1 Hz, H-3), 8.31 (1H, d, J = 8.8 Hz, H-9), 8.32 (1H, d, J = 9.1 Hz, H-4), 8.35 (1H, d, J = 8.8 Hz, H-4), 9.39 (1H, s, H-6), 13.5 (1H, br. s, NH); 13C NMR (100 MHz, DMSO-d6): δ 33.4, 113.0, 114.2, 117.7, 120.4, 121.2, 125.2, 126.6, 126.8 (2C), 128.5, 129.7, 129.8, 131.2 (2C), 130.9, 139.0, 147.7, 155.0; FT-IR (NaCl, cm–1): 3407 (NH), 3214, 3028, 2814, 1618, 1494, 1373; HRMS (TOF-ES): calc for C19H13ClN3 (M + H) 318.0793, found 318.0788 (1.6 ppm).

2-Benzyl-2-methyl-6H-pyrrolo[2,3,4-gh]perimidine (18db). Yellowish orange crystals, mp 223–225 °C (toluene); 1H NMR (400 MHz, DMSO-d6): δ 2.86 (3H, s, Me), 4.68 (2H, s, CH2), 7.23 (1H, t, J = 7.3 Hz, Ph (H-4)), 7.33 (2H, dd, J = 7.7, 7.3 Hz, Ph (H-3,5)), 7.43 (2H, d, J = 7.7 Hz, Ph (H-2,6)), 7.48 (1H, d, J = 9.1 Hz, H-3), 7.69 (1H, d, J = 8.8 Hz, H-9), 8.24 (1H, d, J = 9.1 Hz, H-8), 8.27 (1H, d, J = 8.8 Hz, H-4), 13.12 (1H, br. s, NH); 13C NMR (100 MHz, DMSO-d6): δ 114.0, 114.2, 121.8, 127.1, 130.9, 130.9, 131.2 (2C), 130.9, 139.0, 147.7, 155.0; FT-IR (NaCl, cm–1): 3407 (NH), 3214, 3028, 2814, 1618, 1494, 1373; HRMS (TOF-ES): calc for C19H14N3 (M + H) 318.0793, found 318.0788 (1.6 ppm).
9.1 Hz, H-8), 8.35 (1H, d, J = 8.8 Hz, H-4), 8.69–8.73 (3H, m, ArH), 13.62 (1H, br, s, NH); 13C NMR (100 MHz, DMSO-d6): δ 33.4, 113.0, 118.1, 120.8, 121.4, 121.6, 125.4, 126.9, 128.3 (2C), 128.6 (2C), 128.9 (2C), 129.1 (2C), 129.8, 130.9, 131.4, 133.9, 139.0, 140.2, 148.3, 155.3, 160.0; FT-IR (NaCl, cm⁻¹): 3373 (NH), 2924, 2842, 2366, 1745, 1625, 1535, 1497, 1367, 1242, 1274; HRMS (TOF-ES) calculated for C₂₃H₂₁ClN₃ (M + H) 360.1495, found 360.1501 (1.6 ppm).

7-Pentyl-6H-pyrrolo[2,3,4-g]perimidine (18ca). Yellowish orange crystals, mp 156–158 °C (toluene–petroleum ether); Rᵣ 0.62 (EtOAc/ EtOH 3:1); ¹H NMR (400 MHz, DMSO-d₆): δ 0.87 (3H, t, J = 7.0 Hz, Me), 1.36 (4H, m, CH₂CH₂), 1.93 (2H, m, CH₂), 3.36 (2H + H₂O, m, CH₂ + H₂O), 7.10 (6H, d, J = 9.1 Hz, H-3), 7.80 (1H, d, J = 8.7 Hz, H-6), 8.05 (1H, d, J = 8.7 Hz, H-4), 8.52 (1H, d, J = 9.1 Hz, H-8), 9.38 (1H, s, H-6), 13.24 (1H, br, s, NH); ¹³C NMR (100 MHz, DMSO-d₆): δ 13.7, 20.7, 21.8, 29.4, 31.0, 111.7, 114.1, 117.0, 120.2, 120.5, 120.7, 124.8, 128.1, 131.0, 147.4, 154.7, 154.8; FT-IR (NaCl, cm⁻¹): 3507 (NH), 2958, 2438, 1631, 1572, 1490, 1255, 1190, 1082; HRMS (TOF-ES) calculated for C₁₇H₁₈N₃ (M + H) 264.1495, found 264.1493 (0.8 ppm).

2-(Chlorophenyl)-7-pentyl-6H-pyrrolo[2,3,4-g]perimidine (18ed). Yellowish orange crystals, mp 211–213 °C (toluene–petroleum ether); Rᵣ 0.74 (EtOAc); ¹H NMR (400 MHz, CDCl₃): δ 0.92 (3H, t, J = 7.1 Hz, Me), 1.35–1.47 (4H, m, CH₂CH₂), 1.93–2.00 (2H, m, CH₂), 3.30 (2H, J = 7.7 Hz, CH₃), 5.11 (1H, d, J = 8.6 Hz, Ar (H-3,5)), 7.78 (1H, d, J = 9.2 Hz, H-3), 7.97 (1H, d, J = 8.8 Hz, H-9), 8.16 (1H, d, J = 8.8 Hz, H-4), 8.30 (1H, d, J = 9.2 Hz, H-8), 8.69 (2H, J = 8.6 Hz, Ar (H-2,6)), 9.71 (1H, br, s, NH); ¹³C NMR (100 MHz, CDCl₃): δ 13.9, 22.4, 28.0, 29.8, 31.6, 113.3, 113.5, 119.0, 119.7, 121.4, 122.8, 124.9, 128.6 (2C), 130.1 (2C), 130.3, 135.7, 139.0, 139.1, 148.6, 156.1, 160.6; FT-IR (NaCl, cm⁻¹): 33 907 (NH), 2991, 1621, 1552, 1491, 1360, 1275, 1181, 1080, 840, 800; HRMS (TOF-ES) calculated for C₂₃H₂₁ClN₃ (M + H) 374.1419, found 374.1423 (1.1 ppm).

7-(Chlorophenyl)-2-methyl-6H-pyrrolo[2,3,4-g]perimidine (18fb). Yellowish orange crystals, mp 283–284 °C (toluene); Rᵣ 0.44 (EtOAc/EtOH 3:1); ¹H NMR (400 MHz, CDCl₃): δ 3.00 (3H, s, Me), 7.37 (1H, d, J = 7.9 Hz, Ar (H-4)), 7.45 (1H, dd, J = 7.9, J = 7.7 Hz, Ar (H-5)), 7.68 (1H, d, J = 9.2 Hz, H-3), 7.75 (1H, d, J = 8.8 Hz, H-9), 7.82 (1H, d, J = 8.8 Hz, Ar (H-6)), 7.92 (1H, s, Ar (H-2)), 8.00 (1H, d, J = 8.8 Hz, H-4), 8.40 (1H, d, J = 9.2 Hz, H-8), 9.51 (1H, br, s, NH); ¹³C NMR (100 MHz, CDCl₃): δ 25.8, 111.4, 121.1, 117.9, 119.8, 122.0, 124.3, 125.3, 126.1, 127.7, 129.8, 130.0, 132.4, 133.6, 134.5, 134.8, 147.0, 153.6, 162.6; FT-IR (NaCl, cm⁻¹): 3317 (NH), 2985, 2825, 2240, 1641, 1554, 1490, 1383, 1273, 1181, 840, 800; HRMS (TOF-ES) calculated for C₁₅H₁₃ClN₃ (M + H) 318.0793, found 318.0787 (1.9 ppm).

References


