新型 1,3,4-噁二唑衍生物的合成*

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摘 要：1,3,4-噁二唑广泛应用于医药、农药、材料等诸多领域，该文设计并合成了一系列含 *N*-正癸基咪唑结构单元的 1,3,4-噁二唑化合物，目标产物结构经 NMR，MS 和 EA 鉴定。

关键词：咪唑；1,3,4-噁二唑；合成
中图分类号：O621.3 **文献标志码**：A **文章编号**：0529－6579（2012）03－0068－05

Synthesis of Novel 1,3,4-Oxadiazole Derivatives

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Abstract: 1,3,4-Oxadiazole compounds are a class of significant heterocyclic compounds widely used in medicinal chemistry, pesticide chemistry and materials science. In this work, a series of 1,3,4-oxadiazole derivatives containing imidazole unit were designed, synthesized and their structures were characterized by NMR spectroscopy, Mass and elemental analyses.

Key words: imidazole; 1,3,4-oxadiazoles; synthesis

1 引 言

咪唑衍生物广泛用于医药和农药[1－3]、光电材料[4－12]、不对称有机合成催化剂配体[13]、离子液体[14]、阴离子传感器[15]等领域。本课题组多年从事具有杂环结构的发光材料的设计合成和发光性能的研究[16－18]，曾报道过一系列含有咪唑结构单元的大共轭体系的 2，5-二芳基-1,3,4-噁二唑的合成[19]。但是该类化合物的溶解性能不佳，为改善其溶解性，从而改善在染料等方面的应用性能，增加烷基的长度，合成了系列含 *N*-正癸基咪唑结构的 2，5-二芳基-1,3,4-噁二唑。合成路线和目标产物的结构见图 1。

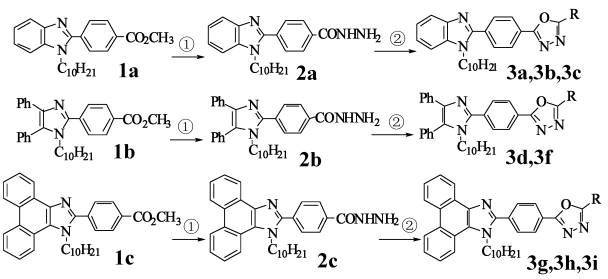

图 1 1,3,4-噁二唑衍生物的合成路线

Fig. 1 The synthetic route of 1,3,4-oxadiazole derivatives.

* **收稿日期**：2011－12－19
基金项目：广东省科技计划资助项目（2003C103006，2007B060401068）
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2　实验部分

2.1　仪器和试剂

温度计未校准，X4 熔点仪，Bruker AVANCE－300 NMR 核磁共振仪，SHIMADZU LCMS－2010A 质谱仪，Elementar Analysensysteme GmbH Vario EL 元素分析仪。其余试剂均为分析纯，未经处理直接使用。

2.2　4-（2-（1-正癸基咪唑基））苯甲酸甲酯 **1a**，**1b**，**1c** 的合成

化合物 **1a**，**1b**，**1c** 参照文献［19］合成，其结构表征如下。

4-(2-(1-正癸基苯并咪唑基)) 苯甲酸甲酯（**1a**）：淡黄色，黏稠液体，产率 75%。^{1}H NMR (DMSO-d_6) δ: 0.87 (t, J = 6.6 Hz, 3H, CH_3), 1.20～1.30 (m, 14H, CH_2), 1.77～1.83 (m, 2H, CH_2), 3.96 (s, 3H, OCH_3), 4.23 (t, J = 7.5 Hz, 2H, NCH_2), 7.27～7.35 (m, 2H, ArH), 7.38～7.44 (m, 1H, ArH), 7.78～7.83 (m, 3H, ArH), 8.19 (d, J = 8.7 Hz, 2H, ArH)。ESI-MS (m/z): 393 $[M + H]^+$。

4-(2-(1-正癸基-4，5-二苯基咪唑基)) 苯甲酸甲酯（**1b**）：无色固体。产率 81%，熔点 81～82 ℃。^{1}H NMR (DMSO-d_6, δ: 0.83 (t, J = 6.9 Hz, 3H, CH_3), 0.86～0.94 (m, 6H, CH_2), 0.96～1.05 (m, 2H, CH_2), 1.07～1.16 (m, 4H, CH_2), 1.18～1.33 (m, 4H, CH_2), 3.89 (s, 3H, OCH_3), 3.94 (t, J = 7.2 Hz, 2H, NCH_2), 7.09～7.22 (m, 3H, ArH), 7.38～7.48 (m, 4H, ArH), 7.50～7.57 (m, 3H, ArH), 7.91 (d, J = 8.1 Hz, 2H, ArH), 8.10 (d, J =8.4 Hz, 2H, ArH)。ESI-MS (m/z): 495 $[M + H]^+$。

4-(2-(1-正癸基菲并［9，10-d］咪唑基)) 苯甲酸甲酯（**1c**）：浅黄色固体，产率 76%，熔点 72～73 ℃。^{1}H NMR ($CDCl_3$) δ: 0.86 (t, J = 6.9 Hz, 3H, CH_3), 1.15～1.34 (m, 14H, CH_2), 1.87～2.00 (m, 2H, CH_2), 3.98 (s, 3H, OCH_3), 4.60 (t, J = 7.2 Hz, 2H, NCH_2), 7.58～7.71 (m, 4H, ArH), 7.84 (d, J = 8.1 Hz, 2H, ArH), 8.19－8.25 (m, 3H, ArH), 8.69 (d, J = 8.1 Hz, 1H, ArH), 8.77 (d, J = 7.8 Hz, 1H, ArH), 8.84 (d, J = 7.8 Hz, 1H, ArH)。ESI-MS (m/z): 493 $[M + H]^+$。

2.3　4-(2-(1-癸基咪唑基))苯甲酰肼 **2a**，**2b**，**2c** 的合成

化合物 **2a**，**2b**，**2c** 参照文献［19］合成，其结构表征如下。

4-(2-(1-正癸基苯并咪唑基)) 苯甲酰肼（**2a**）：无色固体，产率 70%，熔点 95～96℃。^{1}H NMR ($CDCl_3$) δ: 0.86 (t, J = 6.6 Hz, 3H, CH_3), 1.12～1.29 (m, 14H, CH_2), 1.70～1.84 (m, 2H, CH_2), 4.18 (t, J = 6.9 Hz, 2H, NCH_2), 4.72 (s, 2H, NH_2), 7.29～7.35 (m, 2H, ArH), 7.38～7.44 (m, 1H, ArH), 7.63～7.70 (m, 2H, ArH), 7.83～7.88 (m, 3H, ArH), 8.42 (s, 1H, NH)。ESI-MS (m/z): 393 $[M + H]^+$。

4-(2-(1-正癸基-4，5-二苯基咪唑)) 苯甲酰肼（**2b**）：无色固体，产率 82%，熔点 111～112 ℃。^{1}H NMR (DMSO-d_6) δ: 0.83 (t, J = 6.9 Hz, 3H, CH_3), 0.88～0.96 (m, 6H, CH_2), 0.99～1.07 (m, 2H, CH_2), 1.09～1.17 (m, 4H, CH_2), 1.19～1.29 (m, 4H, CH_2), 3.92 (t, J = 7.2 Hz, 2H, NCH_2), 4.54 (s, 2H, NH_2), 7.09～7.21 (m, 3H, ArH), 7.39～7.48 (m, 4H, ArH), 7.50～7.57 (m, 3H, ArH), 7.81 (d, J = 8.4 Hz, 2H, ArH), 7.98 (d, J = 8.1 Hz, 2H, ArH), 9.89 (s, 1H, CONH)。ESI-MS (m/z): 495 $[M + H]^+$。

4-(2-(1-正癸基菲并［9，10－d］咪唑－2)) 苯甲酰肼（**2c**）：无色针状晶体，产率 78%，熔点 197～198 ℃。^{1}H NMR ($CDCl_3$) δ: 0.87 (t, J = 6.6Hz, 3H, CH_3), 1.11～1.32 (m, 14H, CH_2), 1.88～1.99 (m, 2H, CH_2), 4.20 (s, 2H, NH_2), 4.59 (t, J = 7.5 Hz, 2H, NCH_2), 7.61～7.74 (m, 4H, ArH), 7.81 (d, J = 8.4Hz, 2H, ArH), 7.94 (d, J = 8.4 Hz, 2H, ArH), 8.26 (d, J = 7.5 Hz, 1H, ArH), 8.72 (d, J = 7.8 Hz, 1H, ArH), 8.79 (dd, J_1 = 1.5 Hz, J_2 = 9.3 Hz, 1H, ArH), 8.86 (d, J = 7.8 Hz, 1H, ArH)。ESI-MS (m/z): 493 $[M + H]^+$。

2.4　2,5-二芳基-1,3,4-噁二唑衍生物 **3a**，**3b**，**3c**，**3d**，**3e**，**3f**，**3g**，**3h**，**3i** 的合成

参照文献［19］完成合成，粗产物经柱层析后得到纯的目标物 **3a**，**3b**，**3c**，**3d**，**3e**，**3f**，**3g**，**3h**，**3i**。

2-(4-(2(1-正癸基苯并咪唑基)) 苯基) -5-苯基-1,3,4-噁二唑（**3a**）：无色固体，产率 45%，熔点 78～80 ℃。^{1}H NMR ($CDCl_3$) δ: 0.85 (t, J = 6.6 Hz, 3H, CH_3), 1.14～1.23 (m, 14H, CH_2), 1.79～1.87 (m, 2H, CH_2), 4.27 (t, J = 7.5 Hz, 2H, NCH_2), 7.29～7.36 (m, 2H, ArH), 7.40～

7.44（m, 1H, ArH）, 7.50～7.58（m, 3H, ArH）, 7.80～7.86（m, 1H, ArH）, 7.92（d, J = 8.7 Hz, 2H, ArH）, 8.14～8.17（m, 2H, ArH）, 8.31（d, J = 8.7 Hz, 2H, ArH）。^{13}C NMR（$CDCl_3$）δ: 14.0, 22.5, 26.6, 28.9, 29.1, 29.3, 29.3, 29.7, 31.7, 44.8, 110.0, 119.9, 122.4, 122.9, 123.4, 124.6, 126.7, 126.9, 128.8, 129.7, 131.6, 133.6, 135.5, 142.8, 151.8, 163.6, 164.5。元素分析(w/%): $C_{31}H_{34}N_4O \cdot H_2O$, 计算值: C, 74.97; H, 7.31; N, 11.28;实测值: C, 74.69; H, 7.33; N 11.23。ESI-MS（m/z）: 479［M + H］$^+$。

2-(4-(2-(1-正癸基苯并咪唑基))苯基)-5-(2-萘基)-1,3,4-噁二唑（**3b**）: 无色固体, 产率47%, 熔点102～104 ℃。^{1}H NMR（$CDCl_3$）δ: 0.85（t, J = 6.6 Hz, 3H, CH_3）, 1.14～1.32（m, 14H, CH_2）, 1.80～1.87（m, 2H, CH_2）, 4.28（t, J = 7.5 Hz, 2H, NCH_2）, 7.29～7.36（m, 2H, ArH）, 7.54～7.61（m, 3H, ArH）, 7.83～7.92（m, 3H, ArH）, 7.95～8.00（m, 3H, ArH）, 8.36（d, J = 8.1 Hz, 2H, ArH）, 8.65（s, 1H, ArH）。^{13}C NMR（$CDCl_3$）δ: 13.9, 22.4, 26.4, 28.8, 29.0, 29.1, 29.2, 29.5, 31.6, 44.6, 109.9, 119.6, 120.4, 122.3, 122.6, 122.7, 124.3, 126.6, 126.8, 127.4, 127.5, 128.3, 128.4, 128.5, 129.4, 132.2, 133.1, 134.1, 135.3, 142.4, 151.5, 163.4, 164.3。元素分析(w/%): $C_{35}H_{36}N_4O \cdot 0.21H_2O$, 计算值: C, 78.95; H, 6.89; N, 10.52; 实测值: C, 78.42; H, 6.28; N 9.76。ESI-MS（m/z）: 529［M + H］$^+$。

2-(4-(2-(1-正癸基苯并咪唑基))苯基)-5-(2-噻吩基)-1,3,4-噁二唑（**3c**）: 黄色固体, 产率40%, 熔点94～95 ℃。^{1}H NMR（$CDCl_3$）δ: 0.85（t, J = 6.6 Hz, 3H, CH_3）, 1.15～1.24（m, 14H, CH_2）, 1.78～1.85（m, 2H, CH_2）, 4.26（t, J = 7.5Hz, 2H, NCH_2）, 7.18～7.21（m, 1H, ArH）, 7.28～7.36（m, 2H, ArH）, 7.41～7.44（m, 1H, ArH）, 7.59（d, J = 4.8 Hz, 1H, ArH）, 7.81～7.87（m, 2H, ArH）, 7.91（d, J = 8.1Hz, 2H, ArH）, 8.28（d, J = 8.1 Hz, 2H, ArH）。^{13}C NMR（$CDCl_3$）δ: 14.0, 22.6, 26.7, 28.9, 29.2, 29.3, 29.4, 29.8, 31.8, 44.9, 110.0, 119.9, 122.4, 122.9, 124.4, 126.9, 128.1, 129.7, 129.8, 130.2, 133.7, 135.6, 142.9, 151.9, 160.9, 163.2。元素分析(w/%): $C_{29}H_{32}N_4OS$, 计算值: C, 71.87; H, 6.66; N, 11.56;实测值: C, 71.60; H, 6.50; N 11.52。ESI-MS（m/z）: 485［M + H］$^+$。

2-(4-(2-(1-正癸基-4,5-二苯基咪唑基))苯基)-5-苯基-1,3,4-噁二唑（**3d**）: 无色固体, 产率39%, 熔点96～98 ℃。^{1}H NMR（DMSO-d_6）δ: 0.75（t, J = 6.9 Hz, 3H, CH_3）, 0.81～1.17（m, 14H, CH_2）, 1.22～1.36（m, 2H, CH_2）, 3.98（t, J = 6.9 Hz, 2H, NCH_2）, 7.10～7.23（m, 3H, ArH）, 7.41～7.49（m, 4H, ArH）, 7.51～7.58（m, 3H, ArH）, 7.60～7.67（m, 3H, ArH）, 8.02（d, J = 8.4 Hz, 2H, ArH）, 8.12～8.19（m, 2H, ArH）, 8.29（d, J = 8.4 Hz, 2H, ArH）。^{13}C NMR（$CDCl_3$）δ: 14.1, 22.6, 26.1, 28.5, 29.0, 29.1, 29.3, 30.3, 31.8, 44.9, 123.6, 126.2, 126.6, 126.7, 126.9, 127.9, 128.6, 128.9, 129.3, 130.4, 130.8, 131.0, 131.6, 134.2, 134.5, 138.1, 146.0, 163.9, 164.4。ESI-MS（m/z）: 581［M + H］$^+$。

2-(4-(2-(1-正癸基-4,5-二苯基咪唑基))苯基)-5-(2-萘基)-1,3,4-噁二唑（**3e**）: 无色固体, 产率43%, 熔点133～134 ℃。^{1}H NMR（DMSO-d_6）δ: 0.73（t, J = 6.6 Hz, 3H, CH_3）, 0.83～1.20（m, 14H, CH_2）, 1.24～1.36（m, 2H, CH_2）, 3.98（t, J = 7.2 Hz, 2H, NCH_2）, 7.10～7.23（m, 3H, ArH）, 7.42～7.50（m, 4H, ArH）, 7.52～7.59（m, 3H, ArH）, 7.62～7.70（m, 2H, ArH）, 8.04（d, J = 8.4 Hz, 3H, ArH）, 8.14～8.23（m, 3H, ArH）, 8.34（d, J = 8.4 Hz, 2H, ArH）, 8.80（s, 1H, ArH）。^{13}C NMR（$CDCl_3$）δ: 14.2, 22.7, 26.2, 28.7, 29.2, 29.3, 29.4, 30.5, 31.9, 45.0, 120.9, 123.1, 123.8, 126.3, 126.7, 127.1, 127.2, 127.9, 128.0, 128.7, 128.9, 129.4, 130.4, 130.9, 131.1, 132.7, 134.2, 134.6, 138.2, 146.1, 164.1, 164.8。ESI-MS（m/z）: 631［M + H］$^+$。

2-(4-(2-(1-正癸基-4,5-二苯基咪唑基))苯基)-5-(2-噻吩基)-1,3,4-噁二唑（**3f**）: 无色固体, 产率40%, 熔点106～108 ℃。^{1}H NMR（DMSO-d_6）δ: 0.76（t, J = 6.6 Hz, 3H, CH_3）, 0.83～1.22（m, 10H, CH_2）, 1.22～1.36（m, 2H, CH_2）, 3.97（t, J = 7.2 Hz, 2H, NCH_2）, 7.09～7.23（m, 3H, ArH）, 7.32～7.35（m, 1H, ArH）, 7.40～7.50（m, 4H, ArH）, 7.51～7.59（m, 3H, ArH）, 7.96～8.01（m, 4H, ArH）, 8.24（d, J = 8.4 Hz, 2H, ArH）。^{13}C NMR（$CDCl_3$）δ: 14.1,

22.6, 26.1, 28.6, 29.1, 29.2, 29.3, 30.4, 31.8, 44.9, 123.4, 124.9, 126.2, 126.6, 126.9, 127.9, 128.0, 128.6, 128.9, 129.3, 129.7, 130.1, 130.4, 130.8, 131.0, 134.2, 134.6, 138.2, 146.0, 160.8, 163.4。ESI-MS (m/z): 587 $[M + H]^+$。

2-(4-(2-(1-正癸基菲并[9,10-d]咪唑基))苯基)-5-苯基-1,3,4-噁二唑 (**3g**): 浅黄色固体, 产率 36%, 熔点 160 ~ 162 ℃。^{1}H NMR ($CDCl_3$) δ: 0.82 (t, J = 6.6 Hz, 3H, CH_3), 1.07 ~ 1.30 (m, 14H, CH_2), 1.86 ~ 2.03 (m, 2H, CH_2), 4.69 (t, J = 6.9 Hz, 2H, NCH_2), 7.49 ~ 7.74 (m, 7H, ArH), 8.00 (d, J = 7.5 Hz, 2H, ArH), 8.10 ~ 8.20 (m, 2H, ArH), 8.27 (d, J = 7.5 Hz, 1H, ArH), 8.32 (d, J = 7.5 Hz, 2H, ArH), 8.67 (d, J = 7.8 Hz, 1H, ArH), 8.81 (t, J = 7.2 Hz, 2H, ArH)。^{13}C NMR ($CDCl_3$) δ: 14.1, 22.6, 26.2, 28.8, 29.2, 29.3, 29.4, 30.1, 31.8, 47.3, 120.8, 122.7, 122.8, 123.6, 124.3, 124.7, 125.1, 125.8, 126.2, 126.8, 126.9, 127.3, 128.1, 128.9, 129.2, 130.6, 131.7, 150.4, 163.8, 164.6。ESI-MS(m/z): 579 $[M + H]^+$。

2-(4-(2-(1-正癸基菲并[9,10-d]咪唑基))苯基)-5-(2-萘基)-1,3,4-噁二唑 (**3h**): 黄色固体, 产率 30%, 熔点 151 ~ 153 ℃。^{1}H NMR ($CDCl_3$) δ: 0.83 (t, J = 6.6 Hz, 3H, CH_3), 1.17 ~ 1.30 (m, 14H, CH_2), 1.87 ~ 2.06 (m, 2H, CH_2), 4.73 (t, J = 7.2 Hz, 2H, NCH_2), 7.56 ~ 7.74 (m, 6H, ArH), 7.87 ~ 7.94 (m, 1H, ArH), 7.98 ~ 8.05 (m, 4H, ArH), 8.23 (d, J = 7.8, 1H, ArH), 8.30 (d, J = 7.5 Hz, 1H, ArH), 8.39 (d, J = 8.4 Hz, 2H, ArH), 8.61 ~ 8.71 (m, 2H, ArH), 8.83 (d, J = 8.1 Hz, 1H, ArH), 8.87 (d, J = 8.4 Hz, 1H, ArH)。^{13}C NMR ($CDCl_3$) δ: 14.1, 22.6, 26.2, 28.8, 29.2, 29.3, 29.4, 30.1, 31.8, 47.4, 120.7, 120.8, 122.7, 122.8, 122.9, 124.2, 124.7, 125.2, 125.9, 126.1, 126.8, 126.9, 127.2, 127.3, 127.7, 127.8, 128.1, 128.6, 128.8, 129.2, 130.7, 132.6, 134.5, 150.2, 163.6, 164.7。ESI-MS (m/z): 629 $[M + H]^+$。

2-(4-(2-(1-正癸基菲并[9,10-d]咪唑基)苯基)-5-(2-噻吩基)-1,3,4-噁二唑 (**3i**): 黄色固体, 产率 32%, 熔点 118 ~ 119 ℃。^{1}H NMR ($CDCl_3$) δ: 0.79 (t, J = 6.6 Hz, 3H, CH_3), 1.02 ~ 1.07 (m, 14H, CH_2), 1.83 (m, 2H, CH_2), 4.95 (t, J = 7.2 Hz, 2H, NCH_2), 7.20 (t, J = 7.8 Hz, 1H, ArH), 7.39 (m, 1H, ArH), 7.47 (t, J = 7.8 Hz, 1H, ArH), 7.59 ~ 7.75 (m, 4H, ArH), 8.10 (d, J = 7.8 Hz, 2H, ArH), 8.23 (d, J = 8.1 Hz, 1H, ArH), 8.31 (t, J = 8.4 Hz, 3H, ArH), 8.50 (d, J = 7.8 Hz, 1H, ArH), 8.91 (d, J = 8.1 Hz, 1H, ArH)。^{13}C NMR ($CDCl_3$) δ: 13.9, 22.4, 25.8, 28.4, 29.0, 29.1, 29.2, 29.3, 31.6, 48.5, 120.7, 120.8, 121.6, 122.4, 124.1, 124.3, 124.7, 124.9, 126.6, 126.8, 127.4, 127.5, 128.0, 128.1, 128.3, 129.4, 130.2, 130.8, 131.6, 147.0, 160.8, 162.0。ESI-MS (m/z): 585 $[M + H]^+$。

3　结果与讨论

众所周知, 咪唑衍生物的溶解性往往较差, 尤其是具有大共轭体系的咪唑化合物, 如本文中的 4-(2-咪唑基) 苯甲酸甲酯衍生物, 其溶解性更差, 往往不溶于一般的有机溶剂, 使其在合成应用方面受到极大的限制。因此, 本合成研究的首要工作是实现 4-(2-咪唑基) 苯甲酸甲酯衍生物的癸基化, 也是本合成工作的关键所在。为得到目标物, 采用了前期报道的 w = 50% K_2CO_3 - 丁酮 - TBAB 体系[19]。该体系对 4-(2-咪唑基) 苯甲酸甲酯衍生物的长链烷基化具有显著的优势, 产率为 75% 以上, 溶剂可循环使用, 产物的提纯较为简便, 适合批量生产。

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