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# Oxidative Aromatization of 1,3,5-Trisubstituted Pyrazolines Using Hydrogen Tetrachloroaurate as Catalyst Under an Oxygen Atmosphere

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### Oxidative Aromatization of 1,3,5-Trisubstituted Pyrazolines Using Hydrogen Tetrachloroaurate as Catalyst Under an Oxygen Atmosphere

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Today gold is without a doubt the "star metal" in chemistry because of the focus of attention on gold-catalyzed organic transformations in recent years.<sup>1–7</sup> Among various new transformations catalyzed by gold, those involving nucleophilic additions to C-C multiple bonds (alkynes, alkenes or allenes)<sup>8–10</sup> and catalytic C-H bond functionalization<sup>11,12</sup>, have been studied intensively. In contrast, gold-catalyzed oxidation chemistry, in particular gold as heterogeneous and/or homogeneous catalyst for selective oxidation reactions with economic and environmentally benign oxidants, such as dioxygen or hydrogen peroxide, has been less developed.<sup>13</sup> Thus far, representative oxidation transformations are mainly limited in oxidation of monoxide, <sup>14–16</sup> alcohols, <sup>17–24</sup> amines<sup>25,26</sup> and sulfides, <sup>27,28</sup> epoxidations of olefins, <sup>29</sup> oxidative cleavage of carbon-carbon multiple bonds, <sup>30,31</sup> *etc*. Therefore, it is still desirable to extend gold-catalyzed oxidation chemistry.

1,3,5-trisubstituted pyrazoles are one class of important compounds because heterocycles containing pyrazole moieties often exhibit valuable biological and medicinal activities, such as analgestic, anti-inflammatory, antipyretic, anti-arrhythmic, psychoanaleptic, antidiabetic and antibacterial ones.<sup>32–37</sup> Generally, oxidative aromatization of 1,3,5-trisubstituted pyrazolines which could be easily prepared from the condensation of chalcones with arylhydrazines,<sup>38</sup> is the most widely used protocol for synthesis of 1,3,5-trisubstituted pyrazole derivatives. To date, a variety of oxidants have been used for the purpose, *e.g.* zirconium (IV) nitrate,<sup>39</sup> lead (IV) tetraacetate,<sup>40</sup> silver (I) nitrate,<sup>41</sup> bismuth (III) nitrate,<sup>42</sup> manganese (IV) dioxide,<sup>43</sup> potassium permanganate,<sup>44</sup> mercury (II) oxide,<sup>45</sup> iodobenzene diacetate,<sup>46</sup> sodium nitrite and sodium nitrate in acetic acid,<sup>47</sup> trichloroisocyanuric acid,<sup>48</sup> 4-(*p*-chloro)phenyl-1,3,4-triazole-3,5-dione,<sup>49</sup> Pd/C,<sup>50</sup> iodine pentoxide or iodic acid,<sup>51</sup> activated carbon/oxygen system,<sup>52</sup> and N-hydroxyphthalimide (NHPI)/cobalt acetate/oxygen sytem,<sup>53</sup> *etc.* However, these processes have one or more limitations including high catalyst

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a)  $R^1 = C_6H_5$ ,  $R^2 = C_6H_5$ , b)  $R^1 = C_6H_5$ ,  $R^2 = 4-CH_3C_6H_4$ , c)  $R^1 = C_6H_5$ ,  $R^2 = 4-CH_3OC_6H_4$ , d)  $R^1 = C_6H_5$ ,  $R^2 = 4-CIC_6H_4$ , e)  $C_6H_5$ ,  $R^2 = 4-BrC_6H_4$ , f)  $R^1 = C_6H_5$ ,  $R^2 = 4-NO_2C_6H_4$ , g)  $R^1 = C_6H_5$ ,  $R^2 = 2,4-CI_2C_6H_3$ , h)  $R^1 = C_6H_5$ ,  $R^2 = 2-furyl$ , i)  $R^1 = 4-CIC_6H_4$ ,  $R^2 = C_6H_5$ , j)  $R^1 = 4-CIC_6H_4$ ,  $R^2 = 4-CIC_6H_4$ , k)  $R^1 = 4-BrC_6H_4$ ,  $R^2 = C_6H_5$ , l)  $R^1 = 4-BrC_6H_4$ ,  $R^2 = 4-CIC_6H_4$ , m)  $R^1 = 4-CH_3C_6H_4$ ,  $R^2 = 4-CIC_6H_4$ 

#### Figure 1

loading, or using stoichiometric, even excess amounts of oxidants (other than dioxygen) relative to the substrates, long reaction times, high temperature, troublesome for disposal problems, and using toxic reagents. As part of our continued effort in synthesis of useful heterocycles,<sup>54</sup> we now report an efficient oxidative aromatization of 1,3,5-trisubstituted pyrazolines to the corresponding pyrazoles in good to excellent yields by using only 1 mol% of hydrogen tetrachloroaurate as catalyst under oxygen atmosphere.

Initially, 1,3,5-trisubstituted pyrazoline **1a** was chosen as a model substrate for optimization of the reaction conditions (*Figure 1, Table 1*). The reaction hardly occurs in the

| Entry | Catalyst (0.01 mmol)                   | t<br>ol) Solvent<br>HOAc             | Temperature<br>(°C) | Time<br>(hrs)<br>2 | Yield<br>(%) <sup>b</sup><br><5 |  |  |  |  |  |
|-------|--|--------------------------------------|---------------------|--------------------|---------------------------------|--|--|--|--|--|
| 1     | none                                   |                                      | 50                  |                    |                                 |  |  |  |  |  |
| 2     | AuCl                                   | HOAc                                 | 50                  | 2                  | 55                              |  |  |  |  |  |
| 3     | NaAuCl <sub>4</sub> ·2H <sub>2</sub> O | HOAc                                 | 50                  | 2                  | 70                              |  |  |  |  |  |
| 4     | AuCl <sub>3</sub>                      | HOAc                                 | 50                  | 2                  | 83                              |  |  |  |  |  |
| 5     | $HAuCl_4 \cdot 4H_2O(3)$               | HOAc                                 | 50                  | 2                  | 95                              |  |  |  |  |  |
| 6     | 3                                      | HOAc                                 | 40                  | 2                  | 83 <sup>c</sup>                 |  |  |  |  |  |
| 7     | 3                                      | THF                                  | 50                  | 2                  | 91                              |  |  |  |  |  |
| 8     | 3                                      | EtOH                                 | 50                  | 2                  | 87                              |  |  |  |  |  |
| 9     | 3                                      | ClCH <sub>2</sub> CH <sub>2</sub> Cl | 50                  | 4                  | 85                              |  |  |  |  |  |
| 10    | 3                                      | Toluene                              | 50                  | 4                  | 84                              |  |  |  |  |  |
| 11    | 3                                      | CH <sub>3</sub> CN                   | 50                  | 4                  | 70                              |  |  |  |  |  |
| 12    | 3                                      | DMF                                  | 50                  | 4                  | 50                              |  |  |  |  |  |
| 13    | 3                                      | $CH_2Cl_2$                           | 50                  | 4                  | 38                              |  |  |  |  |  |
| 14    | 3                                      | Acetone                              | 50                  | 4                  | 16                              |  |  |  |  |  |

 Table 1

 Optimization of Reaction Conditions<sup>a</sup>

<sup>a</sup>) Carried out on 1 mmol scale of 1a in the presence of catalyst in 2 mL solvent under oxygen atmosphere (balloon).

<sup>b</sup>) Isolated yield.

c) 30% Aqueous H<sub>2</sub>O<sub>2</sub> solution (2 mmol) was used as oxidant.

absence of a gold catalyst (*Entry 1*). Treatment of **1a** with 1 mol% of AuCl at 50°C for 2 h in HOAc under an oxygen atmosphere gave the aromatized product **2a** in 55% yield (*Entry 2*). Under the catalysis of NaAuCl<sub>4</sub>·2H<sub>2</sub>O and AuCl<sub>3</sub>, the yield of **2a** was improved to be 70% and 83%, respectively (*Entries 3, 4*). HAuCl<sub>4</sub>·4H<sub>2</sub>O proved to be the ideal catalyst for conducting this reaction, giving **2a** in 95% yield when employed in HOAc at 50°C for 2 h under an oxygen atmosphere (*Entry 5*). The yield of **2a** decreased to 83% when 30% aqueous H<sub>2</sub>O<sub>2</sub> solution was used as oxidant catalyzed by HAuCl<sub>4</sub>·4H<sub>2</sub>O (*Entry 6*). The reaction was successful in THF, EtOH, toluene and 1,2-dichloroethane, which gave **2a** in good yields (*Entries 7–10*, 84%–91%); while CH<sub>3</sub>OH, CH<sub>3</sub>CN, CH<sub>2</sub>Cl<sub>2</sub> and acetone are not good choice (*Entries 11–14*).

With optimized conditions in hand, we next turned our attention to examine the scope of the reaction and the results are presented in *Table 2*. Various substituted pyrazolines were tested to establish its generality and efficiency, and all reactions proceeded smoothly under similar conditions. The experimental results showed that there is not much difference between substituent containing electron-donating and electron-withdrawing functionalities on  $R^1$  and  $R^2$  with respect to reaction times and yields of products. The present reaction conditions tolerate several functionalities such as halogen, nitro, methoxy and ether groups.

In summary, we have described an efficient protocol for oxidative aromatization of 1,3,5-trisubstituted pyrazolines to the corresponding pyrazoles by using only 1 mol% of HAuCl<sub>4</sub>·4H<sub>2</sub>O as catalyst under oxygen atmosphere. The advantages of the method are lower catalyst loading, good to excellent yields, mild reaction conditions, and simple operational procedures.

#### **Experimental Section**

Melting points are uncorrected. <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were obtained on a Bruker AVANCE III 500 (500 MHz) instrument in CDCl<sub>3</sub> using TMS as internal standard. Chemical shifts ( $\delta$ ) are expressed in ppm and coupling constants J are given in Hz. Mass spectra were obtained on a HP 5989B mass spectrometer. Elemental analyses were performed on an EA-1110 instrument. TLC analyses were performed on Merck silica gel 60 F<sub>254</sub>. GC-MS analyses were performed with an Agilent 6890N GC system equipped with a 5973N mass-selective detector. All gold catalysts were purchased from Aldrich.

#### **General Procedure**

A two-necked flask (25 mL) was charged with 1,3,5-trisubstituted pyrazolines **1** (1.0 mmol), HAuCl<sub>4</sub>·4H<sub>2</sub>O (4.0 mg, 0.01 mmol) and HOAc (2 mL). Then the flask was fitted with a balloon filled with dioxygen and the reaction mixture heated to 50°C and stirred for 2–4 h under oxygen atmosphere. Upon complete consumption of **1** monitored by TLC and GC-MS analyses, the resulting mixture was filtered to remove the gold catalyst, the filtrate was poured into saturated NaHCO<sub>3</sub> solution (10 mL) and extracted with ethyl acetate (40 mL × 3). The organic layer was dried over anhydrous MgSO<sub>4</sub>. After evaporation of solvent under vacuum and the residue was purified by chromatography using hexane/ethyl acetate (15:1) as eluent. All the products are isolated as white or light yellow solids.

The HAuCl<sub>4</sub>·4H<sub>2</sub>O/HOAc catalyst system could be directly reused at least for two cycles with similar activity and chemoselectivity after product 2 had been extracted with ether from the reaction mixture.<sup>28</sup>

|                           | 37 11     | mp.                          | hun p  | 134.0.0.00                | MS                       | Elemental Analysis<br>(Found) |        |        |
|---------------------------|-----------|------------------------------|--|---------------------------|--------------------------|-------------------------------|--------|--------|
| Cmpd                      | Yield (%) | (°C)<br>( <i>lit</i> . mp.)  | <sup>4</sup> H NMR $(\delta)$                                    | $^{13}N NMR$ ( $\delta$ ) | m/z<br>(M <sup>+</sup> ) | C                             | Н      | N      |
| $\frac{1}{2a^{50}}$       | 95        | 139.1–140.0                  | 7.93 (d. 2H, J = 8.5 Hz), 7.94-7.92                              | ()                        | 296                      |                               |        |        |
|                           | 20        | (141–142)                    | (m, 13H), 6.82 (s, 1H)   |                           | 270                      |                               |        |        |
| <b>2b</b> <sup>55</sup> 9 | 97        | 114.5–115.6                  | 7.81 (d, 2H, $J = 8.0$ Hz), 7.38–7.23                            |                           | 310                      |                               | _      | _      |
|                           |           | (116–117)                    | (m, 12H), 6.79 (s, 1H), 2.38 (s, 3H)                             | )                         |                          |                               |        |        |
| <b>2c</b> <sup>50</sup>   | 98        | 79.0–79.8                    | 7.93 (d, 2H, J = 8.0 Hz), 7.43-7.18                              |                           | 326                      | _                             | _      | _      |
|                           |           | (79–80)                      | (m, 10H), 6.83 (d, 2H, J = 8.5 Hz)                               | ,                         |                          |                               |        |        |
| 2450                      | 87        | 114 / 115 2                  | 6.76 (s, 1H), $3.79$ (s, 3H)<br>7 85 (d 2H I = 8 5 Hz) 7 40 7 25 |                           | 330                      |                               |        |        |
| 2u                        | 07        | (114-115)                    | (m, 12H), 6.79 (s, 1H)   |                           | 550                      |                               |        | _      |
| <b>2e</b> <sup>47</sup>   | 89        | 128.7–129.6                  | 7.77 (d, 2H, J = 8.0 Hz), 7.34-7.19                              |                           | 374                      | _                             | _      | _      |
|                           |           | (126–128)                    | (m, 12H), 6.75 (s, 1H)   |                           |                          |                               |        |        |
| <b>2f</b> <sup>50</sup>   | 77        | 142.1-142.9                  | 8.25 (d, 2H, J = 8.5 Hz), 8.05 (d, 2H)                           | ,                         | 341                      | —                             | _      | _      |
|                           |           | (142–144)                    | J = 8.5 Hz), 7.44–7.31 (m, 10H),                                 |                           |                          |                               |        |        |
| 20                        | 05        | 750 765                      | 6.89 (s, 1H)   | 151.90 120                | 00 264                   | 60.05                         | 2.96   | 7 67   |
| 2g                        | 05        | 75.9-70.5                    | (m, 11H) = 6.80 (s, 1H)  | 139.95 135                | .99, 304<br>60           | (69.03                        | (3.84) | (7.70) |
|                           |           |                              | (11, 111), 0.00 (3, 11)  | 134.80, 132               | .85,                     | (0).23)                       | (5.04) | (1.10) |
|                           |           |                              |  | 129.98, 129               | .02,                     |                               |        |        |
|                           |           |                              |  | 128.93, 128               | .76,                     |                               |        |        |
|                           |           |                              |  | 128.21, 127               | .41,                     |                               |        |        |
|                           |           |                              |  | 127.28, 125.              | .88,                     |                               |        |        |
| 2h                        | 81        | 108 2-109 3                  | 7.90 (d. 2H. I = 8.5 Hz) 7.48-7.38                               | 124.13, 107               | .00                      | 79 70                         | 4 93   | 9 78   |
|                           | 01        | 10012 10710                  | (m, 9H), 6.97 (s, 1H), 6.31                                      | 142.60, 140               | .31,                     | (79.86)                       | (4.96) | (9.74) |
|                           |           |                              | (d, 1H, J = 4.0 Hz), 5.96 (d, 1H,                                | 135.79, 132               | .83,                     |                               |        |        |
|                           |           |                              | J = 3.5 Hz)  | 129.14, 128               | .70,                     |                               |        |        |
|                           |           |                              |  | 128.47, 128               | .12,                     |                               |        |        |
|                           |           |                              |  | 126.04, 125               | .87,                     |                               |        |        |
|                           |           |                              |  | 103.41                    | .)0,                     |                               |        |        |
| 2i <sup>56</sup>          | 93        | 134.6-135.4                  | 7.85 (d, 2H, J = 8.0 Hz), 7.40–7.25                              |                           | 330                      | _                             |        | _      |
|                           |           | (137–140)                    | (m, 12H), 6.79 (s, 1H)   |                           |                          |                               |        |        |
| <b>2j</b> <sup>39</sup>   | 92        | 141.4–141.9                  | 7.82 (d, 2H, J = 8.0 Hz), 7.39-7.17                              |                           | 364                      | —                             | —      | _      |
| <b>a</b> 46               | 0.4       | (140–141)                    | (m, 11H), 6.76 (s, 1H)   |                           | 274                      |                               |        |        |
| 2K <sup>10</sup>          | 94        | 100.5 - 101.5<br>(156 - 157) | /./9 (d, 2H, J = 8.5 Hz), $/.55(d 2H J - 8.5 Hz), 7.36-7.25$     |                           | 3/4                      |                               | _      | _      |
|                           |           | (150 157)                    | (a, 2H, 3 = 0.5 Hz), 7.50 7.25<br>(m, 10H), 6.79 (s, 1H)         |                           |                          |                               |        |        |
| 21                        | 91        | 131.1-132.1                  | 7.80 (d, 2H, J = 8.5 Hz), 7.54                                   | 150.97, 143               | .40, 410                 | 61.56                         | 3.44   | 6.84   |
|                           |           |                              | (d, 2H, J = 8.5 Hz), 7.38–7.16                                   | 139.76, 134               | .53,                     | (61.42)                       | (3.41) | (6.88) |
|                           |           |                              | (m, 9H), 6.77 (s, 1H)  | 131.85, 131               | .82,                     |                               |        |        |
|                           |           |                              |  | 129.95, 129               | .13,                     |                               |        |        |
|                           |           |                              |  | 128.85, 128               | .79,<br>34               |                               |        |        |
|                           |           |                              |  | 125.30, 127               | .06.                     |                               |        |        |
|                           |           |                              |  | 105.18                    | - *                      |                               |        |        |
| 2m                        | 92        | 109.5-110.4                  | 7.80 (d, 2H, J = 8.0 Hz), 7.35–                                  | 152.18, 143               | .10, 344                 | 76.63                         | 4.97   | 8.12   |
|                           |           |                              | 7.18 (m, 11H), 6.77 (s, 1H), 2.38                                | 139.94, 137               | .94,                     | (76.79)                       | (4.92) | (8.16) |
|                           |           |                              | (s, 3H)  | 134.34, 130               | .05,                     |                               |        |        |
|                           |           |                              |  | 129.98, 129               | .42,<br>79               |                               |        |        |
|                           |           |                              |  | 127.63. 125               | ., ),<br>.74.            |                               |        |        |
|                           |           |                              |  | 125.36, 105               | .18,                     |                               |        |        |
|                           |           |                              |  | 21.37                     |                          |                               |        |        |

 Table 2

 Yields, mps., Elemental Analysis and Spectral Data of Compounds 2

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