ISSN 1070-4280, Russian Journal of Organic Chemistry, 2018, Vol. 54, No. 10, pp. 1505–1508. © Pleiades Publishing, Ltd., 2018.
Original Russian Text © V.A. Kobelevskaya, A.V. Popov, G.G. Levkovskaya, E.V. Rudyakova, I.B. Rozentsveig, 2018, published in Zhurnal Organicheskoi Khimii, 2018, Vol. 54, No. 10, pp. 1493–1496.

Regioselective Synthesis of 3-[2-(Alkylsulfanyl)ethyl]pyrazoles by Reaction of Alkanethiols with 3-Alkenylpyrazoles

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Received February 28, 2018

Abstract—3-Alkenyl-5-chloropyrazoles reacted with alkanethiols on heating to 60°C to afford in good yields 3-[2-(alkylsulfanyl)ethyl]-5-chloropyrazoles as a result of anti-Markovnikov addition to the alkenyl group.

DOI: 10.1134/S1070428018100111

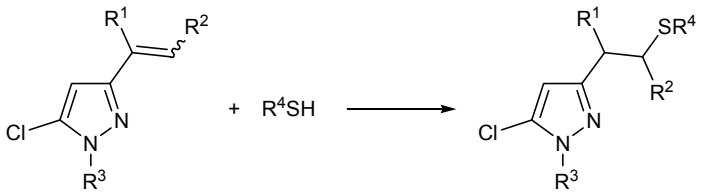
Pyrazole derivatives exhibit a broad spectrum of biological activity and are used for the preparation of materials for modern technologies, as reagents in organic synthesis [1–12], supramolecular [13], polymer [14], and coordination chemistry [15, 16], and in the synthesis of liquid crystals [17] and agrochemicals [4, 5, 7, 11].

Pyrazole derivatives with sulfur-containing substituents were shown to possess biological activity [8] and act as corrosion inhibitors [18]. Pyrazoles derivatives containing sulfide fragments are hemilabile ligands for metal complexes that are promising for commercial applications such as oligomerization of alkenes [19], as well as in cross-coupling reactions [20]. Some of these complexes showed antioxidant activity [21, 22] and can be used as model substrates in biochemical studies [23]. Development of efficient methods for the synthesis of pyrazole derivatives containing sulfide fragments, as well as synthesis of new compounds of this series, constitutes an important problem.

A promising method for the preparation of pyrazoles with alkylsulfanyl groups is based on the reaction of alkenylpyrazoles with sulfur nucleophiles. The addition of alkanethiols to 1-alkenylpyrazoles was reported [24, 25], but this reaction was not selective under conditions of both electrophilic assistance and radical initiation; as a result, mixtures of α- and β-addition products were formed.

We have developed efficient procedures for the synthesis of 3-alkenyl-5-chloropyrazoles [26]. Systematic studies of their reactivity showed that these compounds are highly active in reactions with benzene-

Scheme 1.



1, $R^1 = R^2 = H$, R^3 = Me (**a**), $PhCH_2$ (**b**), $Me_2CH(CH_2)_2$ (**c**); R^1 = Me, $R^2 = H$, $R^3 = PhCH_2$ (**d**); $R^1 = H$, R^2 = Me, $R^3 = PhCH_2$ (**e**); **2**, R^4 = Pr (**a**), Bu (**b**), *s*-Bu (**c**), C_6H_{13} (**d**), $PhCH_2$ (**e**), *i*-PrOC(O)CH_2 (**f**); **3**, $R^1 = R^2 = H$, R^3 = Me, R^4 = Pr (**a**), Bu (**b**), *s*-Bu (**c**), C_6H_{13} (**d**), $PhCH_2$ (**e**); $R^3 = PhCH_2$, R^4 = Bu (**f**), *i*-PrOC(O)CH_2 (**g**); $R^3 = Me_2CH(CH_2)_2$, $R^4 = PhCH_2$ (**h**); R^1 = Me, $R^2 = H$, $R^3 = R^4 = PhCH_2$ (**i**); $R^1 = H$, R^2 = Me, $R^3 = R^4 = PhCH_2$ (**j**).

thiols. The corresponding β-adducts were selectively formed at room temperature in 3 min [27]. 5-Chloro-3-vinylpyrazoles are capable of reacting with propane-1,3-dithiol, 1,3-disulfanylpropan-2-ol, and 1,3-diselanylpropan-2-ol to give linearly linked 3,3′-bispyrazoles containing sulfur or selenium in the side chain [28]. Furthermore, the possibility of addition of alkanethiols to the exocyclic double bond of 3-alkenyl-5-chloropyrazoles was demonstrated [29].

In continuation of our studies on the synthesis of new hemilabile ligands and reagents promising for subsequent transformations, herein we report the reaction of 1-substituted 3-vinyl-, 3-propenyl-, and 3-isopropenyl-5-chloropyrazoles with alkanethiols. By heating equimolar mixtures of 3-alkenyl-5-chloropyrazoles **1a**–**1e** with alkanethiols **2a**–**2f** we obtained the corresponding anti-Markovnikov adducts, 1-R-3-[(2-alkylsulfanyl)ethyl]-5-chloropyrazoles **3a**–**3j** (Scheme 1). The optimal conditions were found using the reaction of 5-chloro-3-ethenyl-1-methyl-1*H*-pyrazole (**1a**) with propane-1-thiol (**2a**) as model. The best yield of **3a** (83%) was obtained when the reactants were heated in benzene for 1 h at 60°C.

The product yield almost did not depend on the substituent on N^1 in the initial pyrazole. In the reactions of 1-methyl- and 1-benzyl-3-vinylpyrazoles **1a** and **1b** with thiols **2a**–**2d**, the optimal reaction time was 1 h, and the yields of the corresponding adducts were 76–85%. However, the reaction rate depended on the initial thiol. For instance, in the reactions of 3-alkenyl-1*H*-pyrazoles **1a** and **1c**–**1e** with phenylmethanethiol (**2e**), the optimal reaction time was 3 h, presumably due to lower nucleophilicity of that thiol. After 1 h at room temperature, the yield of **3e** was as low as 6%, and it increased to 41% after 18 h. Heating of the reactants for 1 h under solvent-free conditions afforded 68% of **3e**. At room temperature under radical initiation (UV irradiation, AIBN) the yield of **3e** in 1 h did not exceed 7%. In all cases, the products were exclusively anti-Markovnikov adducts **3a**–**3j**, and no isomeric α-adducts were detected.

Our results suggest that the reaction of 3-alkenyl-5-chloropyrazoles **1a**–**1e** with thiols can be regarded as nucleophilic addition of sulfur nucleophiles to alkenyl group which is somewhat activated due to effect of electron-deficient pyrazole ring. Electrophilic addition would give rise to the formation of Markovnikov adducts. Neither ultraviolet irradiation nor addition of radical initiators (such as AIBN) affected the reaction course; therefore, radical mechanism of the addition can be ruled out.

The structure of compounds **3a**–**3j** was proved by NMR, IR, and mass spectra and elemental analyses. The ^{1}H and ^{13}C NMR spectra of **3a**–**3j** lacked signals assignable to protons and carbons of alkenyl groups, whereas signals typical of organylsulfanyl fragments were observed. The ^{1}H NMR spectra of **3i** and **3j** characteristically displayed diastereotopicity of the methylene protons due to appearance in their molecules of asymmetric centers as a result of thiol addition to the double bond of the isopropenyl or propenyl group of **1d** or **1e**.

The regioselectivity of the formation of anti-Markovnikov adducts was reliable confirmed by comparison of their spectral data with those of authentic isomeric α-adducts synthesized previously [29] by reaction of thiols with 3-(1-chloroalkyl)pyrazoles.

The described optimized synthesis of functionalized pyrazoles containing alkylsulfanyl groups opens the way to promising reagents, ligands, and biologically active compounds. Compounds **3e** and **3f** were described previously [29], whereas the other pyrazoles **3** were isolated for the first time.

EXPERIMENTAL

The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker DPX-400 spectrometer at 400.61 and 100.13 Hz, respectively, from 3–5% solutions in $CDCl_3$. The chemical shifts were measured relative to tetramethylsilane with an accuracy of 0.01 (^{1}H) or 0.02 ppm (^{13}C). The IR spectra were recorded on a Bruker IFS-25 spectrometer from thin films.

5-Chloro-1-methyl-3-[2-(propylsulfanyl)ethyl]-1*H*-pyrazole (3a). A mixture of 0.143 g (1 mmol) of 5-chloro-3-ethenyl-1-methyl-1*H*-pyrazole (**1a**) and 0.076 g (1 mmol) of propane-1-thiol (**2a**) in 2 mL of benzene was heated for 1 h at 60°C. When the reaction was complete, the solvent was removed under reduced pressure, and the residue was washed with cold hexane and dried under reduced pressure. Yield 0.182 g (83%). IR spectrum, ν, cm^{-1}: 3124, 2954, 2924, 2870, 1993, 1515. ^{1}H NMR spectrum, δ, ppm: 0.99 t (3H, CH_3, J = 7.4 Hz), 1.62 m (2H, CH_2), 2.53 t (2H, CH_2, J = 7.4 Hz), 2.81 m (4H, CH_2), 3.76 s (3H, CH_3), 6.06 s (1H, 4-H). ^{13}C NMR spectrum, δ_C, ppm: 13.46, 22.94, 29.26, 31.51, 34.20, 35.88, 103.39, 127.31, 151.00. Found, %: C 49.53; H 6.88; Cl 16.17; N 12.79; S 14.63. $C_9H_{15}ClN_2S$. Calculated, %: C 49.42; H 6.91; Cl 16.21; N 12.81; S 14.66.

Pyrazoles **3b**–**3j** were synthesized in a similar way.

3-[2-(Butylsulfanyl)ethyl]-5-chloro-1-methyl-1*H*-pyrazole (3b) was synthesized from 0.143 g (1 mmol) of pyrazole **1a** and 0.090 g (1 mmol) of butane-1-thiol (**2b**). Yield 0.198 g (85%). IR spectrum, ν, cm^{-1}: 3125, 2952, 2929, 2866, 1696, 1514. ^1H NMR spectrum, δ, ppm: 0.90 t (3H, CH_3, J = 7.2 Hz), 1.39 m (2H, CH_2), 1.56 m (2H, CH_2), 2.53 t (2H, CH_2, J = 7.4 Hz), 2.78 m (4H, CH_2), 3.76 s (3H, CH_3), 6.04 s (1H, 4-H). ^{13}C NMR spectrum, δ_C, ppm: 13.61, 21.94, 29.21, 31.52, 31.68, 31.80, 35.84, 103.35, 127.25, 150.96. Found, %: C 51.70; H 7.38; Cl 15.19; N 11.99; S 13.74. $C_{10}H_{17}ClN_2S$. Calculated, %: C 51.60; H 7.36; Cl 15.23; N 12.04; S 13.77.

3-[2-(Butan-2-ylsulfanyl)ethyl]-5-chloro-1-methyl-1*H*-pyrazole (3c) was synthesized from 0.143 g (1 mmol) of **1a** and 0.090 g (1 mmol) of butane-2-thiol (**2c**). Yield 0.186 g (80%). IR spectrum, ν, cm^{-1}: 3125, 2962, 2925, 2875, 1692, 1514. ^1H NMR spectrum, δ, ppm: 0.95 t (3H, CH_3, J = 7.3 Hz), 1.25 d (3H, CH_3, J = 6.7 Hz), 1.55 m (2H, CH_2), 2.78 m (5H, CH_2, CH), 3.76 s (3H, CH_3), 6.04 s (1H, 4-H). ^{13}C NMR spectrum, δ_C, ppm: 11.32, 20.76, 29.41, 29.68, 29.77, 35.81, 41.71, 103.34, 127.21, 151.05. Found, %: C 51.70; H 7.38; Cl 15.19; N 11.99; S 13.74. $C_{10}H_{17}ClN_2S$. Calculated, %: C 51.60; H 7.36; Cl 15.23; N 12.04; S 13.77.

5-Chloro-3-[2-(hexylsulfanyl)ethyl]-1-methyl-1*H*-pyrazole (3d) was synthesized from 0.143 g (1 mmol) of **1a** and 0.118 g (1 mmol) of hexane-1-thiol (**2d**). Yield 0.198 g (76%). IR spectrum, ν, cm^{-1}: 3125, 2926, 2856, 1692, 1514. ^1H NMR spectrum, δ, ppm: 0.87 t (3H, CH_3, J = 7.4 Hz), 1.37 m (6H, CH_2), 1.58 m (2H, CH_2), 2.54 t (2H, CH_2, J = 7.4 Hz), 2.81 m (4H, CH_2), 3.78 s (3H, CH_3), 6.05 s (1H, 4-H). ^{13}C NMR spectrum, δ_C, ppm: 22.52, 28.56, 29.25, 29.61, 31.41, 31.57, 32.19, 35.87, 109.39, 127.29, 151.00. Found, %: C 55.20; H 8.09; Cl 13.62; N 10.77; S 12.32. $C_{12}H_{21}ClN_2S$. Calculated, %: C 55.26; H 8.12; Cl 13.59; N 10.74; S 12.29.

3-[2-(Benzylsulfanyl)ethyl]-5-chloro-1-methyl-1*H*-pyrazole (3e) was synthesized from 0.143 g (1 mmol) of **1a** and 0.124 g (1 mmol) of phenylmethanethiol (**2e**); reaction time 3 h. Yield 0.214 g (80%). IR spectrum, ν, cm^{-1}: 3130, 3084, 3061, 3027, 3003, 2941, 2119, 2850, 1601, 1513. ^1H NMR spectrum, δ, ppm: 2.64 t (2H, CH_2, J = 6.9 Hz), 2.76 t (2H, CH_2, J = 6.9 Hz), 3.66 s (2H, CH_2), 3.68 s (3H, CH_3), 5.94 s (1H, 4-H), 7.17–7.26 m (5H, C_6H_5). ^{13}C NMR spectrum, δ_C, ppm: 28.64, 30.67, 35.71, 36.15, 103.25, 126.80, 126.57, 128.32, 128.70, 138.22, 150.62. Found, %: C 58.42; H 5.69; Cl 13.27; N 10.54; S 12.08. $C_{13}H_{15}ClN_2S$. Calculated, %: C 58.53; H 5.67; Cl 13.29; N 10.50; S 12.02.

1-Benzyl-3-[2-(butylsulfanyl)ethyl]-5-chloro-1*H*-pyrazole (3f) was synthesized from 0.219 g (1 mmol) of 1-benzyl-5-chloro-3-ethenyl-1*H*-pyrazole (**1b**) and 0.090 g (1 mmol) of butane-1-thiol (**2b**). Yield 0.253 g (82%). IR spectrum, ν, cm^{-1}: 3131, 3082, 3060, 3027, 3004, 2940, 2933, 2920, 2850, 1600, 1515. ^1H NMR spectrum, δ, ppm: 0.91 t (3H, CH_3, J = 7.3 Hz), 1.43 m (2H, CH_2), 1.57 m (2H, CH_2), 2.60 m (4H, CH_2), 2.81 t (2H, CH_3, J = 6.9 Hz), 5.28 s (2H, CH_2), 6.12 s (1H, 4-H), 7.18–7.36 m (5H, C_6H_5). ^{13}C NMR spectrum, δ_C, ppm: 13.82, 22.09, 22.17, 29.57, 31.80, 32.08, 52.76, 104.18, 127.39, 127.97, 128.07, 128.84, 132.42, 151.79. Found, %: C 62.35; H 6.83; Cl 11.43; N 9.03; S 10.36. $C_{16}H_{21}ClN_2S$. Calculated, %: C 62.22; H 6.85; Cl 11.48; N 9.07; S 10.38.

Propan-2-yl {[2-(1-benzyl-5-chloro-1*H*-pyrazol-3-yl)ethyl]sulfanyl}acetate (3g) was synthesized from 0.219 g (1 mmol) of **1b** and 0.134 g (1 mmol) of isopropyl 2-sulfanylacetate (**2f**); reaction time 3 h. Yield 0.265 g (75%). IR spectrum, ν, cm^{-1}: 3131, 3089, 3065, 3032, 2980, 2933, 2878, 1720, 1606, 1515. ^1H NMR spectrum, δ, ppm: 1.22 d (6H, CH_3, J = 6.1 Hz), 2.83 m (4H, CH_2), 3.03 s (2H, CH_2), 4.92 sept (1H, CH, J = 6.1 Hz), 5.16 s (2H, CH_2), 6.01 s (1H, 4-H), 7.11–7.21 m (5H, C_6H_5). ^{13}C NMR spectrum, δ_C, ppm: 22.34, 29.02, 32.13, 33.79, 52.93, 68.32, 104.37, 126.86, 127.84, 128.13, 128.99, 136.72, 151.12, 169.11. Found, %: C 57.69; H 6.03; Cl 10.09; N 7.92; S 9.04. $C_{17}H_{21}ClN_2O_2S$. Calculated, %: C 57.86; H 6.00; Cl 10.05; N 7.94; S 9.09.

3-[2-(Benzylsulfanyl)ethyl]-5-chloro-1-(3-methylbutyl)-1*H*-pyrazole (3h) was synthesized from 0.199 g (1 mmol) of 5-chloro-3-ethenyl-1-(3-methylbutyl)-1*H*-pyrazole (**1c**) and 0.124 g (1 mmol) of thiol **2e**; reaction time 3 h. Yield 0.274 g (85%). IR spectrum, ν, cm^{-1}: 3132, 3062, 2957, 2928, 2872, 1695, 1600, 1512. ^1H NMR spectrum, δ, ppm: 0.92 d (6H, CH_3, J = 6.6 Hz), 1.66 m (3H, CH_2, CH), 2.65 m (2H, CH_2), 2.78 m (2H, CH_2), 3.70 s (2H, CH_2), 4.04 t (2H, CH_2, J = 7.7 Hz), 5.96 s (1H, 4-H), 7.21–7.29 m (5H, C_6H_5). ^{13}C NMR spectrum, δ_C, ppm: 22.28, 25.61, 28.83, 30.85, 36.23, 38.52, 47.34, 103.20, 126.39, 126.87, 128.39, 128.78, 138.31, 150.69. Found, %: C 63.42; H 7.14; Cl 10.92; N 8.65; S 9.87. $C_{17}H_{23}ClN_2S$. Calculated, %: C 63.24; H 7.18; Cl 10.98; N 8.68; S 9.93.

1-Benzyl-3-[1-(benzylsulfanyl)propan-2-yl]-5-chloro-1*H*-pyrazole (3i) was synthesized from

0.233 g (1 mmol) of 1-benzyl-5-chloro-3-(prop-1-en-2-yl)-1*H*-pyrazole (**1d**) and 0.124 g (1 mmol) of thiol **2e**; reaction time 3 h. Yield 0.278 g (78%). IR spectrum, ν, cm^{-1}: 3134, 3086, 3062, 3029, 2957, 2928, 2870, 1602, 1513. ^{1}H NMR spectrum, δ, ppm: 1.27 d (3H, CH_3, J = 7.0 Hz), 2.51 m (1H, CH_2), 2.69 m (1H, CH_2), 2.93 m (1H, CH), 3.59 s (2H, CH_2), 5.33 s (2H, CH_2), 5.97 s (1H, 4-H), 7.13–7.25 m (10H, C_6H_5). Found, %: C 67.22; H 5.96; Cl 9.97; N 7.83; S 9.02. $C_{20}H_{21}ClN_2S$. Calculated, %: C 67.31; H 5.93; Cl 9.93; N 7.85; S 8.98.

1-Benzyl-3-[2-(benzylsulfanyl)propyl]-5-chloro-1*H*-pyrazole (3j) was synthesized from 0.233 g (1 mmol) of 1-benzyl-5-chloro-3-(prop-1-en-1-yl)-1*H*-pyrazole (**1e**) and 0.090 g (1 mmol) of thiol **2e**; reaction time 3 h. Yield 0.250 g (70%). IR spectrum, ν, cm^{-1}: 3138, 3086, 3068, 3025, 2924, 2854, 1608, 1511. ^{1}H NMR spectrum, δ, ppm: 1.20 d (3H, CH_3, J = 6.3 Hz), 2.65 m (1H, CH), 2.85 m (2H, CH_2), 3.68 s (2H, CH_2), 5.23 s (2H, CH_2), 5.97 s (1H, 4-H), 7.12–7.24 m (10H, C_6H_5). Found, %: C 67.18; H 5.95; Cl 9.95; N 7.86; S 9.06. $C_{20}H_{21}ClN_2S$. Calculated, %: C 67.31; H 5.93; Cl 9.93; N 7.85; S 8.98.

The main results of this study were obtained using the facilities of the Baikal Joint Analytical Center, Siberian Branch, Russian Academy of Sciences.

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