Monatsh Chem (2018) 149:721–722
https://doi.org/10.1007/s00706-018-2149-3

CORRECTION

Correction to: Regioselective synthesis of pyridines by redox alkylation of pyridine *N*-oxides with malonates

Miran Lemmerer[1] · Christopher J. Teskey[1] · Daniel Kaiser[1] · Nuno Maulide[1]

Published online: 23 January 2018

Correction to: Monatsh Chem
https://doi.org/10.1007/s00706-017-2081-y

The original version of this article unfortunately contained mistakes. In section "General procedure" some numbers were missing. The corrected text is given below.

General procedure

All flasks and stirrer bars were flame dried before use. To the *N*-oxide (0.2 mmol, 1.0 equiv.), dissolved in 2 cm^3 dichloromethane was added Tf_2O (0.3 mmol, 1.5 equiv.) at 0 °C. In another flask, a suspension of NaH (0.7 mmol, 3.5 equiv.) in 1 cm^3 tetrahydrofuran was cooled to 0 °C and the malonate (0.7 mmol, 3.5 equiv.) was added. After 15 min, the malonate solution was added to the activated *N*-oxide solution and the mixture was stirred at room temperature for 1 h. The reaction was quenched with NH_4Cl solution and the aqueous phase was extracted with dichloromethane. The combined organic layers were washed with brine before being dried over $MgSO_4$. The solvents were removed under reduced pressure and the crude product was purified by column chromatography.

Dibenzyl 2-(2,6-dimethylpyridin-4-yl)malonate (**6a,** C24H23NO4) The product was prepared according to the general procedure. Purification by column chromatography (EtOAc:heptane = 1:1) yielded the product (41.0 mg, 53%) as a pale yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.28–7.20 (m, 10H), 6.88 (s, 2H), 5.11 (dd, J = 12.0, 18.1 Hz, 4H), 4.56 (s, 1H), 2.43 (s, 6H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): δ = 167.0, 158.4, 141.5, 135.1, 128.7, 128.7, 128.4, 120.9, 67.9, 57.3, 24.6 ppm; IR: $\bar{\nu}$ = 3064, 3033, 2955, 2922, 1732, 1605, 1569, 1497, 1453, 1375, 1297, 1140 cm^{-1}; HRMS (ESI): *m/z* calculated for $[M + H]^+$ 390.1700, found 390.1701.

Diethyl 2-(2,6-dimethylpyridin-4-yl)-2-fluoromalonate (**6b**, $C_{14}H_{18}FNO_4$) The product was prepared according to the general procedure. Purification by column chromatography (EtOAc:heptane = 1:3) yielded the product (34.3 mg, 61%) as a pale yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.19 (s, 2H), 4.33 (q, J = 7.1, 4H), 2.56 (s, 6H), 1.32 (t, J = 7.1 Hz, 6H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): δ = 164.9 (d, J = 25.0 Hz), 158.3, 142.4 (d, J = 22.4 Hz), 116.8 (d, J = 9.0 Hz), 93.2 (d, J = 202.9 Hz), 63.4, 24.8, 14.0 ppm; ^{19}F NMR (659 MHz, $CDCl_3$): − 165.2 ppm; IR: $\bar{\nu}$ = 2983, 2927, 1753, 1604, 1569, 1445, 1412, 1369, 1270, 1230, 1174, 1105, 1044, 1010 cm^{-1}; HRMS (ESI): *m/z* calculated for $[M + H]^+$ 284.1293, found 284.1292.

Diethyl 2-(2-cyanoethyl)-2-(2,6-dimethylpyridin-4-yl)-malonate (**6c**, $C_{17}H_{22}N_2O_4$) The product was prepared according to the general procedure. Purification by column chromatography (EtOAc:heptane = 1:1) yielded the product (48.4 mg, 76%) as a pink liquid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 6.90 (s, 2H), 4.32–4.24 (m, 4H), 2.61–2.57 (m, 2H), 2.54 (s, 6H), 2.37–2.33 (m, 2H), 1.28 (t, J = 7.1 Hz, 6H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ = 168.8, 158.6, 145.0, 119.0, 118.9, 62.6, 61.3, 32.0, 24.8, 14.0, 13.5 ppm; IR: $\bar{\nu}$ = 2982, 2937, 2249, 1728, 1603, 1564, 1445, 1368, 1254, 1188, 1079, 1016 cm^{-1}; HRMS (ESI): *m/z* calculated for $[M + H]^+$ 319.1652, found 319.1651.

The original article can be found online at https://doi.org/10.1007/s00706-017-2081-y.

✉ Nuno Maulide
nuno.maulide@univie.ac.at

[1] Institute of Organic Chemistry, University of Vienna, Währinger Strasse 38, 1090 Vienna, Austria

Diethyl 2-(2,6-dimethylpyridin-4-yl)-2-methylmalonate (**6d**, $C_{15}H_{21}NO_4$) The product was prepared according to the general procedure. Purification by column chromatography (EtOAc:heptane = 1:1) yielded the product (33.6 mg, 60%) as a pale yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 6.94$ (s, 2H), 4.25 (m, 4H), 2.52 (s, 6H), 1.81 (s, 3H), 1.26 (t, $J = 7.2$ Hz, 6H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): $\delta = 170.7$, 157.9, 147.8, 119.1, 62.1, 58.6, 24.8, 22.2, 14.1 ppm; IR: $\bar{\nu} = 2982$, 1728, 1604, 1564, 1447, 1414, 1377, 1253, 1181, 1105, 1017 cm^{-1}; HRMS (ESI): *m/z* calculated for $[M + H]^+$ 280.1543, found 280.1543.

Diethyl 2-allyl-2-(2,6-dimethylpyridin-4-yl)malonate (**7a**, $C_{17}H_{23}NO_4$) The product was prepared according to the general procedure. Purification by column chromatography (EtOAc:heptane = 1:3) yielded the product (43.6 mg, 71%) as a pale yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 7.01$ (s, 2H), 5.75–5.64 (m, 1H), 5.08 (m, 1H), 5.04(s, 1H), 4.29–4.16 (m, 4H), 3.00 (d, $J = 7.1$ Hz, 2H), 2.52 (s, 6H), 1.25 (t, $J = 7.1$, 6H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): $\delta = 169.5$, 157.8, 146.3, 132.5, 119.7, 119.4, 62.4, 62.0, 40.3, 24.8, 14.1 ppm; IR: $\bar{\nu} = 2981$, 2926, 1729, 1602, 1563, 1443, 1414, 1367, 1295, 1270, 1230, 1196, 1162 cm^{-1}; HRMS (ESI): *m/z* calculated for $[M + H]^+$ 306.1700, found 306.1703.

Diethyl 2-allyl-2-(4-methylpyridin-2-yl)malonate (**7b**, $C_{16}H_{21}NO_4$) The product was prepared according to the general procedure. Purification by column chromatography (EtOAc:heptane = 1:10) yielded the product (48.8 mg, 84%) as a pale yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 8.39$ (dd, $J = 0.5$, 5.0 Hz, 1H), 7.56 (app t, $J = 0.7$ Hz, 1H), 7.01–6.99 (m, 1H), 5.82–5.75 (m, 1H), 5.04–4.99 (m, 2H), 4.27–4.20 (m, 4H), 3.12 (d, $J = 7.2$ Hz, 2H), 2.36 (s, 3H), 1.24 (t, $J = 7.1$ Hz, 6H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): $\delta = 169.9$, 156.6, 148.6, 147.1, 133.6, 124.8, 123.5, 118.6, 65.3, 61.7, 40.4, 21.4, 14.1 ppm; IR: $\bar{\nu} = 2980$, 2936, 1729, 1601, 1444, 1298, 1195 cm^{-1}; HRMS (ESI): *m/z* calculated for $[M + H]^+$ 292.1543, found 292.1543.

Diethyl 2-allyl-2-(4-phenylpyridin-2-yl)malonate (**7c**, $C_{21}H_{23}NO_4$) The product was prepared according to the general procedure. Purification by column chromatography (EtOAc:heptane = 1:10) yielded the product (48.0 mg, 68%) as a pale yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 8.59$ (dd, $J = 0.7$, 5.1 Hz, 1H), 7.88 (dd, $J = 0.7$, 1.7 Hz, 1H), 7.65–7.63 (m, 2H), 7.48–7.41 (m, 4H), 5.86–5.79 (m, 1H), 5.06–5.02 (m, 2H), 4.30–4.24 (m, 4H), 3.17 (d, $J = 7.1$ Hz, 2H), 1.26 (t, $J = 7.1$ Hz, 6H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): $\delta = 169.8$, 157.2, 149.2, 148.5, 138.6, 133.3, 129.2, 129.1, 127.3, 122.4, 120.7, 119.0, 65.5, 61.8, 40.5, 14.2 ppm; IR: $\bar{\nu} = 3062$, 2980, 2935, 1729, 1594, 1547, 1467, 1225, 1036 cm^{-1}; HRMS (ESI): *m/z* calculated for $[M + H]^+$ 354.1700, found 354.1699.

Diethyl 2-allyl-2-(4-cyanopyridin-2-yl)malonate (**7d**, $C_{16}H_{18}N_2O_4$) The product was prepared according to the general procedure. Purification by column chromatography (EtOAc:heptane = 1:10) yielded the product (10.3 mg, 17%) as a pale yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 8.72$ (dd, $J = 0.9$, 5.0 Hz, 1H), 7.98 (app t, $J = 1.3$ Hz, 1H), 7.43 (dd, $J = 1.3$, 5.0 Hz, 1H), 5.74–5.63 (m, 1H), 5.04–5.01 (m, 2H), 4.29–4.22 (m, 4H), 3.12 (d, $J = 7.3$ Hz, 2H), 1.25 (t, $J = 7.1$ Hz, 6H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): $\delta = 169.0$, 158.5, 149.6, 132.4, 126.5, 124.0, 120.5, 119.7, 116.8, 65.3, 62.2, 40.3, 14.1 ppm; IR: $\bar{\nu} = 3077$, 2981, 2933, 2239, 1730, 1594, 1467, 1299, 1168, 1044 cm^{-1}; HRMS (ESI): *m/z* calculated for $[M + Na]^+$ 325.1159, found 325.1157.

The original article has been corrected.