Electronic supplementary information (ESI)

Antiviral Mechanism Study of Gossypol and Its Schiff’s Bases Derivatives Based on Reactive Oxygen Species (ROS)

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1. Analyze of the \( \text{H}_2\text{O}_2 \) and \( \text{O}_2^- \)

The most important thing for \( \text{H}_2\text{O}_2 \) or \( \text{O}_2^- \) determination was to find the suited standard curve of each compound.

\[
\text{NH}_2\text{OH} + 2\text{O}_2^- + \text{H}^+ \rightarrow \text{NO}_2^- + \text{H}_2\text{O}_2 + \text{H}_2\text{O}
\]

**Figure 1.** The mechanism of \( \text{O}_2^- \) detection.

The mechanism for the measurement of \( \text{O}_2^- \) is shown above. The concentration of \( \text{O}_2^- \) can be converted to \( \text{NaNO}_2 \) by the equation (1).

\[
(1) \quad c(\text{O}_2^-) = c(\text{NaNO}_2) \times 2
\]

The corresponding \( \text{NaNO}_2 \) values can be obtained by plotting against a standard curve of \( \text{NaNO}_2 \). As we can see in below, the equation (2) reflect the standard curve of \( \text{NaNO}_2 \) solution.

\[
(2) \quad A_{530} = c(\text{NaNO}_2) \times D + E
\]
In this equation, $A_{530}$ stand for the 530 nm absorbance data, and $c(NaNO_2)$ stand for the concentration of NaNO$_2$. The constant $E$ was the absorbance of blank sample ($c(NaNO_2) = 0$), which is a background of this method, and $D$ was related to its the sensitivity.

In theory, different gossypol schiff’s bases may have different $A_{530}$ at the same time, and the same gossypol schiff’s base could have different $A_{530}$ at different time. To make the equation more reasonable, we add a constant $F$, which represents the $A_{530}$ of gossypol schiff’s bases, to the equation (2). On the base of this, the equation (2) could be transfer to (3). For example, the equation suited for compound 11 (t = 1hr, 25 ℃) could be obtain by adding a constant $C$ ($A_{530} = 0.029$) to the standard curve of NaNO$_2$.

\begin{equation}
A_{530} = c(NaNO_2) \times D + E + F
\end{equation}

\[y = 0.006x + 0.0994\]
\[y = 0.006x + 0.0704\]

**Figure 2.** The modified curve of compound 11 (T= 1hr, 25 ℃) was obtain by adding a constant $F$ ($A_{530} = 0.029$) to the stand curve of NaNO$_2$.

Because the O$_2$ can exit in water in a very short time, the results were the sum of O$_2$ in certain period. The equation of the O$_2$ production rate could be describe as equation (5).
(4) \( c(O_2^-) = \frac{(A_{530} - E - F) \times 2}{D} \)

(5) \( rate(O_2^-) = \frac{(A_{530} - E - F) \times 2}{D \times T} \) \( (T < 20 \text{ min}) \)

The \( \text{H}_2\text{O}_2 \) concentration were measured by the equation (6).

(6) \( c(\text{H}_2\text{O}_2) = \frac{A_{560} - B - C}{A} \)

Figure 3. The water-solubility of compound 3-12, 26 (at 25 ℃) and their anti-TMV activity.

2. Data for key compounds (\(^1\text{H} \text{NMR, } \text{^{13}C} \text{NMR and HRMS } \))
Data for 3, yellow solid; yield, 53%; $^1$H NMR (400 MHz, DMSO-d$_6$), δ 13.64 (s, 2H), 9.67 (s, 2H), 8.60 (s, 2H), 7.35−7.22 (m, 12H), 4.86 (s, 2H), 3.66 (s, 2H), 1.83 (s, 6H), 1.42 (s, 12H); $^{13}$C NMR (100 MHz, DMSO-d$_6$), δ 171.8, 170.4, 162.3, 160.5, 146.6, 142.0, 135.5, 131.3, 129.2, 128.8, 128.6, 128.4, 127.4, 127.3, 126.5, 116.9, 104.3, 68.7, 64.4, 27.0, 20.9. HRMS(ESI) m/z calcd for C$_{46}$H$_{43}$N$_2$O$_{10}$ (M−2Na+H)$^-$ 783.29, found 783.29.

Data for 4, yellow solid, moisture absorption easily; yield, 72%; $^1$H NMR (400 MHz, DMSO-d$_6$), δ 13.19 (m, 2H), 9.62 (d, J=12.3Hz, 1H), 9.50 (d, J=12.3Hz, 1H), 8.45 (s, 2H), 7.38−7.09 (m, 14H), 3.96 (s, 2H), 3.68 (m, 2H), 3.09−2.85 (m, 4H), 1.92−1.89 (d, J=13.6H, 6H), 1.43 (t, J=5.6Hz, 12H); $^{13}$C NMR (100 MHz, DMSO-d$_6$), δ 171.5, 170.9, 160.5, 149.9, 146.4, 138.1, 130.7, 129.4, 128.1, 126.5, 126.0, 125.5, 120.5, 116.3, 116.1, 103.0, 65.9, 36.9, 26.4, 20.3. HRMS(ESI) m/z calcd for C$_{48}$H$_{47}$N$_2$O$_{10}$ (M−2Na+H)$^-$ 811.32, found 811.32.
Data for 5, yellow solid, moisture absorption easily; yield, 43%; $^1$H NMR (400 MHz, DMSO-$d_6$), δ 13.28-13.19 (m, 2H), 10.91 (s, 1H), 10.75 (s, 1H), 9.63 (s, 1H), 9.44 (s, 1H), 8.47 (s, 2H), 7.53–6.84 (m, 12H), 4.04 (s, 2H), 3.66 (m, 2H), 3.15-3.06 (m, 4H), 1.88 (d, J=8Hz, 6H), 1.42 (s, 12H); $^{13}$C NMR (100 MHz, DMSO-$d_6$), δ 172.3, 171.1, 161.1, 146.9, 136.6, 131.0, 127.8, 127.0, 125.7, 124.4, 124.2, 121.0, 118.8, 118.6, 116.6, 111.6, 110.7, 103.27, 65.3, 55.1, 31.2, 26.9, 20.9. HRMS(ESI) m/z calcd for C$_{52}$H$_{49}$N$_4$O$_{10}$ (M−2Na+H)$^+$ 889.34, found 889.34.

Data for 6, yellow solid, moisture absorption easily; yield, 39%; $^1$H NMR (400 MHz, DMSO-$d_6$), δ 13.16 (s, 2H), 9.69-9.65 (m, 2H), 8.51 (s, 2H), 7.40-7.39 (m, 2H), 3.89-3.88 (m, 2H), 3.68-3.62 (m, 4H), 1.92 (s, 6H), 1.43–1.41 (m, 12H), 1.05–1.02 (m, 6H); $^{13}$C NMR (100 MHz, DMSO-$d_6$), δ 172.0, 171.3, 161.7, 151.8, 146.7, 131.2, 126.8, 126.2, 122.7, 117.1, 115.9, 103.9, 70.1, 67.7, 55.5, 27.0, 22.7, 20.9, 20.2. HRMS(ESI) m/z calcd for C$_{38}$H$_{43}$N$_2$O$_{12}$ (M−2Na+H)$^+$ 719.28, found 719.28.

Data for 7, yellow solid, moisture absorption easily; yield, 67%; $^1$H NMR (400 MHz, DMSO-$d_6$), δ 13.25 (s, 2H), 9.75 (dd, $J = 21.3$, 12.8 Hz, 2H), 8.52 (s, 2H), 7.39 (d, $J = 6.1$ Hz, 2H), 3.81 – 3.51 (m, 10H), 1.91 (d, $J =10.8$Hz, 6H), 1.43-1.42 (m, 12H); $^{13}$C NMR (100 MHz, DMSO-$d_6$), δ 170.8, 169.8, 161.0, 149.9, 146.4, 130.6, 126.5, 125.4,
120.2, 116.5, 116.1, 103.2, 66.4, 63.8, 60.7, 26.4, 20.4, 20.2. HRMS(ESI) m/z calcd for C₃₆H₃₉N₂O₁₂ (M−2Na+H)⁻ 691.24, found 691.24.

Data for 8, yellow solid; yield, 86%; ¹H NMR (400 MHz, DMSO-d₆), δ 13.25 (s, 2H), 9.74 (s, 2H), 8.47 (s, 2H), 7.37 (d, J = 12.0 Hz, 2H), 4.18-4.09 (m, 2H), 3.80-3.78 (m, 4H), 3.00-2.90 (m, 2H), 1.88 (t, J = 8.0 Hz, 6H), 1.42-1.38 (m, 12H), 0.83 (s, 2H).

Data for 9, yellow solid; yield, 86%; ¹H NMR (400 MHz, DMSO-d₆), δ 13.20 (s, 2H), 9.68 (d, J = 12.5 Hz, 2H), 8.52 (s, 2H), 7.39 (s, 2H), 3.68 (m, 2H), 3.51 (s, 2H), 2.21 (m, 2H), 1.92 (s, 6H), 1.43 (t, J = 6.6 Hz, 12H), 0.88-0.84 (m,12H); ¹³C NMR (100 MHz, DMSO-d₆), δ 172.4, 171.4, 161.3, 150.7, 146.9, 131.1, 126.9, 126.1, 121.3, 117.0, 116.6, 103.5, 71.2, 32.1, 27.0, 20.9, 20.1, 17.7. HRMS(ESI) m/z calcd for C₄₀H₄₇N₂O₁₀ (M−2Na+H)⁺ 715.32, found 715.32.

Data for 10, yellow solid, moisture absorption easily; yield, 82%; ¹H NMR (400 MHz, DMSO-d₆), δ 13.27 (s, 2H), 9.77 (s, 2H), 8.51 (s, 2H), 7.39 (s, 2H), 3.77 (s, 2H), 3.69 (s, 2H), 1.92 (s, 6H), 1.65-1.57 (m, 6H), 1.43 (s, 12H), 0.86 (s, 12H); ¹³C NMR (100
MHz, DMSO-d6), δ 172.9, 171.3, 160.9, 151.0, 146.9, 131.3, 127.0, 126.1, 121.9, 117.3, 116.6, 103.6, 64.0, 43.8, 26.9, 24.6, 23.6, 22.13, 20.8. HRMS(ESI) m/z calcd for C_{42}H_{51}N_{10}O_{10} (M–2Na+H)^+ 743.35, found 743.35.

Data for 11, yellow solid, moisture absorption easily; yield, 43%; \(^1\)H NMR (400 MHz, DMSO-d6), δ 13.22 (s, 2H), 9.71 (s, 2H), 8.47 (s, 2H), 7.56 (s, 2H), 7.37 (s, 2H), 6.86 (d, J = 32.5 Hz, 2H), 4.05 (m, 2H), 3.69–3.66 (m, 2H), 2.75–2.71 (m, 2H), 2.44–2.38 (m, 2H), 1.92 (d, J = 12 Hz 6H), 1.43 (t, J = 6.0 Hz, 12H). \(^{13}\)C NMR (100 MHz, DMSO-d6), δ 172.0, 171.8, 170.7, 161.1, 151.2, 146.5, 130.6, 126.4, 125.5, 120.7, 116.4, 115.8, 103.0, 61.3 54.8, 26.4, 21.8, 20.3, 20.2. HRMS(ESI) m/z calcd for C_{38}H_{43}N_{10}O_{12} (M–2Na+3H)^+ 747.28, found 747.28.

Data for 12, yellow solid, moisture absorption easily; yield, 42%; \(^1\)H NMR (400 MHz, DMSO-d6), δ 13.22 (s, 2H), 9.74 (s, 2H), 8.48 (s, 2H), 7.38 (d, J = 10.4 Hz, 4H), 6.89 (s, 2H), 3.78–3.68 (m, 4H), 2.09–1.92 (m, 14H), 1.45–1.42 (m, 6H); \(^{13}\)C NMR (100 MHz, DMSO-d6), δ 174.3, 172.3, 171.4, 160.5, 151.0, 146.9, 131.2, 127.0, 126.1, 121.0 116.9, 109.9, 103.6, 64.5, 64.3, 56.49, 31.8, 30.8, 27.0, 20.9, 20.7. HRMS(ESI) m/z calcd for C_{40}H_{43}N_{10}O_{12} (M–2Na+3H)^+ 775.31, found 775.31.
Data for 13, yellow solid; yield, 71%; $^1$H NMR (400 MHz, DMSO-d$_6$), δ 13.50-13.44 (m, 2H), 10.93 (d, $J$ = 1.8 Hz, 2H), 9.76 (d, $J$ = 12.5 Hz, 1H), 9.65 (d, $J$ = 12.5 Hz, 1H), 8.45 (s, 2H), 7.84 (s, 1H), 7.61 (s, 1H), 7.47 – 6.92 (m, 12H), 4.78 (s, 6.3 Hz, 2H), 3.67 (s, 8H), 3.43 (s, 4H), 1.92 (s, 6H), 1.42 (d, $J$ = 6.7 Hz, 12H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 173.4, 170.5, 161.7, 148.8, 147.1, 136.3, 131.8, 129.1, 127.7, 126.8, 124.2, 122.2, 119.8, 118.3, 115.7, 114.5, 111.2, 108.9, 103.5, 62.8, 62.33, 53.0, 30.4, 27.4, 20.3, 19.9.

Data for 14, yellow solid; yield, 71%; $^1$H NMR (400 MHz, DMSO-d$_6$), δ 13.26–13.23 (m, 2H), 10.89 (s, 2H), 9.81 (d, $J$ = 12.4 Hz, 2H), 8.40 (s, 2H), 7.78 (brs, 2H), 7.59 (d, 2H), 7.44 (s, 2H), 7.33 (d, $J$ = 7.9 Hz, 2H), 7.21 (s, 2H), 7.05 (t, $J$ = 7.4 Hz, 2H), 6.96 (t, $J$ = 7.4 Hz, 2H), 3.84–3.69 (m, 6H), 3.09 (t, $J$ = 6.4 Hz, 4H), 1.94 (s, 6H), 1.45 (t, $J$ = 5.7 Hz, 12H). $^{13}$C NMR (100 MHz, DMSO--d$_6$), δ 172.2, 162.8, 150.5, 146.7, 136.8, 131.6, 127.4, 126.8, 123.7, 121.5, 120.8, 118.8, 116.8, 116.4, 111.9, 110.8, 103.7, 51.0, 27.0, 26.8, 20.9, 20.7. HRMS(ESI) m/z calcd for C$_{50}$H$_{49}$N$_4$O$_6$ (M–H)$^+$ 801.37, found 801.36.
Data for 15, yellow solid; yield, 76%; $^1$H NMR (400 MHz, DMSO-d$_6$), $\delta$ 13.48–13.43 (m, 2H), 9.73 (d, $J = 12.8$ Hz, 2H), 8.42 (s, 2H), 7.72 (s, 2H), 7.54–7.12 (m, 12H), 5.20 (s, 2H), 3.70-3.51 (m, 8H), 3.02-2.88 (m, 4H), 1.91 (s, 6H), 1.39 (m, 12H); $^{13}$C NMR (101 MHz, DMSO-d$_6$), $\delta$ 172.2, 162.5, 150.01, 146.7, 138.1, 131.6, 129.7, 128.9, 127.3, 126.9, 120.4, 117.0, 116.2, 103.7, 64.4, 63.7, 37.7, 27.0, 20.8, 20.7. HRMS(ESI) m/z calcd for C$_{48}$H$_{51}$N$_2$O$_8$ (M–H)$^+$ 783.37, found 783.36.

Data for 16, yellow solid, moisture absorption easily; yield, 70%; $^1$H NMR (400 MHz, DMSO-d$_6$), $\delta$ 13.16 (m, 2H), 9.62 (d, $J = 12.3$ Hz, 1H), 9.58 (d, $J = 12.3$ Hz, 1H), 8.46 (s, 2H), 7.38–7.09 (m, 14H), 3.96 (s, 2H), 3.68 (m, 2H), 3.09–2.85 (m, 4H), 1.90-1.87 (d, $J = 13.6$ Hz, 6H), 1.42 (t, $J = 5.6$ Hz, 12H).

Data for 17, yellow solid, moisture absorption easily; yield, 41%; $^1$H NMR (400 MHz, DMSO-d$_6$), $\delta$ 13.12 (s, 2H), 9.73–9.66 (m, 2H), 8.51 (s, 2H), 7.40-7.38 (m, 2H), 3.89-3.88 (m, 2H), 3.68-3.62 (m, 4H), 1.92 (s, 6H), 1.43–1.41 (m, 12H), 1.04–1.00 (m, 6H).
Data for **18**, yellow solid, moisture absorption easily; yield, 71%; $^1$H NMR (400 MHz, DMSO-d$_6$), δ 13.25 (s, 2H), 9.70 (m, 2H), 8.50 (s, 2H), 7.39 (d, $J = 6.1$ Hz, 2H), 3.76–3.50 (m, 10H), 1.92 (d, $J = 10.8$Hz, 6H), 1.45-1.42 (m, 12H).

Data for **19**, yellow solid, moisture absorption easily; yield, 46%; $^1$H NMR (400 MHz, DMSO-d$_6$), δ 13.22 (s, 2H), 9.71 (s, 2H), 8.48 (s, 2H), 7.56 (s, 2H), 7.37 (s, 2H), 6.86 (d, $J = 32.5$ Hz, 2H), 4.05 (m, 2H), 3.71–3.64 (m, 2H), 2.74-2.71 (m, 2H), 2.44-2.38(m, 2H), 1.92 (d, $J = 12$ Hz 6H), 1.43 (t, $J = 6.0$ Hz, 12H).

Data for **20**, yellow solid, moisture absorption easily; yield 47%; $^1$H NMR (400 MHz, DMSO-d$_6$), δ 13.28-13.19 (m, 2H), 10.91 (s, 1H), 10.75 (s, 1H), 9.63 (s, 1H), 9.42 (s, 1H), 8.47 (s, 2H), 7.53–6.82 (m, 12H), 4.04 (s, 2H), 3.69-3.63 (m, 2H), 3.39 (m, 2H), 3.16-3.06 (m, 4H), 1.87 (d, $J = 8$Hz, 6H), 1.42 (s, 12H).
Data for 21, yellow solid; yield, 71%; ¹H NMR (400 MHz, DMSO-d₆), δ 13.49-13.44 (s, 2H), 10.93 (s, 2H), 9.76 (d, J = 12.5 Hz, 1H), 9.66 (d, J = 12.5 Hz, 1H), 8.44 (s, 2H), 7.84 (s, 1H), 7.61 (s, 1H), 7.47–6.91 (m, 12H), 4.81-4.76 (m, 2H), 3.67 (d, J = 4.8 Hz, 8H), 3.43 (s, 4H), 1.92 (d, J = 4.8 Hz, 6H), 1.43 (d, J = 6.7 Hz, 12H).

Data for 22, yellow solid, moisture absorption easily; yield, 75%; ¹H NMR (300 MHz, DMSO-d₆), δ 13.47 (dd, J = 4.8, 12.5 Hz, 2H), 10.92 (s, 2H), 9.76 (d, J = 12.6 Hz, 2H), 8.44 (s, 2H), 7.81 (s, 2H), 7.48–6.91 (m, 12H), 4.82–4.76 (m, 2H), 3.66 (s, 8H), 3.46–3.35 (m, 4H), 1.93 (s, 6H), 1.43 (d, J = 6.4 Hz, 12H). ¹³C NMR (100 MHz, DMSO-d₆), δ 238.34, 173.12, 171.35, 170.3, 161.8, 150.2, 146.7, 136.6, 132.0, 127.6, 127.4, 124.7, 121.5, 121.0, 119.0, 118.5, 117.1, 116.2, 112.0, 108.2, 104.4, 62.5, 53.0, 29.4, 27.0, 20.8, 20.7.

Data for 23, yellow solid, moisture absorption easily; yield, 84%; ¹H NMR (300 MHz, DMSO-d₆), δ 13.48 (dd, J = 4.8, 12.5 Hz, 2H), 10.92 (s, 2H), 9.64 (d, J = 12.6 Hz, 2H), 8.44 (s, 2H), 7.61 (s, 2H), 7.48–6.89 (m, 12H), 4.82–4.76 (m, 2H), 3.67 (s, 8H),
3.43–3.35 (m, 4H), 1.92 (s, 6H), 1.42 (d, J = 6.4 Hz, 12H); $^{13}$C NMR (100 MHz, DMSO-$d_6$), δ 238.3, 173.1, 171.4, 170.3, 169.0, 161.8, 150.2, 146.7, 136.6, 132.0, 127.6, 127.4, 124.7, 121.5, 121.0, 119.0, 118.5, 117.1, 116.2, 111.0, 108.2, 104.4, 62.5, 53.0, 29.4, 27.0, 20.8, 20.7.

Data for 24, yellow solid, moisture absorption easily; yield, 84%; $^1$H NMR (300 MHz, DMSO-$d_6$), δ 13.51–13.45 (m, 2H), 10.92 (s, 2H), 9.77 (d, J = 12.6 Hz, 2H), 8.44 (s, 2H), 7.81 (s, 2H), 7.48–6.93 (m, 12H), 4.82–4.76 (m, 2H), 3.66 (s, 8H), 3.46–3.35 (m, 4H), 1.93 (s, 6H), 1.43 (d, J = 6.4 Hz, 12H).

Data for 25, yellow solid, moisture absorption easily; yield, 84%; $^1$H NMR (400 MHz, DMSO-$d_6$), δ 13.48–13.43 (m, 2H), 10.92 (s, 2H), 9.64 (d, J = 12.6 Hz, 2H), 8.43 (s, 2H), 7.61 (s, 2H), 7.48–6.89 (m, 12H), 4.82–4.76 (m, 2H), 3.67 (s, 8H), 3.43–3.35 (m, 4H), 1.92 (s, 6H), 1.42 (d, J = 6.4 Hz, 12H).

Data for 26, yellow solid, moisture absorption easily; yield, 88%; $^1$H NMR (400 MHz, DMSO-$d_6$), δ 13.25-13.18 (s, 2H), 9.75 (t, J = 12.8 Hz, 2H), 8.50 (s, 2H), 7.39 (s, 2H), 3.88–3.79 (m, 2H), 3.72–3.65 (m, 2H), 2.08–1.81 (m, 20H), 1.45–1.38 (m, 12H).
3. Spectrums of key compounds (\textsuperscript{1}H NMR, \textsuperscript{13}C NMR and HRMS)

\textsuperscript{1}H NMR spectrum of compound 3.
$^{13}$C NMR spectrum of compound 3.

HRMS of compound 3.
$^1$H NMR spectrum of compound 4.
\(^{13}\text{C}\) NMR spectrum of compound 4.

HRMS of compound 4.
$^1$H NMR spectrum of compound 5.
HRMS of compound 5.

$^{1}$H NMR spectrum of compound 6.
$^{13}$C NMR spectrum of compound 6.
HRMS of compound 6.
$^1$H NMR spectrum of compound 7.

$^{13}$C NMR spectrum of compound 7.
HRMS of compound 7.

\[ \text{H NMR spectrum of compound 8.} \]
HRMS of compound 8.

\[\text{H NMR spectrum of compound 9.}\]
$^{13}$C NMR spectrum of compound 9.
HRMS of compound 9.

$^1$H NMR spectrum of compound 10.
$^{13}$C NMR spectrum of compound 10.

HRMS of compound 10.
$^1$H NMR spectrum of compound 11.

$^{13}$C NMR spectrum of compound 11.
HRMS of compound 11.
$^1$H NMR spectrum of compound 12.

$^{13}$C NMR spectrum of compound 12.
HRMS of compound 12.

1H NMR spectrum of compound 13.
$^{13}$C NMR spectrum of compound 13.

$^1$H NMR spectrum of compound 14.
$^{13}$C NMR spectrum of compound 14.
HRMS of compound 14.

\[ \text{ESI Scan (0.681-0.698 min; 2 scans) Frag=175.0V Z8.1-10.d Subtract (2)} \]

\[ \text{801.3606} \]

\[ \text{659.2728} \]

\[ 433.2332 \]

\[ \text{Counts (%)} \text{ vs. Mass-to-Charge m/z} \]

\[ 400 \quad 450 \quad 500 \quad 550 \quad 600 \quad 650 \quad 700 \quad 750 \quad 800 \quad 850 \quad 900 \quad 950 \quad 1000 \quad 1050 \quad 1100 \quad 1150 \quad 1200 \]

\[ \text{H NMR spectrum of compound 15.} \]

\[ \text{13C NMR spectrum of compound 15.} \]
HRMS of compound 15.

1H NMR spectrum of compound 16.
$^1$H NMR spectrum of compound 17.
\(^1\text{H NMR spectrum of compound 18.}\)

\(^1\text{H NMR spectrum of compound 19.}\)
$^1$H NMR spectrum of compound 20.

$^1$H NMR spectrum of compound 21.
$^1$H NMR spectrum of compound 22.
$^{13}$C NMR spectrum of compound 22.

$^1$H NMR spectrum of compound 23.

$^{13}$C NMR spectrum of compound 23.
$^1$H NMR spectrum of compound 24.

$^1$H NMR spectrum of compound 25.
$^1$H NMR spectrum of compound 26.