

Supplementary Material

# Target-Guided Isolation of *O*-tigloylcyclovirobuxeine -B from *Buxus sempervirens* L. by Centrifugal Partition Chromatography

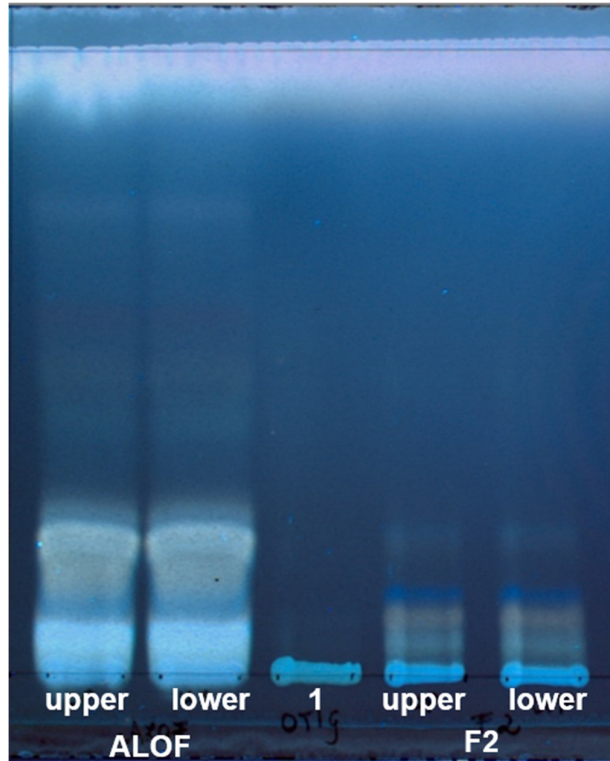
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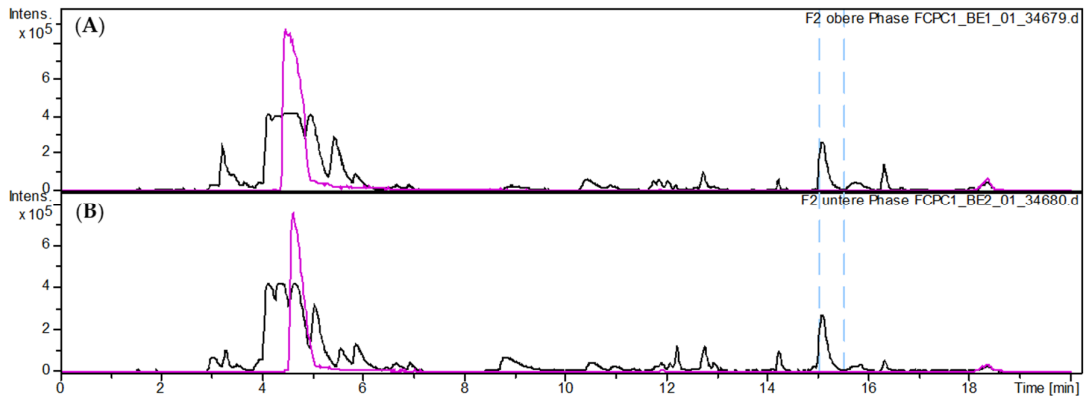
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**Table S1.** Solvent system selection process for the alkaloid fraction (ALOF).

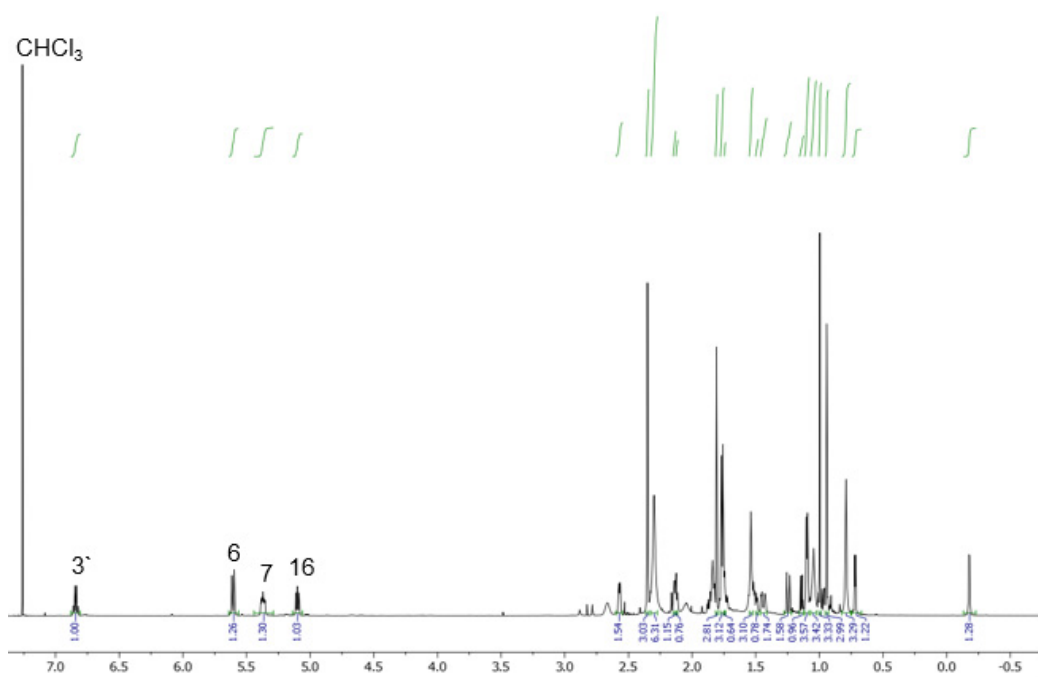
<b>Solvent system</b>	<b>Observation</b>
Chloroform : MeOH : PrOH : H <sub>2</sub> O : NH <sub>4</sub> OH (35 : 65 : 40 : 5 : 1)	No phase separation
Chloroform : MeOH : PrOH : H <sub>2</sub> O : NH <sub>4</sub> OH (35 : 65 : 5 : 40 : 1)	Emulsification
Hexane / ACN : DCM (10 : 7 : 3)	Enrichment in lower phase (TLC)
Hexane / EtOH : H <sub>2</sub> O (6 : 5 : 1)	Enrichment in lower phase (TLC)
Hexane : EtOAc / MeOH : H <sub>2</sub> O (7 : 3 : 7 : 3)	Equal distribution between the two phases (TLC) → K value determination by LC/MS



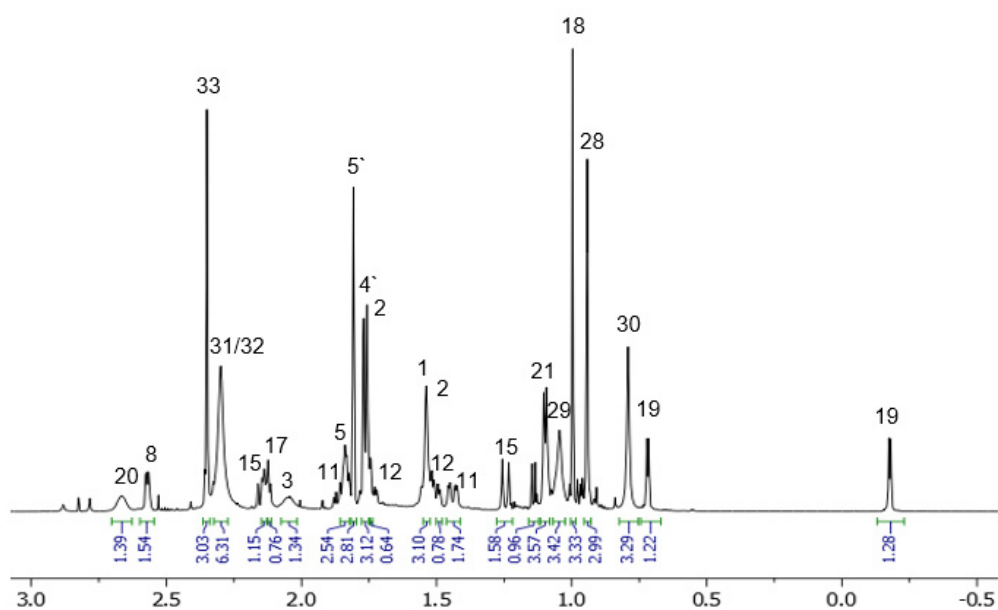
**Figure S1.** Determination of a suitable phase system for the fractionation by TLC. TLC plate of ALOF, O-tigloylcyclovirobuxeine-B (1) and fraction 2 (F2) visualized with Liebermann–Burchard spray reagents (366 nm). Note that these TLC separations were performed in order to check for equal distribution of ALOF and fraction 2 between the two phase, not for an optimal separation of these samples.



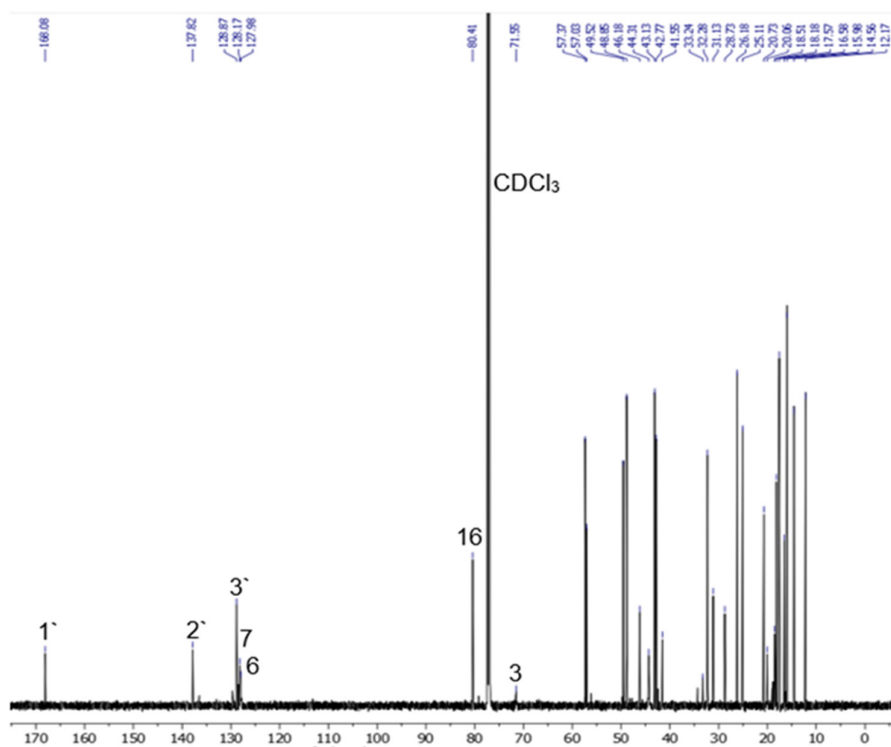
**Figure S2.** Determination of a suitable phase system for the fractionation of fraction 2 by UHPLC/ESI-QqTOF-MS. Base peak chromatogram of  $m/z$  200-1000 (black), Extracted ion chromatogram of  $m/z$  497  $[M + H]^+$  (pink); (A) upper phase; (B) lower phase.



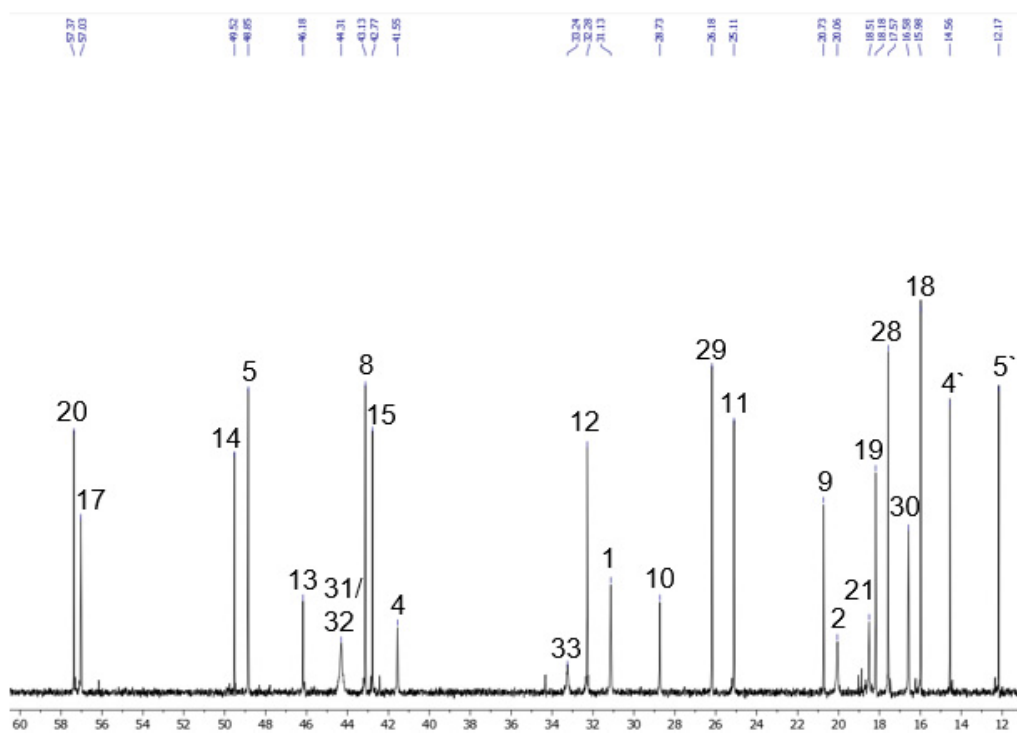
**Figure S3.**  $^1\text{H}$  NMR spectrum of O-tigloylcyclovirobuxeine-B (**1**) ( $\text{CDCl}_3$ , 600 MHz). The assignment of the signals between -0.5 and 3 ppm can be found in the expanded region shown in Figure S4.



**Figure S4.** Detail of the  $^1\text{H}$  NMR spectrum of O-tigloylcyclovirobuxeine-B (**1**) ( $\text{CDCl}_3$ , 600 MHz).



**Figure S5.**  $^{13}\text{C}$  NMR spectrum of O-tigoylcyclovirobuxeine-B (**1**) ( $\text{CDCl}_3$ , 150 MHz). The assignment of the signals between 10 and 60 ppm can be found in the expanded region shown in Figure S6.



**Figure S6.** Detail of the  $^{13}\text{C}$  NMR spectrum of O-tigoylcyclovirobuxeine-B (**1**) ( $\text{CDCl}_3$ , 150 MHz).

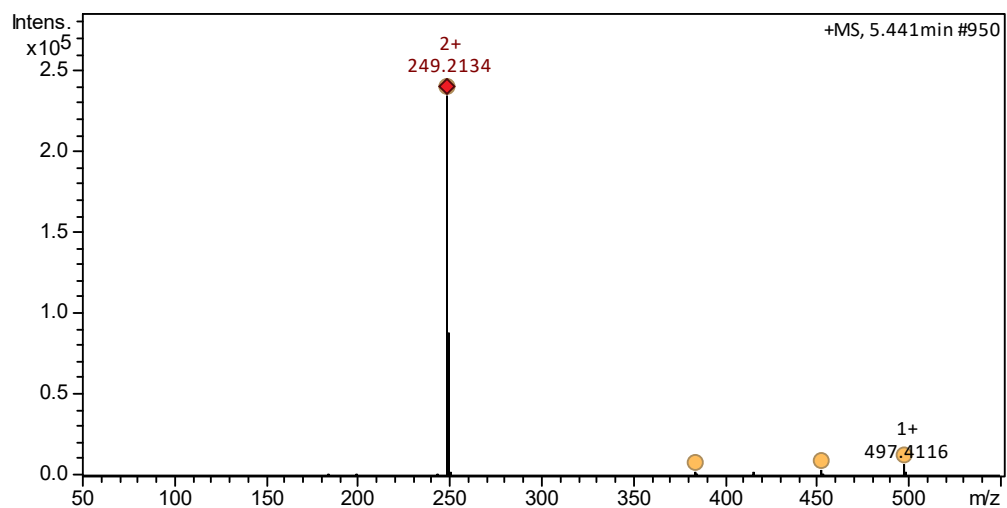


Figure S7. +ESI/QqTOF mass spectrum (full scan) of O-tigloylcyclovirobuxeine-B (1).

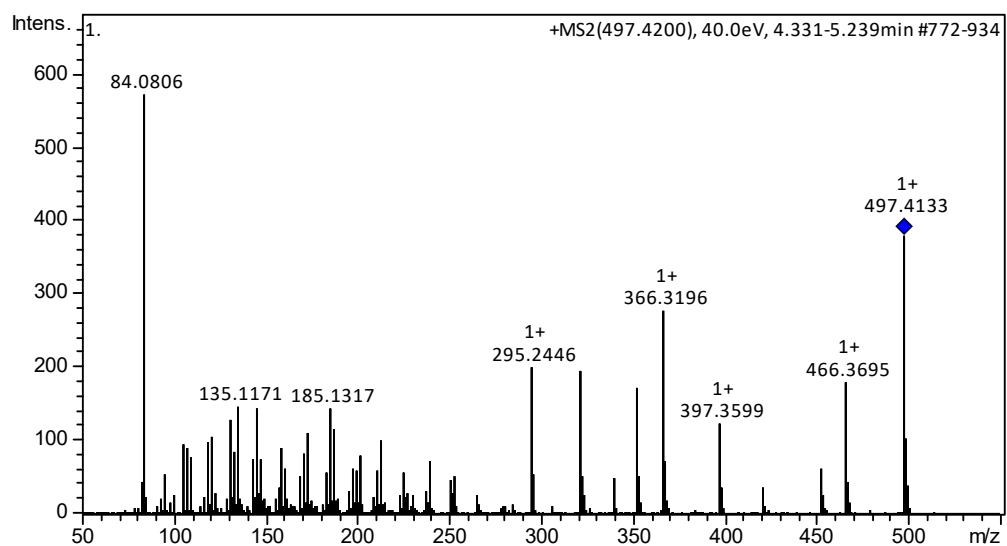
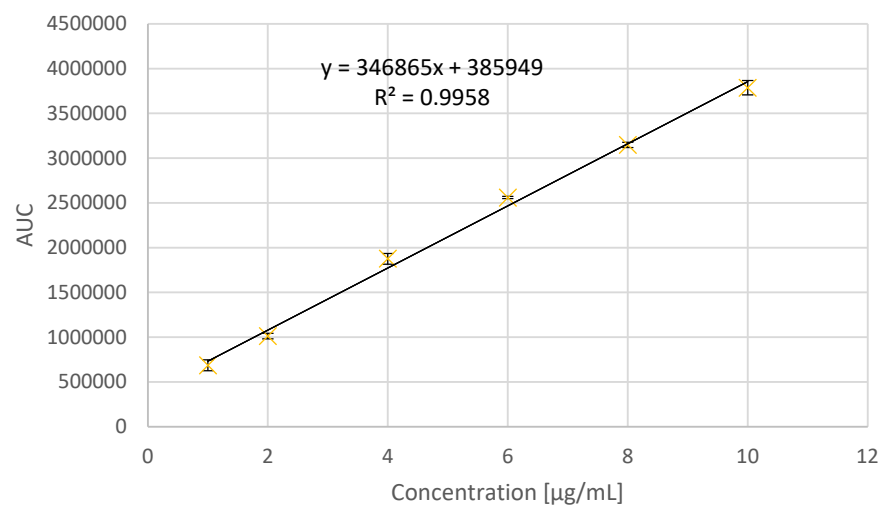


Figure S8. +ESI/QqTOF MS/MS spectrum of the  $[M + H]^+$  ion of O-tigloylcyclovirobuxeine-B (1). An expanded region of this spectrum is shown in Figure 5A of the main manuscript.



**Figure S9.** Calibration line for the quantitative determination of O-tigloylcyclovirobuxeine-B (1). Data were obtained by integration of LC/MS chromatograms (extracted ion chromatograms of the most stable ion,  $[M+2H]^{2+}$ ). Each point represents the mean of three analyses with error bars showing the standard deviation.