

Diterpenoids from *Pinus cembra* isolated by HPCCC and HSCCC assisted by APCI-MS profiling

Matheus L. Silva^{1*}, Pedro H. J. Batista¹, João H. G. Lago² and Gerold Jerz³

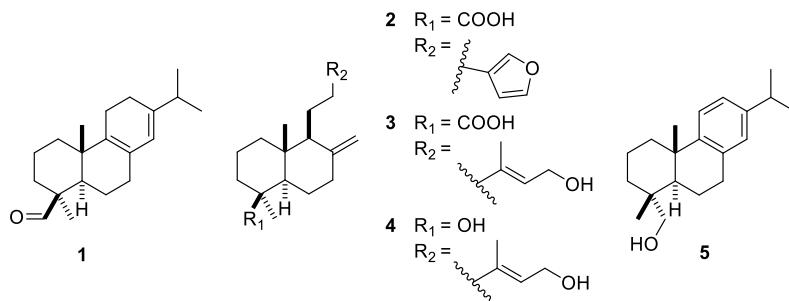
*matheus.silva@inpa.gov.br

¹*Instituto Nacional de Pesquisas da Amazônia, 69060-001, Manaus, AM, Brazil.*

²*Universidade Federal do ABC, 09280-560, Santo André, SP, Brazil.*

³*Institute for Food Chemistry, Technische Universität Braunschweig, 38106 Braunschweig, Germany.*

Pinus cembra, commonly known as Swiss stone pine, is an emblematic alpine conifer with ecological importance and a rich repertoire of secondary metabolites. While its seeds and essential oils are well documented, the chemical profile of cone scales has remained largely unexplored. In this work, air-dried cone scales (675 g) were extracted with *n*-hexane and yielded 8 g of crude extract. 650 mg of the hexane extract was fractionated using reversed-phase High-Performance Countercurrent Chromatography (HPCCC) with a *n*-hexane/ACN (1:1, v/v). 2 g of the same hexane extract was also fractionated with reversed-phase High-Speed Countercurrent Chromatography (HSCCC) with *n*-hexane/ACN/TBME (46:46:6, v/v/v). APCI-MS off-line injection profiling of HSCCC fractions enabled targeted tracking of diterpenoid-rich zones, improving fraction selection and structural assignment ^[1]. Subsequent purification by silica gel and RP-18 column chromatography afforded subfractions of high purity. Structure elucidation was carried out using NMR spectroscopy in combination with APCI-MS/MS fragmentation patterns. This workflow led to the isolation of five abietane diterpenoids: palustral (**1**), lambertianic acid (**2**), isocupressic acid (**3**), agathadiol (**4**), and 4-*epi*-dehydroabietol (**5**). This is the first time ¹³C NMR and MS data for palustral (**1**) shall be published. The findings not only contribute to chemophenetic understanding of *Pinus* genus but also open perspectives for potential bioactivity investigation from the isolated compounds.



Keywords: *Pinus cembra, diterpenes, countercurrent chromatography, APCI-MS profiling, palustral*

^[1] Präßler *et al.*, *J. Chromatogr. A*, 2025

