

# Purification and structural characterization of compounds isolated from *Cystoseira abies-marina*



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## Introduction

In the last three decades the discovery of metabolites with biological activities from macroalgae has significantly increased.<sup>[1]</sup> The ability of seaweeds to produce secondary metabolites of potential interest has been extensively documented. Although the Azores archipelago is rich in algal communities, only two papers have been published concerning the nutritional and pharmacological potential of this resource in the Azorean sea.<sup>[2,3]</sup> Barreto *et al.*<sup>[4]</sup> reported that some extracts from *Cystoseira abies-marina* had a significant antitumour activity (Table 1).

## Results and discussion

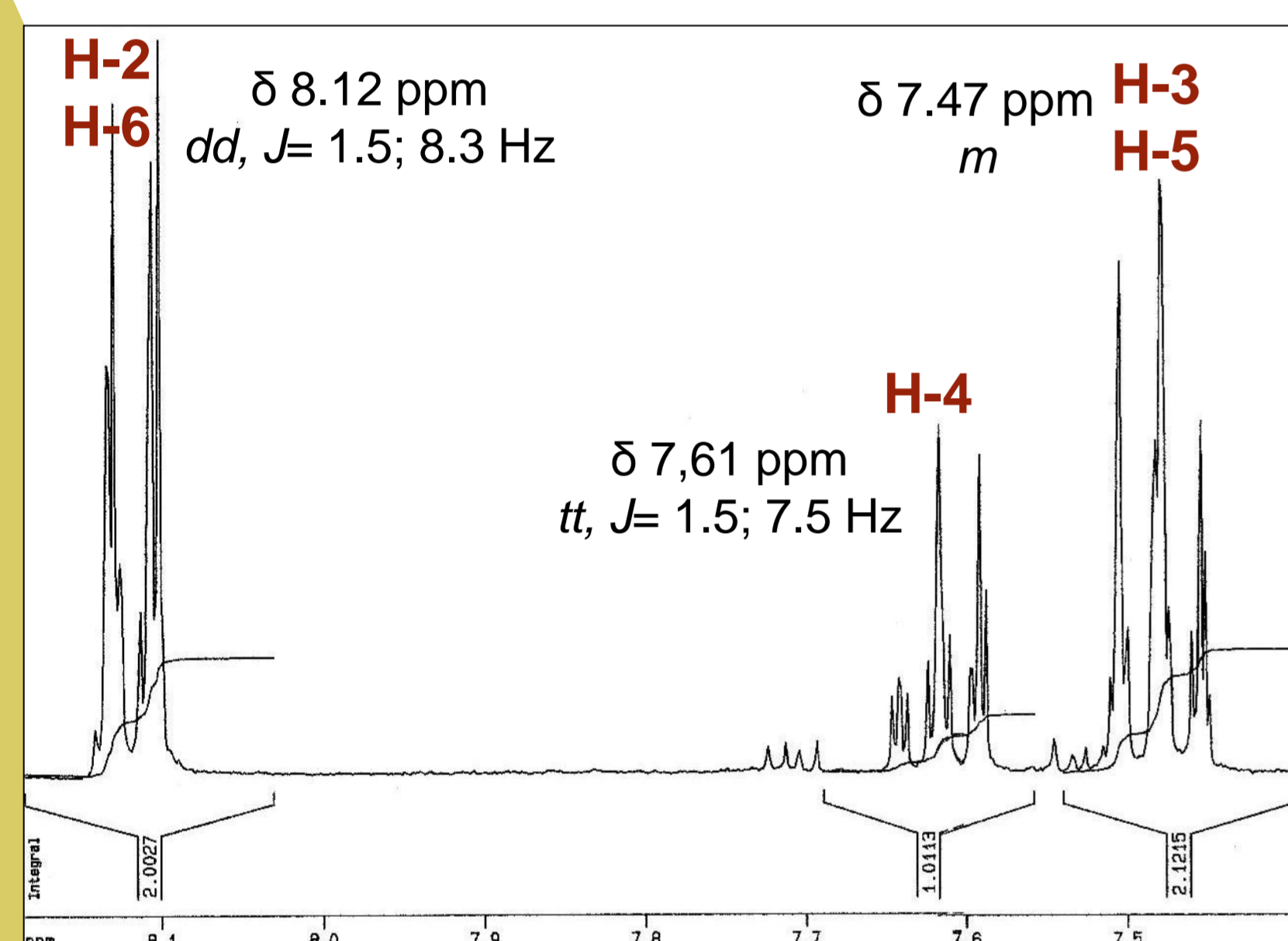
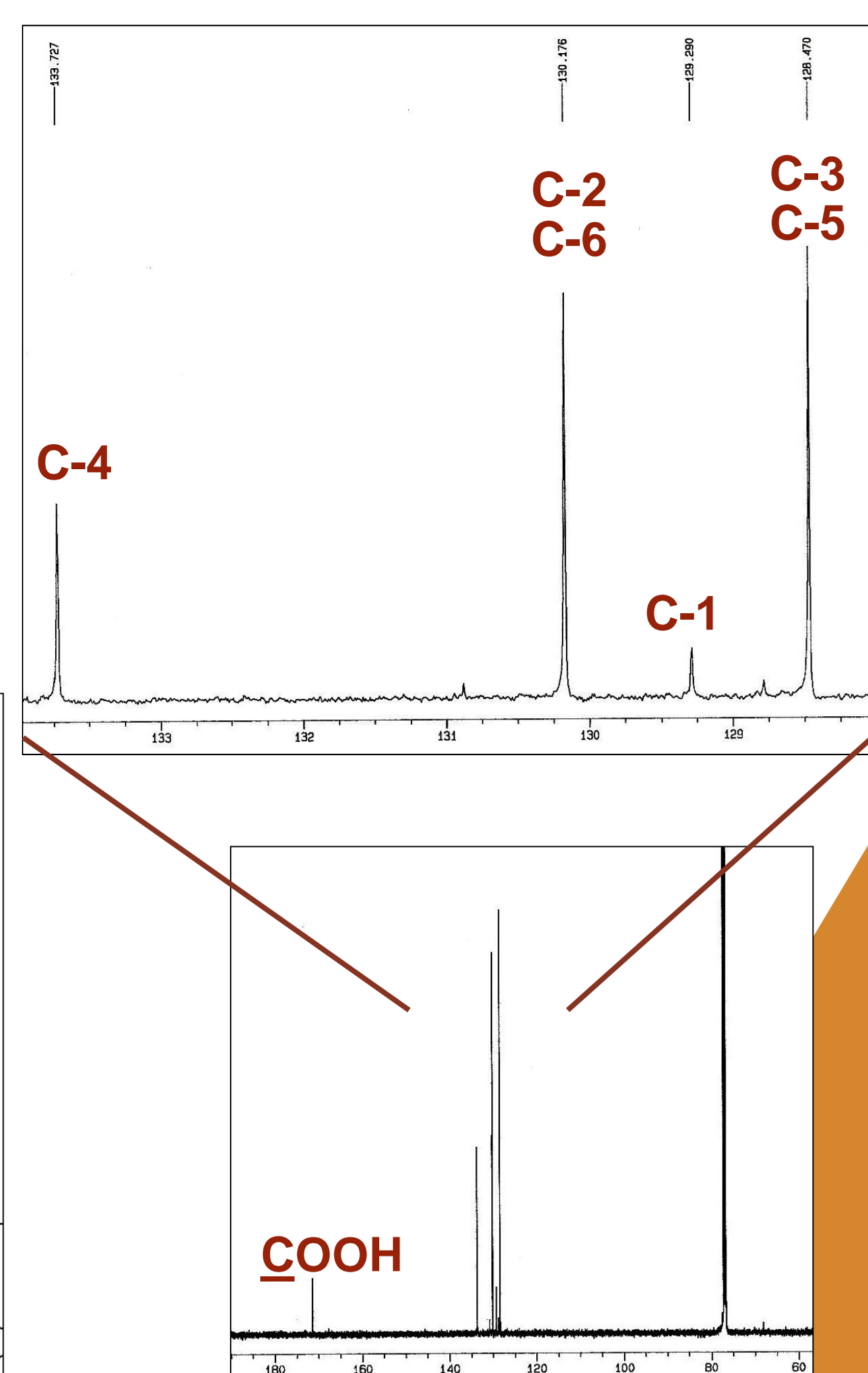
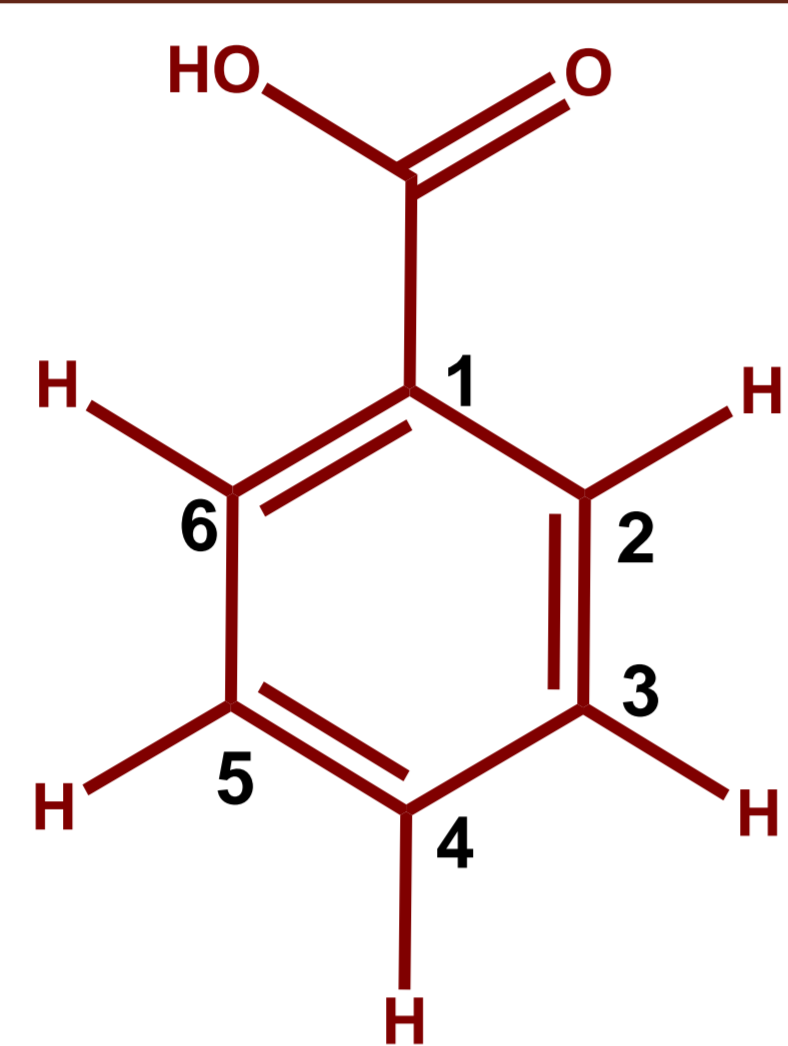
We now report isolation of benzoic acid (**1**) and two norsesquiterpenoids (**2** and **3**) from the brown alga *Cystoseira abies-marina*. Benzoic acid (**1**) was found in the winter collection while **2** and **3** were found in winter and spring collection. The unequivocal structural identification compounds **1-3** was achieved by detailed spectroscopic analysis. The compounds **2** and **3** were, for the first time, reported in nature.

Table 1. Cytotoxicity of *Cystoseira abies-marina* extracts and fractions.

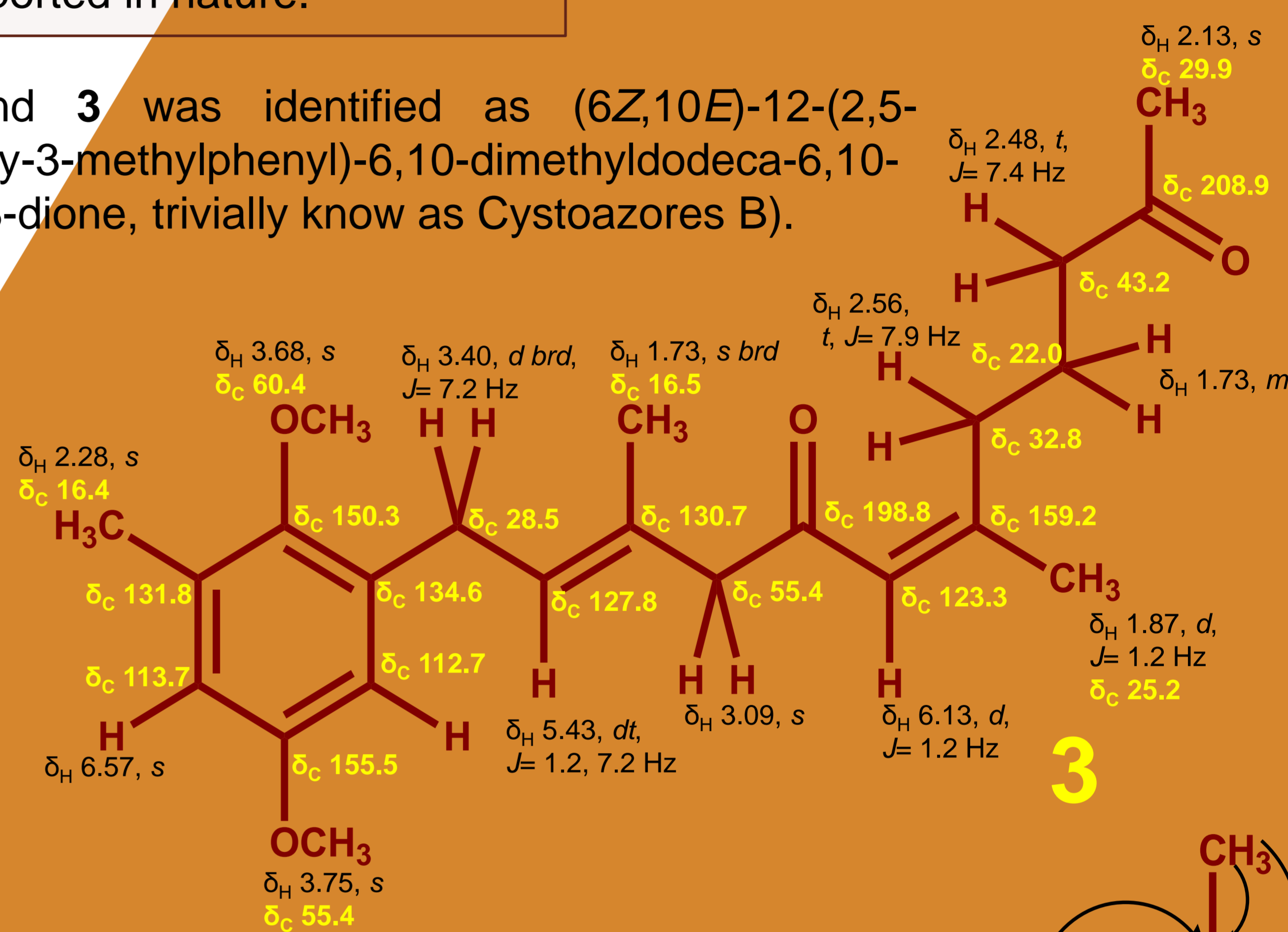
Macroalgae	a)	Cytotoxicity, IC <sub>50</sub> (µg/mL)	
		HeLa	Vero
<i>Cystoseira abies-marina</i>	MF	>200	>200
	DE	8.8 ± 1.29	48.2 ± 1.60
	HF	26.3 ± 2.06	47.8 ± 2.75

<sup>a)</sup>MF: methanol fraction; DE: dichloromethane extract; HF: Hexane fraction.

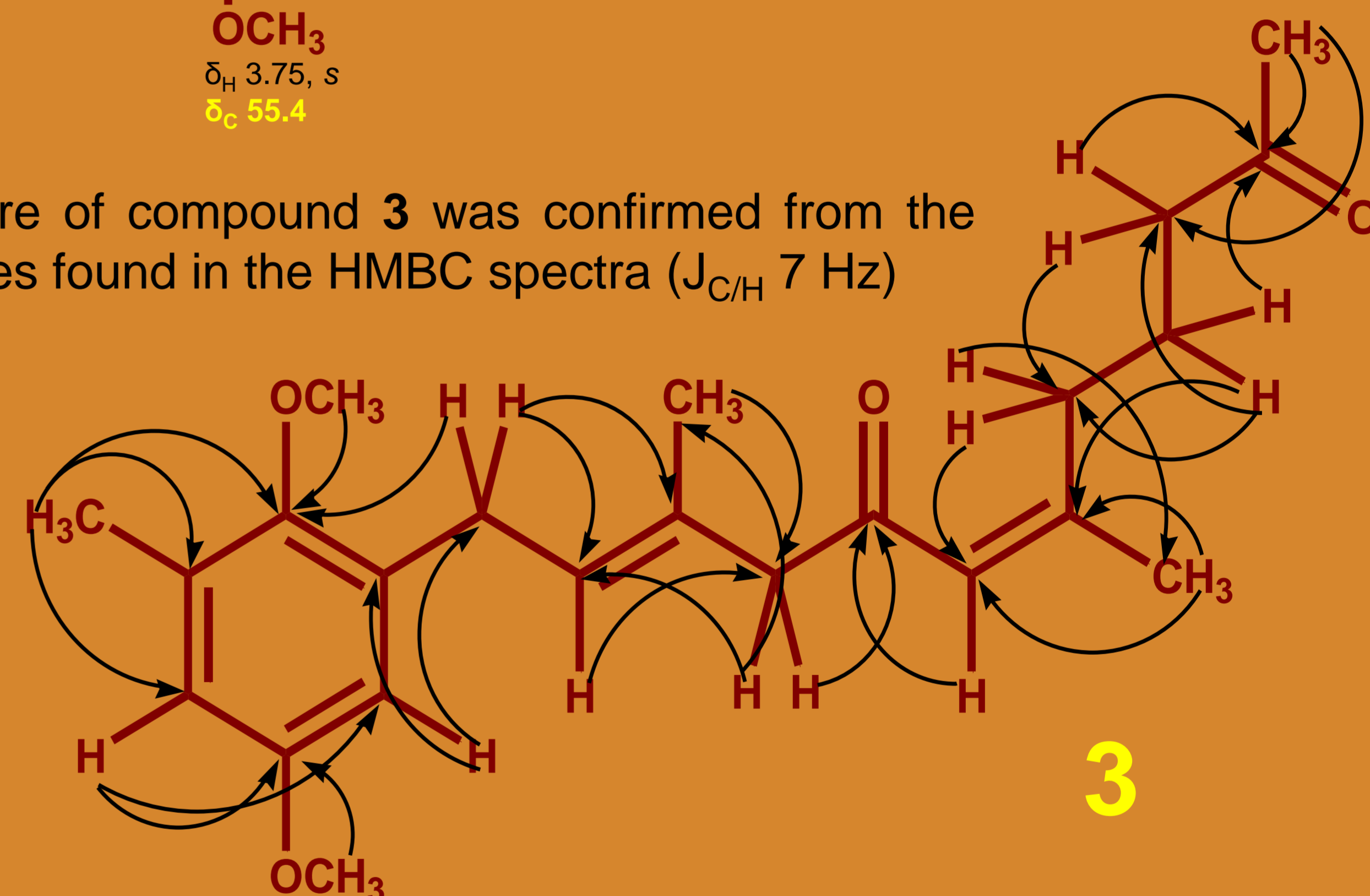
Benzoic acid (**1**) is isolated for the first time from *Cystoseira abies-marina*.



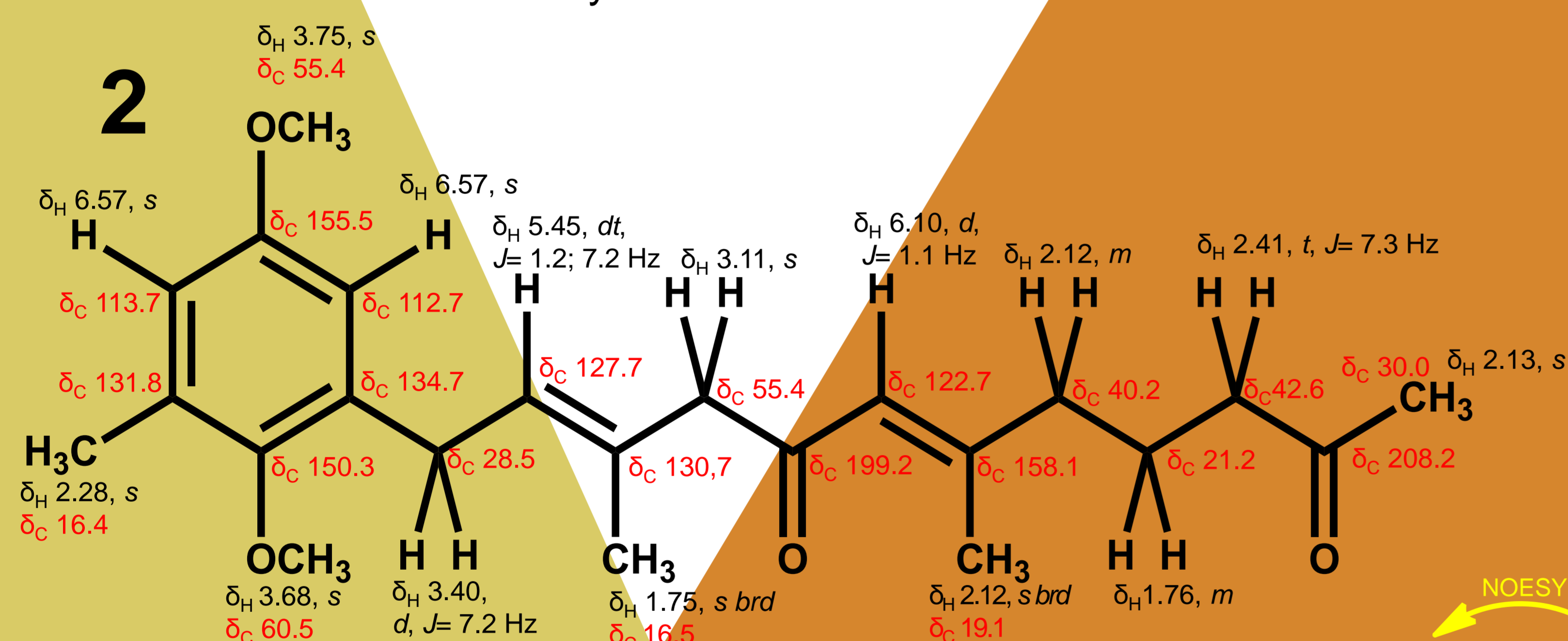
Compound **3** was identified as (6*Z*,10*E*)-12-(2,5-dimethoxy-3-methylphenyl)-6,10-dimethyldodeca-6,10-diene-2,8-dione, trivially known as Cystozores B).



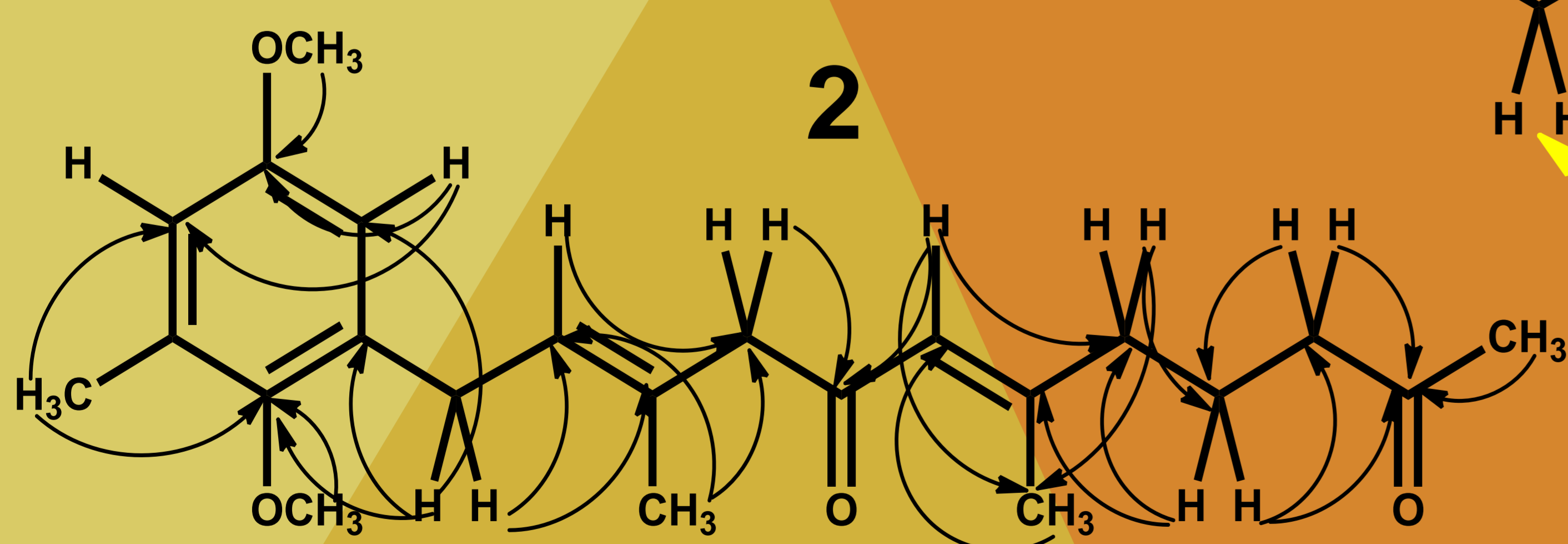
The structure of compound **3** was confirmed from the connectivities found in the HMBC spectra ( $J_{C/H}$  7 Hz)



Compound **2** was identified as (6*E*,10*E*)-12-(2,5-dimethoxy-3-methylphenyl)-6,10-dimethyldodeca-6,10-diene-2,8-dione, trivially known as Cystozores A.



The NOE effects (NOESY spectra) observed for **2** and **3** and the literature data<sup>[5]</sup> allowed to confirm the stereochemistry of the double bonds.



## Material and Methods

*Cystoseira abies-marina* was collected in Mosteiros, São Miguel in Winter 2010 and Spring 2011. After grinding, *Cystoseira abies-marina* was exhaustively extracted with methanol and dichloromethane. The MeOH and CH<sub>2</sub>Cl<sub>2</sub> extract was evaporated to dryness, and then fractionated by column and preparative TLC chromatography on silica gel, eluting with solvent mixtures of different polarity. The structures of the compounds were established by 1D (<sup>1</sup>H, <sup>13</sup>C, DEPT) and 2D (COSY, HSQC, HMBC, NOESY) NMR techniques as well as MS.

The EIMS data of **2** and **3** showed [M]<sup>+</sup> peaks at m/z ??? and ??? respectively, confirming the proposed structures.

## References

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## Acknowledgements

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