

## SUPPLEMENTARY MATERIAL

### A novel pentacyclic triterpene acid from the stem bark of *Combretum fragrans* F. Hoffm (Combretaceae)

Isaac Silvère Gade<sup>a\*</sup>, Jean Noël Nyemb<sup>b\*</sup>, Achi Mahamat<sup>a</sup>, Alex De Théodore Atchade<sup>a\*</sup>, Emmanuel Talla<sup>c</sup>, Sophie Laurent<sup>d</sup>, Céline Henoumont<sup>d</sup> and Alessandro Venditti<sup>e</sup>

<sup>a</sup> Department of Organic Chemistry, Faculty of Science, The University of Yaounde I, Yaounde 812, Cameroon;

<sup>b</sup> Department of Refining and Petrochemistry, National Advanced School of Mines and Petroleum Industries, The University of Maroua, P.O. Box 08 Kaele, Cameroon;

<sup>c</sup> Department of Chemistry, Faculty of Science, University of Ngaoundere, P.O. Box 404 Ngaoundéré

<sup>d</sup> Laboratory of NMR and Molecular Imaging, Department of General, Organic Chemistry and Biomedical, University of MONS, B-7000 Mons, Belgium.

<sup>e</sup> Dipartimento di Chimica, "Sapienza" Università di Roma, Piazzale Aldo Moro 5, Rome, Italy

#### \*Corresponding authors:

Jean Noël Nyemb, Department of Fundamentals Science, Faculty of Mines and Petroleum Industries, University of Maroua, P.O. Box 08 Kaélé, Cameroon;

E-mail: [nyembjeannoel@gmail.com](mailto:nyembjeannoel@gmail.com);

 <http://orcid.org/0000-0001-5069-6737>

Isaac Silvère Gade, Department of Organic Chemistry, Faculty of Science, The University of Yaounde I, P.O. Box 812 Yaounde, Cameroon;

E-mail: [isaac\\_gade@yahoo.fr](mailto:isaac_gade@yahoo.fr) (IS Gade)

Alex De Théodore Atchade, Department of Organic Chemistry, Faculty of Science, The University of Yaounde I, Yaounde 812, Cameroon;

E-mail address: [alexiod@ yahoo.fr](mailto:alexiod@ yahoo.fr) (AT Atchade)

#### Abstract

A phytochemical study was carried out on the stem bark of *Combretum fragrans* F. Hoffm., a medicinal plant belonging to the Combretaceae family and used traditionally in the treatment of various ailments. Column chromatography separation on silica gel of the crude methanol extract from the stem bark of *C. fragrans* led to the isolation of a new pentacyclic triterpene acid, trivially named as fragransin (**1**) along with four known compounds: betulin (**2**), betulinic acid (**3**), bellericagenin B (**4**) and a mixture of  $\beta$ -sitosterol (**5**) and stigmasterol (**6**). Compounds structures were elucidated by extensive spectroscopic analyses including 1D and 2D NMR, mass spectrometry as well as by comparison with the literature data. All compounds were isolated for the first time from *C. fragrans* and their implications in chemosystematic and traditional medicine was also briefly discussed.

**Keywords:** Combretaceae, *Combretum frgarans*, fragransin, pentacyclic triterpenes

QTOF1\_PHIL\_210518\_HRMS\_CFE\_85\_01 67 (1.235) AM (Cen, 6, 80.00, Ht, 9082.0, 525.29, 0.70); Sm (SG, 10x4.00); Cm (40:68)  
TOF MS LD+

5.19e+002

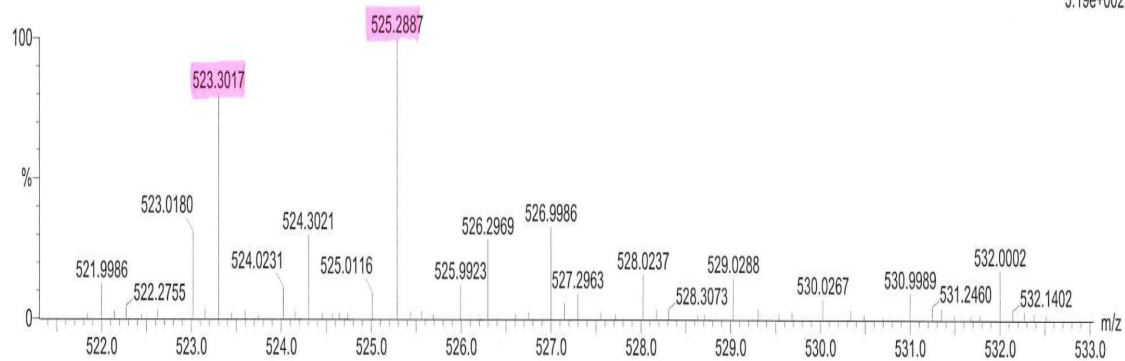


Figure S1a: HRESI-MS(+) Mass Spectrum of compound 1

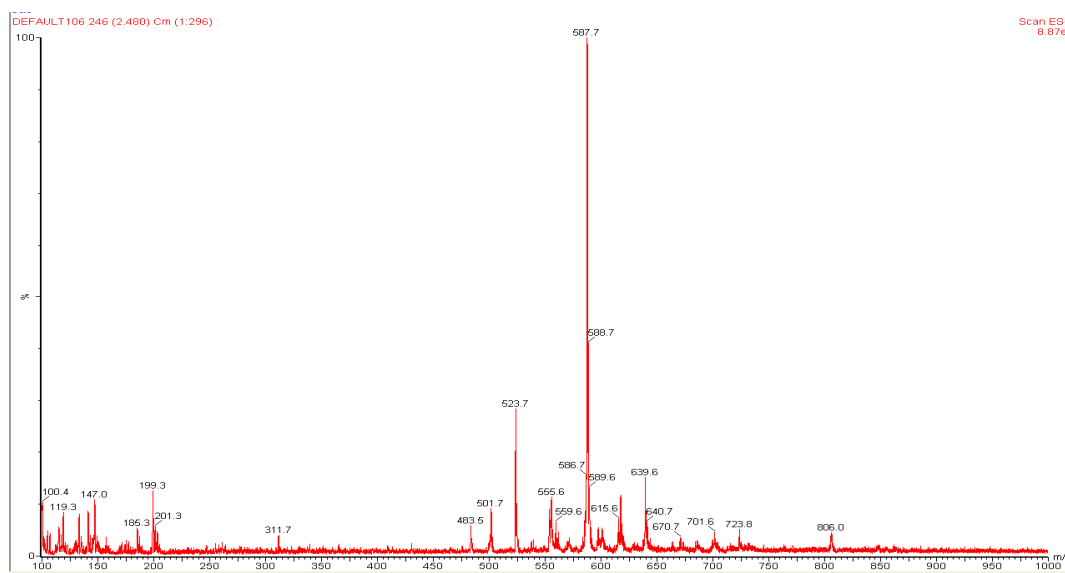


Figure S1b: ESI-MS(+) Mass Spectrum of compound 1

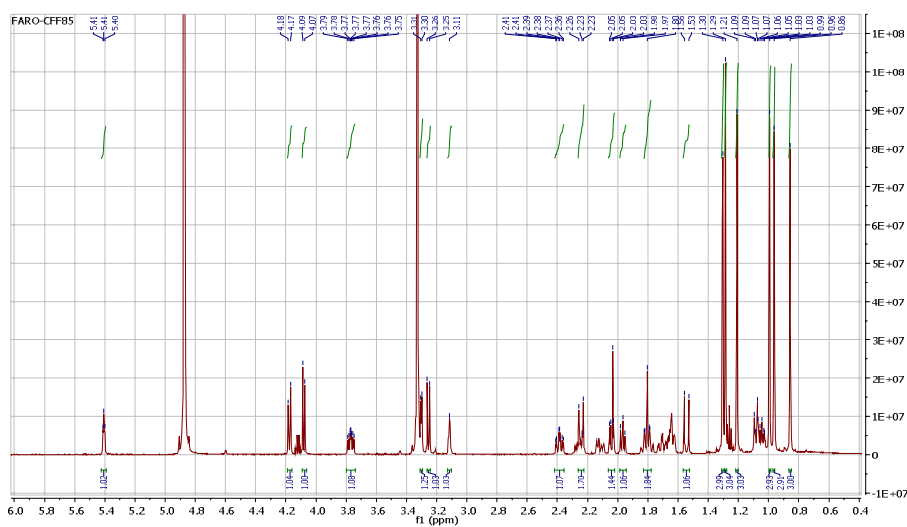
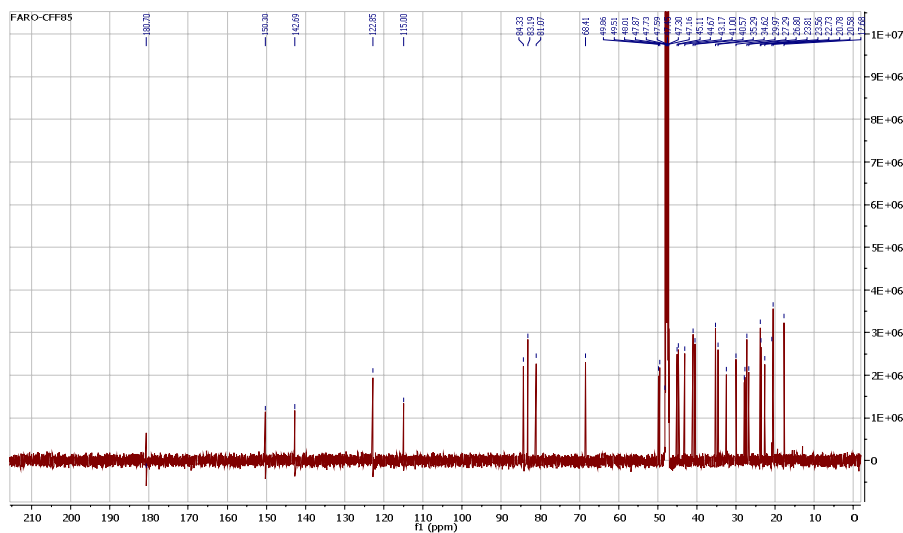
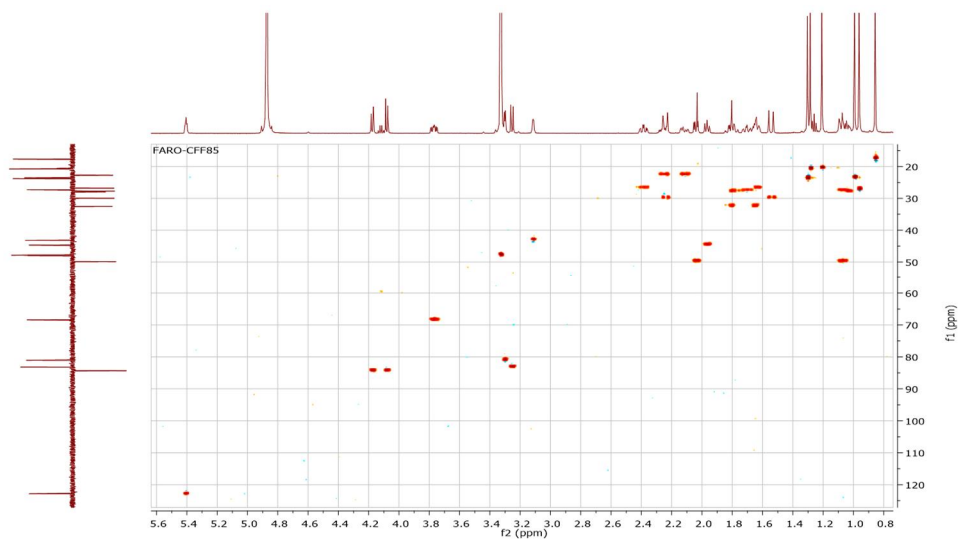


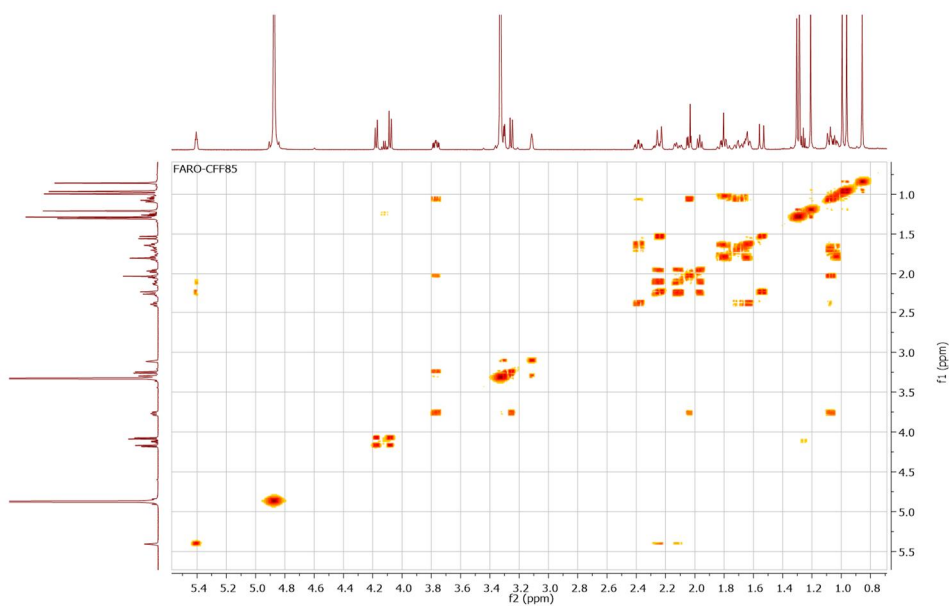
Figure S2: <sup>1</sup>H NMR (600 MHz, MeOD) spectrum of compound 1



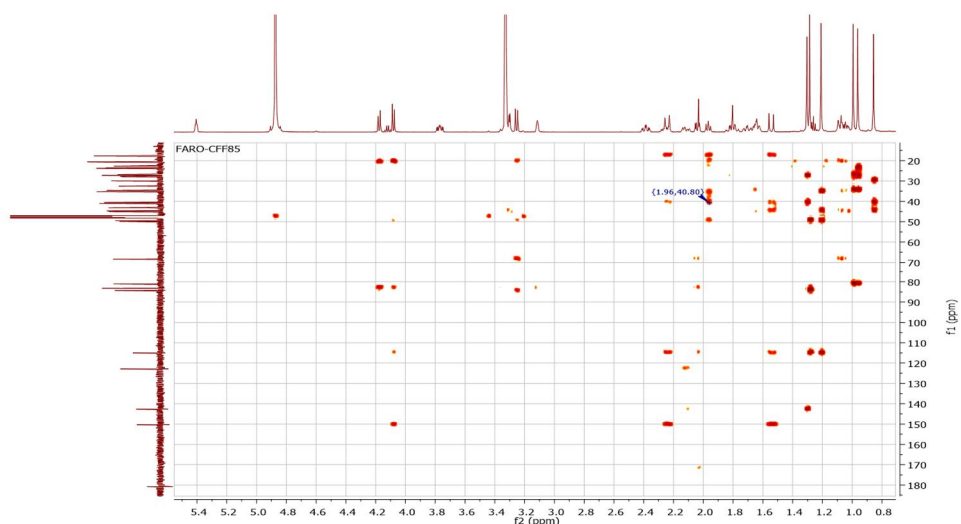
**Figure S3:**  $^{13}\text{C}$  NMR (151 MHz, MeOD) Spectrum of compound **1**



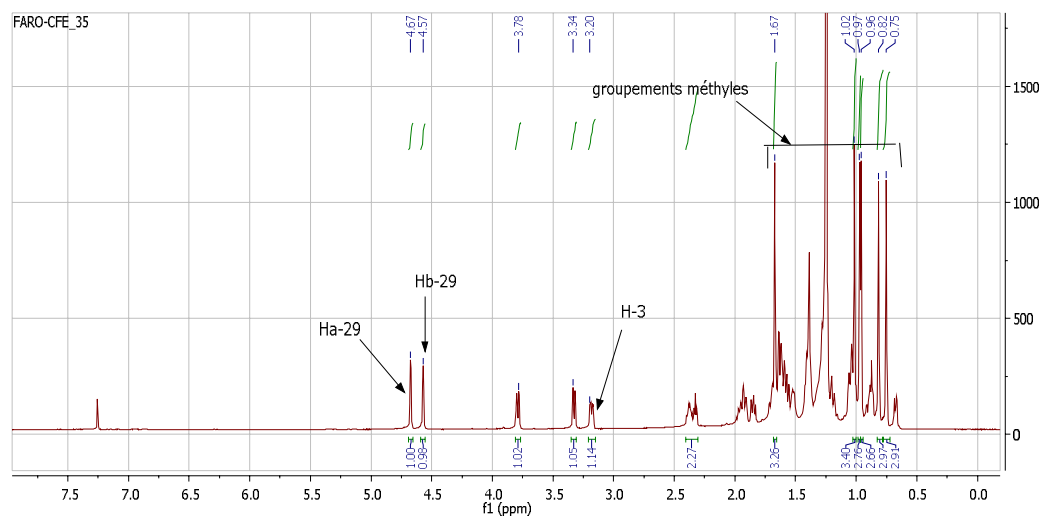
**Figure S4:** HSQC spectrum of compound **1**



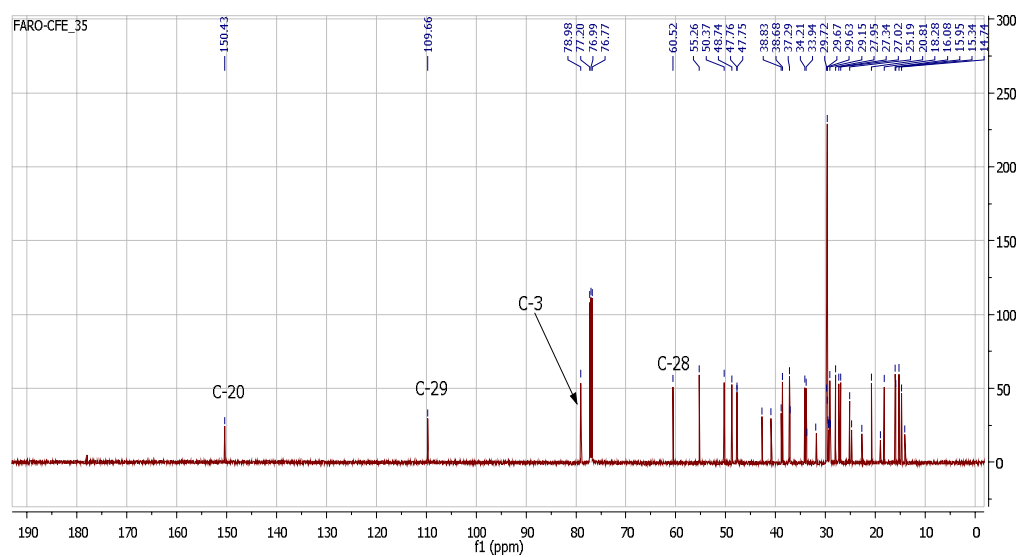
**Figure S5:**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of compound **1**



**Figure S6:** HMBC spectrum of compound **1**



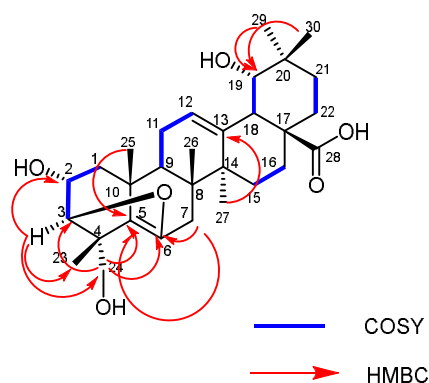
**Figure S7:** <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) spectrum of compound **2**



**Figure S8:** <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>) spectrum of compound **2**.







**Figure S15:** COSY and HMBC correlation of **1**

**Table 1S:**  $^1\text{H}$  (600 MHz,  $\text{CD}_3\text{OD}$ ) and  $^{13}\text{C}$  (150 MHz,  $\text{CD}_3\text{OD}$ ) NMR data and HMBC correlation of compound **1**.

Compound <b>1</b>			
N <sup>o</sup>	$\delta_{\text{H}}$ (Mult., $J$ (Hz))	$\delta_{\text{C}}$	HMBC
1	2.02 $\alpha$ ; 1.06 $\beta$	49.8	
2	3.77 (1H, <i>dddd</i> , $J = 11.5, 9.4$ and 4.2)	68.4	
3	3.25 (1H, <i>d</i> , $J = 9.4$ )	83.2	C2, C24, C23
4	-	49.5	
5	-	115.0	
6	-	150.3	
7	2.23 $\alpha$ (1H, <i>d</i> , $J = 17.6$ ); 1.54 $\beta$ (1H, <i>d</i> , $J = 17.6$ )	29.9	C5, C6, C8, C26
8	-	40.5	
9	1.96 (1H, <i>t</i> , $J = 9.1$ )	44.6	
10	-	46.3	
11	2.23 $\alpha$ ; 2.09 $\beta$	22.7	
12	5.41 ( <i>t</i> , 1H)	122.8	
13	-	142.6	
14	-	41.0	
15	2.37 $\alpha$ ; 1.63 $\beta$	26.8	
16	1.80 $\alpha$ ; 1.03 $\beta$	27.7	
17	-	45.1	
18	3.11 (1H, <i>brs</i> )	43.1	
19	3.30 (1H, <i>d</i> , $j = 3.9$ )	81.0	
20	-	34.6	
21	1.78 $\alpha$ ; 1.64 $\beta$	32.5	
22	-	27.9	
23	1.29 (3H, <i>s</i> )	20.7	C5, C3, C4
24	4.08 $\alpha$ (1H, <i>d</i> , $J = 8.8$ ); 4.18 $\beta$ (1H, <i>d</i> , $J = 8.8$ )	84.3	
25	1.21 (3H, <i>s</i> )	20.5	C5, C4, C1, C9
26	0.86 (3H, <i>s</i> )	17.7	C9, C14, C7
27	1.30 (3H, <i>s</i> )	23.8	C13, C8, C15
28	-	180.0	
29	0.96 (3H, <i>s</i> )	27.3	C19, C20
30	0.99 (3H, <i>s</i> )	23.5	C19, C20