

Full Length Research Paper

Isolation and characterization of 2, 3, 8-tri-me ether ellagic acid from the stem bark of *Irvingia gabonensis* (Baill)

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The stem bark of *Irvingia gabonensis* used variously in ethno medicinal treatments in some parts of Nigeria was studied. A silica gel column chromatography of the methanol extract (using only chloroform) eluted a pale yellow crystalline substance identified by means of spectral analyses as 2,3,8-Tri-Me ether ellagic acid.

Key words: *Irvingia gabonensis*, 2, 3, 8-Tri-Me ether ellagic acid, chromatography, spectral analyses.

INTRODUCTION

The genus *Irvingia gabonensis* (Irvingiaceae) is widely distributed in tropical West Africa and represented by two varieties: (i) Fruits with sweet edible scanty fibrous pulp, hole fluted or cylindrical and (ii) Fruits with bitter inedible very fibrous pulp, hole buttressed, Watt *et al.* (1962). The stem bark is claimed to be very useful in ethno medicinal treatments in some parts of West Africa. Irvine (1935) reported that the shavings of the stem bark are eaten to stop diarrhea or dysentery in French Equatorial Africa while Dalziel (1948) reported that aqueous extract of the bark is rubbed for pain relief by the *Mendes* of Sierra Leone. The powdered kernels is applied to burns and also used as an astringent (Irvine, 1935). Nyahandiyi (1999) reported that the methanol extract from the stem bark has excitatory effect on the phrenic nerve of rats. Raji *et al.* (2001) reported that the methanol extract of its stem bark has antidiahegenic and antiulcer properties on rats. Folklore has it that the aqueous/alcoholic extract of the stem bark finds use in the treatment of dysentery, typhoid fever and fungal (skin) infections. There is no literature available on the chemical compounds isolated from the stem bark of this plant.

MATERIALS AND METHOD

Collection, identification and preparation of plant material

The plant material was collected from Ihiala, Anambra State, Nigeria [6°N and 7°E] in December and identified by Mr. Abdullahi

Musa of the Herbarium, Department of Biological Sciences, Faculty of Science, Ahmadu Bello University, Zaria, Nigeria. A voucher specimen, number 103947, was deposited there. The collected sample was air-dried, pulverized using a mill hammer and stored in polythene bags for use.

Extraction of plant material

Air-dried and pulverized plant material (325.60 g) was defatted with redistilled petroleum spirit (60 – 80°C) by the use of soxhlet extractor to afford 6.00 g (1.85%) of fatty acids and their derivatives. The defatted pulverized plant material was then successively and exhaustively extracted with redistilled chloroform and methanol respectively. The various extracts were concentrated *in vacuo* at 40°C using a *rota vapor*. This gave 0.80g (0.24%) of dry crude chloroform extract and 22.28 g (6.00%) of dry crude methanol extract. The various extracts were stored in a refrigerator until needed for further studies.

Separation and purification of phytochemical constituents from the crude extracts

The methanol fraction was column chromatographed on a silica gel column. The solvent used in packing the silica gel in the column was pure chloroform. Elution of the crude extract in the column with pure and redistilled chloroform gave pale yellow crystals. The crystals were further purified using Merck's precoated preparative thin layer chromatography [PTLC] made of silica gel. The crystals were dried in the dessicator after which the melting point was determined and found to be 296-297°C. The crystals were subjected to and characterized purely by spectral techniques as 2,3,8-Tri-Me ether ellagic acid (Figure 4).

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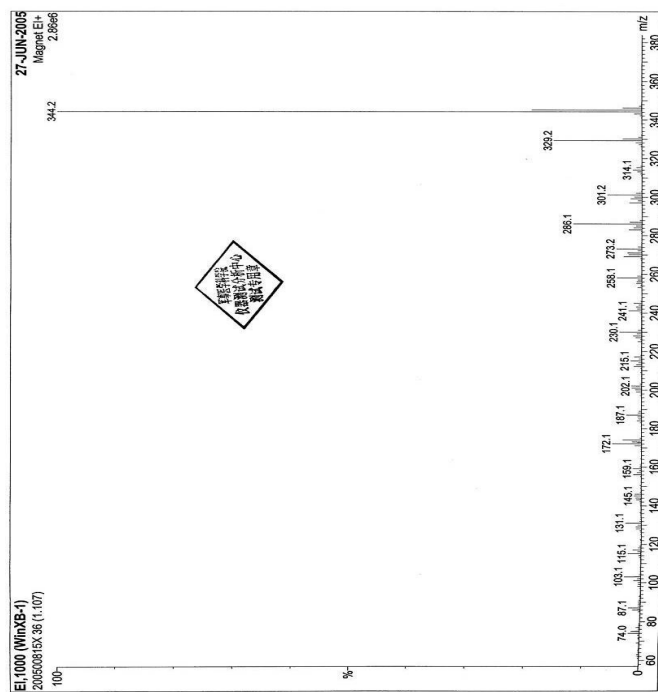


Figure 1. The EI-MS of the 2, 3, 8-Tri-Me ether ellagic acid.

Table 1. ¹³C and ¹H NMR spectral data of the 2, 3, 8-Tri-Me ether ellagic acid

	¹³ C-NMR	¹ H-NMR
1	111.21	-
2	140.96	-
3	140.20	-
4	152.63	-
5	111.66	7.55s
6	112.53	-
7	158.33	-
1'	111.96	-
2'	141.49	-
3'	140.84	-
4'	153.81	-
5'	107.47	7.65s
6'	113.38	-
7'	158.52	-
OMe(4')	56.73	4.01s
OMe(3')	61.31	4.04s
OMe(3)	61.02	4.06s
OH(4)	-	-

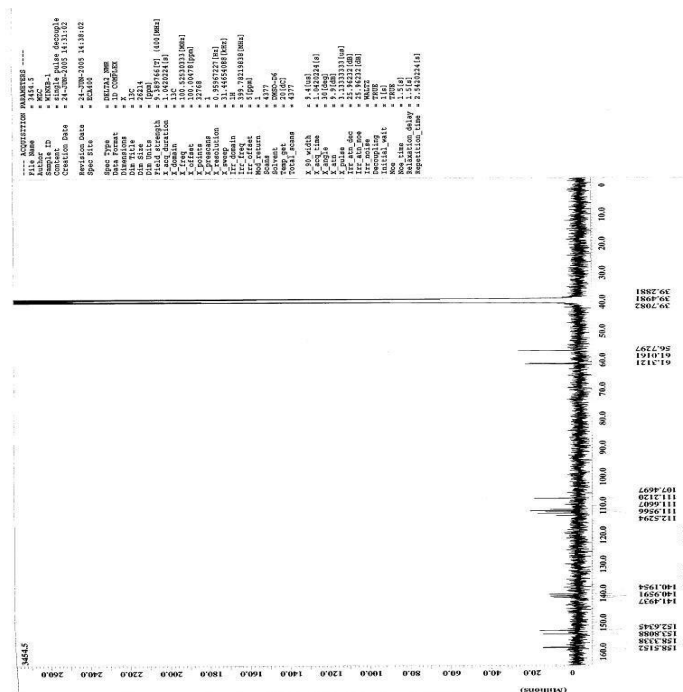


Figure 2. ¹³C-NMR spectrum of Sample

RESULTS AND DISCUSSION

Pale yellow crystals were the major product of the column separation study. The crystals were subjected to Electron

Impact-Mass Spectrograph-, ¹³C-NMR- and ¹H-NMR studies and the results obtained are as shown below:

ELECTRON IMPACT-MASS SPECTROGRAM [EI-MS]:

The Electron Impact-Mass Spectrograph [EI-MS] of the sample, Figure1 has a molecular ion 334.2[M⁺] which is also the base peak. Other prominent peaks obtained are as shown below:

EI-MS, m/z: 74.0, 87.1, 103.1, 115.1, 131.1, 145.1, 159.1, 172.1, 187.1, 202.1, 215.1, 230.1, 241.1, 258.1, 273.2, 286.1, 301.2 314.1, 329.2.

¹³C-NMR (400MHz, DMSO-D₆): The ¹³C-NMR is shown in Figure 2 and the peaks obtained are as recorded below:

δ: 111.21 (C-1), 140.96 (C-2), 140.20 (C-3), 152.63 (C-4), 111.66 (C-5), 112.53 (C-6), 158.33 (C-7), 111.96 (C-1'), 141.49 (C-2'), 140.80 (C-3'), 153.81 (C-4'), 107.47 (C-5'), 113.38 (C-6'), 158.52 (C-7'), 56.73 (OMe-4'), 61.31 (OMe-3') and 61.02 (OMe-3).

¹H-NMR (400 MHz, DMSO-D₆)

The proton NMR is shown in Figure 3 and the peaks are as recorded below:

δ: 7.55 (1H singlet), 7.65 (1H singlet), 4.01 (1H singlet), 4.04 (1H singlet), 4.06 (1H singlet).

Based on the spectral data and coupled with the comparative spectral analyses of the works of Nawwar et

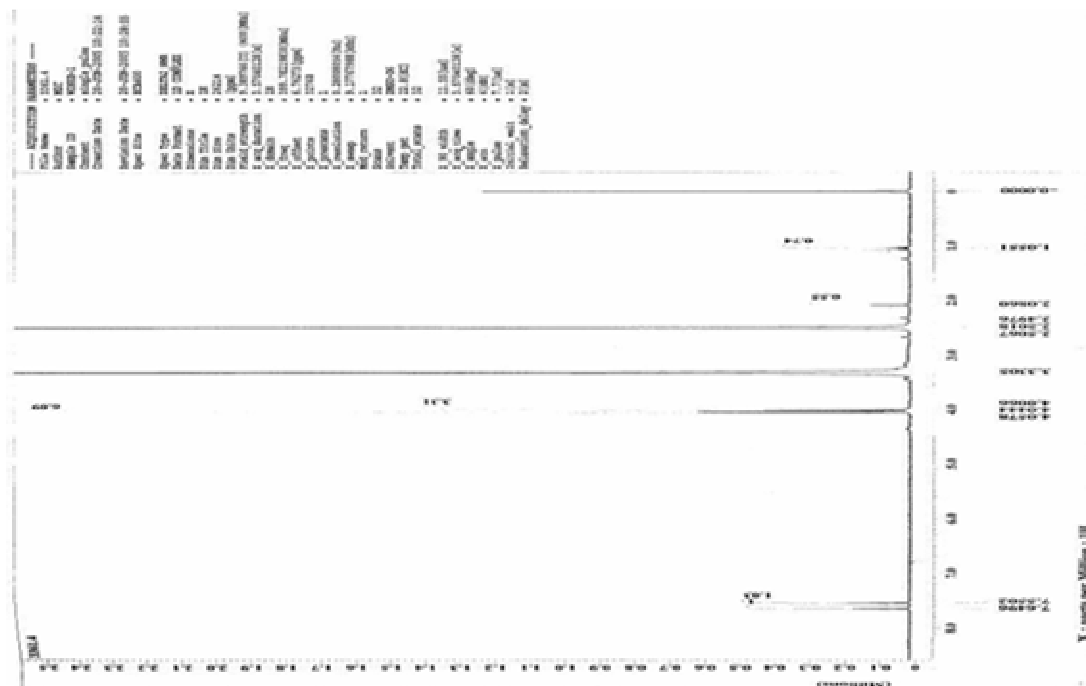


Figure 3. ¹H-NMR spectrum of Sample 1.

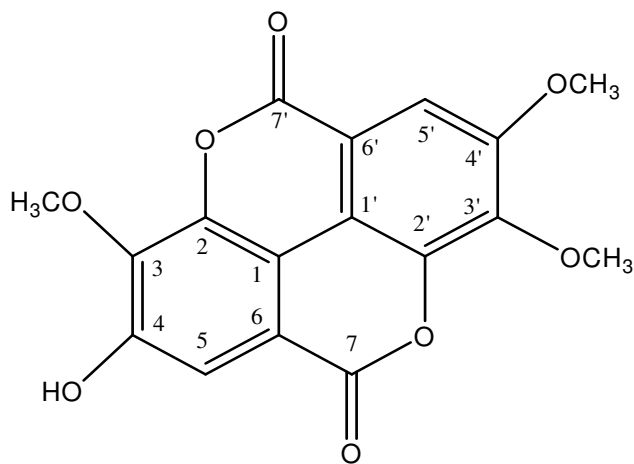


Figure 4. 2, 3, 8-Tri-Me ether ellagic acid Molecular formula. C₁₇H₁₂O₈. Percentage composition: C 59.3%, H 3.51%, O 37.18%. Physical description: Pale yellow needles (DMSO). Melting point: 296 – 297°C.

al. (1994), Tchinda et al. (2003) and Yazaki and Hillis (1976), this component was found to be 2,3,8-Tri-Me ether ellagic acid (Figure 4, Table 1).

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