X-ray diffraction powder data of ambroperuvin, a new pseudoguaianolide from *Ambrosia peruviana*

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Ambroperuvin is a new pseudoguaianolide isolated from *Ambrosia peruviana* Willd., a plant used in traditional medicine in Venezuela. The analysis of the X-ray powder pattern being reported led to an orthorhombic unit cell with space group $P2_12_12_1$ and cell parameters $a = 15.999(6)$ Å, $b = 11.263(2)$ Å, $c = 9.112(2)$ Å. No detectable impurities were observed. © *2017 International Centre for Diffraction Data*. [doi:10.1017/S0885715617000604]

Key words: *Ambrosia peruviana*, ambroperuvin, pseudoguaianolide, X-ray powder diffraction data

I. INTRODUCTION

Secondary metabolites isolated from natural sources, such as plants, lichens, fungi, and marine organisms, constitute an important source of potential pharmaceutical compounds. These materials are usually characterized by spectroscopic and chromatographic techniques. Since polymorphism is an important solid-state phenomenon among pharmaceuticals, X-ray powder diffraction techniques are particularly suitable for characterizing the nature of the crystalline phase, which would be of potential pharmaceutical interest. Perhaps because of the low yields obtained in the process of isolating the secondary metabolites, X-ray powder diffraction techniques are seldom used to characterize these materials. However, with the modern instrumentation having high-intensity sources and more sensitive detectors, a more extensive use of powder diffraction might be possible.

In this contribution, the X-ray powder diffraction data of ambroperuvin (Figure 1), 4α-hydroxy-6β-acetoxy-2βH(3βH)-epoxy-5βMe,7αH,8βH,10αH-pseudoguai-11(13)-en-12,8-olide ($C_{17}H_{22}O_6$) is presented. This compound is a new pseudoguaianolide isolated from the aerial parts of *Ambrosia peruviana* Willd. (Asteraceae), a plant common in Lagunillas, Mérida state, Venezuela. Allelopathic activity has been reported for *A. peruviana* (Anaya and del Amo, 1978). It is widely used for the treatment of skin spots, varicose veins, scars, and menstrual problems, among other conditions, in Venezuela and in other South American countries (Roth and Lindorf, 2002).

The determination of the crystal structure of ambroperuvin was carried out from single-crystal data and is being reported elsewhere. In this report, the X-ray powder diffraction data of this material are presented.

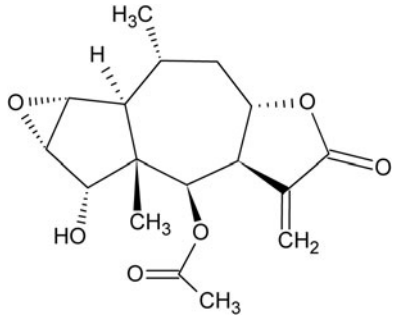

Figure 1. Molecular diagram for ambroperuvin ($C_{17}H_{22}O_6$).

II. EXPERIMENTAL

A. Sample preparation

Plant materials (leaves, stems, and inflorescence) of *A. peruviana* Willd. were collected in San Juan de Lagunillas, Mérida, Venezuela. The plant material was air-dried, ground, and subjected to an exhaustive extraction procedure with acetone. Subsequent extractions with hexane-EtOAc, EtOAc-MeOH, and hexane:dichloromethane mixtures produced a crude product, which was purified via preparative thin layer chromatography. Preliminary characterization of the material was carried out by Fourier transform infrared (FT-IR) on a KBr pellet using a Perkin-Elmer 1600 spectrometer at room temperature. The melting point of this compound was determined with an electro-thermal apparatus.

B. Powder diffraction data collection

A small portion of the title compound was gently ground in an agate mortar and dusted on top of a zero-background specimen holder. The powder diffraction data were collected using the θ-θ geometry at room temperature on a Siemens D5005 diffractometer equipped with a sample spinner, a graphite diffracted beam monochromator, a scintillation

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TABLE I. X-ray powder diffraction data of ambroperuvin ($C_{17}H_{22}O_6$).

No.	d_{calc}	d_{obs}	I/I_o	h	k	l	$2\theta_{obs}$	$2\theta_{calc}$	$\Delta 2\theta$
1	9.2100	9.2209	1000	1	1	0	9.584	9.595	0.011
2	7.9177	7.8812	46	1	0	1	11.218	11.166	−0.052
3	7.0839	7.0790	185	0	1	1	12.494	12.485	−0.009
4	6.4774	6.4740	497	1	1	1	13.667	13.660	−0.007
5	5.6316	5.6292	58	0	2	0	15.730	15.723	−0.007
6	5.3122	5.3094	42	1	2	0	16.684	16.675	−0.009
7	5.3034	5.2874	259	2	1	1	16.754	16.703	−0.051
8	4.8201	4.8229	192	3	1	0	18.381	18.392	0.011
9	4.6050	4.6066	190	2	2	0	19.252	19.259	0.007
10	4.2607	4.2578	73	3	1	1	20.846	20.832	−0.014
11	4.1099	4.1128	71	2	2	1	21.590	21.605	0.015
12	4.0836	4.0860	94	1	1	2	21.733	21.746	0.013
13	3.9999	4.0002	101	4	0	0	22.205	22.207	0.002
14	3.9589	3.9566	49	2	0	2	22.453	22.440	−0.013
15	3.7349	3.7314	48	2	1	2	23.827	23.805	−0.022
16	3.6551	3.6526	50	1	3	0	24.349	24.332	−0.017
17	3.5420	3.5395	96	0	2	2	25.140	25.122	−0.018
18	3.4713	3.4729	91	0	3	1	25.630	25.642	0.012
19	3.4582	3.4588	64	1	2	2	25.736	25.740	0.004
20	3.3924	3.3944	45	1	3	1	26.233	26.249	0.016
21	3.3110	3.3109	37	3	1	2	26.907	26.906	−0.001
22	3.2387	3.2404	38	2	2	2	27.504	27.518	0.014
23	3.0781	3.0762	43	5	1	0	29.003	28.985	−0.018
24	2.9839	2.9850	38	1	0	3	29.910	29.920	0.010
25	2.9093	2.9083	50	3	3	1	30.718	30.707	−0.011
26	2.7732	2.7747	40	1	4	0	32.236	32.254	0.018
27	2.7374	2.7410	35	4	3	0	32.643	32.687	0.044
28	2.6530	2.6534	71	1	4	1	33.752	33.757	0.005
29	2.5505	2.5514	42	5	1	2	35.145	35.158	0.013
30	2.5459	2.5439	41	3	3	2	35.252	35.223	−0.029
31	2.4900	2.4889	30	3	4	0	36.058	36.040	−0.018
32	2.3898	2.3909	40	3	2	3	37.590	37.607	0.017
33	2.3688	2.3686	33	1	4	2	37.957	37.953	−0.004
34	2.2779	2.2777	34	0	0	4	39.534	39.529	−0.005
35	2.2327	2.2332	40	0	1	4	40.355	40.364	0.009
36	2.0751	2.0743	31	3	5	0	43.598	43.580	−0.018
37	2.0550	2.0549	41	4	4	2	44.031	44.030	−0.001
38	2.0034	2.0018	31	1	5	2	45.264	45.225	−0.039
39	1.9794	1.9797	31	4	0	4	45.797	45.803	0.006
40	1.8939	1.8948	32	6	4	1	47.974	47.999	0.025
41	1.8348	1.8351	34	4	4	3	49.640	49.646	0.006
42	1.8293	1.8290	32	3	3	4	49.816	49.806	−0.010

detector, and a long fine-focus Cu tube running at 40 kV and 30 mA. The data were collected over the angular range from 5° to 50° (2θ) with a step size of 0.02° (2θ) and a counting time of 80 s step^{-1}.

III. RESULTS AND DISCUSSION

The FT-IR spectrum obtained was consistent with the functional groups present in the molecule. The melting point determined was 195–197 °C.

The indexing of the pattern recorded was successfully carried out with DICVOL14 (Louër and Boultif, 2014). The resulting orthorhombic unit cell was very similar to the cell obtained from the single-crystal study. The analysis of the entire pattern carried out with the program AIDS*NBS (Mighell *et al*., 1981) led to a unit cell with parameters: a = 15.999(6) Å, b = 11.263(2) Å, c = 9.112(2) Å; V = 1642.0(5) Å^3. The de Wolff (1968) and Smith and Snyder (1979) figures

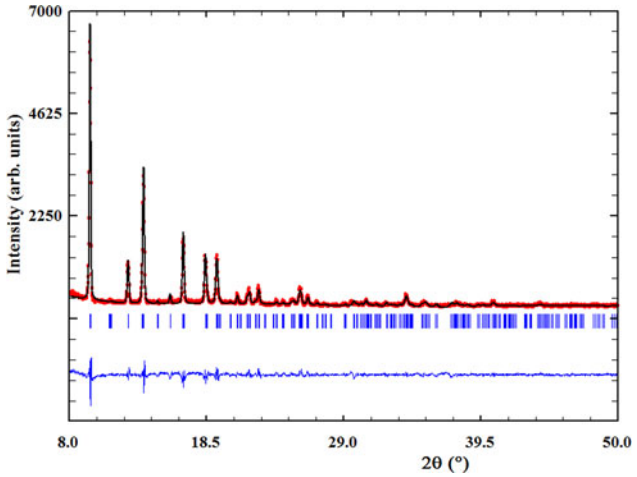

Figure 2. (Color online) The Le Bail profile fitting of the recorded X-ray powder diffraction pattern of ambroperuvin ($C_{17}H_{22}O_6$).

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of merit obtained were $M_{20} = 16.6$; $F_{30} = 24.3$ (0.0158, 78). The powder diffraction data are presented in Table I. Space group analysis using the program DAjust (Vallcorba *et al*., 2012) suggested space group $P2_12_12_1$ as the most likely candidate. This result agrees with the single-crystal structure determination performed on this compound. The fitting of the whole pattern with the Le Bail algorithm implemented in FULLPROF (Rodriguez-Carvajal, 2001), accounts for all the diffraction maxima recorded (see Figure 2).

SUPPLEMENTARY MATERIAL

The supplementary material for this article can be found at https://doi.org/10.1017/S0885715617000604.

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