Communications to the Editor

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STRUCTURE OF ED-1 ISOLATED FROM EMERICELLA DENTATA

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The structure of a new metabolite, ED-1 (1) isolated from *Emericella dentata* together with ED-2, was determined by X-ray analysis.

KEYWORDS — ED-1; fungal metabolite; *Emericella dentata*; structure determination; X-ray analysis

The isolation of two new metabolites, ED-1 (1) and ED-2 (2) from *Emericella dentata* and the structure determination of ED-2 were previously reported.[1] This paper describes the structure determination of 1.

ED-1: colorless needles, mp>300°C, $C_{25}H_{28}O_8$(M^+ m/z 456), $[\alpha]_D^{26}$-202° (c=1.00, pyridine), UV λ_{max}^{MeOH} nm(ε): 233(4200); IR ν_{max}^{KBr} cm^{-1}: 3450, 3350, 1768, 1755, 1690, 1658, 1623; ^{1}H-NMR(in pyridine-d_5) δ: 1.47 (3H, s), 1.53 (3H, s), 1.59 (3H, s), 1.72 (3H, d, J=7), 1.82 (3H, s), 4.66 (1H, d, J=8), 5.57 (1H, q, J=7), 5.61 (1H, s), 5.92 (2H, s), 6.04 (1H, d, J=10), 6.40 (1H, s), 7.08 (1H, d, J=10), 7.88 (1H, d, J=8 disappeared with D_2O).

Comparison of the ^{1}H and ^{13}C-NMR spectra of 1 and 2 showed a close similarity between these metabolites. The molecular formula of 1 indicates that this compound contains two fewer hydrogens than 2. ^{1}H and ^{13}C-NMR spectra of 1 suggested the presence of five methyl (one less than 2) groups. The presence of three carbonyl groups in 1 was suggested from the ^{13}C-NMR and IR spectral data. Catalytic hydrogenation of 1 afforded a dihydro derivative, colorless platelets, mp 278-282°C. Acetylation of 1 gave monoacetate, colorless needles, mp 290-292°C.

Recrystallization of 1 from methanol gave orthorhombic crystals, space group $P2_12_12_1$, with four molecules in a unit cell of dimensions: a=23.997 (5), b=11.581 (6), c=7.974 (4) Å. The intensity data were collected on a Rigaku computer-controled four circle diffractometer with Ni-filtered CuKα radiation and θ-2θ scan technique. The intensities of 1736 independent reflections were measured. The structure was solved by MULTAN[2] followed by Fourier synthesis. The positional and anisotropic temperature factors for non-hydrogen atoms were refined by the block-diagonal least-squares method.[3] The final R value was 0.062. The structure of 1 is shown in Fig.1 with a computer generated perspective drawing.

We determined the structure of 2,[1] including the absolute configuration, and found that 2 corresponds to the desacetyl derivative of austin.[4] Thus, the structure of ED-1, co-metabolite of ED-2, represents as 1, including the absolute configuration.

ACKNOWLEDGMENT The authors are indebted to Miss K. Takizawa of Laboratory for Instrumental Analysis of this Institute for measuring the ^{1}H and ^{13}C-NMR spectra.

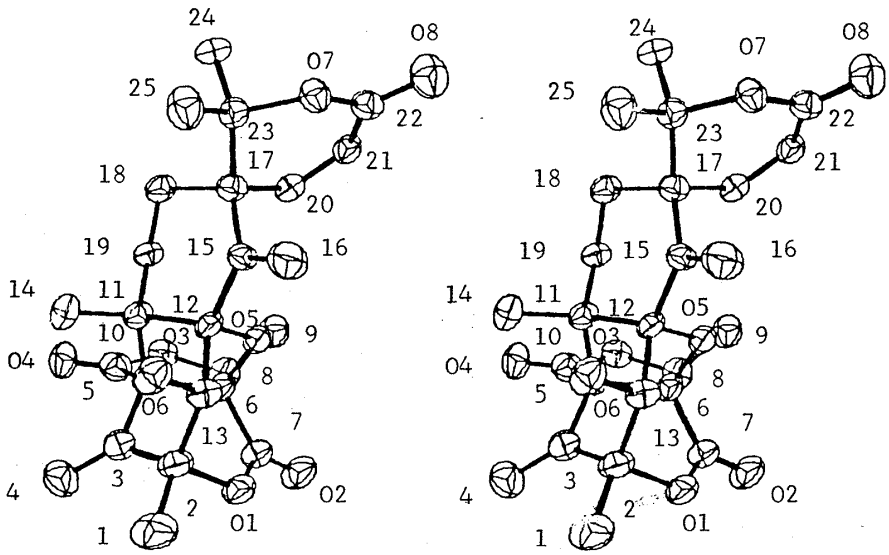
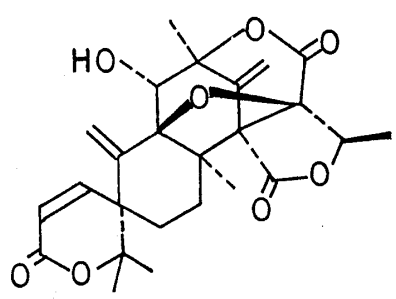
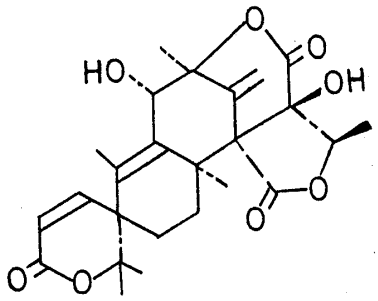


Fig.1. Stereoscopic View of ED-1 (1)
(The hydrogen atoms are omitted from the figure for the sake of clarity.)



ED-1 (1)



ED-2 (2)

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ADDED IN PROOF (April 26,1982) After we have finished the structure determination of ED-1, we received a private communication from Dr. T.J. Simpson of University of Edinburgh[5] in which he described the isolation of austinol and dehydroaustin from *Aspergillus ustus* and *Aspergillus variecolor*. His result indicates that 1 and 2 should be designated as dehydroaustinol and austinol, respectively.

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