structure was solved by MULTAN [3], with the 254 E's > 1.42. After refinement with the 1555 $2\sigma(I)$ observed intensities and location of the hydrogen atoms on a difference map, a weighting scheme was selected to have no dependence of $\langle w\Delta^2F\rangle$ vs. $\langle F_0\rangle$ and vs. $\langle \sin\theta/\lambda\rangle$. A weighted full matrix L.S. anisotropic refinement (fixed isotropic for H atoms) using the 1555 observed Friedel pairs converged to $R = 4.5\%$ and $R_w = 5.7\%$ [4].

The neo-clerodane absolute configuration of salvifarin (Fig. 1 and **2**) was determined comparing the 84 Bijvoet pairs with $\Delta F_c > 0.08$ and with less experimental error, that is $F_0 > 10\sigma(F_0)$, $4 < F_0 < 25$ and $0.22 < \sin\theta/\lambda < 0.58$. The averaged Bijvoet difference was 0.359 for the right enantiomer vs. 0.434 for the wrong one*.

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*A list of atomic parameters, bond distances and angles, torsion angles and $F_0 - F_c$ tables are deposited at the Cambridge Crystallographic Data Centre.

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A COUMARIN ACETYLGLUCOSIDE FROM *VIBURNUM SUSPENSUM*

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Key Word Index—*Viburnum suspensum*; Caprifoliaceae; coumarin; 2′,6′-*O*-diacetylscopolin.

Abstract—A new acetylated coumarin glucoside has been isolated from the leaves of *Viburnum suspensum* and determined as 2′,6′-*O*-diacetylscopolin on the basis of spectral and chemical evidence.

INTRODUCTION

Recently, we have isolated two known flavonol glycosides [1] and several bitter iridoids [2] from the leaves of *Viburnum suspensum* L. Further chromatographic examination of the leaves of the plant gave a new acetylcoumarin glucoside (**1**).

RESULTS

Compound **1** was crystallized as needles, mp 178–179.5°, $[\alpha]_D^{25}$ $-100°$ from methanol. The molecular formula $C_{20}H_{22}O_{11}\cdot 1/2\ H_2O$ was determined on the basis of the elementary analysis and mass spectrum. Absorption bands at 1740, 1620 and 1570 cm^{-1} in the IR spectrum and absorption maxima at 227 nm (ε10 800), 286 nm (ε5600) and 328 nm (ε7600) in the UV spectrum suggested that **1** is a coumarin. Additional evidence for the presence of this carbon skeleton came from the ^{1}H NMR spectrum. The signals corresponding to C-3 and C-4 protons appeared as an AB system at δ6.30 and 7.63 (J = 9.8 Hz). Two singlets at δ7.00 and 7.38 (1H each) were attributable to C-8 and C-5 protons, respectively. The ^{1}H NMR spectrum also showed the presence of two acetyl groups at δ2.07 (3H × 2, *s*) and one methoxyl group at δ3.70 (3H, *s*).

On acetylation with acetic anhydride–pyridine, **1** gave a tetra-acetate (**2**), mp 168–169°, $C_{24}H_{26}O_{13}$. The physical and spectral data of **2** [ν_{max}^{nujol} cm^{-1}: 1770–1730, 1620, 1570, 1505, 920, 890 and 825; δ2.10 (3H × 3, *s*), 2.17 (3H, *s*), 3.95 (3H, *s*), 7.20, 7.40 (1H each, *s*), 6.38 and 7.95 (1H each, *d*, J = 10 Hz)] were identical with those of scopolin acetate [3].

Therefore, two acetyl groups in **1** are located in the glucosyl moiety, and their positions were determined by detailed analysis of the ^{1}H NMR spectrum of **1** with aid of decoupling procedures. A doublet at δ5.43 (1H, J = 8.5 Hz) was assigned to an anomeric proton, which was coupled with a C′-2 proton at δ5.68 (1H, *dd*, J = 8.5 and 8.5 Hz). On irradiation at δ5.68, a double doublet at δ4.23 (1H, *dd*, J = 8 and 8.5 Hz) due to a C′-3 proton collapsed into a doublet ($J = 8$ Hz). A C′-4 proton appeared at δ3.88 as a double doublet ($J = 8$ and 9 Hz) coupled with a C′-3 proton and a C′-5 proton at δ4.10 (1H, *ddd*, $J = 1.8$, 6 and 9 Hz). Two methylene protons of C′-6 occurred at δ4.52 (1H, *dd*, $J = 6$ and 12 Hz) and 4.86 (1H, *dd*, $J = 1.8$ and 12 Hz). The above results showed that the hydroxyl groups at C′-2 and C′-6 in **1** were acetylated because their chemical shifts were in low fields. The new compound is thus the 2′,6′-diacetate of scopolin.

EXPERIMENTAL

All Mps are uncorr. The chemical shifts of ^{1}H NMR spectra are given in δ-values with respect to TMS as the internal standard.

Extraction and isolation. The plants were collected in Kagoshima city and identified by Dr. Sako (Herbarium sample No. 1.). The fresh leaves of *V. suspensum* (1.5 kg) were extracted with MeOH (11 l. × 2). The extracts were evaporated to dryness to afford a dark green material. The material was diluted with H_2O, and extracted with Et_2O and then EtOAc. The EtOAc extract was further extracted with MeOH–$CHCl_3$ (1:19) to give a dark brown residue (17 g). The residue was subjected to column chromatography on silica gel with MeOH–$CHCl_3$ (1:19) and recrystallized from MeOH to give needles of **1** (11 mg), mp 178–179.5°; $[\alpha]_D^{25}$ −100° (MeOH; *c* 0.025); UV λ_{max}^{MeOH} nm (ε): 227 (10800), 286 (5600) and 328 (7600); IR ν_{max}^{nujol} cm^{-1}: 3550, 3250, 1740, 1620, 1570 and 1510; ^{1}H NMR (C_5D_5N): δ2.07 (3H × 2, *s*, COMe), 3.70 (3H, *s*, OMe), 3.88 (1H, *dd*, $J = 8$ and 9 Hz, C′-4 H), 4.10 (1H, *ddd*, $J = 1.8$, 6 and 9 Hz, C′-5 H), 4.23 (1H, *dd*, $J = 8$ and 8.5 Hz, C′-3H), 4.52 (1H, *dd*, $J = 6$ and 12 Hz, C′-6H), 4.86 (1H, *dd*, $J = 1.8$ and 12 Hz, C′-6H), 5.43 (1H, *d*, $J = 8.5$ Hz, C′-1H), 5.68 (1H, *dd*, $J = 8.5$ and 8.5 Hz, C′-2H), 6.30 (1H, *d*, $J = 9.8$ Hz, C-3H), 7.00 (1H, *s*, C-8H), 7.38 (1H, *s*, C-5H) and 7.63 (1H, *d*, $J = 9.8$ Hz, C-4H); MS *m/z*: 438 $[M]^+$. (Found: C, 52.75; H, 5.07%. Calc. for $C_{20}H_{22}O_{11} \cdot 1/2\ H_2O$: C, 52.61; H, 5.30%.)

Acetylation of **1**. Compound **1** was acetylated with Ac_2O–pyridine. The product was recrystallized from EtOH to give **2**, needles, mp 168–169°; IR ν_{max}^{nujol} cm^{-1}: 1770–1730, 1620, 1570, 1505, 920, 890 and 825; ^{1}H NMR ($CDCl_3$): δ2.10 (3H × 3, *s*, COMe), 2.17 (3H, *s*, COMe), 3.95 (3H, *s*, OMe), 6.38 (1H, *d*, $J = 10$ Hz, C-3H), 7.20 (1H, *s*, C-8H), 7.40 (1H, *s*, C-5H) and 7.95 (1H, *d*, $J = 10$ Hz, C-4H). (Found: C, 55.12; H, 5.08%. Calc. for $C_{24}H_{26}O_{13}$: C, 55.17; H, 5.02%.) The IR and ^{1}H NMR spectra were identical with those of scopolin acetate.

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A PHENOL ALLOSIDE FROM *VIBURNUM WRIGHTII*

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Key Word Index—*Viburnum wrightii*; Caprifoliaceae; phenol alloside.

Abstract—A new phenol alloside, *p*-hydroxyphenyl β-D-alloside, has been isolated from the leaves of *Viburnum wrightii* in addition to several known compounds. The structures were elucidated by spectroscopic and chemical methods.

INTRODUCTION

In continuation of our studies of the glycosides in *Viburnum* species, especially bitter components [1–4], we have now investigated *V. wrightii* Miq. From this plant a new non-bitter phenol alloside (**1**) has been isolated together with seven known compounds, α-amyrin palmitate, sitosterol, ursolic acid, *p*-coumaric acid, cosmosiin, 6-*O*-acetylarbutin and arbutin.

RESULTS AND DISCUSSION

The glycoside (**1**) had the molecular formula $C_{12}H_{16}O_7$. The IR spectrum showed absorption bands for a hydroxyl group at 3300 cm^{-1} and a *p*-substituted phenyl group at 1600, 1500 and 830 cm^{-1}. The presence of the *p*-substituted phenyl group was also apparent from the signals at δ 6.95–7.32 (4H, A_2B_2, $J = 10$ Hz) in the ^{1}H NMR spectrum. Four singlets at δ 1.98–2.14 (3H × 4)