APPENDIX 1

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CRYSTAL DATA AND DETAILS OF THE STRUCTURE DETERMINATION.

2-Methyl-4-[2',4',6'-trihydroxy-3'-(2-methylpropanoyl) phenyl] but-2-enyl acetate.

1. **Crystal data**.

Empirical formula $C_{17}H_{22}O_6$

Formula weight 322.35

Crystal system Monoclinic

Space group C2/c (No. 15)

a, b, c, [Angstrom] (a) 13.9411(9); (b) 17.4233 (11); (c) 15.6427 (10)

alpha, beta, gamma [deg.] 90 112.9050 (10) 90

V [Ang ** 3] 3500.0 (4)

Z 8

D (obs), D (calc) [g/cm ** 3] 0.000, 1.224

F (000) 1376

Mu (Moka) [/mm] 0.092

Crystal size 0.30 x 0.30 x 0.60

2. **Collection data**

Temperature (K) 296.2

Radiation [Angstrom] Moka 0.71073

Theta Min-Max [Deg] 5.92, 28.30

Scan, (Type and Range) [Deg] 0.00 + 0.35 Tan (Theta)

Hor. and Vert. Aperture [mm] 0.00 and 0.00

3. **Reference Reflection(s)**

Dataset 18: 18:23:23; 20:20

Tot., Uniq. Data, R (int) 19052, 4294, 0.030

Observed data [I > 2.0 sigma (I)] 3063

4. **Refinement**

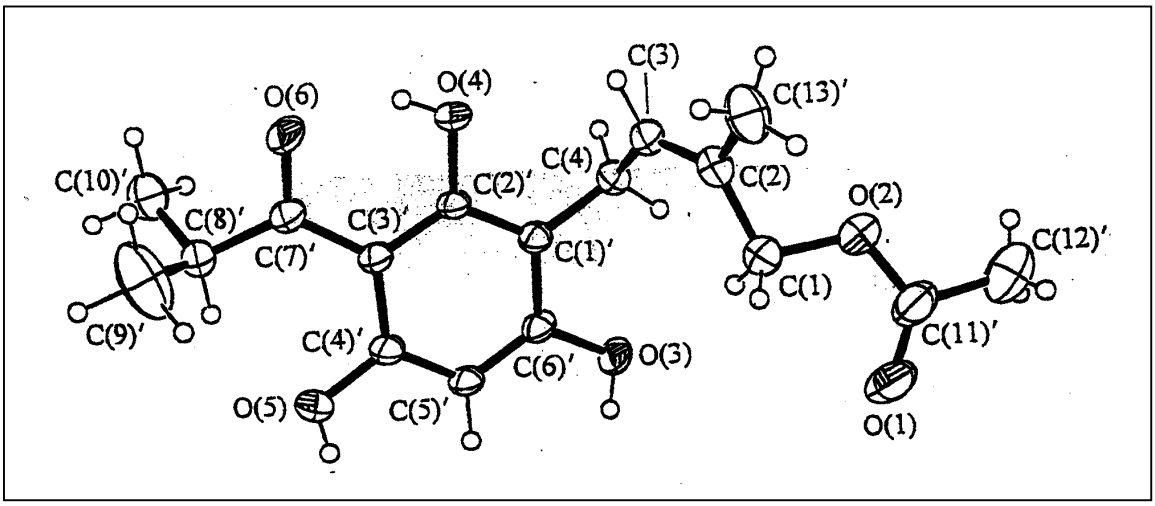
Nref, N par 0, 220

R, wR, S 0.0588, 0.1654, 1.053

w = 0.0961

Max. and Av. Shift/Error 0.05 0.00

Min. and Max resd. Dens. [e/Ang ^ 3]. 0.27, 0.40



Appendix 1 (Figure). The X-ray structure and molecular stereochemistry of the acylated phloroglucinol derivative,caespitate (C_{17} H_{22} O_6), showing the numbering scheme employed.

5. **Structure Solution**

The structure was solved in the monoclinic space group C2/c with the direct methods program SHELXS-97 [1] as implemented by the crystallographic program OSCAIL [2]. The E-map lead to the location of all non-hydrogen atoms; these were refined anisotropically with the program SHELXL-97. A difference Fourier synthesis led to location of all methine, methylene, and methyl hydrogens. All were included as idealized contributors in the least-squares process with standard SHELXL-97 [1] idealization

parameters. No evidence (difference Fourier map) for the inclusion of solvent in the lattice could be found. The final refinement converged to values of: R1 = 0.0588 and wR2 = 0.1654 for the observed 3063 unique reflections [I > 2.0 sigma (I)] and R1 = 0.0823 and wR2 = 0.1829 for all 4294 unique reflections. The maximum and minimum electron densities on the final differences Fourier map were 0.40 and. 0.27 e/A ^ 3, respectively. The final model was plotted using the program ORTEP [3].

Caespitate has a *cis*- double bond in the side chain (App.1. Figure). This is unusual stereochemistry in plant products and may be responsible for the observed activity of the compound (Drewes, personal communication).

REFERENCES

[1] SHELXL-97 : G.M. SHELDRICK, University of Gottingen. (a) G.M. SHELDRICK, *Acta Cryst.* 1990, A46, 467-473. (b) G.M. SHELDRICK, *Acta Cryst* 1993, D 49, 18- 23. (c) G.M. SHELDRICK, T. R.CHNEIDER. In: *Methods in entomology*. Vol. 277. Macromolecular crystallography. Part B. Eds., C.W. Carter and R.M. Sweet. pp 319-343. 1997.

[2] OSCAIL Version 8. P. McARDLE. 1995. Crystallography Centre, chemistry Department, NUI Galway, Ireland. *Journal of Applied Crystallography* 28: 65-65.

[3] ORTEP 3 for Windows V1.01 beta : Louis, J. Farrugia. Department of Chemistry, University of Glasgow, Glasgow G12 8QQ, Scotland. 1998 . (b) ORTEP III. M. N. BURNETT and C. K. JOHNSON. Oak Ridge National Laboratory Report. ORNL-6895. 1996.