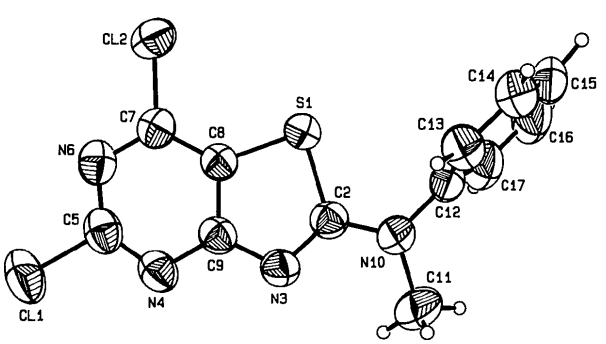


Fig. 1. Perspective drawing of (1) indicating atom labeling. Methyl H atoms H11*A*, H11*B* and H11*C* are shown. The other orientation is rotated approximately 60° with respect to the orientation shown. Thermal ellipsoids are drawn at the 50% probability level.

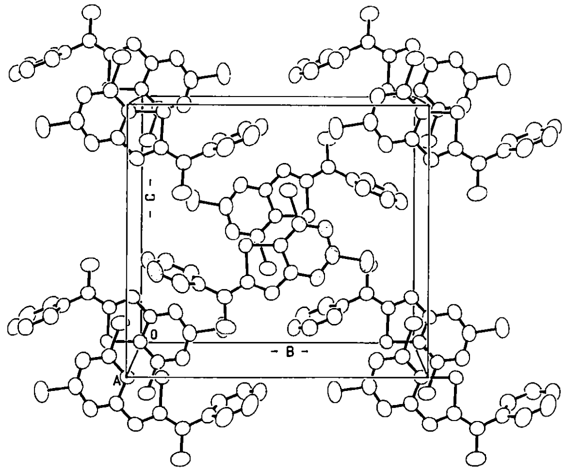


Fig. 2. Perspective drawing of the molecular packing as viewed perpendicular to the *bc* plane. The H atoms have been omitted for clarity. The thiazolopyrimidine rings form layers parallel to the *bc* plane with neighbors 3·49 and 3·60 Å apart. There is essentially no overlap of the thiazolopyrimidine rings although Cl2 is sandwiched between thiazole rings of adjacent molecules.

In the preceding paper we presented the structure of the 8-chloroadenine analog (7-amino-2-chloro-[1,3]thiazolo[4,5-*d*]pyrimidine) (Larson, Anderson, Cottam & Robins, 1989*b*) and we have recently reported the structure of the sodium salt of the 8-aminoguanine analog {2,5-diamino[1,3]thiazolo-[4,5-*d*]pyrimidin-7(6*H*)-one} (Larson, Anderson, Cottam & Robins, 1989*a*). The nucleoside 5-amino-3-$\beta$-D-ribofuranosyl-7(6*H*)-thioxothiazolo[4,5-*d*]pyrimidin-2(3*H*)-one, a 6-thioguanosine analog, has been reported (Nagahara *et al.*, 1989). No other thiazolo[4,5-*d*]pyrimidine crystal structures have been reported (Cambridge Structural Database, 1989).

## References

Cambridge Structural Database (1989). Univ. Chemical Laboratory, Lensfield Road, Cambridge, England.

Cordes, A. W. (1983). Personal communication.

Frenz, B. A. (1985). *Enraf–Nonius SDP-Plus Structure Determination Package*. Version 3·0. Enraf–Nonius, Delft, The Netherlands.

*International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Larson, S. B. (1980). PhD Dissertation, Brigham Young Univ., USA.

Larson, S. B., Anderson, J. D., Cottam, H. B. & Robins, R. K. (1989*a*). *Acta Cryst.* C**45**, 1073–1076.

Larson, S. B., Anderson, J. D., Cottam, H. B. & Robins, R. K. (1989*b*). *Acta Cryst.* C**45**, 1822–1825.

Nagahara, K., Anderson, J. D., Kini, G. D., Dalley, N. K., Larson, S. B., Smee, D. F., Sharma, B. S., Jolley, W. B., Robins, R. K. & Cottam, H. B. (1989). *J. Med. Chem.* In the press.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

---

*Acta Cryst.* (1989). C**45**, 1827–1829

# Structure of the Flavone Centaureidin

By Frank R. Fronczek, Felix J. Parodi and Nikolaus H. Fischer

*Department of Chemistry, Louisiana State University, Baton Rouge, Louisiana* 70803, *USA*

(*Received* 13 *December* 1988; *accepted* 29 *June* 1989)

**Abstract.** 5,7-Dihydroxy-2-(3-hydroxy-4-methoxyphenyl)-3,6-dimethoxy-4*H*-1-benzopyran-4-one, $C_{18}H_{16}O_8$, $M_r = 360·3$, monoclinic, $P2_1/c$, $a = 8·393\,(2)$, $b = 18·356\,(3)$, $c = 10·297\,(2)$ Å, $\beta = 97·964\,(13)°$, $V = 1571·1\,(8)$ Å$^3$, $Z = 4$, $D_x = 1·523$ g cm$^{-3}$, Cu $K\alpha$, $\lambda = 1·54184$ Å, $\mu = 9·85$ cm$^{-1}$, $F(000) = 752$, $T = 295$ K, $R = 0·041$ for 2241 observations (of 3235 unique data). The *A* ring exhibits maximum deviation, 0·013 (2) Å, from planarity, the heterocyclic *B* ring 0·017 (2) Å, and phenyl *C* ring 0·006 (2) Å. The *B* and *C* rings form a dihedral angle of 27·6 (1)°. The methoxy substitution of ring *B* is

0108-2701/89/111827-03$03.00 © 1989 International Union of Crystallography

the same as observed for the antitumor flavone calycopterin [Vijayalakshmi, Rajan, Srinivasan, & Ramachandran Nair (1986). *Acta Cryst.* C**42**, 1752–1754]. Intramolecular hydrogen bonds exist between the carbonyl and a hydroxy moiety of ring *A* with O···O distance 2·580 (2) Å and the angle at hydrogen 153 (2)°, as well as an intermolecular hydrogen bond involving the same OH group as acceptor and the hydroxy group of ring *C* as donor, O···O 2·806 (2) Å, angle at H 159 (2)°. We have isolated crystals of centaureidin, and antitumor active flavone, from *Baccharis salicina* (Asteraceae) which was collected near Corpus Christi, Texas.

**Experimental.** Centaureidin was obtained as colorless needles, data-collection crystal of dimensions 0·08 × 0·16 × 0·20 mm. Space group from absences $h0l$ with $l$ odd and $0k0$ with $k$ odd. Enraf–Nonius CAD-4 diffractometer with graphite monochromator, cell dimensions from setting angles of 25 reflections having $30 > \theta > 20°$. Data collection by $\omega$–$2\theta$ scans designed for $I = 50\sigma(I)$, subject to max. scan time = 120 s. Scan rates varied 0·46–3·30° min$^{-1}$. Reflections having $4 < 2\theta < 150°$, $0 \le h \le 10$, $0 \le k \le 23$, $-12 \le l \le 12$ were measured; corrected for background, Lorentz–polarization and absorption by $\psi$ scans, minimum relative transmission 0·8435; 3235 unique data; $R_{\rm int} = 0·021$ for averaging redundant $0kl$ and $0k\bar{l}$ data. Standard reflections 100, 020, 002, ± 1·4% random variation, no decay correction. Structure solved using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) refinement by full-matrix least squares based on $F$ with weights $w = 4F_o^2[\sigma^2(I) + (0·02F_o^2)^2]^{-1}$ with 2241 data for which $I > 3\sigma(I)$ (994 unobserved reflections), using Enraf–Nonius *SDP* (Frenz & Okaya, 1980). Non-H atoms anisotropic; H atoms located by $\Delta F$, and methyl H atoms were included as fixed contributions while others were refined isotropically. Atomic scattering factors of Cromer & Waber (1974) and anomalous coefficients of Cromer (1974). Final $R = 0·041$, $wR = 0·047$, $S = 2·080$ for 264 variables, extinction coefficient $g = 1·46\,(7) \times 10^{-6}$, where the correction factor $(1 + gI_c)^{-1}$ was applied to $F_c$, max. shift in final cycle $0·02\sigma$, max. residual density 0·21, min. $-0·16$ e Å$^{-3}$. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1,* bond distances, bond angles and selected torsion angles in Table 2. Fig. 1 shows the atom-numbering scheme.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

$$B_{\rm eq} = (8\pi^2/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$$

| | $x$ | $y$ | $z$ | $B_{\rm eq}$ (Å$^2$) |
|---|---|---|---|---|
| O1 | 0·7711 (2) | 0·21879 (7) | 0·5021 (1) | 3·10 (3) |
| O11 | 0·7625 (2) | 0·40786 (7) | 0·6062 (1) | 3·26 (3) |
| O12 | 0·9772 (2) | 0·34701 (8) | 0·8009 (1) | 3·76 (3) |
| O13 | 1·1218 (2) | 0·23410 (8) | 0·9102 (1) | 3·82 (3) |
| O14 | 1·1843 (2) | 0·08311 (8) | 0·8907 (2) | 4·16 (4) |
| O15 | 1·0284 (2) | 0·00853 (8) | 0·6824 (2) | 4·95 (4) |
| O24 | 0·3328 (2) | 0·22053 (8) | 0·1453 (2) | 4·98 (4) |
| O25 | 0·3266 (2) | 0·35263 (8) | 0·0359 (2) | 4·10 (3) |
| C2 | 0·7351 (2) | 0·2914 (1) | 0·5031 (2) | 2·73 (4) |
| C3 | 0·7986 (2) | 0·3343 (1) | 0·6042 (2) | 2·77 (4) |
| C4 | 0·9119 (2) | 0·3065 (1) | 0·7116 (2) | 2·86 (4) |
| C5 | 1·0498 (2) | 0·1945 (1) | 0·8075 (2) | 2·93 (4) |
| C6 | 1·0772 (3) | 0·1206 (1) | 0·8008 (2) | 3·18 (4) |
| C7 | 1·0021 (3) | 0·0813 (1) | 0·6922 (2) | 3·37 (4) |
| C8 | 0·9025 (3) | 0·1140 (1) | 0·5916 (2) | 3·32 (4) |
| C9 | 0·8735 (2) | 0·1877 (1) | 0·6020 (2) | 2·82 (4) |
| C10 | 0·9447 (2) | 0·2294 (1) | 0·7071 (2) | 2·74 (4) |
| C16 | 0·6190 (3) | 0·4238 (1) | 0·6599 (3) | 5·93 (7) |
| C17 | 1·1444 (3) | 0·0779 (1) | 1·0205 (2) | 4·43 (5) |
| C18 | 0·6261 (2) | 0·3118 (1) | 0·3845 (2) | 2·74 (4) |
| C19 | 0·5250 (3) | 0·2587 (1) | 0·3202 (2) | 3·17 (4) |
| C20 | 0·4286 (3) | 0·2744 (1) | 0·2042 (2) | 3·16 (4) |
| C21 | 0·4282 (3) | 0·3440 (1) | 0·1505 (2) | 3·01 (4) |
| C22 | 0·5279 (3) | 0·3970 (1) | 0·2131 (2) | 3·35 (4) |
| C23 | 0·6258 (3) | 0·3810 (1) | 0·3301 (2) | 3·33 (4) |
| C26 | 0·3184 (4) | 0·4219 (1) | −0·0267 (3) | 5·50 (6) |

Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles (°)*

| | | | |
|---|---|---|---|
| O1—C2 | 1·367 (2) | C3—C4 | 1·447 (3) |
| O1—C9 | 1·371 (2) | C4—C10 | 1·443 (3) |
| O11—C3 | 1·384 (2) | C5—C6 | 1·380 (3) |
| O11—C16 | 1·423 (3) | C5—C10 | 1·416 (3) |
| O12—C4 | 1·249 (2) | C6—C7 | 1·405 (3) |
| O13—C5 | 1·354 (2) | C7—C8 | 1·375 (3) |
| O14—C6 | 1·381 (2) | C8—C9 | 1·381 (3) |
| O14—C17 | 1·425 (3) | C9—C10 | 1·391 (3) |
| O15—C7 | 1·360 (2) | C18—C19 | 1·398 (3) |
| O24—C20 | 1·363 (2) | C18—C23 | 1·389 (3) |
| O25—C21 | 1·366 (2) | C19—C20 | 1·376 (3) |
| O25—C26 | 1·422 (3) | C20—C21 | 1·393 (3) |
| C2—C3 | 1·355 (3) | C21—C22 | 1·382 (3) |
| C2—C18 | 1·469 (3) | C22—C23 | 1·392 (3) |
| C2—O1—C9 | 121·1 (1) | O13—C5—C10 | 119·5 (2) |
| C3—O11—C16 | 113·9 (2) | C6—C5—C10 | 120.0 (2) |
| C6—O14—C17 | 116·5 (2) | O14—C6—C5 | 123·6 (2) |
| C21—O25—C26 | 118·7 (2) | O14—C6—C7 | 117·2 (2) |
| O1—C2—C3 | 120·6 (2) | C5—C6—C7 | 119·0 (2) |
| O1—C2—C18 | 110·8 (2) | O15—C7—C6 | 120·3 (2) |
| C3—C2—C18 | 128·6 (2) | O15—C7—C8 | 117·4 (2) |
| O11—C3—C2 | 121·0 (2) | C6—C7—C8 | 122·3 (2) |
| O11—C3—C4 | 117·1 (2) | C7—C8—C9 | 117·7 (2) |
| C2—C3—C4 | 121·9 (2) | O1—C9—C8 | 116·7 (2) |
| O12—C4—C3 | 121·8 (2) | O1—C9—C10 | 120·8 (2) |
| O12—C4—C10 | 122·8 (2) | C8—C9—C10 | 122·6 (2) |
| C3—C4—C10 | 115·4 (2) | C4—C10—C5 | 121·5 (2) |
| O13—C5—C6 | 120·5 (2) | C4—C10—C9 | 120·1 (2) |
| C5—C10—C9 | 118·4 (2) | C19—C20—C21 | 120·3 (2) |
| C2—C18—C19 | 118·9 (2) | O25—C21—C20 | 114·6 (2) |
| C2—C18—C23 | 122·3 (2) | O25—C21—C22 | 125·9 (2) |
| C19—C18—C23 | 118·7 (2) | C20—C21—C22 | 119·5 (2) |
| C18—C19—C20 | 120·7 (2) | C21—C22—C23 | 120·1 (2) |
| O24—C20—C19 | 118·3 (2) | C18—C23—C22 | 120·6 (2) |
| O24—C20—C21 | 121·4 (2) | | |
| C16—O11—C3—C2 | 86·0 (3) | C26—O25—C21—C22 | −0·7 (3) |
| C17—O14—C6—C5 | 67·1 (3) | O1—C2—C18—C19 | 26·1 (3) |

---

* Tables of H-atom parameters, distances and angles involving H atoms, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52077 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

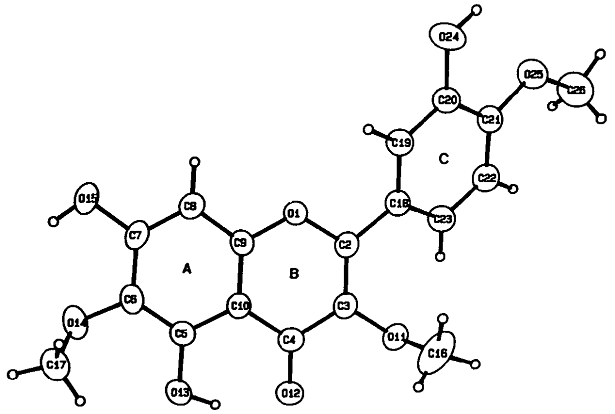


Fig. 1. Numbering scheme with thermal ellipsoids drawn at the 40% probability level. H atoms have arbitrary radius.

**Related literature.** Isolation of centaureidin from *Centaurea* species (Bohlmann & Zdero, 1967). Antitumor activity of centaureidin (Kupchan & Bauerschmidt, 1971). Crystal structure of the pharmacologically active 5,4′-dihydroxy-3,6,7,8-tetramethoxyflavone, calycopterin (Vijayalakshmi, Rajan, Srinivasan & Ramachandran Nair, 1986).

### References

Bohlmann, F. & Zdero, C. (1967). *Tetrahedron Lett.* **33**, 3239–3242.

Cromer, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2.B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Frenz, B. A. & Okaya, Y. (1980). *Enraf–Nonius Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

Kupchan, S. M. & Bauerschmidt, E. (1971). *Phytochemistry*, **10**, 664–666.

Main, P., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Vijayalakshmi, J., Rajan, S. S., Srinivasan, R. & Ramachandran Nair, A. B. (1986). *Acta Cryst.* C**42**, 1752–1754.

---

*Acta Cryst.* (1989). C**45**, 1829–1831

# Structure of the Guaianolide Dehydrocostus Lactone

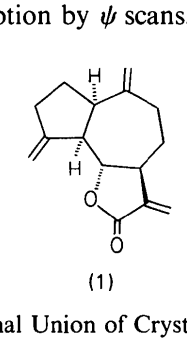
By Frank R. Fronczek, David Vargas, Felix J. Parodi and Nikolaus H. Fischer

*Department of Chemistry, Louisiana State University, Baton Rouge, Louisiana* 70803, *USA*

(*Received* 13 *December* 1988; *accepted* 29 *June* 1989)

**Abstract.** Decahydro-3,6,9-tris(methylene)azuleno-[4,5-*b*]furan-2(3*H*)-one, $C_{15}H_{18}O_2$, $M_r = 230{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}810\,(1)$, $b = 11{\cdot}403\,(1)$, $c = 14{\cdot}240\,(1)$ Å, $V = 1268{\cdot}2\,(3)$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}206$ g cm$^{-3}$, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54184$ Å, $\mu = 5{\cdot}87$ cm$^{-1}$, $F(000) = 496$, $T = 298$ K, $R = 0{\cdot}035$ for 1432 observations (of 1515 unique data). The title compound, which exhibits no molluscicidal acitivity, differs in conformation from its 7$\alpha$-hydroxy analog, 7$\alpha$-hydroxy-3-desoxyzaluzanin C, which is highly active [Fronczek, Vargas, Fischer & Hostettmann (1984). *J. Nat. Prod.* **47**, 1036–1039]. The conformation of the seven-membered ring is a distorted twist-chair, with the pseudodiad axis passing through C8, and asymmetry parameter $\Delta C_2 = 8{\cdot}2°$. The lactone ring is in the half-chair conformation with carbonyl carbon C12 on the local twofold axis, and $\Delta C_2 = 3{\cdot}0°$. The other five-membered ring has a distorted half-chair conformation with the axis passing through C4, and $\Delta C_2 = 7{\cdot}0°$. Crystals of the guaianolide dehydrocostus lactone were isolated from costus oil purchased from Pierre Chauvet S. A., France.

**Experimental.** Dehydrocostus lactone, (1), was obtained as colorless needles, data-collection crystal of dimensions 0·44 × 0·48 × 0·72 mm. Space group from absences $h00$ with $h$ odd, $0k0$ with $k$ odd and $00l$ with $l$ odd. Enraf–Nonius CAD-4 diffractometer with graphite monochromator; cell dimensions from setting angles of 25 reflections having $40 > \theta > 35°$. Data collection by $\omega$–$2\theta$ scans designed for $I = 50\sigma(I)$ subject to max. scan time = 120 s. Scan rates varied $0{\cdot}63 - 4{\cdot}0°$ min$^{-1}$. Reflections having $4 < 2\theta < 150°$, $0 \le h \le 9$, $0 \le k \le 14$, $0 \le l \le 17$ were measured; corrected for background, Lorentz, polarization and absorption by $\psi$ scans, minimum relative



(1)

0108-2701/89/111829-03$03.00 © 1989 International Union of Crystallography