C6—N2—N3—C7	177.4 (2)
N3—N2—C6—C4	−177.01 (18)
C5—C4—C6—O1	5.6 (3)
C3—C4—C6—O1	−171.7 (2)
N2—N3—C7—C8	178.41 (19)
N3—C7—C8—C13	176.0 (2)
N3—C7—C8—C9	−6.4 (3)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1N2···O2*W*	0.87 (2)	2.28 (3)	3.011 (2)	142 (2)
O1*W*—H1*W*1···N3	0.85 (4)	2.25 (4)	3.088 (3)	167 (4)
O2—H1O2···N1^{i}	0.89 (3)	1.87 (3)	2.759 (3)	170 (3)
O2*W*—H1*W*2···O1^{ii}	0.89 (3)	1.92 (3)	2.807 (2)	173 (3)
C7—H7···O1*W*^{iii}	0.99 (2)	2.50 (2)	3.351 (3)	144 (2)

Symmetry codes: (i) $\frac{1}{2} + x, y - \frac{1}{2}, z$; (ii) $1 - x, 1 - y, 1 - z$; (iii) $1 - x, -y, 1 - z$.

The data collection covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω. The crystal-to-detector distance was 4 cm and the detector swing angle was −35°. Coverage of the unique set is over 99% complete. Crystal decay was monitored by repeating 30 initial frames at the end of data collection and analysing the duplicate reflections, and was found to be negligible.

The H atoms were refined because of their involvement in the formation of the supramolecular structure. C—H distances range from 0.94 (2) to 1.01 (2) Å and U_{iso} values for H atoms range from 0.029 (6) to 0.15 (2) Å^2.

Data collection: *SMART* (Siemens, 1996*a*). Cell refinement: *SAINT* (Siemens, 1996*b*). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1996). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

The authors would like to thank the National Nature Science Foundation of China and China Post-Doctoral Science Foundation for support, and the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 190-9609-2801. KC thanks the Universiti Sains Malaysia for a Visiting Post-Doctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1156). Services for accessing these data are described at the back of the journal.

References

Fun, H.-K., Lu, Z.-L., Duan, C.-Y., Tian, Y.-P., You, X.-Z., Gong, X.-Y. & Guo, Y.-M. (1997). *Acta Cryst.* C**53**, 1454–1455.

Fun, H.-K., Lu, Z.-L., Duan, C.-Y., Tian, Y.-P., You, X.-Z., Guo, Y.-M. & Gong, X.-Y. (1997). *Acta Cryst.* C**53**, 1452–1454.

Lu, Z.-L., Duan, C.-Y., Tian, Y.-P., You, X.-Z., Fun, H.-K. & Sivakumar, K. (1996). *Acta Cryst.* C**52**, 1507–1509.

Ma, Y.-X., Lu, Z.-L., Song, Q.-B. & Wu, X.-L. (1994). *J. Coord. Chem.* **32**, 353–359.

Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.

Sheldrick, G. M. (1996). *SHELXTL. Structure Determination Programs.* Version 5. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Siemens (1996*a*). *SMART. Area Detector Control Software.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Siemens (1996*b*). *SAINT. Area Detector and Integration Software.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1999). C**55**, 576–578

Tamarixetin monohydrate

Nor Syarmila Shuib,[a] Teng Wah Sam,[a] Keng Chong Wong,[a] Kandasamy Chinnakali[b]† and Hoong-Kun Fun[b]

[a]*School of Chemical Sciences, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and* [b]*X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia. E-mail: hkfun@usm.my*

(*Received 19 November 1998; accepted 14 December 1998*)

Abstract

In the title compound, 3,5,7,3′-tetrahydroxy-4′-methoxyflavone monohydrate [or 3,5,7-trihydroxy-2-(3-hydroxy-4-methoxyphenyl)-4*H*-1-benzopyran-4-one monohydrate], $C_{16}H_{12}O_7 \cdot H_2O$, there are two independent molecules in the asymmetric unit, linked through OH groups to form an O—H···O hydrogen-bonded dimer. The benzopyran rings are slightly distorted from planarity; in one of the molecules the phenyl ring is coplanar with the pyrone ring, while in the other it is twisted by 4.8 (1)°. The hydroxyl groups are involved in various O—H···O and C—H···O hydrogen bonds and the molecules are stacked parallel to the (220) plane.

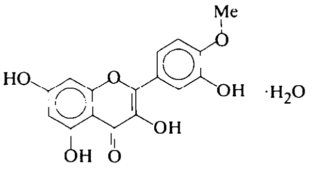
Comment

Chromolaena odorata King and Robinson [formerly *Eupatorium odoratum* (*L.*)] is a perennial shrub native to Central and South America. It has, however, found its way as a troublesome weed into practically every tropical region. In some of these areas, it has acquired a reputation as a medicinal herb for a variety of ailments, including malaria, fever and infections, and as a haemostatic agent. A blood anticoagulating effect in the leaf extract was discovered by us and we speculated that this could be a consequence of the calcium-ion complexing ability of the 3-flavonols found in the extract. It was therefore an important extension of this study that the configurations of some of these

---

† On leave from: Department of Physics, Anna University, Chennai 600 025, India.

3-flavonols be unambiguously established. The crystal structure of one of these 3-hydroxyflavonols, tamarixetin (3,5,7,3′-tetrahydroxy-4′-methoxyflavone), (I)·H_2O, is now reported.



(I)

The asymmetric unit consists of two independent molecules, *A* and *B*, along with two water molecules. Molecules *A* and *B* are related by a pseudo inversion centre at (0.034, 0.463, 0.238) and have different orientations for the OH groups attached to the C7 and C13 positions. Bond lengths and angles observed in these two molecules agree with each other and are comparable with those reported for a closely related structure, quercetin dihydrate (Jin *et al.*, 1990).

In both molecules, the heterocyclic ring is planar, but it is not quite coplanar with the benzene ring; the dihedral angle is 2.9 (1) for *A* and 2.8 (1)° for *B*. In *B*, the phenyl ring is coplanar with the pyrone ring, whereas in *A*, it is slightly twisted and makes a dihedral angle of 4.8 (1)° with the pyrone ring.

In both molecules, there are two intramolecular hydrogen bonds. Each water molecule is linked to either molecule *A* or *B* through a hydrogen bond (O13*A*···O1*W*, O7*B*···O2*W*). Molecules *A* and *B* are linked through O3*A*···O4*B* and O3*B*···O4*A* hydrogen bonds to form a dimeric pair. In the solid state, the molecules are linked (O7*A*···O13*A*^{i} and O13*B*···O7*B*^{i}) to form sheets almost parallel to the (220) plane, with these molecular sheets being linked by (O1*W*···O7*A*^{ii}, O2*W*···O5*A*^{iii} and O2*W*···O7*A*^{iv}) hydrogen bonds involving the water molecules [symmetry codes: (i) $x-1$, $1+y$, z; (ii) $-x$, $1-y$, $-z$; (iii) $-x$, $1-y$, $1-z$; (iv) x, $y-1$, $1+z$]. Full details are given in Table 2. The sheets are stacked in such a way that the molecules of *A* and *B* form separate columns, with a perpendicular distance of 3.555 (3) Å and 3.402 (3) Å for *A* and *B*, respectively, indicating significant π–π stacking interactions.

Experimental

Air-dried leaves of *Chromolaena odorata* King and Robinson were first defatted in a Soxhlet apparatus with petroleum ether (333–353 K) and then re-extracted with acetone in the same apparatus. A quantity of the crude acetone extract (30.6 g) was subjected to column chromatography on silica gel (250 g) with a 1:4 mixture of acetone and chloroform. The fraction (0.63 g) which consisted largely of a single spot with $R_f =$ 0.58 on silica gel thin-layer chromatography [Merck thin-layer

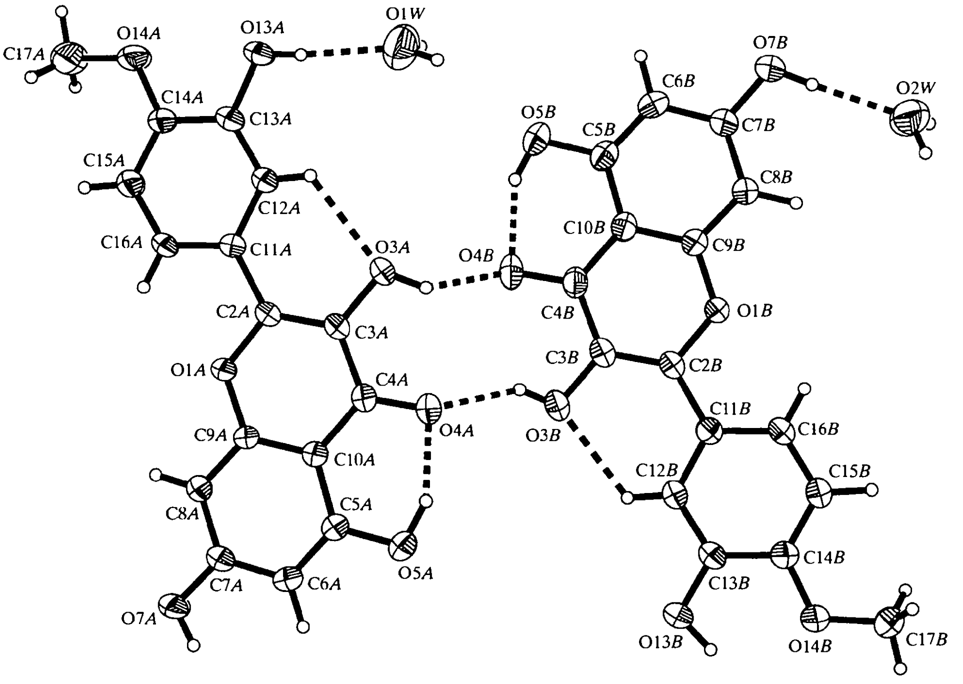


Fig. 1. The structures of the two independent molecules of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme for the non-H atoms, and illustrating the hydrogen-bonded dimer. H atoms are shown as circles of an arbitrary radius and inter- and intramolecular hydrogen bonds are indicated by dashed lines.

chromatography aluminium sheets, product number 1.05554; solvent: hexane–ethyl acetate (1:1)] was further fractionated on a Sephadex LH-20 column with methanol as the eluting solvent, to give a yellow solid (0.43 g). Recrystallization of this solid from ethanol gave tamarixetin (m.p. 532 K), the title compound, as needles.

Crystal data

$C_{16}H_{12}O_7{\cdot}H_2O$
$M_r = 334.27$
Triclinic
$P\bar{1}$
$a = 7.3750\,(1)$ Å
$b = 12.5300\,(2)$ Å
$c = 15.8752\,(2)$ Å
$\alpha = 84.095\,(1)^\circ$
$\beta = 86.380\,(1)^\circ$
$\gamma = 77.933\,(1)^\circ$
$V = 1425.63\,(3)$ Å^3
$Z = 4$
$D_x = 1.557$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 7068 reflections
$\theta = 1.29$–28.28°
$\mu = 0.127$ mm^{-1}
$T = 293\,(2)$ K
Needle
$0.40 \times 0.38 \times 0.18$ mm
Yellow

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
11 223 measured reflections
6571 independent reflections
5400 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.053$
$\theta_{\rm max} = 28.28^\circ$
$h = -9 \rightarrow 9$
$k = -16 \rightarrow 16$
$l = 0 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.163$
$S = 1.184$
6571 reflections
546 parameters
H atoms: see text
$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2 + 0.7772P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$

$\Delta\rho_{\rm max} = 0.47$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.33$ e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 1997)
Extinction coefficient: 0.027 (4)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1A—C9A	1.364 (2)	O1B—C9B	1.358 (2)
O1A—C2A	1.374 (2)	O1B—C2B	1.367 (2)
O3A—C3A	1.356 (2)	O3B—C3B	1.360 (2)
O4A—C4A	1.262 (2)	O4B—C4B	1.268 (2)
O5A—C5A	1.359 (2)	O5B—C5B	1.361 (2)
O7A—C7A	1.363 (2)	O7B—C7B	1.357 (3)
O13A—C13A	1.371 (2)	O13B—C13B	1.366 (2)
O14A—C14A	1.365 (2)	O14B—C14B	1.368 (2)
O14A—C17A	1.420 (3)	O14B—C17B	1.419 (3)

O1A—C2A—C11A—C16A	−5.3 (3)
C3A—C2A—C11A—C12A	−5.5 (3)
C17A—O14A—C14A—C15A	−2.1 (4)
O1B—C2B—C11B—C16B	3.0 (3)
C3B—C2B—C11B—C12B	4.0 (3)
C17B—O14B—C14B—C15B	2.1 (3)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
O5A—H1O5···O4A	0.95 (4)	1.72 (4)	2.603 (2)	153 (3)
C12A—H12A···O3A	0.93 (3)	2.25 (2)	2.847 (2)	122 (2)
O5B—H2O5···O4B	0.90 (4)	1.79 (4)	2.617 (3)	154 (3)
C12B—H12B···O3B	0.93 (3)	2.26 (2)	2.870 (3)	122 (2)
O13B—H13B···O14B	0.91 (3)	2.14 (3)	2.663 (2)	116 (3)
O13A—H13A···O1W	0.81 (3)	1.91 (3)	2.712 (3)	173 (3)
O7B—H2O7···O2W	0.85 (3)	1.96 (3)	2.812 (3)	177 (4)
O3B—H2O3···O4A	0.86 (3)	2.01 (3)	2.768 (2)	147 (3)
O3A—H1O3···O4B	0.89 (3)	1.88 (3)	2.676 (2)	147 (3)
O7A—H1O7···O13A$^{\rm i}$	0.84 (3)	2.03 (3)	2.866 (2)	174 (3)
O13B—H13B···O7B$^{\rm i}$	0.91 (3)	2.03 (4)	2.878 (2)	154 (3)
O1W—H1W1···O7A$^{\rm ii}$	0.89 (6)	2.28 (6)	3.035 (4)	143 (6)
O2W—H1W2···O5A$^{\rm iii}$	0.89 (6)	1.98 (6)	2.876 (3)	176 (6)
O2W—H2W2···O7A$^{\rm iv}$	0.80 (6)	2.38 (6)	3.066 (3)	143 (6)

Symmetry codes: (i) $x-1, 1+y, z$; (ii) $-x, 1-y, -z$; (iii) $-x, 1-y, 1-z$; (iv) $x, y-1, 1+z$.

The data collection covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω. The crystal-to-detector distance was 4 cm and the detector swing angle was −35°. Crystal decay was monitored by repeating 30 initial frames at the end of data collection and analysing the intensity of duplicate reflections, and was found to be negligible.

All the H atoms were located from a difference map and refined isotropically. Since the H atoms of O1W were not stable during refinement, the bond lengths and angles involving them were restrained. Though the ratio of observed reflections to parameters is slightly less than 10 (9.9), the H atoms were refined, since they are involved in the hydrogen bonds.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

The authors would like to thank the Malaysian Government and Universiti Sains Malaysia for research grants R&D Nos. 190-9609-2801 and 190-9601-3401. KC and NSS thank the Universiti Sains Malaysia for a Visiting Post-Doctoral Fellowship and a Post-Graduate Fellowship, respectively.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1249). Services for accessing these data are described at the back of the journal.

References

Jin, G.-Z., Yamagata, Y. & Tomita, K.-I. (1990). *Acta Cryst.* **C46**, 310–313.

Sheldrick, G. M. (1997). *SHELXTL. Structure Determination Programs*. Version 5.10. Bruker Analytical X-ray Systems Inc., Madison, Wisconsin, USA.

Siemens (1996). *SMART* and *SAINT*. *Area Detector Control and Integration Software*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Spek, A. L. (1990). *Acta Cryst.* **A46**, C-34.