Crystalline Structure of Mangiferin, a *C*-Glycosyl-Substituted 9*H*-Xanthen-9-one Isolated from the Stem Bark of *Mangifera indica*

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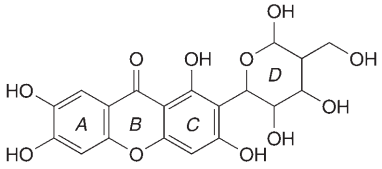
The crystalline structure of mangiferin (=2-β-D-glucopyranosyl-1,3,6,7-tetrahydroxy-9*H*-xanthen-9-one; **1**), a biologically active xanthenone *C*-glycoside, isolated from the stem bark of *Mangifera indica* (Anacardiaceae), was unambiguously determined by single-crystal X-ray diffraction (XRD). The crystal structure is summarized as follows: triclinic, *P*1, $a = 7.6575(5)$, $b = 11.2094(8)$, $c = 11.8749(8)$ Å, $\alpha = 79.967(5)$, $\beta = 87.988(4)$, $\gamma = 72.164(4)^\circ$, $V = 955.3(1)$ Å^3, and $Z = 2$. The structure also shows two molecules in the asymmetric unit cell and five crystallization H_2O molecules. The packing is stabilized by several intermolecular H-bonds involving either the two symmetry-independent mangiferin molecules **1a** and **1b**, or the H_2O ones.

Introduction. – Mangiferin (=2-β-D-glucopyranosyl-1,3,6,7-tetrahydroxy-9*H*-xanthen-9-one; **1**) can be found in various vegetal species, including *Mangifera indica*, species belonging to the Anacardiaceae family. Chemical studies of *M. indica* (mango tree) have made possible the isolation of flavonoids [1][2], xanthenones (mangiferin) [3][4], isomangiferin [5], triterpenoids [6], polysaccharides [7], tannins, polyphenols [8], and volatile compounds [9]. Among these substances, **1** is the principal component, representing 2.6% of the bark's EtOH extract [10].



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Various assays have been reported, either with the mango tree bark extracts or with the isolated mangiferin (**1**), to verify the potential of this vegetal species to control or prevent diseases. The anti-inflammatory activity was verified in the bark's EtOH extract, and *Vimang*® [10], a patented product and traditionally used in Cuba, is an anti-

inflammatory, analgesic, and antioxidant drug [11]. The isolated **1** of *M. indica* exhibited also anticarcinogenic activity in *in vivo* assays with cancerous colon cells [12]. The most studied biological activity of the mango tree bark extract or of **1** itself is the potential antioxidant activity [13]. Complexation studies with Fe^{III} performed recently revealed an increase of efficiency of this activity [14].

Despite the innumerous studies about the biological activities of mangiferin (**1**), there are few reports dealing with its physicochemical properties. Although the molecular structure of **1** is known since 1967 [15] its crystal structure is not reported in the literature. Since weak intermolecular interactions have an important influence on the macroscopic properties of solids we studied the intramolecular geometry of solid **1** and determined its structure by single-crystal X-ray diffraction.

Results and Discussion. – Crystal data of mangiferin **1** are summarized in *Table 1*. It crystallizes in the triclinic space group *P*1 (No. 1) presenting two molecules, **1a** and **1b**, in the asymmetric unit. *Fig. 1* is an ORTEP-3 [16] view of the two molecules **1a** and **1b** wherein the D-glucose moiety is kept in the same spacial orientation to show more clearly the intramolecular differences between molecules **1a** (*Fig. 1*,*a*) and **1b** (*Fig. 1*,*b*). The main difference between them is the torsion angles involving the D-glucopyranose moiety, *i.e.*, in molecule **1a** the angle O(11)–C(15)–C(2)–C(14) is

Table 1. *Crystal Data and Structure Refinement of Mangiferin* (**1**)

Molecular formula	$(C_{19}H_{18}O_{11})_2(H_2O)_5$
M_r	934.75
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system	triclinic
Space group	*P*1
Unit cell dimensions	$a = 7.6575(5)$ Å
	$b = 11.2094(8)$ Å
	$c = 11.8749(8)$ Å
	$\alpha = 79.967\ (5)^\circ$
	$\beta = 87.988\ (4)^\circ$
	$\gamma = 72.164\ (4)^\circ$
Volume	955.3(1) $Å^3$
Z	2
Density (calculated)	1.625 Mg/m^3
Absorption coefficient	0.141 mm^{-1}
Crystal size	$0.06 \times 0.07 \times 0.27$ mm
θ Range for data collection	$3.21 - 26^\circ$
Reflections collected	12702
Independent reflections	3730 (R(int) = 0.0799)
Completeness to $\theta = 26.0^\circ$	99.6%
Refinement method	full-matrix least-squares on F^2
Data/restraints/parameters	3730/3/609
Goodness-of-fit on F^2	1.015
Final R indices ($I > 2\sigma(I)$)	$R_1 = 0.0471$, $wR_2 = 0.1102$
R indices (all data)	$R_1 = 0.0648$, $wR_2 = 0.1214$
Largest diff. peak and hole	0.251 and -0.311 e · $Å^{-3}$

129.28°, and in molecule **1b** the corresponding angle O(22)–C(35)–C(21)–C(34) is 86.60° (see also *Fig. 2*, *a*). Besides these torsion-angle differences, the corresponding OH groups of **1a** and **1b** containing the O(10) and O(21) atoms are arranged differently due to the different torsion angles O(11)–C(19)–C(20)–O(10) (–63.4(5)°) and O(22)–C(39)–C(40)–O(21) (61.2(5)°) (see *Fig. 1* and *Fig. 2*, *b*). Both intramolecular differences, better illustrated in *Fig. 2*, are a consequence of noncovalent H-bond

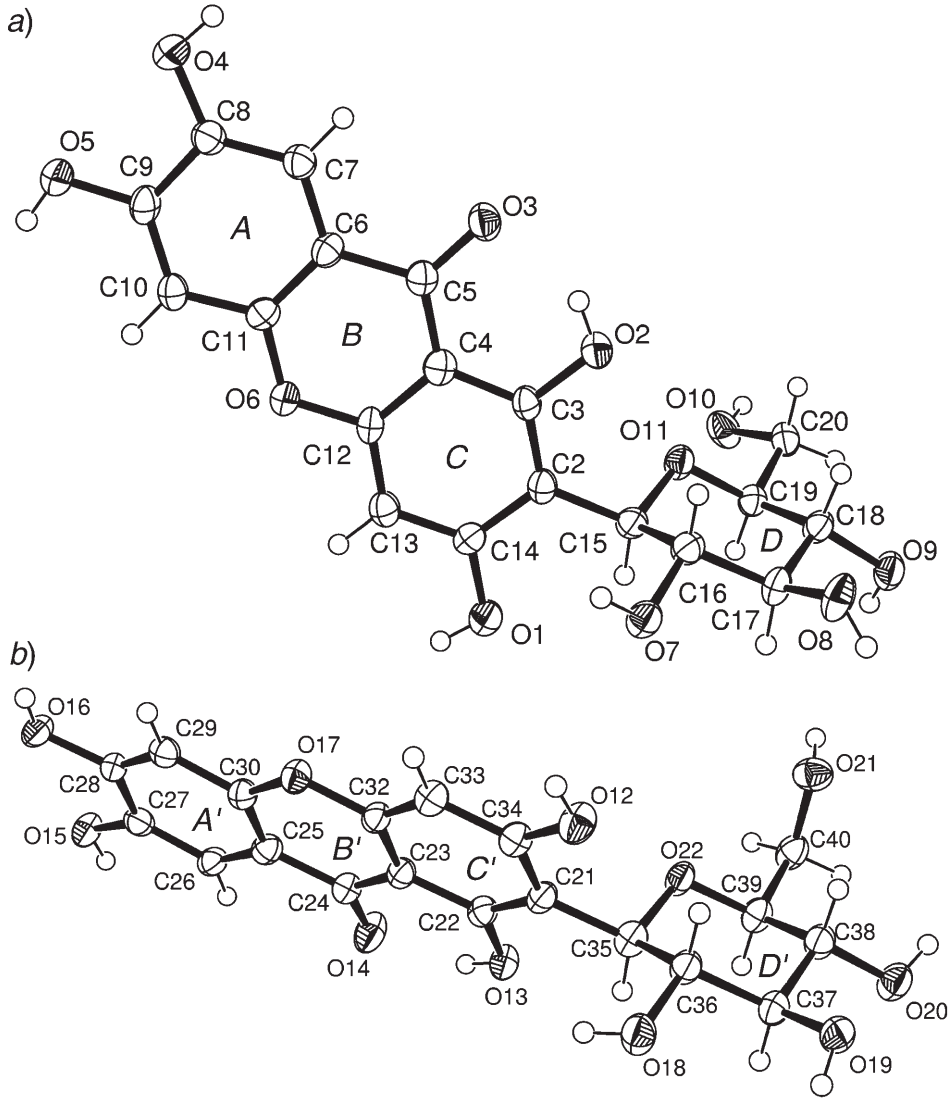

Fig. 1. *ORTEP Plot and atom numbering of mangiferin* (**1**) *showing the two molecules* a) **1a** *and* b) **1b** *in the asymmetric unit, with the xanthenone rings* A, B, *and* C, *and the* D-*glucopyranose moiety* D. The ellipsoids represent the 50% probability level. The H_2O molecules are omitted for clarity.

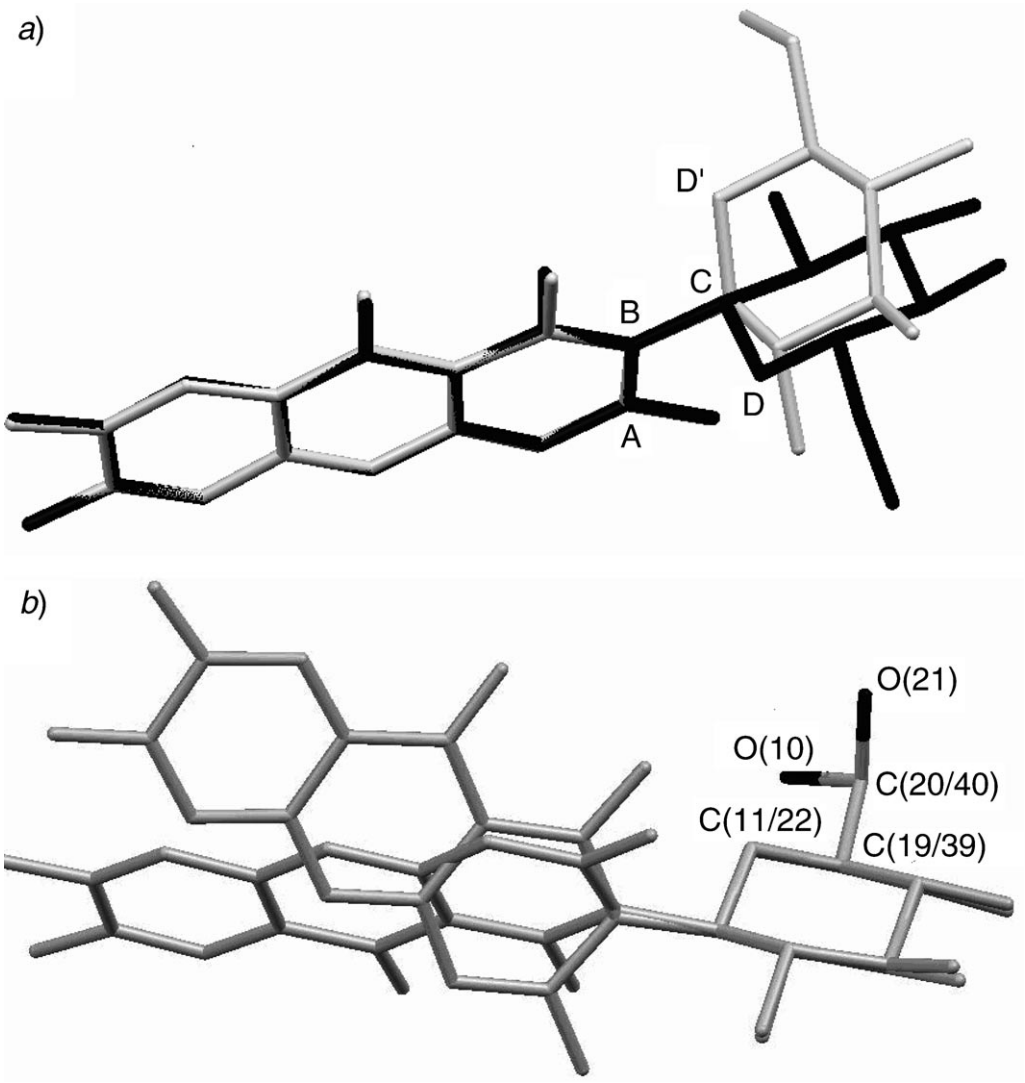

Fig. 2. *Overlay* a) *of the xanthenone and* b) D-*glucopyranose rings showing the differences between molecules* **1a** *and* **1b**. Labels: A = C(14) and C(34), B = C(2) and C(21), C = C(15) and C(35), D = O(11), and D′ = O(22) (see *Fig. 1* for atom numbering).

interactions, which lead to a well-organized intermolecular structure as discussed below. Another important structural feature is the presence of five symmetry-independent crystallization H_2O molecules, which are omitted in *Fig. 1* for clarity.

Not considering the D-glucopyranosyl substituent, the intramolecular geometries of the two molecules **1a** and **1b** are very similar (*Fig. 2,a*). Comparison of these molecules in terms of rings *A*, *B*, and *C*, and of the first neighboring atoms linked to them by the method of *Kabsch* and *Sandar* [17] showed them to be very similar with a root-mean-

square deviation between homologous atoms of 0.037 Å. Another interesting result pointed out by this method is that the transformation relating the two independent three-ring moieties coincides with those belonging to the space group $P1$ (x,y,z and $-x,-y,-z$). This is consistent with a centrosymmetric super-structure when the D-glucopyranose ring is not taken into account. The intramolecular analysis of molecule **1a** shows that all atoms in the rings *A*, *B*, and *C* lie within $-0.096(3)$ Å of the least-squares plane through the three-ring system, including all O-atoms linked to them. The rings *A*, *B*, and *C* are also individually almost planar. The largest deviations from the individual least-squares planes are $0.013(5)$, $0.006(5)$, and $-0.021(5)$ Å for rings *A*, *B*, and *C*, respectively. It is important to emphasize that the same behavior was observed for molecule **1b** considering the rings *A′*, *B′*, and *C′*. Rings *D* and *D′* adopt chair conformation in both molecules as shown in *Figs. 1* and *2*. The weighted average absolute torsion angles [18] in the rings *D* and *D′* are $58(3)^\circ$ and $59(1)^\circ$ for molecules **1a** and **1b**, respectively.

The main geometric parameters are shown in *Table 2*. The observed geometries of the benzene rings *A* and *C* and of the pyran ring *B* agree well with the similar pyranoxanthenone geometry reported for 6-deoxyjacareubin (= 5,10-dihydroxy-2,2-dimethylpyrano[3,2-*b*]xanthen-6(2*H*)-one), a natural xanthenone isolated from leaves of *Vismia latifolia* (Guttiferae family) [19]. To have a rapid access to information on the preferred values of bond lengths, valence angles, and exocyclic torsion angles, the molecular conformation of **1** was analyzed by MOGUL [20], a knowledge base of molecular geometry derived from the *Cambridge Structural Database* (CSD) [21]. This study showed that all bond lengths and bond angles are in agreement with the expected values for a good X-ray-diffraction structure refinement. The analysis also revealed interesting geometrical features due to resonance effects. Indeed, in both molecules **1a** and **1b**, the C–O bond lengths in the pyran ring *B* are longer than the expected ones: C(5)–O(3) is 1.275 Å, and C(24)–O(14) is 1.265 Å, whereas the expected value is 1.225 Å. These geometrical features are due to resonance involving the moiety O(2)–C(3)–C(4)–C(5)–O(3) and O(14)–C(24)–C(23)–C(22)–O(13) in **1a** and **1b**, respectively (see *Fig. 1* and *Table 2*).

The molecules **1a** and **2b** exhibited strong intramolecular H-bonds involving O(2)···O(3) and O(13)···O(14), respectively (*Table 3* and *Fig. 1*). *Fig. 3* shows the

Table 2. *Bond Lengths* [Å] *and Angles* [°] *of Molecule* **1a** *Determined by X-Ray Diffraction* (query value) *and MOGUL Intramolecular Analysis*

Geometry type	Query value	Mean	Geometry type	Query value	Mean
C(2)–C(15)	1.518(2)	1.51(2)	C(14)–C(2)–C(3)	117.7(2)	117(2)
C(4)–C(5)	1.413(3)	1.47(2)	C(17)–C(16)–C(15)	110.3(1)	108(1)
C(6)–C(5)	1.461(2)	1.47(2)	C(19)–O(11)–C(15)	111.1(2)	113(1)
O(1)–C(14)	1.355(2)	1.36(2)	C(2)–C(15)–C(16)	114.6(2)	115(2)
O(2)–C(3)	1.360(3)	1.36(2)	C(3)–C(2)–C(15)	123.7(4)	120(2)
O(3)–C(5)	1.275(2)	1.22(2)	O(3)–C(5)–C(4)	121.9(2)	121(2)
O(4)–C(8)	1.358(2)	1.36(2)	O(3)–C(5)–C(6)	120.7(2)	120(3)
O(5)–C(9)	1.358(2)	1.36(2)	O(11)–C(15)–C(2)	109.5(1)	109(2)
O(6)–C(11)	1.366(2)	1.38(3)			
O(6)–C(12)	1.362(2)	1.38(1)			

Table 3. *Hydrogen-Bonding Angles* [°] *and Distances* [Å] *of Mangiferin* (**1**). D = H-donor and A = H-acceptor.

D–H···A[a])	D–H	H···A	D···A	D–H···A
O(1)–H(1)···O(w5)	0.8200	1.9300	2.745(6)	174.00
O(2)–H(2)···O(3)	0.8200	1.8300	2.571(5)	149.00
O(4)–H(4)···O(w2^{i})	0.8200	1.9000	2.710(5)	167.00
O(5)–H(5)···O(21)	0.8200	1.9000	2.666(5)	155.00
O(7)–H(7a)···O(15^{ii})	0.8200	2.0200	2.770(5)	152.00
O(7)–H(7a)···O(16^{ii})	0.8200	2.3000	2.939(5)	135.00
O(8^{iii})–H(8^{iii})···O(18)	0.8200	2.2600	2.795(5)	123.00
O(9^{iv})–H(9^{iv})···O(19)	0.8200	2.0100	2.814(5)	167.00
O(10)–H(10a)···O(13^{v})	0.8200	2.0000	2.814(5)	173.00
O(12)–H(12)···O(w3^{vi})	0.8200	1.8300	2.633(5)	164.00
O(13)–H(13a)···O(14)	0.8200	1.7500	2.495(5)	150.00
O(15)–H(15a)···O(w4^{vii})	0.8200	1.7900	2.609(5)	173.00
O(16)–H(16a)···O(10)	0.8200	1.8100	2.618(5)	169.00
O(18)–H(18a)···O(5^{vii})	0.8200	2.2800	3.040(5)	155.00
O(19)–H(19a)···O(8^{iii})	0.8200	2.0400	2.830(5)	163.00
O(20)–H(20)···O(w1^{viii})	0.8200	1.9200	2.726(5)	169.00
O(21)–H(21)···O(13^{ii})	0.8200	2.3000	3.036(5)	151.00
O(w1)–H(2w1)···O(20^{iv})	0.8500	2.0400	2.726(5)	138.00
O(w2)–H(2w2)···O(20^{iv})	0.8500	1.9500	2.729(5)	151.00
O(w3)–H(1w3)···O(7)	0.8500	1.9500	2.739(5)	155.00
O(w3)–H(2w3)···O(w1)	0.8500	2.0400	2.858(5)	160.00
O(w4)–H(1w4)···O(9^{ix})	0.8500	1.8800	2.703(5)	164.00
O(w5)–H(1w5)···O(1)	0.8500	2.2500	2.745(6)	117.00

[a]) Symmetry codes: $i = x-1,y,z-1$; $ii = x-1,y,z$; $iii = x-1,y+1,z+1$; $iv = x,y+1,z+1$; $v = x,y+1,z$; $vi = x,y,z-1$; $vii = x+1,y,z$; $viii = x,y-1,z-1$; $ix = x,y-1,z$.

molecular packing of **1** in which the molecules in the crystal are held together by six symmetry-independent intermolecular H-bonds involving O(7)–H(7a)···O(15^{ii}), O(7)–H(7a)···O(16^{ii}), O(5)–H(5)···O(21), O(21)–H(21)···O(13^{ii}), O(18)–H(18a)···O(5^{vii}), and O(16)–H(16a)···O(10) (symmetry codes as in *Fig. 3* and *Table 3*) to form a 1D chain along the [100] direction. *Fig. 3* shows also that molecule **1a** and **1b** are linked together in a 'head-to-tail' manner forming a liner infinite ABABAB ribbon along the [100] direction. The sugar O(7)–H(7a) group acts as intermolecular bifurcated H-bond donor to two OH groups present at ring *A′* (O(15) and O(16)), whereas the O(18)–H(18a) one acts as H-bond donor only to O(5) at ring *A*. Analyzing the packing stabilization in this direction, the O(5)–H(5), O(16)–H(16a), and O(21)–H(21) act either as H-bond donor or receptor, whereas O(10) acts only as H-bond acceptor.

It is important to emphasise that the fact that molecules **1a** and **1b** are found in different conformations (torsion on the D-glucopyranose moiety and on the OH groups O(10) and O(21) as well) in the solid state is the result of crystal-packing noncovalent-interaction forces such as those arising from hindrance effects, *van der Waals* interactions, and mainly H-bonds. In the case of **1**; the latter represent the dominant interaction. *Fig. 4* shows that the parallel chains along [100] form an infinite planar

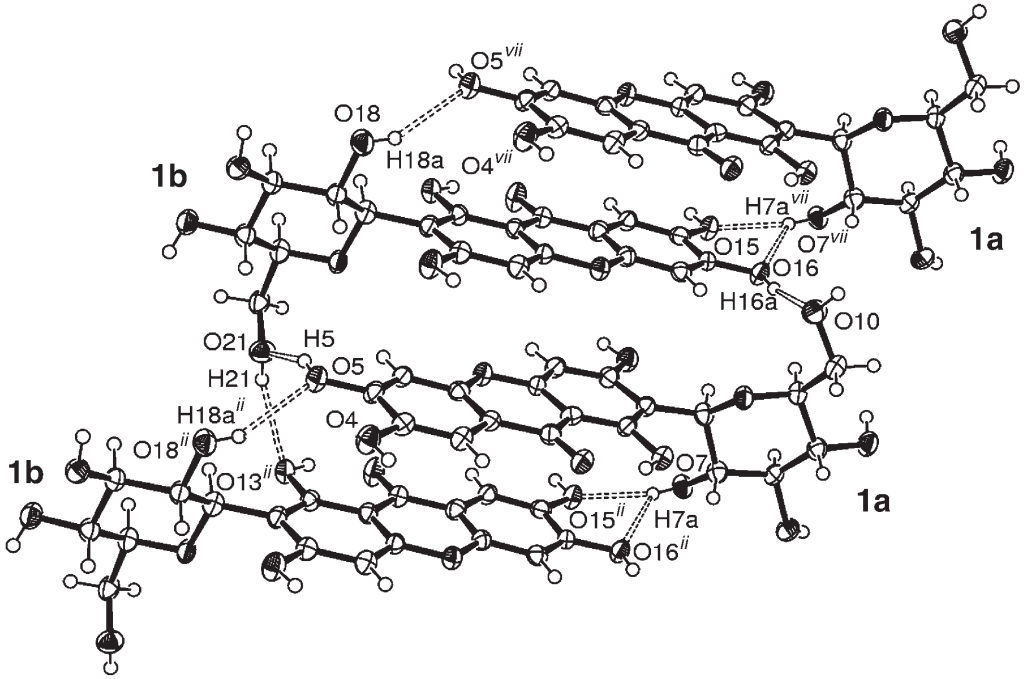

Fig. 3. *Crystal packing of mangiferin* (**1**) *along the [100] direction showing the 1D infinite framework stabilized by H-bonds.* Double-dotted lines represent H-bonds. Symmetry codes: *ii* = $x-1,y,z$; *vii* = $x+1,y,z$.

framework parallel to the (011) plane, connected by three other weak intermolecular H-bonds: O(19)–H(19a)···O(8^{iii}), O(8^{iii})–H($8a^{iii}$)···O(18), O(9^{iv})–H(9^{iv})···O(19) (symmetry codes as in *Fig. 4* and *Table 3*).

Finally, the planes are also linked to one another along [0 – 11] by another H-bond involving O(10)–H(10a)···O(13^{v}) as shown in *Fig. 5*. The result is an extended three-dimensional supramolecular assembly mediated mainly by O–H···O bonding. An interesting observation is that both the O(10)–H(10a) and O(21)–H(21) group act as H-bond donors to O(13), *i.e.*, the O(21)–H(21)···O(13) interaction stabilizes the packing along [100], whereas the O(10)–H(10a)···O(13) one is involved in the interplanar stacking stabilization along [010]. This differing role played in the buildup of the noncovalent structure by packing stabilization explains the variation in the dihedral angles observed in the D-glucopyranose moiety for the O(11)–C(19)–C(20)–O(10) and O(22)–C(39)–C(40)–O(21) moieties when molecules **1a** and **1b** are compared (see *Figs. 1*, *2*, and *5*).

Beyond the ten H-bonds involving the OH groups present in the two independent molecules **1a** and **1b**, the packing is also stabilized by the five crystallization H_2O molecules (OW) present in the structure. All of them act either as H-bond donor or acceptor as shown in *Table 3*. *Fig. 6* shows the H_2O positions in the crystal structure, which are filling two infinite cavities along the *a* axis of the unit cell. It is observed that

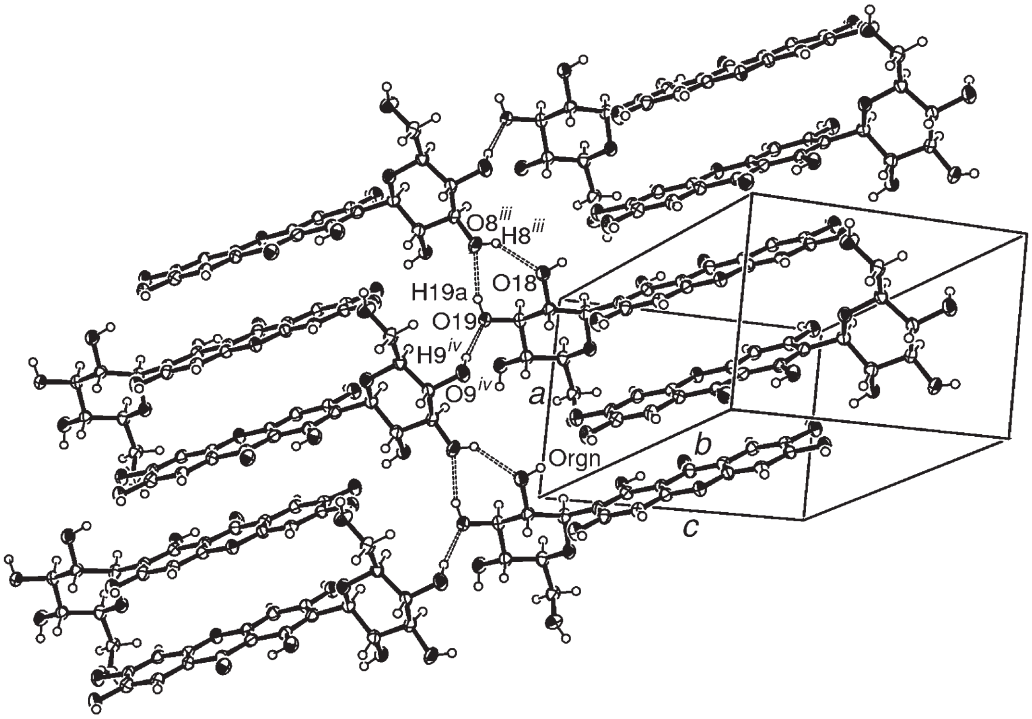

Fig. 4. *Crystal packing of mangiferin* (**1**) *parallel to the (011) plane showing the 2D infinite framework stabilized by H-bonds.* Double-dotted lines represent H-bonds. Symmetry codes: *iii* = $x + 1, y - 1, z - 1$; *iv* = $x, y - 1, z - 1$.

H_2O molecules are involved mainly in the stabilization of the interplanar staking along the [010] direction.

Conclusions. – The single-crystal X-ray-diffraction experiment determined unambiguously the crystal structure and molecular geometry of mangiferin (**1**). In addition, intra- and intermolecular H-bonds were identified, improving the understanding of geometry, localization, and chemical properties.

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Experimental Part

Extraction of Mangiferin (=*2-β-*D*-Glucopyranosyl-1,3,6,7-tetrahydroxy-9*H*-xanthen-9-one*; **1**). The stem bark of *M. indica* was milled and percolated with 50% EtOH/H_2O to give a yellow precipitate which was separated by vacuum filtration: yellow powder. This powder was extracted with 80% EtOH/H_2O and crystallized in EtOH/H_2O: pure **1**. The substance was then analyzed by the usual methods intending to verify its purity. Data: similar to the reported ones [22].

X-Ray Crystallography. The powdered yellow solid obtained after purification was slowly recrystallized at r.t. in 50% EtOH/H_2O: yellow prism single crystals. A suitably sized clear crystal of *ca.*

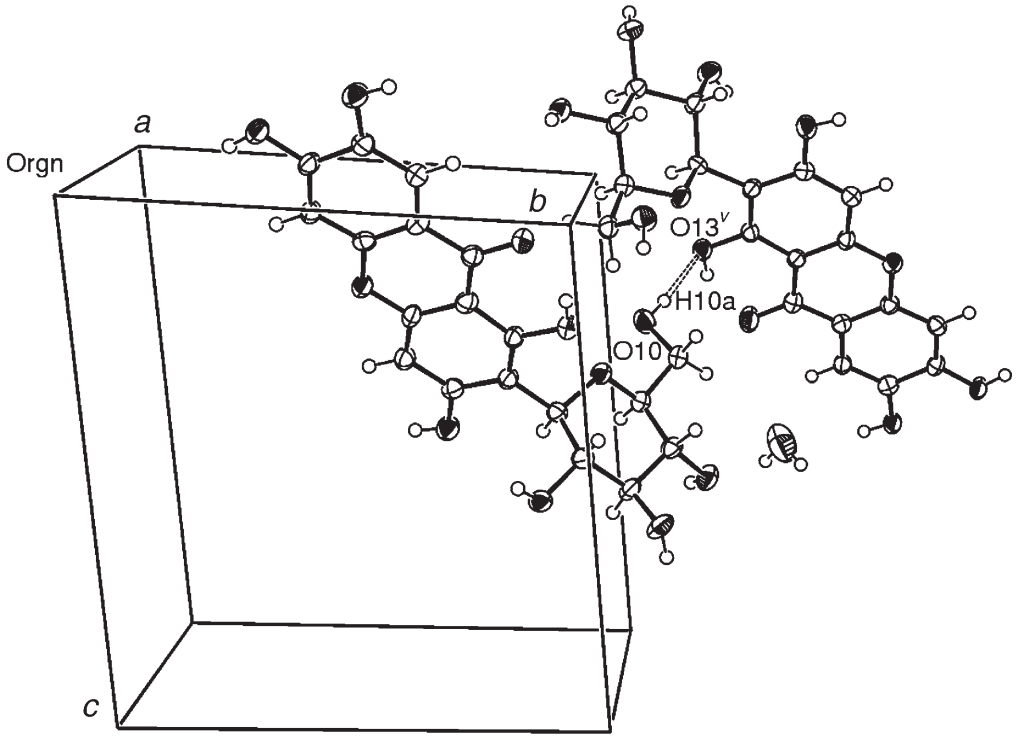

Fig. 5. *Crystal packing of mangiferin* (**1**) *along the [0 – 11] direction showing that the planes parallel to (011) are linked.* Double-dotted lines represent H-bonds. Symmetry code: $v = x,y+1,z$.

$0.06 \times 0.07 \times 0.27$ mm was selected after inspection with a stereoscopic magnifying glass by polarized light. The X-ray diffraction data were collected with MoK_α radiation (λ 0.71073 Å) from a graphite monochromator by using a *Kappa-CCD-Enraf-Nonius* X-ray diffractometer equipped with a low-temp. *Oxford Cryosystem* that allows measurements between 80 and 300 K. The software Collect [23] and Scalepack [24] were used for cell refinement. A total of 11605 *Bragg* reflections were measured to a maximum 2θ of 50.00°. Data reduction was carried out with the software Denzo-SMN, Scalepack, and XdisplayF for visual representation of data. No significant absorption effect ($\mu = 0.141$ mm^{-1}) was revealed, so no absorption correction was applied.

The crystal structure was determined by direct methods with the SHELXS-97 program [25], and the model was refined by full-matrix least-squares procedures on F^2 with SHELXL-97 [26].

The non-H-atoms were refined with anisotropic thermal parameters. All H-atoms at C-atoms were positioned in space and were refined with fixed individual displacement parameters by using the SHELXL-97 riding model. All OH H-atoms were located by difference *Fourier* synthesis and were set as isotropic. The H_2O H-atoms were positioned by the method of *Nardelli* [27] and were kept as rigid group together with its respective O-atoms during the subsequent refinements. The *Flack* parameter was not refined during X-ray crystallographic analysis: The most electron-rich atom is the O-atom, which does not have an anomalous scattering large enough (using MoK_α radiation) to permit determination of the enantiomer present by X-ray diffraction. Therefore, *Friedel* pairs were averaged before refinement, which justify the poor relationship reflection/parameters. Crystal, collection, and structure refinement

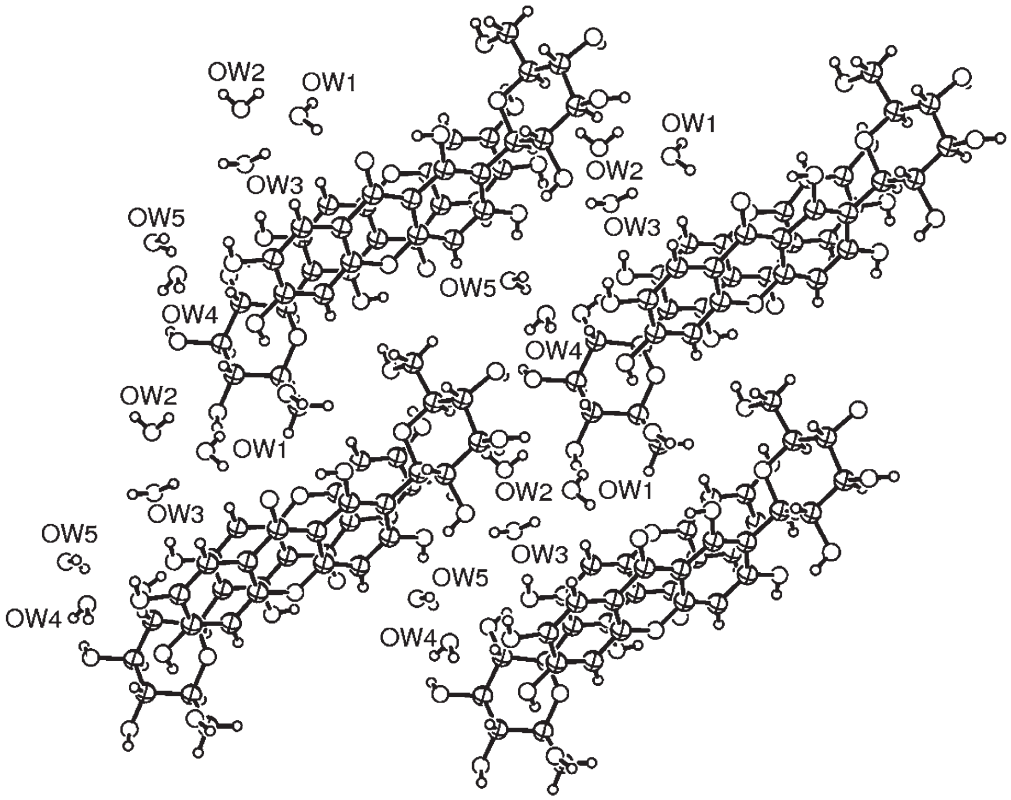

Fig. 6. *Crystal packing of mangiferin* (**1**) *projected onto the* bc *plane showing the molecules* **1a** *and* **1b** *and the position of the five crystallization* H_2O *molecules*

data are summarized in *Table 1*. The program WinGX [28] was used to prepare materials for publication. The programs MERCURY [21] and ORTEP-3 [16] were used to generate the molecular graphics.

CCDC-649851 contains the supplementary data for this paper. These data can be obtained, free of charge, *via* www.ccdc.cam.ac.uk/data_request/cif.

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