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Three ginkgolide hydrates from *Ginkgo biloba* L.: ginkgolide A monohydrate, ginkgolide C sesquihydrate and ginkgolide J dihydrate, all determined at 120 K

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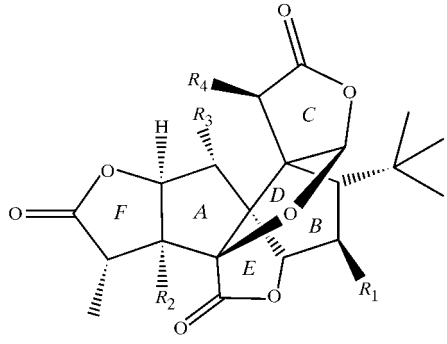
A low-temperature structure of ginkgolide A monohydrate, (1*R*,3*S*,3a*S*,4*R*,6a*R*,7a*R*,7b*R*,8*S*,10a*S*,11a*S*)-3-(1,1-dimethylethyl)-hexahydro-4,7b-dihydroxy-8-methyl-9*H*-1,7a-epoxymethano-1*H*,6a*H*-cyclopenta[*c*]furo[2,3-*b*]furo[3′,2′:3,4]cyclopenta[1,2-*d*]furan-5,9,12(4*H*)-trione monohydrate, $C_{20}H_{24}O_9\cdot H_2O$, obtained from Mo $K\alpha$ data, is a factor of three more precise than the previous room-temperature determination. A refinement of the ginkgolide A monohydrate structure with Cu $K\alpha$ data has allowed the assignment of the absolute configuration of the series of compounds. Ginkgolide C sesquihydrate, (1*S*,2*R*,3*S*,3a*S*,4*R*,6a*R*,7a*R*,7b*R*,8*S*,10a*S*,11*S*,11a*R*)-3-(1,1-dimethylethyl)-hexahydro-2,4,7b,11-tetrahydroxy-8-methyl-9*H*-1,7a-epoxymethano-1*H*,6a*H*-cyclopenta[*c*]furo[2,3-*b*]furo[3′,2′:3,4]cyclopenta[1,2-*d*]furan-5,9,12-(4*H*)-trione sesquihydrate, $C_{20}H_{24}O_{11}\cdot 1.5H_2O$, has two independent diterpene molecules, both of which exhibit intramolecular hydrogen bonding between OH groups. Ginkgolide J dihydrate, (1*S*,2*R*,3*S*,3a*S*,4*R*,6a*R*,7a*R*,7b*R*,8*S*,10a*S*,11a*S*)-3-(1,1-dimethylethyl)-hexahydro-2,4,7b-trihydroxy-8-methyl-9*H*-1,7a-epoxymethano-1*H*,6a*H*-cyclopenta[*c*]furo[2,3-*b*]-furo[3′,2′:3,4]cyclopenta[1,2-*d*]furan-5,9,12(4*H*)-trione dihydrate, $C_{20}H_{24}O_{10}\cdot 2H_2O$, has the same basic skeleton as the other ginkgolides, with its three OH groups having the same configurations as those in ginkgolide C. The conformations of the six five-membered rings are quite similar across ginkgolides A–C and J, except for the *A* and *F* rings of ginkgolide A.

Comment

The highly oxygenated diterpene trilactone-type caged molecules ginkgolide A, B, C and J were isolated from standardized extracts of *Ginkgo biloba* L. and were characterized by detailed high-field two-dimensional NMR studies. They are the major biochemical markers of *Ginkgo biloba*, which is considered an important vascular and neurological botanical and displays potent antagonistic activity against platelet-activating factor (PAF, PAF/acether, AGEPC) (Braquet, 1988; Braquet & Godfroid, 1986; Van Beek *et al.*, 1998).



	R_1	R_2	R_3	R_4
Ginkgolide A, (I)	H	OH	H	OH
Ginkgolide B, (II)	H	OH	OH	OH
Ginkgolide C, (III)	OH	OH	OH	OH
Ginkgolide J, (IV)	OH	OH	H	OH

Out of five reported ginkgolides (Roumestand *et al.*, 1989; Van Beek & Lankhorst, 1996; Weinges *et al.*, 1987), the relative stereochemistries of ginkgolide A, B and C were previously determined by X-ray crystallography (Dupont *et al.*, 1986; Sakabe *et al.*, 1967; Sbit *et al.*, 1987) in a study of the monohydrates of (I) and (II), and the ethanol 1.5-hydrate of (III). In order to prove conclusively the configurations of all 11 asymmetric centers of ginkgolide J, (IV), and especially to ascertain how it correlates with other ginkgolides [(I) and (III)], crystal structure determinations were undertaken. Crystallization from undried solvents yielded hydrates, namely ginkgolide A monohydrate ($C_{20}H_{24}O_9\cdot H_2O$), ginkgolide C sesquihydrate ($C_{20}H_{24}O_{11}\cdot 1.5H_2O$), and ginkgolide J dihy-

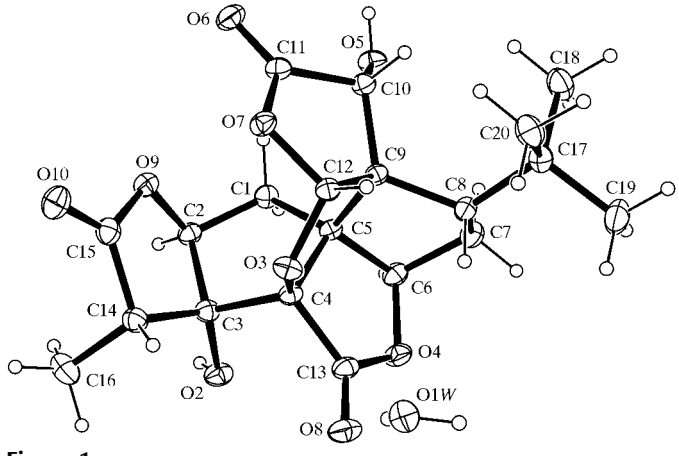


Figure 1
View of ginkgolide A monohydrate showing the atom-numbering scheme and ellipsoids at the 50% probability level.

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drate ($C_{20}H_{24}O_{10}\cdot 2H_2O$), which also allowed direct determination of their comparative caged configurations. The structures reported herein are in agreement with the tentative assignments made using NMR methods.

Our low-temperature determination of ginkgolide A monohydrate, (I).H_2O, with Mo $K\alpha$ data confirms the earlier results of Sbit *et al.* (1987) and represents a substantial improvement in precision over their room-temperature structure. We have also measured room-temperature data with Cu $K\alpha$ radiation at 296 K, and refinement using 3776 reflections (1439 Friedel pairs) yielded $R = 0.028$ and a Flack (1983) parameter of -0.02 (12). We have adopted this absolute structure for all three compounds reported herein, and it is consistent with the absolute configuration reported by Sakabe *et al.* (1967) for the *p*-bromobenzoate ester at C3. Their absolute configuration, determined from the diethanol solvate, was somewhat in doubt because of a rather high R value (0.190) from visually estimated film data with no absorption correction. Our room-temperature cell dimensions for the monohydrate are $a = 8.992$ (2), $b = 12.438$ (2), $c = 17.819$ (3) Å and $V = 1992.9$ (6) Å^3, based on 25 reflections having $22.8 < \theta < 43.1^\circ$, measured on an Enraf–Nonius CAD-4 diffractometer. The results of the Cu $K\alpha$ refinement have been deposited as supplementary data.

Our low-temperature determination of the structure of ginkgolide C as the sesquihydrate, (III)·1.5H_2O, represents an increase in precision by a factor of four over that of Sbit *et al.* (1987) for the sesquihydrate monoethanol solvate at room temperature. For their structure, as well as ours, $Z' = 2$. In both independent molecules of our structure, there is an intramolecular hydrogen bond between OH groups O1 and O5, with O1 as donor. The conformations of all the OH groups agree well between the *A* and *B* molecules, despite the differences in intermolecular hydrogen bonding (Table 3). In the structure of Sbit *et al.*, the intramolecular hydrogen bond is reported to have O5 as the donor and O1 as the acceptor, although they were unable to locate all hydroxy H atoms. In the present structure, there are three independent water molecules, two of which are disordered into pairs of sites. O2*W* occupies sites of occupancy 0.72 (3) and 0.28 (3), separated by 0.641 (17) Å, while O3*W* occupies sites of equal occupancy [0.494 (10) and 0.506 (10)], separated by 0.849 (4) Å. All five water sites accept hydrogen bonds from ginkgolide C molecules; hydrogen bonding between water molecules is less unambiguous because of the inability to locate the water H atoms.

The structure of the trihydroxy compound ginkgolide J, (IV), for which no crystal structure has been previously reported, is shown in Fig. 3 as the dihydrate, *i.e.* (IV)·2H_2O. The configurations of all the asymmetric centers in the basic ginkgolide skeleton are shown to agree with those of ginkgolides A, B, and C. The OH groups have been confirmed to lie at C3, C7, and C10, with the same configurations as the OH groups at those positions in ginkgolide C.

The conformations of the six five-membered rings (*A*–*F* in the *Scheme*) of the ginkgolide skeleton have been discussed by Dupont *et al.* (1986) and Sbit *et al.* (1987). They found that the skeleton was fairly rigid, with the conformations of rings *B*–*E* differing little in ginkgolides A, B, and C, while those of the *A* and *F* rings of ginkgolide A differed from the pattern. They suggested that the anomalous *A*- and *F*-ring conformations of ginkgolide A are related to hydrogen bonding, as ginkgolide A carries no OH group at C1, while ginkgolides B and C do. Our results for the endocyclic torsion angles of the five-membered rings are given in Table 1. We also note little variation (11° or less) across the entire spectrum of torsion angles, except for the *A* and *F* rings of ginkgolide A. Notably, the conformations of the *A* and *F* rings in ginkgolide J, which, like ginkgolide A, carries no OH group at C1, fit the normal pattern.

The hydrogen bonding to the two independent molecules in ginkgolide C sesquihydrate differs (Table 3), which allows direct observation of the conformational differences caused by differences in intermolecular hydrogen bonding. The differences in torsion angles (Table 1) are small, with the overall

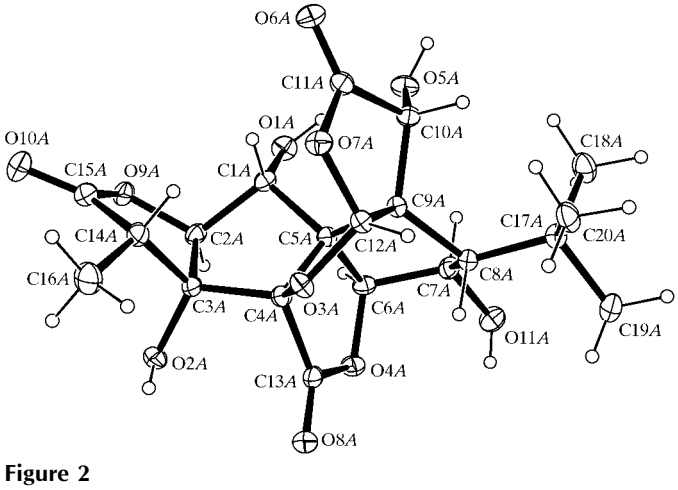

Figure 2
View of one of the two independent molecules of ginkgolide C sesquihydrate showing the atom-numbering scheme and ellipsoids at the 50% probability level.

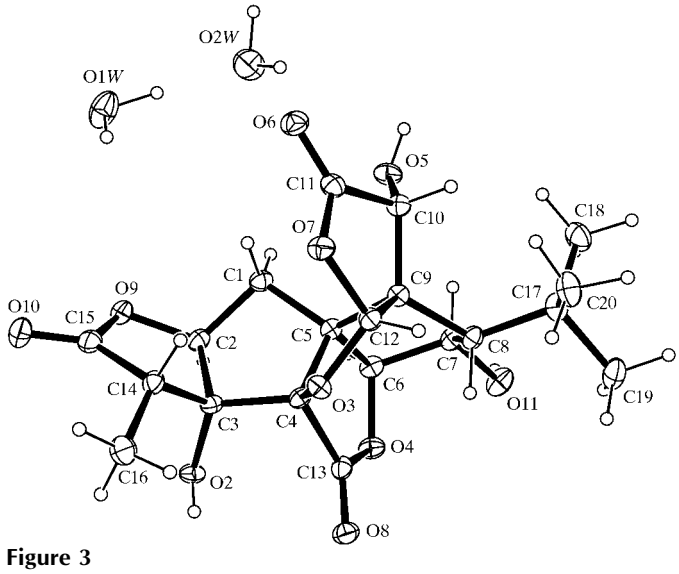

Figure 3
View of ginkgolide J dihydrate showing the atom-numbering scheme and ellipsoids at the 50% probability level.

mean deviation among 30 pairs of torsion angles being only 2.7°. The *A* ring shows the largest deviation, with a mean of 5.1°, with the other rings having mean deviations as follows: *B* 1.6, *C* 1.0, *D* 2.9, *E* 4.1 and *F* 1.2°.

Experimental

Ginkgolides A, B, C and J were isolated in large scale from a standardized extract (Neutrasource Inc., San Carlos, CA) of *Ginkgo biloba* L. (Ginkgoaceae) using a sodium acetate-impregnated silica-gel chromatographic technique (Van Beek & Lelyveld, 1997) combined with a pre-separation by normal column chromatography. Ginkgolide A, (I), was recrystallized from methanol (MeOH) as plates [m.p. 583 K; $[\alpha]_D$ −38 (*c* = 0.069, dioxane) and −56° (*c*, 0.06, MeOH)], ginkgolide C, (III), was recrystallized as plates from acetone/*n*-hexane [m.p. 558 K; $[\alpha]_D$ −16° (*c* = 0.055, dioxane)] and ginkgolide J, (IV), was recrystallized as needles from acetone/*n*-hexane [m.p. 563 K; $[\alpha]_D$ +1.6° (*c*, 0.061, dioxane)]. The initial physical and NMR data, recorded at 500 (^{1}H) and 125 (^{13}C) MHz using a Bruker Avance DRX-500 instrument, were in agreement with those reported in the literature (Roumestand *et al.*, 1989; Van Beek & Lankhorst, 1996; Weinges *et al.*, 1987).

Compound (I)·H_2O

Crystal data

$C_{20}H_{24}O_9\cdot H_2O$
M_r = 426.41
Orthorhombic, $P2_12_12_1$
a = 8.931 (2) Å
b = 12.338 (2) Å
c = 17.779 (3) Å
V = 1959.2 (6) Å^3
Z = 4
D_x = 1.446 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 19 931 reflections
θ = 2.5–33.2°
μ = 0.12 mm^{-1}
T = 120 K
Plate, colorless
0.48 × 0.37 × 0.35 mm

Data collection

KappaCCD diffractometer (with an Oxford Cryosystems Cryostream cooler)
ω scans with κ offsets
19 931 measured reflections
4144 independent reflections
3936 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 33.2°
h = −13 → 13
k = −18 → 18
l = −27 → 27

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.083
S = 1.06
4144 reflections
283 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0419P)^2 + 0.3730P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Compound (III)·1.5H_2O

Crystal data

$C_{20}H_{24}O_{11}\cdot 1.5H_2O$
M_r = 467.42
Monoclinic, $P2_1$
a = 7.4945 (15) Å
b = 12.973 (3) Å
c = 20.934 (4) Å
β = 91.00 (2)°
V = 2035.0 (7) Å^3
Z = 4
D_x = 1.526 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 5587 reflections
θ = 2.5–29.6°
μ = 0.13 mm^{-1}
T = 120 K
Plate, colorless
0.47 × 0.27 × 0.25 mm

Table 1
Hydrogen-bonding geometry (Å, °) for (I)·H_2O.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2O···O1*W*^{i}	0.84	1.89	2.694 (2)	160
O5—H5O···O1*W*^{ii}	0.84	1.93	2.764 (2)	172
O1*W*—H1*W*···O8	0.86 (2)	1.98 (2)	2.831 (2)	175 (2)
O1*W*—H2*W*···O10^{iii}	0.83 (2)	1.90 (2)	2.711 (2)	169 (2)

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

Table 2
Endocyclic torsion angles (°) in the title ginkgolides A, C, and J hydrates.

	Ginkgolide A	Ginkgolide C Molecule *A*	Ginkgolide C Molecule *B*	Ginkgolide J
Ring *A*				
C1—C2—C3—C4	27.84 (12)	−32.96 (16)	−29.01 (17)	−23.50 (16)
C2—C3—C4—C5	−24.67 (12)	11.07 (16)	11.93 (17)	2.51 (16)
C3—C4—C5—C1	12.57 (12)	14.59 (16)	9.08 (17)	19.07 (16)
C4—C5—C1—C2	4.88 (12)	−34.51 (15)	−26.55 (16)	−33.15 (16)
C5—C1—C2—C3	−20.79 (12)	42.12 (15)	34.63 (17)	35.23 (16)
Ring *B*				
C9—C5—C6—C7	−13.21 (12)	−13.92 (16)	−15.29 (16)	−13.94 (17)
C5—C6—C7—C8	35.47 (12)	37.16 (16)	39.63 (15)	38.53 (16)
C6—C7—C8—C9	−43.50 (12)	−45.49 (15)	−48.01 (15)	−47.34 (15)
C7—C8—C9—C5	34.67 (11)	36.04 (14)	37.28 (14)	38.15 (15)
C8—C9—C5—C6	−13.49 (11)	−14.16 (15)	−14.18 (15)	−15.47 (16)
Ring *C*				
C12—C9—C10—C11	−32.36 (11)	−29.85 (15)	−31.09 (15)	−29.39 (15)
C9—C10—C11—O7	29.50 (12)	23.76 (16)	24.11 (16)	24.32 (17)
C10—C11—O7—C12	−13.07 (13)	−6.51 (17)	−6.06 (17)	−8.16 (18)
C11—O7—C12—C9	−8.61 (12)	−13.28 (16)	−14.61 (16)	−11.50 (17)
O7—C12—C9—C10	25.99 (11)	26.92 (15)	28.47 (15)	25.70 (16)
Ring *D*				
O3—C4—C5—C9	16.88 (11)	17.50 (16)	14.16 (16)	22.38 (15)
C4—C5—C9—C12	−23.49 (10)	−24.43 (15)	−23.63 (15)	−27.40 (15)
C5—C9—C12—O3	23.62 (11)	25.12 (16)	26.92 (16)	24.92 (16)
C9—C12—O3—C4	−14.06 (12)	−15.34 (17)	−19.36 (17)	−11.91 (17)
C5—C4—O3—C12	−2.29 (12)	−1.92 (17)	2.69 (18)	−7.17 (16)
Ring *E*				
C13—C4—C5—C6	10.27 (11)	12.25 (16)	9.70 (16)	13.59 (16)
C4—C5—C6—O4	−6.06 (12)	−8.27 (16)	−9.29 (16)	−6.70 (16)
C5—C6—O4—C13	−1.17 (13)	0.33 (17)	5.30 (17)	−3.88 (18)
C6—O4—C13—C4	8.17 (13)	7.94 (18)	1.15 (18)	13.12 (18)
O4—C13—C4—C5	−11.80 (13)	−12.80 (17)	−7.07 (18)	−17.02 (18)
Ring *F*				
O9—C2—C3—C14	29.98 (11)	−31.97 (15)	−30.72 (16)	−25.46 (16)
C2—C3—C14—C15	−24.93 (11)	32.40 (15)	30.85 (15)	29.90 (16)
C3—C14—C15—O9	11.65 (13)	−24.07 (18)	−22.19 (18)	−26.00 (17)
C14—C15—O9—C2	7.87 (13)	3.89 (19)	2.70 (19)	10.22 (18)
C15—O9—C2—C3	−23.99 (12)	18.37 (17)	18.23 (17)	10.03 (17)

Data collection

KappaCCD diffractometer (with an Oxford Cryosystems Cryostream cooler)
ω scans with κ offsets
22 429 measured reflections
5921 independent reflections
5752 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 29.6°
h = −10 → 10
k = −18 → 13
l = −29 → 29

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.086
S = 1.05
5921 reflections
622 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0549P)^2 + 0.4304P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 3
Hydrogen-bonding geometry (Å, °) for (III)·1.5H_2O.

D—H···A	D—H	H···A	D···A	D—H···A
O1A—H1HA···O5A	0.84	2.04	2.749 (2)	142
O1A—H1HA···O4A^{i}	0.84	2.58	3.141 (2)	126
O2A—H2HA···O1W^{ii}	0.84	1.99	2.809 (2)	164
O5A—H5HA···O3W	0.84	2.09	2.839 (3)	148
O5A—H5HA···O3W'	0.84	1.84	2.664 (3)	169
O11A—H11A···O1A^{iii}	0.84	2.29	2.991 (2)	141
O1B—H1HB···O5B	0.84	2.30	2.911 (2)	131
O1B—H1HB···O8B^{iv}	0.84	2.41	3.052 (2)	133
O1B—H1HB···O4B^{iv}	0.84	2.41	2.901 (2)	118
O2B—H2HB···O10A^{ii}	0.84	2.12	2.924 (2)	161
O5B—H5HB···O2W^{iv}	0.84	1.98	2.786 (3)	162
O5B—H5HB···O2W'^{iv}	0.84	2.07	2.821 (8)	149
O11B—H11B···O9B^{v}	0.84	2.37	2.943 (2)	126

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

Compound (IV)·2H_2O

Crystal data

$C_{20}H_{24}O_{10}\cdot 2H_2O$
M_r = 460.42
Orthorhombic, $P2_12_12_1$
a = 11.403 (2) Å
b = 12.950 (3) Å
c = 13.646 (3) Å
V = 2015.0 (6) Å^3
Z = 4
D_x = 1.518 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 14 293 reflections
θ = 2.8–27.9°
μ = 0.13 mm^{-1}
T = 120 K
Needle, colorless
0.25 × 0.22 × 0.17 mm

Data collection

KappaCCD diffractometer (with Oxford Cryosystems Cryostream cooler)
ω scans with κ offsets
14 293 measured reflections
2694 independent reflections
2586 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 27.9°
$h = -14 \rightarrow 15$
$k = -16 \rightarrow 17$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.074
S = 1.03
2694 reflections
309 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0386P)^2 + 0.6511P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.013 (3)

Population parameters of the disordered water sites in ginkgolide C sesquihydrate were refined, with each pair (O2W/O2W' and O3W/O3W') constrained to sum to unity. H atoms were placed in calculated positions, with C—H bond distances in the range 0.96–1.00 Å and thereafter treated as riding. A torsional parameter was refined for each methyl group. OH and H_2O H atoms were placed from difference maps and refined as follows: for ginkgolide A monohydrate and ginkgolide J dihydrate, hydroxy O—H distances were constrained to be 0.82 Å and torsional parameters were refined, while water H-atom positions were refined individually; for ginkgolide C sesquihydrate, hydroxy O—H distances were constrained to be 0.84 Å and torsional parameters were refined, while water H atoms could not be unambiguously placed. For all compounds, U_{iso}(H) = 1.2U_{eq} of the attached atom (1.5 for methyl groups and H atoms on O atoms).

Table 4
Hydrogen-bonding geometry (Å, °) for (IV)·2H_2O.

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2O···O6^{i}	0.84	1.94	2.709 (2)	152
O5—H5O···O1W^{ii}	0.84	1.83	2.659 (2)	167
O11—H11O···O2^{iii}	0.84	2.02	2.810 (2)	156
O1W—H1W···O2W	0.98 (3)	1.76 (3)	2.745 (2)	178 (3)
O1W—H2W···O8^{iv}	0.87 (3)	2.03 (3)	2.896 (2)	175 (3)
O2W—H3W···O10^{ii}	0.96 (3)	1.89 (3)	2.798 (2)	158 (3)
O2W—H3W···O9^{ii}	0.96 (3)	2.67 (3)	3.514 (2)	147 (2)
O2W—H4W···O6	0.81 (3)	2.46 (3)	3.123 (2)	140 (3)
O2W—H4W···O1W^{ii}	0.81 (3)	2.64 (3)	3.263 (2)	135 (3)
O2W—H4W···O5	0.81 (3)	2.67 (3)	3.172 (2)	122 (3)

Symmetry codes: (i) $\frac{3}{2}-x, 1-y, z-\frac{1}{2}$; (ii) $x-\frac{1}{2}, \frac{1}{2}-y, 1-z$; (iii) $x-\frac{1}{2}, \frac{1}{2}-y, -z$; (iv) $\frac{3}{2}-x, 1-y, \frac{1}{2}+z$.

For all three title compounds, data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1627). Services for accessing these data are described at the back of the journal.

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