

Comparison of conventional and synchrotron X-ray structure determinations of the adduct 1,2,4,5-tetrahydroxybenzene–2,5-dihydroxy-1,4-benzoquinone (1/1) and a conventional X-ray structure determination of 1,2,4,5-tetrahydroxybenzene monohydrate

Paul G. Jene, Christopher G. Pernin and James A. Ibers*

Department of Chemistry, Northwestern University, 2145 Sheridan Road, Evanston, IL 60208-3113, USA

Correspondence e-mail: ibers@chem.nwu.edu

Received 20 November 2000

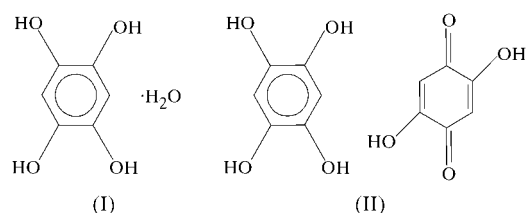
Accepted 16 February 2001

The X-ray structure of 1,2,4,5-tetrahydroxybenzene (benzene-1,2,4,5-tetrol) monohydrate, $C_6H_6O_4 \cdot H_2O$, (I), reveals columns of 1,2,4,5-tetrahydroxybenzene parallel to the *b* axis that are separated by 3.364 (12) and 3.453 (11) Å. Molecules in adjacent columns are tilted relative to each other by 27.78 (8)°. Water molecules fill the channels between the columns and are involved in hydrogen-bonding interactions with the 1,2,4,5-tetrahydroxybenzene molecules. The crystal structure of the adduct 1,2,4,5-tetrahydroxybenzene–2,5-dihydroxy-1,4-benzoquinone (1/1), $C_6H_6O_4 \cdot C_6H_4O_4$, (II), reveals alternating molecules of 1,2,4,5-tetrahydroxybenzene and 2,5-dihydroxy-1,4-benzoquinone (both lying on inversion centers), and a zigzag hydrogen-bonded network connecting molecules in three dimensions. For compound (II), the conventional X-ray determination, (II*a*), is in very good agreement with the synchrotron X-ray determination, (II*b*). When differences in data collection temperatures are taken into account, even the displacement parameters are in very good agreement.

Comment

Derivatized porphyrins have found extensive use as models for the heme active sites of myoglobin and hemoglobin (Mometeau & Reed, 1994). Compound (I), 1,2,4,5-tetrahydroxybenzene monohydrate, was prepared as a precursor in the synthesis of elaborated porphyrins containing a benzene 'cap' linked to the porphyrin by ethoxy linkages (Ma *et al.*, 1993; Johnson *et al.*, 1996; Slebodnick *et al.*, 1996; Jene *et al.*, 1999; Jene & Ibers, 2000). Compound (I) forms pale-pink crystals. The synthesis of (I) is sensitive to the initial reaction conditions and yields products that vary from these crystals to

a gray-black amorphous material. All products work in the capped porphyrin synthesis. To understand these products better, we determined the structures of (I) and of the adduct 1,2,4,5-tetrahydroxybenzene–2,5-dihydroxy-1,4-benzoquinone (1/1), (II). Crystals of (II) are stable and of excellent quality. Consequently, a short wavelength (0.354 Å) data set, (II*b*), was collected from a crystal of (II) on a Rigaku R-AXIS image-plate diffractometer at the DND-CAT at Sector 5 of the Advanced Photon Source (APS). Refinement of this data set provides a check on the APS system and an interesting comparison with the results from a conventional structure determination.



The asymmetric unit of compound (I) contains two half-molecules of 1,2,4,5-tetrahydroxybenzene that sit on inversion centers and two half water molecules that sit on twofold axes (Fig. 1). Selected bond lengths and angles are listed in Table 1. The benzene rings are planar by symmetry. C–C bond lengths in (I) reflect the fully delocalized nature of the benzene ring. The crystal structure is composed of columnar stacking and two-dimensional networks of hydrogen bonding (Fig. 2). Both unique molecules form separate columns parallel to the *b* axis. The first column is formed from atoms C1, C2, C3, O1, and O2 and their symmetry-generated counterparts; the second column is formed from atoms C4, C5, C6, O3, and O4 and their symmetry-generated counterparts. Molecules in different columns are tilted relative to each other by 27.78 (8)°. A ruffled two-dimensional network of hydrogen bonding exists perpendicular to the columns. Inter- and intramolecular hydrogen-bonding interactions are listed in Table 2. The two unique half water molecules, containing atoms O5 and O6, fill neighboring channels of the column structure but are oriented in opposite directions. The O atoms O5 and O6 of the half

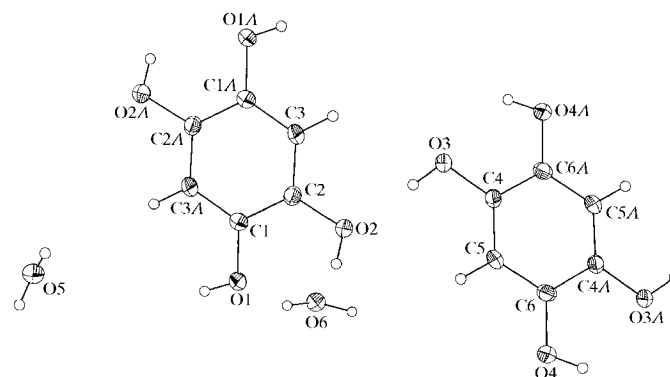


Figure 1
The molecular structure of (I), showing the atom-numbering scheme and 50% displacement ellipsoids. H atoms are drawn as small spheres of arbitrary radii.

water molecules are involved in four hydrogen bonds to four molecules of 1,2,4,5-tetrahydroxybenzene in neighboring columns.

The synthesis (Jones & Shonle, 1945) and crystal structure (Semmingsen, 1977) of 2,5-dihydroxy-1,4-benzoquinone have been known for some time. In addition, dihydroxyquinones substituted at the 3,6-positions have been prepared with fluoro (Krogh Andersen & Krogh Andersen, 1975), chloro (Krogh Andersen, 1967*a,b*), bromo (Robl, 1987), iodo (Robl & Sheldrick, 1988), and additional hydroxy (Klug, 1965) substituents.

Molecules of 1,2,4,5-tetrahydroxybenzene and 2,5-dihydroxy-1,4-benzoquinone in compound (II) (Fig. 3) are both located on inversion centers, and the benzene and quinone portions of these molecules are planar by symmetry. These two molecules are related by pseudosymmetry if the positions of the H atoms are ignored. However, the positions of these H atoms refine well when constraints are released. The molecules are tilted relative to each other by $64.00(5)^\circ$, as measured by their mean planes. The two kinds of molecules alternate to form two unique stacks parallel to the *a* axis and two unique stacks parallel to the *b* axis (Fig. 4). Metrical details are listed in Tables 3–6, and values for discussion are taken from Tables 5 and 6. The interplanar distances between molecules in the stacks are 2.895 (1) and 3.301 (1) Å along the *a* axis, and 3.115 (1) and 3.304 (1) Å along the *b* axis. There is a three-dimensional zigzag hydrogen-bonded network in the structure.

The 2,5-dihydroxy-1,4-benzoquinone component of (II) shows pronounced quinoid character, with both short and long C—O bonds and three varied C—C bond lengths (Tables 3

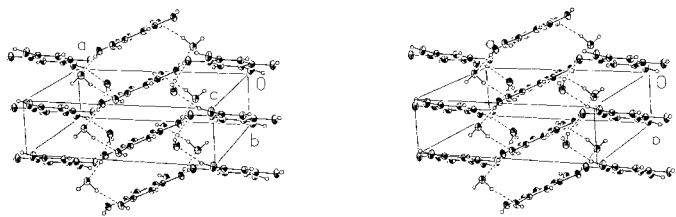


Figure 2

A stereoview of (I), showing columnar stacking, hydrogen-bonding interactions (dashed lines), and the water molecule orientation.

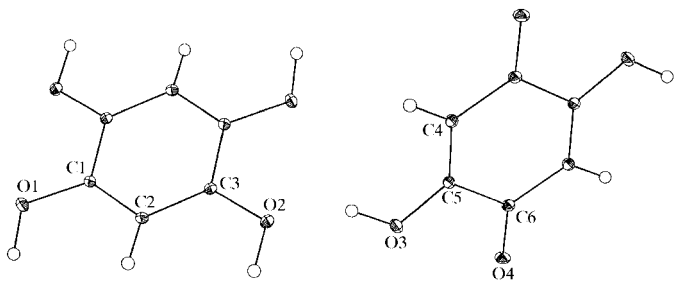


Figure 3

The molecular structure of (II) from the R-AXIS data set, *i.e.* (II*b*), showing the atom-numbering scheme and 50% displacement ellipsoids. H atoms are drawn as small spheres of arbitrary radii. Only unique atoms are labeled.

and 5). These values are similar to those reported earlier by Semmingsen (1977). The difference is an $\sim 180^\circ$ rotation of a hydroxyl group between the structures. The geometry of the 1,2,4,5-tetrahydroxybenzene component of (II) is very similar to that in (I). The exception is one hydroxyl group that is flipped by $\sim 180^\circ$ (Tables 3 and 5). This orientation precludes the possibility of intramolecular hydrogen-bonding interactions and underscores the importance of crystal-packing forces on the orientation of hydroxyl groups in compounds of this type.

For compound (II), the conventional X-ray determination, (II*a*) (Tables 3 and 4), is in very good agreement with the synchrotron X-ray determination, (II*b*) (Tables 5 and 6). There are a few possibly significant differences, *e.g.* C2—C3 [1.389 (2) Å in (II*a*) and 1.3953 (4) Å in (II*b*)], but since (II*a*) is a determination at 163 K and (II*b*) at 110 K it is possible that these arise from differences in vibrational foreshortening of the bonds. In this regard, if a linear correction is applied for the differences in temperature (Debye, 1913; Dunitz *et al.*, 1988), then generally the U_{ij} values from the two determinations agree to within about 10%. Since, in X-ray structure determinations, it is the U_{ij} values that are most sensitive to a variety of systematic errors, we find this agreement very encouraging. The standard uncertainties associated with derived quantities for (II*b*) are roughly 20% of those for (II*a*),

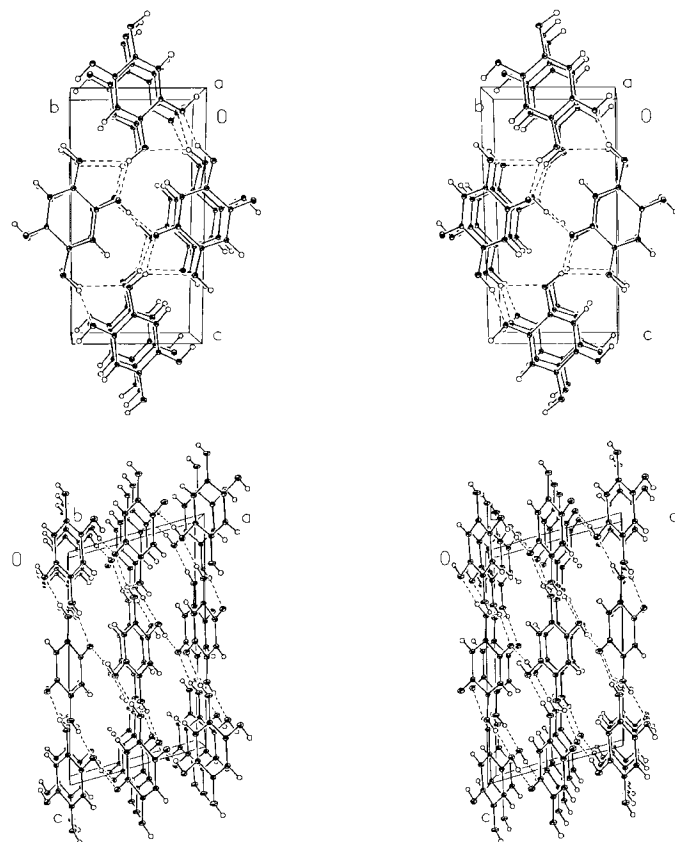


Figure 4

Stereoview of compound (II), data set (II*b*), showing columnar stacking and hydrogen-bonding interactions (dashed lines).

in part because there were 5678 independent reflections in data set (IIb) and 842 in (IIa).

Crystals of (II) are dark and shiny, in sharp contrast with crystals of (I). The dark color probably arises from a charge-transfer interaction between molecules of 1,2,4,5-tetrahydroxybenzene acting as donors and molecules of 2,5-dihydroxy-1,4-benzoquinone acting as acceptors. Chowdhury (1961) has shown that charge-transfer interactions occur between quinones and aromatic hydro-C atoms. Sakurai (1965) has proposed that the color of quinhydrone results from hydroquinone acting as the donor and quinone as the acceptor molecule. One feature of this interaction is abnormally short C...O interactions. Compound (IIb) has short C...O interactions that range from 3.097 (1) to 3.241 (1) Å. The shortest of these is shorter than the 3.2 Å value reported by Sakurai (1965). It is also likely that the gray-black material often present in the synthesis of (I) is a mixture of (I) and (II), with (II) forming by partial air oxidation in solution.

Experimental

Crystals of compound (I) were prepared according to the literature method of Anslow & Raistrick (1939) (m.p. 490–491 K). Compound (II) was prepared by allowing a small quantity of (I) to stand in D₂O. After one month, dark shiny crystals of (II) formed (m.p. > 673 K). Melting points were measured on a Mel-Temp melting point apparatus (Laboratory Instruments, Holliston, Massachusetts).

Compound (I)

Crystal data

C₆H₆O₄·H₂O
M_r = 160.12
 Monoclinic, *P*2₁/*n*
a = 13.487 (3) Å
b = 3.647 (1) Å
c = 13.506 (3) Å
 β = 102.33 (3)°
V = 649.1 (2) Å³
Z = 4

D_x = 1.639 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 623 reflections
 θ = 1.9–28.3°
 μ = 0.15 mm⁻¹
T = 203 (2) K
 Rectangular prism, pale pink
 0.5 × 0.2 × 0.2 mm

Table 1

Selected geometric parameters (Å, °) for (I).

O1–C1	1.386 (2)	C1–C3 ⁱⁱⁱ	1.384 (3)
O2–C2	1.378 (3)	C1–C2	1.392 (3)
O3–C4	1.381 (3)	C2–C3	1.391 (3)
O4–C6	1.379 (3)	C3–C1 ⁱⁱⁱ	1.384 (3)
O5–H5A ⁱ	0.80 (4)	C4–C5	1.389 (3)
O5–H5A	0.80 (4)	C4–C6 ^{iv}	1.392 (3)
O6–H6A	0.95 (5)	C5–C6	1.386 (3)
O6–H6A ⁱⁱ	0.95 (5)	C6–C4 ^{iv}	1.392 (3)
C3 ⁱⁱⁱ –C1–O1	122.50 (19)	O3–C4–C5	122.27 (19)
C3 ⁱⁱⁱ –C1–C2	119.9 (2)	O3–C4–C6 ^{iv}	117.50 (19)
O1–C1–C2	117.57 (19)	C5–C4–C6 ^{iv}	120.2 (2)
O2–C2–C3	118.15 (19)	C6–C5–C4	120.41 (19)
O2–C2–C1	122.5 (2)	O4–C6–C5	118.79 (19)
C3–C2–C1	119.4 (2)	O4–C6–C4 ^{iv}	121.84 (19)
C1 ⁱⁱⁱ –C3–C2	120.7 (2)	C5–C6–C4 ^{iv}	119.4 (2)

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

Data collection

Bruker SMART1000 CCD diffractometer
 ω scans
 3708 measured reflections
 1578 independent reflections
 1270 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.022
 θ_{\max} = 28.3°
h = -17 → 13
k = -4 → 4
l = -11 → 18
 Intensity decay: <2%

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.045
wR (*F*²) = 0.149
S = 1.22
 1578 reflections
 116 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.036P)^2 + 1.0778P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.39 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.26 \text{ e } \text{Å}^{-3}$

Table 2

Hydrogen-bonding geometry (Å, °) for (I).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
O1–H1...O4 ⁱ	0.83	1.96	2.756 (2)	160
O2–H2...O6 ⁱⁱ	0.83	1.92	2.735 (2)	166
O3–H3A...O2 ⁱⁱⁱ	0.83	1.88	2.699 (2)	169
O4–H4...O5 ^{iv}	0.83	1.96	2.767 (2)	163
O5–H5A...O3 ^v	0.77 (3)	2.07 (3)	2.826 (2)	165 (3)
O6–H6A...O1	0.95 (5)	1.91 (5)	2.832 (2)	162 (3)

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

Compound (II), data set (IIa)

Crystal data

C₆H₆O₄·C₆H₄O₄
M_r = 282.20
 Monoclinic, *P*2₁/*n*
a = 7.387 (3) Å
b = 6.060 (1) Å
c = 12.198 (2) Å
 β = 105.78 (2)°
V = 525.4 (3) Å³
Z = 2

D_x = 1.784 Mg m⁻³
 Cu *K*α radiation
 Cell parameters from 25 reflections
 θ = 30.8–38.9°
 μ = 1.34 mm⁻¹
T = 163 (2) K
 Rectangular prism, black
 0.43 × 0.15 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: analytical face-indexed (de Meulenaer & Tompa, 1965)
*T*_{min} = 0.877, *T*_{max} = 0.938
 3379 measured reflections
 842 independent reflections

753 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.021
 θ_{\max} = 63°
h = -8 → 8
k = -6 → 6
l = -14 → 14
 6 standard reflections every 100 reflections
 intensity decay: <2%

Table 3

Selected geometric parameters (Å, °) for (II), data set (IIa).

O1–C1	1.3671 (16)	C2–C3	1.3887 (19)
O2–C3	1.3742 (17)	C3–C1 ⁱ	1.394 (2)
O3–C5	1.3320 (16)	C4–C5	1.341 (2)
O4–C6	1.2355 (17)	C4–C6 ⁱⁱ	1.4395 (19)
C1–C2	1.386 (2)	C5–C6	1.505 (2)
C1–C3 ⁱ	1.394 (2)	C6–C4 ⁱⁱ	1.4395 (19)
O1–C1–C2	124.22 (12)	C5–C4–C6 ⁱⁱ	120.36 (12)
O1–C1–C3 ⁱ	116.06 (12)	O3–C5–C4	126.65 (13)
C2–C1–C3 ⁱ	119.71 (13)	O3–C5–C6	112.26 (12)
C1–C2–C3	119.77 (12)	C4–C5–C6	121.10 (13)
O2–C3–C2	123.18 (12)	O4–C6–C4 ⁱⁱ	123.02 (12)
O2–C3–C1 ⁱ	116.29 (12)	O4–C6–C5	118.44 (12)
C2–C3–C1 ⁱ	120.52 (12)	C4 ⁱⁱ –C6–C5	118.54 (12)

Symmetry codes: (i) *2 - x, 1 - y, -z*; (ii) *2 - x, -y, 1 - z*.

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.114$
 $S = 1.14$
 842 reflections
 94 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0694P)^2 + 0.1389P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.19 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$

Table 4

Hydrogen-bonding geometry (\AA , $^\circ$) for (II), data set (IIa).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H1 \cdots O4 ⁱ	0.84	2.04	2.737 (1)	140
O2—H3 \cdots O4 ⁱⁱ	0.84	1.85	2.686 (2)	172
O3—H5 \cdots O2	0.84	1.95	2.719 (1)	152

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

Compound (II), data set (IIb)

Crystal data

$C_6H_6O_4 \cdot C_6H_4O_4$
 $M_r = 282.20$
 Monoclinic, $P2_1/n$
 $a = 7.382 (1) \text{\AA}$
 $b = 6.065 (1) \text{\AA}$
 $c = 12.197 (1) \text{\AA}$
 $\beta = 105.778 (1)^\circ$
 $V = 525.51 (12) \text{\AA}^3$
 $Z = 2$
 $D_x = 1.783 \text{ Mg m}^{-3}$

Synchrotron radiation
 $\lambda = 0.35400 \text{\AA}$
 Cell parameters from 4909 reflections
 $\theta = 2.6\text{--}25.9^\circ$
 $\mu = 0.09 \text{ mm}^{-1}$
 $T = 110 (2) \text{ K}$
 Rectangular prism, black
 $1.12 \times 0.33 \times 0.20 \text{ mm}$

Data collection

R-Axis IV image-plate diffractometer
 φ scans
 13 465 measured reflections
 5678 independent reflections
 4908 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.032$
 $\theta_{\text{max}} = 25.9^\circ$
 $h = -14 \rightarrow 16$
 $k = -12 \rightarrow 12$
 $l = -25 \rightarrow 23$
 Intensity decay: $<2\%$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.113$
 $S = 1.04$
 5678 reflections
 94 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0738P)^2 + 0.021P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.58 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.48 \text{ e } \text{\AA}^{-3}$

Table 5

Selected geometric parameters (\AA , $^\circ$) for (II), data set (IIb).

O1—C1	1.3635 (3)	C2—C3	1.3953 (4)
O2—C3	1.3710 (3)	C3—C1 ⁱ	1.3994 (4)
O3—C5	1.3289 (3)	C4—C5	1.3533 (4)
O4—C6	1.2386 (3)	C4—C6 ⁱⁱ	1.4423 (4)
C1—C2	1.3923 (4)	C5—C6	1.5011 (4)
C1—C3 ⁱ	1.3994 (4)	C6—C4 ⁱⁱ	1.4423 (4)
O1—C1—C2	124.09 (2)	C5—C4—C6 ⁱⁱ	120.02 (2)
O1—C1—C3 ⁱ	116.21 (2)	O3—C5—C4	126.27 (2)
C2—C1—C3 ⁱ	119.69 (2)	O3—C5—C6	112.78 (2)
C1—C2—C3	119.76 (2)	C4—C5—C6	120.94 (2)
O2—C3—C2	122.87 (2)	O4—C6—C4 ⁱⁱ	122.52 (2)
O2—C3—C1 ⁱ	116.56 (2)	O4—C6—C5	118.44 (2)
C2—C3—C1 ⁱ	120.55 (2)	C4 ⁱⁱ —C6—C5	119.04 (2)

Symmetry codes: (i) $2-x, 1-y, -z$; (ii) $2-x, -y, 1-z$.

Table 6

Hydrogen-bonding geometry (\AA , $^\circ$) for (II), data set (IIb).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H1 \cdots O4 ⁱ	0.84	2.04	2.7316 (4)	140
O2—H3 \cdots O4 ⁱⁱ	0.84	1.85	2.6816 (4)	171
O3—H5 \cdots O2	0.84	1.95	2.7186 (3)	152

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

For compound (II), data set (IIb), an R-Axis IV image-plate detector was used in the BMB-5 beamline hutch of the DuPont–Northwestern–Dow Collaborative Access Team (DND–CAT) at the APS. For all data sets, H atoms were treated as riding, with O–H = 0.83–0.84 \AA and C–H = 0.94–0.95 \AA , except those attached to atoms O5 and O6 in compound (I), which were refined isotropically. The crystals were attached to glass fibers with a minimum amount of silicone cement.

For compound (I), data collection: *SMART* (Bruker, 1998); cell refinement: *SMART* (Bruker, 1998); data reduction: *SAINT-Plus* (Bruker, 1998). For compound (II), data set (IIa), data collection: *CAD-4 Software* (Enraf–Nonius, 1993); cell refinement: *DIRAX* (Duisenberg, 1992); data reduction: local programs. For compound (II), data set (IIb), data collection: *HKL* (Otwinowski & Minor, 1997); cell refinement: *HKL*; data reduction: *HKL*. For all three data sets, program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL/PC* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXTL/PC*.

This research was supported by National Institutes of Health grant No. HL 13157 and an IMGIP fellowship. Portions of this work were performed at the DND–CAT Synchrotron Research Center, located at Sector 5 of the APS. DND–CAT is supported by EI DuPont de Nemours & Co., the Dow Chemical Company, the US National Science Foundation through grant DMR-9304725, the State of Illinois through the Department of Commerce, and the Board of Higher Education through grant IBHE HECA NWU 96. Use of the APS was supported by the US Department of Energy, Basic Energy Sciences, Office of Energy Research, under contract No. W-31-102-Eng-38.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: DA1173). Services for accessing these data are described at the back of the journal.

References

- Anslow, W. K. & Raistrick, H. (1939). *J. Chem. Soc.* pp. 1446–1457.
 Bruker (1998). *SMART* (Version 5.054) and *SAINT-Plus* (Version 6.02a). Bruker AXS Inc., Madison, Wisconsin, USA.
 Chowdhury, M. (1961). *Trans. Faraday Soc.* **57**, 1482–1485.
 Debye, P. (1913). *Verh. Dtsch. Phys. Ges.* **15**, 738–752.
 Duisenberg, A. J. M. (1992). *J. Appl. Cryst.* **25**, 92–96.
 Dunitz, J. D., Schomaker, V. & Trueblood, K. N. (1988). *J. Phys. Chem.* **92**, 856–867.
 Enraf–Nonius (1993). *CAD-4 Software*. Version 1.2. Enraf–Nonius, Delft, The Netherlands.
 Jene, P. G., Chan, D. S., Cooke, B. L. & Ibers, J. A. (1999). *Acta Cryst.* **C55**, 801–806.

- Jene, P. G. & Ibers, J. A. (2000). *Acta Cryst.* **C56**, 246–249.
- Johnson, M. R., Seok, W. K., Ma, W., Slebodnick, C., Wilcoxon, K. M. & Ibers, J. A. (1996). *J. Org. Chem.* **61**, 3298–3303.
- Jones, R. G. & Shonle, H. A. (1945). *J. Am. Chem. Soc.* **67**, 1034–1035.
- Klug, H. (1965). *Acta Cryst.* **19**, 983–992.
- Krogh Andersen, E. (1967a). *Acta Cryst.* **22**, 188–191.
- Krogh Andersen, E. (1967b). *Acta Cryst.* **22**, 191–196.
- Krogh Andersen, E. & Krogh Andersen, I. G. (1975). *Acta Cryst.* **B31**, 384–387.
- Ma, W., Slebodnick, C. & Ibers, J. A. (1993). *J. Org. Chem.* **58**, 6349–6353.
- Meulenaer, J. de & Tompa, H. (1965). *Acta Cryst.* **19**, 1014–1418.
- Momenteau, M. & Reed, C. A. (1994). *Chem. Rev.* **94**, 659–698.
- Otwinowski, Z. & Minor, W. (1997). *Methods Enzymol.* **276**, 307–326.
- Robl, C. (1987). *Z. Kristallogr.* **180**, 249–253.
- Robl, C. & Sheldrick, G. M. (1988). *Z. Kristallogr.* **184**, 295–300.
- Sakurai, T. (1965). *Acta Cryst.* **19**, 320–330.
- Semmingsen, D. (1977). *Acta Chem. Scand. Ser. B*, **31**, 11–14.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1997a). *SHELXL97*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997b). *SHELXTL/PC*. Version 5.101. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Slebođnick, C., Fettinger, J. C., Peterson, H. B. & Ibers, J. A. (1996). *J. Am. Chem. Soc.* **118**, 3216–3224.
- Zaleski, J., Wu, G. & Coppens, P. (1998). *J. Appl. Cryst.* **31**, 302–304.