15-2; **15**, 58735-16-3; **16**, 58735-57-2; isoprene, 78-79-5; 1-acetoxybuta-1,3-diene, 1515-76-0; 1-acetoxy-3-methylbuta-1,3-diene, 17616-47-6; KOAc, 127-08-2; 3-methylcrotonaldehyde, 107-86-8; ammonia, 7664-41-7.

Supplementary Material Available. A table of the atomic coordinates and temperature factors from this analysis (1 page). Ordering information is given on any current masthead page.

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Molecular Geometry Studies. Crystal and Molecular Structure of a 7-Spirocyclopropylbicyclo[2.2.1]heptene Anhydride[1]

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Received December 3, 1975

The molecular geometry of the bridge spirocyclopropyl anhydride (1) has been determined. There are no unusual bond lengths or bond angles which would make it especially unstable. The molecular packing suggests a weak intermolecular oxygen carbon–carbon π bond interaction between half of the molecules in the crystal. The agreement between x-ray crystallographic data and LIS measurements is excellent.

The conformation of molecules with internal strain, which includes derivatives of the bicyclo[2.2.1]heptane series, is of continuing interest.[3] Because these molecules are rigid their molecular geometry is presumably unchanged in the solid state and in solution. Hence, a knowledge of the conformation in the solid state is essential to both an understanding of specific chemical transformations and for substantiating theoretical work.

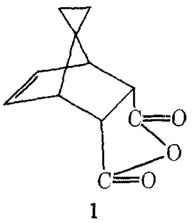
The addition of spiro substituents at the bridge position, as in the anhydride [3′aα,4′α,7′α,7′aα]-3′a,4′,7′,7′a-tetrahydrospiro[cyclopropane-1,8′-[4,7]methanoisobenzofuran]-1′,3′-dione (1), imposes further restrictions or "strain" on the bicyclo[2.2.1]heptane system.

Justification for the determination of the structure of anhydride 1 by x-ray crystallography was threefold:

(1) The structure determination of a bicyclo[2.2.1]heptane system containing a 7-spiro hydrocarbon substituent has not previously been reported. The only compound containing a spiro cyclopropylcyclopentyl grouping whose molecular geometry has been determined is spiro[bicyclo[4.1.0]hepta-2,4-diene-7,1′-(1*H*)indene].[4]

(2) To recognize if any unusual features in the endo anhydride 1, including the cyclopropyl group, are present which would explain its facile rearrangement to the corresponding exo isomer under less stringent thermal requirements than the corresponding rearrangement of the parent compound 5-bicyclo[2.2.1]heptene-2,3-*endo*-dicarboxylic anhydride (2) to 5-bicyclo[2.2.1]heptane-2,3-*exo*-dicarboxylic anhydride (3). Interest in this area derives from a series of kinetic studies on the mechanism of the endo–exo transformation.[5]

(3) Previous NMR-LIS[6] work on anhydride 1 used idealized bond angles and bond lengths for computational results.[5] The PDIGM program provided the location of the complexation site between the shift reagent Eu(fod)$_3$ and the functional group based on the shift data obtained for specific hydrogens.[7,8] Comparison of actual vs. idealized bond length–bond angle values was deemed necessary if a systematic approach using NMR-LIS work to determine molecular geometry is meaningful.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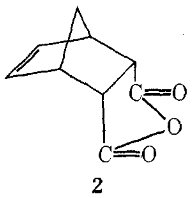
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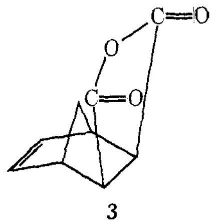
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Discussion

The anhydride 1 crystallizes in an orthorhombic space group, $P2_12_12_1$, the same as the unsubstituted anhydrides 2 and 3.[3a,c] Crystal data are given in Table I.

A major difference between 1 and 2 and 3 is the presence of two molecules in the asymmetric unit in 1. Consequently two sets of molecular dimensions are available and are designated as molecule A and molecule B of anhydride 1. The $C_{11}H_{10}O_3$ molecule is shown in Figure 1 and a stereoscopic view of the asymmetric unit in Figure 2.

Bond distances are givein in Table II, bond angles in Table III, and torsion angles calculated according to the convention of Klyne and Prelog[9] are given in Table IV.

The molecular geometry of the anhydride presents few unusual features. Characteristic bond lengths and bond angles are observed including the long carbon–carbon bonds (C_1–C_2,

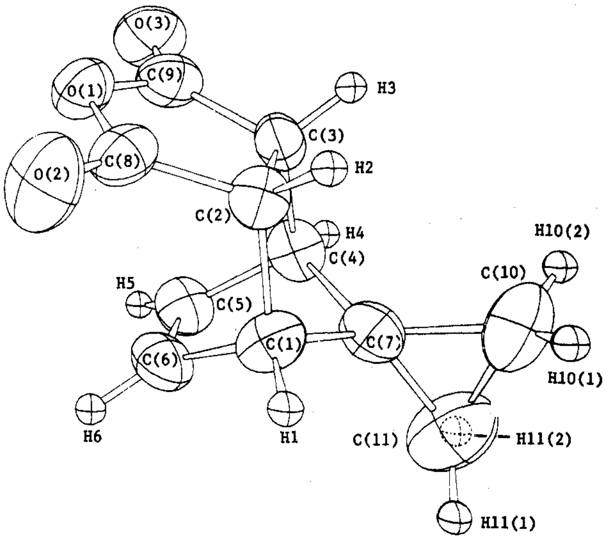

Figure 1. ORTEP drawing of the $C_{11}H_{10}O_3$ molecule. Thermal and positional parameters for molecule B were used to prepare the illustration. Thermal ellipsoids are scaled to include 50% probability; for clarity, hydrogen atoms have been assigned an isotropic temperature factor of 1.0.

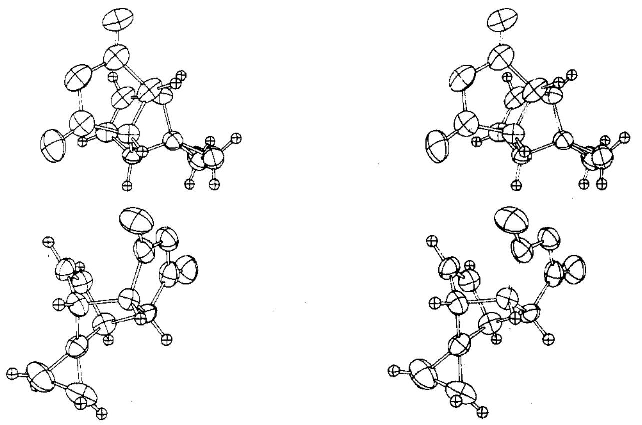

Figure 2. ORTEP stereoscopic drawing of the two molecules in the asymmetric unit illustrating their spatial relationship. The *a* axis is nearly horizontal and positive toward the right, the *c* axis is nearly vertical and positive toward the bottom, and the *b* axis is directed out of the plane of the paper, perpendicularly. Thermal ellipsoids are scaled to include 50% probability; hydrogens have been assigned an isotropic temperature factor of 1.0 for clarity.

C_3–C_4) peculiar to some Diels–Alder adducts and the short carbon–carbon bonds typical of the cyclopropyl ring. However, the two long carbon–carbon bonds to not appear equivalent in either molecule A or molecule B. This is also true for the 7-unsubstituted anhydrides, 2 and 3.[3a,c] Chemical and spectroscopic properties do not support a rationalization of this asymmetry in molecular terms and no explanation can be given at present.

Even the "tortured" spiro carbon (C_7) appears to be bonded normally to its neighbors despite bond angles of ~62 and 95°.

The overall molecular geometry of **1** is very similar to that found for the bridge unsubstituted anhydride **2** by Simonetta and co-workers.[3a]

The equations of atomic planes in the molecule are given in Table V. A comparison of the dihedral angles between certain of these planes for 1A, 1B, and 2 is shown in Figure 3.[10] The substituted anhydride 1 shows a larger A–C angle and a compensatorily smaller A–B angle than in 2. B–C and A–D angles are very similar in all structures.

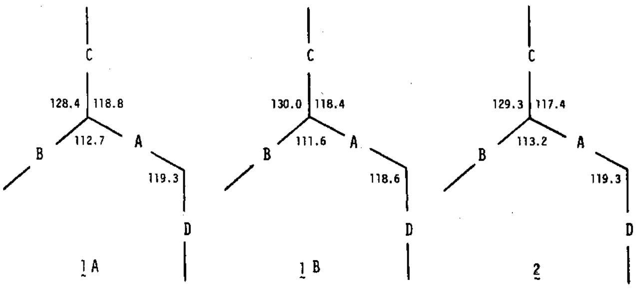

Figure 3. Angles between various atomic planes of anhydrides 1A, 1B, and 2: A = $C_1C_2C_3C_4$ plane; B = $C_1C_4C_5C_6$ plane; C = $C_1C_4C_7$ plane; D = $C_2C_3C_8C_9O$ plane. The dihedral angles between atomic planes for 1A and 1B in this figure are taken as 180 − α, where α is the dihedral angle for the appropriate plane given in Table IV. The dihedral angles for 2 are taken from ref 3a.

Table I. Crystal Data for $C_{11}H_{10}O_3$

a = 11.797 (5)
b = 6.118 (2)
c = 26.086 (15)
Space group $P2_12_12_1$
FW = 190.198
ρ_c = 1.342 g cm^{-3}
$\alpha = \beta = \gamma = 90°$
Z = 8
μ(Cu K) = 8.202 cm^{-1}
ρ_{meas} = 1.286 g cm^{-3}

Table II. Bond Distances (Å) with Standard Deviations in Parentheses

	Molecule A	Molecule B
C(1)–C(2)	1.564 (5)	1.551 (5)
C(1)–C(6)	1.507 (5)	1.509 (6)
C(1)–C(7)	1.527 (5)	1.516 (6)
C(1)–H(1)	0.88 (3)	0.96 (3)
C(2)–C(3)	1.526 (5)	1.519 (6)
C(2)–C(8)	1.518 (6)	1.489 (5)
C(2)–H2	0.89 (3)	0.87 (3)
C(3)–C(4)	1.581 (6)	1.588 (6)
C(3)–C(9)	1.487 (6)	1.493 (6)
C(3)–H(3)	0.91 (3)	0.94 (3)
C(4)–C(5)	1.506 (6)	1.510 (6)
C(4)–C(7)	1.518 (5)	1.523 (6)
C(4)–H(4)	0.94 (3)	1.02 (3)
C(5)–C(6)	1.317 (6)	1.342 (6)
C(5)–H(5)	0.94 (3)	1.06 (3)
C(6)–H(6)	1.00 (3)	0.96 (3)
C(7)–C(10)	1.488 (6)	1.472 (6)
C(7)–C(11)	1.501 (6)	1.477 (6)
C(8)–O(1)	1.388 (6)	1.397 (5)
C(8)–O(2)	1.174 (6)	1.191 (5)
C(9)–O(1)	1.388 (6)	1.376 (5)
C(9)–O(3)	1.186 (6)	1.205 (6)
C(10)–C(11)	1.557 (7)	1.499 (7)
C(10)–H10(1)	1.05 (3)	1.03 (4)
C(10)–H10(2)	1.00 (3)	0.96 (3)
C(11)–H11(1)	1.06 (3)	0.94 (3)
C(11)–H11(2)	1.03 (3)	0.98 (4)

Such a change could result from the bulkier cyclopropyl group (at C_7) in 1 interacting with hydrogens 2 and 3. In any case the differences in dihedral angles are, in our view, minor.

It is clear that the facility of the endo → exo rearrangement in the spiro anhydride 1 cannot be based on any special structural instability in 1. It should also be pointed out that spectral analyses of 7-spiro cyclopropyl bicycloheptadienes and related compounds, for which significantly different geometry from the 7-unsubstituted compounds was assumed, are now in doubt.[11]

There are small differences in molecular geometry between 1A and 1B. Two of these seem prominent enough to be worthy of consideration:

(1) The intramolecular distance between the C_5–C_6 π bond and the ether oxygen (O_1) of the anhydride ring is greater

Table III. Bond Angles with Standard Deviations in Parentheses

	Molecule A	Molecule B
C(2)–C(1)–C(6)	106.4 (3)	105.7(3)
C(2)–C(1)–C(7)	97.8 (3)	99.1 (3)
C(2)–C(1)–H1	113 (2)	119 (2)
C(6)–C(1)–C(7)	99.7 (3)	100.7 (3)
C(6)–C(1)–H1	122 (2)	114 (2)
C(7)–C(1)–H1	114 (2)	115 (2)
C(1)–C(2)–C(3)	105.4 (3)	104.3 (3)
C(1)–C(2)–C(8)	113.8 (3)	112.9 (3)
C(1)–C(2)–H2	111 (2)	109 (2)
C(3)–C(2)–C(8)	104.6 (3)	105.1 (3)
C(3)–C(2)–H2	116 (2)	116 (2)
C(8)–C(2)–H2	107 (2)	110 (2)
C(2)–C(3)–C(4)	102.3 (3)	102.5 (3)
C(2)–C(3)–C(9)	105.0 (3)	104.3 (3)
C(2)–C(3)–H3	111 (2)	109 (2)
C(4)–C(3)–C(9)	114.8 (3)	114.6 (3)
C(4)–C(3)–H3	114 (2)	110 (2)
C(9)–C(3)–H3	110 (2)	115 (2)
C(3)–C(4)–C(5)	106.7 (3)	106.0 (3)
C(3)–C(4)–C(7)	98.3 (3)	98.1 (3)
C(3)–C(4)–H4	115 (2)	113 (2)
C(5)–C(4)–C(7)	100.4 (3)	100.3 (3)
C(5)–C(4)–H4	118 (2)	118 (2)
C(7)–C(4)–H4	116 (2)	118 (2)
C(4)–C(5)–C(6)	107.7 (4)	107.5 (4)
C(4)–C(5)–H5	123 (2)	122 (2)
C(6)–C(5)–H5	127 (2)	129 (2)
C(1)–C(6)–C(5)	108.4 (3)	107.3 (4)
C(1)–C(6)–H6	126 (2)	127 (2)
C(5)–C(6)–H6	126 (2)	123 (2)
C(1)–C(7)–C(4)	95.3 (3)	95.3 (3)
C(1)–C(7)–C(10)	124.3 (3)	125.1 (4)
C(1)–C(7)–C(11)	124.4 (3)	127.0 (4)
C(4)–C(7)–C(10)	126.7 (3)	124.6 (4)
C(4)–C(7)–C(11)	125.1 (3)	125.1 (4)
C(10)–C(7)–C(11)	62.8 (3)	61.1 (3)
C(2)–C(8)–O(1)	108.8 (4)	109.7 (3)
C(2)–C(8)–O(2)	130.1 (4)	131.3 (4)
O(1)–C(8)–O(2)	121.1 (4)	118.9 (4)
C(3)–C(9)–O(1)	110.1 (4)	110.6 (3)
C(3)–C(9)–O(3)	129.8 (4)	129.3 (4)
O(1)–C(9)–O(3)	120.1 (4)	120.0 (4)
C(7)–C(10)–C(11)	59.0 (3)	59.6 (3)
C(7)–C(10)–H10(1)	120 (2)	119 (2)
C(7)–C(10)–H10(2)	118 (2)	120 (2)
C(11)–C(10)–H10(1)	114 (2)	117 (2)
C(11)–C(10)–H10(2)	111 (2)	117 (2)
H10(1)–C(10)–H10(2)	119 (2)	114 (3)
C(7)–C(11)–C(10)	58.2 (3)	59.3 (3)
C(7)–C(11)–H11(1)	126 (2)	114 (2)
C(7)–C(11)–H11(2)	117 (2)	123 (2)
C(10)–C(11)–H11(1)	125 (2)	121 (2)
C(10)–C(11)–H11(2)	121 (2)	120 (2)
H11(1)–C(11)–H11(2)	104 (2)	111 (3)
C(8)–O(1)–C(9)	111.5 (3)	110.2 (3)

Table IV. Torsion Angles Involving Atoms C(1), C(2), C(3), C(4), C(5), C(6), C(7), C(10), C(11)

	Molecule A, deg	Molecule B, deg
C(1)–C(2)–C(3)–C(4)	0.4	0.9
C(2)–C(3)–C(4)–C(5)	66.3	66.3
C(3)–C(4)–C(5)–C(6)	−70.2	−69.4
C(4)–C(5)–C(6)–C(1)	0.5	−0.2
C(5)–C(6)–C(1)–C(2)	68.8	70.6
C(6)–C(1)–C(2)–C(3)	−66.2	−68.1
C(1)–C(7)–C(4)–C(3)	60.2	59.0
C(4)–C(7)–C(1)–C(2)	−59.6	−59.0
C(7)–C(1)–C(2)–C(3)	36.4	35.8
C(7)–C(4)–C(3)–C(2)	−37.3	−37.0
C(1)–C(7)–C(4)–C(5)	−48.6	−49.0
C(4)–C(7)–C(1)–C(6)	48.6	49.0
C(7)–C(1)–C(6)–C(5)	−32.4	−32.2
C(7)–C(4)–C(5)–C(6)	31.8	32.2
C(2)–C(1)–C(7)–C(10)	82.5	80.7
C(2)–C(1)–C(7)–C(11)	160.7	158.5
C(3)–C(4)–C(7)–C(10)	−80.5	−81.0
C(3)–C(4)–C(7)–C(11)	−160.5	−157.5
C(6)–C(1)–C(7)–C(10)	−169.3	−171.3
C(6)–C(1)–C(7)–C(11)	−91.1	−93.5
C(5)–C(4)–C(7)–C(10)	170.7	171.0
C(5)–C(4)–C(7)–C(11)	90.7	94.5
C(1)–C(7)–C(10)–C(11)	114.7	116.8
C(1)–C(7)–C(11)–C(10)	−114.5	−114.0
C(4)–C(7)–C(10)–C(11)	−115.0	−114.7
C(4)–C(7)–C(11)–C(10)	117.4	113.8

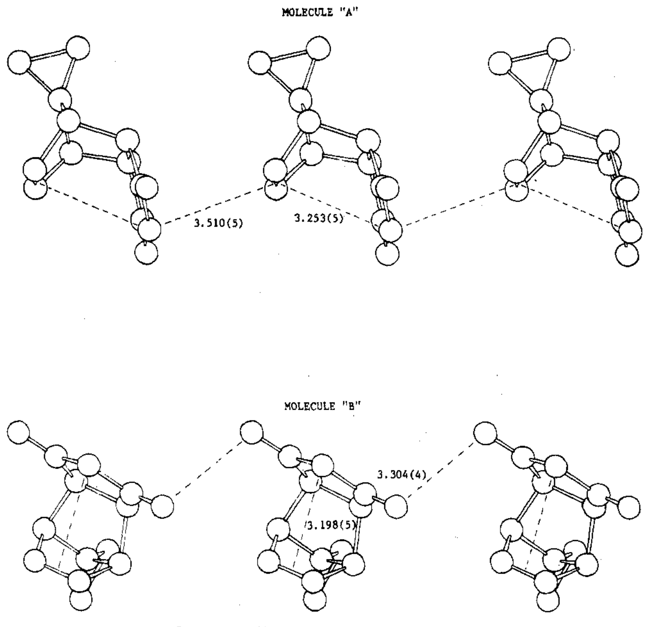

Figure 4. ORTEP drawing illustrating the translational packing along the *b* axis of each of the molecules of the asymmetric unit. The drawings illustrate intramolecular distances and standard deviations involving the ether oxygen atom, O(1), and the midpoint between the π-bonded carbon atoms, C(5) and C(6), for both molecules. Intermolecular distances illustrated involve the same atoms for molecule A, while the drawing for molecule B shows intermolecular distances involving the carbonyl oxygen atoms, O(2) and O(3). All atoms have been assigned an isotropic temperature factor of 3.5, for the sake of clarity.

in 1A than in 1B. This may be due to differences in molecular packing along the crystallographic *b* axis. The packing arrangement and distances are shown in Figure 4. Although the distances involved are slightly greater than the appropriate van der Waals radii, a weak *intramolecular* through-space interaction between the carbon–carbon π-bond and the oxygen may be postulated for both A- and B-type molecules. However, a corresponding *intermolecular* interaction could only occur for A-type molecules.

(2) The most obvious differences between 1A and 1B are the C_{10}–C_{11} bond lengths (A, 1.557; B, 1.499), the C_{10}–C_7–C_{11} bond angles (A, 62.8°; B, 61.1°), and torsion angles involving C_7, C_{10}, and C_{11}. Although the weak through-space interactions proposed above might be accommodated by molecular flexion to produce slight differences between 1A and 1B in other portions of the molecules, such a rationalization for differences occurring in the cyclopropyl portions of the molecules is too oblique for the data to

Table V. Equations of planes and Dihedral Angles

	A^a	B	C	D	Σ
	Molecule A				
(1) C(1),C(4),C(5),C(6)	0.5402	0.7901	0.2898	7.026	2.5×10^{-4}
(2) C(1),C(2),C(3),C(4)	0.8686	−0.2594	0.4223	11.884	6.8×10^{-6}
(3) C(2),C(3),C(8),C(9),O(1)	0.2244	−0.9693	0.1005	2.281	2.4×10^{-4}
(4) C(1),C(4),C(7)	0.2270	−0.9707	0.0783	3.380	
(5) C(7),C(10),C(11)	−0.4489	−0.0249	0.8933	6.971	
(6) C(1),C(2),C(6)	0.7922	0.1200	−0.5984	0.078	
(7) C(2),C(3),C(5),C(6)	0.8547	0.2988	0.4246	12.225	7.5×10^{-5}
(8) C(3),C(4),C(5)	−0.0052	0.0689	0.9976	12.580	
	Molecule B				
(1) C(1),C(4),C(5),C(6)	0.8462	−0.1854	−0.4996	5.147	1.2×10^{-5}
(2) C(1),C(2),C(3),C(4)	0.0375	−0.3558	0.9338	5.276	9.5×10^{-5}
(3) C(2),C(3),C(8),C(9),O(1)	0.8214	−0.4889	0.2936	7.744	1.5×10^{-4}
(4) C(1),C(4),C(7)	0.8335	−0.4654	0.2979	9.246	
(5) C(7),C(10),C(11)	0.3869	0.8668	0.3147	6.384	
(6) C(1),C(2),C(6)	0.1545	0.7096	0.6874	7.109	
(7) C(2),C(3),C(5),C(6)	−0.4719	−0.1060	0.8753	1.121	7.3×10^{-5}
(8) C(3),C(4),C(5)	0.5176	0.8471	−0.1208	4.164	

Dihedral Angles

	Molecule A	Molecule B		Molecule A	Molecule B
(1)–(2)	67.3°	68.4°	(3)–(7)	86.8°	85.5°
(1)–(4)	51.6°	50.0°	(4)–(5)	89.6°	89.4°
(2)–(3)	60.7°	61.4°	(6)–(7)	62.7°	63.0°
(2)–(4)	61.2°	61.6°	(7)–(8)	63.9°	63.9°

[a] The equation of the plane is $Ax + By + Cz - D = 0$, where A, B, and C are direction cosines, D is the perpendicular distance from the plane to the origin, and Σ is the sum of the squares of the deviations of the atoms from the least-squares plane. The coordinate system is described by x along a, y along b, and z along the c axis.

Table VI. Best Fit Positions for Europium Atom from PDIGM

Moment direction[a]	R_{LIS}	r_{O}[b]	R_{LIS}	r_{O}	R_{LIS}	r_{O}
Ether oxygen	9.2	0.58	4.82	0.16	3.88	0.56
Carbonyl oxygen	1.9	2.8	0.25	3.26	0.57	3.44
Anhydride 1	Ref 5		Molecule B		Molecule A	

[a] The direction of the principal magnetic moment is taken to the ether or the carbonyl oxygen. [b] r_{O} is the distance in Å from the europium atom to the nearest oxygen (this is always a carbonyl oxygen).

support. The C_{10}–C_{11} bond distance in B-type molecules could be foreshortened somewhat from thermal effects. (See Table VII, supplementary material.) At any rate, the differences are quite small.

The previous comparison of observed LIS and calculated LIS[6] for the endo anhydride **1** based on the McConnell–Robertson[12] equation and partially idealized atomic coordinates showed clearly that $Eu(fod)_3$ complexed at the carbonyl oxygen rather than the ether oxygen of the anhydride.

When the actual x-ray coordinates of this work for all atoms were used to calculate LIS values the same choice was confirmed. Indeed, the best fit for the europium atom position resulted in a substantially lower agreement factor (R_{LIS}) than in the previous work. Further, the best-fit europium atom position for **1** in this work is more consistent with the best europium position found previously for the bridge-unsubstituted anhydride (**2**), in which coordinates of all atoms had been derived from x-ray crystallography. Table VI gives comparison of the R_{LIS} value and europium–oxygen distance for the partially idealized case and for the two x-ray determined molecules.

In view of such good agreement between x-ray crystallographic and LIS data for this rigid molecule we must conclude that structural determination using LIS measurements, which has been widely discussed,[13] looks promising indeed.

Experimental Section

[3′aα,4′α,7′α,7′aα]-3′a,4′,7′,7′a-Tetrahydrospiro[cyclopropane-1,8′-[4,7]methanoisobenzofuran]-1′,3′-dione (**1**) was synthesized as previously described.[14] Clear, colorless crystals with well-defined faces were obtained by recrystallization from solution in benzene by vapor diffusion of 60–110 °C petroleum ether into the benzene solution. Preliminary Weissenberg photographs of a small crystal mounted on a glass fiber indicated an orthorhombic space group. Systematic absences were $h00$, $h \neq 2n$; $0k0$, $k \neq 2n$; and $00l$, $l \neq 2n$, uniquely defining the space group as $P2_12_12_1$.

Unit cell dimensions were provided by least-squares refinement of 14 independent 2θ values obtained by automatic centering routines on a GE XRD-490 equipped with a scintillation counter detector and interfaced with a Digital PDP-8 minicomputer. Ni-filtered Cu Kα radiation was used. Refined unit cell dimensions are given together with other pertinent crystal data in Table I.

The crystal used for both intensity data collection and determination of the unit cell parameters was mounted along the b axis and was bounded by {0.1.0}, {1.0.1}, and {1.0.1}. The dimensions of the crystal, measured as the perpendicular distances between the above parallel faces, were approximately 3.0 × 0.26 × 0.23 mm, respectively. Intensity data were collected out to a 2θ of 120° using automatic θ–2θ step scans with 0.5-s counting times at 0.05° scan intervals in 2θ, scanning over a 2° total 2θ range with total counting times of 20.5 s per scan. Backgrounds were counted for 10.25 s each on both extremes of the scan range. Three standard reflections were measured at 50 reflection intervals throughout data collection and showed no significant change. A total of 1644 reflections were scanned with 1290 considered observed based on the criteria $I > 3\sigma(I)$. Unobserved data were not included in the refinement.

Lorentz and polarization corrections were applied in the normal manner.[15] Correction of the data for absorption was made by the method of Tompa;[16] transmission factors varied from 0.805 to 0.931. Weights were calculated according to the method of Stout and Jensen;[17] $w(F) = [(k/4LpI)(\sigma^2(I) + (0.02I)^2)]^{-1}$. Scattering factors for nonhydrogen atoms were taken from the literature;[18] the scattering factor curve for hydrogen was that of Stewart, Davidson, and Simpson.[19] No correction was made for extinction.

Structure Analysis and Refinement. The structure was solved by direct methods using the program MULTAN.[15] Using a starting set of six reflections with assigned phases chosen by the program and 150 reflections with E values above 1.55, an E map revealed 26 of the 28 nonhydrogen positions for the two molecules in the asymmetric unit. These 26 positions were refined using full-matrix least squares minimizing $\Sigma w \Delta F^2$; a subsequent Fourier map revealed the positions of the two remaining nonhydrogen atoms. The structure was refined isotropically to an R factor of 12.1% ($R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$). Further refinement, treating the vibration of all atoms anisotropically, reduced the R to 9.0%. A difference Fourier was calculated which showed the positions of all hydrogen atoms. The refinement of all positional parameters and anisotropic thermal parameters for nonhydrogen atoms reduced the R to 3.7%. During these final cycles of refinement, hydrogen atoms were assigned the refined isotropic temperature factors of the carbon atoms to which they were bound; no attempt was made to refine the assigned hydrogen temperature factors. The R factor for all data, including unobserved reflections, was 5.9%; the largest shift divided by the standard deviation was 0.2 at the end of the refinement. The weighted Rw ($Rw = [\Sigma w \Delta F^2]^{1/2}/[\Sigma w F_o^2]^{1/2}$) was 3.9%, and S, the standard deviation of an observation of unit weight ($S = [\Sigma w \Delta F^2/(m - n)]^{1/2}$ where m is the number of observations and n is the number of parameters), was 1.464. A $\delta(R)$ normal probability plot[20] was calculated and was essentially linear with a slope of 1.373 and an intercept of 0.013. A final difference Fourier was calculated and showed no peaks greater than ±0.16 eÅ^{-3}; the largest peaks were found in the vicinity of the C(7) carbon atoms of both molecules in the asymmetric unit.

Acknowledgment. R.E.R.C. gratefully acknowledges financial support for chemicals associated with this project from the Eloise Gerry Fellowship Fund of Sigma Delta Epsilon, Graduate Women in Science. We also wish to thank the Montana State University Computing Center for a grant of computing time.

Registry No.—**1**, 56587-27-0.

Supplementary Material Available. Table VII, positional and thermal parameters and standard deviations (1 page). Ordering information is given on any current masthead page.

References and Notes

(1) The *Chemical Abstracts* nomenclature for compound **1** is [3′aα,4′α,7′α,7′aα]-3′a,4′,7′,7′a-tetrahydrospiro[cyclopropane-1,8′-[4,7]methanoisobenzofuran]-1′,3′-dione.

(2) Eloise Gerry Fellow, 1975–1976.

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(15) Computer programs used were by F. R. Ahmed and co-workers (NRC-2, Data Reduction; NRC-8, Fourier for Distorted and Undistorted Nets; and NRC-12, Scan of Interatomic Distances and Angles; National Research Council, Ottawa, Ontario, Canada), Busing and Levy (ORFLS), Carrol K. Johnson (ORTEP), and Germaine, Main, and Woolfson (MULTAN, 1972 version). These programs were locally modified for use with the XDS Sigma 7 computer. Other programs were written locally by G. D. Smith, C. N. Caughlan, and R. D. Larsen.

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