average shift/error during the final cycle of refinement was 0·27. Atomic positional parameters are presented in Table 1 while interatomic distances and valence angles are given in Table 2.* The numbering scheme used in Fig. 1 and in all tables reflects molecular symmetry and does not correspond to *Chemical Abstracts* notation.

Discussion. Fig. 1 is an *ORTEP* drawing (Johnson, 1971) of the two independent 9,9′-bibicyclo[3.3.1]nonylidene molecules. The molecules exist in the all-chair conformation which leads to a strong transannular interaction between (H3*b*)···H(3*b*′) and H(23*a*)···H(25*b*) of 1·99 (3) and 1·81 (8) Å, respectively. This interaction is responsible for the flattening of the cyclohexane rings as indicated by four torsion angles of less than 55·8° (Bucourt & Hainaut, 1965) and three valence angles of greater than 112°. The short transannular contact is consistent with the observed shifts in the IR and proton NMR spectra.

The double bonds with attached atoms form planar systems which bring atoms H(1′)···H(1″), H(21′)···H(21″) and the symmetry-related pairs within 1·95 (4) and 1·90 (4) Å, respectively. Since the title molecule serves as a model for compounds such as tetraisopropylethylene, it confirms the predicted strong steric interaction expected for tetra-*tert*-butylethylene which has yet to be synthesized. Although the H(1′)···H(1″) and H(21′)···H(21″) distances are short, there are apparently no significant shifts in the IR or proton NMR absorptions for these atoms.

9,9′-Bibicyclo[3.3.1]nonylidene reacts with peroxides to give an epoxide and with singlet oxygen to yield a dioxetane (Keul, 1975). The instability of the compound upon exposure to X-rays in the presence of air is more likely due to reaction with oxygen to give an epoxide.

The 9,9′-bibicyclo[3.3.1]nonylidene molecule can be considered to have D_{2h} symmetry, although not required by crystallographic symmetry.

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Structure of the 1:1 Complex of Resorcinol and Urea

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Abstract. $C_6H_6O_2.CO(NH_2)_2$, orthorhombic, $P2_12_12_1$, $a = 7·142\ (2)$, $b = 7·798\ (2)$, $c = 15·428\ (5)$ Å, $D_m = 1·321$, $D_x = 1·314$ Mg m^{-3}, $Z = 4$. 1297 observed diffractometer-measured intensities were used to determine and refine the structure ($R = 0·050$). The structure is hydrogen-bonded with O—H···O (urea) distances 2·696 and 2·679 Å and N—H···O (resorcinol) distances 2·944 and 3·128 Å. There are no short contacts between molecules of the same type.

Introduction. Urea forms 1:1 complexes with all three benzenediols, the structure of the one with hydroquinone having already been reported (Mahmoud & Wallwork, 1975). The title compound is frequently quoted in textbooks on physical chemistry as a typical intermolecular compound. Colourless crystals were prepared by slow evaporation of a solution in acetone–methanol of an equimolar mixture of resorcinol and urea. A powder photograph of this material showed it to be identical with that obtained by cooling a 1:1 fusion mixture of the two compounds.

Using Cu $K\alpha$ radiation (λ = 1·542 Å) the space group was determined from Weissenberg photographs and the refined cell dimensions from θ values measured on a four-circle diffractometer (Small & Travers, 1961). Intensity data were measured on a Stoe Stadi-2 automatic two-circle diffractometer using graphite-monochromatized Mo $K\alpha$ radiation (λ = 0·71069 Å). Two crystals were used; the layers h = 0 to 10 (1323 reflexions) were measured with one and the layers k = 0 to 2 (552 reflexions) with the other.

The intensity data were merged using the *SHELX* suite of programs (Sheldrick, 1976). The phases were determined with *MULTAN* (Germain, Main & Woolfson, 1971) and the coordinates of all non-H atoms found from the E map. After refinement, using 1297 reflexions for which $I > 2{\cdot}5\sigma(I)$, of the parameters of these atoms (including anisotropic U_{ij}), H-atom positions were obtained from the $F_o - F_c$ map. Isotropic U and coordinates for H were included in the final least-squares cycles. When R = 0·050 the parameter shifts were less than 0·2 e.s.d. for non-H atoms and less than 0·4 e.s.d. for H. Final coordinates and U values are given in Table 1.* Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1962). Bond distances and angles are listed in Table 2.

Table 1. *Fractional atomic coordinates and isotropic thermal parameters* ($\times 10^4$ *for* C, N *and* O; $\times 10^3$ *for* H)

	x	y	z	U_{eq}*/U (Å^2)
C(1)	4211 (3)	1601 (3)	6066 (1)	359 (17)
C(2)	3070 (4)	2467 (3)	5491 (2)	420 (19)
C(3)	5504 (3)	2536 (3)	6555 (1)	287 (14)
C(4)	3189 (4)	4230 (3)	5389 (2)	405 (18)
C(5)	5618 (3)	4300 (3)	6474 (1)	297 (14)
C(6)	4463 (3)	5134 (3)	5887 (1)	337 (15)
C(7)	1037 (3)	3082 (3)	8180 (2)	345 (16)
O(1)	6689 (3)	1748 (2)	7131 (1)	390 (14)
O(2)	4564 (3)	6878 (2)	5764 (1)	442 (15)
O(3)	2689 (2)	3492 (2)	8381 (1)	403 (14)
N(1)	−200 (4)	2619 (4)	8774 (2)	602 (27)
N(2)	498 (4)	3122 (5)	7354 (2)	605 (25)
H(1)	407 (5)	40 (5)	616 (2)	51 (9)
H(2)	217 (6)	182 (5)	512 (2)	60 (10)
H(3)	666 (7)	61 (7)	704 (3)	78 (13)
H(4)	260 (7)	474 (6)	499 (2)	73 (11)
H(5)	650 (5)	491 (4)	682 (2)	42 (7)
H(6)	−119 (9)	241 (8)	863 (4)	92 (17)
H(7)	12 (5)	262 (5)	928 (3)	45 (9)
H(8)	−54 (9)	253 (6)	722 (3)	79 (13)
H(9)	164 (6)	350 (6)	697 (3)	74 (12)
H(10)	545 (5)	725 (4)	612 (2)	31 (6)

* $U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

Table 2. *Bond distances* (Å) *and angles* (°), *not involving* H

C(1)–C(2)	1·381 (3)	C(3)–O(1)	1·369 (3)
C(1)–C(3)	1·393 (3)	C(7)–N(1)	1·323 (4)
C(2)–C(4)	1·386 (3)	C(7)–N(2)	1·332 (4)
C(4)–C(6)	1·383 (3)	C(7)–O(3)	1·261 (3)
C(6)–C(5)	1·388 (3)	C(6)–O(2)	1·375 (2)
C(5)–C(3)	1·391 (3)		
C(3)–C(1)–C(2)	119·0 (2)	C(1)–C(3)–O(1)	122·1 (2)
C(1)–C(2)–C(4)	121·4 (2)	C(4)–C(6)–O(2)	117·5 (2)
C(2)–C(4)–C(6)	118·9 (2)	C(5)–C(6)–O(2)	121·5 (2)
C(4)–C(6)–C(5)	121·0 (2)	O(3)–C(7)–N(1)	121·5 (3)
C(6)–C(5)–C(3)	119·3 (2)	O(3)–C(7)–N(2)	120·0 (2)
C(5)–C(3)–C(1)	120·4 (2)	N(2)–C(7)–N(1)	118·5 (2)
C(5)–C(3)–O(1)	117·5 (2)		

Discussion. An extensive system of hydrogen bonds is formed between the component molecules of this compound; there are no hydrogen bonds between molecules of the same kind. The shortest hydrogen bonds are those in which the two hydroxyl groups act as donors [O(2)–H(10)···O(3) and

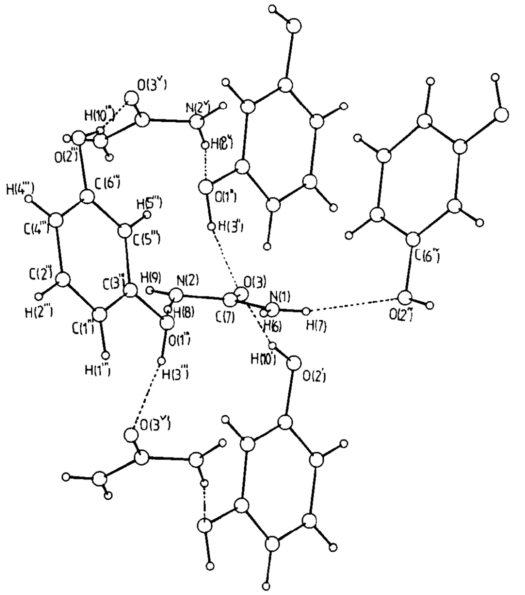

Fig. 1. View of the structure of urea–resorcinol (1:1) approximately along **a** showing hydrogen bonds. For symmetry code see Table 3.

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* Lists of structure factors, anisotropic thermal parameters and bond distances involving H have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38049 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Hydrogen-bond geometry*

A—H$\cdots B$	A—H (Å)	H$\cdots B$ (Å)	$A\cdots B$ (Å)	A—$\hat{\rm H}\cdots B$ (°)
O(1)—H(3)$\cdots$O(3$^{\rm i}$)	0·90 (5)	1·83 (4)	2·696 (3)	160 (5)
O(2)—H(10)$\cdots$O(3$^{\rm ii}$)	0·89 (3)	1·82 (3)	2·679 (3)	163 (3)
N(2)—H(8)$\cdots$O(1$^{\rm iii}$)	0·90 (6)	2·07 (5)	2·944 (4)	163 (6)
N(1)—H(7)$\cdots$O(2$^{\rm iv}$)	0·81 (4)	2·34 (3)	3·128 (4)	166 (4)

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

O(1)—H(3)$\cdots$O(3)] to the urea O. Each urea O accepts hydrogen bonds from the resorcinol molecules related by **b** thus forming infinite chains parallel to that direction. Weaker cross-links between the chains are formed by N—H$\cdots$O bonds. The shortest of these, N(2)—H(8)$\cdots$O(1), joins together chains related by a 2_1 axis through $(0,y,\frac{3}{4})$ and a further, longer bond N(1)—H(7)$\cdots$O(2) connects chains related by a 2_1 axis through $(x,\frac{3}{4},\frac{1}{4})$. In this manner four of the six possible H are involved in hydrogen bonding and a three-dimensional network is formed, Fig. 1. The geometry of the hydrogen bonds is given in Table 3. The intermolecular distances are similar to those reported for the hydroquinone–urea complex (Mahmoud & Wallwork, 1975) in which the shortest distances (2·683 and 2·702 Å) were also from hydroxyl groups to urea O with longer N—H$\cdots$O bonds (2·980, 3·049 and 3·048 Å) to the phenol O. Short contacts between molecules of the same type were absent in that structure also.

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3-Benzoyl-5,8-dihydroxyflavone,* a New Synthetic Contact Allergen

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Abstract. $C_{22}H_{14}O_5$, monoclinic, $P2_1/c$, a = 10·149 (4), b = 18·124 (7), c = 9·393 (4) Å, β = 100·54 (5)°, (λ = 0·7107 Å, T = 293 K), V = 1698·6 Å^3, Z = 4, M_r = 358·35, D_x = 1·401 Mg m^{-3}, $F(000)$ = 744, μ(Mo $K\alpha$) = 0·108 mm^{-1}; R_1 = 8·2% for 1288 observed reflections. The structure has been elucidated by X-ray analysis. The benzo-4-pyrone system is nearly coplanar. The conformation of the molecule is characterized by three planes: the *A,B* rings, the phenyl ring *C* and the benzoyl ring *D*. The angle between rings *A* and *C* is 32 (2)°, *A* and *D* 82 (2)°, and *C* and *D* 113 (1)°. The hydroxyl H at O(5) is hydrogen-bonded to the C(4) carbonyl group. Intermolecular hydrogen bonds of the type O—H$\cdots$O form endless chains with alternate molecules along **b**.

Introduction. The naturally occurring flavone primetin (I) has been shown to possess a sensitizing capacity. It crystallizes as a polytypic structure with twinning effects (Jarchow & Schmalle, 1981). These effects led to abnormally short as well as long bonds being observed in the structure (Jarchow, Schmalle, Hausen & Schulz, 1983). To obtain more suitable crystals and to collect further material for testing purposes, (I) was synthesized in several steps (Schaefer & Krebs, 1981). In the first step 3-benzoyl-5-hydroxyflavone (II) was obtained. Finally, a third compound (III) was detected as a by-product during purification of primetin (I)

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* 3-Benzoyl-5,8-dihydroxy-2-phenyl-4H-1-benzopyran-4-one.

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