Crystal and Molecular Structure of μ-Carbonyl-μ-(1—3-η : 1-σ, 4—2'-η-{1,3-dimethyl-4-[5-oxo-4-phenyl-2(5*H*)-furan-2-ylidene]but-1-ene-1,3-diyl})-bis(dicarbonylcobalt) (*Co–Co*)

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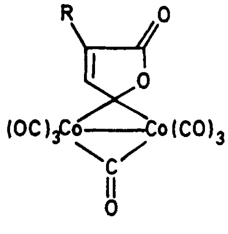
The crystal and molecular structure of the title compound have been determined by *X*-ray diffraction methods from photographic data. Crystals are triclinic tablets, space group $P\bar{1}$, with $Z = 2$ in a cell with dimensions $a = 9.41(3)$, $b = 9.98(3)$, $c = 11.90(3)$ Å, $\alpha = 97.3(3)$, $\beta = 100.9(3)$, and $\gamma = 108.3(3)°$. The structure has been solved by Patterson and Fourier methods, and refined by least-squares techniques to *R* 0.096 for 946 reflections. The cobalt–cobalt distance is 2.48 Å.

REACTION between a butenolide complex, (1; R = H, Me, or Ph) and a monosubstituted acetylene, L, gives a mixture of products which includes two isomeric compounds of formula [(1) − 2 CO]L_2. In the case of the butenolide (1; R = Ph) and methylacetylene, both isomers, denoted A and B,[1] have been obtained pure. This paper reports the structure of the B isomer. The A isomer has so far provided only multiply twinned crystals that have frustrated *X*-ray analysis.



(1)

EXPERIMENTAL

The title compound forms red tablets (m.p. 184 °C). The crystal selected was bounded by (1 0 $\bar{1}$) and ($\bar{1}$ 0 1), with {0 1 0}, {0 0 1}, and {0 1 1} displayed.

Crystal Data.—$C_{21}H_{14}Co_2O_7$, $M = 496.2$, Triclinic, $a = 9.41(3)$, $b = 9.98(3)$, $c = 11.90(3)$ Å, $\alpha = 97.3(3)$, $\beta = 100.9(3)$, $\gamma = 108.3(3)°$, $U = 1\,002$ Å^3, $D_m = 1.603 \pm 0.005$ g cm^{-3}, $Z = 2$, $D_c = 1.644$ g cm^{-3}, $F(000) = 500$, space group $P\bar{1}$, Cu-K_α radiation, $\lambda = 1.541\,8$ Å, μ(Cu-K_α) = 136.1 cm^{-1}. Crystal dimensions, *ca.* 0.1 mm along each axial direction.

Crystallographic Measurements.—The parameters of the unit cell were found from rotation and Weisenberg photographs about *a* with Cu-K_α radiation, and from precession photographs with Mo-K_α ($\lambda = 0.710\,7$ Å) radiation. The intensity data for the layers 0—7 *kl* were collected as equi-inclination multiple-film Weissenberg photographs. The intensities for 981 reflections were measured and corrected for absorption at the S.R.C. Microdensitometer Laboratory. In each case these were corrected for time of exposure, polarization, and Lorentz factors.

Structure Determination.—The known molecular weight, the density, and the volume of the unit cell gave $Z = 2$, and the space group was taken as $P\bar{1}$. A three-dimensional Patterson map gave co-ordinates for the cobalt atoms and successive rounds of structure-factor calculations and Fourier syntheses revealed the remaining carbon and oxygen atoms. Scattering factors for the atom types were taken from ref. 2, and throughout the calculations were corrected for the real and imaginary components of anomalous dispersion. The co-ordinates and temperature factors for the atoms (anisotropic for cobalt) were optimized by block-diagonal least-squares methods; the course of the calculations was that used for tricarbonyl(2-methylindolyl)-manganese,[3] except that the final weighting scheme was $w^{\frac{1}{2}} = 1.0$ if $|F_o| \leqslant 35.0$, otherwise $w^{\frac{1}{2}} = 35.0/|F_o|$. The final discrepancy ratios were *R* 0.096, R' ($= \Sigma w\Delta^2/\Sigma wF_o^2$) 0.132. Observed and calculated structure factors, temperature parameters, and evidence of convergence of the refinement are listed in Supplementary Publication No. SUP 22681 (13 pp.).‡ An attempt to continue refinement with anisotropic temperature parameters for all atoms produced little change in bond lengths, but gave implausible temperature parameters, possibly a consequence of the low ratio of intensities (981) to parameters (270 atomic and temperature parameters). When refinement had converged, a difference-Fourier map was computed and searched for peaks of maximum density $\geqslant |0.5|$ e Å^{-3}. Sixteen were found, nine with positive density, seven with negative density; the extreme values were 0.7 and −0.6 e Å^{-3}. One, of height 0.6 e Å^{-3}, was the hydrogen at C(20); C–H 1.04 Å, H–C(20)–C angles 118 [C(19)] and 129° [C(22)]. No other peak was chemically significant.

DISCUSSION

Table 1 gives the final co-ordinates of the atoms, and standard derivations derived from the final least-squares matrix. Figure 1 shows the non-standard crystallographic numbering of the molecule and Figure 2 is a stereoscopic drawing of the molecule, seen normal to the plane through Co(1), Co(2), and C(15). Table 2 lists interatomic distances, valency angles, and some non-bonded interactions; Table 3 gives mean values of the parameters for selected types of bond, together with the means of some previously measured values, and the mean estimated standard deviations for bond lengths and angles. Table 4 lists radial and angular co-ordinates in stereographic projections of the bond vectors about the two cobalt atoms and Table 5 comparative data for bond lengths within the furanone ring for different compounds.

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‡ For details see Notices to Authors No. 7, *J.C.S. Dalton*, 1979, Index issue.

Table 6 gives deviations of atoms from least-squares planes through selected groups of atoms.

The title compound is derived from the butenolide (**1**; R = Ph); the crystal and molecular structures of the analogue (**1**; R = H) have been described by Mills and Robinson.[4] The cobalt–cobalt distance in the title compound (2.48 Å) is similar to that in (**1**; R = H), and in other organometallic compounds containing a cobalt–cobalt bond. Neither cobalt atom has octahedral co-ordination. For the terminal carbonyl groups the mean C–Co–C angle (98°) is similar to that (101°) for dicobalt octacarbonyl,[5] and for those in (**1**; R = H) (101° at each cobalt atom).* The bond angles at C(10) and C(11) depart from 180° by two standard deviations; this may indicate disorder, as the highest temperature parameters are systematically those for the oxygen atoms of the carbonyl ligands. The Co–C distances

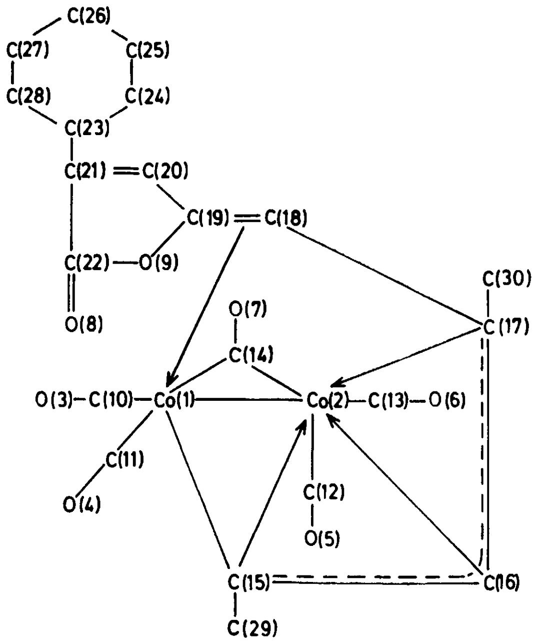

FIGURE 1 A molecule of the title compound showing the crystallographic numbering used

TABLE 1

Final atomic co-ordinates ($\times 10^4$) with standard deviations in parentheses

Atom	*x/a*	*y/b*	*z/c*
Co(1)	5 671(5)	2 102(4)	2 132(3)
Co(2)	3 562(5)	3 098(4)	1 773(3)
O(3)	5 486(30)	−647(27)	2 629(22)
O(4)	7 516(26)	2 152(23)	408(19)
O(5)	380(26)	1 562(23)	1 477(19)
O(6)	3 438(27)	5 253(24)	364(20)
O(7)	3 579(27)	1 223(24)	−231(19)
O(8)	11 003(22)	3 211(20)	4 176(16)
O(9)	8 937(18)	3 585(16)	3 226(13)
C(10)	5 469(32)	386(30)	2 371(23)
C(11)	6 823(31)	2 072(28)	1 116(22)
C(12)	1 639(31)	2 157(28)	1 594(22)
C(13)	3 457(35)	4 324(32)	941(25)
C(14)	3 995(36)	1 905(33)	751(26)
C(15)	4 218(24)	2 239(22)	3 128(18)
C(16)	4 352(27)	3 630(25)	3 540(20)
C(17)	5 443(26)	4 646(24)	3 062(19)
C(18)	6 692(28)	4 237(26)	2 724(21)
C(19)	7 443(28)	3 568(25)	3 438(20)
C(20)	7 523(26)	3 303(23)	4 607(18)
C(21)	8 874(26)	3 090(24)	5 036(19)
C(22)	9 667(30)	3 216(27)	4 171(21)
C(23)	9 356(28)	2 705(26)	6 177(20)
C(24)	8 930(31)	3 249(28)	7 107(23)
C(25)	9 432(36)	2 923(34)	8 210(26)
C(26)	10 317(37)	2 113(33)	8 330(27)
C(27)	10 802(39)	1 533(36)	7 402(29)
C(28)	10 268(30)	1 823(27)	6 292(21)
C(29)	3 262(31)	1 044(29)	3 626(23)
C(30)	5 631(33)	6 291(30)	3 240(24)

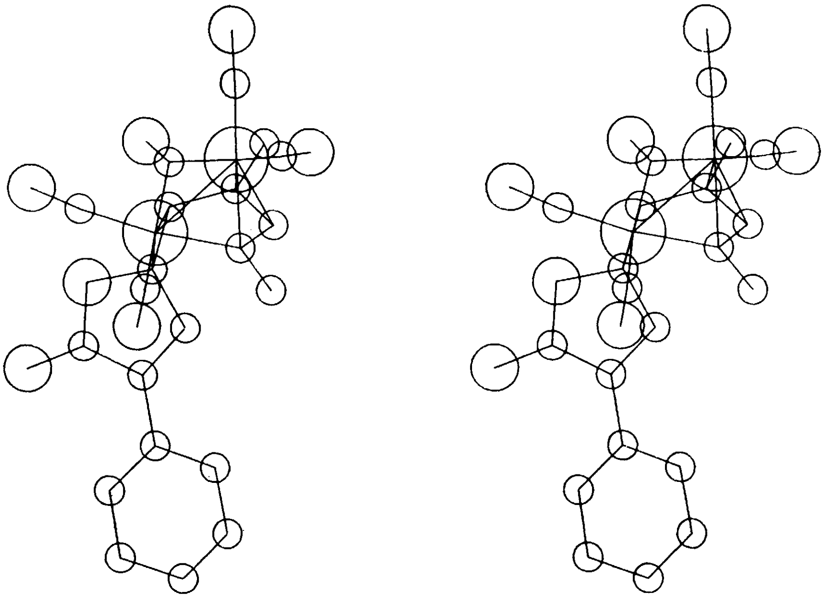

FIGURE 2 Stereoscopic drawing showing the molecule viewed normal to the plane through Co(1), Co(2), and C(15). Co(1) lies to the left; in front of it are C(18) (upper) and C(19) (lower)

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* Values calculated from published data.

TABLE 2

Selected valency parameters

(*a*) Bond lengths (Å)

Co(1)–Co(2)	2.48	O(9)–C(19)	1.47
Co(1)–C(10)	1.73	O(9)–C(22)	1.36
Co(1)–C(11)	1.77	C(15)–C(16)	1.37
Co(1)–C(14)	1.99	C(15)–C(29)	1.51
Co(1)–C(15)	2.00	C(16)–C(17)	1.46
Co(1)–C(18)	2.01	C(17)–C(18)	1.47
Co(1)–C(19)	2.06	C(17)–C(30)	1.58
Co(2)–C(12)	1.71	C(18)–C(19)	1.37
Co(2)–C(13)	1.68	C(19)–C(20)	1.44
Co(2)–C(14)	1.79	C(20)–C(21)	1.37
Co(2)–C(15)	2.01	C(21)–C(22)	1.38
Co(2)–C(16)	2.03	C(21)–C(23)	1.49
Co(2)–C(17)	2.13	C(23)–C(24)	1.35
O(3)–C(10)	1.12	C(23)–C(28)	1.41
O(4)–C(11)	1.15	C(24)–C(25)	1.42
O(5)–C(12)	1.12	C(25)–C(26)	1.33
O(6)–C(13)	1.22	C(26)–C(27)	1.40
O(7)–C(14)	1.20	C(27)–C(28)	1.42
O(8)–C(22)	1.26		

(*b*) Bond angles (°)

(*i*) Round Co(1)

Co(2)–Co(1)–C(10)	126	C(11)–Co(1)–C(14)	86
Co(2)–Co(1)–C(11)	121	C(11)–Co(1)–C(15)	173
Co(2)–Co(1)–C(14)	46	C(11)–Co(1)–C(18)	93
Co(2)–Co(1)–C(15)	52	C(11)–Co(1)–C(19)	95
Co(2)–Co(1)–C(18)	75	C(14)–Co(1)–C(15)	88
Co(2)–Co(1)–C(19)	107	C(14)–Co(1)–C(18)	105
C(10)–Co(1)–C(11)	95	C(14)–Co(1)–C(19)	144
C(10)–Co(1)–C(14)	107	C(15)–Co(1)–C(18)	85
C(10)–Co(1)–C(15)	90	C(15)–Co(1)–C(19)	89
C(10)–Co(1)–C(18)	148	C(18)–Co(1)–C(19)	39
C(10)–Co(1)–C(19)	109		

(*ii*) Round Co(2)

Co(1)–Co(2)–C(12)	126	C(13)–Co(2)–C(14)	98
Co(1)–Co(2)–C(13)	126	C(13)–Co(2)–C(15)	161
Co(1)–Co(2)–C(14)	53	C(13)–Co(2)–C(16)	123
Co(1)–Co(2)–C(15)	52	C(13)–Co(2)–C(17)	92
Co(1)–Co(2)–C(16)	77	C(14)–Co(2)–C(15)	94
Co(1)–Co(2)–C(17)	72	C(14)–Co(2)–C(16)	129
C(12)–Co(2)–C(13)	100	C(14)–Co(2)–C(17)	118
C(12)–Co(2)–C(14)	99	C(15)–Co(2)–C(16)	40
C(12)–Co(2)–C(15)	94	C(15)–Co(2)–C(17)	69
C(12)–Co(2)–C(16)	103	C(16)–Co(2)–C(17)	41
C(12)–Co(2)–C(17)	139		

(*iii*) Carbonyl ligands

Co(1)–C(10)–O(3)	172	Co(2)–C(12)–O(5)	179
Co(1)–C(11)–O(4)	173	Co(2)–C(13)–O(6)	177
Co(1)–C(14)–O(7)	134	Co(2)–C(14)–O(7)	144

(*iv*) Internal angles within the ring systems

C(16)	111	C(23)	121
C(17)	118	C(24)	119
C(18)	119	C(25)	121
		C(26)	123
O(9)	106	C(27)	116
C(19)	104	C(28)	120
C(20)	110		
C(21)	107		
C(22)	113		

(*v*) Other angles

Co(1)–C(15)–C(16)	113	Co(1)–C(19)–O(9)	110
Co(1)–C(15)–C(29)	127	Co(1)–C(19)–C(18)	68
Co(2)–C(15)–C(16)	71	Co(1)–C(19)–C(20)	117
Co(2)–C(15)–C(29)	129	C(20)–C(21)–C(23)	127
C(16)–C(15)–C(29)	119	C(22)–C(21)–C(23)	126
Co(2)–C(17)–C(16)	66	O(8)–C(22)–O(9)	116
Co(2)–C(17)–C(18)	98	O(8)–C(22)–C(21)	131
Co(2)–C(17)–C(30)	120	C(22)–C(23)–C(24)	139
C(16)–C(17)–C(18)	118	C(22)–C(23)–C(28)	121
C(16)–C(17)–C(30)	122		
C(18)–C(17)–C(30)	118		

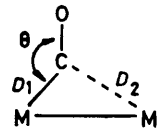
TABLE 2 (*Continued*)

(*c*) Intermolecular contacts (Å); limits O · · · O <3.30, O · · · C <3.40, C · · · C <3.60

O(3) · · · O(7$^{\mathrm{I}}$)	3.18	O(8) · · · C(16$^{\mathrm{IV}}$)	3.29
O(4) · · · O(5$^{\mathrm{IV}}$)	3.02	O(8) · · · C(20$^{\mathrm{V}}$)	3.32
O(4) · · · O(6$^{\mathrm{II}}$)	3.18	O(9) · · · C(24$^{\mathrm{V}}$)	3.29
O(4) · · · O(7$^{\mathrm{I}}$)	3.16	C(10) · · · O(7$^{\mathrm{I}}$)	3.25
O(5) · · · O(4$^{\mathrm{III}}$)	3.02	C(11) · · · O(7$^{\mathrm{I}}$)	3.20
O(5) · · · O(8$^{\mathrm{III}}$)	3.27	C(12) · · · O(8$^{\mathrm{III}}$)	3.34
O(6) · · · O(4$^{\mathrm{II}}$)	3.18	C(16) · · · O(8$^{\mathrm{III}}$)	3.29
O(7) · · · O(3$^{\mathrm{I}}$)	3.18	C(20) · · · O(8$^{\mathrm{V}}$)	3.32
O(7) · · · O(4$^{\mathrm{I}}$)	3.16	C(20) · · · C(22$^{\mathrm{V}}$)	3.56
O(7) · · · O(10$^{\mathrm{I}}$)	3.25	C(21) · · · C(22$^{\mathrm{V}}$)	3.44
O(7) · · · C(11$^{\mathrm{I}}$)	3.20	C(22) · · · C(20$^{\mathrm{V}}$)	3.56
O(8) · · · O(5$^{\mathrm{IV}}$)	3.27	C(22) · · · C(21$^{\mathrm{V}}$)	3.44
O(8) · · · C(12$^{\mathrm{IV}}$)	3.34	C(24) · · · O(9$^{\mathrm{V}}$)	3.29

* The Roman numeral superscripts refer to the following equivalent positions: I $1 - x, -y, -z$; II $1 - x, 1 - y, -z$; III $-1 + x, y, z$; IV $1 + x, y, z$; V $2 - x, 1 - y, 1 - z$.

involving the carbonyl groups resemble those for [$Co_4(CO)_{10}(C_2H_5CCC_2H_5)$].[6] In both cases the average Co–C distance for the terminal ligands is short and the same asymmetry is shown by the bridging carbonyl, much greater than in (1; R = H). For the title compound, the asymmetry, $(D_2 - D_1)/D_2$ (0.11), and θ (144°) [see (2)] fit the curve for normally asymmetric carbonyl bridges.[7] If a metal (terminal) carbonyl fragment can be represented as (3a), then the bonding at the bridging carbonyl in the title compound is better represented as (3b) than as (3c); since the bond Co(2)–C(14) (1.79 Å) is comparable in length with the other Co–C (terminal carbonyl) bonds, while the distance Co(1)–C(14) (1.99 Å)



(2)

TABLE 3

Means of selected valency parameters. Figures in parentheses are means of previously measured values

(*a*) Bond lengths (Å)

Co–Co	2.48 (2.45) ^{a} (2.524) ^{b} (2.46) ^{c}
Co–C (terminal carbonyl)	1.72 (1.81) ^{a} (1.80) ^{b} (1.73) ^{c}
C–O (terminal carbonyl)	1.15 (1.15) ^{a} (1.17) ^{b} (1.18) ^{c}
Co–C (sp^2) σ bond	2.00 (2.01) ^{c}
μ bond	2.04 (2.08) ^{d} (2.10) ^{e}
π-allyl system	2.05 (2.09) ^{d} (2.04) ^{f} (2.08)
C–C (π-allyl system)	1.42 (1.43) ^{d} (1.41) ^{f} (1.40)

(*b*) Mean estimated standard deviations

(*i*) Bond lengths (Å)

Co–Co	0.01
Co–C, O–C, C–C [O(8) to C(22) inclusive]	0.03
Carbonyl groups, benzene ring, and bonds to C(29) and C(30)	0.04

(*ii*) Bond angles (°)

Co–Co–C	1.4	C–Co–C	2.2
Co–C–C	2.4	Co–C–O (carbonyl)	3.6
Light atoms (as above)	3.1	Benzene ring	4.5

[a] Ref. 5. [b] Ref. 6. [c] Ref. 7. [d] L. Porri, B. Vitalli, and M. Zocchi, *Chem. Comm.*, 1969, 276. [e] G. Allegra, F. L. Guidice, G. Natta, U. Giannini, G. Fagherazzi, and P. Pino, *Chem. Comm.*, 1967, 1263. [f] O. S. Mills and G. Robinson, *Proc. Chem. Soc.*, 1964, 187.

is similar to that for a Co–C(alkene) bond. Co(1) is linked by a single bond to C(15) and, at C(18) and C(19), to the π electrons of a double bond. Table 4 shows C(11) and C(15) mutually *trans*. If these are taken as axial, and the other ligands chosen are C(10), and the midpoints of the bonds Co(2)–C(14), and of C(18)–C(19), the co-ordination approaches trigonal bipyramidal with vectors displaced towards C(15). Co(2) is bonded to the major ligand at C(15), C(16), and C(17), which form a π-allyl system. The mean Co–C distance (2.04 Å) is similar to that found in other systems, and the variation in the Co–C distances is unexceptional. None of the sites of the π-allyl system, nor the midpoints of their bonds, is *trans* to any of the carbonyl groups.

TABLE 4

Stereographic projections of the bond vectors about the cobalt atoms [a]

Vector	Radius	Azimuth	
(*a*) From Co(1)			
C(15)	0	0	Dot
C(18)	92	0	Dot
C(19)	98	39	Dot
M(18–19) [b]	95	19	Dot
C(10)	100	148	Dot
Co(2)	49	−74	Dot
C(14)	96	−105	Dot
M(2–14) [b]	66	−90	Dot
C(11)	7	−77	Ring
(*b*) From Co(2)			
C(12)	0	0	Dot
C(13)	83	0	Ring
C(14)	85	99	Ring
C(15)	94	−166	Ring
C(16)	80	−127	Ring
C(17)	37	−105	Ring
Co(1)	51	150	Ring
M(15–16) [b]	86	−147	Ring
M(16–17) [b]	56	−118	Ring

[a] The limiting radius = 100; vectors ending in the northern hemisphere or on the equator are labelled Dot, those ending in the southern hemisphere, Ring. [b] M indicates the midpoint of the bond between the atoms indicated.

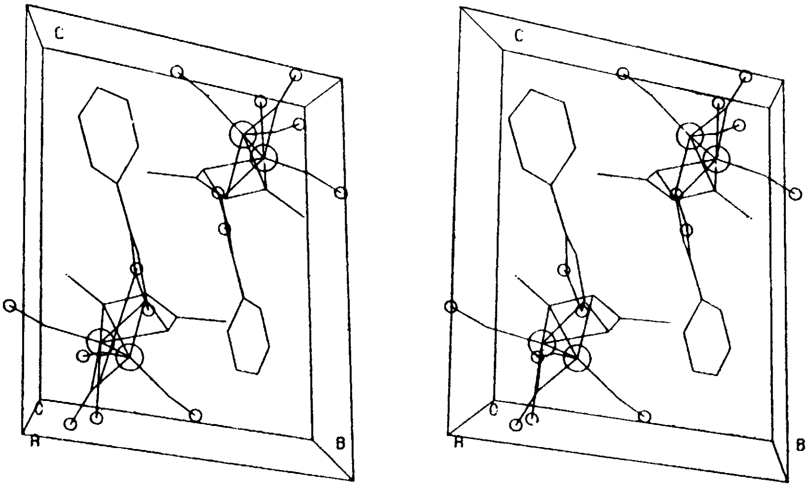


FIGURE 3 The unit-cell contents, viewed down the *a* axis

The title compound apart, a dihydrofuranone ring occurs in (1; R = H) linked at the site matching C(19) to two cobalt atoms, and bonded at the site matching C(20) to a saturated carbon at the 17-position of a steroid, in digitoxigenin,[8a] and in $\Delta^{8,14}$-anhydrodigitoxigenin.[8b] If the values for the steroid derivatives are taken as standard, then both cobalt compounds have a common set of deviations; bonds corresponding to O(8)–C(22) and C(20)–C(21) are lengthened while those corresponding to C(19)–C(20) and C(21)–C(22) are shortened. The two cobalt compounds differ; in the title compound the distance O(9)–C(19) is relatively long, while in (1; R = H) it is noticeably short. The pattern can be rationalized by assuming that cobalt is a net electron donor with oxygen the recipient. In the title compound electron-release occurs through a bridge-bond into the π system, resulting in relatively large changes in the lengths of the conjugated bonds; in (1; R = H) the release occurs along σ bonds into the σ system, shortening particularly the bond

TABLE 5

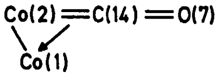
Bond lengths (Å) within 2-oxo-2,5-dihydrofuran systems

Bond corresponding to	Title compound	(1; R = H)	D [a]	A [b]
O(8)–C(22)	1.26	1.23	1.178	1.202
O(9)–C(19)	1.47	1.40	1.451	1.438
O(9)–C(22)	1.36	1.42	1.391	1.365
C(19)–C(20)	1.44	1.42	1.484	1.499
C(20)–C(21)	1.37	1.37	1.358	1.317
C(21)–C(22)	1.38	1.44	1.463	1.457

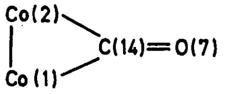
[a] Digitoxigenin (ref. 8*a*). [b] $\Delta^{8,14}$-Anhydrodigitoxigenin (ref. 8*b*).

$M{=}C{=}O$

(3a)



(3b)



(3c)

O(9)–C(19), and the heterocyclic π system then acts as a weak acceptor. In the title compound, unlike the three analogues, the five-membered heterocyclic ring is not planar.[5,8]

The effect of co-ordination to cobalt at C(18) and C(19) distorts the alkene from its ideal geometry. If a reference plane is chosen through Co(1), C(18), and C(19), then the dihedral angles between this plane, and the planes through C(18), C(19), and their substituents are: for C(17), 98°; for O(9), 100°; and for C(20), 110°; that is, all substituents are displaced away from the cobalt.[9]

TABLE 6

Mean planes, and deviations (Å) of atoms from mean planes in square brackets *

Plane (1): O(8), O(9), C(19)—(23)

[O(8) −0.02, O(9) −0.02, C(19) 0.02, C(20) −0.02, C(21) 0.00, C(22) 0.05, *C*(23) 0.05]

Plane (2): C(21), C(23)—(28)

[*C*(21) −0.07, C(23) −0.01, C(24) 0.00, C(25) 0.01, C(26) 0.00, C(27) −0.01, C(28) 0.01]

* Italicized atoms were not used to locate the mean plane.

The atoms of the phenyl group are coplanar within experimental error; C(21) lies 0.07 Å out of the plane. The best plane through the phenyl group is inclined at 32° to the plane through C(20), C(21), and C(22).

Figure 3 shows the contents of the unit cell. Relatively, the closest intermolecular contacts are O · · · O; there are only four C · · · C contacts less than 3.6 Å. This open packing results in high thermal parameters for the atoms of the phenyl group, and correspondingly large standard deviations in their bond parameters.

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