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Carbon Compounds of the Transition Metals, VII; The Structure of a γ-lactone Bridged Cobalt Carbonyl (triclinic modification).

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The compound of empirical composition $Co_2(CO)_9HCCH$, formed by the reaction of $Co_2(CO)_6HCCH$ with CO under pressure, has been shown to contain an unsaturated lactone ring which bridges the metal atoms. The structure has been determined by three-dimensional Fourier methods and refined by least-squares techniques. The compound crystallised in the triclinic system, spacegroup P$\bar{1}$, with unit-cell dimensions a = 7.279 ± 0.003, b = 8.589 ± ± 0.004, c = 12.560 ± 0.006 Å, α = 94°39′ ± 6′, β ± 115°26′ ± 3′, γ = 93°22′ ± 13′ *and two molecules per unit cell. The average bond distances found were Co-Co 2.45 ± 0.01₅, Co-C(sp² bridging) 1.93 ± 0.08, Co-C(sp³ bridging) 2.02 ± 0.08, Co-C(terminal CO) 1.81 ± 0.08 and C-O(terminal) 1.15 ± 0.10 Å, where the limits quoted are* three *standard deviations. The structure of this molecule is similar in geometry to that found subsequently for $Co_2(CO)_8$.*

Introduction

It has been shown from infra-red measurements[1,2] that both bridging and non-bridging CO groups occur in $Co_2(CO)_8$ and that the absorption frequencies which correspond to the bridging groups (ca. 1860 cm^{-1}) are absent when two molecules of CO are displaced by one molecule of acetylene.[3] This derivative, dicobalt acetylene hexacarbonyl $Co_2(CO)_6HCCH$, reacts further with CO under more energetic conditions[4] to form dicobalt acetylene nonacarbonyl, $Co_2(CO)_9HCCH$, which once more exhibits absorption bands in the 1800 cm^{-1} region. It was the object of the present investigation to determine the overall geometry of this latter complex.

Experimental Section

Crystal Data. Dicobalt acetylene nonacarbonyl, $Co_2(CO)_9HCCH$, mol. wt. 396.1, *triclinic*, $a = 7.279 \pm \pm 0.003$, $b = 8.589 \pm 0.004$, $c = 12.560 \pm 0.006$ Å, $\alpha = 94°39' \pm 6'$, $\beta = 115°26' \pm 3'$, $\gamma = 93°22' \pm \pm 13'$, $U = 703 \pm 0.5$ Å,[3] $D_m = 1.86$ g. cm^{-1}, $D_c = 1.87$ g. cm^{-1}, $Z = 2$. Systematic absences: none. Spacegroup $P\bar{1}$ (assumed) Linear absorption coefficient (Co-K_α radiation) $\mu = 55.4$ cm^{-1}.

A sample of the material was kindly supplied by Dr. H. W. Sternberg. Suitably small crystals ($\mu t < 1$) were cut and used without absorption correction. Oscillation and Weissenberg photographs failed to reveal any detectable symmetry elements. From the arbitrarily chosen axes the Delauney reduced cell was obtained.

The values of the unit cell parameters were refined by a least-squares method similar to that suggested by Cohen.[5-7] Measurements were made on accurately set $0k1$ and $h1l$ Weissenberg photographs which had been calibrated with NaCl traces. Measurements were restricted to those spots whose α_1, α_2 doublet was resolved (Co-K_α radiation) and from these a 7×7 matrix was formed which involved, in addition to the unit-cell parameters, one parameter which makes allowance for absorption and eccentricity corrections. This latter parameter evaluated very small. Estimates of the standard deviations of the unit-cell parameters were obtained from the diagonal elements of the inverse matrix and the limits quoted in this paper are three times the standard deviations. All reflexions were given unit weight.

Partial three-dimensional data were collected by the multiple film technique from the levels $h0l$ to $h3l$ and from $0kl$ Weissenberg photographs. In this way slightly more than 1000 reflexions were recorded which we considered to be sufficient for a determination of molecular geometry since accurate values of bond lengths were not the prime aim in the analysis. Subsequently it became clear that the crystal had decomposed whilst the $h3l$ data were being collected; the refinement was based on 939 reflexions of which 99 were too weak to be estimated.

Solution and Refinement. The structure was solved by conventional Fourier methods. Individual positional and isotropic thermal parameters were refined by least-squares methods. The matrix approximation used

(1) J. W. Cable, R. S. Nyholm and R. K. Sheline, *J. Am. Chem. Soc.*, 76, 3373 (1954).
(2) R. A. Friedel, I. Wender, S. L. Shufler and H. W. Sternberg, *J. Am. Chem. Soc.*, 77, 3951 (1955).
(3) H. Greenfield, H. W. Sternberg, R. A. Friedel, J. Wotiz, R. Markby and I. Wender, *J. Am. Chem. Soc.*, 78, 120 (1956).
(4) H. W. Sternberg, J. G. Shukys, C. D. Donne, R. Markby, R. A. Friedel and I. Wender, *J. Am. Chem. Soc.*, 81, 2339 (1959).
(5) M. U. Cohen, *Rev. Sci. Inst.*, 6, 68 (1935).
(6) M. U. Cohen, *Rev. Sci. Inst.*, 7, 155 (1936).
(7) M. U. Cohen, *Z. Krist.* (A), 94, 288 (1936).

was a 4×4 matrix for each atom and the overall scale-factor was obtained from a 2×2 matrix formed with the overall temperature factor. The individual values of b_i were corrected for their interaction with the overall scale-factor.[8] Six cycles of this treatment reduced $R = \Sigma||F_o|-|F_c||/\Sigma|F_o|$ from 25.6% to 11.2% (non-zero F_o terms only) and 28.5% to 12.5% (all terms), at which stage the shifts were small (~0.01 Å). The cobalt atoms were refined anisotropically and the R values were reduced to 10.4 and 11.7 respectively. Full anisotropic refinement of the whole molecule was attempted and whilst this reduced R still further, to 8.8 and 10.1% respectively, we concluded that the intensity data did not warrant this analysis since the *bij* of some of the light atoms then corresponded to complex thermal vibrational modes.

Discussion

Positional and thermal parameters are listed in Table I together with standard deviations for similar atoms. These values lead to the following average standard deviations in bond lengths and angles:

	σ (Å)		σ (°)
Co-Co	0.00_5	Co-C-O	2.3
Co-C	0.02_5	Co-C-Co	1.2
C-O	0.03_2	lactone ring	2.0
C-C	0.03_1		

The more important distances and angles are shown in Figs. 2 and 3 whilst Fig. 1 shows the molecule in perspective.

Table I.

	x/a	y/b	z/c	B
Co_1	0.3972	.7657	.1803	
Co_2	.4506	.5084	.2543	
C_3	.1797	.8153	.2072	4.90
O_4	.0476	.8691	.2234	6.42
C_5	.5661	.9477	.2024	6.14
O_6	.6666	.0542	.2184	7.47
C_7	.2561	.7473	.0236	5.10
O_8	.1597	.7423	-.0805	7.21
C_9	.5632	.6141	.1596	3.88
O_{10}	.6738	.5838	.1184	5.13
C_{11}	.3419	.3365	.1404	5.35
O_{12}	.2780	.2397	.0677	6.86
C_{13}	.6612	.4248	.3535	4.28
O_{14}	.7993	.3644	.4229	5.82
C_{15}	.2432	.4936	.2967	3.88
O_{16}	.1134	.4822	.3251	6.17
C_{17}	.5457	.7270	.3493	3.89
O_{18}	.4578	.7629	.4272	3.78
C_{19}	.6186	.8377	.5349	5.08
O_{20}	.5737	.8746	.6166	5.19
C_{21}	.8088	.8328	.5255	4.44
C_{22}	.7601	.7691	.4122	4.02

Averaged values of the standard deviations for similar atoms

σ_{cobalt} = .003 Å; σ_{carbon} = .025 Å; σ_{oxygen} = .020 Å

Allowance was made for anisotropic thermal motion of the cobalt atoms by the expression

$$\exp -(b_{11}h^2+b_{22}k^2+b_{33}l^2+b_{12}hk+b_{13}hl+b_{23}kl)$$

	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
Co_1	.0204	.0080	.0065	.0033	.0106	.0011
Co_2	.0199	.0077	.0062	—.0015	.0100	—.0004

(8) A. A. Hock and O. S. Mills, *Acta Cryst.*, *14*, 139 (1961).

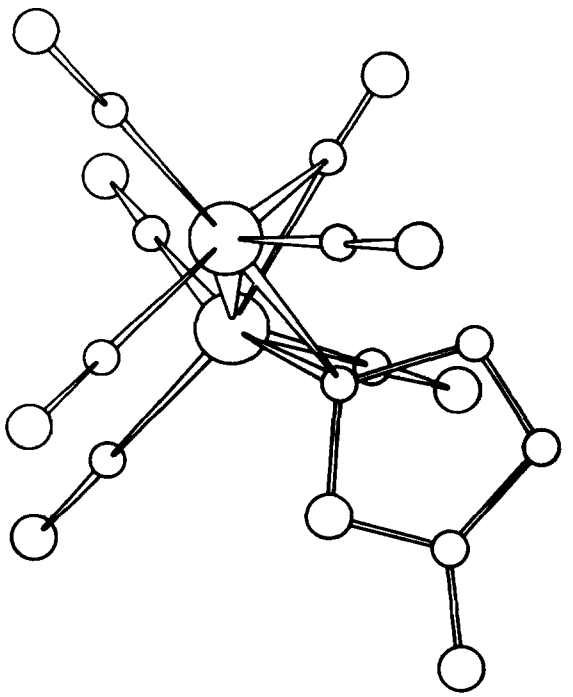


Figure 1. Perspective view of the molecule.

The carbonyl groups fall into four types. Six occur as linear, terminal carbonyls which form, with the two cobalt atoms, two approximately trigonal $Co(CO)_3$ groups oriented in an eclipsed configuration similar to the $Fe(CO)_3$ groups in iron enneacarbonyl.[9] One carbonyl is a bridging group and is staggered with respect to the terminal carbonyl groups. The remaining two carbonyl groups have combined with the original acetylene to form a γ-lactone ring which bridges the metal atoms. Within experimental error this ring is planar and the least-squares plane defined by the lactone ring and the bridging carbonyl group is perpendicular to the cobalt-cobalt vector. The molecular symmetry is *m*. None of the atoms which define this plane are more than 0.05 Å (~ 2σ) from the plane.

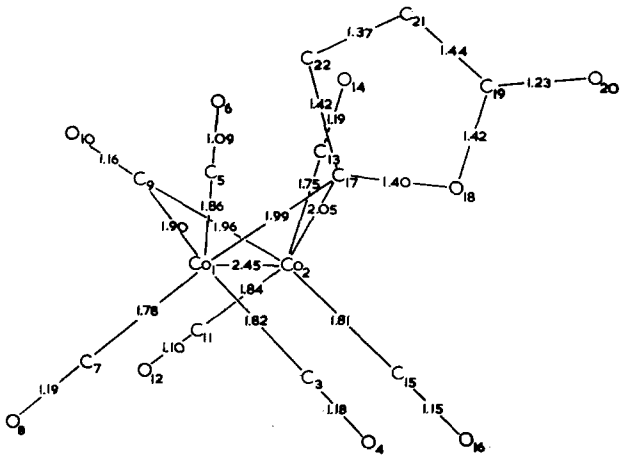


Figure 2. Interatomic distances.

(9) H. M. Powell and R. V. G. Ewens, *J. Chem. Soc.*, 286 (1939).

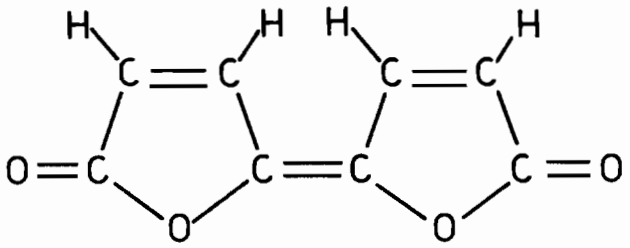
The values listed are h, k, l, $10F_o$ and $10F_c$. The common value of h and k is listed at the head of each group of reflections with varying l.

l	$10F_o$	$10F_c$
0 0		
1	63	-13
2	609	-732
3	422	-466
4	35	36
5	360	352
6	146	-122
7	64	-43
8	71	-61
9	65	-51
10	54	-63
11	99	83
12	101	101
0 1		
0	483	-519
1	442	531
2	580	615
3	694	-728
4	544	-497
5	151	142
6	343	309
7	0	-29
8	74	-44
9	89	-88
10	167	148
11	115	-55
12	23	13
-1	550	-546
-2	114	95
-3	53	55
-4	572	574
-5	575	589
-6	56	-64
-7	478	-472
-8	285	-299
-9	118	134
-10	134	142
-11	0	12
-12	180	-197
0 2		
0	381	-350
1	60	-76
2	671	-706
3	216	249
4	291	268
5	315	-311
6	157	-130
7	125	92
8	352	328
9	81	-31
10	37	-59
11	81	-75
-1	577	-544
-2	797	-737
-3	529	543
-4	567	516
-5	343	-354
-6	386	-343
-7	31	-10
-8	364	352
-9	194	193
-10	162	-171
-11	51	-36
-12	33	-30
0 3		
0	795	-768
1	152	-105
2	757	760
3	106	-85
4	37	26
5	228	193
6	35	-38
7	169	-167
8	168	-157
9	117	131
10	61	84
11	50	-40
-1	608	-569
-2	70	-40
-3	82	-11
-4	816	-787
-5	172	-131
-6	351	321
-7	268	259
-8	34	13
-9	29	-48
-10	34	17
-11	18	-37
-12	35	20
0 4		
0	627	619
1	60	-49
2	433	-398
3	150	108
4	630	587
5	0	39
6	197	-83
7	96	71
8	179	-135
9	48	-35
10	44	-51
-1	523	436
-2	104	-67
-3	0	-7
-4	30	15
-5	89	48
-6	50	-39
-7	74	-64
-8	0	-22
-9	183	-138
-10	0	-15
-11	58	72
0 5		
0	72	-68
1	335	297
2	238	225
3	167	-163
4	543	-506
5	90	91
6	144	169
7	70	87
8	57	-71
9	34	-18
-1	192	-172
-2	326	297
-3	430	-388
-4	133	109
-5	174	161
-6	216	199
-7	97	-86
-8	269	-284
-9	0	8
-10	108	129
-11	23	22
0 6		
0	87	-60
1	122	-82
2	115	-87
3	167	166
4	155	143
5	269	-274
6	88	-121
7	132	131
8	185	184
-1	215	-240
-2	181	-190
-3	0	29
-4	399	390
-5	33	-29
-6	200	-196
-7	79	-68
-8	122	122
-9	104	100
-10	28	-22
0 7		
0	242	-257
1	136	-127
2	130	149
3	35	35
4	0	9
5	27	32
6	102	71
7	150	-182
-1	34	73
-2	294	262
-3	137	157
-4	286	-285
-5	194	-182
-6	34	37
-7	79	69
-8	50	75
0 8		
0	277	275
1	50	-77
2	74	-151
3	28	86
4	124	154
5	25	7
-1	87	107
-2	104	-116
-3	51	-69
-4	60	-57
-5	114	131
-6	32	-28
-7	72	78
0 9		
0	49	-45
1	77	118
2	48	61
-1	0	-54
-2	50	36
-3	27	2
-4	57	-53
1 0		
0	861	-1237
1	318	-328
2	318	339
3	555	578
4	0	27
5	279	-302
6	0	2
7	251	-243
8	93	-101
9	69	87
10	164	183
11	739	-731
-1	316	392
-2	630	713
-3	81	61
-4	53	25
-5	236	-220
-6	33	42
-7	182	179
-8	37	-21
-9	173	178
-10	21	6
-11	100	-119
-12	81	-94
-13	78	116
1 1		
0	614	687
1	187	206
2	754	-737
3	551	-597
4	672	636
5	332	398
6	371	-335
7	131	-137
8	29	42
9	47	47
10	4	-23
11	98	98
-1	539	606
-2	448	-445
-3	393	432
-4	0	24
-5	0	-30
-6	147	-162
-7	280	295
-8	147	148
-9	166	-147
-10	184	202
-11	0	47
-12	195	222
-13	16	-17
1 -1		
0	132	110
1	331	408
2	67	83
3	263	-260
4	96	79
5	340	-359
6	244	-260
7	215	212
8	331	373
9	90	-107
10	168	-191
11	88	-108
12	502	-558
-2	280	-272
-3	382	337
-4	261	223
-5	421	-338
-6	326	-292
-7	120	-85
-8	283	262
-9	109	97
-10	152	-140
-11	54	43
-12	0	-23
1 2		
0	0	5
1	660	666
2	279	285
3	184	-165
4	609	-633
5	114	94
6	552	509
7	148	122
8	270	-265
9	0	5
10	34	63
11	0	14
-1	148	-129
-2	574	-497
-3	441	-388
-4	83	-46
-5	207	186
-6	354	368
-7	90	-77
-8	309	-269
-9	0	-15
-10	166	153
-11	0	-28
-12	48	-46
-13	24	-25
1 -2		
0	0	13
1	529	-658
2	859	1034
3	83	126
4	541	-551
5	22	38
6	380	390
7	261	258
8	243	-263
9	70	-84
10	29	17
11	79	72
-1	437	-449
-2	101	74
-3	17	19
-4	123	-119
-5	348	-331
-6	226	197
-7	294	-270
-8	330	-315
-9	55	58
-10	142	148
-11	0	12
-12	78	-76
1 3		
0	69	29
1	363	309
2	178	-179
3	172	159
4	97	-92
5	51	24
6	222	-182
7	144	147
8	98	119
9	90	-86
10	168	-165
-1	1069	-1093
-2	115	125
-3	324	321
-4	238	287
-5	86	-95
1 -3		
0	355	409
1	297	316
2	223	-196
3	327	-348
4	227	212
5	268	247
6	166	-192
-1	50	23
-2	102	-85
-3	0	-4
-4	0	2
-5	210	-188
-6	107	89
-7	219	194
-8	153	89
-9	208	-198
-10	49	-69
-11	102	108
-12	109	123
2 0		
0	110	79
1	84	52
2	333	-345
3	0	18
4	60	-72
5	240	232
6	52	-98
7	18	-6
8	187	198
9	52	-53
10	181	-176
-1	340	-360
-2	672	-796
-3	290	-336
-4	481	500
-5	19	-19
-6	87	-45
-7	25	4
-8	129	-112
-9	33	4
-10	74	58
-11	166	186
-12	27	22
-13	126	-134
2 1		
0	0	9
1	133	142
2	329	305
3	475	502
4	261	-256
5	341	-326
6	56	-68
7	132	130
8	0	-23
9	0	29
10	0	24
-1	193	-197
-2	516	508
-3	274	-293
-4	563	-546
-5	135	152
-6	68	-61
-7	348	-309
-8	120	100
-9	263	255
-10	176	169
-11	53	-68
-12	139	-183
-13	76	92
2 -1		
0	73	-119
1	449	-464
2	176	-188
3	75	93
4	99	33
5	72	-94
6	241	217
7	80	62
8	119	-133
9	0	-32
10	92	122
-1	366	372
-2	271	280
-3	467	-476
-4	570	-548
-5	767	761
-6	510	454
-7	30	-32
-8	409	-374
-9	56	-53
2 2		
0	209	191
1	555	-536
2	168	-162
3	74	-53
4	430	436
5	215	215
6	284	-283
7	252	-257
8	87	74
9	100	101
-1	429	409
-2	51	33
-3	894	726
-4	199	187
-5	401	-336
-6	242	-212
-7	142	112
-8	337	311
-9	286	-270
-10	182	-173
-11	5	-19
-12	77	97
-13	30	36
2 -2		
0	294	314
1	30	11
2	372	-387
3	201	-223
4	296	312
5	169	85
6	227	-206
7	143	-154
8	116	125
9	97	97
10	0	-21
-1	677	733
-2	568	535
-3	69	-56
-4	83	-78
-5	354	-377
-6	168	-139
-7	485	480
-8	0	41
-9	312	-295
-10	146	-137
-11	34	47
-12	132	134
2 -3		
0	526	-514
1	221	-194
2	302	286
3	546	606
4	93	96
5	358	-387
6	0	-2
-1	135	130
-2	308	297
-3	72	54
-4	257	-229
-5	200	-147
-6	195	179
-7	293	-273
3 0		
0	152	-124
1	234	-210
2	49	71
3	0	-19
4	141	142
5	54	-46
6	112	101
7	33	20
8	69	-72
9	78	9
-1	672	706
-2	156	-179
-3	360	-348
-4	571	-646
-5	156	150
-6	272	299
-7	180	-197
-8	114	83
-9	0	-2
-10	25	-13
-11	102	-110
-12	21	-8
-13	114	147
3 1		
0	630	597
1	208	185
2	219	-195
3	344	-324
4	166	177
5	226	226
6	96	-88
7	35	-45
8	78	-69
9	0	-21
-1	190	-90
-2	560	-531
-3	79	-87
-4	264	253
-5	0	-22
-6	136	150
-7	145	114
-8	202	-189
-9	172	-159
-10	0	20
-11	121	140
-12	105	110
-13	121	-142
3 -1		
0	49	-50
1	0	-27
2	186	-181
3	98	75
4	225	224
5	79	65
6	121	-115
7	74	-48
8	76	94
9	102	100
-1	339	-332
-2	53	51
-3	606	605
-4	219	253
-5	259	-268
-6	138	-153
-7	203	182
-8	310	286
-9	101	-79
-10	235	-221
-11	56	69
-12	0	-15
-13	43	35
3 2		
0	0	24
1	0	8
2	252	225
3	22	15
4	352	-351
5	179	-184
6	72	77
7	120	140
8	0	-12
-1	69	-49
-2	113	112
-3	174	-150
-4	294	258
-5	483	413
-6	64	-67
-7	471	-339
-8	271	-282
-9	378	334
-10	197	192
-11	99	-121
-12	22	-47
-13	0	-5
3 -2		
0	155	-128
1	66	-90
2	392	323
3	244	240
4	396	-418
5	159	-152
6	0	-21
7	96	77
8	46	57
9	110	-112
-1	186	-169
-2	149	-140
-3	81	-62
-4	387	358
-5	157	154
-6	126	-90
-7	382	-352
-8	0	-44
-9	475	474
-10	95	91
-11	157	-145
-12	165	-154
-13	46	43
4 0		
0	97	-97
1	144	166
2	91	69
3	51	32
4	0	9
5	40	40
6	109	-130
7	18	-6
-1	340	-364
-2	141	162
-3	428	493
-4	322	345
-5	216	-199
-6	292	-302
-7	269	253
-8	107	106
-9	97	-97
-10	48	54
-11	84	-88
-12	59	-76
-13	79	-67
4 1		
0	201	-191
1	297	-288
2	100	-113
3	345	346
4	87	-36
5	80	-75
6	0	33
7	87	-14
-1	140	118
-2	326	330
-3	187	-186
-4	128	-89
-5	0	-10
-6	214	202
-7	225	-195
-8	164	168
-9	173	116
-10	156	-177
-11	127	-161
-12	0	8
-13	100	137
4 -1		
0	97	84
1	86	49
2	185	180
3	0	1
4	241	-233
5	122	-137
6	66	65
7	141	136
-1	251	244
-2	80	-76
-3	243	-238
-4	0	29
-5	333	318
-6	304	-293
-7	100	-101
-8	257	-252
-9	301	282
-10	186	176
-11	74	-72
-12	0	35
-13	25	-18
4 2		
0	248	232
1	145	130
2	220	-206
3	244	-218
4	185	174
5	135	142
6	35	-46
7	93	-116
-1	93	-88
-2	136	-120
-3	216	-205
-4	127	-134
-5	268	-242
-6	0	10
-7	423	365
-8	99	79
-9	307	-306
-10	0	8
-11	132	165
-12	17	30
-13	0	-22
4 -2		
0	55	-64
1	268	268

2	196	-181		5	0		-13	94	-109	-13	68	110	1	130	-125	-4	0	-18	
3	226	-236	0	183	176		5	-1			5	-2	2	141	-126	-5	164	162	
4	168	195	1	147	-171	0	112	108	0	173	-162	3	38	47	-6	0	32		
5	100	86	2	0	-24	1	56	2	1	222	-198	-1	273	279	-7	42	-30		
6	75	86	3	71	-60	2	185	-193	2	58	60	-2	138	-114	-8	50	-63		
7	138	-103	4	46	-52	3	39	-16	3	228	241	-3	191	-158	-9	150	-167		
-1	0	2	5	33	-31	4	50	49	4	36	21	-4	0	-56	-10	134	153		
-2	241	-266	-1	177	169	5	136	140	5	96	-82	-5	170	159	-11	114	138		
-3	26	-54	-2	99	-81	-1	28	0	-1	250	243	-6	135	100	-12	19	-33		
-4	310	-271	-3	374	-354	-2	170	155	-2	134	121	-7	27	-21		6	-2		
-5	80	-94	-4	114	122	-3	60	70	-3	97	-86	-8	0	-45	0	51	58		
-6	277	235	-5	462	512	-4	318	-321	-4	52	-58	-9	25	-20	1	110	120		
-7	293	265	-6	120	129	-5	320	-302	-5	38	-32	-10	0	-58	2	125	105		
-8	149	-121	-7	252	-241	-6	338	303	-6	223	-210	-11	29	-58	-1	191	-172		
-9	406	-387	-8	137	-132	-7	157	126	-7	125	-114	-12	89	115	-2	0	8		
-10	0	-3	-9	35	-31	-8	0	12	-8	181	176		6	-1	-3	135	124		
-11	278	297	-10	76	73	-9	256	-268	-9	115	108	0	70	-67	-4	80	-52		
-12	52	47	-11	54	46	-10	0	-32	-10	0	-9	1	78	-84	-5	0	49		
-13	49	-55	-12	54	67	-11	78	79	-11	180	-170	2	99	93	-6	92	106		
	4	-3	-13	30	19	-12	0	14	-12	31	2	3	106	110	-7	0	16		
0	204	-211		5	1		5	2		6	0	-1	0	-40	-8	0	-44		
1	421	-435	0	40	41	0	208	-179	0	107	-112	-2	0	-48	-9	84	-82		
2	53	20	1	224	226	1	175	-179	1	0	8	-3	69	-71	-10	73	92		
3	237	219	2	114	125	2	141	153	2	31	-24	-4	233	244	-11	93	87		
4	108	100	3	175	-184	3	207	200	3	14	-7	-5	80	68	-12	42	-39		
5	62	-59	4	27	-6	4	0	-9	-1	0	-24	-6	152	-161		7	0		
6	0	-8	5	15	9	5	69	-91	-2	206	215	-7	88	-93	0	42	55		
-1	332	337	-1	293	-298	-1	108	105	-3	59	40	-8	27	-15	-1	0	-3		
-2	273	253	-2	158	-141	-2	41	20	-4	161	-150	-9	147	158	-2	112	-119		
-3	103	-99	-3	253	220	-3	23	-31	-5	276	-317	-10	0	7	-3	71	-51		
-4	305	-286	-4	123	116	-4	106	91	-6	0	0	-11	95	-103	-4	45	38		
-5	170	-127	-5	103	-86	-5	44	-41	-7	162	177	-12	34	-22	-5	185	185		
-6	188	149	-6	28	-16	-6	175	-172	-8	24	8		6	2	-6	66	-76		
-7	91	82	-7	0	55	-7	121	-131	-9	46	52	0	43	33	-7	195	-199		
-8	102	101	-8	0	-14	-8	64	67	-10	92	-98	1	196	194	-8	42	26		
-9	167	145	-9	111	86	-9	247	281	-11	51	54	2	0	-20	-9	41	40		
-10	218	-202	-10	106	133	-10	156	-168	-12	55	-58	-1	93	-99	-10	16	25		
-11	93	-111	-11	62	69	-11	98	-119		6	1	-2	59	-46	-11	19	-15		
-12	0	50	-12	125	-159	-12	27	-37	0	81	80	-3	37	75		7	1		

0	54	-66	-2	44	54														
-1	106	-120	-3	89	-95														
-2	27	66	-4	26	33														
-3	149	160	-5	26	35														
-4	0	15	-6	0	11														
-5	78	-76	-7	76	72														
-6	110	-97	-8	35	46														
-7	53	24	-9	0	5														
-8	29	-44	-10	152	-152														
-9	0	-12																	
-10	73	86																	
-11	0	-10																	
	7	-1																	
0	50	16																	
-1	54	47																	
-2	0	-16																	
-3	0	16																	
-4	108	120																	
-5	77	-57																	
-6	163	155																	
-7	95	99																	
-8	94	-85																	
-9	107	-108																	
-10	127	97																	
-11	105	118																	
	7	2																	
-1	74	105																	
-2	0	21																	
-3	76	-90																	
-4	0	5																	
-5	0	-9																	
-6	0	4																	
-7	0	19																	
-8	115	138																	
-9	88	94																	
-10	100	-137																	
	7	-2																	
0	31	7																	
-1	0	27																	

The metal atoms are bonded together with a separation of 2.45 Å. Though this is less than that reported for $Co_2(CO)_8$[10] it is in close agreement with values found in the orthorhombic modification of $Co_2(CO)_9HCCH$[11], $Co_2(CO)_4(Bu^tCCH)_2HCCH$[12] and in $Co_4(CO)_{10}(C_2H_5CCC_2H_5)$[13] which are 2.43 and 2.45 Å in the first two cases and 2.43 and 2.55 in the last. The bond lengths in the lactone ring are in good agreement with those reported for similar ring compounds and correspond formally to a double bond between atoms C_{21} and C_{22}. The presence of this synthesised lactone ring is in accord with the independent observations of Albanesi and Tovaglieri[14] and of Sauer *et al.*[15] who report that cobalt octacarbonyl to form bifurandione



The synthesis of this di-lactone is rational from the moiety present in our structure. It is significant that, in the structure reported here, the acetylene residue is no longer bonded directly to the metal atoms, as was the case in the precursor,[16] but is separated by one molecule of carbon monoxide.

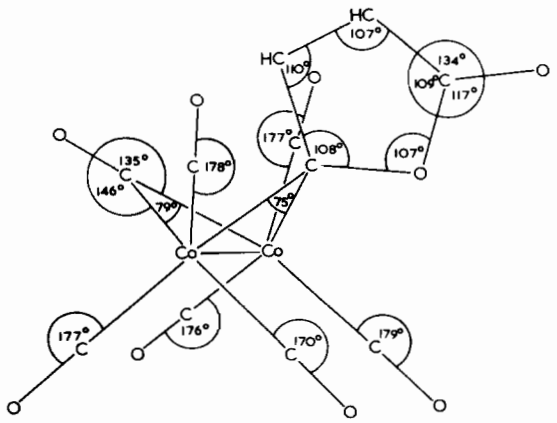


Figure 3. Interatomic angles. In addition to those shown in the diagram the following are also useful:

Co1-C17-C22	120	Co2-C17-C22	117
Co1-C17-O18	119	Co2-C17-O18	115

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