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The X-ray Structure of Bis(tricobalt enneacarbonyl)acetone

BY G. ALLEGRA AND S. VALLE

Istituto di Chimica Industriale del Politecnico e Centro Nazionale di Chimica delle Macromolecole, Sez. 1, Piazza L. da Vinci, 32, Milano, Italy

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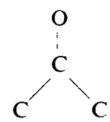
The crystal and molecular structure of a complex with the formula $[Co_3(CO)_9C]_2CO$ (space group $P2_12_12_1$; $a=31{\cdot}35\pm0{\cdot}13$, $b=9{\cdot}87\pm0{\cdot}04$, $c=9{\cdot}87\pm0{\cdot}04$ Å; $Z=4$) has been elucidated through application of a new method for sign determination to the X-ray data on two-dimensional projections. The complex turns out to be bis(tricobalt enneacarbonyl)acetone; the two (Co_3C) tetrahedra show seemingly significant differences in the lengths of the Co–C bonds and also in the two central C–C bonds (1·60 and 1·42 Å). The central



group is strictly planar and is differently oriented with respect to the two tetrahedra; the distorted tetrahedral coordination around the C atoms departs from threefold symmetry as one of the three Co–C–C angles ($\sim150°$) strongly exceeds the value of the other two ($\sim120°$) in order to reduce the intramolecular steric hindrance between the (CO) groups belonging to opposite tetrahedra.

Introduction

In recent years the synthesis and properties of tricobalt enneacarbonyl methane and of some of its derivatives [$Co_3(CO)_9CX$ where X = H, halogen, COOR ...] have been reported (Dent, Duncanson, Guy, Reed & Shaw, 1961; Ercoli, Santambrogio & Tettamanti Casagrande, 1962; Bor, Markó & Markó, 1962). Recently the synthesis and crystal structure of the first example of a bis adduct have been concisely described (Allegra, Mostardini Peronaci & Ercoli, 1966), namely bis(tricobalt enneacarbonyl) acetone (I; Fig. 1). This molecule, whose structural resolution has been performed through the application of a new method for sign determination (Allegra, 1965) to X-ray data on two two-dimensional crystallographic projections, contains two identical $(OC)_9Co_3C$– units joined together by a binding CO group. Together with the case of $Co_3(CO)_9CCH_3$ (Sutton & Dahl, 1967), it represents the first example of a detailed structural resolution for molecules containing such units.

It is the purpose of the present paper to discuss in more detail both the approach to sign determination that has been used and the molecular geometry of (I), as derived from the three-dimensional X-ray refinement.

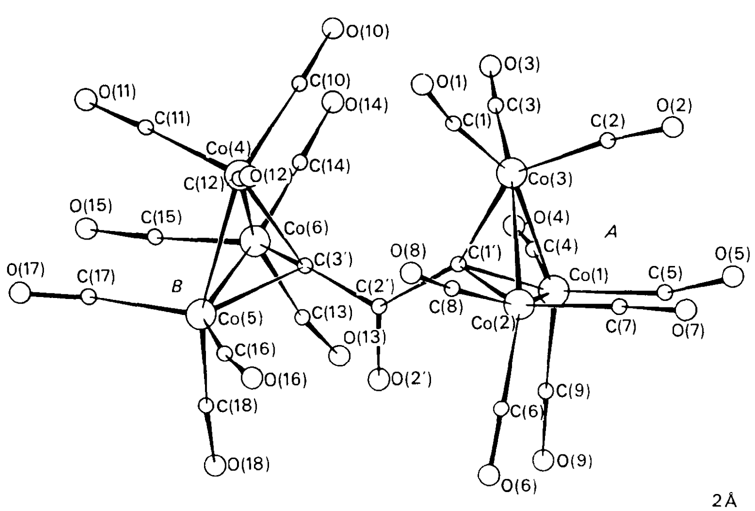


Fig. 1. Molecular conformation of bis(tricobalt enneacarbonyl)acetone, from the X-ray refinement.

Preliminary data

(I) has been synthesized by warming an anhydrous toluene solution of $(OC)_9Co_3CBr$ at about 90°C; it appears as a brown crystalline precipitate, m.p. = 125°C with decomposition, together with other higher-melting products. Good crystals of (I) may be precipitated from a toluene solution (Allegra *et al.*, 1966).

Three-dimensional (*hkl*) intensities were collected by the usual multiple film Weissenberg equi-inclination

Table 1. *The 7 most probable sign combinations for the 24 hk0 and h0l base reflexions, and the corresponding Π values*

				(*hk*O) reflexions					
(*hk*)	(16 7)	(9 6)	(10 4)	(14 3)	(15 1)	(12 6)	(15 5)	(14 5)	
|*E*|	2·40	2·03	2·38	2·35	2·14	2·77	2·60	2·38	
1st combination	+	+	−	+	−	+	+	−	
2nd combination	+	+	−	+	−	+	+	−	
3rd combination	+	+	+	−	−	−	−	−	
4th combination	+	+	−	+	−	+	+	−	
5th combination	+	+	+	−	−	−	−	−	
6th combination	+	+	+	−	−	−	−	−	
7th combination	+	+	−	+	−	+	+	−	
(*hk*)	(18 4)	(18 2)	(5 5)	(6 5)	(30 2)	(31 2)	(27 2)	(1 6)	
|*E*|	2·25	1·85	1·80	1·77	2·24	2·19	2·11	1·75	
1st combination	+	−	+	+	+	+	+	−	
2nd combination	+	−	+	+	+	+	−	−	
3rd combination	−	−	−	+	+	−	−	−	
4th combination	+	−	+	+	+	+	+	−	
5th combination	−	−	−	+	+	−	+	−	
6th combination	−	−	−	+	+	−	−	−	
7th combination	+	−	+	+	+	+	−	−	
(*hk*)	(21 6)	(25 1)	(3 7)	(24 3)	(20 0)	(4 7)	(19 8)	(17 1)	*Π*
|*E*|	1·84	1·72	1·63	1·90	1·86	1·65	1·63	1·61	
1st combination	−	−	−	−	+	−	+	+	255·3
2nd combination	−	−	−	−	+	−	+	+	253·3
3rd combination	−	−	+	−	+	−	+	+	247·1
4th combination	−	−	−	+	+	−	+	+	242·7
5th combination	−	−	+	−	+	−	+	+	233·1
6th combination	−	−	−	−	+	−	+	+	227·5
7th combination	−	−	−	+	+	−	+	+	226·0
				(*h*O*l*) reflexions					
(*hl*)	(15 4)	(4 5)	(8 4)	(12 5)	(14 1)	(22 1)	(10 6)	(13 5)	
|*E*|	2·11	2·28	2·66	2·26	1·97	2·24	2·47	2·31	
1st combination	+	+	+	−	+	−	−	+	
2nd combination	+	+	−	−	−	+	+	−	
3rd combination	+	+	+	−	+	−	−	+	
4th combination	+	+	−	−	−	+	+	−	
5th combination	+	+	−	−	−	+	+	−	
6th combination	+	+	−	−	−	+	+	+	
7th combination	+	+	−	−	−	+	+	−	
(*hl*)	(18 6)	(18 2)	(14 5)	(16 7)	(4 7)	(16 1)	(18 4)	(17 5)	
|*E*|	2·71	2·17	1·82	1·92	1·90	1·75	1·81	1·76	
1st combination	+	−	−	+	−	+	−	−	
2nd combination	−	−	−	+	−	−	+	+	
3rd combination	+	−	−	+	−	+	−	−	
4th combination	−	−	−	+	−	−	+	+	
5th combination	−	−	−	−	−	−	+	+	
6th combination	−	−	−	+	−	−	+	+	
7th combination	−	−	−	+	−	−	+	+	
(*hl*)	(32 1)	(2 9)	(27 4)	(20 0)	(3 6)	(22 5)	(5 10)	(28 4)	*Π*
|*E*|	2·07	2·15	1·78	1·85	1·62	1·62	1·75	1·63	
1st combination	+	+	−	+	+	+	−	+	248·3
2nd combination	−	−	−	+	+	+	+	−	220·3
3rd combination	+	+	+	+	+	+	−	+	214·9
4th combination	−	+	−	+	+	+	+	−	211·0
5th combination	−	−	−	+	+	+	+	−	208·4
6th combination	−	+	+	+	+	+	+	−	204·9
7th combination	−	−	+	+	+	+	+	−	198·6

technique, using the Co $K\alpha$ radiation. The absorption factor for the crystals investigated, which were approximately cylindrical in shape, was estimated to be about $\mu R = 1{\cdot}8$. This has been neglected, and therefore the corresponding effect is included in the average thermal factor. By visual estimation, 1653 observable intensities were measured, out of 2206 included in the limiting sphere (layers with $k=0, \ldots 4$ and $l=0, \ldots 4$). The orthorhombic unit cell has the following dimensions:

$$a = 31{\cdot}35 \pm 0{\cdot}13;\ b = 9{\cdot}87 \pm 0{\cdot}04;\ c = 9{\cdot}87 \pm 0{\cdot}04\ \text{Å};$$
$$Z = 4;\ D_{\text{calc}}(\simeq D_{\text{exp}}) = 1{\cdot}98\ \text{g.cm}^{-3}.$$

The space group is $P2_12_12_1$, from the systematic extinction of the odd index reflexions on the reciprocal lattice axes. Therefore, the whole molecule represents the asymmetric unit of the structure.

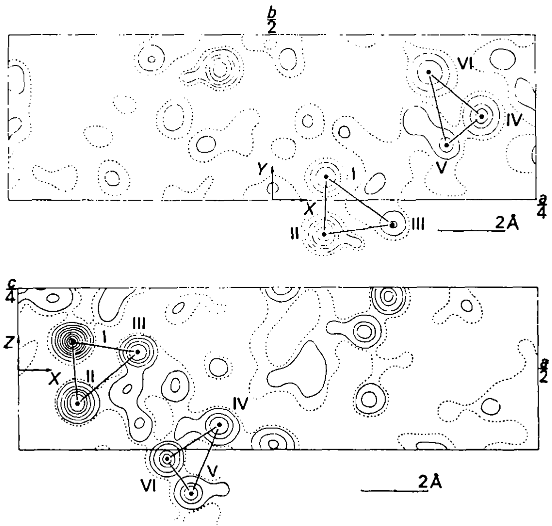


Fig. 2. *ab* (above) and *bc* (below) electron density projections, obtained from the statistical sign determination. Contours are drawn at arbitrary levels; the symbols I to VI attached to the heaviest peaks correspond to the 6 cobalt atoms (see text).

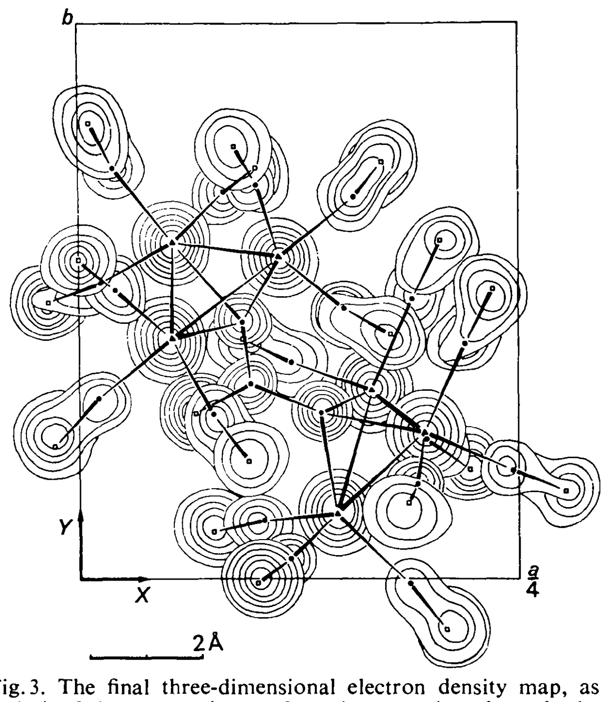


Fig. 3. The final three-dimensional electron density map, as derived from superimposed sections passing through the atomic centres. Contours are drawn at 2,4,6...e.Å^{-3} around the C (black circles) and the O (squares) atoms; at 5,10,15...e.Å^{-3} around the Co atoms (black triangles).

Method of solution

We first tried to solve the structure from the (*ab*) and (*ac*) 'sharpened' Patterson projections, but any attempt to identify coherently the 78 independent Co–Co vectors was unsuccessful. The same projections were then investigated from the viewpoint of statistical methods; even if the space group is acentric, they are centrosymmetrical and the projecting cell edges are short enough (9·87 Å in both cases) as to suggest improbable overlap among Co atoms.

In a paper on the phase problem (Allegra, 1965), a new joint probability distribution of a set of structure factor signs is proposed [expression (10), quoted paper]; it shows analogies with other general expressions, such as those given by Bertaut [1955, expression (III·1)], and also by Naya, Nitta & Oda [1964, expressions (1) and (25)]. Two general formulae of possible practical interest were derived from the proposed probability distribution (Allegra, 1965): expression (18) for the probability of a single sign, and expression (23), which for our purposes can be written as follows [see also expression (25)]:

$$\text{Most probable set of } (s_1s_2\ldots s_m) \equiv \max \Pi(s_1s_2\ldots s_m)$$
$$= \max \left\{ \Sigma\, s_is_js_k\alpha_{ijk} + \Sigma\, s_is_js_ks_l\alpha_{ijn}\alpha_{kln} \right\}, \quad (1)$$

where s_i is the sign of the ith reflexion ($1 \le i \le m$), and α_{ijk} is Woolfson's hyperbolic tangent (Woolfson, 1954) which relates three structure factors whose reciprocal vectors are such that $\mathbf{H}_i + \mathbf{H}_j + \mathbf{H}_k = 0$:

$$\alpha_{ijk} = \text{Th}\ \{E_iE_jE_k\sigma_3/\sigma_2^{3/2}\};\ (\sigma_n = \Sigma f_i^n). \quad (2)$$

Here, contrary to the previous paper (Allegra, 1965), E_i, instead of $|A_i|$, denotes the absolute value of a normalized structure factor, f_i is the scattering factor of the ith atom at rest, and the sum is extended to all the atoms contained in the unit cell.

The criterion of maximizing an expression, related to the sign probability distribution, with respect to all possible combinations of the signs, has already been suggested by other authors (see, for instance, Cochran & Douglas, 1955). However, expression (1) allows the introduction of a comparatively large number of Sayre triads without inceasing too much the number of signs to be permuted; we have given, in the Appendix, a concise description of the computing programs which perform the maximization of (1).

Remembering that $\Pi(s_1 \ldots s_m)$ stands for the expression in parentheses on the right hand side of (1), the sign combination characterized by the highest Π value (see Table 1) was considered both on the (*ab*) and on the (*ac*) projection; the two combinations proved to be coherent between themselves, as long as the signs of the common $0k0$ reflexions were correctly related. The corresponding two-dimensional Fourier syntheses (Fig. 2) proved also to be coherent, since a satisfactory correspondence was observed among the x coordinates of the 6 strongest peaks. Furthermore, examination of the three-dimensional peak coordinates suggested a reasonable pattern of cobalt-to-cobalt intramolecular distances; in fact (see Fig. 2) $d_{\text{I--II}} \simeq d_{\text{II--III}} \simeq d_{\text{III--I}} \simeq d_{\text{IV--V}} \simeq d_{\text{V--VI}} \simeq d_{\text{VI--IV}} = 2{\cdot}50 \pm 0{\cdot}12$ Å, *i.e.* a figure close to the Co–Co bond length found in many cobalt clusters (Corradini, 1959; Gardner Sumner, Klug & Alexander, 1964).

Therefore the six peaks were attributed to the structurally independent cobalt atoms; they appeared to be arranged in two sets of three atoms, each set being an approximately equilateral triangle with sides of 2·50 Å, while the two sets were about 5 Å apart. Progressive two-dimensional Fourier syntheses lead to detection of most of the Co-coordinated carbonyl groups; no bridging carbonyl was detected, in accord with infrared data. Evidence for the C atoms belonging to the Co_3C tetrahedra was also found, but some uncertainty remained as to the bridging group between the two tetrahedra. This was finally resolved by the three-dimensional Fourier synthesis (Fig. 3).

Table 2. *Final list of the fractional coordinates and thermal factors* (Å^2) *and corresponding standard deviations*

	x/a	y/b	z/c	$\bar{B}$	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
CO(1)	0·0526	0·0664	0·0769		5·205	5·621	3·879	0·376	0·228	−0·280
CO(2)	0·0530	−0·1091	−0·1007		4·519	4·468	5·081	−0·202	−0·303	−0·275
CO(3)	0·1149	−0·0827	0·0495		5·275	5·086	4·903	0·029	−0·592	0·605
CO(4)	0·1957	0·2354	−0·1667		5·003	6·086	7·621	−0·326	−0·334	−0·063
CO(5)	0·1660	0·1588	−0·3876		5·570	5·884	5·175	0·005	1·027	−0·296
CO(6)	0·1459	0·3815	−0·2899		5·114	4·445	7·250	0·047	0·910	−0·292
C(1′)	0·0913	0·0327	−0·0710	3·09						
C(2′)	0·0974	0·1491	−0·1820	3·35						
C(3′)	0·1380	0·1999	−0·2201	3·47						
O(2′)	0·0649	0·1996	−0·2331	4·47						
C(1)	0·1558	−0·1900	−0·0406	4·52						
C(2)	0·1016	−0·2129	0·1796	7·02						
C(3)	0·1507	0·0135	0·1548	4·93						
C(4)	0·0766	0·2039	0·1676	4·31						
C(5)	0·0208	−0·0224	0·2078	6·02						
C(6)	0·0120	−0·0367	−0·2125	3·71						
C(7)	0·0189	−0·2434	−0·0125	5·56						
C(8)	0·0817	−0·1995	−0·2356	5·46						
C(9)	0·0127	0·1747	0·0120	5·31						
C(10)	0·1915	0·3293	−0·0067	6·56						
C(11)	0·2458	0·3032	−0·2262	7·20						
C(12)	0·2187	0·0747	−0·1037	8·24						
C(13)	0·1039	0·3953	−0·4183	5·38						
C(14)	0·1192	0·4627	−0·1649	6·88						
C(15)	0·1867	0·5076	−0·3489	7·56						
C(16)	0·1891	−0·0056	−0·3941	5·23						
C(17)	0·1974	0·2482	−0·5082	4·47						
C(18)	0·1203	0·1073	−0·4843	5·79						
O(1)	0·1721	−0·2547	−0·1034	7·18						
O(2)	0·0895	−0·2817	0·2500	8·73						
O(3)	0·1778	0·0590	0·2180	9·52						
O(4)	0·0974	0·2881	0·2227	8·69						
O(5)	−0·0001	−0·0759	0·2758	8·30						
O(6)	−0·0165	−0·0011	−0·2635	5·69						
O(7)	0·0066	−0·3243	0·0426	8·90						
O(8)	0·0997	−0·2443	−0·3123	6·74						
O(9)	−0·0142	0·2359	−0·0280	6·13						
O(10)	0·1863	0·3660	0·0865	11·54						
O(11)	0·2754	0·3427	−0·2724	10·72						
O(12)	0·2346	−0·0231	−0·0681	7·36						
O(13)	0·0754	0·4147	−0·4877	6·86						
O(14)	0·1009	0·5061	−0·0599	6·58						
O(15)	0·2074	0·5905	−0·4015	9·48						
O(16)	0·2056	−0·1102	−0·3582	8·77						
O(17)	0·2217	0·3035	−0·5844	7·38						
O(18)	0·0924	0·0672	−0·5250	6·17						

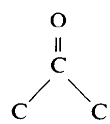
Table 2 (*cont.*)

	$\sigma(x)/a$	$\sigma(y)/b$	$\sigma(z)/c$	$\sigma(\bar{B})$	$\sigma(B_{11})$	$\sigma(B_{22})$	$\sigma(B_{33})$	$\sigma(B_{12})$	$\sigma(B_{13})$	$\sigma(B_{23})$
CO(1)	0·00013	0·00048	0·00046		0·182	0·199	0·186	0·170	0·165	0·228
CO(2)	0·00013	0·00047	0·00046		0·184	0·185	0·185	0·163	0·165	0·213
CO(3)	0·00013	0·00048	0·00046		0·172	0·200	0·191	0·169	0·161	0·232
CO(4)	0·00017	0·00062	0·00064		0·257	0·279	0·293	0·236	0·236	0·306
CO(5)	0·00014	0·00054	0·00052		0·204	0·232	0·219	0·190	0·190	0·251
CO(6)	0·00014	0·00049	0·00054		0·182	0·200	0·234	0·172	0·186	0·245
C(1′)	0·00069	0·00249	0·00256	0·42						
C(2′)	0·00078	0·00287	0·00282	0·50						
C(3′)	0·00077	0·00273	0·00281	0·47						
O(2′)	0·00062	0·00223	0·00231	0·44						
C(4)	0·00085	0·00304	0·00305	0·54						
C(5)	0·00116	0·00421	0·00412	0·84						
C(6)	0·00088	0·00313	0·00318	0·58						
C(7)	0·00112	0·00417	0·00412	0·79						
C(8)	0·00099	0·00357	0·00366	0·67						
C(9)	0·00093	0·00342	0·00336	0·62						
C(10)	0·00134	0·00487	0·00485	1·06						
C(11)	0·00128	0·00451	0·00475	0·96						
C(12)	0·00142	0·00501	0·00500	1·10						
C(13)	0·00096	0·00352	0·00348	0·65						
C(14)	0·00113	0·00401	0·00393	0·79						
C(15)	0·00126	0·00447	0·00445	0·89						
C(16)	0·00096	0·00342	0·00347	0·63						
C(17)	0·00087	0·00331	0·00331	0·57						
C(18)	0·00103	0·00379	0·00364	0·70						
O(1)	0·00081	0·00281	0·00295	0·61						
O(2)	0·00116	0·00426	0·00433	1·03						
O(3)	0·00110	0·00367	0·00383	0·91						
O(4)	0·00104	0·00384	0·00383	0·87						
O(5)	0·00097	0·00301	0·00296	0·65						
O(6)	0·00072	0·00273	0·00266	0·54						
O(7)	0·00091	0·00322	0·00323	0·71						
O(8)	0·00078	0·00282	0·00272	0·58						
O(9)	0·00083	0·00308	0·00299	0·64						
O(10)	0·00119	0·00423	0·00425	1·03						
O(11)	0·00115	0·00413	0·00418	1·00						
O(12)	0·00080	0·00295	0·00286	0·61						
O(13)	0·00082	0·00297	0·00280	0·61						
O(14)	0·00079	0·00289	0·00277	0·57						
O(15)	0·00100	0·00346	0·00349	0·77						
O(16)	0·00092	0·00328	0·00325	0·71						
O(17)	0·00083	0·00301	0·00297	0·63						
O(18)	0·00076	0·00280	0·00267	0·56						

12 cycles of least-squares refinement reduced the disagreement factor to $R=0·127$ on the 1653 observed reflexions; the block diagonal approximation has been utilized, except in the last two cycles (full matrix) (Immirzi, 1967). With the exception of the cobalt atoms, isotropic thermal factors have been assumed. The independent structural parameters are given in Table 2, together with their standard errors; in Table 3 the full list of observed and calculated structure factors is reported.

Discussion of the structure

The geometrical parameters of the molecule are shown in Fig. 4 and in Table 4. In Table 5 the average values of the different kinds of bond lengths and angles are compared with those found for the analogous compound $Co_3(CO)_9C_2H_3$ (Sutton & Dahl, 1967); the differences appear to be insignificant throughout. In the same Table the average standard deviations (σ), as they result from the least-squares refinement, are also reported; allowance has been made for the standard errors in the unit-cell parameters. With few exceptions, the single standard deviations do not depart from the average by more than 10%. The central



group is strictly planar. Of the two $[CO)_9Co_3C]$ groups, one of them (*i.e.* *B* in Fig. 4) does not deviate from threefold symmetry, within experimental error, while the other shows differences in the Co–C(tetrahedral) distances. Another remarkable difference occurs in the orientation of the central planar group with respect to the Co atoms; while its plane is nearly orthogonal to the Co(1)–Co(2) bond for the *A* group, it is virtually parallel to Co(4)–Co(6) of group *B*; the shortest Co–C tetrahedral bond length (1·81 Å) is contained in the

Table 3. *List of observed and calculated structure factors (electrons/unit cell)*

PH stands for the calculated phase, expressed as a fraction of 2π, multiplied by the factor 10000. The asterisk means 'less than'

H	K	L	FO	FC	PH
0	0	0	* 0	1756	0
2	0	0	41	53	0
4	0	0	111	153	5000
6	0	0	60	64	0
8	0	0	123	124	5000
10	0	0	148	142	0
12	0	0	31	18	0
14	0	0	9	12	5000
16	0	0	42	43	0
18	0	0	104	90	0
20	0	0	136	138	0
22	0	0	* 9	6	5000
24	0	0	47	47	5000
26	0	0	7	11	5000
28	0	0	67	62	5000
30	0	0	41	32	5000
32	0	0	32	25	5000
34	0	0	5	2	0
1	1	0	125	153	2500
2	1	0	178	194	5000
3	1	0	169	193	2500
4	1	0	192	229	0
5	1	0	123	138	2500
6	1	0	49	52	5000
7	1	0	85	88	2500
8	1	0	29	33	5000
9	1	0	* 5	0	2500
10	1	0	120	124	0
11	1	0	28	31	7500
12	1	0	45	41	0
13	1	0	51	33	7500
14	1	0	44	41	0
15	1	0	156	155	7500
16	1	0	79	67	5000
17	1	0	101	91	7500
18	1	0	25	23	0
19	1	0	81	68	2500
20	1	0	54	52	0
21	1	0	53	43	2500
22	1	0	41	29	5000
23	1	0	* 9	8	2500
24	1	0	40	32	0
25	1	0	66	58	2500
26	1	0	19	5	0
27	1	0	53	43	2500
28	1	0	27	15	0
29	1	0	25	21	7500
30	1	0	* 8	7	0
31	1	0	28	22	7500
32	1	0	39	30	5000
33	1	0	25	21	7500
34	1	0	* 5	8	0
0	2	0	40	44	5000
1	2	0	69	74	7500
2	2	0	133	127	0
3	2	0	45	35	2500
4	2	0	* 4	4	5000
5	2	0	104	106	2500
6	2	0	57	59	5000
7	2	0	65	59	7500
8	2	0	87	84	0
9	2	0	61	65	7500
10	2	0	75	89	5000
11	2	0	83	92	2500
12	2	0	85	81	5000
13	2	0	52	45	2500
14	2	0	36	29	5000
15	2	0	59	59	7500
16	2	0	26	23	0
17	2	0	8	6	2500
18	2	0	99	77	0
19	2	0	* 8	12	7500
20	2	0	* 8	1	0
21	2	0	53	43	7500
22	2	0	24	24	0
23	2	0	64	53	2500
24	2	0	* 8	7	5000
25	2	0	* 8	8	2500
26	2	0	* 8	9	5000
27	2	0	62	52	7500
28	2	0	* 8	-4	0
29	2	0	18	16	2500

H	K	L	FO	FC	PH
30	2	0	54	44	5000
31	2	0	44	37	2500
32	2	0	* 5	1	5000
33	2	0	19	13	7500
34	2	0	19	20	0
1	3	0	13	12	2500
2	3	0	68	62	5000
3	3	0	121	109	7500
4	3	0	91	86	5000
5	3	0	43	41	2500
6	3	0	100	111	0
7	3	0	51	61	7500
8	3	0	105	115	5000
9	3	0	62	55	2500
10	3	0	95	74	0
11	3	0	17	24	2500
12	3	0	62	60	0
13	3	0	28	27	7500
14	3	0	150	161	5000
15	3	0	41	38	2500
16	3	0	* 7	7	5000
17	3	0	15	26	2500
18	3	0	36	33	0
19	3	0	45	52	7500
20	3	0	37	27	0
21	3	0	31	22	7500
22	3	0	25	9	0
23	3	0	37	44	2500
24	3	0	65	59	5000
25	3	0	33	27	7500
26	3	0	23	21	0
27	3	0	* 8	2	7500
28	3	0	16	9	0
29	3	0	29	28	2500
30	3	0	10	9	5000
31	3	0	21	15	7500
32	3	0	15	11	0
33	3	0	* 4	5	7500
0	4	0	31	28	5000
1	4	0	93	100	7500
2	4	0	18	14	5000
3	4	0	25	21	2500
4	4	0	98	100	0
5	4	0	15	16	7500
6	4	0	* 6	2	5000
7	4	0	112	108	7500
8	4	0	18	7	5000
9	4	0	52	53	7500
10	4	0	161	178	0
11	4	0	40	39	7500
12	4	0	41	43	0
13	4	0	* 7	14	7500
14	4	0	36	43	5000
15	4	0	24	26	7500
16	4	0	* 8	9	0
17	4	0	44	42	7500
18	4	0	101	96	5000
19	4	0	* 8	3	2500
20	4	0	27	22	5000
21	4	0	56	49	2500
22	4	0	* 9	6	5000
23	4	0	27	27	2500
24	4	0	13	14	5000
25	4	0	31	27	7500
26	4	0	12	11	0
27	4	0	8	10	7500
28	4	0	18	17	5000
29	4	0	14	13	7500
30	4	0	25	21	0
31	4	0	* 5	2	2500
32	4	0	10	7	0
1	5	0	13	17	2500
2	5	0	39	42	0
3	5	0	75	62	7500
4	5	0	70	55	5000
5	5	0	118	119	7500
6	5	0	114	128	5000
7	5	0	30	37	2500
8	5	0	74	64	0
9	5	0	* 4	4	7500
10	5	0	22	26	0
11	5	0	80	70	7500

H	K	L	FO	FC	PH
12	5	0	36	35	0
13	5	0	21	16	2500
14	5	0	119	117	0
15	5	0	125	119	2500
16	5	0	24	12	5000
17	5	0	69	59	2500
18	5	0	30	22	0
19	5	0	65	55	7500
20	5	0	40	36	0
21	5	0	16	15	7500
22	5	0	38	26	5000
23	5	0	* 5	12	7500
24	5	0	* 5	7	5000
25	5	0	43	38	7500
26	5	0	20	17	5000
27	5	0	14	14	7500
28	5	0	13	6	5000
29	5	0	* 3	1	2500
30	5	0	31	21	0
0	6	0	105	104	5000
1	6	0	94	98	2500
2	6	0	44	48	5000
3	6	0	21	20	7500
4	6	0	22	25	0
5	6	0	* 5	3	7500
6	6	0	70	62	0
7	6	0	35	32	7500
8	6	0	40	34	0
9	6	0	102	89	7500
10	6	0	24	26	5000
11	6	0	62	52	7500
12	6	0	128	102	0
13	6	0	9	8	7500
14	6	0	7	9	0
15	6	0	59	60	2500
16	6	0	54	56	5000
17	6	0	13	15	7500
18	6	0	16	15	5000
19	6	0	38	26	2500
20	6	0	54	43	5000
21	6	0	57	55	2500
22	6	0	* 5	3	0
23	6	0	14	9	7500
24	6	0	13	9	0
25	6	0	27	19	7500
26	6	0	14	10	5000
27	6	0	* 3	4	7500
28	6	0	9	10	0
1	7	0	44	45	7500
2	7	0	30	31	5000
3	7	0	76	60	7500
4	7	0	76	69	5000
5	7	0	17	12	7500
6	7	0	39	28	5000
7	7	0	* 5	5	2500
8	7	0	* 5	7	5000
9	7	0	13	1	7500
10	7	0	21	16	0
11	7	0	38	32	7500
12	7	0	* 5	4	0
13	7	0	81	45	2500
14	7	0	19	11	0
15	7	0	32	30	2500
16	7	0	80	44	0
17	7	0	13	12	7500
18	7	0	* 5	5	5000
19	7	0	* 5	9	2500
20	7	0	42	35	5000
21	7	0	20	19	7500
22	7	0	22	15	0
23	7	0	22	14	7500
24	7	0	31	27	5000
25	7	0	11	12	2500
26	7	0	28	19	5000
0	8	0	22	23	0
1	8	0	61	46	2500
2	8	0	39	26	5000
3	8	0	21	23	7500
4	8	0	* 5	6	5000
5	8	0	48	40	7500
6	8	0	50	42	0
7	8	0	24	19	2500

H	K	L	FO	FC	PH
8	8	0	13	8	5000
9	8	0	* 9	12	7500
10	8	0	* 5	2	0
11	8	0	* 5	4	7500
12	8	0	* 5	8	5000
13	8	0	* 5	6	2500
14	8	0	* 5	6	0
15	8	0	47	36	7500
16	8	0	* 4	6	5000
17	8	0	21	15	2500
18	8	0	40	30	5000
19	8	0	40	31	2500
20	8	0	31	22	0
21	8	0	* 3	3	7500
22	8	0	* 3	2	0
23	8	0	10	7	2500
1	9	0	15	14	7500
2	9	0	34	28	5000
3	9	0	21	15	7500
4	9	0	33	29	0
5	9	0	43	32	2500
6	9	0	36	29	0
7	9	0	* 5	2	2500
8	9	0	6	4	0
9	9	0	25	23	7500
10	9	0	* 4	2	5000
11	9	0	31	17	2500
12	9	0	13	17	5000
13	9	0	29	24	7500
14	9	0	7	10	0
15	9	0	30	26	7500
16	9	0	* 3	5	0
17	9	0	23	14	2500
18	9	0	21	16	0
19	9	0	* 2	2	7500
0	10	0	22	16	0
1	10	0	29	18	7500
2	10	0	12	12	0
3	10	0	8	9	7500
4	10	0	8	12	5000
5	10	0	8	7	7500
6	10	0	24	18	5000
7	10	0	6	7	7500
8	10	0	6	9	0
9	10	0	43	28	2500
10	10	0	14	16	5000
11	10	0	32	23	2500
12	10	0	19	11	5000
13	10	0	24	18	7500
1	0	1	128	171	7500
2	0	1	140	181	2500
3	0	1	189	247	2500
4	0	1	168	205	2500
5	0	1	108	112	7500
6	0	1	96	98	2500
7	0	1	22	21	7500
8	0	1	22	25	2500
9	0	1	98	101	2500
10	0	1	68	66	2500
11	0	1	12	19	2500
12	0	1	* 7	12	2500
13	0	1	* 7	2	7500
14	0	1	156	150	7500
15	0	1	76	61	7500
16	0	1	124	108	7500
17	0	1	61	62	2500
18	0	1	14	18	2500
19	0	1	15	13	7500
20	0	1	28	26	2500
21	0	1	51	42	7500
22	0	1	99	89	2500
23	0	1	62	49	2500
24	0	1	20	21	2500
25	0	1	* 11	6	2500
26	0	1	26	26	2500
27	0	1	30	24	2500
28	0	1	34	31	2500
29	0	1	* 10	3	7500
30	0	1	21	17	7500
31	0	1	* 8	4	7500
32	0	1	35	32	7500
33	0	1	14	14	2500

H	K	L	FO	FC	PH
34	0	1	20	16	7500
0	1	1	123	128	7500
1	1	1	245	255	1072
2	1	1	86	81	5317
3	1	1	80	75	8986
4	1	1	118	122	2593
5	1	1	142	153	6732
6	1	1	109	105	9682
7	1	1	107	103	5056
8	1	1	116	109	5973
9	1	1	50	45	3667
10	1	1	111	104	3366
11	1	1	168	159	5030
12	1	1	44	47	7194
13	1	1	30	33	5232
14	1	1	23	27	2081
15	1	1	42	42	1170
16	1	1	53	51	3636
17	1	1	79	79	853
18	1	1	104	92	7630
19	1	1	86	73	9010
20	1	1	36	29	2226
21	1	1	48	38	1165
22	1	1	39	33	3960
23	1	1	22	20	5880
24	1	1	23	25	8515
25	1	1	46	37	7005
26	1	1	33	30	35
27	1	1	* 10	3	5449
28	1	1	21	18	5083
29	1	1	44	35	5556
30	1	1	17	14	4388
31	1	1	42	36	4342
32	1	1	25	20	445
33	1	1	10	7	1612
34	1	1	11	7	7903
0	2	1	104	89	5000
1	2	1	127	105	585
2	2	1	93	64	3476
3	2	1	114	94	8356
4	2	1	107	102	2561
5	2	1	127	122	4094
6	2	1	44	43	9117
7	2	1	28	12	2037
8	2	1	20	16	44
9	2	1	121	106	7655
10	2	1	148	144	788
11	2	1	23	23	814
12	2	1	68	72	8571
13	2	1	56	59	8739
14	2	1	63	68	5023
15	2	1	106	100	4351
16	2	1	102	97	7441
17	2	1	44	47	4520
18	2	1	21	29	9302
19	2	1	59	52	344
20	2	1	81	74	3949
21	2	1	19	19	768
22	2	1	39	35	4807
23	2	1	56	46	5833
24	2	1	43	40	934
25	2	1	23	20	2017
26	2	1	20	15	2132
27	2	1	24	23	9330
28	2	1	* 9	3	3136
29	2	1	* 8	8	4257
30	2	1	40	37	9973
31	2	1	12	14	3504
32	2	1	18	18	8463
33	2	1	15	12	6144
34	2	1	* 4	15	9853
0	3	1	101	108	7500
1	3	1	36	31	7017
2	3	1	191	151	5673
3	3	1	94	77	3712
4	3	1	143	124	2551
5	3	1	165	154	8305
6	3	1	123	107	3968
7	3	1	143	141	7828
8	3	1	92	82	7569
9	3	1	95	103	3279
10	3	1	62	60	8595

H	K	L	FO	FC	PH
11	3	1	36	30	9218
12	3	1	31	36	4516
13	3	1	77	67	2053
14	3	1	36	36	2763
15	3	1	101	95	3060
16	3	1	50	52	8862
17	3	1	53	44	8623
18	3	1	* 9	9	6127
19	3	1	16	19	2899
20	3	1	40	36	5048
21	3	1	41	35	6451
22	3	1	37	30	2040
23	3	1	33	25	8600
24	3	1	22	17	4001
25	3	1	36	35	8527
26	3	1	42	36	6457
27	3	1	24	25	6738
28	3	1	14	16	37
29	3	1	28	27	2229
30	3	1	20	18	705
31	3	1	* 7	5	701
32	3	1	15	11	3662
33	3	1	* 4	2	6408
0	4	1	119	105	5000
1	4	1	35	35	2456
2	4	1	44	42	2343
3	4	1	44	31	7369
4	4	1	117	97	8227
5	4	1	84	79	7454
6	4	1	55	61	6278
7	4	1	116	123	4251
8	4	1	83	83	9900
9	4	1	85	88	7689
10	4	1	66	64	9401
11	4	1	142	140	8899
12	4	1	21	22	524
13	4	1	70	75	2511
14	4	1	68	77	2294
15	4	1	33	30	6858
16	4	1	38	37	2984
17	4	1	45	43	8634
18	4	1	24	31	5852
19	4	1	58	49	1849
20	4	1	38	32	5761
21	4	1	* 10	9	2467
22	4	1	23	22	6898
23	4	1	21	17	3597
24	4	1	30	25	8435
25	4	1	33	29	6273
26	4	1	21	22	9802
27	4	1	* 9	7	356
28	4	1	25	22	8707
29	4	1	16	15	515
30	4	1	15	10	4109
31	4	1	14	17	7590
32	4	1	13	15	1991
0	5	1	70	71	2500
1	5	1	120	104	5654
2	5	1	94	90	301
3	5	1	32	35	7330
4	5	1	67	68	4506
5	5	1	81	73	808
6	5	1	67	63	3935
7	5	1	34	28	8705
8	5	1	76	67	7285
9	5	1	64	63	8457
10	5	1	75	68	7341
11	5	1	77	83	1715
12	5	1	26	24	5028
13	5	1	71	57	9208
14	5	1	19	17	1782
15	5	1	30	29	7893
16	5	1	36	30	29
17	5	1	93	71	3966
18	5	1	35	34	3321
19	5	1	88	82	5445
20	5	1	38	34	3234
21	5	1	* 6	4	4831
22	5	1	19	18	1276
23	5	1	19	17	7717
24	5	1	18	16	3996
25	5	1	22	17	8122

above plane. Seemingly significant differences are also observed between the two C–C bond distances (1·60 and 1·42 Å); any attempt to start a new least-squares cycle from a more symmetrical configuration of the C–C–C sequence has resulted in the same final arrangement. On the other hand, inspection of the three-dimensional Fourier syntheses (see Fig. 3) shows a satisfactory spherical symmetry of the electron density distribution on the three carbon atoms. However, it is difficult to conclude that the above differences are really significant and they are contrary to expectation; a possible explanation could lie in a strong interaction among the parameters of the tetrahedral carbon atoms and those of the surrounding Co atoms, leading to high values of the error covariances.

The large number of *a priori* equivalent but crystallographically independent bond lengths and angles present in this structure has suggested a comparison between the average standard deviations resulting from the refinement, and the corresponding quantities directly evaluated from the internal mean square deviation of the individual parameters. Table 5 reports the two

Table 3 (*cont*.)

H	K	L	FO	FC	PH
26	5	1	21	19	6218
27	5	1	15	14	2177
28	5	1	21	18	7666
29	5	1	• 5	11	8952
30	5	1	• 4	5	63
0	6	1	73	61	0
1	6	1	31	34	6122
2	6	1	76	61	7220
3	6	1	39	35	5781
4	6	1	30	27	4905
5	6	1	65	57	4124
6	6	1	39	33	8647
7	6	1	64	61	5876
8	6	1	51	42	7441
9	6	1	22	20	460
10	6	1	37	28	4419
11	6	1	37	28	8602
12	6	1	32	40	4541
13	6	1	38	39	9417
14	6	1	89	71	1593
15	6	1	70	52	1007
16	6	1	89	69	2539
17	6	1	59	36	8296
18	6	1	23	17	5623
19	6	1	• 8	7	6092
20	6	1	49	46	8809
21	6	1	21	21	3035
22	6	1	• 7	6	1070
23	6	1	17	15	6327
24	6	1	28	28	4963
25	6	1	21	19	5011
26	6	1	14	13	7227
27	6	1	14	12	4139
28	6	1	• 4	8	8579
0	7	1	38	42	2500
1	7	1	89	67	5460
2	7	1	58	49	2628
3	7	1	39	31	774
4	7	1	34	31	8214
5	7	1	44	42	2584
6	7	1	41	34	7744
7	7	1	20	15	413
8	7	1	• 8	7	6940
9	7	1	• 8	10	8990
10	7	1	37	26	6610
11	7	1	16	13	8197
12	7	1	38	34	7716
13	7	1	36	30	471
14	7	1	• 8	10	7025
15	7	1	32	25	7457
16	7	1	53	53	4330
17	7	1	38	38	6070
18	7	1	• 8	16	1804
19	7	1	41	34	3533
20	7	1	22	21	1097
21	7	1	16	14	9502
22	7	1	15	16	4190
23	7	1	12	8	8944
24	7	1	• 5	8	8517
25	7	1	31	26	3089
26	7	1	• 4	10	9311
0	8	1	• 8	11	0
1	8	1	61	51	7761
2	8	1	31	22	8502
3	8	1	46	43	4091
4	8	1	16	16	5477
5	8	1	26	19	5931
6	8	1	• 8	13	2753
7	8	1	30	28	14
8	8	1	20	18	5629
9	8	1	23	15	3883
10	8	1	45	35	5717
11	8	1	31	28	4536
12	8	1	22	19	9996
13	8	1	18	13	1655
14	8	1	• 7	12	2981
15	8	1	27	20	9130
16	8	1	17	16	4951
17	8	1	• 7	7	738
18	8	1	• 6	8	9493
19	8	1	12	14	7171
20	8	1	• 5	8	8176
21	8	1	29	25	7044
22	8	1	• 4	7	9076
23	8	1	10	12	3516
0	9	1	• 8	14	2500
1	9	1	21	20	1058
2	9	1	• 8	8	4795
3	9	1	15	13	4812
4	9	1	38	33	8679
5	9	1	• 7	10	9653
6	9	1	41	36	9430
7	9	1	18	15	2257
8	9	1	32	26	3874
9	9	1	22	17	5824
10	9	1	27	24	2919
11	9	1	• 6	5	7561
12	9	1	• 6	9	8631
13	9	1	• 6	6	9186
14	9	1	45	38	5454
15	9	1	• 5	6	2453
16	9	1	15	12	4952
17	9	1	• 5	2	7507
18	9	1	• 4	4	586
19	9	1	10	12	8151
0	10	1	17	16	5000
1	10	1	19	16	5777
2	10	1	• 6	10	2612
3	10	1	12	10	7747
4	10	1	18	17	514
5	10	1	36	31	135
6	10	1	14	10	5612
7	10	1	11	16	183
8	10	1	13	16	4289
9	10	1	10	16	1645
10	10	1	18	16	9706
11	10	1	24	23	3190
12	10	1	• 4	6	6539
13	10	1	8	12	5302
0	0	2	17	19	0
1	0	2	110	101	5000
2	0	2	109	74	0
3	0	2	110	97	5000
4	0	2	23	26	0
5	0	2	160	166	0
6	0	2	50	54	5000
7	0	2	44	45	5000
8	0	2	56	24	5000
9	0	2	37	41	5000
10	0	2	31	17	0
11	0	2	45	29	0
12	0	2	86	79	5000
13	0	2	54	51	5000
14	0	2	69	92	5000
15	0	2	27	12	5000
16	0	2	44	46	0
17	0	2	48	46	0
18	0	2	109	121	0
19	0	2	38	48	5000
20	0	2	• 12	21	5000
21	0	2	23	13	5000
22	0	2	• 12	21	5000
23	0	2	36	35	0
24	0	2	30	34	0
25	0	2	• 12	5	0
26	0	2	• 12	5	0
27	0	2	24	14	0
28	0	2	28	34	5000
29	0	2	26	26	5000
30	0	2	26	20	5000
31	0	2	• 9	8	5000
32	0	2	• 8	1	5000
33	0	2	• 7	10	0
34	0	2	• 5	1	0
0	1	2	31	23	2500
1	1	2	138	130	3441
2	1	2	90	70	174
3	1	2	105	99	2785
4	1	2	103	90	6889
5	1	2	63	38	9704
6	1	2	69	60	3911
7	1	2	105	103	5849
8	1	2	108	110	93
9	1	2	258	273	655
10	1	2	64	41	9819
11	1	2	52	49	9951
12	1	2	38	21	3794
13	1	2	93	101	5855
14	1	2	75	72	6806
15	1	2	68	76	9073
16	1	2	43	37	326
17	1	2	75	76	5988
18	1	2	49	53	2226
19	1	2	34	39	5008
20	1	2	34	33	8036
21	1	2	34	32	1741
22	1	2	61	60	8698
23	1	2	41	43	4356
24	1	2	34	29	3654
25	1	2	• 12	14	199
26	1	2	25	19	4018
27	1	2	32	33	1233
28	1	2	• 11	5	8815
29	1	2	• 11	11	2095
30	1	2	19	9	9714
31	1	2	29	29	8651
32	1	2	• 8	8	5822
33	1	2	20	22	7516
34	1	2	• 4	4	6304
0	2	2	127	114	0
1	2	2	115	106	4380
2	2	2	40	41	5933
3	2	2	106	85	1126
4	2	2	213	181	5571
5	2	2	54	54	1724
6	2	2	340	328	5862
7	2	2	185	175	3780
8	2	2	162	150	6709
9	2	2	49	49	9491
10	2	2	75	80	2210
11	2	2	40	43	5485
12	2	2	110	112	3038
13	2	2	104	113	1274
14	2	2	36	44	4315
15	2	2	65	64	435
16	2	2	85	94	2451
17	2	2	66	74	6483
18	2	2	• 11	6	7488
19	2	2	33	40	879
20	2	2	55	63	8522
21	2	2	39	50	3257
22	2	2	• 11	16	383
23	2	2	• 12	11	3464
24	2	2	44	45	7605
25	2	2	26	29	9266
26	2	2	53	54	7656
27	2	2	• 11	18	5614
28	2	2	• 11	9	2814
29	2	2	• 10	1	3979
30	2	2	17	20	1811
31	2	2	• 8	11	8826
32	2	2	• 7	6	1727
33	2	2	• 6	7	1175
0	3	2	49	52	2500
1	3	2	136	129	5150
2	3	2	107	93	1896
3	3	2	53	37	4858
4	3	2	81	57	5400
5	3	2	95	88	3645
6	3	2	125	132	5286
7	3	2	64	58	183
8	3	2	60	10	9996
9	3	2	137	152	9201
10	3	2	37	44	9755
11	3	2	59	72	603
12	3	2	78	84	7229
13	3	2	21	22	9420
14	3	2	33	20	2967
15	3	2	57	59	5783
16	3	2	46	54	3617
17	3	2	37	38	8318
18	3	2	34	35	6299
19	3	2	43	47	4670
20	3	2	63	61	2430
21	3	2	86	92	5266
22	3	2	29	20	2983
23	3	2	• 11	15	14
24	3	2	42	44	6792
25	3	2	23	21	274
26	3	2	23	22	4929
27	3	2	29	28	51
28	3	2	19	16	9814
29	3	2	22	24	9258
30	3	2	23	22	8455
31	3	2	• 7	4	2554
32	3	2	15	16	4892
33	3	2	• 3	12	475
0	4	2	56	65	5000
1	4	2	140	132	359
2	4	2	62	52	4468
3	4	2	68	64	4750
4	4	2	82	75	7676
5	4	2	119	119	4503
6	4	2	100	100	8281
7	4	2	45	46	7921
8	4	2	50	46	9386
9	4	2	51	49	6714
10	4	2	57	62	4830
11	4	2	50	46	8717
12	4	2	47	39	1456
13	4	2	34	39	1048
14	4	2	93	105	1751
15	4	2	38	40	1437
16	4	2	47	47	4855
17	4	2	36	35	8361
18	4	2	28	29	4183
19	4	2	24	10	3107
20	4	2	24	26	1807
21	4	2	36	36	2270
22	4	2	54	54	8116
23	4	2	26	33	4666
24	4	2	27	32	6736
25	4	2	27	25	6332
26	4	2	• 11	11	6940
27	4	2	• 10	14	7937
28	4	2	18	18	232
29	4	2	• 8	7	6012
30	4	2	• 7	9	2220
31	4	2	14	20	781
0	5	2	55	54	2500
1	5	2	73	77	8340
2	5	2	31	22	3558
3	5	2	137	141	6079
4	5	2	58	71	5161
5	5	2	35	35	1431
6	5	2	• 10	10	557
7	5	2	29	21	400
8	5	2	33	28	6589
9	5	2	30	30	7585
10	5	2	46	59	8092
11	5	2	44	43	9678
12	5	2	50	52	419
13	5	2	53	64	2985
14	5	2	23	21	3304
15	5	2	23	22	2861
16	5	2	24	25	6918
17	5	2	• 12	14	4561
18	5	2	38	42	582
19	5	2	• 12	2	1657
20	5	2	23	22	1217
21	5	2	23	25	6589
22	5	2	23	25	3792
23	5	2	• 11	22	6100
24	5	2	21	28	5447
25	5	2	20	21	1028
26	5	2	23	15	7694
27	5	2	• 9	4	6478
28	5	2	• 8	5	880
29	5	2	• 7	11	7358
30	5	2	• 5	7	7347
0	6	2	53	52	5000
1	6	2	27	32	4335
2	6	2	51	52	4931
3	6	2	85	94	1569
4	6	2	22	18	1291
5	6	2	22	35	9509
6	6	2	53	53	791
7	6	2	111	101	5570
8	6	2	• 11	15	5768
9	6	2	33	33	1106
10	6	2	23	25	8098
11	6	2	37	46	1188
12	6	2	37	39	837
13	6	2	38	31	6620
14	6	2	24	26	8495
15	6	2	24	25	7420
16	6	2	24	23	6310
17	6	2	23	29	3083
18	6	2	23	25	5159
19	6	2	• 11	15	2169
20	6	2	22	16	690
21	6	2	• 11	6	9977
22	6	2	• 10	13	168
23	6	2	• 10	19	1839
24	6	2	• 9	4	2579
25	6	2	• 8	7	5301
26	6	2	• 8	14	1837
27	6	2	13	21	6757
28	6	2	• 5	11	8245
0	7	2	• 12	10	7500
1	7	2	29	19	489
2	7	2	58	52	6259
3	7	2	47	47	8884
4	7	2	• 12	11	1390
5	7	2	33	34	5565
6	7	2	47	48	2790
7	7	2	33	32	2583
8	7	2	24	32	5598
9	7	2	24	19	4723
10	7	2	24	26	3622
11	7	2	44	43	5832
12	7	2	33	27	2554
13	7	2	• 12	12	8574
14	7	2	• 12	5	2618
15	7	2	28	36	2184
16	7	2	22	20	6398
17	7	2	22	28	2108
18	7	2	• 11	18	7999
19	7	2	• 10	9	8940
20	7	2	• 10	7	6736
21	7	2	• 9	20	9535
22	7	2	21	20	5795
23	7	2	• 8	10	482
24	7	2	• 7	10	3487
25	7	2	17	22	5471
26	7	2	• 4	4	4016
0	8	2	33	35	5000
1	8	2	24	25	1853
2	8	2	33	29	7728
3	8	2	29	28	94
4	8	2	58	57	1846
5	8	2	40	40	9627
6	8	2	33	29	3456
7	8	2	23	24	9920
8	8	2	• 11	9	5900
9	8	2	• 11	16	4206
10	8	2	• 11	11	9742
11	8	2	• 11	5	2518
12	8	2	22	20	8191
13	8	2	• 11	19	6233
14	8	2	• 10	15	6671
15	8	2	38	40	4801
16	8	2	• 10	11	131
17	8	2	• 9	14	3732
18	8	2	• 9	4	8021
19	8	2	• 8	9	397
20	8	2	21	18	585[illegible]
21	8	2	• 7	17	8827
22	8	2	• 6	6	2415
23	8	2	• 4	6	8887
0	9	2	52	51	7500
1	9	2	• 11	6	1303
2	9	2	• 11	9	6508
3	9	2	• 11	16	226
4	9	2	• 11	5	3263
5	9	2	21	14	7661
6	9	2	20	18	8930
7	9	2	32	21	4910
8	9	2	20	30	2166
9	9	2	39	18	3292
10	9	2	• 9	14	2888
11	9	2	• 9	9	8933
12	9	2	• 9	16	3408
13	9	2	• 9	9	5794
14	9	2	• 8	5	487
15	9	2	• 8	11	1037
16	9	2	14	13	8400
17	9	2	• 6	7	4139
18	9	2	15	17	6910
0	10	2	• 8	5	5000
1	10	2	• 8	7	2821
2	10	2	• 8	11	3010
3	10	2	16	19	8188
4	10	2	19	14	7134
5	10	2	15	19	8849
6	10	2	• 8	9	3585
7	10	2	21	25	1400
8	10	2	• 7	7	1848
9	10	2	13	15	1239
10	10	2	• 6	14	8825
11	10	2	16	18	5234
12	10	2	• 5	7	5063
13	10	2	• 3	9	4463
1	0	3	14	6	2500
2	0	3	47	22	7500
3	0	3	108	79	2500
4	0	3	85	70	7500
5	0	3	98	102	7500
6	0	3	117	108	7500
7	0	3	63	75	2500
8	0	3	90	93	2500
9	0	3	40	41	2500
10	0	3	63	46	2500
11	0	3	94	103	7500
12	0	3	35	35	7500
13	0	3	29	26	7500
14	0	3	35	43	2500
15	0	3	85	96	2500
16	0	3	46	53	7500
17	0	3	• 11	8	7500
18	0	3	21	15	7500
19	0	3	20	13	7500
20	0	3	29	31	2500
21	0	3	21	15	2500
22	0	3	23	21	7500
23	0	3	44	39	7500
24	0	3	25	5	2500
25	0	3	21	10	7500
26	0	3	27	18	7500
27	0	3	34	41	2500
28	0	3	• 10	5	2500
29	0	3	• 10	7	7500
30	0	3	19	18	2500
31	0	3	22	26	7500
32	0	3	17	16	7500
33	0	3	• 5	7	2500
0	1	3	156	116	7500
1	1	3	111	88	7006
2	1	3	75	39	2722
3	1	3	34	20	9619
4	1	3	169	139	4819
5	1	3	138	119	7356
6	1	3	112	94	1333
7	1	3	109	75	7256
8	1	3	119	115	453
9	1	3	61	55	8689
10	1	3	131	147	7119
11	1	3	43	51	2230
12	1	3	62	69	3705
13	1	3	109	117	2826
14	1	3	64	70	273
15	1	3	52	17	5140
16	1	3	47	53	8638
17	1	3	51	55	9591
18	1	3	45	50	2417
19	1	3	32	37	2424
20	1	3	29	35	6178
21	1	3	49	57	5578
22	1	3	21	26	2375
23	1	3	61	63	7677
24	1	3	• 12	10	291
25	1	3	31	32	9555

series of results, for the different parameters, together with their average value, and with the number of non-equivalent determinations, assumed as statistically independent; the ratios between the two different estimates of the s.d. are also reported. The latter figures are always larger than unity. We have limited ourselves to the data resulting from at least 6 different determinations, with the exception of the Co–C–O bond angles, whose values are seriously affected by the arbitrary choice of their upper limit (see at 180°) and are therefore statistically meaningless. The squares of the remaining ratios are always larger than the corresponding F-values at the 5% level of significance (Johnson & Leone, 1964), with the only exception of the Co–Co distances; it seems worth stressing that the lowest ratios correspond to geometrical parameters which are expected to be scarcely affected by crystal packing effects, namely most bond lengths and some of the bond angles which correspond to the Co_3C tetrahedra. Four of these are in the range 1·30 to 1·55; the larger ratios, for $d_{\text{Co-C(tetrahedral)}}$, and Co–C–Co (2·17 and 3·09 respectively), are related to the differences, discussed above, between the two tetrahedra. The difference between the C_A–Co–C_E and the

Table 3 (*cont.*)

H	K	L		FO	FC	PH
26	1	3		27	30	7633
27	1	3	•	11	7	2709
28	1	3		19	15	3346
29	1	3	•	10	4	7123
30	1	3	•	9	15	6600
31	1	3		22	16	2481
32	1	3	•	7	7	7435
33	1	3		10	12	2945
0	2	3		27	46	0
1	2	3		103	99	814
2	2	3		227	220	4609
3	2	3		29	17	1360
4	2	3		147	136	5820
5	2	3		89	88	5405
6	2	3		116	117	781
7	2	3		97	97	3776
8	2	3		125	128	8934
9	2	3		70	54	7824
10	2	3		57	61	9602
11	2	3		61	56	9932
12	2	3		63	71	975
13	2	3		34	42	5842
14	2	3		71	84	7291
15	2	3		31	32	8380
16	2	3		66	75	4546
17	2	3		25	28	1434
18	2	3		26	32	5197
19	2	3		49	53	3835
20	2	3		64	71	5689
21	2	3		47	21	7878
22	2	3		45	53	4171
23	2	3	•	11	3	9442
24	2	3		31	31	8139
25	2	3		36	38	4679
26	2	3		27	36	125
27	2	3		18	11	7319
28	2	3		28	28	160
29	2	3		20	20	8616
30	2	3	•	8	6	8742
31	2	3		13	13	6881
32	2	3		11	10	2733
33	2	3	•	3	6	6518
0	3	3		34	39	2500
1	3	3		18	16	9898
2	3	3		74	77	5716
3	3	3		40	47	7584
4	3	3		57	58	990
5	3	3		111	118	6923
6	3	3		39	56	2568
7	3	3		125	113	7093
8	3	3		117	129	6102
9	3	3		29	27	8704
10	3	3		46	54	8570
11	3	3		82	84	3034
12	3	3		39	54	9463
13	3	3		102	109	2793
14	3	3		27	34	1884
15	3	3		60	57	636
16	3	3		45	49	176
17	3	3		29	32	3211
18	3	3		61	65	8471
19	3	3		58	60	2779
20	3	3		66	67	3358
21	3	3		35	42	8478
22	3	3	•	11	15	3553
23	3	3		54	61	7239
24	3	3		27	28	7093
25	3	3		22	28	7333
26	3	3		19	16	5270
27	3	3		18	13	8995
28	3	3		18	16	7075
29	3	3	•	8	7	2595
30	3	3		14	9	2461
31	3	3		15	16	2499
32	3	3	•	4	7	8393
0	4	3		52	60	5000
1	4	3		17	14	800
2	4	3		78	89	7093
3	4	3		70	64	2343
4	4	3		118	131	3397
5	4	3		65	65	3892
6	4	3		27	18	8995

H	K	L		FO	FC	PH
7	4	3		59	64	6419
8	4	3		95	98	9313
9	4	3		52	58	8277
10	4	3		79	89	9831
11	4	3		36	31	7911
12	4	3		21	24	6323
13	4	3		37	37	9922
14	4	3		20	17	2156
15	4	3	•	11	11	3789
16	4	3		49	52	7595
17	4	3		41	44	3613
18	4	3		31	35	4785
19	4	3		37	41	1581
20	4	3		21	23	5115
21	4	3	•	11	17	7195
22	4	3		29	34	6024
23	4	3		23	22	3387
24	4	3		23	32	3340
25	4	3	•	10	6	1253
26	4	3		18	21	9662
27	4	3		24	28	7277
28	4	3		22	27	9453
29	4	3	•	7	8	9042
30	4	3	•	6	7	8152
31	4	3	•	4	4	3011
0	5	3		110	115	7500
1	5	3		22	12	5627
2	5	3		48	59	2984
3	5	3		18	8	6
4	5	3		41	49	3488
5	5	3		26	22	7671
6	5	3		23	33	7034
7	5	3		74	76	8341
8	5	3		38	45	3695
9	5	3		27	32	4933
10	5	3		34	41	9005
11	5	3		56	54	3620
12	5	3		45	55	299
13	5	3		32	36	9366
14	5	3		21	16	573
15	5	3		25	24	3970
16	5	3		25	31	5728
17	5	3		25	22	3099
18	5	3		25	27	1002
19	5	3		21	16	8353
20	5	3		20	25	9193
21	5	3	•	10	14	9862
22	5	3		19	23	4115
23	5	3	•	9	12	987
24	5	3		21	22	4646
25	5	3		26	29	6473
26	5	3	•	8	16	8092
27	5	3	•	7	9	5729
28	5	3		12	8	1922
29	5	3	•	5	15	2743
0	6	3		20	20	5000
1	6	3		20	24	7692
2	6	3		20	25	1238
3	6	3		49	40	2877
4	6	3		57	56	8355
5	6	3		40	43	1656
6	6	3		70	74	3453
7	6	3		32	33	9536
8	6	3		25	14	4375
9	6	3		33	37	4395
10	6	3		53	60	8518
11	6	3	•	11	11	3908
12	6	3		21	20	2675
13	6	3		21	17	2724
14	6	3		25	23	5598
15	6	3		25	32	3277
16	6	3		21	13	1634
17	6	3		20	18	7886
18	6	3	•	10	12	791
19	6	3	•	10	17	8007
20	6	3	•	9	11	6664
21	6	3		18	10	2380
22	6	3	•	9	9	1453
23	6	3	•	8	12	3668
24	6	3	•	7	11	9763
25	6	3	•	7	12	7724
26	6	3		12	16	3259

H	K	L		FO	FC	PH
27	6	3	•	5	7	9965
0	7	3		21	18	2500
1	7	3		25	25	3443
2	7	3		75	79	9649
3	7	3		29	21	3268
4	7	3		29	31	750
5	7	3		33	31	9607
6	7	3	•	11	7	1343
7	7	3		55	56	3233
8	7	3		39	30	9153
9	7	3		21	16	4490
10	7	3		49	57	2667
11	7	3		32	36	8237
12	7	3		21	13	6194
13	7	3	•	10	8	7572
14	7	3		24	27	6561
15	7	3		20	10	1830
16	7	3		19	19	3962
17	7	3	•	9	9	6627
18	7	3	•	9	5	8198
19	7	3		17	11	6228
20	7	3		16	18	9341
21	7	3	•	8	8	2898
22	7	3		17	18	8582
23	7	3	•	6	13	1900
24	7	3	•	5	8	1810
25	7	3	•	4	17	1985
0	8	3		20	21	0
1	8	3		45	48	6503
2	8	3		20	16	158
3	8	3		25	27	7331
4	8	3	•	10	10	1633
5	8	3		24	29	886
6	8	3		24	24	7955
7	8	3		34	42	2751
8	8	3		31	35	4946
9	8	3	•	10	13	3021
10	8	3		23	23	5057
11	8	3		26	25	3836
12	8	3		18	22	8399
13	8	3		22	27	2868
14	8	3		17	19	2411
15	8	3	•	8	5	7163
16	8	3		16	8	2631
17	8	3		28	36	7012
18	8	3		14	15	9561
19	8	3		14	17	6617
20	8	3		12	13	8802
21	8	3		10	7	6120
22	8	3	•	3	9	9155
0	9	3	•	9	10	2500
1	9	3		18	18	5328
2	9	3		18	16	821
3	9	3		21	22	15
4	9	3		27	29	9249
5	9	3		17	17	2947
6	9	3		21	22	26
7	9	3		17	18	2987
8	9	3		16	24	1243
9	9	3		16	16	9496
10	9	3		19	14	8781
11	9	3		15	21	8359
12	9	3		22	23	4485
13	9	3	•	7	7	7081
14	9	3		28	26	4661
15	9	3		14	8	4645
16	9	3	•	5	3	5827
17	9	3	•	4	4	8633
0	10	3	•	6	2	0
1	10	3	•	6	10	9278
2	10	3	•	6	7	777
3	10	3		20	18	6974
4	10	3		12	12	7642
5	10	3	•	6	7	4576
6	10	3	•	6	6	9311
7	10	3		21	21	1476
8	10	3	•	5	7	7190
9	10	3	•	5	6	2007
10	10	3		10	9	3869
11	10	3	•	3	9	3890
0	0	4		14	11	0
1	0	4	•	9	7	5000

H	K	L		FO	FC	PH
2	0	4		141	125	0
3	0	4		36	30	5000
4	0	4		15	5	5000
5	0	4		20	19	5000
6	0	4		34	31	5000
7	0	4		65	43	5000
8	0	4		187	197	0
9	0	4		26	28	0
10	0	4		84	80	0
11	0	4		86	97	5000
12	0	4		50	56	0
13	0	4		20	18	0
14	0	4		65	58	0
15	0	4		99	109	0
16	0	4	•	10	11	5000
17	0	4		38	38	5000
18	0	4		73	76	5000
19	0	4		49	50	5000
20	0	4		28	26	5000
21	0	4		50	45	5000
22	0	4	•	10	11	5000
23	0	4		36	35	0
24	0	4		19	25	5000
25	0	4	•	10	5	5000
26	0	4		23	20	0
27	0	4		34	34	5000
28	0	4		29	25	0
29	0	4	•	8	6	0
30	0	4		9	12	5000
31	0	4	•	6	6	5000
32	0	4	•	5	2	0
0	1	4		91	83	2500
1	1	4		79	76	6571
2	1	4		44	34	2190
3	1	4		50	43	8983
4	1	4		114	99	6595
5	1	4		67	58	6353
6	1	4		34	14	2127
7	1	4		132	127	8725
8	1	4		56	32	2511
9	1	4		88	83	9644
10	1	4		32	23	7019
11	1	4		84	82	2689
12	1	4		65	69	8810
13	1	4		59	67	904
14	1	4		45	59	9827
15	1	4		12	5	7865
16	1	4		24	27	3261
17	1	4		30	35	3185
18	1	4		36	38	1974
19	1	4		31	30	4336
20	1	4		32	31	1595
21	1	4		36	39	6121
22	1	4		40	31	5424
23	1	4		29	26	8227
24	1	4		23	19	6102
25	1	4		18	15	7311
26	1	4		17	21	977
27	1	4		31	36	9792
28	1	4	•	8	7	1890
29	1	4	•	8	9	1126
30	1	4		11	12	6722
31	1	4		17	19	4598
32	1	4		13	15	9100
0	2	4		33	34	5000
1	2	4		40	32	9216
2	2	4		63	71	6761
3	2	4		115	125	3604
4	2	4		39	22	6068
5	2	4		46	27	6010
6	2	4		98	104	8428
7	2	4		76	83	8303
8	2	4		102	107	7584
9	2	4		41	41	3741
10	2	4		33	30	1060
11	2	4		30	34	7343
12	2	4		106	114	2147
13	2	4		42	49	8236
14	2	4		46	30	3794
15	2	4		42	45	1049
16	2	4		24	16	6676
17	2	4		42	50	697

H	K	L		FO	FC	PH
18	2	4		52	54	2291
19	2	4		37	45	4997
20	2	4		22	25	4546
21	2	4		26	27	2666
22	2	4		51	54	7118
23	2	4	•	9	9	1719
24	2	4		34	41	8038
25	2	4		21	24	7036
26	2	4		12	14	8446
27	2	4	•	8	11	7525
28	2	4	•	7	10	2223
29	2	4		19	20	5603
30	2	4		11	13	1178
31	2	4	•	5	7	9795
0	3	4		42	38	7500
1	3	4		43	69	2287
2	3	4		65	73	3405
3	3	4		140	145	5038
4	3	4		43	39	3070
5	3	4		38	38	7300
6	3	4		58	55	5634
7	3	4		80	89	731
8	3	4		40	33	7437
9	3	4		81	95	9853
10	3	4		44	19	1079
11	3	4		66	72	9145
12	3	4		19	20	8716
13	3	4		28	30	3501
14	3	4		25	26	7825
15	3	4		13	13	8883
16	3	4		47	55	3795
17	3	4		50	52	6016
18	3	4		30	32	1618
19	3	4		22	22	4963
20	3	4		18	19	928
21	3	4	•	9	11	4039
22	3	4		21	22	4726
23	3	4		33	35	5360
24	3	4		12	9	3375
25	3	4		15	21	1113
26	3	4	•	8	6	5579
27	3	4		27	29	135
28	3	4		9	8	5192
29	3	4		12	14	9022
30	3	4	•	5	10	8301
31	3	4	•	3	5	1373
0	4	4		24	25	0
1	4	4		18	16	7244
2	4	4		82	89	1292
3	4	4		56	71	8355
4	4	4		61	68	8717
5	4	4	•	8	4	2821
6	4	4		81	85	7334
7	4	4		67	82	5009
8	4	4		64	60	6530
9	4	4		51	55	6744
10	4	4		36	31	3589
11	4	4		39	37	1671
12	4	4		73	78	2293
13	4	4		65	73	9766
14	4	4		33	37	3002
15	4	4		33	39	7056
16	4	4		14	10	3783
17	4	4		31	42	5186
18	4	4		33	40	313
19	4	4		29	34	52
20	4	4		15	19	2439
21	4	4	•	9	11	5306
22	4	4	•	9	9	6932
23	4	4		19	21	4605
24	4	4		26	27	8223
25	4	4		15	13	2315
26	4	4		33	35	5326
27	4	4		13	15	6438
28	4	4		14	18	4756
29	4	4	•	5	5	4644
0	5	4		29	31	7500
1	5	4		14	10	5504
2	5	4		24	22	7403
3	5	4		29	32	2501
4	5	4		54	63	1186
5	5	4		52	51	3426

H	K	L		FO	FC	PH
6	5	4		28	21	9202
7	5	4		40	45	1571
8	5	4		71	72	7471
9	5	4		41	40	807
10	5	4		47	51	829
11	5	4		49	59	8411
12	5	4		13	6	5952
13	5	4		56	61	7901
14	5	4		48	39	5170
15	5	4		27	31	4835
16	5	4		23	29	71
17	5	4		13	12	8815
18	5	4	•	7	6	7854
19	5	4	•	7	4	8517
20	5	4	•	7	5	4486
21	5	4		24	31	4523
22	5	4		30	36	614
23	5	4		24	25	1900
24	5	4		10	7	1549
25	5	4		10	8	1555
26	5	4		13	14	7188
27	5	4	•	4	4	7841
28	5	4		11	17	9156
0	6	4		20	8	5000
1	6	4		42	36	9293
2	6	4		30	29	1387
3	6	4		38	37	8308
4	6	4		42	45	162
5	6	4		15	16	9170
6	6	4		44	41	6528
7	6	4		43	42	3053
8	6	4		30	27	3939
9	6	4		21	14	3900
10	6	4	•	7	9	5749
11	6	4		36	38	252
12	6	4		38	39	5773
13	6	4		31	32	3554
14	6	4		13	18	1316
15	6	4		34	37	5396
16	6	4		18	21	1021
17	6	4		16	16	7741
18	6	4		12	8	6792
19	6	4		13	13	8391
20	6	4		22	20	9601
21	6	4		18	17	9109
22	6	4		13	11	142
23	6	4		9	6	5623
24	6	4		14	13	4283
25	6	4		13	13	5372
26	6	4	•	3	8	5575
0	7	4		36	32	7500
1	7	4		21	25	7561
2	7	4		42	39	9232
3	7	4		23	22	9514
4	7	4		16	11	8244
5	7	4		33	36	1829
6	7	4		13	17	1234
7	7	4		37	44	5260
8	7	4		23	29	509
9	7	4		13	14	7118
10	7	4		16	16	4634
11	7	4		22	27	692
12	7	4		42	50	3688
13	7	4		18	19	5810
14	7	4		12	9	2360
15	7	4		13	12	8513
16	7	4		28	33	6771
17	7	4	•	6	8	1704
18	7	4		23	25	6418
19	7	4		16	10	7671
20	7	4		10	11	9248
21	7	4		9	9	9818
22	7	4		10	5	2073
23	7	4		7	8	952
0	8	4		14	14	5000
1	8	4		21	25	8951
2	8	4	•	7	5	2572
3	8	4		35	41	22
4	8	4		12	2	7679
5	8	4		12	8	8819
6	8	4		22	26	1922
7	8	4	•	7	4	9096

C_E–Co–C_E average bond angles (A = axial; E = equatorial carbonyl groups; see Table 5) is significant at the 1% level, even considering the e.s.d. derived from comparison of the results [s in Table 5; t test]; instead, the differences between d_{Co-C_A} and d_{Co-C_E} and between C_A–Co–Co and C_E–Co–Co are not significant, even at the 10% level, so that no separate estimate has been given.

For both tetrahedra the bond coordination around C atoms departs markedly from threefold symmetry, mainly because of the differences in the C–C–Co angles; for each tetrahedron, two of these are contained in the range $121 \pm 3{\cdot}5°$, and the third within $148 \pm 1°$ (see Table 4). As can be seen from Figs. 1 and 4, such distortion allows the relaxation of the O···O distances involving atoms belonging to different tetrahedra, all of which in fact exceed 3·15 Å; note that the dihedral angle between the Co_3 planes of the two tetrahedra should, in the absence of any distortion, approach 57° [$\simeq 180° - $(C–C–C)], instead of the experimental value of 26°.

The overall external shape of the molecule, defined as the envelope of the van der Waals spheres of its oxygen atoms, is markedly ellipsoidal; the O···O

Table 3 (*cont.*)

H	K	L		FO	FC	PH
8	8	4		24	20	775
9	8	4		40	40	1175
10	8	4	•	6	9	6228
11	8	4		15	16	4659
12	8	4		15	12	7295
13	8	4		21	23	5357
14	8	4		16	13	8243
15	8	4		21	17	3894
15	8	4		17	20	3706
17	8	4		10	15	5914
18	8	4	•	4	8	7074
19	8	4		8	11	7684
20	8	4	•	3	9	6952
0	9	4		15	8	7500
1	9	4	•	6	4	6248
2	9	4		21	25	7651
3	9	4		16	16	9055
4	9	4		14	14	7710
5	9	4		15	12	8049
6	9	4		15	15	1676
7	9	4	•	5	7	8796
8	9	4		13	13	1842
9	9	4		20	24	5235
10	9	4		11	12	2903
11	9	4		12	13	5243
12	9	4	•	4	5	1204
13	9	4		18	19	910
14	9	4		10	10	1756
15	9	4	•	3	7	9098
0	10	4		6	8	0
1	10	4		8	10	833
2	10	4		13	14	6808
3	10	4	•	3	5	787
4	10	4	•	3	15	4191
5	10	4		12	6	9086
6	10	4	•	3	12	1674
7	10	4	•	2	16	9146
1	0	5		82	70	2500
2	0	5		31	25	7500
3	0	5		72	69	7500
4	0	5		143	141	7500
5	0	5		21	35	2500
6	0	5	•	11	12	7500
7	0	5		53	54	7500
8	0	5		31	32	7500
9	0	5		29	28	2500
10	0	5	•	11	0	7499
11	0	5		25	27	2500
12	0	5		113	110	2500
13	0	5		111	114	7500
14	0	5		84	82	2500
15	0	5	•	13	11	2500
16	0	5		36	34	2500
17	0	5		70	60	2500
18	0	5		37	37	2500
19	0	5		40	33	2500
20	0	5	•	13	21	2500
21	0	5	•	13	2	2500
22	0	5		49	45	7500
23	0	5	•	12	7	2500
24	0	5		39	38	7500
25	0	5		21	18	2500
26	0	5	•	11	4	2500
27	0	5		20	8	7500
28	0	5	•	9	13	7500
29	0	5	•	8	11	7500
30	0	5	•	7	1	2500
0	1	5		97	85	2500
1	1	5		50	41	5893
2	1	5		92	83	5136
3	1	5		120	103	4380
4	1	5		45	41	4909
5	1	5		26	25	9907
6	1	5		20	18	2057
7	1	5		128	135	8959
8	1	5		50	50	7351
9	1	5		35	35	2614
10	1	5	•	12	18	4269
11	1	5		58	63	9708
12	1	5		43	46	3650
13	1	5		45	50	9449
14	1	5		56	54	8616

H	K	L		FO	FC	PH
15	1	5	•	13	11	4164
16	1	5		52	40	339
17	1	5		61	51	5078
18	1	5		42	41	2191
19	1	5		54	49	4357
20	1	5		34	35	4850
21	1	5		24	28	5289
22	1	5	•	13	13	5561
23	1	5		27	21	5976
24	1	5	•	12	13	2515
25	1	5	•	11	14	8227
26	1	5	•	11	14	7020
27	1	5		26	25	9150
28	1	5		17	15	7060
29	1	5		31	25	1158
30	1	5		16	16	2802
0	2	5		23	30	5000
1	2	5		79	81	4542
2	2	5		75	69	7520
3	2	5		50	46	3580
4	2	5		43	48	6288
5	2	5		64	74	8716
6	2	5		36	43	6372
7	2	5		69	74	4677
8	2	5		56	57	400
9	2	5		33	37	6273
10	2	5		51	54	535
11	2	5		78	85	9131
12	2	5		63	64	3734
13	2	5	•	11	19	9207
14	2	5		20	17	3473
15	2	5	•	11	21	4235
16	2	5		34	31	954
17	2	5		39	42	1940
18	2	5		21	21	2029
19	2	5	•	11	12	1639
20	2	5		29	26	4972
21	2	5		27	27	6526
22	2	5		34	27	5736
23	2	5	•	10	12	3961
24	2	5		18	6	7760
25	2	5	•	9	5	5571
26	2	5		23	18	9022
27	2	5	•	8	9	1694
28	2	5		14	11	530
29	2	5	•	7	4	7705
30	2	5	•	5	8	3164
0	3	5		83	88	7500
1	3	5		63	69	3546
2	3	5		23	27	2631
3	3	5		59	62	9823
4	3	5		62	68	4322
5	3	5		46	62	7636
6	3	5	•	10	10	6076
7	3	5		51	57	5854
8	3	5	•	11	15	9913
9	3	5		34	39	9482
10	3	5	•	11	10	6000
11	3	5		67	79	2690
12	3	5	•	11	8	1027
13	3	5		36	42	4658
14	3	5		30	34	7595
15	3	5	•	11	18	9765
16	3	5	•	11	9	3458
17	3	5		21	24	1359
18	3	5		30	31	2722
19	3	5	•	11	8	4761
20	3	5	•	11	7	6385
21	3	5	•	11	23	1917
22	3	5	•	10	2	8452
23	3	5		18	22	7436
24	3	5	•	10	11	3569
25	3	5		31	31	7177
26	3	5	•	8	5	8982
27	3	5		17	16	1577
28	3	5	•	7	6	6596
29	3	5	•	5	11	4133
0	4	5		38	36	0
1	4	5		31	42	8401
2	4	5		44	47	3502
3	4	5		35	42	8878
4	4	5		45	54	3801

H	K	L		FO	FC	PH
5	4	5		23	21	2704
6	4	5		39	38	1452
7	4	5		28	33	9029
8	4	5		63	63	992
9	4	5		23	23	7782
10	4	5		42	57	490
11	4	5	•	12	9	4515
12	4	5		41	44	7626
13	4	5		24	20	8053
14	4	5		48	55	6758
15	4	5		29	25	2685
16	4	5		24	29	2499
17	4	5		23	26	6853
18	4	5	•	12	5	9397
19	4	5		23	18	8999
20	4	5	•	11	13	6709
21	4	5		26	23	1905
22	4	5		38	43	3373
23	4	5	•	10	7	7834
24	4	5		18	14	1206
25	4	5		17	18	617
26	4	5		18	18	9370
27	4	5	•	6	8	1639
28	4	5		12	14	2453
0	0	6		104	105	5000
1	0	6	•	11	11	5000
2	0	6		70	61	5000
3	0	6		85	79	5000
4	0	6	•	12	12	5000
5	0	6		27	15	5000
6	0	6		52	46	0
7	0	6		37	33	0
8	0	6		47	35	0
9	0	6		47	49	5000
10	0	6		111	108	0
11	0	6		36	36	5000
12	0	6	•	13	5	0
13	0	6		61	56	0
14	0	6	•	13	12	0
15	0	6		45	37	0
16	0	6	•	13	4	0
17	0	6	•	13	6	0
18	0	6		87	90	5000
19	0	6	•	13	6	5000
20	0	6		25	20	5000
21	0	6	•	12	18	5000
22	0	6	•	12	20	5000
23	0	6		29	21	5000
24	0	6	•	11	4	5000
25	0	6		20	16	0
26	0	6		28	26	0
27	0	6	•	8	7	5000
28	0	6	•	7	11	0
0	1	6	•	12	1	7500
1	1	6		51	51	9029
2	1	6		44	43	3285
3	1	6		68	60	7174
4	1	6		24	17	370
5	1	6		60	59	7396
6	1	6		69	62	4690
7	1	6		40	46	7266
8	1	6		30	36	9879
9	1	6	•	12	9	6235
10	1	6		31	22	1391
11	1	6		56	48	1766
12	1	6		78	73	6803
13	1	6		58	51	3112
14	1	6		71	62	8691
15	1	6		75	66	3101
16	1	6		50	46	3453
17	1	6		59	49	741
18	1	6		41	34	2724
19	1	6	•	13	18	8953
20	1	6	•	12	24	9579
21	1	6		24	22	5655
22	1	6	•	12	16	6491
23	1	6	•	11	18	8079
24	1	6	•	11	15	32
25	1	6	•	10	18	7782
26	1	6	•	9	10	4751
27	1	6		27	21	6011
28	1	6	•	7	12	6141

H	K	L		FO	FC	PH
0	2	6		85	81	5000
1	2	6		34	21	4214
2	2	6		115	112	2864
3	2	6		19	20	3995
4	2	6		34	38	4853
5	2	6		20	22	8728
6	2	6		52	53	7475
7	2	6	•	11	13	4354
8	2	6		50	43	1182
9	2	6		46	38	9313
10	2	6		36	38	5315
11	2	6		21	23	746
12	2	6		44	43	9302
13	2	6		10	12	9707
14	2	6		26	17	9699
15	2	6		26	24	9811
16	2	6		26	35	6163
17	2	6	•	11	10	615
18	2	6	•	11	15	7454
19	2	6	•	11	12	4774
20	2	6		39	41	4316
21	2	6	•	10	12	2822
22	2	6	•	10	7	3065
23	2	6		18	20	9440
24	2	6		16	14	9741
25	2	6	•	8	3	9168
26	2	6	•	5	4	7475
27	2	6	•	7	7	2682
28	2	6		19	20	9792
0	3	6		61	66	2500
1	3	6	•	11	23	7670
2	3	6	•	11	16	8314
3	3	6	•	11	15	1658
4	3	6		45	55	6234
5	3	6		50	53	5520
6	3	6		68	69	1278
7	3	6		39	32	3481
8	3	6	•	11	6	290
9	3	6		21	25	1494
10	3	6		36	39	5773
11	3	6		30	30	7744
12	3	6		32	28	2100
13	3	6		47	40	5152
14	3	6		21	25	4541
15	3	6		26	23	4510
16	3	6		29	29	8234
17	3	6		20	31	9725
18	3	6	•	11	13	4131
19	3	6	•	11	14	1251
20	3	6	•	10	15	5433
21	3	6	•	10	20	4532
22	3	6	•	10	11	9042
23	3	6	•	9	7	7105
24	3	6	•	6	6	4567
25	3	6	•	8	11	420
26	3	6	•	7	12	9292
27	3	6		11	5	5961
0	4	6		55	54	0
1	4	6		33	33	9152
2	4	6	•	12	14	3110
3	4	6		50	44	9182
4	4	6		24	25	2233
5	4	6	•	12	11	8170
6	4	6		29	32	8642
7	4	6		34	42	2999
8	4	6		48	47	5787
9	4	6		29	33	581
10	4	6		24	31	3039
11	4	6		24	13	2393
12	4	6		29	28	4288
13	4	6		33	42	4798
14	4	6	•	12	12	7205
15	4	6	•	11	19	6985
16	4	6	•	11	15	334
17	4	6	•	11	11	624
18	4	6	•	11	10	203
19	4	6	•	10	12	4983
20	4	6		28	28	211
21	4	6		19	19	8940
22	4	6		17	11	4054
23	4	6		20	20	9398
24	4	6		15	14	6868

H	K	L		FO	FC	PH
25	4	5		12	12	4677
26	4	5	•	5	7	9488
1	0	7		58	53	2500
2	0	7		65	58	7500
3	0	7	•	11	11	7500
4	0	7		80	75	7500
5	0	7		26	15	7500
6	0	7		49	44	7500
7	0	7	•	13	11	2500
8	0	7	•	13	13	2500
9	0	7		45	39	7500
10	0	7	•	11	7	2500
11	0	7	•	13	11	7500
12	0	7	•	11	8	7500
13	0	7	•	13	5	2500
14	0	7		28	24	2500
15	0	7		36	22	7500
16	0	7		56	52	2500
17	0	7		47	43	2500
18	0	7	•	12	15	7500
19	0	7		23	25	2500
20	0	7		26	20	7500
21	0	7		25	29	7500
22	0	7	•	10	1	7500
23	0	7	•	9	8	2500
24	0	7		29	23	7500
25	0	7	•	8	7	2500
26	0	7		20	21	7500
0	1	7	•	13	14	2500
1	1	7		83	72	4690
2	1	7		80	69	5476
3	1	7	•	13	19	1015
4	1	7	•	13	10	5995
5	1	7		26	30	1342
6	1	7	•	13	20	1657
7	1	7	•	13	12	9323
8	1	7	•	13	18	2773
9	1	7		34	27	8829
10	1	7	•	13	13	1914
11	1	7		63	56	1214
12	1	7	•	13	10	8193
13	1	7		52	47	8166
14	1	7	•	13	18	1141
15	1	7		40	40	7588
16	1	7		23	27	9511
17	1	7		23	21	4255
18	1	7	•	12	10	6929
19	1	7		26	29	5921
20	1	7		18	14	4205
21	1	7		20	16	4708
22	1	7	•	10	6	8243
23	1	7		17	15	2517
24	1	7		13	9	7769
25	1	7	•	8	8	9175
26	1	7		18	17	2899
0	2	7		21	15	0
1	2	7		36	28	871
2	2	7		63	52	8924
3	2	7	•	11	19	9306
4	2	7		30	27	7291
5	2	7		21	19	3722
6	2	7		76	66	5896
7	2	7		36	26	9460
8	2	7	•	11	7	2300
9	2	7		42	39	1771
10	2	7	•	11	10	7756
11	2	7		36	32	4726
12	2	7		55	57	5558
13	2	7		42	35	6516
14	2	7		29	24	4331
15	2	7	•	11	9	1799
16	2	7		31	32	645
17	2	7		19	17	6472
18	2	7	•	10	17	9639
19	2	7	•	10	13	4926
20	2	7	•	9	21	9542
21	2	7	•	9	12	2767
22	2	7	•	8	15	7899
23	2	7		22	25	9019
24	2	7		13	13	6038
25	2	7	•	6	4	279
26	2	7	•	4	9	7455

H	K	L		FO	FC	PH
0	3	7	•	11	3	2500
1	3	7	•	11	16	2089
2	3	7		34	33	9185
3	3	7		74	75	3271
4	3	7		34	34	7928
5	3	7		21	27	2725
6	3	7		21	21	773
7	3	7		21	20	8979
8	3	7		63	60	1752
9	3	7		36	32	3350
10	3	7		24	19	4489
11	3	7		21	8	4900
12	3	7		25	22	5258
13	3	7		20	26	7949
14	3	7	•	11	10	149
15	3	7		28	28	7767
16	3	7		23	19	6237
17	3	7	•	10	12	6100
18	3	7	•	10	11	8724
19	3	7		17	20	2345
20	3	7		20	17	9981
21	3	7		15	14	2325
22	3	7		17	16	6744
23	3	7	•	7	7	3026
24	3	7	•	6	15	8449
25	3	7	•	4	9	3861
0	4	7		38	44	0
1	4	7		38	38	7446
2	4	7		24	13	9417
3	4	7		24	21	773
4	4	7		24	20	4613
5	4	7		29	25	9630
6	4	7		23	24	1490
7	4	7		23	23	1031
8	4	7	•	12	12	6517
9	4	7	•	12	13	3178
10	4	7		28	28	5527
11	4	7		23	19	4615
12	4	7	•	11	7	3192
13	4	7		22	21	3276
14	4	7		21	25	6803
15	4	7	•	10	10	6482
16	4	7		24	23	8304
17	4	7		19	20	6752
18	4	7		29	34	285
19	4	7		24	29	6524
20	4	7		16	10	3186
21	4	7	•	7	4	107
22	4	7	•	6	16	6174
23	4	7	•	5	11	533
0	0	8		30	24	5000
1	0	8		26	21	0
2	0	8		26	17	5000
3	0	8		30	21	5000
4	0	8		26	26	0
5	0	8		45	37	5000
6	0	8		40	40	0
7	0	8		42	30	0
8	0	8		36	27	5000
9	0	8	•	13	14	5000
10	0	8		25	10	5000
11	0	8		23	12	5000
12	0	8		36	35	0
13	0	8	•	12	7	0
14	0	8	•	12	12	0
15	0	8	•	11	9	0
16	0	8	•	11	1	5000
17	0	8	•	11	12	5000
18	0	8	•	10	5	5000
19	0	8		29	29	5000
20	0	8		29	25	5000
21	0	8	•	8	7	0
22	0	8	•	7	7	5000
23	0	8	•	6	1	0
0	1	8		34	25	2500
1	1	8		65	47	9843
2	1	8	•	13	8	4514
3	1	8		46	33	6284
4	1	8		42	28	8602
5	1	8		34	31	8137
6	1	8		24	23	9581
7	1	8	•	13	13	2411

intermolecular distances all exceed 3·15 Å. From analysis of the atomic thermal parameters (see Table 2), the molecule should be affected by a fairly strong rigid oscillation around its centre of gravity, in addition to the intramolecular atomic vibrations (Cruickshank, 1956).

Conclusions

The above structural resolution represents, in our opinion, an interesting example of the usefulness of direct methods when there are more than 3–4 crystallographically independent heavy atoms; even a three-dimensional Patterson approach would not have been trivial at all in the present case. In spite of the substantial similarity between the method applied here and other methods, such as the symbolic addition procedure (Karle & Karle, 1964), the simultaneous use of many Sayre terms, even of a comparatively low power, in a fully automatic procedure has probably been effective in reaching a rapid solution of the phase problem. Other two and three-dimensional problems are now under study with the same method; the complete structural resolution of hydrazonium hydrazinedithiocarboxylate has been obtained through a two-dimensional

Table 3 (*cont.*)

8	1	8		28	29	2758
9	1	8		30	21	5167
10	1	8	•	13	3	434
11	1	8	•	12	17	8089
12	1	8	•	12	22	6430
13	1	8	•	12	19	4744
14	1	8	•	12	18	3948
15	1	8	•	11	15	2425
16	1	8	•	11	15	452
17	1	8	•	11	9	564
18	1	8	•	10	10	4318
19	1	8		30	22	9834
20	1	8		25	16	4419
21	1	8		21	16	9262
22	1	8	•	7	11	9742
23	1	8	•	6	7	5746
0	2	8		33	35	5000
1	2	8		21	12	4765
2	2	8		30	29	3273
3	2	8		47	46	8011
4	2	8		42	31	1998
5	2	8	•	11	22	9758
6	2	8		41	37	2180
7	2	8		46	40	2653
8	2	8	•	11	2	6832
9	2	8		28	22	1996
10	2	8		20	14	9038
11	2	8	•	11	18	4090
12	2	8	•	10	4	7451
13	2	8	•	10	8	4382
14	2	8		26	28	8149
15	2	8		18	17	7324
16	2	8		34	37	7388
17	2	8	•	9	5	2712
18	2	8		14	15	5090
19	2	8	•	8	6	8943
20	2	8		23	19	2441
21	2	8		20	23	6754
22	2	8	•	6	1	1536
23	2	8	•	4	8	9104
0	3	8		20	18	7500
1	3	8		46	53	9511
2	3	8		20	21	7558
3	3	8	•	11	13	8976
4	3	8		25	22	124
5	3	8		25	28	5011
6	3	8		30	29	934
7	3	8		28	23	7737
8	3	8		20	21	1059
9	3	8		27	26	5913
10	3	8	•	10	15	3896
11	3	8		38	40	4398
12	3	8		32	34	4011
13	3	8	•	10	2	7221
14	3	8	•	10	15	5633
15	3	8	•	9	3	9105
16	3	8	•	8	15	6999
17	3	8		26	23	228
18	3	8		20	17	4487
19	3	8		24	28	9092
20	3	8	•	6	8	5981
21	3	8	•	5	8	9862
22	3	8	•	3	19	9120
0	4	8	•	11	8	5000
1	4	8	•	11	15	7214
2	4	8	•	11	22	1932
3	4	8	•	11	13	8238
4	4	8		27	38	2664
5	4	8		27	31	9318
6	4	8	•	11	11	2443
7	4	8		21	13	2041
8	4	8		21	16	556
9	4	8		41	44	3110
10	4	8	•	10	4	1883
11	4	8		20	23	634
12	4	8	•	9	11	6319
13	4	8		13	20	5967
14	4	8		21	22	7748
15	4	8		27	34	4870
16	4	8	•	8	7	8163
17	4	8	•	8	7	8031
18	4	8	•	7	9	3864
19	4	8		12	12	7780
20	4	8	•	4	7	2345
1	0	9	•	12	1	7500
2	0	9		57	50	7500
3	0	9		20	21	7500
4	0	9		41	31	2500
5	0	9		21	20	7500
6	0	9		34	28	2500
7	0	9		23	15	2500
8	0	9	•	11	6	2500
9	0	9		24	23	2500
10	0	9	•	11	6	2500
11	0	9	•	11	7	2500
12	0	9	•	10	13	7500
13	0	9	•	10	3	2500
14	0	9	•	10	2	7500
15	0	9	•	9	6	7500
16	0	9	•	8	11	2500
17	0	9		16	14	7500
18	0	9		14	14	2500
19	0	9	•	6	1	2500
0	1	9		45	35	7500
1	1	9	•	12	9	4156
2	1	9	•	12	8	1821
3	1	9		24	22	658
4	1	9	•	12	6	291
5	1	9		22	19	2022
6	1	9	•	12	19	8353
7	1	9	•	11	9	3385
8	1	9		42	37	1955
9	1	9	•	11	11	5729
10	1	9	•	11	12	2994
11	1	9	•	11	14	5772
12	1	9	•	10	16	5762
13	1	9		22	17	8699
14	1	9	•	10	11	5087
15	1	9		31	26	8368
16	1	9	•	9	7	3761
17	1	9	•	8	12	5924
18	1	9	•	7	5	5296
19	1	9	•	6	10	2810
0	2	9		35	27	0
1	2	9	•	10	7	8917
2	2	9	•	10	21	9224
3	2	9	•	10	10	8088
4	2	9		26	21	7097
5	2	9		32	22	9865
6	2	9		26	21	2702
7	2	9		36	34	324
8	2	9		18	13	3818
9	2	9		17	17	3692
10	2	9		34	30	5603
11	2	9		23	21	4433
12	2	9		19	22	4584
13	2	9	•	8	9	1919
14	2	9		14	4	4190
15	2	9	•	8	10	5566
16	2	9		18	21	781
17	2	9	•	6	12	5637
18	2	9		17	14	9902
0	3	9	•	10	5	7500
1	3	9	•	10	6	3360
2	3	9		22	22	7199
3	3	9		25	25	3725
4	3	9	•	10	12	9424
5	3	9		25	25	2149
6	3	9		17	11	560
7	3	9		19	21	2263
8	3	9	•	8	12	2316
9	3	9	•	9	8	2624
10	3	9		35	40	2366
11	3	9		18	18	9826
12	3	9	•	8	5	6611
13	3	9		17	17	7503
14	3	9		13	16	6373
15	3	9		29	28	6404
16	3	9	•	6	11	4176
17	3	9	•	5	4	8508
0	4	9		23	27	0
1	4	9		18	22	6130
2	4	9		18	15	969
3	4	9	•	9	7	8258
4	4	9	•	9	6	7821
5	4	9		21	21	180
6	4	9		21	24	7305
7	4	9		17	14	9136
8	4	9		17	15	3019
9	4	9	•	8	9	9986
10	4	9	•	8	16	4489
11	4	9	•	7	12	3460
12	4	9		14	12	4920
13	4	9		12	14	4830
14	4	9		11	21	2578
15	4	9		13	5	9214
0	0	10		21	18	0
1	0	10		19	9	5000
2	0	10	•	10	5	0
3	0	10		24	21	0
4	0	10		21	10	0
5	0	10		35	29	0
6	0	10		20	14	0
7	0	10	•	9	4	0
8	0	10		21	20	5000
9	0	10	•	8	5	0
10	0	10		31	24	5000
11	0	10	•	7	9	5000
12	0	10	•	7	4	0
13	0	10		21	18	5000
0	1	10		17	13	7500
1	1	10	•	10	4	6891
2	1	10		27	23	6425
3	1	10		17	18	33
4	1	10		14	14	9689
5	1	10		28	19	3096
6	1	10		23	20	673
7	1	10		28	24	2964
8	1	10	•	9	9	2787
9	1	10	•	8	3	5705
10	1	10		20	18	2885
11	1	10		18	17	5810
12	1	10	•	7	10	2837
13	1	10	•	6	3	4133
0	2	10	•	8	6	5000
1	2	10	•	8	4	8659
2	2	10	•	8	6	686
3	2	10	•	8	17	7571
4	2	10		20	20	1687
5	2	10	•	8	9	9096
6	2	10	•	8	8	3054
7	2	10		18	21	920
8	2	10		12	13	4363
9	2	10		16	15	2331
10	2	10	•	6	7	3784
11	2	10	•	6	11	2020
12	2	10		11	19	7482
13	2	10		13	6	6189
0	3	10	•	7	5	7500
1	3	10	•	7	8	9861
2	3	10	•	7	14	4885
3	3	10	•	7	6	9140
4	3	10	•	7	8	70
5	3	10	•	7	2	1193
6	3	10		17	20	9545
7	3	10		14	15	5252
8	3	10	•	6	9	908
9	3	10	•	5	8	5720
10	3	10	•	5	7	5134
11	3	10	•	3	12	3683
0	4	10		16	17	5000
1	4	10	•	6	5	9765
2	4	10	•	6	11	2959
3	4	10	•	6	5	9403
4	4	10		11	5	1327
5	4	10	•	5	12	6236
6	4	10		9	13	2693
7	4	10		8	6	7998

approach (Braibanti, Manotti Lanfredi, Tiripicchio & Logiudice, 1969).

The above reported comparison between the e.s.d., either obtained from internal comparison of chemically equivalent molecular parameters or from the average of the values resulting from the least-squares refinement, suggests that the latter values are always lower by an amount of at least 30–50%. Among the factors which may contribute to the observed discrepancy are the influence of the crystal packing, and, perhaps, the neglecting of any anisotropic thermal effect on the light atoms, and the introduction of the weighting scheme derived from an approximate formula given by Cruickshank (1965).

APPENDIX

A sequence of four independent computing programs, written in FORTRAN IV language, covers the mathematical processing of the data; the initial input is represented by the list of structure factors, on an arbitrary scale, while the final output consists of the most probable sign combinations applied to the strongest structure factors, after scale reduction. Up to 30 sign combinations may be obtained, in a format suitable for direct Fourier synthesis processing. The programs are now suitable for any centrosymmetrical space group, both in two and three dimensions.

In the following, a concise description of the four programs is given. More detailed instructions can be sent from the author on request, together with the programs themselves, which are prepared on punched cards.

(I) *Evaluation of normalized structure factors*

From the $F'(hkl)$ non-scaled structure factors the $E(hkl)$ normalized structure factors ($\bar{E}^2=1$) are derived by first calculating both the K scale factor and the B average temperature factor through a least-squares derivation of Wilson's plot (Wilson, 1942). No correction for possible non-linearity of the plot at low $\sin\theta$ (Rogers, 1965), has been applied hitherto. $E(hkl)$ is then obtained from the relationship:

$$E(hkl)=\frac{F'(hkl)}{K\cdot\sigma_2^{1/2}\cdot\exp\left(-B\frac{\sin^2\theta}{\lambda^2}\right)}\,;(\sigma_n=\Sigma f_i^n)\,. \quad (1a)$$

Printing of the results comprises the B and K values, and a full list of the reflexions, together with the scaled and the normalized structure factors. The reflexions with $E>{}^2E$ (arbitrarily chosen limit) are also punched on cards, in decreasing order of E values. The progressive index number for each reflexion is also punched.

(II) *Search for the* $(\mathbf{H}_i,\mathbf{H}_j,\mathbf{H}_k)$ *triads with* $\mathbf{H}_i+\mathbf{H}_j+\mathbf{H}_k=0$ $[\mathbf{H}_i\equiv(h_i,k_i,l_i)]$

All the different Sayre triads of reflexions are selected in this program, subject to the condition that at least two of their E factors are larger than another proper limit, ${}^1E(>{}^2E)$. Then the α_{ijk} values are calculated [see expression (2)], and the output is both printed and punched on cards; on the latter the Bragg indices are omitted, and the three reflexions of each triad are only recorded by their corresponding index numbers (see preceding section).

(III) *Calculation of the most probable sets of signs of the base reflexions*

Either 12, or 18, or 24 base reflexions are chosen among those with highest normalized factors, in decreasing order (they must be in any case $>{}^1E$), following the additional criterion that they must appear in many Sayre groups. The first two base reflexions

Table 4. *Bond lengths and angles*

Bond lengths

Co(1)	Co(2)	2·46$_4$Å	Co(4)	C(3′)	1·92Å
Co(2)	Co(3)	2·45$_6$	Co(5)	C(3′)	1·92
Co(3)	Co(1)	2·46$_0$	Co(6)	C(3′)	1·94
Co(1)	C(1′)	1·93	Co(4)	C(10)	1·84
Co(2)	C(1′)	1·87	C(10)	O(10)	1·00
Co(3)	C(1′)	1·81	Co(4)	C(11)	1·81
Co(1)	C(4)	1·79	C(11)	O(11)	1·11
C(4)	O(4)	1·19	Co(4)	C(12)	1·85
Co(1)	C(5)	1·85	C(12)	O(12)	1·14
C(5)	O(5)	1·08	Co(5)	C(16)	1·79
Co(1)	C(9)	1·77	C(16)	O(16)	1·21
C(9)	O(9)	1·11	Co(5)	O(17)	1·80
Co(2)	C(6)	1·84	C(17)	O(17)	1·20
C(6)	O(6)	1·08	Co(5)	C(18)	1·80
Co(2)	C(7)	1·91	C(18)	O(18)	1·04
C(7)	O(7)	1·04	Co(6)	C(13)	1·83
Co(2)	C(8)	1·84	C(13)	O(13)	1·14
C(8)	O(8)	1·04	Co(6)	C(14)	1·69
Co(3)	C(1)	1·89	C(14)	O(14)	1·26
C(1)	O(1)	1·03	Co(6)	C(15)	1·88
Co(3)	C(2)	1·86	C(15)	O(15)	1·17
C(2)	O(2)	1·04	C(1′)	C(2′)	1·60
Co(3)	C(3)	1·80	C(2′)	C(3′)	1·42
C(3)	O(3)	1·15	C(2′)	O(2′)	1·24
Co(4)	Co(5)	2·48$_8$			
Co(5)	Co(6)	2·48$_2$			
Co(6)	Co(4)	2·44$_9$			

Bond angles

Co(1)	Co(2)	Co(3)	60·0°	Co(5)	Co(4)	C(3′)	49·5°
Co(2)	Co(3)	Co(1)	60·2	Co(4)	C(3′)	C(2′)	147·0
Co(3)	Co(1)	Co(2)	59·8	Co(5)	C(3′)	C(2′)	124·4
Co(3)	Co(1)	C(1′)	46·7	Co(6)	C(3′)	C(2′)	122·4
Co(2)	Co(1)	C(1′)	48·5	Co(4)	C(3′)	Co(5)	81·0
Co(3)	Co(2)	C(1′)	47·0	Co(4)	C(3′)	Co(6)	78·9
Co(1)	Co(2)	C(1′)	50·6	Co(5)	C(3′)	Co(6)	80·2
Co(2)	Co(3)	C(1′)	49·1	C(5)	Co(1)	C(4)	103·7
Co(1)	Co(3)	C(1′)	50·9	C(5)	Co(1)	C(9)	99·1
Co(3)	C(1′)	C(2′)	148·8	C(4)	Co(1)	C(9)	91·2
Co(2)	C(1′)	C(2′)	120·5	C(7)	Co(2)	C(6)	98·7
Co(1)	C(1′)	C(2′)	118·1	C(7)	Co(2)	C(8)	105·5
Co(3)	C(1′)	Co(1)	82·4	C(6)	Co(2)	C(8)	95·5
Co(3)	C(1′)	Co(2)	83·9	C(2)	Co(3)	C(3)	96·1
Co(2)	C(1′)	Co(1)	81·0	C(2)	Co(3)	C(1)	95·2
C(1′)	C(2′)	C(3′)	122·9	C(3)	Co(3)	C(1)	98·3
C(1′)	C(2′)	O(2′)	118·0	C(5)	Co(1)	Co(3)	102·8
O(2′)	C(2′)	C(3′)	119·1	C(5)	Co(1)	Co(2)	99·6
Co(4)	Co(5)	Co(6)	59·0	C(4)	Co(1)	C(1′)	104·2
Co(5)	Co(6)	Co(4)	60·6	C(4)	Co(1)	Co(3)	100·1
Co(6)	Co(4)	Co(5)	60·4	C(9)	Co(1)	C(1′)	106·0
Co(4)	Co(5)	C(3′)	49·5	C(9)	Co(1)	Co(2)	99·9
Co(6)	Co(5)	C(3′)	50·3	C(6)	Co(2)	C(1′)	104·6
Co(5)	Co(6)	C(3′)	49·5	C(6)	Co(2)	Co(1)	98·6
Co(4)	Co(6)	C(3′)	50·2	C(8)	Co(2)	C(1′)	99·4
Co(6)	Co(4)	C(3′)	50·9	C(8)	Co(2)	Co(3)	95·9
C(7)	Co(2)	Co(1)	99·2	C(17)	Co(5)	Co(6)	87·8

must be linearly independent mod. 2, and they are given two arbitrary signs.

Suppose 24 base reflexions are being considered: the corresponding index numbers are fed into the computer, together with the signs of the first two reflexions. With reference to expression (1), the α_{ijk} terms are selected, where i, j and k all correspond to the base set, and stored in different sections of the computer depending on whether they are all comprised in the group of the first 12 reflexions, or partly in the group 1 to 12, partly in the group 13 to 18, or at least one of them belongs to the group 19 to 24. Following analogous selection criteria, the products $\alpha_{ijn} . \alpha_{khn}$ are computed, where n need not belong to the base set. The program then proceeds through three further steps: in the first, only the first 12 reflexions are considered. Accounting for the two previously fixed signs, ten of them are still undetermined: they are combined in all possible ways ($2^{10}=1024$), and for each combination the corresponding $\Pi(s_i s_j s_k \ldots)$ is evaluated. Always

Table 4 (*cont.*)

C(7)	Co(2)	Co(3)	103·9
C(1)	Co(3)	C(1′)	108·8
C(1)	Co(3)	Co(2)	101·1
C(3)	Co(3)	Co(1)	96·7
C(3)	Co(3)	C(1′)	107·6
C(2)	Co(3)	Co(1)	99·2
C(2)	Co(3)	Co(2)	99·6
C(10)	Co(4)	C(11)	98·9
C(10)	Co(4)	C(12)	99·8
C(11)	Co(4)	C(12)	95·1
C(16)	Co(5)	C(17)	101·7
C(16)	Co(5)	C(18)	92·7
C(17)	Co(5)	C(18)	103·1
C(13)	Co(6)	C(14)	96·5
C(13)	Co(6)	C(15)	103·1
C(14)	Co(6)	C(15)	104·4
C(10)	Co(4)	C(3′)	105·1
C(10)	Co(4)	Co(6)	94·8
C(11)	Co(4)	Co(5)	98·8
C(11)	Co(4)	Co(6)	100·1
C(12)	Co(4)	C(3′)	107·7
C(12)	Co(4)	Co(5)	100·4
C(16)	Co(5)	C(3′)	114·3
C(16)	Co(5)	Co(4)	99·0
C(17)	Co(5)	Co(4)	103·2
C(18)	Co(5)	C(3′)	98·8
C(18)	Co(5)	Co(6)	104·8
C(13)	Co(6)	C(3′)	102·9
C(13)	Co(6)	Co(5)	88·8
C(14)	Co(6)	C(3′)	96·7
C(14)	Co(6)	Co(4)	103·4
C(15)	Co(6)	Co(4)	96·3
C(15)	Co(6)	Co(5)	107·1
Co(1)	C(4)	O(4)	171·6
Co(1)	C(5)	O(5)	174·1
Co(1)	C(9)	O(9)	175·4
Co(2)	C(6)	O(6)	168·8
Co(2)	C(7)	O(7)	167·8
Co(2)	C(8)	O(8)	175·4
Co(3)	C(1)	O(1)	166·6
Co(3)	C(2)	O(2)	171·6
Co(3)	C(3)	O(3)	169·5
Co(4)	C(10)	O(10)	169·7
Co(4)	C(11)	O(11)	174·6
Co(4)	C(12)	O(12)	176·8
Co(5)	C(16)	O(16)	160·7
Co(5)	C(17)	O(17)	174·2
Co(5)	C(18)	O(18)	169·7
Co(6)	C(13)	O(13)	171·7
Co(6)	C(14)	O(14)	170·4
Co(6)	C(15)	O(15)	169·2

referring to expression (1), the indices *i*, *j*, *k* and *l* are now included in the set which corresponds to the first 12 base reflexions. Suppose, for the sake of clarity, that the above indices run from 1 to 12, and so on: $\Pi(s_1 \ldots s_{12})$ may be taken as linearly related to the overall probability. Only those sign combinations which correspond to the highest Π values are stored; their total number may be chosen in the range 30 to 60. In the second step each $(s_1 \ldots s_{12})$ combination is tested against the next $s_{13} \ldots s_{18}$ signs; the latter are also permuted in all possible ways, and each time $\Pi(s_1 \ldots s_{10})$ is evaluated in keeping with expression (1), for a total of (30 to 60) $\times\ 2^6 = 1920$ to 3840 sign combinations. The 30 of these with highest Π are stored, and each tested against the $s_{19} \ldots s_{24}$ remaining signs (1920 combinations; third step). Finally, the 30 most probable $(s_1 \ldots s_{24})$ sign combinations are printed and punched on cards, together with their resulting Π value. The α_{ijn} terms, where n does not belong to the base set, are also punched with their i,j,n indices.

Other kinds of progressive factorization could be devised, for the same purpose of avoiding the permutation of 24 signs in 2^{22} different ways. In fact, a typical figure for the total time of computing, using the above program, is 15 minutes for as many as 100 reflexions with $E_i > {}^2E$ and 260 Sayre triads to be used in expression (1) (IBM-7040).

(IV) *Sign generation for the non-base reflexions*

The input to this program consists of the punched cards output of the programs I and III. The signs of the non-base reflexion are determined for each of the basic sign combinations; some of the latter ones, however, can be discarded from the beginning. The prob-

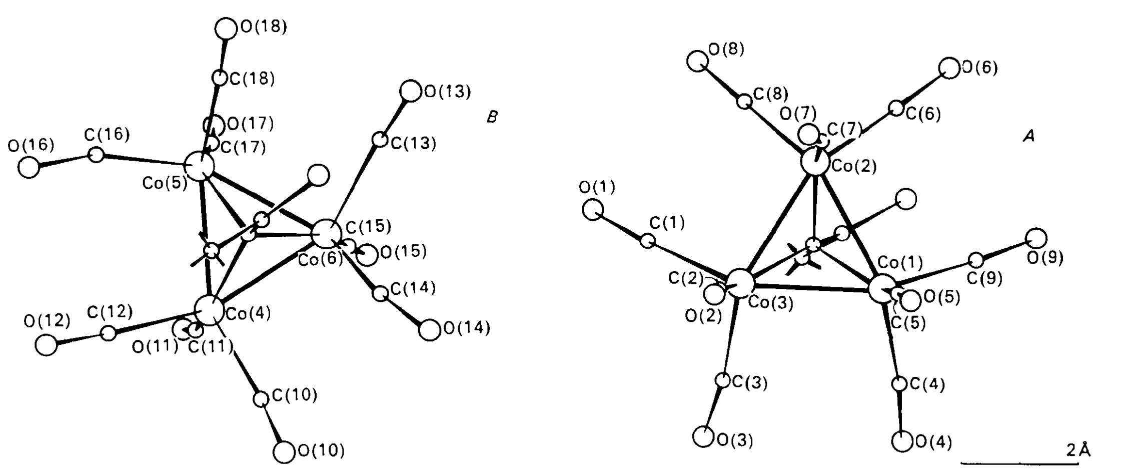


Fig. 4. Conformation of the *A* and *B* tetrahedra (see Fig. 1), viewed perpendicularly to the planes of the cobalt atoms.

Table 5. *Average values of bond lengths and angles, compared with those of the analogous* $Co_3(CO)_9C_2H_3$,* *and comparisons between the error standard deviations obtained for our compound from internal comparison of the data (s) and those resulting from the least-squares refinement* (σ)

Symbols of the atoms: C_A and C_E are carbonyl C atoms in axial and equatorial position with respect to the Co_3 triangle; C_T are tetrahedral atoms.

	Distance, or angle (average value)	Corresponding average values for $Co_3(CO)_9C_2H_3$	s	σ	s/σ	Corresponding number of determinations
Co–Co	$2·46_6$ Å	$2·46_8$ Å	0·015 Å	0·012 Å	1·32	6
Co–C_A (or Co–C_E)	$1·82_3$	1·80	0·053	0·038	1·39	18
C_A–(or C_E–)O	$1·11_2$	1·10	0·074	0·048	1·54	18
Co–C_T	$1·89_4$	1·90	0·050	0·024	2·17	6
C_T–C	1·51	1·53	0·127	0·036	3·53	2
C–O (central group)	1·24	—	—	0·033	—	1
C_T–Co–Co	49·4°	49·4°	1·32°	0·87°	1·52	12
C_A–Co–C_E	100·3	102·2	3·72	0·83	4·48	12
C_E–Co–C_E	95·7	97·2	3·18	0·93	3·42	6
C_A–(or C_E–)Co–Co (when < 130°)	99·2	98·2	4·49	0·71	6·32	24
C_E–Co–C_T	104·7	102·7	4·86	0·75	6·47	12
Co–C_T–Co	81·3	81·1	1·70	0·55	3·09	6
Co–C–O	171·0	175·0	3·76	18·50	0·20	18
C_T–C–C_T	122·9	–	—	1·29	—	1
C_T–C–O	118·5	–	1·18	1·23	0·96	2
Co–C_T–C	130·2	131·3	13·64	1·55	8·80	6

*Sutton & Dahl (1967).

able value of s_m (E_m must be $>{}^2E$) is assumed to be equal to the sign of

$$S_m = \sum_{i,j} s_i s_j \alpha_{ijm} ,$$

where s_i and s_j are the (known) signs of a pair of base reflexions. When all the S_m are calculated, s_m is accepted only if $|S_m|$ exceeds a prefixed value (for instance 0·8). The program output consists of the printed-punched list of the (h,k,l) indices, together with the (signed) $F(hkl)$ scaled structure factors, with a format suitable for direct Fourier synthesis processing.

Less than 5 minutes is required for computing, considering 10 base combinations of 24 signs, and 100 reflexions with $E>{}^2E$ and 260 Sayre triads (IBM-7040).

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