are less affected by the ring strain. All C–P–C bond angles are very similar to those found in substituted non-coordinated phosphines (103°) (Daly, 1970). Thus the hybridization of the phosphorus atoms seems not to differ significantly from the tetrahedral sp^3 type. It is obvious on the other hand from the bonding angles of the heavy atom framework, *e.g.* the acute P–Ni–P angle (Fig. 3), that the orbital overlap has to take place through bent bonds, the region of maximum overlap being displaced by about 0·3 Å from the Ni–P axis, assuming exact tetrahedral hybridisation of the nickel (see Fig. 4). The contacts Ni–C(1) (2·963 Å) and Ni–C(2) (2·983 Å) seem to be too large to have any influence on the overall molecular arrangement.

All cyclohexyl rings are in the chair conformation and bonded equatorially to the phosphorus. The P–C distances are in the range of our previous measurements on similar compounds (Barnett & Krüger, 1972; Krüger & Tsay, 1972). The average C–C distance in the cyclohexyl rings is 1·529 Å. Fig. 5 shows a stereoscopic view of the unit-cell along the $-b$ axis.

Non-bonded distances

There are no abnormally short intermolecular distances. Thus it seems that the observed molecular skeleton is not affected by packing forces. A list of contacts less than 4 Å is given in Table 5.

Table 5. *Interatomic contacts less than* 4 Å

C(15)–C(64)	3·802 Å
C(24)–C(55)	3·810
C(33)–C(25)	3·998
C(46)–C(13)	3·959
C(54)–C(63)	3·934
C(63)–C(74)	3·941
C(64)–C(76)	3·945
C(65)–C(86)	3·719

All computations were carried out on a PDP-10 time-sharing computer with local versions of commonly available crystallographic programs. Some programs were of local origin.

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The Crystal and Molecular Structure of Monofluoroacetic Acid

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Crystals of monofluoroacetic acid are monoclinic with $a=4\cdot30$, $b=7\cdot55$, $c=9\cdot98$ Å and $\beta=85\cdot2°$. The structure was solved by application of the $B_{3,0}$ formula to three-dimensional photographic data. It consists of centrosymmetrical dimers formed by carboxyl group coupling. The carbon–carbon distance is found to be extremely short.

Introduction

As a part of our research program on the structure and conformation of substituted mono and dicarboxylic acids the structures of tartronic acid (van Eijck, Kanters & Kroon, 1965) and monofluoromalonic acid (Roelofsen, Kanters, Kroon & Vliegenthart, 1971) were determined. These structures are remarkably similar in that the molecules form carboxyl-group coupled polymeric chains which are packed in almost identical ways. Strong chain interactions are absent in the two structures. This is reflected in the fact that the alcoholic hydroxyl group in tartronic acid is not hydrogen bonded (Kanters, Kroon, Peerdeman & Vliegenthart, 1967). In the recently determined structure of glycollic acid (Ellison, Johnson & Levy, 1971; Pijper, 1971) the alcoholic hydroxyl group plays an important part in the hydrogen bond scheme, thus excluding an isotypical structure for monofluoroacetic acid. Our interest in the molecular conformation, which may be the same in both molecules, prompted us to investigate the crystal structure of monofluoroacetic acid.

Experimental

After sublimation *in vacuo* under strict anhydrous conditions, a block-shaped crystal (dimensions 0·3 × 0·6 × 0·8 mm) was transferred to a climate box and mounted along its longest dimension in a sealed capillary tube. The crystals belong to the monoclinic

Table 1. *Positional and thermal parameters*

The temperature factors for the heavy atoms are of the form $\sum_i \sum_j h_i h_j \beta_{ij} \times 10^{-4}$. The estimated standard deviations are 0·005 Å for the heavy atoms and 0·05 Å for the hydrogen atoms.

	x	y	z	β_{11}	β_{22}	β_{33}	$2\beta_{12}$	$2\beta_{23}$	$2\beta_{31}$
F(1)	−0·0374	−0·0752	0·3546	591	233	97	−75	24	160
O(2)	0·5477	0·1319	0·1469	522	175	94	−207	−42	94
O(3)	0·2305	−0·0914	0·1057	514	182	71	−252	2	46
C(4)	0·1964	0·0440	0·3202	448	175	75	−6	−2	88
C(5)	0·3232	0·0202	0·1812	386	142	76	28	31	15
H(6)	0·680	0·095	0·065	3 Å^2					
H(7)	0·100	0·180	0·330	3 Å^2					
H(8)	0·380	0·010	0·375	3 Å^2					

Table 2. *Observed and calculated structure factors*

For each reflexion the order of entries is h, k, l and values of F_{obs} and F_{calc} on absolute scale.

0	0	4	56.0	-60.8
		6	24.1	23.3
		8	3.5	2.3
		10	2.6	-1.4
		12	1.8	1.8
0	1	2	16.7	-14.6
		3	2.2	1.7
		4	14.4	13.2
		5	11.6	-10.5
		6	5.8	5.3
		7	5.1	-5.0
		8	9.7	-9.9
		9	4.5	4.8
		11	2.5	2.5
		12	3.5	4.1
0	2	1	2.9	-2.4
		2	19.1	-17.5
		3	18.9	17.5
		4	21.4	-18.3
		5	2.6	1.7
		6	13.3	11.5
		7	13.2	-12.4
		8	4.1	-4.0
		11	3.2	2.8
		12	2.1	1.6
0	3	1	14.4	-12.7
		2	9.7	-10.0
		3	11.7	11.4
		4	15.8	14.5
		5	5.7	5.8
		6	4.3	3.6
		7	6.8	-6.5
		8	11.1	-11.5
		11	2.4	2.2
		12	4.4	5.3
0	4	0	17.0	-15.9
		1	9.1	8.9
		2	5.3	-4.3
		3	16.8	15.8
		4	15.1	14.6
		5	6.9	-7.2
		6	2.3	1.9
		7	9.0	-9.4
		8	6.0	-7.0
		9	4.8	5.8
		10	2.1	2.3
		11	1.0	.6
0	5	1	5.0	-4.7
		2	4.5	4.4
		3	2.0	-1.4
		4	1.9	-1.0
		5	8.3	7.6
		9	3.3	-3.7
0	6	0	6.5	-7.3
		1	2.3	-1.6
		2	2.5	-2.0
		3	3.0	3.1
		4	6.2	6.2
		5	1.4	-2.0
		6	2.6	2.2
		7	2.7	-2.8
		8	3.0	-3.2
		9	3.5	4.9
0	7	1	3.9	4.0
		3	7.9	-8.1
		4	2.6	-2.8
		5	2.9	3.0
		7	3.1	3.0
		8	2.2	2.8
0	8	1	6.9	-7.1
		3	2.1	-2.1
		5	3.7	3.8
0	9	1	1.8	2.3
		2	1.8	-2.5
		3	2.3	-3.3
1	0	-12	2.4	2.3
		-10	1.7	1.5
		-8	4.1	-4.1
		-6	8.0	-7.6
		-4	5.2	3.3
		-2	17.2	17.1
		2	17.9	-17.7
		4	12.3	-12.0
		6	8.0	7.8
1	0	8	8.1	7.8
		10	1.5	-1.6
		12	3.0	-2.9
1	1	-11	3.8	4.6
		-10	2.0	1.7
		-8	3.2	3.0
		-7	12.2	-11.1
		-6	5.8	-5.0
		-5	11.1	-9.5
		-4	5.5	-5.2
		-3	40.1	43.0
		-2	1.8	-.9
		-1	4.0	4.3
		2	16.2	16.0
		3	28.7	31.3
		4	11.7	-11.6
		5	2.3	1.3
		6	7.0	-6.7
		7	14.8	-13.2
		8	5.0	4.7
		9	12.7	12.5
1	2	-11	3.5	3.8
		-10	1.6	-1.4
		-9	5.4	-5.1
		-8	2.6	2.2
		-7	3.1	2.2
		-5	12.7	11.5
		-4	9.7	-9.6
		-3	31.0	-31.0
		-2	9.4	8.7
		-1	6.5	-6.1
		0	13.1	13.1
		2	11.4	-11.3
		3	4.3	-3.7
		4	10.0	-9.6
		5	28.5	-29.6
		6	5.3	5.7
		7	4.6	3.8
		8	4.6	4.6
		9	8.4	8.6
		10	1.9	-2.1
		11	1.3	-1.0
		12	1.7	-1.3
1	3	-11	3.5	-3.1
		-10	4.2	3.8
		-9	1.8	-1.9
		-8	8.0	8.6
		-7	11.1	11.5
		-6	14.6	-14.3
		-5	10.5	-10.4
		-4	9.7	-9.1
		-3	7.9	-7.5
		-2	14.1	13.5
		-1	18.7	18.8
		0	6.0	6.0
		1	8.9	-9.1
		2	8.7	7.8
		4	9.5	-8.2
		5	1.8	2.0
		6	6.6	-6.2
		7	4.3	-3.5
		8	4.9	4.2
		9	3.5	2.9
1	4	-10	3.4	-2.9
		-9	1.6	-1.2
		-8	5.4	5.4
		-7	1.7	1.6
		-6	5.2	4.6
		-5	2.8	2.1
		-4	10.7	-9.7
		-3	13.4	-12.6
		-2	2.1	-2.8
		0	5.2	4.8
		1	20.1	19.6
		2	2.8	2.0
		3	4.1	-3.2
		4	2.4	2.2
		5	11.1	-10.2
		6	2.8	-1.8
		7	1.6	1.0
		8	1.8	-1.8
		9	2.9	2.7
1	5	-10	3.0	2.2
		-9	1.2	-1.0
1	5	-8	6.7	7.5
		-7	11.1	11.9
		-6	11.8	-12.5
		-5	5.3	-4.7
		-3	13.3	-12.5
		-2	15.7	14.9
		-1	8.7	7.7
		0	9.6	-9.3
		1	2.1	1.9
		2	2.8	-2.3
		4	3.7	4.0
		5	2.0	2.1
		7	3.1	-2.9
1	6	-4	1.7	1.9
		-3	1.8	1.1
		-1	2.5	.9
		0	9.2	-9.5
		1	4.4	-4.2
		2	5.5	5.7
		4	9.7	9.8
		5	4.0	4.1
		6	6.1	-6.8
		8	2.9	-3.4
		9	1.6	-2.0
1	7	-7	2.0	2.0
		-6	2.6	-3.1
		-4	4.9	4.8
		-2	1.7	1.9
		0	8.9	-9.0
		2	3.7	3.2
		3	3.0	2.8
		4	2.7	2.2
		7	1.9	-2.2
1	8	-5	1.2	1.3
		-4	1.7	2.1
		-3	2.0	1.8
		-2	2.3	1.8
		-1	2.3	-2.2
		0	4.9	-5.6
		1	2.4	-2.3
		3	1.6	1.3
		4	3.9	4.8
		5	1.4	1.5
		6	2.7	-3.5
2	0	-10	4.1	-5.6
		-8	8.6	9.2
		-6	1.7	-1.8
		-4	1.8	-2.0
		-2	7.1	7.0
		2	10.2	-11.8
		4	13.9	-12.1
		6	20.4	18.6
		8	4.9	4.4
		10	10.2	-11.8
2	1	-10	3.1	3.3
		-9	3.9	-4.0
		-8	2.7	2.0
		-7	4.6	-4.3
		-6	15.8	-13.5
		-5	11.9	10.7
		-4	1.5	.8
		-3	10.4	10.3
		-2	24.4	26.8
		-1	12.4	-14.2
		2	16.9	-19.8
		3	6.7	4.6
		4	9.2	8.8
		5	7.7	7.4
		6	4.7	4.1
		7	1.2	1.0
		8	3.2	-2.4
		9	3.5	-3.3
2	2	-10	.7	-.1
		-9	.9	-.4
		-8	3.0	3.6
		-7	8.4	-8.4
		-6	7.5	-7.1
		-5	3.1	2.2
		-4	8.3	7.4
		-3	5.8	6.1
		-2	3.9	3.5
		-1	4.4	5.0
		0	9.6	-10.0
2	2	1	1.4	-.9
		2	4.8	4.3
		3	14.0	-13.2
		4	7.2	-6.1
		5	2.3	1.4
		6	4.8	4.1
		7	5.8	5.7
		8	5.0	4.6
		9	1.0	-.2
		10	4.9	-5.6
2	3	-10	3.2	3.9
		-8	2.6	2.1
		-7	6.1	-5.9
		-6	17.3	-16.0
		-5	4.8	4.2
		-3	6.4	5.8
		-2	27.2	29.7
		-1	11.3	-10.8
		0	9.3	-8.6
		1	2.9	2.8
		2	21.5	-21.7
		3	7.6	7.2
		4	9.8	8.1
		5	5.7	-5.1
		6	4.4	3.9
		7	1.1	-1.3
		8	2.1	-1.6
		9	1.7	1.8
		10	1.5	1.6
2	4	-9	3.0	-2.9
		-8	1.2	-.9
		-7	4.3	-4.8
		-6	9.2	-9.1
		-5	9.9	9.2
		-4	10.9	9.8
		-3	9.0	-6.3
		-2	2.2	-2.1
		-1	3.5	-2.9
		0	10.1	-8.9
		1	14.3	15.0
		2	15.4	16.0
		3	10.2	-10.3
		4	3.9	-3.8
		5	5.7	-5.9
		6	8.4	-8.6
		7	5.9	6.5
		8	4.6	4.6
		10	.6	1.1
2	5	-6	1.9	-1.2
		-5	4.3	-3.9
		-4	2.0	-1.7
		-3	3.2	-3.2
		-2	2.7	2.3
		-1	1.6	1.7
		0	2.2	1.8
		1	8.8	9.0
		2	1.6	-1.1
		3	1.0	-1.1
		5	7.0	-7.4
		6	1.3	-.8
		7	1.9	1.6
		8	1.0	.9
		9	1.5	2.0
2	6	-6	3.2	-3.1
		-5	6.5	6.9
		-4	1.7	.9
		-3	7.9	-8.8
		-2	2.7	-2.1
		-1	3.5	-3.5
		0	1.1	.5
		1	14.0	12.4
		2	6.8	6.9
		3	4.2	-4.8
		4	3.4	-3.4
		5	2.9	-3.0
		6	3.4	-4.0
		7	2.3	2.8
		8	1.8	2.2
2	7	-6	2.2	2.6
		-5	3.3	-3.9
		-4	1.2	-1.4
		-3	2.3	-3.1
		-2	3.6	-3.9
2	7	-1	2.7	3.2
		0	2.0	2.0
		1	3.7	4.2
		2	2.4	2.3
		3	2.5	-3.1
		5	1.9	-2.3
		7	1.8	2.6
2	8	-3	1.4	-2.2
		1	1.8	2.2
		3	1.2	-1.3
3	0	-8	3.0	3.3
		-6	6.7	6.4
		-4	5.6	-4.4
		4	4.0	4.3
		6	1.5	-1.0
		8	3.2	-3.6
		10	.7	.7
3	1	-9	4.4	-4.3
		-8	1.2	-1.4
		-7	5.4	-5.7
		-6	3.1	3.3
		-5	19.1	19.3
		-4	.8	.8
		-3	7.3	-7.1
		-2	.7	.3
		4	2.6	2.5
		5	1.3	.5
		6	2.2	2.2
		7	3.7	-4.2
		8	1.1	-.5
		9	2.1	-2.4
3	2	-9	7.0	-6.9
		-8	2.6	3.0
		-7	2.9	3.1
		-6	3.5	3.2
		-5	10.9	11.0
		-4	6.3	-5.8
		-3	7.2	-7.0
		-2	1.2	-.4
		-1	6.7	-7.1
		0	5.7	4.6
		1	7.9	7.5
		2	4.0	-3.9
		3	3.3	-3.4
		4	1.9	1.1
		5	3.5	-2.7
		6	3.2	2.9
		7	5.6	5.8
		8	2.3	-2.4
3	3	-8	1.3	-1.9
		-6	1.6	2.0
		-5	4.5	4.2
		-3	6.4	-5.7
		-2	6.6	6.9
		-1	4.1	4.5
		0	3.9	-4.9
		1	4.6	3.3
		2	12.0	-11.2
		3	11.7	-10.5
		4	7.6	7.7
		5	5.2	5.3
		6	3.3	4.1
		7	2.2	2.9
		8	2.1	-2.7
		9	3.3	-3.7
3	4	-6	1.2	-1.6
		-5	3.5	3.6
		-4	1.6	-1.6
		-2	4.3	5.1
		-1	7.6	-7.3
		0	4.3	3.6
		2	7.8	-8.1
		3	1.6	-1.4
		4	1.7	-1.5
		6	4.5	4.8
		7	2.5	2.8
		8	1.0	-.6
3	5	-6	1.8	-2.0
		-5	1.7	-2.1
		-4	2.0	-1.7
		-3	2.4	-1.2
		-2	8.4	9.1
		-1	8.3	8.7
3	5	0	2.8	-3.0
		1	.9	-1.0
		2	8.2	-9.0
		3	8.8	-9.7
		4	6.5	7.3
		5	3.9	3.7
		6	.8	.7
		7	2.2	3.1
		8	1.8	-3.0
3	6	-5	2.3	-2.6
		-4	3.8	3.4
		-3	2.6	2.8
		-1	1.0	1.1
		0	1.1	.2
		1	2.1	-1.9
		2	1.0	-.8
		3	1.0	1.0
		4	.9	-1.2
3	7	-3	.7	-.2
		-2	2.0	2.0
		-1	1.9	1.8
		0	1.6	1.7
		1	.8	.7
		2	1.9	-1.7
		3	1.7	-1.7
		4	1.6	1.8
4	0	-6	1.8	.1
		-4	16.5	-17.2
		-2	11.4	11.0
		4	5.4	3.9
		6	.8	-.9
		8	2.5	-2.1
4	1	-6	2.4	-2.1
		-5	2.0	-2.1
		-4	2.4	2.1
		-2	1.5	1.1
		-1	.7	-.7
		3	3.5	3.3
		4	4.6	4.6
		5	2.9	-3.0
		6	1.1	-1.0
		7	2.2	-2.1
		8	1.9	-2.4
4	2	-6	1.1	1.6
		-5	.9	.5
		-4	8.1	-8.1
		-3	1.9	.3
		-2	6.0	5.4
		-1	4.9	-5.4
		0	1.1	1.4
		2	4.8	-4.3
		3	5.7	6.3
		4	5.8	4.9
		5	1.4	-1.1
		6	2.5	-2.4
		7	1.8	-2.2
		8	1.5	-1.4
4	3	-5	1.9	2.5
		-4	3.5	3.5
		-3	.9	.8
		-1	3.4	-4.2
		0	2.8	-5.9
		2	1.9	1.9
		3	4.3	4.3
		4	4.7	5.1
		5	1.5	-1.3
		6	1.1	-1.0
		7	1.5	-1.6
4	4	-4	1.5	1.9
		-3	.9	-.8
		-1	3.6	-4.3
		0	3.9	-4.3
		1	1.5	1.6
		2	.9	.3
		3	3.6	3.9
		4	3.8	4.0
		5	2.5	-2.8
		6	2.3	-2.1
4	5	-2	2.3	-2.0
		-1	1.1	.4
		1	2.3	-2.7
		2	.8	.6
		4	.7	-.6

system and have cell dimensions: $a=4\cdot30\pm0\cdot01$, $b=7\cdot55\pm0\cdot02$, $c=9\cdot98\pm0\cdot02$ Å and $\beta=85\cdot2\pm0\cdot1°$ as measured on precession photographs of the *h*0*l* and *hk*0 zones, taken with Mo $K\alpha$-radiation ($\lambda=0\cdot7107$ Å). Assuming the presence of four molecules in the unit cell the calculated density is 1·60 g.cm^{-3}. The space group $P2_1/c$ was unambiguously deduced from the systematic extinctions. Retigraph photographs were taken (layers with $h=0$ to 4) with Cu $K\alpha$-radiation at 20 °C. The intensities of 500 independent reflexions were measured by means of a photodensitometer. Corrections for Lorentz–polarization factors were applied; absorption was neglected ($\mu=18\cdot6$ cm^{-1}).

Structure determination

The data from the various layers were roughly put on a common arbitrary scale by using intensities measured on precession photographs. They were brought to an absolute scale with Wilson's method (1942) and were converted into E values. Triple products $E_{\mathbf{h}_1}\,E_{\mathbf{h}_2}\,E_{\mathbf{h}_1+\mathbf{h}_2}$, with E values larger than unity, were established. Values of these origin invariants were calculated by applying the $B_{3,0}$ formula (Karle & Hauptman, 1958). The expression used was: $E_{\mathbf{h}_1}E_{\mathbf{h}_2}E_{\mathbf{h}_1+\mathbf{h}_2}=AB+C$, in which A is a positive constant, C is a small correction term and B stands for

$$\langle(E^2_{\mathbf{k}}-1)\,(E^2_{\mathbf{k}+\mathbf{h}_1}-1)\,(E^2_{\mathbf{k}+\mathbf{h}_1+\mathbf{h}_2}-1)\rangle_{\mathbf{k}}\,.$$

k running over all reciprocal space. In the sign evaluation by a symbolic procedure carried out by hand, triple products with the highest calculated positive values were used. One single solution was obtained, and the corresponding Fourier synthesis with 133 terms gave starting parameters for the subsequent least-squares refinement. This was carried out in a block-diagonal approximation in which the layer scaling factors, positional parameters and individual anisotropic temperature factors were allowed to vary ($R=0\cdot106$). As there is a 1:1 correlation between the layer scaling factors and the β_{11} components of the vibrational expressions (Lingafelter & Donohue, 1966), these components were given the values obtained by the isotropic refinement and were kept constant throughout the refinement. A difference electron density synthesis indicated in the neighbourhood of the expected positions of the hydrogen atoms. Their positional parameters and B values were not refined. (The B values were kept fixed at 3·0 Å^2.)

Final parameters are shown in Table 1. The observed and calculated structure factors are listed in Table 2. Atomic scattering factors were in an analytical form, taken from Doyle & Turner (1968) for F, O and C and from Moore (1963) for H. The final R value is 0·092.

Discussion of the structure

The bond lengths and bond angles of the fluoroacetic molecule are shown in Table 3. The carbon–carbon bond is extremely short compared to the average value for an sp^3–sp^2 carbon–carbon bond, whereas the other bond lengths and bond angles do not deviate from normal values. Short carbon–carbon bond lengths are, however, found in some other acetic acid derivatives, as Table 4 reveals.

Table 3. *Bond lengths and bond angles in the monofluoroacetic acid molecule*

The e.s.d.'s are given in parentheses and refer to the least significant digits.

C(4)–F(1)	1·37 (1) Å	O(2)–H(6)	1·00 (7) Å
C(5)–O(2)	1·31 (1)	C(4)–H(7)	1·11 (7)
C(5)–O(3)	1·22 (1)	C(4)–H(8)	1·03 (7)
C(4)–C(5)	1·46 (1)		
F(1)–C(4)–C(5)	111·4 (5)°	F(1)–C(4)–H(7)	109 (4)°
C(4)–C(5)–O(3)	123·8 (5)	F(1)–C(4)–H(8)	106 (4)
C(4)–C(5)–O(2)	112·3 (5)	C(5)–C(4)–H(7)	108 (4)
O(2)–C(5)–O(3)	123·9 (5)	C(5)–C(4)–H(8)	103 (4)
C(5)–O(2)–H(6)	113 (4)	H(8)–C(4)–H(7)	119 (6)

As in glycollic acid, the molecule is planar as far as the non-hydrogen atoms are concerned (the angle between the plane through C(5)O(2)O(3) and C(5)C(4)-F(1) being 0·3°), with the C=O bond in the *syn* position with respect to the carbonyl oxygen atom. The molecule has the same conformation in the solid state as the most predominant one found in the gaseous state by microwave techniques (van Eijck, 1971).

The molecules are hydrogen-bonded across centres of symmetry (Fig. 1) thus forming planar dimers (O...O = 2·64 Å). This type of hydrogen bonding is not observed in formic acid (Holtzberg, Post & Fankuchen, 1953) acetic acid (Nahringbauer, 1970) or glycollic acid (Pijper, 1971) where the molecules are linked in a polymeric chain. The structure of monofluoroacetic acid is very similar to that of propionic acid (Strieter & Templeton, 1962) and higher homo-

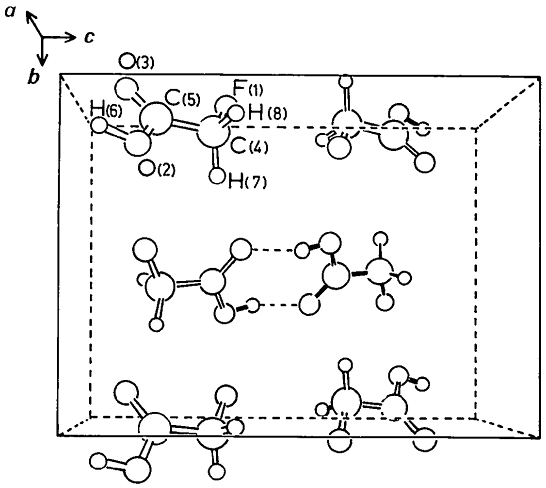

Fig. 1. Perspective view along the *a* axis of the structure of monofluoroacetic acid. Hydrogen bonds are shown as broken lines.

Table 4. C–C *bond lengths in acetic acid and derivatives*

Compound	C–C	e.s.d.	References
Acetic acid	1·478 Å	0·006 Å	Nahringbauer (1970).
$CH_3COONH_4.CH_3COOH$	1·488	0·005	Nahringbauer (1969).
$C_6H_5N_2Cl.CH_3COOH$	1·470	0·016	Rømming & Tjørnhom (1968).
Propionic acid	1·502	0·01	Strieter *et al.* (1962).
Glycollic acid	1·508	0·003	Pijper (1971).
	1·498	0·003	
Fluoroacetic acid	1·46	0·01	Present work.

logues. One short intermolecular contact is worth mentioning, namely the C(4)–H(7)...O(3) $(-x, \frac{1}{2}+y, \frac{1}{2}-z)$ contact, with a C···O distance of 3·36 Å and H···O of 2·29 Å.

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The Crystal and Molecular Structure of Dicarbonylnitrosyltriphenylphosphinecobalt(0), $C_{20}H_{15}CoNO_3P$

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Dicarbonylnitrosyltriphenylphosphinecobalt(0), $C_{20}H_{15}CoNO_3P$, crystallizes in the triclinic system with $a = 11·055$ (2), $b = 11·024$ (4), $c = 10·260$ (2) Å, $\alpha = 121·07$ (2), $\beta = 101·01$ (2), $\gamma = 105·14$ (2)°, $D_m = 1·39$ (5), $D_x = 1·42$ g.cm^{-3}, $Z = 2$; space group is $P\bar{1}$. The structure was determined from 2006 'observed' intensities collected on a G.E. XRD-5 diffractometer using Mo $K\alpha$ radiation. Nitrogen and carbon atoms in the nitrosyl and carbonyl groups are disordered. Least-squares refinement of the parameters led to a final R of 3·2% when composite 'NC atoms' ($\frac{2}{3}$ C, $\frac{1}{3}$ N) were refined and also when disordered nitrogen and carbon atoms were independently refined at each location. The coordination around cobalt is slightly distorted from tetrahedral; angles between the carbonyl and nitrosyl groups average approximately 113·4° and angles between the phosphorus and the nitrosyl or carbonyl groups average approximately 105·0°. The structure consists of discrete molecular units with no indication of intermolecular hydrogen bonding.

Introduction

Tricarbonylnitrosylcobalt(0) and its triphenylphosphine derivatives have been of interest for some time. (Cable & Sheline, 1956; Beck & Lottes, 1965; Beck & Melnikoff, 1965). These cobalt species are isoelectronic with nickel tetracarbonyl and are postulated to have a similar molecular structure. The tetrahedral configuration of nickel tetracarbonyl was established by the electron-diffraction study of Brockway & Cross (1935) and by the X-ray diffraction study of Ladell, Post & Fankuchen (1952); the tetrahedral configuration of the '*pseudo*-nickel carbonyls'* $Co(CO)_3NO$ and $Fe(CO)_2(NO)_2$ was established by the electron-diffraction study of

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* Name applied to the series of compounds $Ni(CO)_4$, $Co(CO)_3NO$, $Fe(CO)_2(NO)_2$, $Mn(CO)(NO)_3$, and $Cr(NO)_4$ by Huber & Anderson (1931).