require 18 *half-filled* pentagonal tunnels per unit cell and this very closely resembles the twinned structure depicted in Fig.6. Provided the molar ratio is 1:1, the difference between these two structures is that $\frac{1}{6}$ of the 18 pentagonal tunnels in the twinned $Nb_8W_9O_{47}$ structure are *completely* filled. This is not unexpected since the difference between metal: oxygen ratios in the two compounds $Nb_8W_9O_{47}$ and $Nb_6W_8O_{39}$ is less than 1%.

The structure of a crystal of the compound $Nb_8W_9O_{47}$ can thus be described in terms of the ninefold tetragonal tungsten-bronze subcell unit outlined above together with an intergrowth phase of probable composition $Nb_6W_8O_{39}$. Alternatively, a similar desscription involves a threefold tetragonal tungsten-bronze subcell unit which is twinned about the [130] axis. It is probable that both twinning and intergrowth of phases occur in the one crystal.

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The Crystal and Molecular Structure of Cyclopropanecarboxamide*

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Cyclopropanecarboxamide, C_4H_7NO, crystallizes in space group $P2_1/c$, with $a_0 = 6.919$, $b_0 = 8.271$, $c_0 = 16.313$ Å, $\beta = 90.22°$, and two molecules in the asymmetric unit. The structure was solved by direct methods and was refined by full-matrix least-squares, with visually estimated three-dimensional photographic intensity data. The two independent molecules have essentially the same dimensions and conformation, which are comparable with those for related compounds. Hydrogen bonds link the molecules three-dimensionally, each molecule being involved in four such bonds. The two molecules show nearly identical librational patterns, the predominant motion for each being a 9.6° libration about an axis close to the molecular axis of minimum inertia.

Introduction

This investigation was begun in 1954 but, for various reasons, was not completed until recently. The original purpose was to provide precise structural data on a three-membered ring compound. Although no such

structural studies of crystalline compounds had then been reported, several precise determinations have since appeared, together with several careful electron diffraction and microwave analyses. The most precise of the crystallographic studies (Fritchie, 1966; Hartman & Hirshfeld, 1966) have even provided evidence to support the bent-bond model for the three-membered ring (Coulson & Moffitt, 1949; Coulson & Goodwin, 1962). The present study, based on visually estimated photographic data is not accurate enough for that, but does provide reasonably precise molecular dimensions, further data on the hydrogen-bonding schemes of amides, and information of interest in

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connection with studies of molecular motion in crystals, already reported in part (Schomaker & Trueblood, 1968).

Experimental

A sample of cyclopropanecarboxamide, C_4H_7NO, originally prepared by Schlatter (1941), was kindly supplied to us by E. R. Buchman. Preliminary photographs showed the crystals to be monoclinic; the systematic extinctions, $0k0$ absent with k odd and $h0l$ absent with l odd, uniquely determine the space group as $P2_1/c$. The cell dimensions were obtained from zero-level Weissenberg photographs taken with Cu K radiation and calibrated with superimposed sodium chloride ($a_0 = 5\cdot639$ Å) powder photographs. The wavelengths used were $1\cdot5418$ for Cu $K\alpha$, $1\cdot5405$ for Cu $K\alpha_1$, and $1\cdot3922$ Å for Cu $K\beta$. The cell parameters are $a_0 = 6\cdot919 \pm 0\cdot007$, $b_0 = 8\cdot271 \pm 0\cdot008$, $c_0 = 16\cdot313 \pm 0\cdot016$ Å and $\beta = 90\cdot22 \pm 0\cdot02°$. The observed density, measured by flotation in a mixture of carbon tetrachloride and benzene, is $1\cdot203$ g.cm^{-3}; that calculated for eight molecules of C_4H_7NO per unit cell is $1\cdot211$ g.cm^{-3}. Thus the asymmetric unit consists of two independent molecules.

Intensities were estimated visually from multiple-film equi-inclination Weissenberg photographs taken with Cu $K\alpha$ radiation around **a** ($h = 0$ thru 4) and **b** ($k = 0$ thru 6). The crystals used for intensity measurements were shaped into approximate cylinders of about $0\cdot5$ mm diameter by dissolving irregularities with pyridine. The linear absorption coefficient for Cu $K\alpha$ radiation is about $0\cdot83$ mm^{-1} and the corresponding maximum error in relative values of the structure factor amplitudes because of absorption is only a few per cent. No corrections for absorption were made. The intensities of those reflections too weak to be estimated were evaluated at $I_{min}/3$ (Hamilton, 1955). After Lorentz, polarization, and Tunell corrections had been applied, inter-film and inter-layer scaling factors were determined by the method of Hamilton, Rollett & Sparks (1965). The scaled data set consisted of 2006 unique values of F_o^2, including 260 which were too weak to be estimated. This constitutes about 94 per cent of the independent diffraction maxima in the Cu $K\alpha$ sphere.

All calculations were made on an IBM 7094 computer with programs written in these laboratories. The atomic scattering factors used at first were those of Hoerni & Ibers (1954) for neutral nitrogen and oxygen, that of McWeeny (1954) for graphite carbon, and the exact quantum mechanical results quoted in *International Tables for X-ray Crystallography* (1962) for hydrogen. However, after the analysis was thought to be completed, it was discovered that (because β is so near $90°$) an indexing error had been made, with all hkl and $hk\bar{l}$ reflections interchanged. At this point, as discussed below, two further least-squares cycles were done, with proper indexing and with the carbon f curve changed to that for the valence state (Hoerni & Ibers, 1954)

and the hydrogen f curve changed to that for bonded hydrogen (Stewart, Davidson, & Simpson, 1965).

Determination and refinement of the structure

A scale factor and average temperature factor were obtained from a Wilson (1942) plot. After many unsuccessful efforts to solve the structure from sharpened Patterson distributions, a direct-methods computer program based on iterative application of Sayre's (1952) equation was written (Long, 1965). The first trial with this program [ACA (new) No. 2] yielded the correct structure immediately. The program uses a starting set of seven signs, with three arbitrarily assigned to fix the origin, and all combinations of the other four considered, giving sixteen solutions. The normalized structure-factor amplitudes, $|E|$, corresponding to the starting set of reflections included the five largest and two others nearly as large. In all, 287 $|E|$ values with $|E| \geq 1\cdot50$ were employed; in the most consistent solution, all signs were correct, as judged by comparison with the signs at the end of the refinement. The twelve highest peaks in a Fourier synthesis based on these E values were in reasonable mutual positions for identification with the twelve heavy atoms in the two molecules of cyclopropanecarboxamide in the asymmetric unit. The average deviation in positions from those obtained at the end of the refinement was only $0\cdot08$ Å, with a maximum deviation of $0\cdot30$ Å; furthermore four of the six highest peaks proved to be the oxygen and nitrogen atoms. Thus this initial E map was a very good representation of the structure.

The two distinct molecules in the asymmetric unit will be designated A and B; the numbering scheme for identification of the atoms in each is indicated in Figs. 2 and 3.

The structure was refined first by one cycle of Fourier synthesis, phasing with all twelve atoms found in the E map, and then with successive cycles of full-matrix least-squares. The program used was a modified version of ACA (old) No. 317 (Gantzel, Sparks, & True-
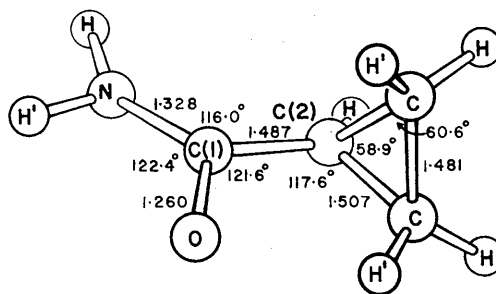
Fig. 1. Average bond distances and angles involving the heavy atoms, after corrections for molecular libration. See also Table 4. (The plane of the amide group is nearly normal to the ring plane, as discussed in the text; it has been represented here as twisted somewhat to facilitate labelling of the drawing.)

Table 1. *Observed and calculated structure factors*

The running index is *l*. F_o values marked with U were below the minimum observable intensity; the listed values are 0·58 $F_{\min}$. See text for significance of E and *.

H= 0, K= 0		
2	87	-74
4	460E	-773
6	4U	-4
8	422E	-446
10	93	93
12	112	106
14	71	-72
16	7U	3
18	55	52
20	60	65
H= 0, K= 1		
1	235	269
2	73	71
3	192	229
4	544E	-490
5	215	-217
6	14	13
7	68	-65
8	68	-65
9	151	-141
10	28	27
11	58	62
12	174	175
13	69	65
14	16	-5
15	90	-87
16	37	33
17	40	35
18	6U	-13
19	51	55
20	16	-20
H= 0, K= 2		
0	287	-354
1	366	-431
2	82	-81
3	113	104
4	127	-129
5	86	-90
6	68	58
7	306	-312
8	160	174
9	99	93
10	107	-101
11	117	121
12	28	27
13	30	27
14	34	26
15	8	0
16	6U	-8
17	14	-14
18	12	1
19	5U	4
20	25	-28
H= 0, K= 3		
1	131	-153
2	336	-376
3	73	-76
4	162	173
5	281	298
6	133	129
7	10	0
8	47	-45
9	117	-115
10	131	133
11	14	5
12	36	-30
13	29	22
14	128	-128
15	20	14
16	37	35
17	10	6
18	37	37
19	4U	-3
20	20	-27
H= 0, K= 4		
0	33	31
1	288	-336
2	52	49
3	168	-173
4	78	70
5	99	95
6	155	156
7	5U	-4
8	89	-91
9	78	77
10	231	-231
11	25	22
12	18	12
13	66	-71
14	69	69
15	15	14
16	16	15
17	23	27
18	15	13
19	9	-7
H= 0, K= 5		
1	5U	8
2	87	-86
3	44	-48
4	114	-119
5	94	89
6	115	-123
7	100	-105
8	34	-36
9	8	0
10	40	39
11	48	45
12	52	53
13	69	-67
14	50	45
15	19	18
16	29	29
17	6	-6
18	10	-8
H= 0, K= 6		
0	192	-209
1	11	-14
2	11	8
3	107	-104
4	45	-43
5	26	27
6	80	-79
7	15	-19
8	173	173
9	113	-124
10	74	80
11	93	94
12	47	-49
13	29	26
14	10	-9
15	10	10
16	23	-26
17	27	33
H= 0, K= 7		
1	121	-117
2	20	20
3	61	58
4	208	215
5	42	-38
6	72	-68
7	65	61
8	98	-100
9	70	66
10	50	44
11	11	2
12	5U	-4
13	9	8
14	15	1
15	23	-27
H= 0, K= 8		
0	120	117
1	34	26
2	29	-21
3	94	83
4	38	-37
5	38	36
6	11	7
7	29	-28
8	25	-21
9	17	-16
10	48	44
11	5U	-4
12	20	-15
13	14	15
H= 0, K= 9		
1	6U	-6
2	27	20
3	60	50
4	69	-69
5	26	-23
6	13	13
7	46	-39
8	39	34
9	25	23
10	7	-8
11	9	-0
H= 0, K= 10		
0	51	-45
1	16	-14
2	10	8
3	28	-20
4	15	13
5	10	-0
6	31	-25
H= 1, K= 0		
18	54	-52
16	29	32
14	43	34
12	7U	13
10	129	116
8	43	44
6	18	24
4	159	-141
2	242	-195
0	86	-76
-2	320	-319
-4	13	-17
-6	35	-30
-8	251	252
-10	82	80
-12	168	-153
-14	42	38
-16	23	12
-18	39	-43
H= 1, K= 1		
19	14	-18
18	66	-62
17	49	44
16	29	-34
15	7U	7
14	69	68
13	43	-31
12	7U	3
11	84	76
10	116	-122
9	190	186
8	18	-25
7	161	-155
6	163	-177
5	352	-426
4	273	312
3	245	236
2	384E	920
1	207	-197
0	285	-339
-1	130	-131
-2	380E	-559
-3	440E	728
-4	52	-52
-5	134	-128
-6	88	-83
-7	68	-67
-8	110	92
-9	103	80
-10	19	-3
-11	121	-100
-12	88	-73
-13	6U	-0
-14	16	14
-15	11	11
-16	88	81
-17	15	-12
-18	40	34
-19	11	11
-20	45	-47
H= 1, K= 2		
20	14	-13
19	8	-13
18	22	-23
17	81	-87
16	96	96
15	43	43
14	36	33
13	96	103
12	93	-106
11	136	-132
10	102	103
9	65	63
8	128	-102
7	8	-19
6	206	-211
5	55	53
4	170	161
3	305*	432
2	286*	-517
1	306*	-573
0	166	199
-1	283*	-382
-2	420E	805
-3	18	8
-4	134	139
-5	180	165
-6	14	-5
-7	219	213
-8	27	-14
-9	94	-88
-10	166	-148
-11	46	-49
-12	151	-145
-13	54	-51
-14	49	42
-15	14	16
-16	81	80
-17	63	62
-18	34	-31
-19	36	-38
-20	25	24
H= 1, K= 3		
19	20	-19
18	34	-35
17	10	-9
16	6U	-9
15	81	75
14	11	-7
13	27	-27
12	111	115
11	6U	-8
10	48	53
9	5U	8
8	34	-38
7	211	-218
6	139	143
5	191	179
4	268	-303
3	134	127
2	89	-101
1	234	-291
0	74	-77
-1	26	-25
-2	351*	-569
-3	136	142
-4	297	336
-5	240	253
-6	231	234
-7	70	-62
-8	97	107
-9	134	-144
-10	89	91
-11	6U	-7
-12	141	-147
-13	28	-22
-14	50	-49
-15	22	28
-16	15	-14
-17	12	-13
-18	26	-27
-19	7	-3
H= 1, K= 4		
19	3U	3
18	4U	-1
17	37	-42
16	53	-57
15	20	17
14	6U	5
13	26	-27
12	92	84
11	6U	-8
10	118	-115
9	149	151
8	158	150
7	107	107
6	111	119
5	126	-133
4	214	-256
3	150	-158
2	174	179
1	95	-102
0	88	-82
-1	132	-148
-2	115	-120
-3	78	82
-4	98	-111
-5	161	179
-6	185	-189
-7	92	-94
-8	150	156
-9	96	99
-10	88	88
-11	90	83
-12	63	59
-13	91	-85
-14	6U	-9
-15	35	-32
-16	75	-77
-17	5U	-10
-18	9	5
-19	22	-23
H= 1, K= 5		
18	3U	-8
17	14	-17
16	28	-26
15	25	-33
14	38	39
13	8	-10
12	55	-132
11	92	100
10	29	-32
9	26	-26
8	280	291
7	79	-79
6	99	-94
5	53	49
4	46	-42
3	69	73
2	110	120
1	102	113
0	153	-167
-1	86	-91
-2	100	101
-3	178	-149
-4	35	34
-5	25	-25
-6	148	-158
-7	19	-23
-8	80	-74
-9	94	107
-10	84	88
-11	86	87
-12	86	87
-13	21	-16
-14	30	-30
-15	32	-30
-16	14	12
-17	11	-13
-18	14	-14
H= 1, K= 6		
16	24	-24
15	24	-26
14	5U	-10
13	6U	6
12	6U	-9
11	6U	7
10	24	20
9	10	14
8	43	-45
7	64	66
6	72	-70
5	64	-63
4	64	63
3	4U	6
2	48	41
1	96	101
0	111	118
-1	58	-62
-2	28	27
-3	20	15
-4	152	-161
-5	55	-50
-6	10	9
-7	84	-87
-8	47	45
-9	80	78
-10	6U	2
-11	36	35
-12	22	-20
-13	10	-12
-14	27	-24
-15	8	-9
-16	19	19
H= 1, K= 7		
10	36	31
9	6U	1
8	6U	7
7	50	44
6	6U	-13
5	25	-25
4	44	-39
3	14	-23
2	52	-50
1	73	73
0	49	44
-1	29	33
-2	6U	-7
-3	42	-43
-4	6U	-5
-5	6U	1
-6	70	72
-7	6U	-14
-8	13	-12
-9	48	-50
-10	6U	1
-11	32	34
-12	9	9
-13	5U	4
-14	18	-18
-15	25	-22
H= 1, K= 8		
9	13	-13
8	41	37
7	6U	-7
6	135	128
5	11	-15
4	15	-16
3	22	-22
2	34	-32
1	6U	-0
0	6U	-7
-1	27	-20
-2	119	-114
-3	46	44
-4	26	25
-5	47	44
-6	69	70
-7	38	-38
-8	44	-48
-9	11	13
-10	5U	-1
-11	8	12
-12	33	28
-13	13	-14
H= 1, K= 9		
8	4U	-3
7	86	77
6	47	-44
5	18	17
4	18	13
3	13	-8
2	75	72
1	36	-29
0	10	14
-1	57	-55
-2	13	-23
-3	27	27
-4	19	-16
-5	5U	11
-6	30	-28
-7	16	-15
-8	4U	-3
-9	18	-15
-10	3U	1
-11	2U	10
H= 1, K= 10		
6	23	26
5	3U	25
4	3U	-2
3	33	31
2	37	-33
1	27	-29
0	27	25
-1	33	29
-2	22	25
-3	4U	7
-4	3U	-2
-5	37	-37
H= 2, K= 0		
20	46	50
18	56	-57
16	126	-126
14	115	106
12	51	51
10	21	-5
8	195	177
6	203	-168
4	274	-224
2	4U	-23
0	88	76
-2	103	-77
-4	171	-161
-6	316	314
-8	94	83
-10	51	50
-12	82	-81
-14	206	-204
-16	45	46
-18	73	77
-20	14	-16
H= 2, K= 1		
20	4U	-3
19	5U	5
18	21	-21
17	61	-63
16	63	57
15	59	-58
14	7U	-1
13	115	116
12	41	-39
11	50	51
10	79	68
9	6U	-10
8	190	-206
7	34	38
6	103	109
5	212	-215
4	364	457
3	187	185
2	270	-291
1	219	215
0	125	-125
-1	433E	-606
-2	171	-167
-3	318	-348
-4	20	-29
-5	352	372
-6	223	211
-7	239	225
-8	35	37
-9	18	-26
-10	42	40
-11	16	13
-12	32	-29
-13	74	-69
-14	6U	-12
-15	83	-82
-16	5U	-6
-17	52	52
-18	49	-47
-19	15	14
-20	3U	-1
H= 2, K= 2		
20	3U	-24
19	26	26
18	5U	-6
17	32	-30
16	19	-14
15	60	-61
14	34	-24
13	21	16
12	105	100
11	59	45
10	18	-15
9	47	-50
8	35	-37
7	87	-74
6	310	374
5	114	116
4	269	-302
3	80	66
2	286	-347
1	77	85
0	171	187
-1	9	-3
-2	179	-169
-3	331	-388
-4	74	61
-5	99	-98
-6	26	-5
-7	155	153
-8	4U	-13
-9	15	-10
-10	83	76
-11	83	76
-12	5U	3
-13	26	16
-14	37	36
-15	49	-41
-16	11	-8
-17	19	-15
-18	39	-40
-19	3U	-6
-20	3U	-7
H= 2, K= 3		
19	8	7
18	35	36
17	22	24
16	81	-85
15	19	21
14	100	-111
13	56	-56
12	96	99
11	52	-59
10	89	-78
9	102	92
8	19	4
7	192	202
6	248	245
5	31	-34
4	154	-178
3	208	-212
2	66	62
1	115	-105
0	107	115
-1	3U	6
-2	102	-108
-3	123	145
-4	196	-211
-5	154	165
-6	83	-85
-7	194	-207
-8	9	16
-9	99	-105
-10	56	-60
-11	122	125
-12	118	115
-13	75	78
-14	76	72
-15	13	-9
-16	87	-85
-17	41	-44
-18	31	31
-19	13	-11
H= 2, K= 4		
18	12	17
17	5U	15
16	10	-11
15	80	-90
14	56	60
13	58	-54
12	62	-66
11	51	49
10	119	-119
9	25	24
8	141	144
7	49	45
6	19	-12
5	116	110
4	23	-21
3	79	-75
2	29	25
1	4U	-1
0	78	-85
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-2	158	172
-3	110	-113
-4	75	93
-5	154	-167
-6	50	-45
-7	7	-9
-8	65	-64
-9	11	2
-10	46	-44
-11	58	59
-12	49	51
-13	66	67
-14	31	-26
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-16	10	8
-17	13	-14
-18	14	16
H= 2, K= 5		
17	16	19
16	23	25
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14	48	-52
13	50	-47
12	42	-38
11	6U	-11
10	71	75
8	24	-8
9	36	31
7	68	-63
6	30	27
5	5U	3
4	36	30
3	32	26
2	92	-94
1	24	22
0	103	115
-1	91	96
-2	19	21
-3	34	38
-4	185	-199
-5	45	-44
-6	38	38
-7	41	-43
-8	75	81
-9	35	-33
-10	48	43
-11	20	23
-12	11	8
-13	20	18
-14	33	-31
-15	5U	-2
-16	9	-11
-17	20	-16
H= 2, K= 6		
16	9	13
15	18	18
14	24	-32
13	17	-14
12	29	30
11	15	-18
10	14	11
9	84	-80
8	67	-62
7	38	32
6	11	12
5	175	171
4	37	32
3	61	-54
2	21	-21
1	93	-104
0	72	70
-1	12	-12
-2	55	53
-3	40	-39
-4	111	-108
-5	78	76
-6	76	-72
-7	50	52
-8	60	63
-9	40	-44
-10	5U	5
-11	14	-16
-12	18	-13
-13	24	24
-14	48	49
-15	4U	5
-16	3U	-3
H= 2, K= 7		
10	92	-79
9	28	-20
8	9	3
7	85	-76
6	71	71
5	16	19
4	28	17
3	82	78
2	5U	1
1	59	58
0	5U	3
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-2	55	-57
-3	38	-37
-4	23	-25
-5	5U	-2
-6	81	82
-7	5U	-5
-8	9	-11
-9	18	17
-10	78	-85
-11	19	-26
-12	15	12
-13	9	8
-14	34	37
-15	2U	34
H= 2, K= 8		
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8	4U	-1
7	20	14
6	47	-41
5	5U	7
4	42	41
3	30	32
2	11	11
1	5U	4
0	5U	5
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-2	58	55
-3	53	51
-4	13	-14
-5	9	-3
-6	35	-35
-7	23	-22
-8	8	-1
-9	17	-12
-10	12	-12
-11	42	-35
-12	20	-19
H= 2, K= 9		
7	39	-36
6	25	-20
5	9	7
4	10	9
3	73	66
2	34	-29
1	37	-32
0	10	0
-1	11	14
-2	34	30
-3	32	26
-4	25	22
-5	35	-34
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-7	32	29
-8	18	-15
-9	16	-15
H= 2, K= 10		
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3	14	-13
2	15	-11
1	18	-17
0	11	8
-1	41	37
-2	9	-7
-3	7	-6
-4	18	-20
H= 3, K= 0		
18	63	-61
16	7U	-0
14	84	-77
12	51	40
10	249	226
8	190	-166
6	30	-32
4	184	154
2	146	-125
0	132	-127
-2	187	-170
-4	87	-72
-6	123	-122
-8	178	162
-10	158	145
-12	109	-116
-14	71	22
-16	32	38
-18	55	-58
H= 3, K= 1		
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18	7	6
17	11	10
16	43	45
15	53	-48
14	72	-68
13	110	-108
12	47	-46
11	57	57
10	33	-30
9	129	120
8	14	18
7	41	-41
6	201	198
5	101	81
4	109	91
3	61	58
2	37	-36
1	207	-211
0	326	-359
-1	92	87
-2	68	60
-3	185	-168
-4	169	139
-5	108	-88
-6	188	-173
-7	118	111
-8	32	35
-9	29	-25
-10	60	-53
-11	17	-7
-12	72	-71
-13	94	86
-14	130	124
-15	20	-15
-16	12	9
-17	48	-44
-18	32	-31
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H= 3, K= 2		
19	3U	-3
18	56	60
17	21	21
16	28	-24
15	88	92
14	90	-94
13	52	-50
12	12	-17
11	131	-144
10	79	-74
9	76	-71
8	25	14
7	52	51
6	230	241
5	272	277
4	134	140
3	135	135
2	143	-162
1	182	-201
0	46	-46
-1	224	-229
-2	230	247
-3	167	177
-4	198	-205
-5	207	223
-6	138	-137
-7	164	-152
-8	124	123
-9	112	-101
-10	61	-57
-11	25	-27
-12	49	46
-13	38	39
-14	54	51
-15	94	94
-16	61	-63
-17	10	-6
-18	4U	1
-19	41	-46
H= 3, K= 3		
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17	36	37
16	31	34
15	6U	-0
14	86	89
13	54	-50
12	100	-109
11	25	-16
10	89	-91
9	5U	3
8	73	58
7	42	35
6	169	-171
5	4U	0
4	45	47
3	76	69
2	206	228
1	18	10
0	4U	7
-1	65	-77
-2	160	191
-3	39	37
-4	4U	-5
-5	35	-34
-6	114	-109
-7	13	8
-8	82	-78
-9	15	6
-10	5U	11
-11	37	-32
-12	58	56
-13	43	45
-14	11	15
-15	9	-1
-16	15	13
-17	23	-24
-18	9	-10
-19	1U	12
H= 3, K= 4		
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16	39	46
15	27	25
14	49	57
13	43	-41
12	35	-36
11	46	-44
10	41	38
9	84	78
8	23	-23
7	124	-118
6	13	9
5	87	-94
4	64	67
3	142	193
2	181	-219
1	38	34
0	19	-22
-1	78	83
-2	107	117
-3	53	54
-4	40	36
-5	136	-137
-6	5U	-2
-7	88	-92
-8	45	-48
-9	35	37
-10	47	48
-11	47	50
-12	16	-12
-13	22	-22
-14	37	-31
-15	16	11
-16	46	49
-17	4U	8
-18	2U	-5
H= 3, K= 5		
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15	41	44
14	50	-52
13	47	53
12	21	-25
11	32	31
10	50	52
9	61	-57
8	33	-33
7	74	-65
6	7	-5
5	7	17
4	26	27
3	35	30
2	85	86
1	81	90

Table 1 (cont.)

```
  0  5U -12      -14 105 10C        2  88  96       -1  12   8       12  74 -22      -12 3C -13       10   8  -9
 -1  87 -94      -16  75  75        1 161 167       -2  21  19       11  74  76      -14 26  26        9  24  -4
 -2  25 -22      -18  22 -27        0  41  40       -3   9   6       10  6U   8                        8   8   8
 -3  71 -73                        -1  41  49       -4  20 -22        9  28  31      H= 6, K= 1        7  25 -27
 -4  21 -24      H= 4, K= 1        -2 135 135       -5  3U  -1        8  79  88      15  27  31         6  5U   7
 -5 106 125      18  44  43        -3 137 -146      -6  7U   6        7  73 -71      14  5U  -1         5  48 -5C
 -6 113 141      17  19 -18        -4  95 -102                        6   8   2      13  1C  13         3  17  18
 -7  29  35      16  6U  11        -5  38  37       H= 5, K= 0        5  46 -44      12  21  27         2  36  37
 -8  58  6C      15  53  51        -6 146 -153      16  13 -12        4  91  93      11  42 -38         0  25  25
 -9  10  -5      14  74  73        -7  29  24       14  31 -23        3  19 -21      10  57 -5C        -1  6U  -6
-10  53  61      13  29  25        -8  54  51       12 133 139        2 150 -161      9  73  72        -2  39 -37
-11  38 -39      12 113 111        -9  33 -29       10  49  46        1  37 -38       8  5C  50        -3  52 -56
-12  83 -91      11 152 -159      -10  48  53        8 232 -216       0 157 -179      7  1A   9        -4  5U   1
-13  43 -44      10 162 -166      -11  19  20        6  66  65       -1  3U  -C       6 101  95        -5  5U   2
-14  37  36       9  7U  -9       -12  12 -10        4 1C4  99       -2 135 146       5  99 -95        -6  5U  -3
-15  37  40       8  64 -59       -13  23 -20        2 108 -83       -3  45  44       4 162 -158       -7  36  30
-16  28  27       7  24 -16       -14  23 -20        0  11  20       -4  47 -50       3  7U  -6        -8  15 -18
-17  16  15       6  53 -46       -15  19 -17       -2  18 -30       -5  4U  -4       2  7U   5        -9  4U   1
                  5  56 -45       -16   8  -5       -4  8C -71       -6  49  49       1  62 -57       -10  5   9
H= 3, K= 6        4  4C -33       -17  18  26       -6  86  82       -7  64  69       0  16 -14
15   8 -11        3 262 294                        -8 109 108       -8  66  73      -1  34   4       H= 7, K= 5
14  35  39        2  39  32       H= 4, K= 5       -10  28 -18       -9  39  41      -2  84 -78        8  17  13
13  20 -24        1 107 115       16  2U -15       -12  25 -17      -10  47 -52      -3 113 103        7  15  12
12  57  65        0  94 -97       15  4U -11       -14  3U   0      -11  49 -51      -4 137 138        6  1C  -4
11   7  12       -1 142 -148      14  5U   9       -16  36 -37      -12  18 -19      -5  7U  -8        5  16 -16
10  95 -94       -2 265 270       13  5U  -3                       -13  18 -19      -6  38  31        4  33  36
 9  41  37       -3 144 -139      12  54  55       H= 5, K= 1      -14  13 -14      -7  16  -9        3  25 -24
 8  38 -40       -4  72  77       11  37  40       16  15 -15      -15  13 -12      -8  7U  -7        2  27  28
 7  19 -17       -5  60  59       10   8  -5       15  10  10                       -9  7U  -5        1  25 -25
 6  1C  -5       -6  48  57        9  36 -33       14  47 -45      H= 6, K= 0       -10  24  22        0  45 -43
 5  65 -66       -7  33  32        8  49 -49       13  65  64      14  14  14       -11  16 -13       -1  11  12
 4  5U   6       -8  52  40        7  5U   8       12  18 -25      13  20 -23       -12  18 -16       -2   6   3
 3  90  85       -9  92 -81        6  41 -39       11  16 -14      12  5U   3       -13  5U  -4       -3  22   2
 2 110 103      -10 278 -200       5  39 -36       10  14 -13      11  55 -56       -14  20 -17       -4  4U  -2
 1  78  72      -11  48  -9        4 118 -124       9  54 -55      10  45  47       -15  12  11       -5  29 -24
 0  13  16      -12  27  12        3  47 -47        8  9C  87       9  51  55                         -6   7 -11
-1  48 -49      -13  16  16        2  54 -59        7  66 -66       8  67  72      H= 6, K= 2        -7  33 -39
-2   7 -11      -14  41  42        1 107 115        6 126 114       7  64  71      14  14  14        -8  3U   0
-3  14  12      -15  32  27        0 189 201        5  17  12       6  95 -92      13  20 -23
-4  33 -32      -16  32 -26       -1   9 -18        4  62 -59       5  38  39      12  5U   3       H= 7, K= 6
-5  62 -67      -17  36  36       -2  87 101        3  47  46       4  91 -98      11  55 -56        4  43  43
-6  33  37      -18  34  40       -3  5U -17        2 109 -104      3  61 -63      10  45  47        3   6  -9
-7  28 -31      -19  0U -78       -4  48 -56        1  17 -17       2  61 -63       9  51  55        2  15 -11
-8  42  46                        -5  21  22        0  55  57       1  11 -17       8  67  72        1  30 -31
-9  62  72      H= 4, K= 2        -6  39  39       -1 121 115       0  6U   4       7  64  71        0  20 -19
-10 64 -70      18  3U  -6        -7  16 -22       -2  67  65                       6  95 -92       -1  18  18
-11 16 -20      17  4U   1        -8  12 -15       -3  79 -64      H= 6, K= 1       5  38  39       -2  48  55
-12 24 -23      16  46 -45        -9  28  26       -4  57 -51      15  27  31       4  31 -29       -3  24  28
-13 17 -18      15  53  53       -10  53 -56       -5  49 -41      14  5U  -1       3  80 -85       -4  3U   4
-14 4U  -4      14  49  40       -11  35 -34       -6  89 -85      13  1C  13       2  89  87       -5  16 -20
-15 3U  -2      13  4U  39       -12  5U   5       -7 139 139      13  1C  13       1  91 -90
-16 1U   9      12  12 -17       -13   7  -7       -8  61 -61      11  55 -56       0 138 -138      H= 8, K= 0
                 11  12   9       -14  17 -11       -9  91 -85      10  45  47      -1  7U   7        8  57  59
H= 3, K= 7      10  7U   2       -15  3U   1       -10  77  27       9  51  55      -2  12   8        6  21 -21
10  16 -17       9  81 -79       -16  2U -1C       -11  48 -43       8  67  72      -3  12  1C        4  6U  11
 9  5U   9       8 116 117                        -12 104  90       7  64  71      -4  65  66        2  62  63
 8  5U  -2       7  62 -61       H= 4, K= 6       -13  46  44        6  95 -92      -5  68  68        0  46 -47
 7  6U  -6       6  11  -5       14  25  29       -14  28  24        5  38  39      -6  91 -81       -2  6U   5
 6  59 -51       5  79  75       13  28  32       -15  19 -17        4  31 -29      -7  36 -31       -4  18 -14
 5  73 -68       4  73 -67       12  39  48       -16  37 -28        3  80 -85      -8  44 -19       -6  41 -49
 4  29  17       3 162 -162      11  25 -29       -17   8   9        2  89  87      -9  19 -27       -8  19  17
 3  5U  -2       2 127 -120      10  33  37                         1  91 -90      -10 76  77
 2  17  22       1  73  73        9  25 -17       H= 5, K= 2        0 138 -138      -11 56  4C      H= 8, K= 1
 1  51  47       0  12  -7        8  14 -14       16  12  15       -1  7U   7       -12  6  -9        7  17  18
 0  21  23      -1 164 172        7  51  49       15  35  34                       -13 4U -11        6  70 -76
-1  5U  -4      -2  14 -10        6  86 -90       14  31 -12       H= 6, K= 3      -14 15 -14        5  12  25
-2  5U  -5      -3  74 -74        5  15  -7       13  57 -58       14  3U  -1                         4  5U   7
-3  48  52      -4  56  54        4 117 -11       12  25  25       13  4U  -3      H= 6, K= 3        3  1C  -4
-4  5U  -5      -5 146 154        3 10C -104      11  16  22       12   8  -3      14  3U  -1         2  12  13
-5  49  45      -6  59  47        2  5U  -4       10  35 -15       11  23  29      13  4U  -3         1  32 -24
-6  46  43      -7  36 -31        1  40 -36        9 104 114       10  23 -28      12   8  -3         0  32  26
-7  38 -41      -8 103 -103       0  30 -34        8 107 -111       9  17  15      11  23  29        -1  43  43
-8  27 -26      -9 117 -116      -1  35  12        7  88 -83        8  36  39      10  23 -28        -2  47  49
-9  51 -56      -10 67  67       -2  88  92        6  3U  -6        7  37 -13       9  17  15        -3  5U  -1
-10 4U   1      -11 52  47       -3  5U  -3        5  58  54        6  27  36       8  36  39        -4  19 -22
-11 4U   7      -12 54  48       -4  43  43        4  67 -64        5  16 -16       7  37 -13        -5  48 -52
-12 4U  -2      -13 28 -18       -5  96 104        3  81 -66        4  6U   4       6  27  36        -6  35 -36
-13 3U   1      -14 76 -76       -6  20  18        2  81  77        3  18  17       5  16 -16        -7   9 -10
-14 29 -33      -15 20 -14       -7  23  24        1 114 -127       2  18  -7       4  6U   4        -8  25 -22
                -16 20  21       -8  54 -67        0 168 178        1  6U -15       3  18  17
H= 3, K= 8      -17 27  28       -9  37 -39       -1 138 140        0  8U  -9       2  18  -7       H= 8, K= 2
 8  14  19      -18 3U   6       -10 35 -37       -2  21  25       -1  6U  -9       1  6U -15        8  28 -35
 7  48 -44                      -11  30  36       -3  25  21       -2  22 -23       0  8U  -9        7  10  18
 6  35 -34      H= 4, K= 3      -12   9   6       -4  50  48       -3  55 -56       6  3U  -6        6  4U  -7
 5   9 -44      17   7   6      -13  30  36       -5  18  14       -4  19 -21       4  26  28        5  15 -20
 4  67 -70      16  1C -16      -14  29 -31       -6  8C -78       -5  37 -32       3  28  19        4  21  26
 3  39  36      15  5U  -2                        -7  6U -4C       -6  11 -14       2  18  -1        3  27  29
 2  5U   7      14  38  38      H= 4, K= 7       -8 126 -124      -7  1C  25       1   5  -1        2  12 -11
 1  31 -36      13  49 -51       8  41  41        -9  84 -33       -8  5U  11       0  23 -25        1  36  34
 0  28  19      12  44  46       7  47 -47       -10 18  18       -9  55  62      -1  4U  -5        0  11  -4
-1  25 -24      11  6U  -9       6  75  72       -11 48  47       -10 14 -18      -2  4U  -6        -1  25  22
-2  5U   3      10  77 -76       5  5U   C       -12 4U  -C       -11 14 -12      -3  3U  -3        -2  5U  -3
-3  42  41       9  7U  69       4  28 -29       -13 3U  -6       -12 15 -18      -4  5U  -5        -3  17  15
-4  66  65       8  34 -28       3  5U   C       -14  4  -3       -13 3U   7      -6  17 -18        -4   4   7
-5  15  14       7  99  98       2  74 -71       -15 1C   C                                         -5  6U -10
-6  26 -26       6 105 107       1  75 -75       -16 14  16      H= 6, K= 4      H= 7, K= 0        -6  14 -49
-7  4U   6       4  25 -23       0  27  21                       13  18  21      -10 6U  -7        -7   9  15
-8  40 -43       3 113 -115     -1  5U  12       H= 5, K= 3      12  21  19       12  48  45
-9  4U  -1       2 147 148      -2  37 -13       16  5U  27      11  55 -13       10  41 -42      H= 8, K= 3
-10 40  40       1  5U  98      -3  36 -31       15  4U  14      10  5U   5        8  17  21        6   3  10
-11 13 -12       0  14  19      -4  32 -30       14  19 -16       9  5U   9        6  95 108        5  3U   7
                -1  21  -2      -5  13 -12       13  49 -57       8  35 -38        5  66 -66        4  26  28
H= 3, K= 9      -2 168 -177     -4  3C -4C       12  43  48       7  26  29        2  33 -34        3  28  19
 6   6   4      -3  34  34      -3  24 -44       11  42 -37       6  39 -37        1  19 -13        2  18  -1
 5  56 -40      -4  29  25      -4 148 151       10  57  61       5  6U   9       -2  66 -77        1   5  -1
 4  14 -12      -5   8  1C     -5  16 -11        9  6U  -4        4  4C  39       -3  74  74        0  23 -25
 3  58 -53      -6  2C  20      -6  5U  -5        8  61  60        3  6U  16       -4  56  56       -1  4U  -5
 2  37 -29      -7  77  83      -7  16 -34        7   6   1        2  2C  20       -6  4C  60       -2  4U  -6
 1  4U   6      -8  65 -69      -8  93 101        6 128 137        1  3U  36       -8  25 -34       -3  3U  -3
 0  4U   5      -9  19 -22      -9  24  18        5 116 -116       0  53  51      -12 28  27       -4  5U  -5
-1  38  36      -10 39  37     -10 4U  -5        4 107 115       -1  6U -64                        -5  2U  10
-2  26  23      -11 1C  -8     -11 78 -25        3 20A 210       -2  31   5      H= 7, K= 1
-3  16   9      -12 22  2C     -12 79 -77        2  42 -42       -3  62 -66      13  17  29
-4  19  -9                     -13 3U  -1        1  6U  -9       -4  62 -67      12  4U -17
-5  4U   9      H= 4, K= 4     -14  9   4        0  54 -77       -5  41  43      11  5U  -6
-6  16 -13      16  13  -9     -15  6  -8       -1  28 -44       -6  25  49      10  10   8
-7  26 -24      15  20 -18     -16 32 -16       -2  48  41       -7  27  30       9  35 -33
-8  26  25      14  24 -27                      -3  28  14       -8  6U   9       7  56  58
                13  42  46     H= 5, K= 0       -4  17 -17       -9  2C -22       5  22 -27
H= 4, K= 0      12  6U -16     14  13  -7       -5   9   4       -10  7 -15       5  46  46
18  11   4      11  49  51     12  11 -28       -6  19 -18       -11 5U  -8       4  18 -20
16  80  76      10  21 -27     10  36  35       -7  18  18       -12 8 -13       3  51 -46
14  13  -5       9  65 -71      8  74 -71                                        2  26  31
12  46 -47       8  18 -11      6  59  52       H= 5, K= 4      H= 6, K= 5       1  29 -29
10 239 -235      7  51 -50      4  31  21       13  3U  20      12  3U  20       0  17 -14
 8 179 -168      6 106 -111     2 153 -145      12  21  19      11  4U  -5      -1  22 -21
 6 241 211       5  24 -18      0  8U -16       11  6U  13      10  74 -73      -2  36  33
 4 197 171       4  52  48     -2  6C  61       10  5U   7       9  4C -42      -3  16 -16
 2 423E 363      3   6  -6     -4 205 190        9  5U  -38      8  4U  38      -4  18 -18
 0 229 -198                    -6  38  36        7  76  29       7  5U  -6      -5  43  38
-2 196 -165                    -8 117 -133       6  39 -37       6  12  16      -6  6U  -2
-4 247 179                    -10 39 -32         3  6U  16       5  53  55      -7  46  42
-6 283 -254                                      4  4C  4U                      -8  6U   5
-8  91 -76                                        3   8  17                     -9  13 -21
-10 54  41                                        2  18  18                    -10 5U  -1
-12 114 -106                                      1   2   C                    -11 5U   3
                                                 0  19 -16                    -12 27  21
                                                -1  37 -25                    -13 2U   2
                                                -2  32  4C
                                                -3  21  16                    H= 7, K= 2
                                                -4  45  44                    12   6 -31
                                                -5  24  2C                    11  22  21
                                                -6  3U   2                    10  36  32
                                                -7  14 -14                     9  18  19
                                                -8  14  10                     8  5U -11
                                                -9   9  -6                     7  10  25
                                               -10  13  -1C                    6  21 -22
                                                                               5  6U  -8
                                               H= 5, K= 5                      4  45  42
                                                5  13  -7                      3   8  17
                                                4  2U  -4                      2  43 -33
                                                3   6   7                      1  37 -31
                                                2   3U   7                     0  19 -16
                                                1   1  -9                     -1  37  34
                                                0 116 -116                    -2  55 -52
                                               -1  78 -26                     -3  74 -69
                                               -2  10A 210                    -4  14  17
                                               -3  6U  -66                    -5  5U   4
                                               -4  17 -17                     -6  5U   7
                                               -5   9   9                     -7  14  16
                                               -6  19 -18                     -8  4C  99
                                                                              -9  4U  11
                                               H= 6, K= 0                    -10  3U  -0
                                               14  13  -1
                                               12  11 -28                    H= 7, K= 3
                                               10  36  35                    11  3U  1C
                                                8  74 -71                    10  10  13
                                                6  59  52                     9  17  19
                                                4  31  21                     8  65 -73
                                                2 153 -145                    7  5U   3
                                                0  8U -16                     6  41  41
                                               -2  6C  61                     5  24 -26
                                               -4 205 190                     4  49  49
                                               -6  38  36                     3  28 -25
                                               -8 117 -133                    2  35 -36
                                              -10  39 -32                     1  32  33
                                                                             0  35  32
                                                                            -1  50  48
                                                                            -2  43 -45
                                                                            -3  73 -70
                                                                            -4  34 -14
                                                                            -5  5U   6
                                                                            -6  7U   4
                                                                            -7   8   9
                                                                            -8  45  47
                                                                            -9  15  18
                                                                           -10   5   8
                                                                           -11   5   8
```

blood, unpublished), which minimizes $\Sigma w(|F_o| - K|F_c|)^2$; the weights, w, were chosen as $1/\sigma^2(F_o)$ as estimated by the scaling program. At an intermediate state with the usual discrepancy index, R, equal to 0·15, the fourteen hydrogen atoms were added at their expected positions, assuming normal molecular geometry; with no other changes in parameters, R dropped to 0·12. At this juncture, the nine highest-intensity reflections, most of which seemed to show appreciable secondary extinction, were removed; they are marked with an E in Table 1. In addition, five others of the strongest low-order reflections for which there were large discrepancies were omitted (* in Table 1); the precision of estimating these had been low. (Nine of these fourteen reflections have $h = 1$.) The hydrogen atoms were assigned isotropic temperature factors (Table 2), which were not allowed to vary, and then the hydrogen positions and all the parameters of the heavy atoms were refined by full-matrix least-squares. After convergence had been achieved, the aforementioned indexing error was discovered; it was corrected, the f tables were modified as described above, and two further full-matrix cycles were done. The parameter shifts were very small, and, in the second cycle, the maximum shifts were less than one-third of the corresponding estimated standard deviations and the average shifts were less than 0·1σ. The final value of R for all reflections is 0·12; with the omission of the fourteen doubt-

ful reflections mentioned above, it is 0·094, and with the further omission of the 260 unobserved reflections it is 0·087. The final position and temperature-factor parameters are given in Table 2, together with their standard deviations as estimated from the inverse of the matrix of the normal equations.

A final difference synthesis was calculated, with the contributions of all atoms subtracted. However, the data are not sufficiently precise [$\sigma(\varrho)$ is of the order of 0·12 e.Å^{-3}] for details such as bond peaks (Fritchie, 1966; Hartman & Hirshfeld, 1966) to be discernible. The peaks in the map varied in height from about 0·36 to $-0·35$ e.Å^{-3}, each about three times the approximate e.s.d. of the electron density. These extreme peaks were situated about 1 Å from O(B) and about 0·5 Å from O(A) respectively. There were a few negative peaks in the range $-0·25$ to $-0·3$ e.Å^{-3} near some of the sites of the atoms. There were no consistent indications of bond peaks; a peak of height 0·27 e.Å^{-3} was located not far from the axis of the cyclopropane ring of molecule B, about 1 Å from the plane of the ring, but no comparable features were present on the other side of this ring or in similar positions on the ring of molecule A.

The molecular structure

The temperature-factor parameters of the two distinct molecules (Table 2) were used in separate analyses of

Table 2. *Positional and thermal parameters**

	x	y	z	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
C(1A)	6281,3	6138,3	4024,1	166,5	106,3	30,1	$-17,6$	$-12,3$	5,3
C(2A)	5094,4	7339,3	4473,2	253,7	153,4	39,1	88,8	$-30,4$	$-20,3$
C(3A)	2971,5	7354,5	4337,2	233,8	220,6	56,2	81,11	51,6	$-13,5$
C(4A)	4145,5	8642,4	3973,2	291,8	136,4	67,2	94,10	$-17,6$	1,4
O(A)	5762,2	5571,2	3356,1	193,4	148,3	30,1	$-25,5$	$-17,2$	$-10,2$
N(A)	7912,3	5716,3	4384,1	217,6	175,4	38,1	95,7	$-57,4$	$-32,3$
C(1B)	$-1522,4$	7445,3	1605,1	162,5	125,3	33,1	$-46,6$	$-21,3$	18,3
C(2B)	$-0330,4$	6136,3	1947,2	183,6	164,4	38,1	$-9,8$	6,4	33,3
C(3B)	1811,4	6300,5	1915,2	188,7	216,7	75,2	23,10	$-66,6$	72,6
C(4B)	0867,5	5164,5	1368,2	279,8	196,6	62,2	106,11	33,6	1,5
O(B)	$-0926,2$	8308,2	1031,1	182,4	216,4	40,1	$-42,6$	$-16,3$	84,3
N(B)	$-3245,3$	7659,3	1926,1	191,5	148,4	37,1	21,7	14,3	27,3

				B (Å^2)					
H(2A)	558,5	765,4	503,2	4					
H(3A)	216,6	763,5	473,2	5					
H'(3A)	250,6	654,5	411,2	5					
H(4A)	428,6	961,5	425,2	5					
H'(4A)	436,4	868,5	331,2	5					
H(NA)	814,5	602,4	499,2	4					
H'(NA)	864,5	496,4	410,2	4					
H(2B)	$-079,5$	564,4	236,2	4					
H(3B)	254,6	586,4	246,2	5					
H'(3B)	222,5	730,5	167,2	5					
H(4B)	096,5	412,5	146,2	5					
H'(4B)	073,6	555,4	088,2	5					
H(NB)	$-363,5$	705,4	226,2	4					
H'(NB)	$-421,5$	854,5	178,2	4					

* C, O, N parameters $\times 10^4$; H position-parameters $\times 10^3$. The e.s.d. for each parameter is separated by a comma and is given in units of the least significant digit of the corresponding parameter. The temperature factor is in the form

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

the rigid-body motion of each molecule (Schomaker & Trueblood, 1968), with the results shown in Table 3. The librational tensors, L, are remarkably similar with the principal motion for each molecule corresponding to a root-mean-square libration amplitude of about 9·6° about an axis close to the axis of minimum inertia and close also to the direction of one of the N–H···O hydrogen bonds. Further discussion of the implications of the results in Table 3 is deferred until after the hydrogen-bonding scheme has been described. The librational tensors of Table 3 were used to calculate corrections for the intramolecular distances on the assumption that each molecule is reasonably rigid (Cruickshank, 1956, 1961); the bond distances and bond angles both before and after correction are shown in Table 4 and the average corrected distances and angles are illustrated in Fig. 1.

The dimensions of the two independent molecules agree with one another within experimental error, and are also, except for the C–O distance and the angles involving the nitrogen atom in the amide group, consistent with the corresponding features (uncorrected for libration) of the molecule of cyclopropyl carbohydrazide given in the last column of Table 4 (Chesnut & Marsh, 1958). Chesnut & Marsh estimated that the thermal motion correction to their C–O distance would lengthen it to about 1·23 Å, and of course the presence of the additional –NH$_2$ group in the hydrazide group makes comparison of the angles involving the amide nitrogen atom not very meaningful. The dimensions of the amide group in the present molecule are, in fact, typical of those found in many amides. For example, the average C=O and C–N distances in three recent precise studies of amides (Ammon & Jensen, 1967; Hamilton, 1965; Hughes & Small, 1962) were 1·257 and 1·333 respectively, in good agreement with the present values of 1·260 and 1·328.

The average C–C distance in the cyclopropyl rings in the present structure, including librational corrections, is 1·50 Å (1·498 Å), the same as that (uncorrected) in the corresponding hydrazide, 1·497 Å (Chesnut & Marsh, 1958), and somewhat less than

Table 3. *Rigid-body motion parameters**

		Molecule A			Molecule B	
L, σ(L) [(°)^2]	59,6	−39,7	−5,4	60,12	−33,12	8,5
		47,5	7,3		53,8	−14,3
			25,3			16,4
T, σ(T), (Å^2 × 10^{-4})	427,13	−30,14	−18,12	399,20	−56,21	−60,20
		376,15	10,14		493,24	67,22
			430,17			425,29
S, σ(S) [Å-(°) × 10^{-2}]	31,8	22,5	−43,5	18,14	−27,9	4,8
	−36,5	−21,6	1,4	−7,7	14,10	40,6
	14,3	17,3	−10,	−13,5	−26,5	−32,

Principal axes:	Value	Direction cosines			Value	Direction cosines		
L	93 (°)^2	−749,	652,	120	93 (°)^2	719,	−667,	194
	25	236,	093,	967	25	682,	625,	−379
	13	620,	752,	−224	11	132,	405,	905
Reduced T	0·0441 Å^2	−748,	603,	275	0·0542 Å^2	−563,	683,	465
	0·0367	362,	024,	932	0·0369	374,	713,	−593
	0·0302	556,	797,	−236	0·0289	737,	160,	657
Reduced S	0·21 Å-(°)	852,	−458,	−253	0·33 Å-(°)	902,	−430,	037
	−0·03	204,	737,	−644	−0·12	396,	792,	−466
	−0·18	481,	498,	722	−0·21	173,	434,	884

$[\Sigma (\varDelta U_{ij})^2/(m-s)]^{1/2}$	0·0024 Å^2	0·0039 Å^2
Symmetrizing origin: ϱ_1	0·84 Å	0·89 Å
ϱ_2	−0·31	0·23
ϱ_3	−0·10	−0·08
Coordinates of displaced axes: $^2\varrho_1$	0·69	0·87
$^3\varrho_1$	1·12	0·94
$^1\varrho_2$	−0·34	0·24
$^3\varrho_2$	−0·04	0·18
$^1\varrho_3$	−0·10	0·00
$^2\varrho_3$	−0·06	−0·40
Effective screw translations: Parallel to $L1$	0·020 Å	0·029 Å
$L2$	−0·026	−0·014
$L3$	−0·016	−0·065

* Notation is that of Schomaker & Trueblood (1968). Some values differ slightly (but not significantly) from those given there because of the indexing error and f-table changes (see text). The reference axes are a, b, c*. The first T and S listed are relative to the center of mass; the *reduced* T and S are those corresponding to equations (20) and (12) of Schomaker & Trueblood. The positions of the symmetrizing origin and the displacements of the axes are relative to the center of mass, measured parallel to the principal axes of L. All direction cosines have been multiplied by 10^3.

those in crystalline bicyclopropyl at $-105°$, $1\cdot507$ Å (uncorrected) (Eraker & Rømming, 1967) and crystalline *sym*-tricyanocyclopropane, $1\cdot518$ Å (corrected) (Hartman & Hirshfeld, 1966), as well as in gaseous cyclopropane, $1\cdot510$ Å (Bastiansen, Fritsch, & Hedberg, 1964) and cyclopropyl cyanide, $1\cdot513$ Å (Friend & Dailey, 1958). This suggests that possibly the librational corrections to the ring bond lengths in the present structure (which averaged $0\cdot018$ Å) were somewhat underestimated, and makes questionable the significance of the observation that the bond opposite the amide group is the shortest ring bond in each of the independent molecules in this structure, as well as in the hydrazide. It is noteworthy in this connection that in bicyclopropyl at $-105°$, where there is little evidence of large molecular motion, the unique 'back' ring bond is very slightly (although scarcely significantly) longer than the other two ring bonds (Eraker & Rømming, 1967).

The two independent molecules have the same conformation in the present structure, with the planes of the amide group and the ring (Table 5) nearly orthogonal. The angle between the normals to these planes is $87\cdot8°$ in molecule A and $88\cdot3°$ in molecule B; in the hydrazide (Chesnut & Marsh, 1958) the corresponding angle is $89\cdot4°$. In each molecule, the oxygen atom lies over the three-membered ring. This conformation is also the predominant one found by vapor-phase electron diffraction for cyclopropyl methyl ketone and cyclopropanecarboxylic acid chloride (Bartell, Guillory, & Parks, 1965), as well as for the corresponding aldehyde (Bartell & Guillory, 1965). Indeed, the average intramolecular $O\cdots C(3)$ and $O\cdots C(4)$ distance in the present molecule, $2\cdot92$ Å (Table 7), is essentially identical with that found for the aldehyde, $2\cdot925$ Å, by Bartell & Guillory. Katon, Feairheller & Miller (1968) confirm, with evidence from infrared spectra, that the vapor of the acid chloride has the *S-cis* conformation (with the carbonyl oxygen over the ring, as in the present molecule), but conclude from the spectrum of the solid that crystals of the acid chloride contain the molecule in the *S-trans* conformation, differing by a $180°$ rotation about the exocyclic C–C bond. There is no good evidence on the barrier heights in compounds of this type, but Bartell & Guillory conclude that it is above $2\cdot5$ kcal per mole.

Table 4. *Bond distances and angles**

Distance	Uncorrected		Corrected†		Average	$C_3H_5CONHNH_2$ (uncorrected‡)
	A	*B*	*A*	*B*		
C(1)–O	1·239 Å	1·249 Å	1·254 Å	1·265 Å	1·260 Å	1·213 Å
C(1)–N	1·317	1·316	1·328	1·327	1·328	1·329
C(1)–C(2)	1·484	1·470	1·495	1·479	1·487	1·478
C(2)–C(3)	1·484	1·489	1·499	1·504	1·502	1·493
C(2)–C(4)	1·501	1·493	1·515	1·510	1·512	1·520
C(3)–C(4)	1·467	1·450	1·490	1·472	1·481	1·478
Angle						
N–1–O	122·5°	121·6°	122·8°	122·0°	122·4°	124·5°
2 –1–O	121·9	121·3	122·0	121·3	121·6	121·9
2 –1–N	115·6	117·1	115·2	116·8	116·0	113·6
1 –2–3	118·8	118·4	118·3	118·1	118·2	118·8
1 –2–4	117·0	117·9	116·6	117·5	117·0	118·3
3 –2–4	58·9	58·2	59·3	58·5	58·9	58·8
2 –3–4	61·1	61·1	60·9	61·0	61·0	61·5
2 –4–3	60·0	60·7	59·8	60·6	60·2	59·7

Distance			Angle		
C(2)–H	1·00 Å	0·85 Å	1 –2–H	116°	116°
C(3)–H	0·89	1·00	3 –2–H	118	117
C(3)–H′	0·84	0·96	4 –2–H	117	117
C(4)–H	0·92	0·88	2 –3–H	121	114
C(4)–H′	1·09	0·86	2 –3–H′	116	113
N——H	1·03	0·78	4 –3–H	117	119
N——H′	0·93	1·02	4 –3–H′	129	116
			H–3–H′	106	120
Angle			2 –4–H	108	118
1 –N–H	119°	120°	2 –4–H′	120	109
1 –N–H′	115	127	3 –4–H	119	120
H–N–H′	124	113	3 –4–H′	120	112
			H–4–H′	116	122

* See Fig. 1 for identities of atoms. Carbon atoms are identified by number only in giving angles. E.s.d.'s for bond distances are: C–O, C–N, 0·006 Å; C–C, 0·007 Å; C–H, N–H, 0·06 Å. E.s.d.'s for angles involving heavy atoms are 0·6°; X–X–H, 3°; H–X–H, 5°.

† Libration corrections using data of Table 3 and Cruickshank's (1956, 1961) method.

‡ Chesnut & Marsh (1958).

Hydrogen bonding and molecular packing

Each hydrogen atom of each amide group participates in a N–H$\cdots$O hydrogen bond to a molecule of the other kind (Table 6); thus there are four unique N–H$\cdots$O bonds in the structure, joining A molecules and B molecules. There are no hydrogen bonds linking symmetry-equivalent molecules directly, and thus no hydrogen bonds linking pairs of molecules around centers of symmetry, a structural feature found in some, but by no means all, other amide crystals.

The length of each of the hydrogen bonds (Table 6) is within 0·05 Å of the average of 2·96 Å reported by Fuller (1959) for twenty-two similar bonds between –NH$_2$ and C=O groups (when the latter are not incorporated in a ring). The N–H$\cdots$O and H–N$\cdots$O angles listed in Table 6 are also quite typical (Donohue, 1968). The hydrogen atoms of the amide groups lie essentially in the amide-group planes (Table 5), although the deviation of H(N) of molecule A by 0·21 Å

may be significant. The acceptor oxygen atoms of these hydrogen bonds also lie not far from the planes of the amide groups (Table 5); even the greatest departure, that of O(A), IV (in hydrogen bond 4 of Table 6 and Figs. 2 and 3), corresponds to only an 11° deviation of the N$\cdots$O line from the amide-group plane. These approximate coplanarities, together with the fact that C–N$\cdots$O angles (Table 6) are not far from 120° and the fact that the deviations of the hydrogen bonds from linearity are not large, are consistent with a picture of a trigonally hybridized nitrogen atom.

A common picture of the oxygen atom of a carbonyl group is that it is also trigonally hybridized, with two unshared electron pairs in sp^2 orbitals lying in the amide-group plane, at 120° to the C–O line. It is then sometimes inferred that the acceptor angles at the oxygen atom (N$\cdots$O=C) should be 120°, with the lone pairs acting as acceptors for the hydrogen bonds in the planes of the *accepting* amide groups. However, Donohue (1968) has surveyed a number of relevant structures

Table 5. *Planes of rings and amide groups**

	Molecule A		Molecule B	
	Ring	Amide group	Ring	Amide group
$l_1 \times 10^4$	-1198	-4764	0903	3856
$l_2 \times 10^4$	5520	-7409	-7181	6432
$l_3 \times 10^4$	8252	4735	6900	6615
D (Å)	$-8·953$	2·712	1·474	$-5·283$
Deviations (Å)				
C(2)	0	0·00	0	$-0·01$
C(3)	0	0·59	0	0·61
C(4)	0	$-0·87$	0	$-0·83$
C(1)	$-1·25$	0	$-1·24$	0
O	$-2·37$	0	$-2·36$	0
N	$-1·10$	0	$-1·11$	0
H(N)		0·21		$-0·07$
H'(N)		0·00		0·05
O(B), III (110)		0·30		
O(B), IV (1$\bar{1}$0)		0·16		
O(A), I ($\bar{1}$00)				0·16
O(A), IV				0·57
N(B), I (100)		$-2·72$		
N(B), IV (0$\bar{1}$0)		2·40		
N(A), 3 ($\bar{1}$1$\bar{1}$)				$-1·56$
N(A), 4 (100)				1·64

* Calculated by the method of Schomaker, Waser, Marsh & Bergman (1959). The equation of each plane is in the form $l_1X + l_2Y + l_3Z + D = 0$, with X, Y and Z orthogonalized coordinates in Å parallel to **a**, **b** and **c***. The three atoms for which the deviation is 0 for each plane were used to define that plane. The parameters used were those of Table 2 and do not include any allowance for librational distortion. The last eight atoms listed are in the nearby molecules with which hydrogen bonds are formed (see Table 6).

Table 6. *Hydrogen bonds**

	N$\cdots$O	Angles			
Bond	Distance	N–H$\cdots$O	H–N$\cdots$O	C–N$\cdots$O	N$\cdots$O=C
1. N(A)–H $\cdots$O(B), III (110)	2·92 Å	168°	8°	125°	139°
2. N(A)–H'$\cdots$O(B), IV (1$\bar{1}$0)	2·96	154	18	133	142
3. N(B)–H $\cdots$O(A), I ($\bar{1}$00)	2·98	168	9	116	114
4. N(B)–H'$\cdots$O(A), IV	3·01	168	8	125	127

* The different equivalent positions are identified as follows: I, x,y,z; II, $-x,-y,-z$; III, $x,\frac{1}{2}-y,\frac{1}{2}+z$; IV, $-x,\frac{1}{2}+y,\frac{1}{2}-z$. The triples of numbers in parentheses represent translations parallel to **a**, **b** and **c** respectively.

and concludes that 'the notion of the 120° angle is also quite unimportant in hydrogen bonds of this type. Not only is there wide variation of the angles NH···O=C from 120°, but also there are large deviations of the NH donors from the plane of the carbonyl system'. Examination of Table 6 might seem to belie this state-

ment, for two of the N···O=C angles are close to 120°, and the other two deviate much less than many of those he cited. However, the data in Table 5 show that the near-120° angles of Table 6 have no connection with possible sp^2 hybridization of the oxygen atom, for the donating nitrogen atoms in those bonds
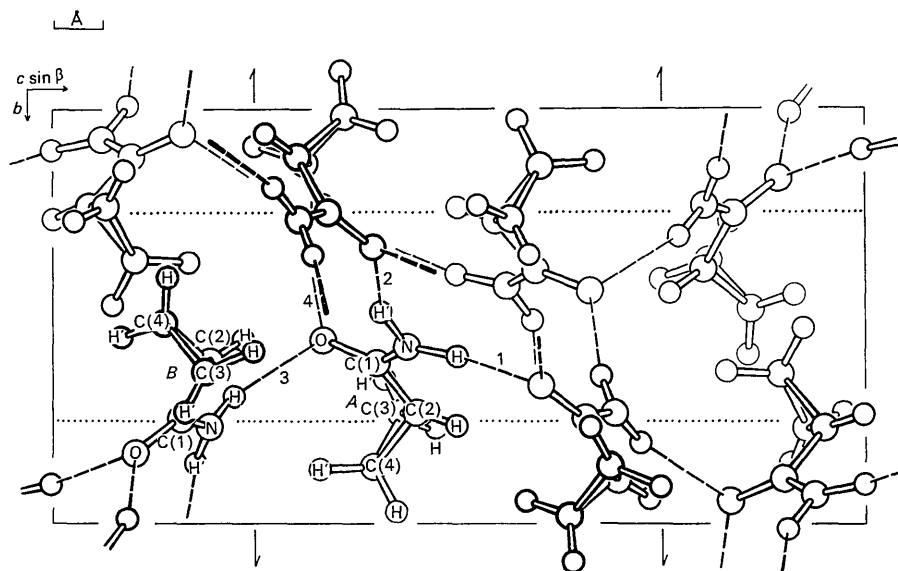
Fig. 2. A view of the structure in the −a direction. The numbers beside the hydrogen bonds correspond to the order in which these bonds are listed in Table 6. (The molecule labelled B here is at x+1, y, z in terms of the coordinates of Table 2.)
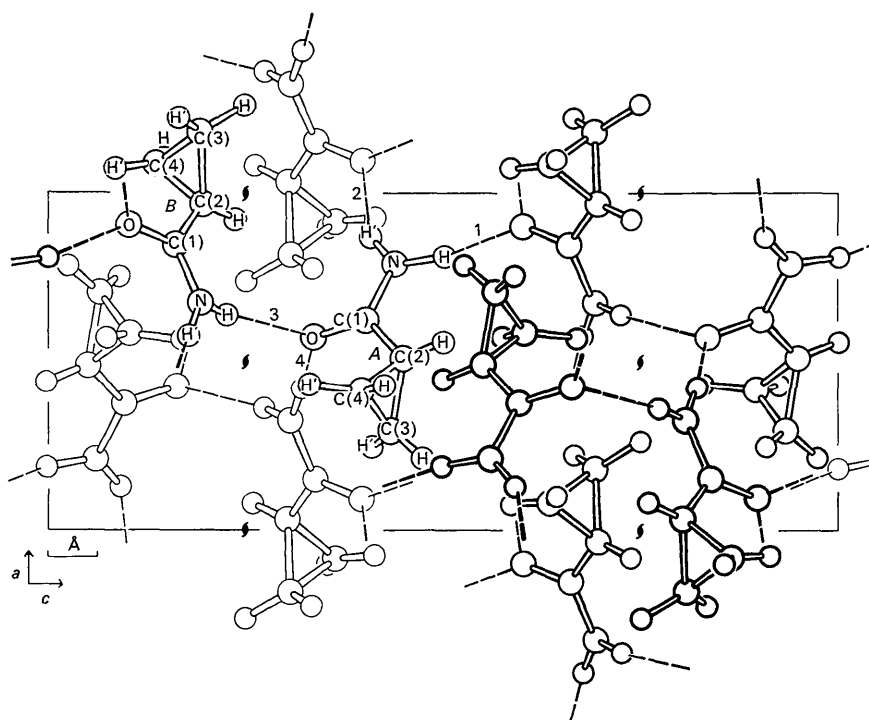
Fig. 3. A view of the structure in the −b direction. The numbers beside the hydrogen bonds correspond to the order in which these bonds are listed in Table 6. (The molecule labelled B here is at x+1, y, z in terms of the coordinates of Table 2.)

(3 and 4 of Table 6) are 2·7 and 2·4 Å from the planes of the accepting amide groups. The fact that they are far from these planes is also evident in Figs. 2 and 3. The donating nitrogen atoms in the other two bonds also lie more than 1·5 Å from the planes of the accepting amide groups, thus confirming Donohue's view about the irrelevance to hydrogen-bond patterns of the trigonal-hybridization picture of carbonyl oxygen atoms.

Because cyclopropanecarboxamide is a rather small and compact molecule, it is not surprising that in a structure held together by an efficient hydrogen-bonding network linking every molecule to four neighbors there are comparatively few other intermolecular distances close to the sum of the van der Waals radii. Fig. 4 is a representation of the packing in the structure, viewed from the same direction as in Fig. 3. Table 7 lists the shortest intermolecular distances (other than those involved in the hydrogen bonds). Actually, all of the intermolecular distances in Table 7 exceed the sum of the van der Waals radii for the atoms involved, although the contact of O(A) with H(B) (which is quite evident in Fig. 2, near hydrogen bond 3) is at a distance comparable to this sum. The fact that there are no close contacts other than those involved in the hydrogen bonding suggests that, subject to the constraints imposed by the hydrogen bonding system, there might be some freedom of molecular motion in the crystal. That this is indeed true is shown in the next section.

Table 7. *Some non-bonded distances of interest*

(a) Intramolecular distances

	Molecule A	Molecule B
H(N)···H(2)	2·2 Å	2·3 Å
N···H(2)	2·51	2·48
O···H'(3)	2·70	2·55
O···H'(4)	2·75	2·56
O···H'(N)	2·38	2·59
O···C(3)	2·91	2·90
O···C(4)	2·95	2·93

(b) Shortest intermolecular distances*

From atom at x,y,z	To atom	Distance
H(2A)	H(4A), II (121)	2·6 Å
H'(NB)	H(3B), IV	2·6
H(NB)	H'(4A), I ($\bar{1}$00)	2·6
H'(4B)	H(3A), III (01$\bar{1}$)	2·6
H'(NA)	H'(3B), IV (1$\bar{1}$0)	2·6
H(NA)	H'(3A), II (111)	2·6
O(A)	H(3B), I	2·67
N(B)	H(3B), IV	2·87
C(1A)	H'(NB), IV (0$\bar{1}$0)	2·89
O(A)	H(2B), I (100)	2·89
O(B)	H'(3A), IV	2·90
N(A)	C(3A), II (111)	3·34

* Not including the hydrogen bonds of Table 6. All H···H distances less than or equal to 2·6 Å, all C,O,N···H distances less than or equal to 2·90 Å, and all C,O,N···C,O,N distances less than or equal to 3·50 Å are included. The symbols for equivalent positions are explained in Table 6.
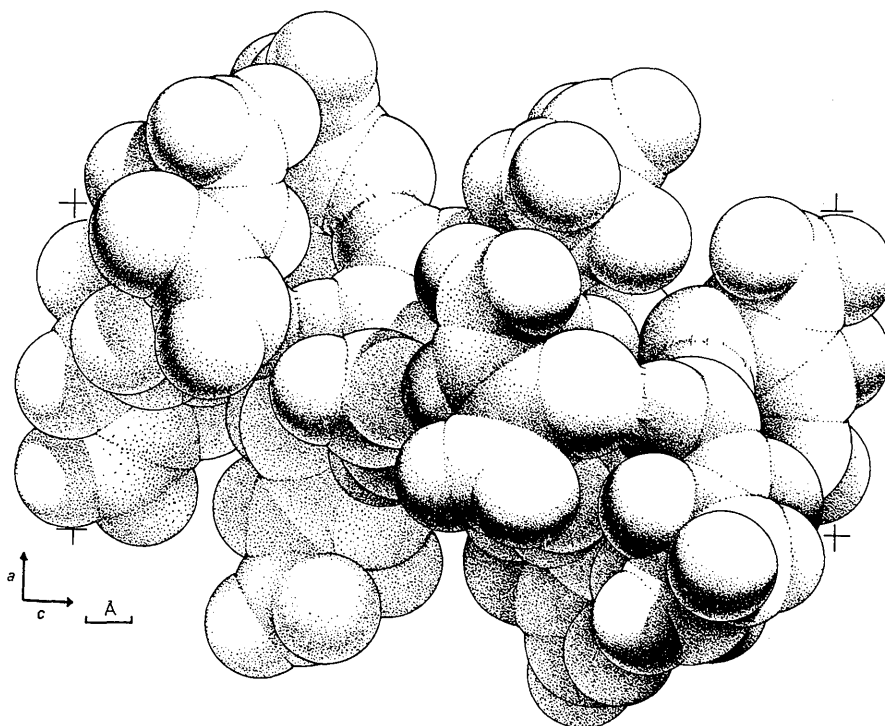

Fig. 4. A packing drawing of the structure in the $-\mathbf{b}$ direction, corresponding to the view in Fig. 3.

Table 8. *Mean-square atomic displacement tensors* (Å$^2 \times 10^4$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1A)	403, −10	366, 3	410, 24	−24, 18	−36, −14	17, 0
C(2A)	613, 7	530, 7	530, −5	127, 19	−88, −28	−67, 32
C(3A)	563, 14	762, 9	757, −4	118, −25	142, 28	−44, −27
C(4A)	706, 8	471, −16	903, 3	136, −10	−52, 16	2, −1
O(A)	468, 19	512, 4	399, −8	−36, −20	−49, −4	−35, −1
N(A)	527, −37	607, −7	512, −11	138, 18	164, 2	−108, −3
C(1B)	393, 6	432, −24	442, 69	−67, 10	−63, 20	61, 11
C(2B)	444, −37	568, 56	510, −13	−14, −21	15, 45	114, 5
C(3B)	458, −8	748, 5	1016, 12	32, −5	−192, −42	247, 5
C(4B)	675, 42	679, −47	837, −11	153, 1	90, −14	5, −20
O(B)	442, 5	747, 27	545, −33	−62, −10	−48, −17	287, 14
N(B)	462, −8	511, −17	505, −24	31, 25	38, 8	93, −16

* The first number in each entry is the observed U_{ij}, corresponding to the b_{ij} of Table 2; the second number is the first minus the U_{ij} calculated from the rigid-body parameters of Table 3. The reference axes are a, b and c*. The r.m.s. values of U_{ij} are 0·0016 Å^2 for molecule A and 0·0026 Å^2 for molecule B, corresponding respectively to somewhat less than 3 and 5 per cent of the average U_{ii}.

Molecular motion

The remarkable similarity of the librational tensors for the two independent molecules (Table 3) provides strong support for the belief that the thermal parameters of Table 2 are meaningful and are not mere artifacts. On the other hand, as discussed elsewhere (Trueblood, 1969), the problem of differentiating between internal torsional motion around each C(1)–C(2) bond and overall librational motion of each molecule as a rigid body cannot be resolved clearly with the present data. Schomaker (1967) has pointed out that the motion of two rigid bodies coupled at one point (*e.g.* about a bond) can be described with 34 independent parameters (there being one overall **T**, and two independent **L**'s and **S**'s); since we have only 36 independent pieces of data for each molecule, the temperature factors of Table 2, no attempt has been made to apply a coupled-rigid-body treatment.

The quality of the fit of the rigid-body analysis is indicated in Table 8, which lists the individual atomic displacement tensors, **U**, and the deviations of those calculated from the rigid-body parameters. The fit is distinctly better for molecule A than for B, but is well within the precision of the data (indicated in Table 2) for each. The principal apparent librational motion is about an axis which is close to the axis of minimum inertia for each molecule (Table 9), an effect which is characteristic of the motion of many different molecules in crystals as deduced from X-ray data (Trueblood, 1969). When cyclopropanecarboxamide is in the 90°-conformation, as it approximately is in this structure, the axis of minimum inertia is along a line in the plane of the amide group, from a point somewhat below the midpoint of the C(3)–C(4) bond and passing not far from C(1). As is evident in Fig. 2, this is also approximately the direction of the hydrogen bond involving H'(N) of each molecule, and indeed (as shown in Table 9), the principal librational motion is not far from each of these bonds. Whether these bonds play a structural role in the motion, *i.e.* whether there is

some twisting of the molecules about a relatively rigid hydrogen-bond framework, is not clear, but the data do suggest that this may occur.

Table 9. *Angular deviations of the principal librational axis*

From	Molecule A	Molecule B
Axis of minimum inertia	2°	11°
N–H'···O	7	11
C(1)–C(2)	24	15

* See Table 3 for direction cosines of this axis. The hydrogen-bond direction used is that of the N···O line; the hydrogen bonds involved are numbered 2 and 4 in Table 6, Fig. 2 and Fig. 3.

The possibility of a significant contribution of torsion about C(1)–C(2) to the largest apparent librational amplitude is indicated by the fact that (Table 9) the axis of this motion deviates by only about 24° and 15° from the C(1)–C(2) direction in the two molecules. The conformation found here is clearly the preferred one for molecules of this kind, but diffraction data unfortunately provide little information about the magnitude of the barrier or shape of the potential function for motion about this bond.

The other parameters of the assumed rigid-body motion are given in Table 3. Since the translational and screw tensors, **T** and **S**, vary with the origin chosen for the calculation, although **L** does not (Schomaker & Trueblood, 1968), the corresponding reduced quantities, which are origin-independent, are given. The mutual displacements of the non-intersecting librational axes corresponding to the reduced **T** are seen to amount to up to about 0·4 Å (*e.g.* for molecule A, the difference in the displacements of axes $L2$ and $L3$ parallel to $L1$ is 0·69–1·12=0·43 Å).

Burke Laing, for assistance and advice; Bonnie Warn and Sandra Smith, who did many of the final calculations; Maryellin Reinecke for the illustrations; the UCLA Campus Computing Network for its cooperation, and the National Science Foundation and Air Force Office of Scientific Research for their generous support.

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The Crystal Structure of Diphenyl Disulphide

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The crystal structure of diphenyl disulphide has been determined by three-dimensional X-ray analysis. Crystals are orthorhombic and belong to the space group $P2_12_12_1$. The unit cell has dimensions $a = 23·78$, $b = 8·13$, $c = 5·64$ Å, and contains four molecules. Positional and temperature factor parameters for the sulphur and carbon atoms have been refined by Fourier and full-matrix least-squares methods on 963 independent reflexions. In the final stages hydrogen atoms were included in fixed positions, the final R value being 9·0%. The molecule has a non-planar configuration, and the planes of the benzene rings are inclined at an angle of 77·3° to each other. The S–S bond length of 2·03 Å is slightly shorter than the normal single-bond distance, suggesting 15% of double-bond character. The S–C bond lengths of 1·79 and 1·81 Å are close to the normal single bond distance.

Introduction

Certain organic disulphides are used as additives to lubricating oils, since their presence increases the load bearing properties of the oil, especially at high pressures.

Allum & Ford (1965) have shown that extreme pressure activity of organic disulphides increases along the series:

phenyl < n-butyl < s-butyl < t-butyl < benzyl,

and suggest that the load bearing properties under these conditions are structural phenomena probably depending on the strength of the sulphur–carbon bond. Furthermore, Allum & Forbes (1967) have shown that the anti-wear properties of organic disulphides in-