applied force and the net plane used were the same for Fig. 3 as for Fig. 2.

Position *a* covers the regions *F* and *D*. Elliptic and hook-shaped fringes are recognized, although a parallel-sided crystal was used. Similar ones appear also in the top and bottom parts of sections *b* and *c*. Incidentally, if the crystal is in the shape of a parallel slab and undistorted, a system of parallel fringes should be expected (Kato, 1961*a*, *b*; Authier & Lang, unpublished). The appearance of non-parallel fringes is understandable by the dynamical diffraction theory on slightly deformed crystals (Kato, 1963, 1964*a*, *b*). According to the theory, the fringe distance should decrease with increase of the strain gradient.* In fact, the fringe position is determined by an effective thickness *Z* which is proportional to the real thickness and the strain gradient (*cf.* equation (30) of Kato, 1964*b*). Since the strain gradient is maximum on the central line *PP′* of Fig. 2 in every column section of the crystal, higher order fringes will spring up from the central part of the section pattern. In other words, the central part is effectively thick. Since the fringes in the traverse pattern are essentially the loci of the apices of the hook-shaped and elliptic fringes, they may be called the 'fringes of equal strain-gradient', just as the terms 'equal thickness fringes' and 'equal inclination fringes' are used in electron microscopy. The fact that the behaviour of fringe phenomena is the same for the *hkl* and $\bar{h}\bar{k}\bar{l}$ reflexions is also expected from the theory.

The dark background in the section pattern *a* accounts for the appearance of the region *D* in the traverse pattern. In general, the darkness of traverse patterns corresponds to the integrated intensity of crystals. The enhancement of the integrated intensity in distorted crystals is expected from the previous theory (Kato, 1964*b*). According to the theory, however, the departure from Friedel's law can not be expected unless the Borrmann absorption is taken into account. In the case of our region *D* the Borrmann absorption has nothing to do with the observation, because the sign of the indices for the net plane in which the enhancement is observed is opposite to the sign predicted by the theory under the experimental conditions described above.

In the central part of section pattern *b*, a broad line of high intensity is recognized. This broad line appears also in section pattern *c*. From the geometrical correspondence between the section and traverse patterns we know that the region *G* corresponds to the central broad line in the section pattern. For this reason the regions *D* and *G* were distinguished above, although they look similar in the traverse pattern so far as the intensity is concerned.

Finally, in section pattern *c*, a pair of dark images appears on either side of the central dark line. In pattern *b* the corresponding one is recognized markedly only on the right side of the central line. These pair images correspond to the region *E* in the traverse pattern.

In conclusion, the fringe phenomena can be explained by assuming the contraction of fringe distance in distorted crystals. The contraction has been predicted theoretically. The regions *D*, *E* and *G* can not be understood straightforwardly by the previous theory.

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* Although the conclusion was derived in the special case of homogeneous bending, it can be shown that the theory is applicable correctly to any type of small distortion in which the displacement can be represented in a quadratic form of coordinates (Kato & Ando, 1966).

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Bond lengths and thermal vibrations in *m*-dinitrobenzene.

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X-ray analysis of *m*-dinitrobenzene crystals (Trotter, 1961*a*, *b*) has established the main features of the molecular structure: the carbon and nitrogen atoms all lie in one plane, but the nitro groups are twisted about 11° out of this plane. The bond distances and valency angles were, however, less precisely measured, and showed variations which were unlikely to be real. A full-matrix least-squares refinement (unpublished), in which a scale-factor parameter for each layer was refined, suggested that much of the trouble in obtaining satisfactory results might be associated with inaccuracies in the scale factors relating the various *hkL* layers ($L=0\rightarrow3$), rather than in the refinement methods, and to overcome possible errors a new set of intensity data has now been measured by counter methods, and more accurate values of the bond distances and angles and of the thermal parameters have been obtained.

The cell parameters, remeasured on the General Electric Goniostat, are: $a=13\cdot257$, $b=14\cdot048$, $c=3\cdot806$ Å. The intensities of all 823 reflexions with $2\theta(\text{Cu } K\alpha)\le146°$ (minimum $d=0\cdot81$ Å) were measured with a scintillation counter, using the θ–2θ scan; 783 reflexions had intensities above background. The structure was refined by block-diagonal least-squares, minimizing $\Sigma w(|F_o|-|F_c|)^2$, with $\sqrt{w}=|F_o|/3$ when $|F_o|<3$ and $\sqrt{w}=1$ when $|F_o|\ge3$. Hydrogen atoms were refined with isotropic, and the heavier atoms with anisotropic thermal parameters; the final R was 0·097 for the observed reflexions (structure factors are listed in Table 1, and comparison with the previous F_o values (Trotter, 1961*a*) indicates that the inter-layer scaling in the previous data contained inaccuracies). The final positional and thermal

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* Alfred P. Sloan Foundation Fellow.

Table 1. *Measured and calculated structure factors*

Columns are h, $10F_o$, $10|F_c|$

h	$10F_o$	$10\lvert F_c\rvert$
HK0		
K= 0		
2	259	267
4	331	380
6	527	539
8	55	36
10	107	112
12	35	30
14	25	34
16	32	34
K= 1		
1	117	115
2	280	270
3	601	629
4	51	50
5	164	155
6	8	10
7	383	371
8	34	39
9	9	15
10	27	20
11	10	15
12	11	3
13	74	93
14	28	23
15	11	12
16	16	1
K= 2		
0	291	286
1	305	299
2	247	244
3	17	19
4	242	233
5	373	365
6	146	136
7	170	163
8	101	104
9	34	41
10	45	54
11	26	9
12	49	57
13	67	70
14	7	15
15	6	1
16	23	20
K= 3		
1	523	522
2	380	360
3	272	251
4	109	75
5	134	130
6	26	3
7	72	73
8	96	96
9	53	50
10	96	103
11	37	41
12	11	6
13	7	14
14	14	6
15	6	10
16	13	13
K= 4		
0	311	314
1	431	406
2	141	132
3	145	138
4	36	31
5	127	129
6	26	5
7	94	87
8	137	131
9	55	51
10	117	123
11	45	41
12	17	21
13	22	14
14	6	1
15	10	23
16	35	40
K= 5		
1	102	102
2	236	234
3	155	145
4	153	160
5	25	25
6	22	43
7	43	50
8	21	26
9	28	33
10	241	243
11	39	34
12	46	47
13	31	31
14	9	3
15	9	6
K= 6		
0	369	355
1	180	160
2	26	0
3	528	497
4	78	78
5	164	156
6	17	0
7	30	24
8	25	28
9	158	164
10	55	57
11	102	103
12	54	63
13	40	30
14	9	7
15	39	44
K= 7		
1	67	56
2	155	147
3	171	162
4	51	40
5	159	161
6	52	54
7	99	93
8	164	177
9	8	2
10	122	129
11	77	73
12	29	32
13	12	6
14	24	13
15	8	2
K= 8		
0	178	190
1	97	99
2	98	83
3	23	7
4	71	45
5	170	160
6	6	1
7	22	17
8	154	159
9	94	93
10	128	123
11	79	73
12	13	12
13	24	25
14	24	26
K= 9		
1	45	75
2	100	83
3	58	56
4	75	61
5	14	13
6	18	15
7	13	10
8	167	158
9	52	45
10	48	43
11	30	33
12	32	29
13	6	15
14	9	5
K=10		
0	18	10
1	102	105
2	46	37
3	43	50
4	102	99
5	11	16
6	21	18
7	4	6
8	45	41
9	30	21
10	38	39
11	34	18
12	24	21
13	5	8
K=11		
1	37	42
2	72	73
3	46	44
4	30	30
5	30	28
6	16	15
7	38	42
8	29	36
9	19	3
10	11	4
11	17	12
12	15	11
K=12		
0	164	185
1	30	20
2	128	119
3	12	23
4	<4	7
5	39	36
6	67	69
7	19	18
8	43	37
9	61	61
10	17	11
11	10	18
K=13		
1	51	59
2	95	88
3	18	13
4	21	23
5	67	63
6	7	1
7	<4	7
8	56	53
9	<3	6
10	29	32
11	25	23
K=14		
0	<4	3
1	95	89
2	68	63
3	51	46
4	7	5
5	24	25
6	7	15
7	24	24
8	13	16
9	4	18
K=15		
1	23	22
2	6	3
3	53	45
4	4	6
5	19	22
6	6	12
7	8	11
8	11	10
K=16		
0	7	6
1	26	22
2	<3	7
3	6	3
4	44	40
5	<3	1
6	5	7
K=17		
1	20	22
2	6	2
3	2	4
HK1		
K= 0		
1	761	737
3	197	198
5	266	241
7	208	189
9	77	76
11	72	82
13	35	38
15	23	27
K= 1		
1	389	389
2	273	245
3	343	307
4	106	104
5	91	73
6	142	134
7	161	145
8	74	72
9	30	34
10	36	45
11	13	15
12	25	33
13	27	25
14	47	50
15	17	24
16	18	19
K= 2		
0	905	967
1	557	581
2	293	298
3	151	160
4	190	176
5	120	112
6	290	276
7	96	89
8	85	78
9	160	160
10	50	58
11	43	43
12	37	43
13	10	12
14	11	15
15	16	12
16	25	22
K= 3		
1	513	531
2	172	175
3	113	96
4	76	73
5	270	267
6	80	73
7	90	86
8	<7	11
9	71	77
10	89	93
11	100	99
12	31	37
13	35	43
14	20	20
15	17	15
K= 4		
0	244	250
1	51	47
2	226	223
3	332	302
4	306	287
5	175	172
6	185	170
7	86	73
8	30	31
9	126	128
10	78	82
11	40	29
12	38	43
13	22	20
14	13	10
15	27	29
K= 5		
1	145	124
2	86	85
3	134	134
4	75	77
5	23	37
6	99	105
7	12	14
8	46	49
9	106	106
10	74	85
11	127	135
12	38	39
13	17	9
14	24	26
15	17	19
K= 6		
0	220	233
1	140	138
2	198	185
3	92	95
4	169	160
5	102	105
6	98	100
7	73	70
8	91	96
9	111	117
10	150	143
11	55	52
12	18	26
13	12	13
14	13	27
15	19	16
K= 7		
1	46	37
2	142	142
3	145	136
4	147	144
5	34	38
6	27	29
7	13	21
8	51	56
9	117	113
10	85	77
11	53	56
12	20	14
13	17	11
14	23	23
K= 8		
0	136	123
1	62	72
2	133	120
3	134	123
4	72	86
5	76	74
6	32	23
7	37	31
8	<4	9
9	125	124
10	61	60
11	27	25
12	33	31
13	32	35
14	21	20
K= 9		
1	63	56
2	33	26
3	46	38
4	29	27
5	21	21
6	41	39
7	55	56
8	93	87
9	57	52
10	23	18
11	27	27
12	33	34
13	6	7
K=10		
0	120	127
1	157	161
2	25	31
3	34	19
4	36	49
5	72	76
6	50	44
7	83	87
8	37	41
9	74	70
10	12	8
11	20	16
12	16	14
13	16	10
K=11		
1	54	63
2	60	58
3	22	19
4	24	21
5	13	16
6	23	29
7	<4	8
8	71	75
9	18	10
10	14	10
11	31	31
12	33	32
K=12		
0	30	27
1	135	122
2	75	77
3	47	40
4	11	14
5	28	32
6	40	41
7	46	45
8	16	25
9	23	20
10	6	2
11	9	14
K=13		
1	60	58
2	27	28
3	58	56
4	20	21
5	29	33
6	23	22
7	19	13
8	16	16
9	25	33
10	15	17
K=14		
0	56	53
1	57	45
2	47	47
3	40	37
4	24	27
5	10	8
6	28	20
7	9	7
8	15	15
9	<2	14
K=15		
1	27	30
2	30	26
3	44	34
4	13	13
5	34	27
6	26	23
7	7	7
K=16		
0	11	13
1	20	18
2	29	26
3	9	10
4	10	11
5	14	15
K=17		
1	6	6
HK2		
K= 0		
0	93	97
2	138	124
4	113	90
6	33	22
8	52	48
10	94	97
12	43	40
14	25	21
K= 1		
1	74	53
2	44	41
3	77	77
4	86	78
5	110	113
6	53	55
7	56	53
8	58	52
9	53	50
10	80	76
11	24	26
12	10	12
13	39	43
14	15	12
K= 2		
0	364	376
1	159	181
2	93	80
3	201	191
4	106	99
5	91	82
6	125	128
7	110	108
8	56	67
9	104	99
10	71	77
11	10	14
12	21	23
13	19	13
14	20	25
K= 3		
1	51	57
2	86	87
3	65	57
4	101	94
5	<7	5
6	96	97
7	60	43
8	47	37
9	37	34
10	134	124
11	71	70
12	14	21
13	27	28
14	10	15
K= 4		
0	226	209
1	229	217
2	147	152
3	236	227
4	105	83
5	74	53
6	104	103
7	55	51
8	26	32
9	121	108
10	76	58
11	32	36
12	<7	6
13	33	32
14	14	15
K= 5		
1	119	117
2	134	126
3	28	26
4	20	26
5	142	124
6	53	52
7	27	15
8	51	59
9	30	34
10	79	80
11	15	16
12	13	15
13	20	20
14	9	12
K= 6		
0	69	61
1	48	48
2	90	99
3	59	61
4	25	35
5	31	23
6	39	43
7	20	19
8	33	30
9	82	67
10	9	11
11	7	12
12	11	6
13	24	28
K= 7		
1	97	100
2	55	56
3	54	56
4	33	25
5	31	26
6	17	41
7	45	42
8	22	34
9	25	23
10	24	23
11	36	34
12	33	31
13	16	21
K= 8		
0	130	147
1	88	92
2	11	11
3	29	23
4	38	38
5	19	25
6	70	70
7	51	43
8	13	23
9	35	34
10	22	22
11	43	38
12	35	25
K= 9		
1	46	46
2	27	22
3	28	15
4	28	24
5	40	39
6	30	32
7	41	50
8	33	46
9	41	35
10	23	18
11	54	51
12	4	5
K=10		
0	182	174
1	55	58
2	132	120
3	6	4
4	57	63
5	52	43
6	25	28
7	10	6
8	59	59
9	22	19
10	26	38
11	22	14
K=11		
1	66	67
2	29	28
3	21	20
4	16	21
5	35	37
6	17	17
7	50	49
8	37	25
9	35	39
10	10	10
K=12		
0	<4	5
1	25	24
2	22	28
3	4	1
4	20	24
5	22	25
6	7	7
7	32	36
8	16	20
9	10	13
K=13		
1	9	13
2	25	24
3	16	17
4	8	6
5	3	2
6	26	24
7	10	13
8	18	20
K=14		
0	33	24
1	32	27
2	21	18
3	4	5
4	6	13
5	20	19
6	14	18
K=15		
1	13	11
2	34	32
3	12	11
4	29	24
HK3		
K= 0		
1	65	50
3	126	89
5	76	69
7	20	24
9	125	109
11	27	14
K= 1		
1	13	8
2	50	39
3	11	14
4	65	51
5	25	13
6	29	23
7	11	12
8	71	70
9	26	15
10	74	66
11	36	32
12	31	31
K= 2		
0	13	15
1	50	61
2	45	29
3	72	50
4	94	78
5	19	27
6	16	26
7	11	13
8	50	44
9	44	34
10	61	61
11	28	28
12	13	3
K= 3		
1	17	24
2	29	26
3	23	11
4	36	23
5	11	3
6	11	11
7	7	16
8	43	36
9	42	35
10	42	32
11	22	24
12	23	19
K= 4		
0	78	74
1	94	94
2	67	75
3	15	17
4	17	7
5	30	21
6	50	50
7	49	43
8	55	49
9	49	37
10	30	24
11	13	11
12	9	6
K= 5		
1	75	79
2	8	9
3	25	26
4	29	31
5	51	44
6	15	9
7	45	38
8	11	13
9	28	19
10	9	12
11	13	15
K= 6		
0	126	127
1	24	21
2	58	66
3	19	24
4	4	18
5	8	9
6	64	57
7	15	4
8	11	10
9	12	16
10	16	11
11	10	9
K= 7		
1	4	5
2	35	34
3	33	44
4	15	13
5	13	15
6	8	14
7	21	21
8	19	24
9	16	17
10	16	14
K= 8		
0	96	109
1	62	84
2	49	54
3	<4	7
4	28	39
5	33	38
6	25	25
7	24	26
8	20	20
9	11	9
10	22	17
K= 9		
1	36	39
2	15	18
3	13	16
4	24	23
5	6	9
6	<4	12
7	22	31
8	12	12
9	17	19
K=10		
0	13	22
1	20	23
2	<4	13
3	4	9
4	24	27
5	<3	15
6	21	24
7	11	24
K=11		
1	9	9
2	17	21
3	<3	11
4	6	13
5	3	7
6	17	17
7	12	14
K=12		
0	33	36
1	17	14
2	15	16
3	17	16
4	4	8
5	12	15
K=13		
1	5	5
2	12	12
3	10	10
HK4		
K= 0		
0	34	23
2	72	46
4	57	42
6	22	14
8	48	40
K= 1		
1	15	3
2	19	10
3	12	16
4	19	10
5	17	9
6	13	3
7	22	17
8	24	17
K= 2		
0	<4	15
1	<4	19
2	15	10
3	47	31
4	12	5
5	13	14
6	14	12
7	16	15
8	15	1
K= 3		
1	9	23
2	<4	3
3	8	13
4	12	9
5	13	8
6	12	1
7	28	21
8	16	12
K= 4		
0	17	47
1	4	25
2	<4	8
3	<4	7
4	16	16
5	13	9
6	29	25
7	17	14
K= 5		
1	<4	5
2	16	25
3	3	12
4	22	14
5	14	0
6	17	12
7	13	4
K= 6		
0	<3	25
1	30	75
2	<3	5
3	6	22
4	<3	9
5	34	25
6	10	7
K= 7		
1	<3	25
2	<3	14
3	<3	14
4	<3	14
5	<3	12
K= 8		
0	29	41
1	<3	16
2	<3	16
3	<3	14
4	<2	16
K= 9		
1	<2	0
2	<2	17

parameters are listed in Table 2, and the thermal motion is analysed in Table 3 in terms of rigid-body vibrations for the aromatic ring, and of mean square displacements for the nitro groups. Table 3 indicates that the ring is only approximately represented as a rigid body, and that the nitro groups exhibit very marked torsional movement, as noted previously for other nitro compounds (Trueblood, Goldish & Donohue, 1961). The measured bond distances were corrected for errors due to angular oscillations; the corrections increase all the bond lengths, C–C by 0·012 Å, C–N by 0·007 Å, N(1)–O(1) by 0·033 Å, N(1)–O(2) by 0·023 Å, N(2)–O(3) by 0·016 Å, N(2)–O(4) by 0·039 Å, and leave the valency angles virtually unchanged. Table 4 gives the uncorrected and corrected distances and the angles, together with their standard deviations.

The main features of the molecular structure previously determined are confirmed, each nitro group being rotated out of the aromatic plane by about 13°, and the directions of the rotations being such that the molecular symmetry is *m*. In addition it is now apparent that the bond distances

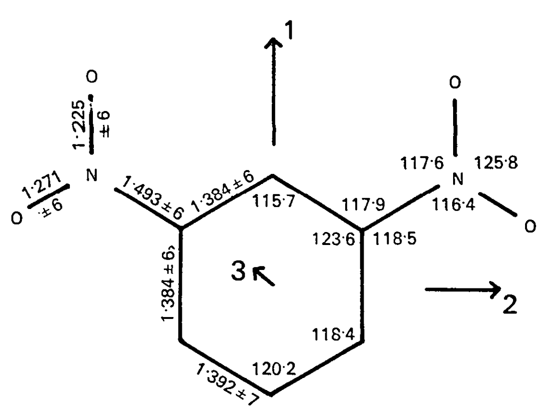

Fig. 1. *Mean bond lengths and standard deviations* (Å), *and valency angles* ($\sigma \sim 0{\cdot}4°$).

Table 2. *Final fractional positional parameters w.r. to crystal axes* ($\times 10^4$ *for* C, N, O; $\times 10^3$ *for* H), *and thermal parameters,* U_{ij} *being the components of the mean square vibration tensors* (*in* 10^{-2} Å^2) *w.r. to the molecular axes* 1, 2 *and* 3 (Fig. 1)

Atom	x	y	z	U_{11}	U_{12}	U_{13}	U_{22}	U_{23}	U_{33}
C(1)	1399	3652	−0298	3·99	2·37	−0·43	4·31	1·32	5·12
C(2)	1800	4493	−1473	4·30	1·83	0·82	4·08	−1·32	5·00
C(3)	2828	4576	−1198	4·55	1·86	−0·60	4·77	1·19	4·85
C(4)	3450	3860	−0037	6·33	4·26	1·17	5·92	−0·19	7·89
C(5)	3000	3042	1257	3·48	3·46	0·03	9·21	2·35	8·46
C(6)	1971	2934	1101	5·97	1·39	2·10	5·35	−1·50	7·63
N(1)	0288	3531	−0416	7·64	3·02	0·78	4·81	−0·50	7·64
N(2)	3297	5469	−2506	8·72	1·24	1·29	5·76	−1·09	7·42
O(1)	−0067	2850	1268	11·48	4·07	−1·01	4·60	−0·39	17·33
O(2)	−0200	4108	−1986	6·97	5·51	−0·70	7·59	0·31	11·72
O(3)	2756	6009	−4133	9·91	−0·26	0·57	6·74	−1·58	11·58
O(4)	4161	5627	−1512	13·72	6·35	2·70	4·44	−0·40	17·81
H(2)	139	489	−360	$B = 5·0$ Å^2					
H(4)	409	399	029	4·3					
H(5)	326	246	080	8·9					
H(6)	164	239	141	9·6					

Standard deviations

C, N, O	$\sigma(x) = 0·0057–0·0075$ Å	H	$\sigma(x) = 0·07–0·09$ Å
	$\sigma(y) = 0·0056–0·0080$		$\sigma(y) = 0·07–0·09$
	$\sigma(z) = 0·0082–0·0127$		$\sigma(z) = 0·09–0·13$
	$\sigma(U_{ij}) = 0·003–0·007$ Å^2		$\sigma(B) = 2·0–3·2$ Å^2

Table 3. *Analysis of thermal motion*

Aromatic ring treated as a rigid body

$$\mathbf{T} = \begin{pmatrix} 3·82 & 2·53 & 0·24 \\ & 4·15 & 0·16 \\ & & 4·14 \end{pmatrix} 10^{-2}\ \text{Å}^2 \qquad \omega = \begin{pmatrix} 27·3 & 1·3 & -9·6 \\ & 34·7 & -3·5 \\ & & 32·5 \end{pmatrix} \text{deg}^2$$

R.M.S. translations = 0·20, 0·20, 0·20 Å

R.M.S. oscillations = 5·2°, 5·9°, 5·7°

Values of $\overline{u^2}$ in various directions (10^{-2} Å^2)

	⊥ to molecular plane	∥ to N –O bonds		⊥ to N–O bonds		∥ to C–N bonds
N(1)	7·6	O(1), 6·3	O(2), 7·6	6·5	4·8	4·8
N(2)	7·4	O(4), 6·0	O(3), 8·7	7·9	5·8	5·6
O(1)	17·3	6·5		9·7		—
O(2)	11·7	7·0		7·6		—
O(3)	11·6	9·9		6·7		—
O(4)	17·8	5·4		13·5		—

Table 4. *Bond distances and valency angles* ($\sigma \sim 0·6°$)

Atoms	Distance (Å) Uncorr.	Corr.	σ
C(1)–C(2)	1·372	1·384	0·009
C(2)–C(3)	1·372	1·384	0·008
C(3)–C(4)	1·374	1·386	0·009
C(6)–C(1)	1·369	1·381	0·010
C(4)–C(5)	1·385	1·397	0·010
C(5)–C(6)	1·374	1·386	0·009
C(1)–N(1)	1·484	1·491	0·008
C(3)–N(2)	1·487	1·494	0·009
N(1)–O(1)	1·243	1·276	0·009
N(1)–O(2)	1·197	1·220	0·009
N(2)–O(3)	1·214	1·230	0·009
N(2)–O(4)	1·227	1·266	0·009
C(2)–H(2)	1·12		0·07
C(4)–H(4)	0·87		0·07
C(5)–H(5)	0·90		0·09
C(6)–H(6)	0·90		0·09

Atoms	Angle
C(6)–C(1)–C(2)	123·1°
C(1)–C(2)–C(3)	115·7
C(2)–C(3)–C(4)	124·0
C(3)–C(4)–C(5)	117·6
C(4)–C(5)–C(6)	120·2
C(5)–C(6)–C(1)	119·1
C(2)–C(1)–N(1)	118·3
C(6)–C(1)–N(1)	118·6
C(2)–C(3)–N(2)	117·5
C(4)–C(3)–N(2)	118·4
C(1)–N(1)–O(1)	116·6
C(1)–N(1)–O(2)	118·3
C(3)–N(2)–O(3)	116·8
C(3)–N(2)–O(4)	116·1
O(1)–N(1)–O(2)	125·1
O(3)–N(1)–O(4)	126·5

and valency angles also conform to symmetry *m*, and the mean distances and angles are shown in Fig. 1.

The C–C bond distances are all equal within experimental error, the average value of 1·386 ± 0·004 Å not being significantly different from the distance in benzene (1·392 Å). The mean C–N length of 1·493 ± 0·006 Å is similar to that in nitrobenzene, 1·486 ± 0·02 Å (Trotter, 1959). The N–O bond distances average 1·271 ± 0·006 Å for N(1)–O(1) and N(2)–O(4), and 1·225 ± 0·006 Å for N(1)–O(2) and N(2)–O(3). Since these averages differ by 5σ, the structure contains two types of N–O bond, one longer and the other shorter than, with their mean (1·248 Å) equal to, the N–O distance in, for example *p*-nitroaniline, 1·247 ± 0·005 Å (Trueblood, Goldish & Donohue, 1961). The valency angles are all quite similar to those in other nitro compounds. The hydrogen atom parameters are not very accurate; the mean C–H distance is 0·95 Å.

All the calculations were performed with our own programs on the IBM 7040 computer, and we are indebted to the staff of the U.B.C. Computing Centre for assistance, and the National Research Council of Canada for financial support and for the award of a research studentship to C.S.W.

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Structure of *P,P′,P″,P‴*-Tetrakistricarbonylnickel-tetraphosphorus hexaoxide, $P_4O_6[Ni(CO)_3]_4$.

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The reaction of P_4O_6 with an excess of nickel carbonyl produces $P_4O_6[Ni(CO)_3]_4$, a white powder, with evolution of four equivalents of CO. As described elsewhere (Riess & Van Wazer, 1965) the symmetric bird-cage structure of P_4O_6, which has four unshared pairs of electrons (one on each phosphorus atom) coordinates four nickel tricarbonyl groups.

Since satisfactory single crystals of this substance could not be grown, an attempt has been made to determine the structure from powder intensity data. A General Electric XRD-5 diffractometer with Cu $K\alpha$ radiation was used. Of the 27 measurable diffraction peaks observed up to a maximum $\sin\theta$ value of 0·62, only 19 were due to the diffracted spectrum from a single set of planes.

The pattern could be indexed on the basis of a cubic unit cell with $a = 8{\cdot}811 \pm 0{\cdot}003$ Å. The absence of systematic extinctions indicated four possible space groups: $P23$, $Pm3$, $P\bar{4}3m$, and $Pm3m$. The density, determined on the powdered sample with a Beckman air comparison pycnometer, is 1·865 g.cm^{-3}. This value may be compared with a calculated density of 1·919 g.cm^{-3} for one molecule in each unit cell. Accordingly, two of the possible space groups can be eliminated if it is assumed that no disorder is present, since $Pm3$ and $Pm3m$ do not have four equivalent positions.

For the initial trials it was assumed that the correct space group was $P\bar{4}3m$ with the atoms placed in the Wyckoff positions indicated in Table 2. This still leaves two possible molecular configurations which differ merely in a rotation by 60° of the $Ni(CO)_3$ groups. Standard bond lengths and angles (*Tables of Interatomic Distances*, 1958) were taken to calculate a set of coordinates. The only doubtful distance is the length of the Ni---P bond. Instead of a value of 2·26 Å as previously reported (Scatturin & Turco, 1958), a bond length of 2·15 Å corresponding to the sum of the covalent radii (Pauling, 1960) was selected. Consequently the values shown in Table 1 were adopted with linear Ni---C---O groups and tetrahedral valencies around the nickel atom. These parameters yield the coordinates shown in Table 2.

Table 1. *Assumed molecular dimensions*

P---O	1·64 Å	OPO	101°
C---O	1·14	POP	124
Ni---C	1·82		
Ni---P	2·15		

Table 2.

	Wyckoff positions		Atomic coordinates (Å)		
			X	Y	Z
Ni	4 (*e*)	$X_1X_1X_1$	2·265	2·265	2·265
P	4 (*e*)	$X_2X_2X_2$	1·024	1·024	1·024
O(1)	6 (*f*)	X_300	1·794	0	0
O(2)	12 (*i*)	$X_4X_4Z_1$	1·672	1·672	5·104
C	12 (*i*)	$X_5X_5Z_2$	1·904	1·904	4·012

A calculation of the 19 available structure factors demonstrated that the model represented in Fig. 1 was correct. The scattering factors were taken from *International Tables for X-ray Crystallography* (1962) with the Ni curve corrected for the real part of the dispersion. Attempts were made to refine the model by least-squares but no satisfactory convergence could be obtained. This was due to the paucity of observed reflexions and the inability to measure more accurately the true intensities because of the relative low degree of crystallinity and possible effects induced by preferred orientation during sample preparation. All calculations were carried out on an Elliott 803B computer with the programming system of Daly, Stephens & Wheatley (1966).