CHRISTENSEN, A. T. & WITMORE, W. G. (1969). *Acta Cryst.* B**25**, 73.
DOLLASE, W. A. (1965). *J. Amer. Chem. Soc.* **87**, 979.
DONOHUE, J. (1968). In *Structural Chemistry and Molecular Biology*, Ed. A. RICH and N. DAVIDSON, p. 443. San Francisco: Freeman.
EMERSON, K. (1966). *Acta Cryst.* **21**, 970.
FEHER, F. & LINKE, K. H. (1964). *Z. anorg. allgem. Chem.* **327**, 151.
FOSS, O. (1967). In *Selected Topics in Structure Chemistry*. Ed. P. ANDERSEN *et al.*, p. 145. Oslo: Universitetsforlaget.
FOSS, O. & HAUGE, S. (1963). *Acta Chem. Scand.* **17**, 1807.
International Tables for X-ray Crystallography (1962). Vol. III, p. 201. Birmingham: Kynoch Press.
OHLBERG, S. M. & VAUGHN, P. A. (1954). *J. Amer. Chem. Soc.* **71**, 2649.
PAULING, L. (1960). *The Nature of the Chemical Bond*, 3rd ed. Ithaca, New York: Cornell Univ. Press.
PIERCE, L., NELSON, K. & THOMAS, C. (1965). *J. Chem. Phys.* **43**, 3423.
RYCHNOVSKY, V. & BRITTON, D. (1968). *Acta Cryst.* B**24**, 725.
STOUT, G. H. & JENSEN, L. H. (1968). *X-ray Structure Determination*, pp. 409–412. New York: Macmillan.
SUTTON, L. E. (1958). *Interatomic Distances and Configuration in Molecules and Ions*. London: The Chemical Society.
THOMAS, L. F., SHERRARD, E. I. & SHERIDAN, J. (1965). *Trans. Faroday Soc.* **51**, 1311.

Acta Cryst. (1971). B**27**, 786

The Crystal Structure of Picryl Chloride

BY JEAN S. WILLIS, JAMES M. STEWART, HERMAN L. AMMON AND HUGH S. PRESTON
Department of Chemistry, University of Maryland, College Park Maryland 20742, *U.S.A.*

AND RICHARD E. GLUYAS AND P. M. HARRIS
Department of Chemistry, The Ohio State University, Colombus, Ohio 43210, *U.S.A.*

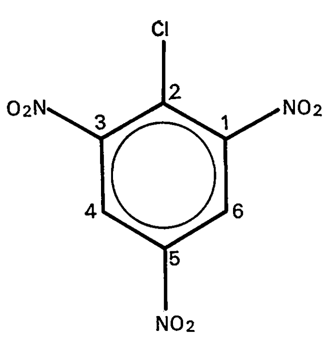
(*Received* 9 *April* 1970)

The crystal structure of picryl chloride, $C_6H_2(NO_2)_3Cl$, has been determined in separate film and diffractometer X-ray investigations. The compound crystallizes in the monoclinic space group $P2_1/a$, $Z=4$, with unit-cell dimensions $a=11{\cdot}020$, $b=6{\cdot}795$, $c=14{\cdot}964$ Å and $\beta=124{\cdot}15°$; $\varrho_{\text{meas}}=1{\cdot}797$ g.cm^{-3}, $\varrho_{\text{X-ray}}=1{\cdot}805$ g.cm^{-3} for $Z=4$. Both sets of data were refined by full-matrix least-squares methods using anisotropic temperature factors for the C, O, N and Cl atoms; H atoms were included with isotropic temperature factors in the counter data calculations. The film and diffractometer R values are 0·127 and 0·043 respectively. In most cases, the differences between the two lists of atomic parameters are less than 3σ, where σ is that of the film parameters. The two nitro groups adjacent to the chlorine atom are rotated by 33 and 81° out of the plane of the benzene ring.

Introduction

Interest in the steric interactions between nitro groups and other substituents has led to a number of structure investigations of nitro aromatics (see Holden & Dickinson, 1969, and references therein). The picryl halides (2-halo-1,3,5-trinitrobenzenes*) have been the subject of several studies during the last forty years: the structure of picryl iodide was reported by Huse & Powell (1940); Hertel & Römer (1933) proposed a packing model for picryl chloride based on a centered lattice; Kitaigorodskii (1948) reported a corrected space group and cell dimensions for the compound; and Gol'der, Zhdanov & Umanskii (1953) published a partial structure. In the latter determination, only an outline of the picryl chloride skeleton in a projection was reported and the positions of two of the oxygen atoms were not specified.

Our own work on picryl chloride, 2-chloro-1,3,5-trinitrobenzene,* has spanned a period of ten years and the results of two separate determinations, one with film and another with counter data, are reported herein. A preliminary account of part of this work has been published (Gluyas & Harris, 1959).



Experimental

Commercially available picryl chloride (Eastman Kodak Co.) recrystallized from a chloroform–carbon tet-

---

* The present *Chemical Abstracts* numbering scheme for picryl halides places the halogen on C(2).

rachloride mixture as yellow plates. The preliminary diffraction survey and film study were done at The Ohio State University. The specimens examined were found to crystallize in the monoclinic system described by Kitaigorodskii (1948) and density measurements (neutral buoyancy technique in aqueous KI) indicated four molecules per unit cell. The diffraction data were recorded with Ni-filtered Cu radiation ($\lambda = 1{\cdot}5418$ Å) on equi-inclination Weissenberg films for levels $h0l$–$h4l$ and $0kl$–$3kl$, and the intensities were visually estimated and corrected for spot size by direct measurement. These films were measured twice and the data were intercorrelated to give 1097 observed reflections with a maximum sin θ value of 0·98. Standard deviations were computed for the individual intensities using the correlation data. The structure was solved with the Patterson-heavy atom technique. Gluyas & Harris (1959) have reported the details of the film data collection, structure solution and preliminary refinement steps. The structure has been further refined* with full-matrix least squares using anisotropic temperature factors for the carbon, nitrogen, oxygen and chlorine atoms to a residual of 0·127 ($R = \sum |F_o| - |F_c| / \sum |F_o|$). Hydrogen atoms were not included.

The film work revealed large thermal parameters for a few of the oxygen atoms and, several years later, the diffraction data were recollected at the University of Maryland to provide a check of these values. The measurements were made on a Datex automated, General Electric diffractometer with a quarter circle goniostat and with Nb-filtered Mo radiation ($\lambda = 0{\cdot}71069$ Å). The crystal specimen had approximate dimensions of $0{\cdot}15 \times 0{\cdot}2 \times 0{\cdot}25$ mm and was mounted parallel to *b*. Unit-cell parameters were obtained by least-squares refinement based on the measured Bragg angles of 16 planes. Crystal data are reported in Table 1. The intensity data were collected using the 2θ scan method with a scan rate of 2° min^{-1} and 20-sec background counts. Of the 2657 unique data measured to a sinθ of 0·5 (2θ of 60°), 1310 reflections were found to be at least three standard deviations above background and were coded as 'observed'.

The data were reduced in the normal way, a scale and overall isotropic temperature factor were calculated with the *X-ray* 67 subprogram *DATFIX*, and the structure was 're-solved' using the symbolic addition procedure code *PHASE* (*X-ray* 67). The C, N, O and Cl atoms were all revealed in the first *E* map, computed with 332 terms, duplicating the film data solution and the structure was refined by full-matrix least squares using isotropic temperature factors to an *R* of 0·174. Following three cycles of anisotropic refinement, a difference synthesis was calculated and the two hydrogen atoms were located in the map. Further refinement with anisotropic thermal parameters for C, N, O and Cl and isotropic terms for H, resulted in an *R* of 0·043. The quantity minimized in the least-squares calculations was $\sum w(F_o - F_c)^2$, and the weights were computed from

$$w = [0{\cdot}25/\mathrm{MAXF}(\sigma(F_o),\ 0{\cdot}0033|F_o|,\ 0{\cdot}25]^2,$$

where MAXF refers to the Fortran maximum value function and the σ values are the structure factor standard deviations which were estimated from counting statistics. The unobserved data were not used in the refinement.

Although the thermal parameters of the oxygen atoms were still rather large, approaching root-mean-square amplitudes of 0·5 Å in a few cases, a careful examination of a difference map showed no evidence of disorder but corroborated the large anisotropy. The final difference map showed no electron density maxima greater than 0·29 e.Å^{-3}, and the largest density in the vicinities of the oxygen atoms was 0·21 e.Å^{-3}. The atomic form factors used were: carbon, nitrogen and oxygen (Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal, 1955); chlorine (*International Tables for X-ray Crystallography*, 1962); hydrogen (Stewart, Davidson & Simpson, 1965). The structure factor data are reported in Table 2.

---

* These calculations were done with *ORXLS* (Busing & Levy, 1959) on the University of Washington (Seattle, Washington) IBM 709 computer. In the later stages of this work, all computations were done on the University of Maryland UNIVAC 1108 machine using the *X-ray* 67 programs.

Table 1. *Crystal data*

Picryl chloride: 2-chloro-1,3,5-trinitrobenzene, $C_6H_2N_3O_6Cl$
Mol.wt.: 247·6
Monoclinic crystal system, diffractometer unit-cell parameters: $a = 11{\cdot}020\ (4)$ Å
$b = 6{\cdot}795\ (1)$
$c = 14{\cdot}964\ (4)$
$\beta = 124{\cdot}15\ (2)°$

Cell volume: 910·6 Å^3
Measured density: 1·797 g.cm^{-3}
Calculated density: 1·805 g.cm^{-3} for $Z = 4$
$F(000)$: 640 *e*
Absent spectra: $0k0$ for k odd, $h0l$ for h odd
Space group: $P2_1/a$ (C_{2h}^5)

An alternative unit cell with a β angle closer to 90° can be obtained with the transformation matrix $\bar{1}00/0\bar{1}0/101$. The cell parameters for the transformed cell are: $a = 11{\cdot}020$, $b = 6{\cdot}795$, $c = 12{\cdot}658$ Å, $\beta = 101{\cdot}94°$.

Table 2. *Structure factor listing for the diffractometer investigation*

The data are listed in blocks with constant h and k. Within each block the columns are l, $10F_o$ and $10F_c$. The observed data are flagged with an $*$.

0,0,L		
2	560	-554
3	372	391
4	0*	-10
5	399	412
6	498	496
7	188	-198
8	132	-142
9	345	-339
10	0*	24
11	48*	-49
12	31*	28
13	45	-51
14	35*	-20
15	36*	-22
16	17*	-12
17	22*	2
2,0,L		
0	729	-714
1	166	-153
2	662	-671
3	144	147
4	416	420
5	282	287
6	310	-303
7	73	67
8	99	99
9	89	98
10	39	36
11	99	89
12	29*	24
13	0*	0
14	25*	-29
15	13*	20
-1	277	262
-2	399	386
-3	430	419
-4	662	-672
-5	63	-68
-6	191	198
-7	473	476
-8	28	29
-9	85	82
-10	67	-59
-11	55	68
-12	73	-75
-13	25*	12
-14	40	-45
-15	21*	-11
-16	20*	-6
-17	39	-31
-18	45	-38
4,0,L		
0	0*	38
1	46	39
2	101	95
3	146	140
4	195	195
5	102	96
6	55	59
7	43	-41
8	78	84
9	34	26
10	80	75
11	16*	-14
12	45	-49
13	0*	21
-1	62	-54
-2	520	522
-3	113	-112
-4	21*	-42
-5	868	-864
-6	0*	-35
-7	297	290
-8	152	-149
-9	66	-71
-10	388	-391
-11	174	-168
-12	380	397
-13	121	-126
-14	25*	-42
-15	50	44
-16	86	-78
-17	25*	21
-18	60	60
-19	7*	-7
6,0,L		
0	254	-253
1	74	77
2	92	91
3	7*	26
4	62	54
5	9*	-20
6	59	-62
7	29*	26
8	39	24
9	9*	5
10	13*	-26
11	18*	13
-1	95	-95
-2	39	-35
-3	145	143
-4	304	307
-5	256	-252
-6	643	-633
-7	18*	17
-8	131	122
-9	15*	-3
-10	169	169
-11	73	-75
-12	394	-398
-13	6*	-12
-14	251	250
-15	45	24
-16	7*	-44
-17	22*	-18
-18	0*	-22
-19	0*	2
-20	47	39
8,0,L		
0	194	-197
1	128	126
2	70	68
3	57	-63
4	24*	-26
5	49	-51
6	26*	-24
7	24*	24
8	29*	25
-1	103	-108
-2	21*	6
-3	0*	-6
-4	172	168
-5	14*	14
-6	120	119
-7	8*	-7
-8	266	-265
-9	111	101
-10	136	144
-11	66	67
-12	75	-78
-13	34	-36
-14	74	-68
-15	40	-38
-16	25*	25
-17	0*	4
-18	18*	0
-19	15*	6
-20	47	-40
10,0,L		
0	109	112
1	16*	-7
2	24*	-9
3	25*	-20
4	61	-57
5	32*	38
-1	15*	1
-2	127	-128
-3	96	-98
-4	22*	-11
-5	79	86
-6	3*	-2
-7	60	-61
-8	37	-35
-9	108	-103
-10	118	113
-11	60	61
-12	15*	-10
-13	34	20
-14	17*	9
-15	9*	-11
-16	88	79
-17	26*	16
-18	27*	-42
-19	25*	-16
-20	0*	30
12,0,L		
0	19*	9
1	0*	-3
-1	19*	11
-2	23*	24
-3	14*	7
-4	51	-50
-5	26*	24
-6	54	58
-7	8*	-7
-8	21*	-9
-9	57	-52
-10	17*	-18
-11	52	47
-12	60	-53
-13	31*	35
-14	16*	-8
-15	49	-45
-16	26*	-20
-17	0*	7
-18	53	57
-19	9*	8
14,0,L		
-4	31*	-37
-5	33*	-40
-6	63	62
-7	47	43
-8	31*	-25
-9	13*	11
-10	42	-37
-11	37	-35
-12	81	80
-13	0*	22
-14	0*	-24
-15	23*	-28
-16	0*	2
-17	0*	16
0,1,L		
1	350	-336
2	337	-338
3	497	-514
4	198	210
5	93	104
6	376	-370
7	113	-107
8	97	100
9	32	-20
10	40	40
11	66	69
12	122	120
13	28*	12
14	16*	3
15	17*	14
16	23*	19
17	26*	16
1,1,L		
0	127	111
1	377	377
2	405	-404
3	470	-476
4	27	-21
5	21	2
6	134	141
7	15*	-9
8	120	-113
9	0*	18
10	0*	22
11	60	59
12	36	23
13	33*	-35
14	0*	8
15	0*	21
16	0*	-8
-1	46	-57
-2	89	-84
-3	489	-492
-4	242	-237
-5	325	320
-6	465	473
-7	197	-184
-8	134	-141
-9	52	-54
-10	8*	3
-11	142	140
-12	12*	-2
-13	27*	-32
-14	48	31
-15	42	-39
-16	26*	21
-17	35	30
2,1,L		
1	442	441
2	315	321
3	146	-140
4	266	281
5	67	76
6	69	71
7	64	64
8	141	147
9	79	72
10	76	73
11	36	30
12	42	-48
13	21*	22
14	21*	18
15	2*	0
-1	572	560
-2	417	-407
-3	826	836
-4	113	112
-5	810	815
-6	252	249
-7	170	164
-8	115	121
-9	35	-32
-10	97	96
-11	0*	-1
-12	175	-173
-13	88	88
-14	33*	-29
-15	15*	-7
-16	0*	-12
-17	19*	-6
-18	24*	-17
3,1,L		
0	394	-378
1	775	-781
2	159	153
3	49	48
4	149	142
5	184	177
6	139	-139
7	129	-130
8	50	-41
9	50	51
10	28*	22
11	48	-47
12	22*	-6
13	0*	11
14	16*	-11
-1	1123	1138
-2	238	234
-3	70	-58
-4	507	-505
-5	518	-525
-6	221	208
-7	339	324
-8	118	115
-9	43	37
-10	167	-171
-11	136	-134
-12	74	78
-13	197	199
-14	15*	-36
-15	22*	-29
-16	24*	-8
-17	24*	-22
-18	13*	11
4,1,L		
0	91	-97
1	48	52
2	62	-62
3	98	-97
4	135	-124
5	38	36
6	29	-25
7	37	-33
8	37	-40
9	0*	3
10	28*	-22
11	8*	-9
12	8*	9
13	0*	5
-1	143	-135
-2	253	254
-3	41	48
-4	128	-117
-5	134	-135
-6	434	438
-7	230	-221
-8	121	131
-9	32	35
-10	68	-55
-11	32	42
-12	192	198
-13	110	-105
-14	25*	-30
-15	31*	-27
-16	37	45
-17	35	-28
-18	18*	-2
-19	8*	-17
5,1,L		
0	98	-103
1	267	-265
2	113	112
3	162	156
4	34	-29
5	115	-121
6	55	-47
7	20*	-2
8	0*	4
9	0*	4
10	21*	10
11	9*	4
12	25*	-17
-1	54	-55
-2	75	79
-3	324	328
-4	127	131
-5	0*	19
-6	92	-76
-7	200	-187
-8	356	353
-9	100	98
-10	32	-33
-11	0*	-2
-12	29	-31
-13	77	-79
-14	31	27
-15	28*	7
-16	20*	3
-17	5*	2
-18	24*	-4
-19	38	-34
6,1,L		
0	241	-235
1	11*	9
2	72	74
3	82	-81
4	18*	16
5	102	-102
6	60	-56
7	17*	-4
8	10*	23
9	20*	0
10	31*	-18
11	0*	4
-1	79	71
-2	139	-139
-3	107	98
-4	96	-96
-5	16*	18
-6	193	-188
-7	247	243
-8	32	22
-9	94	93
-10	232	-230
-11	38	-40
-12	169	166
-13	21*	19
-14	51	-60
-15	99	91
-16	83	-83
-17	3*	15
-18	0*	1
-19	27*	19
-20	12*	-14
7,1,L		
0	37	-34
1	124	129
2	48	45
3	41	38
4	38	-24
5	79	-69
6	13*	-2
7	57	57
8	2*	9
9	0*	-9
-1	224	-220
-2	31	-31
-3	137	132
-4	149	147
-5	27	21
-6	325	-334
-7	12*	-4
-8	34	24
-9	118	117
-10	125	127
-11	187	-187
-12	82	-76
-13	161	170
-14	29	-10
-15	44	33
-16	0*	6
-17	55	-53
-18	13*	-8
-19	54	47
-20	0*	1
8,1,L		
0	54	61
1	0*	0
2	44	-34
3	4*	0
4	98	-93
5	0*	17
6	59	68
7	8*	-8
8	29*	-25
-1	54	53
-2	101	106
-3	26	25
-4	132	132
-5	92	-90
-6	77	-77
-7	86	-77
-8	55	58
-9	77	-67
-10	11*	1
-11	43	-32
-12	138	-128
-13	131	-124
-14	102	109
-15	0*	7
-16	11*	-27
-17	27*	-20
-18	41	33
-19	25*	-22
-20	28*	17
9,1,L		
0	36	31
1	29*	30
2	0*	19
3	45	-45
4	0*	-10
5	32*	40
6	15*	-1
7	19*	-18
-1	21*	15
-2	44	-44
-3	93	-92
-4	69	68
-5	180	188
-6	0*	18
-7	167	-162
-8	52	-48
-9	11*	13
-10	55	58
-11	65	63
-12	42	37
-13	160	-157
-14	77	-82
-15	52	58
-16	43	40
-17	25*	20
-18	2*	-10
-19	57	-55
-20	16*	13
10,1,L		
0	46	57
1	24*	-26
2	15*	-19
3	0*	8
4	24*	32
5	14*	0
-1	29*	-31
-2	10*	-2
-3	18*	-21
-4	39	33
-5	33	-36
-6	7*	1
-7	121	116
-8	80	-80
-9	48	41
-10	24*	27
-11	30	20
-12	74	71
-13	0*	-9
-14	46	-51
-15	14*	1
-16	23*	7
-17	0*	-2
-18	45	-35
-19	27*	15
-20	9*	-11
11,1,L		
0	52	48
1	17*	14
2	26*	-22
3	11*	8
-1	0*	-5
-2	35	-35
-3	47	-43
-4	19*	-11
-5	68	62
-6	35	34
-7	0*	11
-8	27*	-27
-9	79	-80
-10	13*	-25
-11	105	108
-12	29*	31
-13	39	-40
-14	14*	-4
-15	0*	-3
-16	16*	0
-17	20*	-2
-18	21*	-10
-19	44	35
-20	29*	-12
12,1,L		
0	0*	-14
1	0*	3
-1	8*	5
-2	7*	-25
-3	15*	-17
-4	49	42
-5	29*	24
-6	98	-96
-7	0*	-22
-8	50	48
-9	54	-53
-10	0*	40
-11	4*	5
-12	77	-73
-13	17*	16
-14	24*	31
-15	0*	13
-16	16*	8
-17	12*	1
-18	13*	6
-19	14*	-6
13,1,L		
-1	30*	30
-2	10*	22
-3	0*	-10
-4	28*	-24
-5	47	-52
-6	14*	12
-7	78	82
-8	12*	8
-9	48	-49
-10	29*	-27
-11	15*	-13
-12	34	26
-13	45	40
-14	0*	4
-15	42	-43
-16	17*	-20
-17	26*	30
-18	5*	18
14,1,L		
-4	9*	-13
-5	20*	-1
-6	46	53
-7	9*	0
-8	47	-49
-9	0*	-9
-10	21*	19
-11	26*	23
-12	11*	-11
-13	7*	-2
-14	8*	-10
-15	28*	-24
-16	8*	11
-17	18*	-10
15,1,L		
-8	10*	5
-9	25*	31
-10	13*	-19
-11	0*	-9
-12	16*	8
-13	25*	-11
-14	0*	4
-15	0*	14
0,2,L		
0	545	-544
1	62	60
2	638	-656
3	600	-634
4	336	-341
5	198	205
6	146	146
7	41	37
8	106	-111
9	0*	3
10	0*	4
11	199	199
12	36*	18
13	37	-38
14	7*	-3
15	18*	7
16	0*	11
1,2,L		
0	213	209
1	748	-743
2	312	315
3	19	23
4	114	113
5	38	-44
6	42	-44
7	18*	-12
8	15*	-15
9	0*	4
10	19*	18
11	141	-144
12	18*	30
13	0*	11
14	37	-32
15	0*	6
-1	327	-321
-2	52	-56
-3	58	-61
-4	57	-64
-5	146	152
-6	234	-221
-7	69	-68
-8	198	-193
-9	50	-39
-10	0*	2
-11	168	179
-12	76	-74
-13	112	-115
-14	34*	34
-15	33	38
-16	13*	-7
-17	46	34
2,2,L		
0	214	214
1	44	55
2	168	-169
3	15*	-12
4	192	196
5	48	46
6	0*	7
7	213	-208
8	48	-50
9	23*	-22
10	13*	16
11	67	-75
12	33	-31
13	14*	-13
14	15*	-6
15	0*	2
-1	34	-35
-2	639	637
-3	567	-577
-4	51	38
-5	13*	-21
-6	381	-371
-7	195	200
-8	64	64
-9	285	-283
-10	48	62
-11	131	-123
-12	32*	42
-13	96	94
-14	12*	-4
-15	0*	-12
-16	20*	21
-17	7*	5
-18	30*	21
3,2,L		
0	73	-68
1	385	-381
2	129	128
3	305	302
4	13*	5
5	51	-52
6	172	-175
7	26*	-23
8	57	-56
9	0*	4
10	32*	16
11	0*	1
12	42	-37
13	21*	-9
14	0*	-1
-1	561	-561
-2	23	-23
-3	413	-416
-4	300	-293
-5	105	-108
-6	507	488
-7	89	-82
-8	233	-224
-9	31	-23
-10	26	35
-11	41	31
-12	80	76
-13	42	34
-14	49	43
-15	57	-67
-16	0*	1
-17	11*	17
-18	11*	15
4,2,L		
0	193	-206
1	401	-400
2	107	-106
3	116	114
4	0*	17
5	50	-53
6	151	-153
7	101	-96
8	0*	3
9	45	45
10	28*	-31
11	7*	-5
12	10*	-13
13	25*	-4
-1	357	361
-2	107	118
-3	182	191
-4	240	-277
-5	137	-135
-6	182	175
-7	232	227
-8	88	93
-9	3*	-13
-10	0*	-22
-11	0*	6
-12	0*	2
-13	71	75
-14	0*	2
-15	0*	-5
-16	15*	-13
-17	17*	4
-18	27*	23
-19	8*	-3
5,2,L		
0	199	198
1	309	303
2	66	-65
3	87	83
4	17*	14
5	174	-173
6	28*	31
7	137	134
8	43	43
9	29*	-34
10	8*	-2
11	0*	-15
12	15*	-1
-1	657	663
-2	78	78
-3	67	-57
-4	223	-224
-5	171	-175
-6	190	193
-7	47	42
-8	0*	-9
-9	19*	-13
-10	147	-154
-11	11*	-4
-12	61	-68
-13	53	47
-14	22*	21
-15	28*	-24
-16	15*	11
-17	23*	16
-18	0*	-7
-19	2*	-1
6,2,L		
0	136	-136
1	97	92
2	0*	73
3	95	96
4	84	-85
5	127	-121
6	65	74
7	34*	35
8	19*	-4
9	0*	1
10	9*	-15
-1	324	-323
-2	95	112
-3	286	281
-4	135	136
-5	60	-64
-6	80	-82
-7	27	-23
-8	148	146
-9	252	253
-10	0*	1
-11	16*	-16
-12	51	-47
-13	29	-26
-14	56	55
-15	0*	0
-16	0*	11
-17	25*	-14
-18	90	-70
-19	22*	24
7,2,L		
0	47	49
1	78	76
2	12*	4
3	65	-62
4	0*	3
5	97	101
6	0*	-1
7	17*	-34
8	6*	10
9	16*	-2
-1	73	-70
-2	42	-48
-3	111	107
-4	10*	-11
-5	129	-132
-6	129	126
-7	0*	11
-8	45	45
-9	121	126
-10	4*	28
-11	18*	32
-12	20*	25
-13	40	37
-14	37	32
-15	19*	-13
-16	10*	-22
-17	10*	-7
-18	24*	14
-19	22*	-6
-20	26*	-23
8,2,L		
0	74	82
1	43	42
2	53	46
3	30*	-22
4	8*	7
5	27*	28
6	13*	-14
7	0*	5
8	0*	-2
-1	36	-32
-2	120	-119
-3	39	42
-4	53	60
-5	41	41
-6	43	-43
-7	215	-206
-8	25	-28
-9	78	72
-10	39	48
-11	80	-79
-12	36	35
-13	19*	29
-14	0*	11
-15	46	38
-16	15*	7
-17	0*	-2
-18	69	57
-19	0*	-12
-20	24*	-5
9,2,L		
0	41	41
1	15*	13
2	20*	-19
3	12*	8
4	8*	-12
5	4*	-22
6	15*	15
-1	3*	1
-2	43	49
-3	57	64
-4	8*	7
-5	92	-88
-6	75	-80
-7	39	35
-8	19*	3
-9	68	55
-10	86	84
-11	225	-234
-12	60	-64
-13	153	161
-14	21*	-8
-15	0*	-24
-16	21*	33
-17	0*	-14
-18	17*	-8
-19	17*	5
-20	42	35
10,2,L		
0	40	40
1	0*	9
2	13*	-14
3	24*	-16
4	0*	-8
5	0*	-1
-1	43	41
-2	41	-41
-3	30*	-35
-4	12*	-19
-5	121	121
-6	172	170
-7	102	-98
-8	99	-99
-9	45	38
-10	20*	-13
-11	136	134
-12	40	-37
-13	143	-136
-14	22*	-22
-15	0*	16
-16	0*	5
-17	0*	12
-18	46	-42
-19	7*	-13
-20	0*	4
11,2,L		
0	14*	14
1	29*	-24
2	0*	-18
3	0*	-4
-1	37	-32
-2	0*	1
-3	20*	-13
-4	18*	16
-5	125	133
-6	1*	-13
-7	159	-161
-8	12*	8
-9	77	80
-10	0*	11
-11	46	49
-12	17*	19
-13	82	-83
-14	74	-73
-15	63	72
-16	29*	29
-17	0*	-11
-18	18*	-6
-19	16*	-21
12,2,L		
0	16*	-18
1	0*	1
-1	0*	3
-2	40	32
-3	27*	-31
-4	17*	-16
-5	20*	-26
-6	19*	-26
-7	83	88
-8	14*	22
-9	69	-64
-10	18*	-19
-11	21*	-17
-12	91	85
-13	33	42
-14	28*	-26
-15	14*	-27
-16	0*	0
-17	4*	12
-18	17*	13
-19	0*	-1
13,2,L		
-2	18*	-19
-3	14*	-17
-4	0*	16
-5	0*	0
-6	0*	-9
-7	40	46
-8	20*	0
-9	51	-58
-10	19*	9
-11	26*	18
-12	16*	2
-13	15*	13
-14	0*	8
-15	17*	-28
-16	18*	0
-17	23*	-3
-18	12*	-4
14,2,L		
-5	24*	-5
-6	11*	-2
-7	0*	3
-8	0*	7
-9	0*	-2
-10	13*	-14
-11	0*	-17
-12	0*	1
-13	28*	24
-14	5*	-7
-15	17*	0
-16	25*	-14
15,2,L		
-9	18*	0
-10	9*	-6
-11	22*	13
-12	2*	6
-13	16*	-8
0,3,L		
1	235	244
2	458	-468
3	175	186
4	151	152
5	285	285
6	203	198
7	269	-270
8	10*	1
9	52	49
10	37	-32
11	73	98
12	12*	-19
13	58	-63
14	42	-42
15	0*	3
16	26*	-24
1,3,L		
0	505	500
1	18	20
2	245	-244
3	85	81
4	255	251
5	87	88
6	93	89
7	16*	1
8	170	-170
9	0*	17
10	20*	19
11	35*	-46
12	45	48
13	14*	-21
14	40	-37
15	21*	-19
-1	359	355
-2	25	-23
-3	126	-119
-4	42	-43
-5	160	158
-6	46	-46
-7	94	104
-8	61	-60
-9	73	-74
-10	13*	22
-11	13*	-5
-12	46	-44
-13	1*	-2
-14	41	-36
-15	0*	12
-16	5*	4
2,3,L		
0	369	-347
1	159	-148
2	117	107
3	200	203
4	128	122
5	92	-84
6	243	-239
7	145	-145
8	38	40
9	99	101
10	11*	-3
11	84	-82
12	50	-45
13	22*	24
14	21*	13
-1	204	-203
-2	445	-441
-3	343	-347
-4	10*	8
-5	19	22
-6	9*	-3
-7	90	-89
-8	83	-86
-9	131	-133
-10	28	37
-11	61	58
-12	0*	-17
-13	29*	-37
-14	15*	13
-15	22*	-16
-16	30*	28
-17	0*	4
3,3,L		
0	151	-132
1	43	43
2	236	-230
3	85	87
4	290	283
5	107	-105
6	144	-145
7	80	79
8	84	93
9	3*	1
10	83	74
11	15*	13
12	39	-47
13	11*	13
-1	268	-253
-2	424	422
-3	0*	-2
-4	55	-56
-5	195	-191
-6	43	47
-7	173	167
-8	91	96
-9	141	-139
-10	80	-83
-11	18*	27
-12	76	86
-13	80	-89
-14	19*	-3
-15	23*	-22
-16	36	-24
-17	0*	-4
-18	46	38
4,3,L		
0	109	-119
1	19*	15
2	163	154
3	79	83
4	90	-94
5	151	-153
6	141	146
7	72	73
8	0*	1
9	14*	13
10	31*	-24
11	22*	20
12	35	28
-1	123	130
-2	61	65
-3	244	-234
-4	196	-204
-5	249	238
-6	195	-199
-7	115	111
-8	6*	4
-9	166	-159
-10	19*	-16
-11	37	-38
-12	71	78
-13	98	102
-14	91	-98
-15	0*	2
-16	24*	-14
-17	16*	20
-18	42	36
5,3,L		
0	79	73
1	100	96
2	0*	0
3	87	82
4	36	-42
5	103	98
6	35	32
7	9*	-21
8	52	46
9	6*	10
10	26*	-33
11	21*	12
-1	188	-193
-2	32	23
-3	167	160
-4	285	-289
-5	62	-67
-6	204	-209
-7	53	-51
-8	23	-25
-9	17*	-16
-10	141	-141
-11	9*	2
-12	56	-53
-13	70	71
-14	52	54
-15	0*	0
-16	0*	-18
-17	41	44
-18	0*	-19
-19	34*	25
6,3,L		
0	36	-39
1	190	195
2	101	102
3	30*	-36
4	0*	-1
5	0*	17
6	37	-24
7	46	41
8	19*	10
9	0*	-7
10	29*	12
-1	186	190
-2	106	98
-3	94	-91
-4	61	-64
-5	107	-104
-6	103	101
-7	231	223
-8	37	42
-9	167	-167
-10	99	-105
-11	37	-36
-12	25*	31
-13	38	-49
-14	19*	2
-15	0*	3
-16	0*	-13
-17	37	-30
-18	0*	5
-19	37	20
7,3,L		
0	110	-120
1	19*	9
2	93	92
3	18*	-15
4	34*	-19
5	32*	-39
6	0*	-15
7	0*	3
8	14*	12
9	8*	5
-1	54	-50
-2	25*	33
-3	18*	18
-4	180	190
-5	15*	22
-6	94	-85
-7	123	-129
-8	176	179
-9	16*	12
-10	44	44
-11	33	34
-12	125	-135
-13	98	-106
-14	73	79
-15	17*	-13
-16	0*	-6
-17	57	-57
-18	12*	6
-19	13*	-6
8,3,L		
0	140	136
1	28*	-27
2	9*	-5
3	53	-56
4	20*	-25
5	34	26
6	20*	19
7	0*	-5
-1	154	-159
-2	103	110
-3	126	127
-4	50	59
-5	159	-169
-6	275	-279
-7	152	152
-8	131	131
-9	71	70
-10	58	55
-11	37	-34
-12	30	-33
-13	40	43
-14	93	92
-15	19*	13
-16	5*	-25
-18	38	-32
-19	0*	-3
9,3,L		
0	76	77
1	12*	7
2	21*	-28
3	20*	-16
4	35	-32
5	16*	8
6	30*	23
-1	0*	2
-2	156	-158
-3	20*	0
-4	24*	27
-5	78	-80
-6	108	110
-7	80	76
-8	222	-228
-9	15*	11
-10	48	49
-11	60	57
-12	38	42
-13	14*	-4
-14	17*	-4
-15	20*	20
-16	0*	-8
-17	53	52
-18	8*	5
-19	22*	-8
10,3,L		
0	28*	-31
1	0*	-14
2	0*	-19
3	13*	7
4	16*	9
-1	4*	-16
-2	50	-54
-3	47	48
-4	28*	30
-5	0*	17
-6	49	46
-7	122	-116
-8	67	-67
-9	62	62
-10	15*	22
-11	48	-43
-12	46	-38
-13	16*	0
-14	16*	-21
-15	21*	13
-16	9*	16
-17	15*	-12
-18	41	28
-19	32*	16
11,3,L		
0	3*	-2
1	7*	2
2	0*	-6
-1	0*	6
-2	15*	23
-3	22*	-15
-4	22*	-26
-5	11*	8
-6	24*	22
-7	6*	-14
-8	9*	-13
-9	12*	-5
-10	53	52
-11	36	-34
-12	35	-42
-13	15*	24
-14	5*	6
-15	22*	-19
-16	4*	16
-17	12*	1
-18	25*	-19
-19	18*	-13
12,3,L		
0	21*	9
-1	15*	-6
-2	8*	-5
-3	25*	-8
-4	0*	17
-5	30*	33
-6	18*	9
-7	43	-41
-8	16*	-6
-9	0*	13
-10	17*	24
-11	88	84
-12	40	-46
-13	90	-89
-14	49	49
-15	24*	17
-16	16*	15
-17	11*	-5
-18	49	-41
13,3,L		
-3	19*	-5
-4	22*	-17
-5	20*	-11
-6	43	31
-7	32*	24
-8	7*	-18
-9	21*	-4
-10	29*	-29
-11	9*	-11
-12	51	58
-13	23*	15
-14	53	-55
-15	20*	6
-16	9*	2
-17	25*	-22
14,3,L		
-6	9*	19
-7	20*	19
-8	17*	-26
-9	30*	-24
-10	0*	-1
-11	0*	2
-12	45	44
-13	28*	21
-14	40	-38
-15	26*	-21
0,4,L		
0	63	-66
1	106	104
2	150	150
3	63	-71
4	0*	13
5	238	243
6	0*	18
7	225	-232
8	0*	3
9	67	73
10	31*	17
11	22*	21
12	29*	-31
13	31*	-25
14	13*	4
15	26*	3
1,4,L		
0	499	-488
1	111	-111
2	138	-140
3	184	183
4	211	212
5	58	-60
6	245	-243
7	56	55
8	48	50
9	70	71
10	45	32
11	15*	10
12	0*	-12
13	20*	29
14	24*	17
-1	98	-92
-2	344	-337
-3	163	-164
-4	349	355
-5	133	134
-6	112	116
-7	67	-69
-8	163	-167
-9	0*	20
-10	144	143
-11	0*	-8
-12	57	49
-13	49	-48
-14	24*	-14
-15	0*	1
-16	0*	8
2,4,L		
0	77	-93
1	167	-161
2	0*	-3
3	50	44
4	0*	11
5	208	-220
6	70	74
7	112	113
8	68	-67
9	72	70
10	26*	21
11	26*	-34
12	36	28
13	35	39
-1	279	275
-2	57	-58
-3	80	-80
-4	86	-86
-5	27	32
-6	261	257
-7	15*	-12
-8	0*	-2
-9	34	-40
-10	18*	-30
-11	72	70
-12	14*	10
-13	0*	5
-14	0*	-15
-15	27*	-6
-16	0*	-4
3,4,L		
0	44	-45
1	155	-162
2	128	126
3	128	111
4	35	-41
5	72	60
6	25*	-19
7	60	-58
8	135	126
9	0*	1
10	21*	16
11	1*	15
12	24*	-18
-1	389	377
-2	74	-72
-3	108	-119
-4	97	92
-5	186	178
-6	204	191
-7	145	138
-8	162	-153
-9	23*	-25
-10	15*	18
-11	23*	23
-12	24*	-34
-13	19*	2
-14	49	-40
-15	24*	-10
-16	4*	3
-17	46	-30
4,4,L		
0	103	-111
1	69	-66
2	78	79
3	50	43
4	43	41
5	46	-53
6	18*	-32
7	32*	-10
8	21*	23
9	21*	-12
10	21*	10
11	34*	31
-1	23	18
-2	64	56
-3	96	95
-4	0*	-42
-5	103	-108
-6	34	-42
-7	87	86
-8	67	74
-9	30	33
-10	17*	-6
-11	22*	-21
-12	9*	-11
-13	47	36
-14	10*	0
-15	26*	-39
-16	17*	-15
-17	38	38
5,4,L		
0	15*	-20
1	102	99
2	121	127
3	7*	10
4	143	-137
5	59	-60
6	3*	-7
7	0*	14
8	23*	13
9	0*	0
10	43	-44
-1	138	130
-2	151	156
-3	156	-149
-4	219	-222
-5	174	-168
-6	173	181
-7	180	182
-8	17*	7
-9	34	-31
-10	106	-108
-11	17*	19
-12	192	200
-13	24*	27
-14	31*	-38
-15	32	-17
-16	11*	-5
-17	0*	15
-18	17*	19
6,4,L		
0	55	61
1	35	41
2	64	65
3	9*	-12
4	40	-40
5	40	-49
6	0*	-7
7	16*	26
8	13*	0
9	8*	7
-1	63	-64
-2	43	-44
-3	25	25
-4	22*	-29
-5	184	189
-6	0*	-7
-7	297	-309
-8	47	44
-9	40	44
-10	45	-51
-11	85	-85
-12	36	37
-13	40	41
-14	37	-42
-15	0*	14
-16	32*	28
-17	13*	-17
-18	33*	33
7,4,L		
0	11*	3
1	32*	32
2	13*	-14
3	66	-68
4	37	-37
5	0*	0
6	30*	20
7	0*	4
8	21*	-2
-1	24*	-17
-2	135	137
-3	108	107
-4	97	-99
-5	59	54
-6	157	-159
-7	95	-101
-8	220	220
-9	46	-45
-10	109	-108
-11	74	-75
-12	118	-115
-13	31	19
-14	48	56
-15	25*	-22
-16	21*	-19
-17	23*	-16
-18	0*	1
8,4,L		
0	4*	-11
1	43	-53
2	6*	-3
3	0*	-10
4	7*	-16
5	20*	8
6	14*	15
-1	92	102
-2	42	-43
-3	77	-77
-4	86	78
-5	11*	-13
-6	62	-58
-7	135	136
-8	18*	31
-9	2*	-9
-10	0*	-6
-11	0*	2
-12	0*	-10
-13	37	-37
-14	26*	-8
-15	27*	25
-16	34	-35
-17	34*	-36
-18	43	-31
9,4,L		
0	72	82
1	10*	14
2	43	-39
3	26*	-21
4	16*	-2
5	23*	23
-1	24*	-21
-2	46	40
-3	51	54
-4	49	47
-5	23*	-25
-6	107	-98
-7	9*	-13
-8	107	104
-9	49	55
-10	25*	-20
-11	20*	21
-12	54	-48
-13	13*	8
-14	29*	26
-15	19*	16
-16	8*	-6
-17	27*	-31
-18	14*	-5
10,4,L		
0	38	36
1	28*	33
2	18*	-21
3	6*	-8
-1	40	-57
-2	5*	-7
-3	8*	2
-4	8*	-10
-5	38	29
-6	17*	-20
-7	40	-33
-8	26*	-25
-9	30*	-25
-10	10*	22
-11	17*	12
-12	45	45
-13	22*	-29
-14	11*	-21
-15	20*	18
-16	8*	14
-17	44	46
-18	14*	13
11,4,L		
0	26*	-28
1	24*	-14
-1	40	-39
-2	61	-66
-3	22*	8
-4	49	59
-5	28*	25
-6	26*	-21
-7	54	-54
-8	36	-38
-9	17*	17
-10	38	48
-11	23*	25
-12	23*	-4
-13	76	-75
-14	19*	7
-15	6*	16
-16	19*	14
-17	39	31
12,4,L		
-1	37	42
-2	0*	13
-3	20*	-26
-4	0*	-10
-5	0*	-4
-6	23*	2
-7	37	34
-8	29*	12
-9	7*	-19
-10	16*	-9
-11	32*	-24
-12	23*	-12
-13	53	51
-14	15*	-13
-15	15*	-17
-16	15*	20
-17	48	-44
13,4,L		
-4	0*	7
-5	18*	19
-6	0*	6
-7	19*	-5
-8	28*	-25
-9	29*	-13
-10	0*	15
-11	22*	11
-12	19*	-9
-13	15*	6
-14	14*	-10
-15	9*	7
14,4,L		
-9	0*	-5
-10	0*	-15
-11	35	32
-12	10*	7
0,5,L		
1	185	-188
2	142	-148
3	109	107
4	118	121
5	146	-143
6	25*	-21
7	3*	4
8	99	-93
9	73	72
10	24*	32
11	46	-38
12	9*	7
13	15*	15
14	0*	12
1,5,L		
0	26*	-22
1	122	-116
2	27	-15
3	45	44
4	112	-113
5	102	102
6	37	31
7	102	-102
8	57	59

Table 2 (*cont.*)

	1,5,L	
9	0*	6
10	0*	0
11	34	25
12	16*	-6
13	0*	-1
-1	103	-99
-2	12*	-12
-3	83	-79
-4	36	37
-5	28	-32
-6	154	-155
-7	18*	19
-8	74	69
-9	22*	-24
-10	24*	24
-11	29*	13
-12	0*	17
-13	0*	-7
-14	22*	17
-15	24*	19
	2,5,L	
0	198	-190
1	249	-248
2	31	28
3	202	196
4	109	109
5	81	-80
6	58	-64
7	0*	-7
8	24*	17
9	43	32
10	6*	8
11	0*	-4
12	33*	-24
-1	100	-101
-2	131	-134
-3	168	-173
-4	19*	-17
-5	369	363
-6	95	94
-7	270	-275
-8	71	-75
-9	3*	4
-10	47	51
-11	113	113
-12	16*	-22
-13	42	-53
-14	0*	-1
-15	30*	20
	3,5,L	
0	70	73
1	0*	2
2	41	42
3	64	63
4	27*	-22
5	22*	-6
6	98	-102
7	31*	-16
8	27*	33
9	0*	-7
10	19*	-10
11	41	-34
-1	7*	-15
-2	95	-104
-3	80	76
-4	76	81
-5	35	34
-6	70	-77
-7	46	50
-8	118	-120
-9	47	-44
-10	38	44
-11	17*	14
-12	14*	-7
-13	0*	-10
-14	24*	-7
-15	17*	19
-16	15*	-21
	4,5,L	
0	7*	-3
1	103	103
2	131	134
3	25*	22
4	58	-56
5	58	-53
6	43	-47
7	30*	24
8	0*	23
9	16*	-8
10	22*	-24
-1	236	240
-2	65	69
-3	166	-173
-4	100	-103
-5	120	-120
-6	155	158
-7	300	302
-8	53	-54
-9	157	-167
-10	26*	-20
-11	29*	23
-12	29*	17
-13	53	52
-14	14*	-23
-15	32*	-35
-16	19*	-15
	5,5,L	
0	54	-38
1	10*	27
2	14*	7
3	54	-46
4	56	-62
5	47	-49
6	28*	40
7	21*	-6
8	20*	11
9	10*	-8
-1	0*	-6
-2	0*	-3
-3	21*	3
-4	68	72
-5	137	-133
-6	227	225
-7	115	114
-8	63	-67
-9	57	53
-10	50	41
-11	19*	-13
-12	54	49
-13	0*	0
-14	0*	4
-15	44	-42
-16	0*	12
-17	34*	-28
	6,5,L	
0	38	29
1	31*	27
2	38	37
3	35	-29
4	51	-51
5	51	-41
6	0*	23
7	63	58
8	0*	-10
-1	20*	-20
-2	140	142
-3	54	52
-4	149	-153
-5	68	-75
-6	27*	25
-7	25*	25
-8	58	64
-9	24*	-2
-10	19*	-11
-11	14*	8
-12	41	40
-13	41	36
-14	33	31
-15	9*	-15
-16	0*	-3
-17	51	51
	7,5,L	
0	28	-25
1	70	73
2	0*	-7
3	22*	-17
4	12*	-2
5	0*	14
6	30*	31
7	21*	2
-1	28*	-23
-2	15*	3
-3	30	-35
-4	0*	11
-5	0*	11
-6	65	-74
-7	14*	15
-8	49	-45
-9	44	39
-10	17*	-16
-11	25*	-20
-12	37	36
-13	0*	15
-14	0*	-6
-15	44	38
-16	19*	3
-17	28*	22
	8,5,L	
0	73	70
1	30*	25
2	21*	-33
3	49	-44
4	11*	7
5	57	53
-1	17*	-10
-2	44	50
-3	93	94
-4	18*	1
-5	44	-44
-6	27*	-28
-7	33	-39
-8	64	62
-9	105	98
-10	30*	-42
-11	113	-114
-12	74	-66
-13	69	76
-14	0*	13
-15	0*	-15
-16	0*	-8
-17	59	-48
	9,5,L	
0	43	54
1	7*	-5
2	16*	-25
3	0*	-8
-1	33*	41
-2	0*	-9
-3	17*	-16
-4	32	-36
-5	26*	19
-6	80	85
-7	22*	-19
-8	0*	-9
-9	27*	-22
-10	18*	14
-11	10*	-13
-12	27*	-31
-13	28*	20
-14	0*	10
-15	18*	-18
-16	29*	-24
-17	39	-33
	10,5,L	
0	23*	26
1	0*	-5
2	16*	-25
-1	32*	-19
-2	22*	-20
-3	25*	20
-4	41	53
-5	0*	-16
-6	9*	-24
-7	35	-34
-8	0*	-7
-9	52	50
-10	26*	29
-11	28*	14
-12	11*	3
-13	66	-70
-14	27*	26
-15	38	38
-16	20*	-7
-17	25*	18
	11,5,L	
-1	8*	1
-2	55	58
-3	0*	3
-4	28*	-43
-5	0*	1
-6	0*	0
-7	9*	0
-8	23*	33
-9	12*	4
-10	22*	-15
-11	24*	18
-12	27*	-27
-13	6*	0
-14	11*	12
-15	5*	-18
-16	30*	29
	12,5,L	
-3	0*	-1
-4	12*	3
-5	61	58
-6	24*	18
-7	67	-67
-8	32*	-39
-9	25*	27
-10	34	31
-11	19*	-1
-12	0*	-7
-13	9*	1
-14	35	-30
-15	8*	0
	13,5,L	
-8	24*	25
-9	0*	6
-10	0*	-13
-11	0*	-9
-12	24*	19
	0,6,L	
0	58	-55
1	44	-48
2	19*	17
3	93	100
4	64	68
5	119	-109
6	0*	16
7	0*	2
8	30*	-20
9	38	35
10	16*	5
11	19*	-11
12	22*	-10
13	22*	-23
	1,6,L	
0	91	-89
1	135	-131
2	43	41
3	125	127
4	134	134
5	13*	23
6	141	-138
7	78	-77
8	41	50
9	18*	18
10	43	43
11	48	-48
12	58	-57
-1	52	-51
-2	175	-179
-3	51	-51
-4	53	-54
-5	167	173
-6	138	140
-7	124	-124
-8	73	-68
-9	27*	33
-10	0*	1
-11	47	38
-12	33*	18
-13	29*	-21
-14	45	-37
	2,6,L	
0	0*	-15
1	47	50
2	55	60
3	13*	20
4	14*	6
5	0*	-15
6	55	-55
7	9*	18
8	16*	11
9	2*	14
10	0*	-9
11	31*	-25
-1	38	-50
-2	69	-75
-3	68	69
-4	28	33
-5	0*	9
-6	22*	-27
-7	65	-58
-8	37	-27
-9	50	47
-10	16*	5
-11	10*	10
-12	12*	-4
-13	0*	-6
-14	15*	-1
	3,6,L	
0	82	-79
1	8*	15
2	57	45
3	47	54
4	36	-29
5	90	-91
6	49	-36
7	0*	11
8	35	44
9	11*	15
10	39	-47
-1	41	39
-2	14*	-22
-3	212	-209
-4	16*	29
-5	98	103
-6	185	-182
-7	19*	-11
-8	56	57
-9	93	-92
-10	16*	16
-11	31	33
-12	0*	-1
-13	0*	-13
-14	24*	14
-15	20*	20
	4,6,L	
0	28	31
1	18*	16
2	57	56
3	34	-40
4	27*	-19
5	30*	-33
6	26*	-25
7	64	65
8	27*	21
9	27*	-29
-1	30	-22
-2	68	-74
-3	22*	-16
-4	6*	28
-5	60	54
-6	58	53
-7	117	-109
-8	34	-43
-9	16*	-14
-10	0*	-11
-11	27*	23
-12	7*	-3
-13	18*	-25
-14	0*	-8
-15	10*	2
	5,6,L	
0	110	-119
1	131	133
2	82	79
3	25*	-17
4	72	-74
5	18*	-24
6	90	90
7	11*	17
8	19*	-13
-1	133	135
-2	9*	-22
-3	20*	18
-4	124	-121
-5	44	-42
-6	165	162
-7	84	87
-8	45	-51
-9	104	-107
-10	60	-59
-11	8*	9
-12	51	52
-13	13*	-5
-14	39	-39
-15	37	-30
	6,6,L	
0	18*	19
1	23*	30
2	0*	-1
3	33	-30
4	10*	-26
5	39	36
6	0*	21
7	22*	0
-1	0*	12
-2	31*	-29
-3	66	-64
-4	0*	3
-5	0*	23
-6	21*	-19
-7	94	95
-8	17*	8
-9	81	-80
-10	0*	1
-11	49	42
-12	26*	16
-13	21*	13
-14	22*	-17
-15	15*	-14
-16	0*	-16
	7,6,L	
0	96	100
1	49	46
2	19*	-25
3	26*	-33
4	16*	26
5	0*	0
-1	24*	-22
-2	79	84
-3	65	64
-4	0*	-9
-5	124	-120
-6	67	-66
-7	74	81
-8	76	81
-9	47	52
-10	33	-38
-11	10*	-12
-12	0*	-12
-13	31*	27
-14	57	53
-15	9*	-6
-16	0*	21
	8,6,L	
0	46	50
1	0*	2
2	17*	-33
3	0*	-15
-1	21*	10
-2	4*	0
-3	22*	-30
-4	14*	-16
-5	0*	11
-6	10*	-17
-7	21*	22
-8	13*	17
-9	42	-41
-10	15*	5
-11	34	-16
-12	0*	-4
-13	29*	28
-14	8*	5
-15	22*	7
	9,6,L	
0	27*	12
1	26*	-18
2	11*	-27
-1	61	-55
-2	26*	-25
-3	43	39
-4	92	84
-5	22*	-22
-6	98	-98
-7	17*	-15
-8	27*	23
-9	34	33
-10	6*	7
-11	57	-57
-12	38	31
-13	0*	-12
-14	9*	-12
-15	39	35
	10,6,L	
-1	26*	20
-2	25*	17
-3	0*	10
-4	10*	-21
-5	45	-41
-6	17*	16
-7	49	42
-8	21*	2
-9	12*	-9
-10	16*	-2
-11	13*	1
-12	14*	-17
-13	6*	10
-14	23*	24
	11,6,L	
-3	0*	2
-4	24*	19
-5	15*	7
-		
-7	0*	-10
-8	68	-72
-9	3*	4
-10	53	57
-11	23*	24
-12	61	-58
-13	0*	-20
	12,6,L	
-8	19*	8
-9	0*	17
-10	0*	-7
	0,7,L	
1	20*	-27
2	36	-38
3	36	30
4	81	78
5	36	-35
6	11*	-21
7	8*	3
8	29*	-34
9	32*	39
10	23*	26
11	13*	-33
	1,7,L	
0	47	-48
1	0*	10
2	57	48
3	57	58
4	39	36
5	74	-84
6	105	-104
7	0*	12
8	59	62
9	30*	23
10	8*	-16
-1	62	-66
-2	30*	31
-3	105	108
-4	26*	11
-5	61	61
-6	15*	7
-7	73	-79
-8	8*	-8
-9	30*	27
-10	37	41
-11	13*	-7
-12	44	-38
	2,7,L	
0	80	-79
1	104	-106
2	47	44
3	26*	30
4	54	56
5	0*	-11
6	111	-114
7	37	33
8	59	56
9	13*	-11
-1	49	-53
-2	61	-58
-3	44	-45
-4	26*	29
-5	120	116
-6	38	41
-7	53	-60
-8	50	-51
-9	17*	12
-10	22*	27
-11	58	55
-12	18*	-10
	3,7,L	
		-61
1	65	64
2	54	56
3	10*	-1
4	92	-90
5	46	-49
6	107	97
7	22*	18
8	23*	-3
-1	57	-61
-2	14*	-22
-3	43	-43
-4	41	49
-5	32	25
-6	23*	24
-7	26*	-17
-8	38	-35
-9	29*	27
-10	39	36
-11	0*	8
-12	21*	20
-13	11*	-6
	4,7,L	
0	41	-44
1	62	63
2	77	72
3	0*	0
4	58	-60
5	10*	-8
6	0*	12
7	30*	21
-1	112	108
-2	19*	13
-3	42	-46
-4	57	-56
-5	53	-57
-6	79	77
-7	50	54
-8	33	-22
-9	31*	-31
-10	19*	-13
-11	11*	-9
-12	34	27
-13	35	30
	5,7,L	
0	29	41
1	73	73
2	0*	3
3	21*	-26
4	0*	0
5	0*	2
6	0*	7
-1	15*	-18
-2	33	-41
-3	61	-63
-4	15*	23
-5	100	104
-6	62	-65
-7	44	-48
-8	0*	0
-9	41	-39
-10	10*	-12
-11	15*	15
-12	16*	12
-13	17*	-13
-14	20*	-9
	6,7,L	
0	56	53
1	42	38
2	21*	-2
3	23*	-24
4	33*	-39
5	0*	7
-1	27*	19
-2	55	54
-3	47	42
-4	84	76
-5	23*	-29
-6	61	65
-7	14*	-20
-8	0*	-14
-9	24*	24
-10	48	-54
-11	28*	16
-12	20*	9
-13	22*	-23
-14	21*	-10
	7,7,L	
0	57	58
1	21*	-17
2	37	-41
3	36	-35
-1	67	73
-2	0*	-22
-3	41	-43
-4	66	-62
-5	17*	12
-6	133	129
-7	13*	17
-8	31*	-42
-9	36	-38
-10	9*	-7
-11	15*	11
-12	7*	3
-13	22*	20
	8,7,L	
0	45	54
1	13*	-17
-1	21*	-24
-2	22*	26
-3	32*	15
-4	39	33
-5	0*	-10
-6	83	-75
-7	21*	-5
-8	68	74
-9	0*	15
-10	25*	-19
-11	53	-52
-12	8*	7
-13	34*	36
	9,7,L	
-1	13*	20
-2	48	50
-3	12*	-13
-4	23*	-26
-5	16*	-16
-6	45	-40
-7	15*	20
-8	70	69
-9	0*	9
-10	50	-54
-11	19*	-7
-12	9*	18
	10,7,L	
-4	39	44
-5	29*	24
-6	32*	-34
-7	16*	-18
-8	0*	-1
-9	4*	7
-10	25*	23
-11	0*	8
	0,8,L	
0	56	-51
1	6*	-29
2	0*	5
3	55	61
4	40	34
5	59	-62
6	70	-74
7	35	34
8	38	32
9	15*	10
	1,8,L	
0	96	-97
1	30*	-36
2	5*	4
3	46	51
4	26*	23
5	85	-84
6	0*	-9
7	23*	25
8	18*	10
-1	21*	-26
-2	65	-64
-3	17*	3
-4	25*	25
-5	53	50
-6	34	-33
-7	71	-69
-8	12*	9
-9	27*	19
-10	9*	2
	2,8,L	
0	56	-58
1	25*	26
2	81	82
3	18*	-8
4	28*	-23
5	9*	8
6	21*	-34
7	0*	1
-1	54	-61
-2	7*	-2
-3	28*	22
-4	70	72
-5	0*	15
-6	38	-42
-7	44	-40
-8	29*	-27
-9	0*	25
-10	41	42
	3,8,L	
0	25*	-19
1	30*	25
2	43	39
3	21*	14
4	8*	-30
5	0*	-2
6	15*	17
-1	29*	39
-2	0*	2
-3	17*	-18
-4	23*	-22
-5	44	44
-6	54	56
-7	24*	-16
-8	18*	-25
-9	14*	-14
-10	46	42
-11	0*	19
	4,8,L	
0	64	56
1	79	72
2	26*	20
3	0*	-11
4	77	-77
5	30*	-21
-1	52	-57
-2	75	-78
-3	9*	-16
-		
-5	79	76
-6	0*	10
-7	34	-27
-8	31*	-26
-9	0*	-7
-10	18*	15
-11	38	35
	5,8,L	
0	34	30
1	43	40
2	0*	-1
3	36	-35
-1	20*	16
-2	38	34
-3	22*	-6
-4	19*	-21
-5	59	-60
-6	14*	5
-7	84	80
-8	0*	-12
-9	9*	-20
-10	31*	-20
-11	12*	8
	6,8,L	
0	66	66
1	26*	-15
2	63	-55
-1	41	38
-2	25*	-27
-3	28*	-33
-4	28*	-20
-5	0*	-18
-6	49	47
-7	54	51
-8	21*	-29
-9	45	-47
-10	0*	6
-11	20*	21
	7,8,L	
0	23*	-7
-1	46	-48
-2	24*	13
-3	42	39
-4	12*	2
-5	19*	6
-6	20*	-27
-7	29*	-29
-8	23*	20
-9	0*	7
-10	0*	6
	8,8,L	
-3	14*	3
-4	68	-65
-5	22*	-2
-6	57	63
-7	23*	-9
-8	24*	-23
-9	0*	12
	0,9,L	
1	11*	-13
2	0*	-3
3	9*	11
4	0*	7
5	0*	11
	1,9,L	
0	66	-66
1	30*	32
		4
3	34*	39
4	0*	-4
-1	72	-75
-2	0*	9
-3	60	59
-4	31*	33
-5	53	-50
-6	26*	-31
	2,9,L	
0	0*	-25
1	21*	7
2	54	44
3	0*	12
-1	11*	12
-2	16*	-20
-3	11*	-15
-4	17*	16
-5	19*	-4
-6	19*	-15
	3,9,L	
0	47	39
1	55	52
2	10*	23
-1	34*	-31
-2	57	-54
-3	6*	-5
-4	14*	30
-5	76	76
-6	0*	-13
	4,9,L	
0	59	52
-1	18*	20
-2	40	37
-3	23*	-19
-4	10*	-5
-5	25*	19
-6	0*	-8
	5,9,L	
-3	45	-42
-4	25*	8
-5	29*	28

Discussion

Comparison of the film and diffractometer results

The atomic coordinates and temperature factors derived from the film and diffractometer data are given in Tables 3 and 4. The precision of the determined parameters is primarily influenced by (1) the number of observations and (2) the accuracy of the measurements (Bennett, Cotton, Weaver, Williams & Watson, 1967). Clearly, the parameters obtained from the counter data gain in (1), and probably also in (2), over the film data. Estimated standard deviations in the film parameters are about three to five times larger than the corresponding counter data values. The differences between the two parameter lists are in most cases less than 1σ, where the σ values referred to are those of the film parameters, and with the exception of U_{22} for C(5), U_{12} for O(3) and U_{22} for Cl, all of the differences are less than 3σ. Two previous investigations which led to a comparison of the parameters derived from film and diffractometer studies have also found a 3σ agreement: *e.g.* thiourea (Truter, 1967); *trans*-dichlorobis(dimethyl sulfoxide)palladium(II) (Bennett *et al.*, 1967).

Bond lengths obtained from the two determinations are given in Table 5. The differences between the two sets of distances are in all cases less than 2σ, where the σ is that computed from the film parameters.

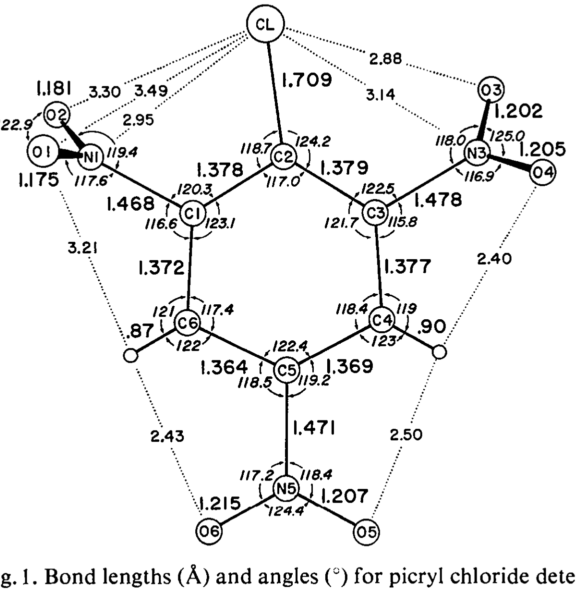


Fig. 1. Bond lengths (Å) and angles (°) for picryl chloride determined from the diffractometer study.

Table 3. *Fractional coordinates and temperature factors* (Å^2) *from the film study of picryl chloride**

Estimated standard deviations are given in parentheses and the temperature factors are in the form exp $[-2\pi^2(h^2a^{*2}U_{11}+\ldots 2klb^*c^*U_{23})]$.

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl	0·0958 (4)	0·0526 (6)	0·1811 (4)	0·069 (2)	0·034 (2)	0·096 (3)	−0·021 (2)	0·050 (2)	−0·028 (3)
C(1)	0·2147 (13)	0·3721 (20)	0·1572 (10)	0·051 (7)	0·030 (8)	0·047 (7)	−0·014 (6)	0·031 (6)	−0·027 (9)
C(2)	0·1828 (13)	0·2740 (20)	0·2235 (11)	0·042 (7)	0·029 (8)	0·061 (8)	−0·011 (6)	0·029 (6)	−0·023 (9)
C(3)	0·2243 (14)	0·3640 (20)	0·3211 (10)	0·056 (8)	0·023 (7)	0·050 (7)	−0·009 (7)	0·035 (6)	−0·002 (9)
C(4)	0·3011 (13)	0·5366 (21)	0·3548 (10)	0·047 (7)	0·041 (9)	0·040 (7)	0·011 (7)	0·025 (6)	0·008 (9)
C(5)	0·3316 (12)	0·6277 (17)	0·2846 (9)	0·046 (6)	0·013 (7)	0·038 (6)	−0·009 (6)	0·024 (5)	−0·006 (7)
C(6)	0·2899 (13)	0·5492 (21)	0·1839 (10)	0·050 (7)	0·048 (9)	0·041 (7)	0·003 (7)	0·023 (6)	0·011 (9)
N(1)	0·1740 (16)	0·2863 (21)	0·0523 (10)	0·085 (9)	0·060 (9)	0·044 (8)	−0·022 (8)	0·028 (7)	−0·022 (9)
N(3)	0·1934 (15)	0·2817 (19)	0·3980 (11)	0·081 (9)	0·042 (8)	0·074 (9)	−0·004 (7)	0·056 (8)	0·000 (10)
N(5)	0·4120 (14)	0·8119 (19)	0·3187 (11)	0·077 (8)	0·048 (9)	0·071 (8)	−0·030 (7)	0·040 (7)	−0·022 (10)
O(1)	0·2691 (18)	0·1979 (28)	0·0511 (12)	0·132 (12)	0·195 (18)	0·108 (12)	0·036 (13)	0·072 (11)	−0·081 (16)
O(2)	0·0513 (16)	0·3060 (23)	−0·0265 (10)	0·136 (12)	0·122 (13)	0·049 (7)	0·004 (10)	0·007 (8)	−0·044 (11)
O(3)	0·0800 (13)	0·1887 (18)	0·3597 (10)	0·089 (9)	0·073 (9)	0·111 (9)	−0·025 (7)	0·064 (8)	0·000 (10)
O(4)	0·2815 (14)	0·3099 (19)	0·4942 (10)	0·122 (10)	0·099 (10)	0·061 (7)	−0·032 (8)	0·063 (7)	0·000 (10)
O(5)	0·4262 (14)	0·9040 (18)	0·3961 (10)	0·124 (10)	0·068 (9)	0·081 (8)	−0·054 (8)	0·065 (8)	−0·073 (10)
O(6)	0·4598 (13)	0·8705 (17)	0·2632 (10)	0·120 (9)	0·053 (8)	0·107 (9)	−0·048 (7)	0·082 (8)	−0·015 (9)

* The reduction and refinement of the film data were done using the cell parameters determined at The Ohio State University: $a=11·07$, $b=6·83$, $c=14·72$ Å, $\beta=124·2°$.

Table 4. *Fractional coordinates and temperature factors* (Å^2) *from the diffractometer study of picryl chloride*

Estimated standard deviations are given in parentheses and the temperature factors are in the form exp $[-2\pi^2(h^2a^{*2}U_{11}+\ldots 2klb^*c^*U_{23})]$.

	x	y	z	U or U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl	0·0956 (1)	0·0524 (1)	0·1810 (1)	0·0729 (6)	0·0418 (4)	0·1021 (7)	−0·0188 (5)	0·0529 (6)	−0·0205 (5)
C(1)	0·2150 (3)	0·3716 (4)	0·1570 (2)	0·047 (2)	0·046 (2)	0·039 (2)	−0·002 (1)	0·025 (2)	−0·010 (1)
C(2)	0·1813 (3)	0·2758 (4)	0·2227 (2)	0·042 (2)	0·033 (1)	0·054 (2)	0·000 (1)	0·027 (1)	−0·005 (1)
C(3)	0·2247 (3)	0·3648 (4)	0·3209 (2)	0·044 (2)	0·040 (2)	0·046 (2)	0·004 (1)	0·030 (1)	0·005 (1)
C(4)	0·2998 (3)	0·5405 (5)	0·3529 (3)	0·048 (2)	0·045 (2)	0·039 (2)	−0·002 (2)	0·026 (2)	−0·007 (2)
C(5)	0·3304 (3)	0·6279 (4)	0·2840 (2)	0·041 (2)	0·038 (1)	0·041 (2)	−0·006 (1)	0·025 (1)	−0·007 (1)
C(6)	0·2886 (3)	0·5477 (5)	0·1853 (3)	0·051 (2)	0·044 (2)	0·044 (2)	0·000 (2)	0·032 (2)	0·001 (2)
N(1)	0·1741 (4)	0·2851 (5)	0·0518 (3)	0·077 (2)	0·064 (2)	0·056 (2)	−0·011 (2)	0·034 (2)	−0·024 (2)
N(3)	0·1927 (3)	0·2797 (4)	0·3979 (3)	0·073 (2)	0·051 (2)	0·069 (2)	0·003 (2)	0·049 (2)	0·011 (2)
N(5)	0·4115 (3)	0·8151 (4)	0·3170 (2)	0·068 (2)	0·055 (2)	0·061 (2)	−0·023 (2)	0·039 (2)	−0·016 (2)
O(1)	0·2647 (4)	0·1973 (7)	0·0501 (3)	0·135 (3)	0·230 (4)	0·101 (2)	0·030 (3)	0·065 (2)	−0·076 (3)
O(2)	0·0533 (4)	0·3051 (5)	−0·0265 (2)	0·122 (3)	0·132 (3)	0·059 (2)	0·007 (2)	0·014 (2)	−0·037 (2)
O(3)	0·0805 (3)	0·1891 (4)	0·3598 (2)	0·086 (2)	0·085 (2)	0·104 (2)	−0·018 (2)	0·066 (2)	0·012 (2)
O(4)	0·2808 (3)	0·3101 (4)	0·4944 (2)	0·120 (2)	0·106 (2)	0·057 (2)	−0·024 (2)	0·056 (2)	0·002 (2)
O(5)	0·4260 (3)	0·9035 (4)	0·3938 (2)	0·126 (2)	0·077 (2)	0·088 (2)	−0·050 (2)	0·071 (2)	−0·048 (2)
O(6)	0·4585 (3)	0·8709 (4)	0·2639 (2)	0·112 (2)	0·077 (2)	0·096 (2)	−0·042 (2)	0·075 (2)	−0·018 (2)
H(4)	0·328 (3)	0·591 (4)	0·418 (2)	0·040 (8)					
H(6)	0·304 (3)	0·608 (4)	0·141 (2)	0·052 (9)					

Table 5. *Film and diffractometer bond lengths*

Estimated standard deviations are in parentheses.

	Film*	Diffractometer	Corrected diffractometer†
Cl—C(2)	1·703 (13) Å	1·709 (3) Å	
C(1)–C(2)	1·379 (24)	1·378 (5)	
C(1)–C(6)	1·387 (19)	1·372 (4)	
C(2)–C(3)	1·379 (21)	1·379 (5)	
C(3)–C(4)	1·366 (19)	1·377 (4)	
C(4)–C(5)	1·398 (23)	1·369 (6)	
C(5)–C(6)	1·384 (19)	1·364 (5)	
C(1)–N(1)	1·463 (21)	1·468 (5)	
C(3)–N(3)	1·462 (26)	1·478 (6)	
C(5)–N(5)	1·450 (17)	1·471 (4)	
N(1)–O(1)	1·217 (28)	1·175 (6)	1·286 Å
N(1)–O(2)	1·195 (16)	1·181 (4)	1·250
N(3)–O(3)	1·220 (19)	1·202 (4)	1·235
N(3)–O(4)	1·199 (17)	1·205 (4)	1·249
N(5)–O(5)	1·229 (22)	1·207 (5)	1·248
N(5)–O(6)	1·258 (26)	1·215 (6)	1·248
C(4)–H(4)		0·90 (3)	
C(6)–H(6)		0·87 (4)	

* Calculated using the diffractometer cell parameters.

† Bond length corrections using the riding motion model described by Busing & Levy (1964). The corrections were evaluated in terms of the principal axes displacements shown in Table 7.

Description of the structure

Fig. 1 gives the values of the intramolecular bond lengths and angles and also some of the close nonbonded approaches which were determined from the diffractometer study. The benzene ring is planar within experimental error and the maximum deviation of the Cl atom and three N atoms from the ring plane is 0·08 Å (Table 6). The molecule shows several features which are characteristic of other nitro-aromatics (Holden & Dickinson, 1969; Cady, 1967): (1) O–N–O angles of about 124°; (2) enlarged interior angles in the benzene ring at the nitro group carbon atoms (Carter, McPhail & Sim, 1966); (3) foreshortened aromatic C–C distances (average 1·373 Å; 1·378 Å in 2,4,6-trinitrophenetole, Gramaccioli, Destro & Simonetta, 1968). The N–O, C–N and C–Cl distances are normal.

Table 6. *Least-squares planes and deviations* (Å)

	Plane 1	Plane 2	Plane 3	Plane 4
C(1)	0·001	0·000		
C(2)	0·003			
C(3)	−0·004		0·002	
C(4)	0·001			
C(5)	0·004			−0·001
C(6)	−0·005			
Cl	0·078*			
N(1)	0·026*	−0·002		
N(3)	−0·031*		−0·007	
N(5)	0·019*			0·003
O(1)	−0·980*	0·001		
O(2)	1·064*	0·001		
O(3)	−0·622*		0·002	
O(4)	0·525*		0·002	
O(5)	−0·209*			−0·001
O(6)	0·252*			−0·001

Table 6 (*cont.*)

Plane 1: $8·1966x - 3·3552y - 0·6574z = 0·4110$
Plane 2: $-5·3889x - 5·7193y + 6·8196z = -2·2138$
Plane 3: $-5·9507x + 5·7050y + 3·7290z = 1·9391$
Plane 4: $6·7269x - 3·3386y + 2·5192z = 0·8425$

* These atoms were not included in the definition of the plane.

Perhaps the most interesting aspect of the structure is the orientation of the nitro groups relative to the benzene ring. The angle between the plane of the nitro group on C(1) and that of the benzene ring is 81°, a position in which both Cl···O distances exceed the sum of the respective van der Waals radii (3·2 Å; Pauling, 1960). Thus the 2·95 Å N(1)···Cl separation represent the major interaction between this nitro group and the chlorine atom. In contrast to this, the C(3) nitro group–benzene angle is only 33° and now the atom closest to the chlorine atom is O(3) at 2·88 Å. The N(3)···Cl distances is 3·14 Å. The third nitro group, on C(5), lies at an angle of 13° to the benzene ring plane, a value which is typical of aromatic nitro groups with no *ortho* substituents (Holden & Dickinson, 1969; Holden, 1969).

The Cl atom does not occupy a symmetrical position between the C(1) and C(3) nitro groups, a situation which can be readily appreciated by considering the Cl–C(2)–C(1) and Cl–C(2)–C(3) angles of 118·7 and 124·2°. Similar relative displacements have been found for C(2) substituents in other 1,3-dinitro-aromatics, *e.g.* 2,4,6-trinitrophenetole (Gramaccioli, Destro & Simonetta, 1968); 2,4-dichloro-1,3,5-trinitrobenzene (Holden & Dickinson, 1967); 4-methyl-3,5-dinitrobenzoic acid (Grant & Richards, 1969). In all of these examples, as in picryl chloride, the two *ortho* nitro groups are rotated by different amounts out of the benzene ring plane and the interposed substituent is displaced toward the group with the largest rotation.

It does not seem possible to rationalize the unsymmetrical disposition of the C(1) and C(3) nitro groups in picryl chloride on the basis of molecular packing arguments. The only intermolecular distance shorter than the sum of the appropriate van der Waals radii is the 3·09 Å Cl···O separation. This distance, which is between molecules related by a center of symmetry (see Fig. 2), involves the nitro group on C(1). It is unlikely, however, that the interaction is responsible for the large out-of-plane rotation of this nitro group, since a smaller angle would actually increase the Cl···O distance.

The orientation of an aromatic nitro group is probably determined by a balance between (1) steric interactions with *ortho* substituents and (2) mesomeric interactions with the aromatic nucleus. Extremes are represented by (1) picryl iodide where steric effects predominate and both *ortho* nitro groups are 80° to the ring plane (Huse & Powell, 1940), and (2) nitro-aromatics with no *ortho* substituents in which resonance effects predominate (Holden & Dickinson (1969) have found an average value of 6° for this arrangement). In cases where the nitro group has an *ortho* substituent of intermediate size the situation is not clear, but Holden & Dickinson (1969) have reported an average rotation of 40°. With chlorine as the substituent, angles close to this mean have been found in: picryl chloride, C(1)–$NO_2=33°$, 1-chloro-2,4-dinitrobenzene, C(2)–$NO_2=39°$ (Watson, 1960); and 2,4-dichloro-1,3,5-trinitrobenzene, C(1)–NO_2=C(5)–$NO_2=37°$ (Holden & Dickinson, 1967). In two of these compounds, the chlorine atom is adjacent to a second nitro group and this group is almost normal to the benzene ring plane: C(3)–$NO_2=81°$ in picryl chloride and C(3)–$NO_2=75°$ in 2,4-dichloro-1,3,5-trinitrobenzene. Thus it would appear that an energy minimum is achieved in picryl chloride by (1) a normal (30–40°) rotation of one nitro group, (2) a displacement of the chlorine atom away from this group, and (3) a large rotation of the second nitro group. This is in contrast to the more symmetrical case in which the chlorine atom would be equidistant from both nitro groups, and the two groups would be rotated by equal amounts (presumably greater than 40°) out of the benzene ring plane.

The magnitudes of the principal axes of the thermal ellipsoids are given in Table 7. In the case of each oxygen atom, the direction of principal vibration is almost perpendicular to the respective C–NO_2 plane, suggesting that an important component of the motions of the nitro groups is an oscillation about the C–N bond. The largest displacements are shown by the two oxygen atoms on C(1)–NO_2, the 81° nitro group, and may arise from the relative absence of steric interactions involving these atoms.

Table 7. *The root-mean-square amplitudes of the principal axes of the thermal ellipsoids for the diffractometric data*

	R.m.s. displacement (Å)		
	Axis 1	Axis 2	Axis 3
Cl	0·181	0·263	0·329
C(1)	0·173	0·219	0·233
C(2)	0·178	0·203	0·239
C(3)	0·187	0·193	0·226
C(4)	0·177	0·217	0·223
C(5)	0·179	0·191	0·217
C(6)	0·182	0·211	0·228
N(1)	0·188	0·286	0·289
N(3)	0·199	0·241	0·281
N(5)	0·193	0·238	0·292

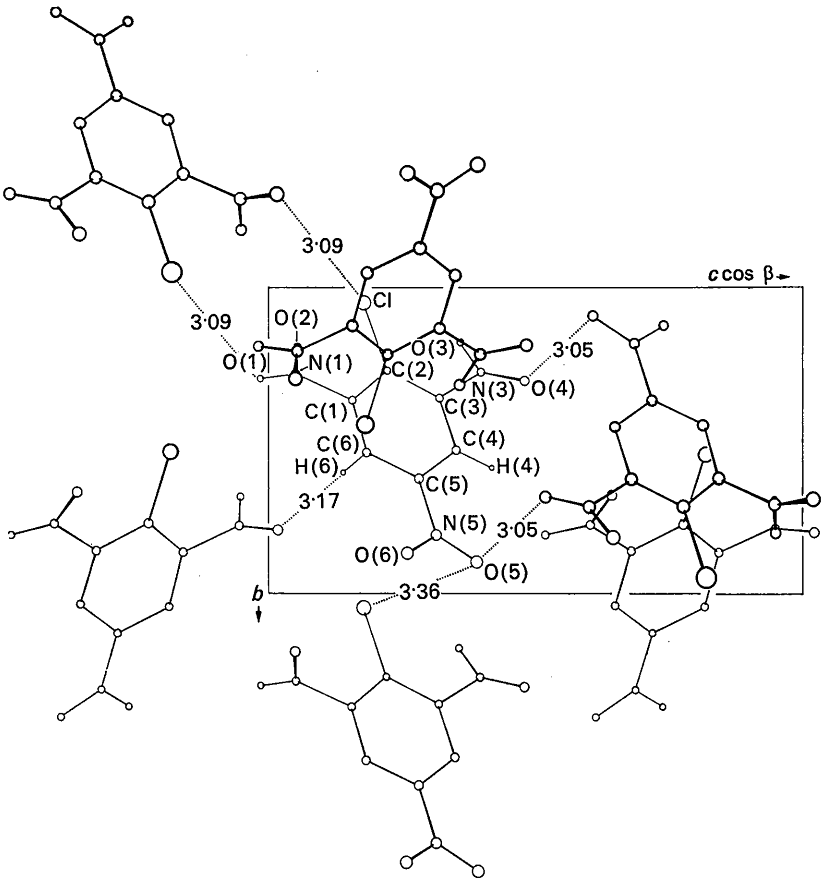


Fig. 2. Picryl chloride packing diagram viewed along [100] showing the shortest intermolecular distances (Å).

Table 7 (*cont.*)

	Axis 1	Axis 2	Axis 3	Plane normal*
O(1)	0·193	0·359	0·549	0·548
O(2)	0·208	0·348	0·458	0·434
O(3)	0·201	0·320	0·333	0·325
O(4)	0·205	0·298	0·378	0·369
O(5)	0·185	0·283	0·398	0·398
O(6)	0·203	0·274	0·376	0·375

* R.m.s. displacement normal to the respective C–NO_2 plane.

Correctons have been applied to the six N–O distances using the riding motion algorithm of Busing & Levy (1964) (Table 5). An over-correction in the case of N(1)–O(1) may be indicated.

The two hydrogen atoms do not appear to be involved in intermolecular hydrogen bond formation since the shortest distance is 3·17 Å between H(6) and O(1) (Fig. 2). The closest H···O approaches are of the intramolecular type and are between H(4) and C(3)–NO_2 and C(5)–NO_2, and between H(6) and C(5)–NO_2. These contacts are shown in Fig. 1.

This work was supported in part by the Air Force Office of Scientific Research, AF18(600)–1371, through the Research Foundation of The Ohio State University and the National Science Foundation, GP-15791. The UNIVAC 1108 computer time for this project was supported under NASA Grant NSG-398 and through the facilities of the Computer Science Center of the University of Maryland. We are grateful to the Biochemistry Section, Walter Reed Army Hospital, for the use of the automatic diffractometer.

References

BENNETT, M. J., COTTON, F. A., WEAVER, D. L., WILLIAMS, R. J. & WATSON, W. H. (1967). *Acta Cryst.* **23**, 788.

BERGHUIS, J., HAANAPPEL, IJ. M., POTTERS, M., LOOPSTRA, B. O., MACGILLAVRY, C. H. & VEENENDAAL, A. L. (1955) *Acta Cryst.* **8**, 478.

BUSING, W. R. & LEVY, H. A. (1959). *ORXLS*, Report 59–4–37, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

BUSING, W. R. & LEVY, H. A. (1964). *Acta Cryst.* **17**, 142.

CADY, H. H. (1967). *Acta Cryst.* **23**, 601.

CARTER, O. L., MCPHAIL, A. T. & SIM, G. A. (1966). *J. Chem. Soc.* (*A*), p. 822.

GLUYAS, R. E. & HARRIS, P. M. (1959). *Structures of Trinitro-Aromatic Crystals and Related Substances*, Ohio State University Research Foundation Report AFOSF-TR-59-165, p. 31.

GOL'DER, G. A., ZHDANOV, G. S. & UMANSKII, M. M. (1953). *Dokl. Akad. Nauk SSSR*, **92**, 311; Reported in 1954, *Chem. Abstr.* **48**, 13331*c*.

GRAMACCIOLI, C. M., DESTRO, R. & SIMONETTA, M. (1968). *Acta Cryst.* B**24**, 129.

GRANT, D. F. & RICHARDS, J. P. G. (1969). *Acta Cryst.* B**25**, 564.

HERTEL, E. & ROMER, G. H. (1933). *Z. Phys. Chem., Abt. B*, **22**, 267.

HOLDEN, J. R. (1969). *Acta Cryst.* A**25**, part S3, S154.

HOLDEN, J. R. & DICKINSON, C. (1967). *J. Phys. Chem.* **71**, 1129.

HOLDEN, J. R. & DICKINSON, C. (1969). *J. Phys. Chem.* **73**, 1199.

HUSE, G. & POWELL, H. M. (1940). *J. Chem. Soc.* p. 1398.

International Tables for X-ray Crystallography (1962). Vol. III, p. 202. Birmingham: Kynoch Press.

KITAIGORODSKII, A. I. (1948). *Izv. Akad. Nauk SSSR, Otdel. Khim. Nauk*, 278; Reported in 1949, *Chem. Abstr.* **43**, 929*a*.

PAULING, L. (1960). *The Nature of the Chemical Bond*, 3rd ed., p. 260. Ithaca: Cornell Univ. Press.

STEWART, R. F., DAVIDSON, E. & SIMPSON, W. (1965). *J. Chem. Phys.* **42**, 3175.

TRUTER, M. R. (1967). *Acta Cryst.* **22**, 556.

WATSON, K. J. (1960). *Nature, Lond.* **188**, 1102.

X-ray 67, *Program System for X-ray Crystallography* (1967). Computer Science Center, University of Maryland, Technical Report 67–58.