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The Crystal Structure of 10-Methyl-9-{[(2-chloroethyl)thio]methyl}anthracene (ICR-358)

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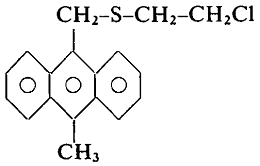
The crystal structure of 10-methyl-9-{[(2-chloroethyl)thio]methyl}anthracene (ICR-358), an anthracene sulfur mustard having mild antitumor activity, has been studied. The crystals are triclinic, space group $P\bar{1}$, with $Z=2$ for $C_{18}H_{17}ClS$ (M.W. 300·83), cell dimensions a = 9·411 (2), b = 11·984 (3) c = 7·835 (2) Å, α = 107·62 (3), β = 109·48 (3), γ = 101·54° (3), V = 748·0 Å^3. The calculated and measured densities are 1·337 and 1·327 g.cm^{-3} respectively. The structural formula is:



Three-dimensional diffractometer data were collected with Cu $K\alpha$ radiation. Of the 2790 reflections scanned, 2296 were above the threshold of measurement. The structure was solved by multiple-vector superpositions in the Patterson function and refined by a full-matrix least-squares procedure to the final residual R = 0·046. There is an angle of 8·7° between the planes of the outer benzene rings, each of which is planar within experimental error. In the packing in the unit cell two of the ring systems of one molecule partially overlap two from another molecule.

Introduction

The compound, 10-methyl-9-{[(2-chloroethyl)thio]methyl}anthracene (ICR-358) (Peck, O'Connell & Creech, 1967) was prepared by Dr Richard Peck of this Institute and was found to be a mild antitumor agent against the ascites form of Sarcoma 37. It is a monofunctional sulfur mustard derived from anthracene and was prepared and tested because of its analogy to the monofunctional nitrogen mustards. This study was undertaken to determine accurate molecular parameters for such an anthracene derivative.

Experimental

The compound was crystallized from a benzene–hexane mixture as large, deep yellow rectangular prisms. The crystals appeared to decompose on prolonged contact with the mother liquor in a closed container. The crystal data are summarized in Table 1.

Table 1. *Crystal data for 10-methyl-9-{[(2-chloroethyl)thio]methyl)}anthracene*

Formula: $C_{18}H_{17}ClS$	F.W. 300·85
Crystal system and space group:	Triclinic, $P\bar{1}$ (no absent spectra)
a = 9·411 (2) Å	α = 107·62 (3)°
b = 11·984 (3)	β = 109·48 (3)
c = 7·835 (2)	γ = 101·54 (3)
V = 748·0 Å^3	$F(000)$ = 316
D_x = 1·337 g.cm^{-3}	Z = 2
D_m = 1·327 g.cm^{-3} (in aqueous KI)	μ(Cu $K\alpha$) = 33·8 cm^{-1}
λ(Cu Kα) = 1·5418 Å	

A roughly spherical crystal, 0·2 mm diameter, was used to collect three-dimensional data on a Syntex automated diffractometer with monochromatic Cu $K\alpha$ radiation using the θ–2θ scan technique. Intensities were measured for 2790 reflections (those in the range $\sin\theta/\lambda$ = 0 to 0·606 Å^{-1}). These comprised 81·4% of the 3426 theoretically accessible in the copper sphere. The raw data were corrected for intensity loss from X-irradiation by means of a curve derived from the loss in intensity of the measured standard reflections as a function of time (23% over 95 hours). Values for $\sigma(I)$ were derived from counting statistics and measured instrumental uncertainties. There were 494 reflections for which the measured intensity, I_{obs}, was less than $3\sigma(I)$ and these were considered to be unobserved. For these 'unobserved reflections', when $I \geq \sigma(I)$, the measured value of I was used in computing F and when $I < \sigma(I)$, F was computed using the value 0·77 $\sigma(I)$. The intensity data were converted to structure amplitudes by application of Lorentz and polarization factors and a spherical absorption correction (Johnson, 1963), and placed on an absolute scale with a Wilson plot.

Structure determination and refinement

Starting with the highest peak in the Patterson map, four vector superpositions on other persisting large peaks gave the trial structure with an initial residual of 0·43. Five cycles of isotropic block-diagonal least-squares refinement reduced this value to 0·22 and a subsequent cycle of full-matrix least-squares aniso-

Table 2. *Final atomic parameters*

Positional parameters are given as fractions of cell edges $\times 10^4$.
Anisotropic temperature factors are expressed as:

$$\exp\left[-\tfrac{1}{4}(h^2a^{*2}B_{11}+k^2b^{*2}B_{22}+l^2c^{*2}B_{33}+2hka^*b^*B_{12}+2hla^*c^*B_{13}+2klb^*c^*B_{23})\right].$$

and isotropic temperature factor as:

$$\exp(-B\sin^2\theta/\lambda^2) \text{ with } B \text{ values given in Å}^2.$$

The standard deviations for each parameter, determined from the inverted full matrix, are given in parentheses and apply to the last specified digits.

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}	B
S	1646·1 (11)	2691·3 (7)	−2465·6 (12)	4·65 (2)	3·45 (2)	3·61 (2)	1·26 (3)	2·43 (3)	1·73 (4)	3·90
Cl	2776·4 (12)	5576·0 (7)	−4595·3 (13)	10·11 (5)	5·23 (3)	5·88 (3)	0·48 (6)	3·97 (6)	3·19 (7)	7·08
C(1)	1718 (3)	4532 (2)	−3860 (4)	5·21 (10)	4·61 (10)	4·75 (10)	1·55 (18)	2·46 (14)	2·60 (24)	4·86
C(2)	2750 (3)	3865 (2)	−2981 (4)	3·75 (8)	5·02 (10)	3·86 (8)	1·19 (16)	2·19 (12)	2·30 (21)	4·21
C(3)	1765 (3)	3588 (2)	−0058 (4)	3·70 (8)	3·06 (8)	3·48 (8)	0·57 (14)	1·91 (11)	1·40 (18)	3·41
C(4)	0767 (2)	2745 (2)	0517 (3)	3·27 (7)	2·61 (8)	2·62 (7)	0·55 (13)	1·56 (9)	0·97 (15)	2·83
C(5)	1433 (2)	2074 (2)	1559 (3)	3·07 (7)	2·94 (8)	2·39 (7)	0·69 (13)	1·31 (9)	0·86 (16)	2·80
C(6)	3054 (3)	2100 (2)	2050 (4)	3·34 (7)	4·76 (11)	3·47 (8)	1·12 (13)	1·88 (11)	1·78 (21)	3·86
C(7)	3671 (3)	1425 (3)	3006 (4)	3·74 (7)	6·02 (12)	3·85 (9)	2·37 (14)	1·88 (12)	2·08 (24)	4·54
C(8)	2731 (3)	0664 (2)	3572 (4)	5·09 (10)	4·84 (10)	3·63 (9)	2·44 (20)	1·95 (12)	2·17 (22)	4·52
C(9)	1204 (3)	0618 (2)	3174 (4)	4·57 (8)	3·62 (9)	3·19 (8)	1·40 (16)	1·90 (11)	1·71 (19)	3·79
C(10)	0486 (3)	1319 (2)	2179 (3)	3·41 (8)	2·85 (8)	2·32 (7)	0·74 (13)	1·27 (9)	1·02 (15)	2·86
C(11)	−1106 (3)	1283 (2)	1786 (3)	3·35 (8)	2·97 (8)	2·63 (7)	0·46 (14)	1·43 (10)	1·07 (16)	2·98
C(12)	−2050 (3)	0588 (2)	2590 (4)	4·10 (8)	4·99 (9)	4·75 (9)	0·76 (16)	2·25 (12)	2·91 (22)	4·61
C(13)	−1794 (3)	1899 (2)	0638 (3)	2·97 (7)	2·93 (8)	2·78 (7)	0·62 (13)	1·39 (10)	1·09 (16)	2·89
C(14)	−3442 (3)	1806 (2)	0047 (4)	3·17 (7)	4·74 (10)	4·02 (8)	1·01 (15)	1·81 (14)	2·00 (20)	3·98
C(15)	−4133 (3)	2328 (2)	−1163 (4)	3·16 (8)	5·26 (10)	5·06 (10)	1·60 (16)	1·52 (11)	2·42 (23)	4·49
C(16)	−3235 (3)	2992 (2)	−1889 (4)	4·27 (10)	4·40 (10)	4·43 (9)	1·81 (18)	1·59 (12)	2·35 (23)	4·37
C(17)	−1662 (3)	3141 (2)	−1335 (4)	4·30 (9)	3·64 (9)	3·76 (8)	1·48 (16)	2·06 (12)	2·08 (20)	3·90
C(18)	−0865 (3)	2608 (2)	−0051 (3)	3·38 (7)	2·64 (8)	2·77 (7)	0·84 (13)	1·51 (10)	1·08 (16)	2·93

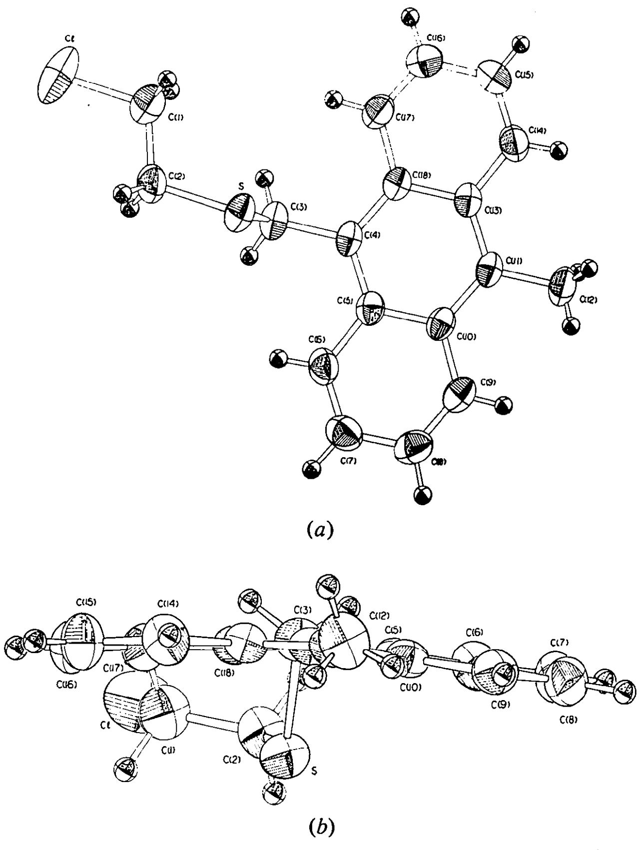


Fig. 1. Views of the molecule showing the thermal ellipsoids. (*a*) Perpendicular to the least-squares plane through central ring system. (*b*) View in (*a*) rotated 90°.

Table 2 (*cont.*)

Positional parameters are given as fractions of cell edges $\times 10^3$.

	x	y	z	B
H(11)	141 (3)	503 (3)	−293 (5)	5·8 (8)
H(12)	075 (3)	388 (3)	−517 (5)	6·0 (8)
H(21)	372 (3)	451 (3)	−175 (5)	6·3 (8)
H(22)	309 (3)	343 (2)	−405 (4)	4·8 (7)
H(31)	146 (2)	431 (2)	−007 (3)	4·6 (6)
H(32)	285 (3)	397 (2)	093 (4)	4·7 (6)
H(6)	368 (3)	257 (2)	171 (4)	5·4 (6)
H(7)	474 (3)	149 (2)	332 (4)	5·2 (6)
H(8)	326 (3)	021 (3)	419 (5)	5·6 (8)
H(9)	062 (3)	012 (2)	358 (4)	4·7 (6)
H(121)	−295 (5)	005 (4)	180 (7)	13·3 (15)
H(122)	−171 (5)	001 (4)	281 (6)	11·4 (14)
H(123)	−226 (6)	111 (4)	344 (8)	15·8 (19)
H(14)	−406 (3)	140 (2)	050 (4)	5·7 (8)
H(15)	−511 (3)	226 (3)	−153 (5)	5·5 (8)
H(16)	−368 (3)	335 (2)	−269 (4)	5·8 (7)
H(17)	−115 (3)	351 (2)	−193 (3)	4·4 (6)

tropic refinement gave $R=0·19$. The positions of twelve of the seventeen hydrogen atoms were derived from a difference Fourier synthesis. These were included but not refined in two further least-squares anisotropic cycles which resulted in $R=0·069$. The remaining five hydrogen atoms were located in a difference Fourier map and refinement of all atoms proceeded for two more cycles with the hydrogen atoms treated isotropically. The weights used in the refinement were $1/(\sigma^2(F_o))$. The unobserved reflections were assigned zero weight throughout the refinements. The quantity minimized was $\sum w\{||F_o|-|F_c||\}^2$. The

Table 3. *Observed and calculated structure factors*

Each entry lists, in order l, $10|F_o|$, $10F_c$. Unobserved reflections are denoted by * and those apparently affected by extinction by +. Values of $10|F_o|$ have been corrected for absorption. $\Delta f''$ was not used in computing values of F_c but the R value was unaffected by its exclusion. Therefore, values of F_c have phase angles of 0 or 180° only in this listing.

-11,0,L		
3	35	34
4	41	44
5	17	20
-11,1,L		
2	19	21
3	18	-15
4	51	-53
5	9*	-10
-11,2,L		
2	20	20
3	39	35
4	54	-49
5	36	-39
-11,3,L		
1	14	6
2	15	-18
3	7*	5
4	43	38
5	7*	0
-11,4,L		
1	7*	5
2	17	17
3	47	-40
4	21	-20
-11,5,L		
1	10*	2
2	32	26
3	7*	6
4	14*	-15
-11,6,L		
2	7*	-8
3	7*	9
-10,-4,L		
4	10*	7
5	21	23
-10,-3,L		
2	14	11
3	18	25
4	7*	-6
5	8*	-10
6	31	31
-10,-2,L		
1	44	-48
2	54	-48
3	16	11
4	20	19
5	32	-35
6	16	-10
-10,-1,L		
1	7*	-1
2	29	-32
3	18	-12
4	47	44
5	25	29
6	19	-15
7	26	-27
-10,0,L		
0	12*	-6
1	47	44
2	19	20
3	26	-32
4	32	-31
5	39	41
6	8*	6
7	36	-37
-10,1,L		
0	37	-39
1	47	-45
2	54	54
3	16*	16
4	34	-31
5	53	-54
6	12*	3
-10,2,L		
-1	26	24
0	29	31
1	20	-17
2	58	-55
3	62	59
4	64	71
5	25	-22
6	48	-52
-10,3,L		
-1	10*	-10
0	13*	12
1	33	32
2	32	-34
3	91	-93
4	36	41
5	31	35
6	20	-21
-10,4,L		
-1	12*	-15
0	15*	-16
1	19	14
2	22	20
3	9*	11
4	61	-62
5	8*	-12
6	50	53
-10,5,L		
-1	11*	5
0	11*	0
1	21	-24
2	31	31
3	93	90
4	46	50
5	51	-57
-10,6,L		
-1	8*	9
0	10*	15
1	43	-41
2	35	-37
3	21	25
4	16	11
5	18	-16
-10,7,L		
-1	21	17
0	10*	6
1	20	16
2	15*	-12
3	53	-56
4	53	-59
-10,8,L		
0	29	-32
1	8*	10
2	63	60
3	7*	-6
-10,9,L		
0	29	-25
1	18	-24
2	7*	1
-9,-6,L		
4	87	83
5	69	72
6	31	-31
-9,-5,L		
2	7*	-6
3	36	-36
4	21	29
5	44	45
6	19	-15
7	109	-110
-9,-4,L		
1	21	16
2	39	44
3	8*	10
4	55	-56
5	18	-9
6	14*	-18
7	9*	12
-9,-3,L		
0	7*	-7
1	30	-27
2	9*	17
3	8*	-13
4	24	-23
5	65	-65
6	13*	3
7	28	31
-9,-2,L		
0	12*	5
1	20	20
2	39	-34
3	12*	-16
4	19	19
5	19	17
6	47	-52
7	25	19
-9,-1,L		
-1	51	-52
0	8*	2
1	51	55
2	18*	22
3	61	-59
4	61	64
5	60	59
6	34	-30
7	38	-40
-9,0,L		
-1	8*	-8
0	76	-79
1	12*	15
2	9*	1
3	26	-23
4	28	-26
5	9*	2
6	54	49
7	8*	18
-9,1,L		
-2	8*	-14
-1	12*	17
0	74	80
1	23	-9
2	28	-23
3	53	48
4	23	30
5	54	-57
6	36	37
7	132	136
-9,2,L		
-2	32	-36
-1	26	-24
0	80	80
1	85	95
2	74	-73
3	9*	-9
4	92	86
5	11*	-17
6	38	-37
7	39	-37
-9,3,L		
-2	20	-17
-1	38	-39
0	16*	-21
1	43	-50
2	38	-46
3	47	-50
4	49	-51
5	33	28
6	29	-30
7	124	-131
-9,4,L		
-2	27	26
-1	31	36
0	19	-19
1	85	-91
2	22	-29
3	59	66
4	14*	-12
5	47	-49
6	68	67
-9,5,L		
-2	9*	-11
-1	66	61
0	23	28
1	25	-28
2	28	35
3	103	101
4	30	37
5	18	-13
6	21	-15
-9,6,L		
-2	24	-29
-1	29	-28
0	39	31
1	18*	28
2	10*	25
3	20	11
4	9*	-2
5	8*	-1
6	15	-18
-9,7,L		
-2	22	24
-1	9*	-20
0	43	-37
1	44	42
2	25	30
3	59	-59
4	64	-65
5	28	26
-9,8,L		
-2	25	18
-1	9*	12
0	15*	-16
1	56	-59
2	20	16
3	9*	8
4	49	-50
-9,9,L		
-2	33	-32
-1	27	24
0	29	26
1	32	-29
2	41	-42
3	26	27
4	44	46
-9,10,L		
-1	7*	-4
0	24	26
1	20	21
2	7*	6
-8,-8,L		
4	106	-107
5	7*	6
-8,-7,L		
2	19	-17
3	11*	6
4	55	44
5	89	-88
6	9*	1
7	56	55
-8,-6,L		
1	14	-1
2	54	-55
3	11*	-14
4	29	30
5	73	67
6	88	-91
7	27	-35
-8,-5,L		
0	7*	-1
1	25	29
2	45	43
3	57	-61
4	25	-26
5	20	16
6	83	77
7	28	-21
8	73	-72
-8,-4,L		
0	43	-45
1	9*	14
2	54	53
3	44	38
4	27	-29
5	29	-36
6	42	44
7	78	80
8	11*	-4
-8,-3,L		
-1	12*	16
0	8*	-7
1	67	-66
2	54	-51
3	75	77
4	115	119
5	10*	-3
6	57	-63
7	20	21
8	9*	4
-8,-2,L		
-2	20	17
-1	8*	5
0	44	44
1	55	58
2	52	-55
3	26	-28
4	127	131
5	14*	16
6	22	-20
7	50	-49
8	8*	15
-8,-1,L		
-2	44	44
-1	77	-78
0	59	-59
1	71	71
2	53	54
3	35	-36
4	220	-218
5	101	-102
6	9*	-10
7	8*	2
8	59	-58
-8,0,L		
-2	39	-46
-1	38	-38
0	70	-73
1	95	-94
2	60	57
3	49	50
4	116	-116
5	221	-223
6	85	90
7	57	59
8	35	-39
-8,1,L		
-3	16	14
-2	35	-38
-1	9*	-1
0	115	120
1	37	41
2	102	-101
3	85	79
4	99	100
5	178	176
6	96	96
7	63	65
8	32	32
-8,2,L		
-3	8*	14
-2	9*	-3
-1	19	19
0	92	97
1	90	92
2	23	20
3	146	-145
4	36	40
5	188	187
6	106	106
7	125	-122
8	32	-33
-8,3,L		
-3	16	10
-2	14*	24
-1	16*	16
0	47	-46
1	33	-39
2	19*	20
3	26	29
4	126	-131
5	32	-30
6	56	-57
7	114	-118
-8,4,L		
-3	31	-33
-2	31	24
-1	26	29
0	39	-41
1	114	-124
2	10*	-12
3	65	61
4	54	57
5	77	-79
6	94	-96
7	8*	4
-8,5,L		
-4	22	23
-3	9*	13
-2	89	-87
-1	16*	-16
0	50	42
1	10*	-8
2	44	-45
3	14*	-8
4	35	31
5	34	34
6	37	-35
7	10*	22
-8,6,L		
-4	23	19
-3	57	58
-2	9*	4
-1	70	-73
0	10*	6
1	86	83
2	26	35
3	48	-48
4	28	-26
5	32	39
6	22	24
-8,7,L		
-4	20	-19
-3	9*	5
-2	36	34
-1	62	70
0	53	-58
1	62	-60
2	83	80
3	15*	14
4	32	-31
5	22	1
6	31	30
-8,8,L		
-3	73	-73
-2	51	-49
-1	74	67
0	35	39
1	47	-48
2	31	-31
3	53	47
4	13*	9
5	25	-26
-8,9,L		
-3	9*	14
-2	42	-44
-1	20	-16
0	39	32
1	88	90
2	42	43
3	49	-49
4	44	38
-8,10,L		
-3	11*	8
-2	44	47
-1	23	-21
0	72	-71
1	36	25
2	10*	12
3	55	-54
-8,11,L		
-2	19	21
-1	29	33
0	38	-39
1	65	-64
2	56	-60
-8,12,L		
-1	24	20
0	33	39
-7,-9,L		
3	40	-43
4	22	-25
5	8*	16
6	21	15
-7,-8,L		
2	21	-17
3	64	62
4	10*	19
5	60	-61
6	29	35
7	33	32
-7,-7,L		
1	41	44
2	11*	-7
3	10*	-5
4	79	72
5	19	14
6	105	-107
7	80	93
8	28	36
-7,-6,L		
0	17*	17
1	83	86
2	29	26
3	47	-54
4	10*	16
5	11*	-6
6	39	39
7	111	-108
8	17	15
-7,-5,L		
-1	9*	16
0	10*	4
1	20	20
2	85	-85
3	36	-32
4	57	-58
5	24	19
6	27	22
7	73	66
8	145	-148
-7,-4,L		
-2	19	-18
-1	33	38
0	46	41
1	164	-163
2	163	-159
3	54	-54
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5	119	-118
6	152	-152
7	17*	15
8	66	68
-4,3,L		
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-3	216	212
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1	169	173
2	294	-301
3	76	-78
4	135	137
5	58	60
6	19*	-22
7	18	18
8	59	63
-4,4,L		
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0	68	-66
1	126	-122
2	67	67
3	9*	-15
4	115	-114
5	86	85
6	82	83
7	41	45
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-6	14*	15
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-3	43	-45
-2	86	-92
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0	27	-33
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3	142	142
4	14*	16
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7	23	25
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-1	46	-47
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1	53	-56
2	140	-154
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4	31	28
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2	85	81
3	115	-117
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5	36	33
6	29	25
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2	59	60
3	56	57
4	72	-75
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-6	17	-14
-5	69	76
-4	66	67
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-2	57	55
-1	36	42
0	13*	-16
1	138	-139
2	47	-45
3	18	-1
4	27	29
5	57	-61
-4,10,L		
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-5	26	21
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-3	56	54
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1	48	41
2	63	-63
3	19	24
4	40	39
-4,11,L		
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-5	69	-62
-4	19	-27
-3	52	-50
-2	10*	-2
-1	108	-107
0	20	17
1	63	66
2	58	58
3	7*	1
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-3	60	-68
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-1	67	65
0	89	-87
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2	36	38
-4,13,L		
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-3	35	-31
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-1	64	63
0	36	39
1	42	-46
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2	28	-27
3	11*	-11
4	47	-48
5	90	93
6	44	38
-3,-11,L		
0	25	-28
1	75	-75
2	38	35
3	24	-26
4	31	-31

Table 3 (*cont.*)

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4	68	70
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-6	57	-55
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2	90	-91
3	13*	-13
4	8*	-3
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-5	67	-62
-4	26	-32
-3	20	22
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1	28	-25
2	70	73
3	21	-23
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0	68	-87
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0	33	-30
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-1	13	-2
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2	8*	-2
3	32	34
4	63	-65
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3	113	111
4	30	31
5	63	-67
6	41	44
7	29	33
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0	22	19
1	14*	-4
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3	96	102
4	13*	0
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6	12*	5
7	15	-5
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1	58	59
2	12*	3
3	111	-116
4	225	-227
5	13*	-13
6	43	-49
7	25	-26
8	21	-23
-1,-9,L		
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-2	58	-61
-1	77	-78
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2	127	123
3	234	-228
4	120	-125
5	30	30
6	134	139
7	123	-130
8	11*	-13
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3	191	187
4	13*	-5
5	361	364
6	104	102
7	66	72
8	32	-32
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2	18	-19
3	176	-181
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6	80	-80
7	64	-63
8	103	108
9	23	-21
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-1	98	-99
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2	216	-205
3	56	-58
4	142	-144
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6	362	-370
7	144	-146
8	61	57
9	39	38
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-3	10*	-1
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-1	108	-106
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1	227	-228
2	387	401
3	147	-150
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5	167	-173
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7	39	42
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4	18	-26
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8	62	63
9	108	-114
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-2	16	4
-1	170	-174
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1	20	-21
2	42	-36
3	211	-217
4	242	245
5	9*	-18
6	101	-98
7	57	51
8	17*	13
9	22	18
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-6	44	-44
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0	126	-130
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2	50	-54
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4	75	78
5	64	64
6	76	80
7	77	-78
8	14*	-22
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3	41	43
4	161	-164
5	40	-39
6	38	41
7	101	101
8	86	-86
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3	99	100
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5	219	-215
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-6	31	31
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3	335	-325
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-7	16*	-16
-6	112	109
-5	22	4
-4	150	-143
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4	93	-96
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7	80	81
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3	303	301
4	117	118
5	132	-129
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2	129	-134
3	138	-133
4	231	-224
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-6	149	143
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1	10*	-22
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3	234	-233
4	181	-181
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6	86	88
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-7	62	-64
-6	22	-15
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-4	219	-215
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-6	9*	-1
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2	74	-78
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4	87	94
5	41	48
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-1	67	-70
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-6	62	61
-5	66	-70
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-3	98	-94
-2	88	91
-1	10*	14
0	139	-140
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2	47	47
3	15*	16
-1,11,L		
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-6	51	-51
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-4	20	17
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1	86	-88
2	15*	16
-1,12,L		
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-3	10*	16
-2	10*	-25
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0	15*	-9
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-5	59	-56
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-3	9*	-10
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1	107	-108
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3	17*	-18
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5	65	-69
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6	68	-72
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2	170	168
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4	132	128
5	30	32
6	10*	13
7	36	-45
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-2	9*	-12
-1	23	-22
0	96	-94
1	174	173
2	60	-56
3	192	-185
4	225	-223
5	87	45
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7	35	-34
8	64	-62
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-1	40	-34
0	46	-45
1	113	-113
2	177	174
3	234	-234
4	185	-180
5	39	-43
6	49	49
7	43	45
8	41	-40
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-3	44	-46
-2	68	-68
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0	67	-60
1	24	33
2	152	-155
3	204	206
4	186	182
5	12*	-17
6	20*	-28
7	24	25
8	51	53
0,-7,L		
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-4	35	39
-3	14*	-21
-2	28	16
-1	136	-139
0	148	147
1	9*	15
2	70	-68
3	192	-194
4	210	214
5	199	196
6	161	-157
7	26	-25
8	20*	-26
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-4	62	-60
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2	125	122
3	128	-123
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5	29	-25
6	169	168
7	97	-94
8	10*	-16
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0	58	-48
1	72	-79
2	71	73
3	231	226
4	60	-57
5	26	-32
6	58	-60
7	157	158
8	55	-52
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-4	77	-80
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0	178	185
1	96	-97
2	288	-288
3	138	-133
4	317	317
5	128	-130
6	44	-47
7	68	-71
8	87	86
0,-3,L		
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-5	96	-92
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2	275	-272
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5	108	109
6	114	-112
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1	64	-62
2	270	277
3	167	-175
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5	177	-173
6	109	108
7	11*	0
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0	21	-18
1	242	249
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3	325	341
4	46	-49
5	10*	-11
6	72	70
7	90	88
8	23	-20
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6	27	-22
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5	74	68
6	104	-106
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1	38	37
2	45	45
3	98	-100
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6	98	98
7	91	-93
8	16	6
9	7*	-13
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0	43	-49
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6	9*	3
7	104	103
8	109	-111
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1	247	-245
2	64	-59
3	35	-29
4	173	174
5	201	-202
6	60	59
7	10*	-19
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9	32	-29
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1	183	189
2	213	-221
3	173	161
4	134	-126
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6	88	-87
7	17*	-23
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9	110	113
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7	37	-36
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0	247	-246
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4	364	-364
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0	160	154
1	100	-100
2	8*	-21
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6	116	-114
7	23	23
8	17	5
9	50	-50
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0	7*	7
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6	99	102
7	129	-128
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9	72	75
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2	117	116
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5	186	171
6	12*	-14
7	27	28
8	94	-90
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-6	30	-28
-5	99	99
-4	51	-52
-3	35	14
-2	88	-87
-1	149	144
0	12*	-1
1	248	-264
2	147	156
3	104	100
4	159	157
5	139	-137
6	26	-30
7	9*	6
8	54	55
-3,2,L		
-7	21	19
-6	17	-18
-5	112	-106
-4	139	142
-3	20	15
-2	117	-114
-1	7*	2
0	170	173
1	139	147
2	340	-357
3	68	67
4	18	-14
5	20	-13
6	167	-169
7	35	34
8	39	40
-3,3,L		
-7	27	24
-6	9*	-8
-5	73	-72
-4	102	-108
-3	147	150
-2	29	24
-1	305	-310
0	8*	10
1	37	36
2	190	197
3	388	-408
4	10*	17
5	47	45
6	32	31
7	57	-52
8	8*	-9
-3,4,L		
-7	34	-36
-6	29	33
-5	35	31
-4	19	-11
-3	87	-83
-2	113	108
-1	219	227
0	247	-256
1	192	208
2	51	50
3	146	148
4	142	-143
5	26	16
6	68	71
7	49	50
-3,5,L		
-7	11*	-7
-6	37	32
-5	66	70
-4	82	75
-3	73	-68
-2	83	-87
-1	95	-88
0	198	205
1	178	-188
2	8*	3
3	26	-21
4	137	135
5	141	-136
6	76	-78
7	50	53
-3,6,L		
-7	75	77
-6	10*	-4
-5	21	10
-4	21	31
-3	117	113
-2	105	-109
-1	120	-113
0	74	-70
1	184	187
2	213	-219
3	10*	-19
4	79	77
5	58	56
6	21	-19
-3,7,L		
-7	58	62
-6	18*	24
-5	166	-172
-4	9*	-1
-3	57	60
-2	137	133
-1	51	-56
0	166	164
1	105	105
2	93	91
3	49	-47
4	11*	7
5	77	74
6	100	103
-3,8,L		
-7	25	-31
-6	84	-88
-5	18*	6
-4	139	-139
-3	26	-27
-2	36	-35
-1	110	101
0	23	10
1	27	-27
2	28	-30
3	51	58
4	50	-48
5	73	-77
-3,9,L		
-7	39	-39
-6	49	-52
-5	34	31
-4	189	200
-3	115	-117
-2	106	-105
-1	17*	15
0	39	39
1	160	-160
2	129	-123
3	44	46
4	27	21
5	45	-48
-3,10,L		
-7	50	47
-6	40	-32
-5	73	78
-4	61	69
-3	129	129
-2	50	-50
-1	43	-44
0	44	38
1	18*	-16
2	18*	-17
3	12*	-3
4	41	41
-3,11,L		
-7	17	-23
-6	43	49
-5	26	-21
-4	63	-61
-3	60	-65
-2	78	77
-1	11*	12
0	88	-84
1	54	51
2	81	84
3	25	25
-3,12,L		
-6	20	-7
-5	62	61
-4	50	-66
-3	80	-76
-2	10*	15
-1	60	64
0	26	24
1	56	-55
2	61	60
-3,13,L		
-5	11*	2
-4	33	32
-3	9*	5
-2	36	-34
-1	21	17
0	22	17
1	8*	11
-3,14,L		
-4	9*	-5
-3	31	34
-2	8*	9
-1	54	-55
-2,-13,L		
2	9*	-2
3	24	-28
4	9*	-4
5	69	70
6	82	-83
-2,-12,L		
1	19	4
2	44	44
3	47	-48
4	32	-25
5	41	-44
6	143	143
7	75	-75
-2,-11,L		
-1	15*	16
0	31	-34
1	18	-16
2	40	-34
3	100	100
4	74	-74
5	42	-39
6	67	-66
7	121	124
8	44	-43
-2,-10,L		
-2	27	-23
-1	35	-34
0	83	81
1	43	-43
2	16*	4
3	26	-21
4	138	138
5	90	-90
6	10*	-15
7	67	-65
8	97	100
-2,-9,L		
-2	56	-61
-1	55	-56
0	17*	-26
1	143	139
2	139	-140
3	25	-29
4	80	-81
5	151	159
6	60	55
7	25	-30
8	46	-46
-2,-8,L		
-3	30	-20
-2	65	65
-1	20*	-25
0	107	105
1	29	-34
2	187	187
3	85	-85
4	71	-67
5	74	69
6	214	220
7	39	42
8	55	-57
9	6*	-2
-2,-7,L		
-4	9*	-4
-3	51	-53
-2	83	-77
-1	225	226
0	11*	18
1	32	-35
2	92	-90
3	266	257
4	26	21
5	109	-102
6	47	-51
7	97	-94
8	36	32
9	33	-32
-2,-6,L		
-4	76	75
-3	10*	-12
-2	51	-47
-1	115	-113
0	123	126
1	345	-325
2	247	-236
3	60	-65
4	107	112
5	55	58
6	388	-392
7	179	-177
8	10*	0
9	52	56
-2,-5,L		
-5	35	38
-4	14*	7
-3	113	115
-2	44	42
-1	31	23
0	290	-280
1	71	73
2	95	91
3	192	-189
4	9*	7
5	60	67
6	143	143
7	89	-86
8	165	160
9	43	41
-2,-4,L		
-5	10*	-4
-4	49	-45
-3	65	-62
-2	126	123
-1	63	70
0	26	-27
1	10	-14
2	559	533
3	422	394
4	292	-246
5	24	20
6	38	36
7	345	357
8	63	61
9	25	-22
-2,-3,L		
-6	41	-37
-5	47	42
-4	59	-55
-3	109	-107
-2	125	-120
-1	54	53
0	92	97
1	9*	5
2	55	46
3	61	-60
4	255	257
5	295	-291
6	48	-47
7	12*	6
8	54	55
9	153	-156
-2,-2,L		
-6	10*	-12
-5	34	-42
-4	136	133
-3	78	71
-2	151	-158
-1	51	57
0	69	66
1	212	214
2	284	-290
3	187	-182
4	79	-79
5	297	298
6	136	-128
7	44	-46
8	110	-110
9	24	-30
-2,-1,L		
-7	30	34
-6	22	8
-5	28	-32
-4	66	-63
-3	69	62
-2	158	170
-1	262	-271
0	43	-41
1	204	-208
2	180	174
3	351	-354
4	9*	-6
5	66	-67
6	157	162
7	43	-35
8	66	-65
-2,0,L		
-7	58	-54
-6	43	45
-5	63	64
-4	81	-82
-3	140	-144
-2	49	49
-1	511*	545
0	753*	-803
1	116	-121
2	58	53
3	286	302
4	111	-118
5	84	-77
6	44	-47
7	85	78
8	10*	-7
-2,1,L		
-7	10*	5
-6	46	-44
-5	60	69
-4	125	126
-3	200	-206
-2	106	117
-1	77	74
0	550*	611
1	265	-279
2	8*	25
3	14*	-17
4	219	219
5	35	-35
6	46	-44
7	31	27
8	25	24
-2,2,L		
-7	42	43
-6	13*	-5
-5	45	-45
-4	114	-110
-3	143	154
-2	217	-220
-1	161	-168
0	390*	-411
1	724*	786
2	210	-208
3	279	-285
4	55	50
5	69	72
6	9*	3
7	47	-44
8	6*	8
-2,3,L		
-8	26	-29
-7	27	-19
-6	100	94
-5	24	-25
-4	86	-85
-3	20	-30
-2	620	629
-1	349	-367
0	231	-249
1	30	-30
2	376	386
3	205	198
4	20	-2
5	49	51
6	35	34
7	16*	15
-2,4,L		
-8	92	-95
-7	43	-52
-6	10*	-13
-5	86	89
-4	106	-104
-3	199	179
-2	142	128
-1	379	390
0	57	-60
1	98	-109
2	133	-125
3	240	230
4	139	142
5	198	-200
6	25	18
7	36	35
-2,5,L		
-8	27	20
-7	42	-38
-6	22	-14
-5	102	-100
-4	73	69
-3	11*	-17
-2	177	-168
-1	437	-426
0	197	203
1	44	44
2	297	-307
3	141	-128
4	182	-172
5	85	-79
6	90	-91
7	8*	12
-2,6,L		
-8	17	21
-7	192	190
-6	10*	-8
-5	58	-56
-4	9*	7
-3	140	145
-2	272	-249
-1	379	-378
0	8*	6
1	162	161
2	115	120
3	130	-134
4	89	-88
5	9*	-6
6	111	115
-2,7,L		
-8	10*	11
-7	10*	-7
-6	117	120
-5	29	-30
-4	57	-52
-3	84	63
-2	53	56
-1	130	121
0	21	-8
1	31	32
2	52	53
3	152	155
4	16*	0
5	128	131
6	91	97
-2,8,L		

final refinement resulted in a residual, R, of 0·046 and a weighted R value of 0·052.

The atomic scattering factors used for chlorine, sulfur and carbon atoms were those in *International Tables for X-ray Crystallography* (1962) and for hydrogen atoms those of Stewart, Davidson & Simpson (1965). The real component of anomalous dispersion correction for Cl and S, 0·348 and 0·319 respectively, are those listed by Cromer & Liberman (1970). Computer programs used were the X-ray 70 System (Stewart, Kundell & Baldwin, 1970), *UCLALS*4 (Full-Matrix Least-Squares) (Gantzel, Sparks, Long & Trueblood, 1969), modified by H. L. Carrell and 'A Distance and Angle Program' (Shiono, 1971).

The final atomic parameters are in Table 2 and the corresponding structure factors in Table 3. Two views of the molecule with thermal ellipsoids are illustrated in Fig. 1 (Johnson, 1965).

Discussion of the structure

The interatomic distances and interbond angles in this molecule are shown in Fig. 2. A comparison of the distances found with those given for anthracene (Cruickshank, 1956), 9,10-bis(chloromethyl)anthracene (Gabe & Glusker, 1971) and 10-chloromethyl-2,3,9-trimethylanthracene (Chomyn, Glusker, Berman & Carrell, 1972) is shown in Table 4. The C–Cl distance 1·792 Å is intermediate between values of 1·768 and 1·779 Å listed by Karle & Estlin (1969) for β-chloroethyl triptycene and values of 1·810 and 1·806 Å for 9,10-bis(chloromethyl)anthracene and 10-chloromethyl-2,3,9-trimethylanthracene respectively. The two C–S bonds are 1·806 and 1·819 Å, the one nearer the ring being the longer by four estimated standard deviations of each bond length.

The deviations of atoms from various planes through the molecule are shown in Table 5. The total ring system is not planar, as seen in Figs. 1(*b*) and 3, but buckled by 8·7° so that one 'end' of the anthracene molecule is 0·5 Å from the plane of the ring at the other 'end' (see planes 2 and 3) although each of the two outer benzene rings is nearly planar. The value of 8·7° may be compared with values of 1·2° for 10-chloromethyl-2,3,9-trimethylanthracene (Chomyn *et al.*, 1972), possibly a typical value for an anthracene derivative, and 14·1° for 1:2,5:6-dibenzanthraquinone (Entwistle, Iball, Motherwell & Thompson, 1969). The non-planarity of the ring system seems to be

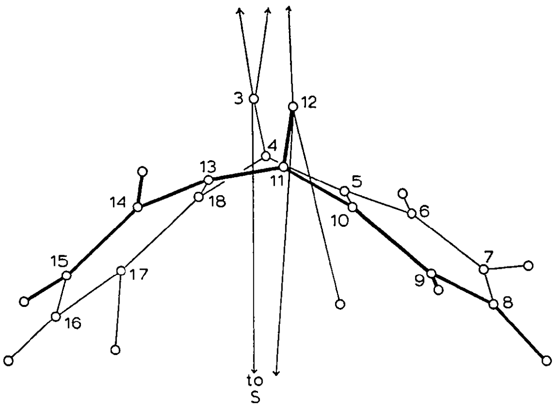
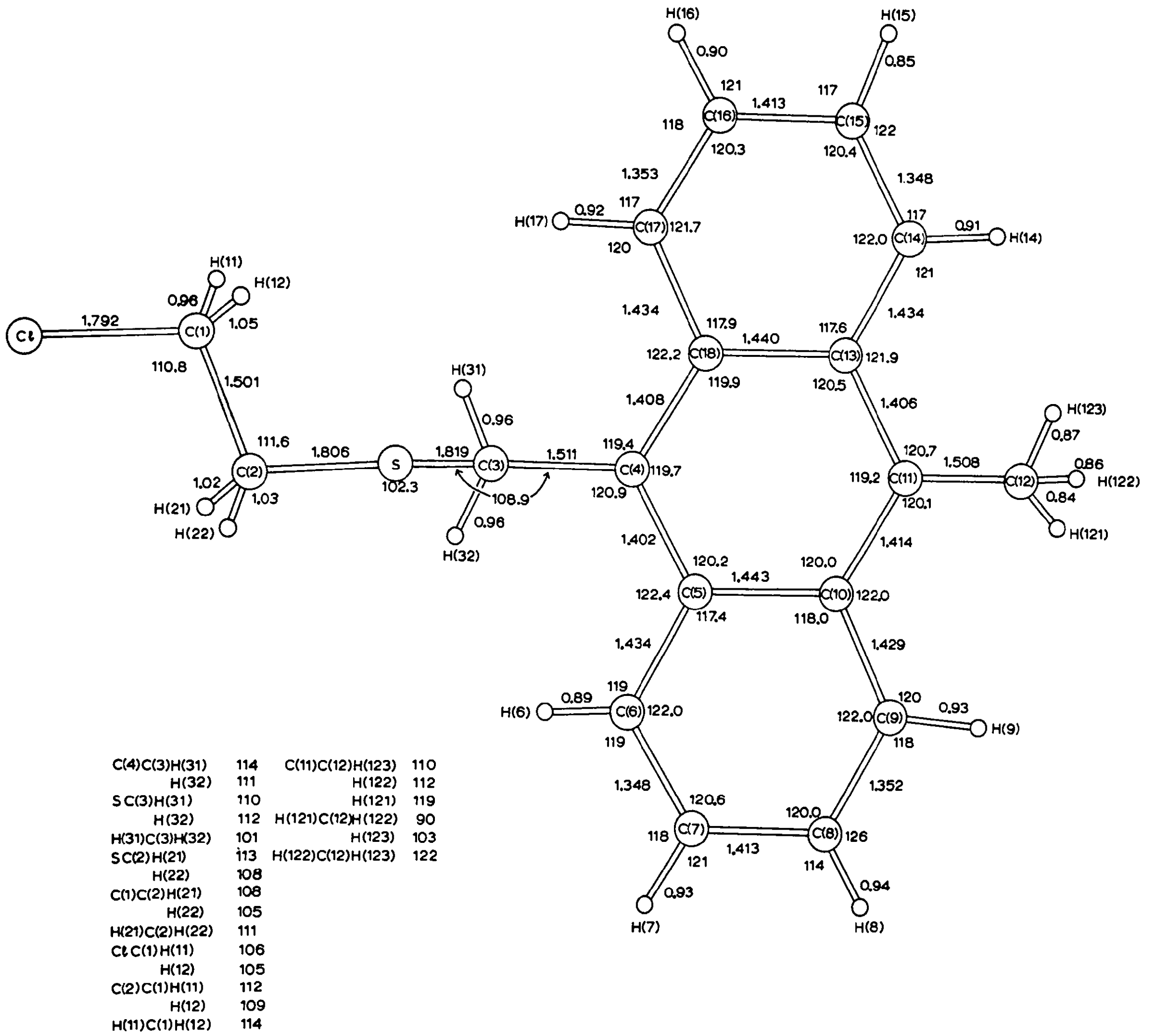


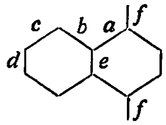
Fig. 3. Schematic diagrams of the buckling of the ring system. The vertical scale is exaggerated ten times.



C(4)C(3)H(31)	114	C(11)C(12)H(123)	110
H(32)	111	H(122)	112
SC(3)H(31)	110	H(121)	119
H(32)	112	H(121)C(12)H(122)	90
H(31)C(3)H(32)	101	H(123)	103
SC(2)H(21)	113	H(122)C(12)H(123)	122
H(22)	108		
C(1)C(2)H(21)	108		
H(22)	105		
H(21)C(2)H(22)	111		
ClC(1)H(11)	106		
H(12)	105		
C(2)C(1)H(11)	112		
H(12)	109		
H(11)C(1)H(12)	114		

Fig. 2. Interatomic distances and interbond angles. E.s.d. values are 0·003 Å for C–S and C–Cl, 0·004 Å for C–C, 0·03 Å for C–H, 0·1° for the C–S–C angle, 0·3° for C–C–C angles and 5° for H–C–H angles.

Table 4. *A comparison of the bond lengths in anthracene and in* ICR-358



	Anthracene†	ICR-450‡	ICR-489§	ICR-358 (This work)
e.s.d. (Å)	0·004	0·002	0·002	0·003
(*a*)	1·397	1·412	1·414	1·402
	1·396	1·407	1·399	1·408
			1·410	1·406
			1·411	1·414
(*b*)	1·435	1·432	1·428	1·434
	1·427	1·440	1·436	1·429
			1·429	1·434
			1·429	1·434
(*c*)	1·360	1·355	1·348	1·353
	1·368	1·351	1·353	1·348
			1·350	1·348
			1·340	1·352
(*d*)	1·417	1·397	1·395	1·413
				1·413
(*e*)	1·434	1·428	1·434	1·443
			1·442	1·440
(*f*)		1·485*	1·493*	1·511
			1·507	1·508

* C–CH_2Cl
† Cruickshank, 1956.
‡ Gabe & Glusker, 1971.
§ Chomyn, Glusker, Berman & Carrell, 1972.

caused by factors affecting the central ring rather than the outer rings. From a comparison with bond angles and bond lengths in 9,10-bis(chloromethyl)anthracene and 10-chloromethyl-2,3,9-trimethylanthracene it appears that the tendency for each carbon atom in the central ring system to have a planar configuration of atoms around it overrides the tendency for the central ring system to be planar.

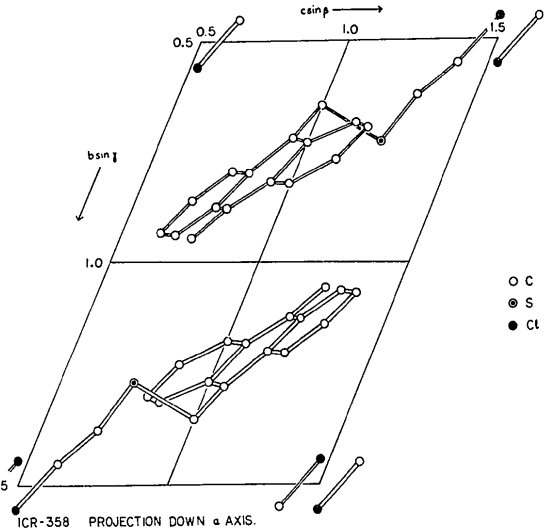


Fig. 4. Packing of molecules in the unit cell viewed down **a**.

Table 5. *Some least squares planes through parts of the molecule*

An asterisk denotes atoms used to calculate the planes. All deviations from the planes are in Å.

	Plane 1	Plane 2	Plane 3	Plane 4	Plane 5
Cl	−0·808	−0·556	−1·291	*−0·044	−0·925
C(1)	−0·947	−0·773	−1·361	* 0·043	−1·058
C(2)	−1·144	−1·089	−1·458	* 0·045	−1·241
S	−1·461	−1·483	−1·705	*−0·044	−1·551
C(3)	0·219	0·165	−0·005	1·698	0·131
C(4)	* 0·123	0·012	−0·049	1·782	* 0·040
C(5)	* 0·064	−0·163	*−0·012	1·932	*−0·007
C(6)	* 0·027	−0·268	* 0·005	1·950	−0·035
C(7)	*−0·066	−0·472	* 0·004	2·059	−0·117
C(8)	*−0·124	−0·582	*−0·006	2·167	−0·170
C(9)	*−0·072	−0·469	*−0·001	2·176	−0·126
C(10)	* 0·037	−0·243	* 0·010	2·075	*−0·030
C(11)	* 0·108	−0·109	0·032	2·101	* 0·033
C(12)	0·205	−0·069	0·179	2·373	0·135
C(13)	* 0·086	*−0·015	−0·086	1·870	*−0·001
C(14)	* 0·040	* 0·008	−0·185	1·770	−0·056
C(15)	*−0·073	* 0·006	−0·390	1·459	−0·180
C(16)	*−0·144	*−0·013	−0·509	1·223	−0·256
C(17)	*−0·065	* 0·006	−0·383	1·342	−0·169
C(18)	* 0·056	* 0·008	−0·165	1·671	*−0·036

Deviations of hydrogen atoms from Plane 1.

H(11)	−0·14	H(32)	+0·74	H(9)	−0·10	H(14)	+0·10
H(12)	−1·81	H(6)	+0·06	H(121)	−0·35	H(15)	−0·12
H(21)	−0·32	H(7)	−0·06	H(122)	−0·12	H(16)	−0·22
H(22)	−1·98	H(8)	−0·22	H(123)	+0·97	H(17)	−0·20
H(31)	+0·73						

Equations of the planes

Plane	
1	$-0·18607X+0·80176Y+0·56794Z=\ 1·74295$
2	$-0·21949X+0·83705Y+0·50117Z=\ 2·02159$
3	$-0·16016X+0·76719Y+0·62111Z=\ 1·76265$
4	$-0·17508X+0·70275Y+0·68956Z=-0·31926$
5	$-0·18141X+0·79808Y+0·57460Z=\ 1·80916$

Where X, Y, and Z are coordinates in orthogonal Ångstrom space with X measured parallel to **a**, Y perpendicular to **a** in the plane of **a** and **c**, and Z perpendicular to the plane of **a** and **c**.

The packing of the molecules in the unit cell is illustrated in Fig. 4 and the surroundings of the sulfur and chlorine atoms are shown in Fig. 5. The sulfur atom is near $C(9^{III})$ at 3·39 Å and $H(9^{III})$ at 3·08 Å. The chlorine atom packs with $C(4^{III})$, $C(6^{VIII})$, $C(14^{IV})$ and $C(16^{VII})$ of different space group-related molecules around it.

In Fig. 6 the extent of overlap of some molecules is illustrated. It can be seen that two of the three rings (of molecules I and II) directly overlap each other (distance 3·6 Å apart) so that the methyl group also is overlapped by an adjacent molecule. The side chains then stack above (from molecule IV) and below these pairs of molecules.

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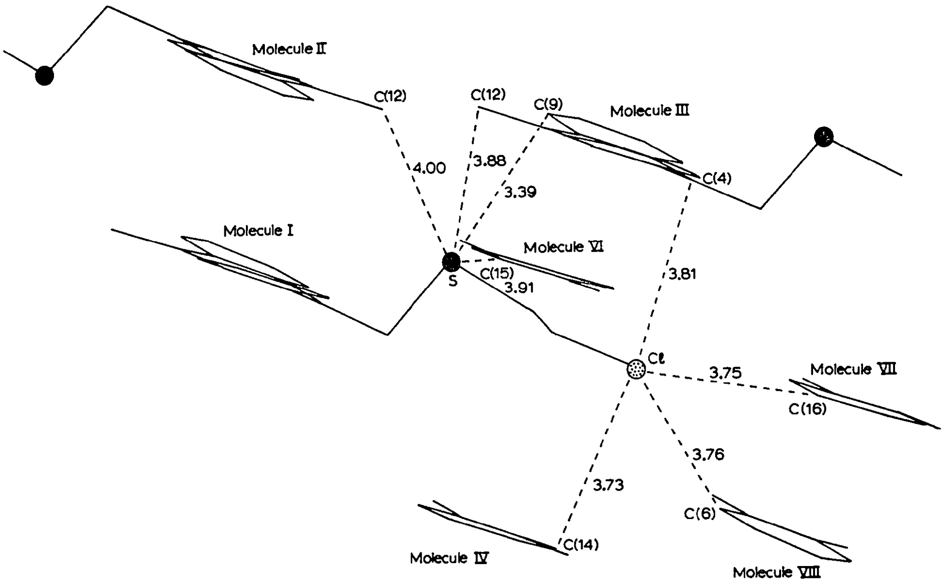

Fig. 5. Atomic surroundings of chlorine and sulfur atoms.

Code:

I	x	y	z
II	$-x$	$-y$	$-z$
III	x	y	$z-1$
IV	$-x$	$1-y$	$-z$
V	x	y	$z+1$
VI	$x+1$	y	z
VII	$-x$	$1-y$	$-z-1$
VIII	$1-x$	$1-y$	$-z$

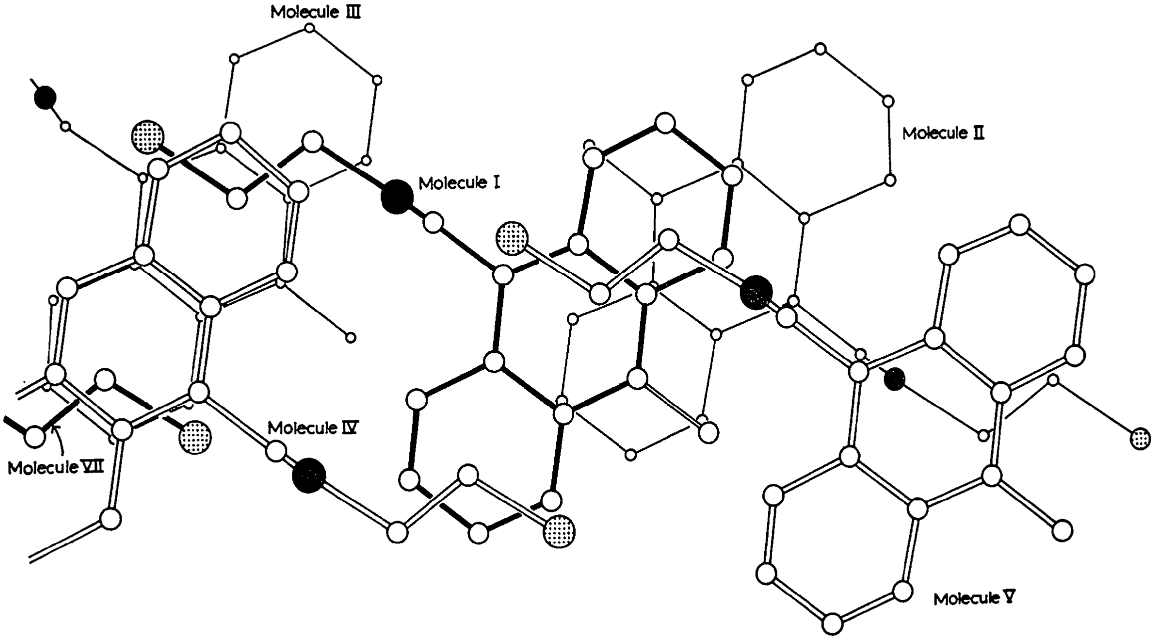

Fig. 6. View of molecular packing perpendicular to the least-squares plane through the central ring system of molecule I. Molecules IV and V lie above this plane and molecules II and III lie below it.

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The Crystal and Molecular Structure of Potassium Pentachloronitrosylruthenate(II), $K_2[Ru(NO)Cl_5]$

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The structure of potassium pentachloronitrosylruthenate(II), $K_2Ru(NO)Cl_5$, has been refined by least-squares techniques on three-dimensional X-ray data collected by counter methods. The material crystallizes in the space group $P2_12_12_1$ (D_2^4) of the orthorhombic system with four molecules in a cell of dimensions a = 10·363(4), b = 13·292(10), and c = 6·880(4) Å. The observed and calculated densities are 2·70 (2) and 2·729 g.cm^{-3} respectively. Least-squares refinement of the structure has yielded a final value for the conventional R value (on F) of 0·040 for 1347 independent reflections having $F^2 > 3\sigma(F^2)$. The $[Ru(NO)Cl_5]^{2-}$ anion is a slightly distorted octahedron with the Ru–Cl bond length of 2·359(2) Å *trans* to the nitrosyl group shorter than the average *cis* Ru–Cl bond length of 2·372 (8) Å. The Ru–N–O group is approximately linear, with an Ru–N–O bond angle of 176·8 (9)° and Ru–N and N–O bond distances of 1·747 (6) and 1·112 (7) Å, respectively. These values suggest that the nitrosyl group is bonded as NO^+. The coordination around the Ru atom is similar to that found in the isomorphous ammonium salt of this anion.

Introduction

The structure of potassium pentachloronitrosylruthenate(II), $K_2Ru(NO)Cl_5$, has been previously investigated (Khodashova & Bokii, 1960). On the basis of their photographic data, these workers assigned the complex to the space group *Pnma* and reported an unusually long N–O bond length of 1·25 Å. Recently completed structural work on the ammonium salt of this anion (Veal & Hodgson, 1972) gives different values for the bond parameters of the anion, demonstrating a significant shortening of the Ru–Cl bond *trans* to the nitrosyl and a more reasonable N–O bond length of 1·131 (3) Å. Preliminary work in our laboratory suggested that the potassium salt was isomorphous with the ammonium salt and that the correct space group for both was $P2_12_12_1$ rather than *Pnma*.

A detailed infrared study of this complex (Durig, McAllister, Willis & Mercer, 1966) has been reported. These workers conclude from vibrational spectroscopy that in ions of the type $[Ru(NO)X_5]^{2-}$ the Ru–X bond (X= halide) *trans* to the nitrosyl is weaker than the bonds *cis* to the nitrosyl. Recently reported molecular orbital calculations for the complexes $Mn(CO)_5X$ (X= Cl, Br, I), however, have shown (Fenske & DeKock, 1970) that the M–X bond is entirely σ in nature, and that the halogen successfully competes with the carbonyl for σ electron density. Since CO and NO^+ are isoelectronic and Cl^- is a better σ bonder than NO^+, arguments of this type would predict that the Ru–Cl bond *trans* to the NO^+ should be strengthened rather than weakened. The structure of the ammonium salt (Veal & Hodgson, 1972), $(NH_4)_2Ru(NO)Cl_5$, is con-

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