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The Crystal and Molecular Structure of Unsolvated μ-Oxo-bis-[*N*,*N*′-ethylenebis(salicylaldiminato)iron (III)]

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The crystal and molecular structure of unsolvated [Fe(salen)]$_2$O (salen = *N*,*N*′-ethylenebis(salicylaldiminato) anion) has been determined by single crystal X-ray analysis and refined to an R of 0·052 with diffractometer data. The crystals are triclinic, space group $P\bar{1}$, with $Z=2$ and $a=10{\cdot}949$, $b=11{\cdot}064$, $c=13{\cdot}812$ Å, $\alpha=112{\cdot}10$, $\beta=106{\cdot}85$, $\gamma=77{\cdot}41°$. The molecular conformation is very similar to that observed for the same molecule in the two solvated crystal structures [Fe(salen)]$_2$O.2py and [Fe(salen)]$_2$O.CH$_2$Cl$_2$. The Fe–O–Fe bridge is symmetrical within experimental error with an Fe–O (bridge) bond length of 1·78 Å and an Fe–O–Fe angle of 145°.

Introduction

Considerable attention has recently been given to oxygen-bridged iron(III) complexes of the type LFe–O–FeL where the attached ligand (L) can be a Schiff base, porphyrin, diimine or HEDTA. The complexes behave as discrete binuclear clusters, and the large anti-ferromagnetic exchange between the two iron(III) centres appears to be chiefly a property of the Fe–O–Fe bridge but virtually independent of the nature of L ($2J = c.a - 200$ cm^{-1} in all cases).

The crystal structures of several molecules containing such an Fe–O–Fe bridging arrangement have now been determined and these are documented in Table 1. Two solvated structures of [Fe(salen)]$_2$O have been reported (one with pyridine and the other with dichloromethane as lattice solvent) and in both cases the Fe–O–Fe angle is approximately 140°, considerably less than the Fe–O–Fe angles in the other structures listed in Table 1 which range from 164–175°. From the now considerable data available it is still not clear what controls the Fe–O–Fe angle in a given compound, for ligand repulsions, electronic and crystal-packing effects can all conceivably play an important role. The effect of crystal packing can best be investigated by examining the same molecule in different packing arrangements and for this reason we have determined the crystal structure of unsolvated [Fe(salen)]$_2$O. There have been numerous previous attempts to obtain single-crystal samples of this unsolvated dimer in order to make a rigorous interpretation of its electronic structure. However all such attempts were unsuccessful and comparisons of its spectral and magnetic properties have perforce been made with the solvated structures. The single crystals of unsolvated [Fe(salen)]$_2$O were obtained here by hydrolysis of Fe(salen)acetate in methanol, the method of preparation first used by Pfeiffer, Breith, Lübbe & Tsumaki (1933).

Experimental

An attempt by Mr A. van den Bergen of this Department to recrystallize slowly from methanol a sample of Fe(salen)acetate prepared by the method of Lewis, Mabbs, Richards & Thornley (1969), resulted in large, beautifully formed, deep red, air-stable crystals. Subsequent chemical analysis proved that the material was unsolvated [Fe(salen)]$_2$O (calc. C, 58·21, H, 4·27, N, 8·49%; found: C, 58·13, H, 4·44, N, 8·39%) and its formation can be rationalized in terms of the equilibrium 2Fe(salen) acetate + H$_2$O $\rightleftharpoons$ 2CH$_3$COOH + [Fe(salen)]$_2$O. As [Fe(salen)]$_2$O is relatively insoluble in methanol, the slow absorption of water from the atmosphere causes crystals of [Fe(salen)]$_2$O to be gradually deposited from the solution.

Preliminary rotation, oscillation and Weissenberg photographs taken with Cu $K\alpha$ radiation indicated that the crystals were triclinic. The unit-cell parameters were obtained with one of the standard programs of

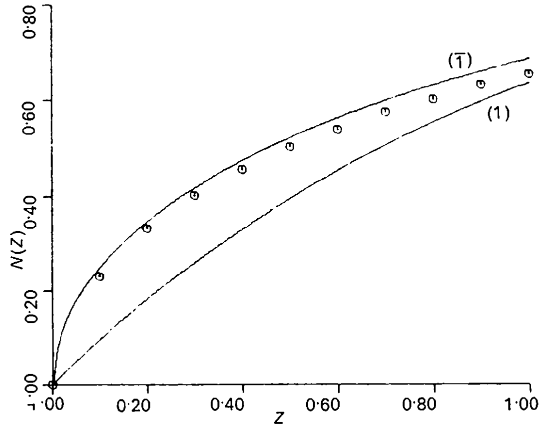

Fig. 1. Zero moment test for [Fe(salen)]$_2$O compared with the theoretical curves for centric ($\bar{1}$) and acentric (1) distributions.

Table 1. Fe–O–Fe *geometries**

	Fe–O–Fe (°)	d (Å)†	Fe–O (Å)		Reference
$[\mathrm{Fe(salen)}]_2\mathrm{O}\,.\,2\mathrm{py}$	139·1	0·57 0·55	1·820 1·773		Gerloch, McKenzie & Towl (1969)
$[\mathrm{Fe(salen)}]_2\mathrm{O}\,.\,\mathrm{CH_2Cl_2}$	142·4	0·57 0·55	1·791 1·797	(i) (ii)	Coggon, McPhail, Mabbs & McLachlan (1971) Atovmyan, D'yachenko & Soboleva (1970)
$[\mathrm{Fe(HEDTA)}]_2\mathrm{O}^{2-}$	165·0	0·36	1·80 1·79		Lippard, Schugar & Walling (1967)
$[\mathrm{Fe(TPP)}]_2\mathrm{O}$	174·5	0·50	1·763		Hoffman, Collins, Day, Fleischer, Srivastava & Hoard (1972)
[Fe(*N*-n-propyl-sal)$_2$]$_2$O	164	0·51 0·55	1·78 1·76		Davies & Gatehouse (1972)
[Fe(*N*-*p*-chlorophenyl-sal)$_2$]$_2$O	175	0·54 0·52	1·76 1·77		Davies & Gatehouse (1973)

* HEDTA = *N*-Hydroxyethyl-ethylenediaminetriacetato anion. TPP = $\alpha,\beta,\gamma,\delta$-Tetraphenylporphinato anion. sal = Salicylaldiminato anion.

† d = displacement of the iron atoms from the coordination plane.

Table 2. *Final positional and thermal parameters**

Numbers in parentheses here and in succeeding tables are estimated standard deviations in the least significant digits. The B values are the parameters in the isotropic temperature factor expression $\exp[-B(\sin\theta)^2/\lambda^2]$. The U_{ij} are $\times 10^2$ and the atomic scattering factors for Fe*A*, Fe*B* are expressed as $f=f_0 \exp[-2\pi^2(U_{11}a^{*2}h^2+\ldots+2U_{23}b^*c^*kl+\ldots)]$.

(*a*) Isotropic temperature factors

	x/a	y/b	z/c	B		x/a	y/b	z/c	B
O*A*	−0·0446 (9)	0·1660 (9)	0·3510 (7)	4·0 (2)	O*B*	0·5042 (9)	0·2052 (9)	0·5618 (7)	4·2 (2)
C(1)*A*	−0·0624 (15)	0·0720 (15)	0·2561 (12)	4·3 (4)	C(1)*B*	0·5342 (14)	0·2208 (15)	0·6656 (12)	4·1 (3)
C(2)*A*	−0·1523 (18)	0·1037 (18)	0·1710 (14)	6·0 (4)	C(2)*B*	0·6148 (15)	0·1184 (16)	0·6991 (13)	4·8 (4)
C(3)*A*	−0·1768 (21)	0·0046 (21)	0·0666 (17)	8·1 (6)	C(3)*B*	0·6513 (18)	0·1375 (18)	0·8107 (14)	6·1 (4)
C(4)*A*	−0·1108 (21)	−0·1226 (22)	0·0552 (17)	8·3 (6)	C(4)*B*	0·6095 (18)	0·2508 (18)	0·8864 (14)	6·2 (5)
C(5)*A*	−0·0249 (19)	−0·1559 (19)	0·1388 (15)	6·9 (5)	C(5)*B*	0·5246 (18)	0·3503 (18)	0·8534 (15)	6·3 (5)
C(6)*A*	0·0014 (15)	−0·0556 (16)	0·2427 (12)	4·7 (4)	C(6)*B*	0·4842 (15)	0·3363 (15)	0·7410 (12)	4·4 (4)
C(7)*A*	0·0847 (15)	−0·0968 (15)	0·3275 (13)	4·6 (4)	C(7)*B*	0·3946 (15)	0·4410 (15)	0·7132 (12)	4·2 (4)
N*A*	0·1246 (11)	−0·0221 (12)	0·4226 (9)	4·1 (3)	N*B*	0·3463 (11)	0·4362 (11)	0·6158 (9)	3·7 (3)
C(8)*A*	0·2083 (15)	−0·0823 (16)	0·5033 (13)	4·9 (4)	C(8)*B*	0·2494 (15)	0·5484 (15)	0·5949 (12)	4·5 (4)
O′*A*	0·0264 (9)	0·3415 (9)	0·5600 (7)	4·2 (2)	O′*B*	0·4644 (9)	0·2168 (9)	0·3561 (7)	3·7 (2)
C(1′)*A*	0·0509 (14)	0·4070 (14)	0·6650 (12)	3·8 (3)	C(1′)*B*	0·4415 (14)	0·2431 (14)	0·2663 (11)	3·8 (3)
C(2′)*A*	−0·0075 (16)	0·5370 (16)	0·6996 (13)	4·8 (4)	C(2′)*B*	0·4970 (16)	0·1501 (16)	0·1804 (13)	5·1 (4)
C(3′)*A*	0·0151 (18)	0·6094 (18)	0·8095 (15)	6·5 (5)	C(3′)*B*	0·4782 (17)	0·1760 (18)	0·0848 (14)	5·8 (4)
C(4′)*A*	0·0934 (20)	0·5534 (20)	0·8847 (16)	7·7 (5)	C(4′)*B*	0·4037 (18)	0·2951 (18)	0·0734 (14)	6·1 (4)
C(5′)*A*	0·1548 (20)	0·4221 (20)	0·8524 (16)	7·3 (5)	C(5′)*B*	0·3522 (17)	0·3858 (17)	0·1558 (14)	5·6 (4)
C(6′)*A*	0·1346 (15)	0·3487 (16)	0·7390 (13)	4·8 (4)	C(6′)*B*	0·3715 (14)	0·3612 (14)	0·2541 (11)	3·9 (3)
C(7′)*A*	0·2065 (15)	0·2205 (16)	0·7105 (13)	4·8 (4)	C(7′)*B*	0·3269 (14)	0·4656 (14)	0·3386 (12)	3·9 (3)
N′*A*	0·2044 (11)	0·1481 (11)	0·6128 (9)	4·1 (3)	N′*B*	0·3319 (11)	0·4622 (11)	0·4306 (9)	3·7 (3)
C(8′)*A*	0·2893 (16)	0·0193 (16)	0·5908 (13)	5·1 (4)	C(8)′*A*	0·2903 (14)	0·5862 (15)	0·5129 (12)	4·3 (4)
Oxo	0·2298 (9)	0·2181 (9)	0·4268 (7)	3·7 (2)					

(*b*) Anisotropic temperature factors

	x/a	y/b	z/c	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Fe*A*	0·1049 (2)	0·1851 (2)	0·4687 (2)	3·31 (13)	3·35 (13)	4·90 (14)	0·01 (10)	1·22 (11)	1·68 (11)
Fe*B*	0·3762 (2)	0·2883 (2)	0·4717 (2)	3·36 (13)	3·33 (13)	4·48 (14)	−0·06 (10)	1·15 (11)	1·64 (11)

Table 3. *Idealized positional coordinates for the hydrogen atoms**

	x/a	y/b	z/c		x/a	y/b	z/c
HC(2)*A*	−0·201	0·199	0·183	HC(2)*B*	0·647	0·031	0·643
HC(3)*A*	−0·242	0·026	0·001	HC(3)*B*	0·712	0·063	0·837
HC(4)*A*	−0·128	−0·196	−0·021	HC(4)*B*	0·641	0·262	0·968
HC(5)*A*	0·021	−0·253	0·127	HC(5)*B*	0·490	0·435	0·911
HC(7)*A*	0·116	−0·198	0·310	HC(7)*B*	0·368	0·524	0·775
HC(8)*A*	0·151	−0·113	0·537	HC(8)*B*	0·157	0·519	0·563
HC(8)*A*	0·269	−0·164	0·466	HC(8)*B*	0·250	0·629	0·666
HC(2′)*A*	−0·068	0·580	0·644	HC(2′)*B*	0·552	0·063	0·189
HC(3′)*A*	−0·028	0·707	0·835	HC(3′)*B*	0·519	0·108	0·022
HC(4′)*A*	0·107	0·609	0·967	HC(4′)*B*	0·388	0·314	0·001
HC(5′)*A*	0·213	0·380	0·909	HC(5′)*B*	0·299	0·473	0·147
HC(7′)*A*	0·263	0·185	0·772	HC(7′)*B*	0·286	0·554	0·324
HC(8′)*A*	0·368	0·026	0·565	HC(8′)*B*	0·367	0·643	0·552
HC(8′)*A*	0·323	−0·007	0·661	HC(8′)*B*	0·212	0·640	0·476

* The hydrogen atoms are labelled by placing an H before the label of the associated carbon atom.

a Philips PW1100 computer-controlled X-ray diffractometer. This program investigates rows in the reciprocal lattice through the origin and scans the four most intense reflexions in the positive and negative θ region. The centres of gravity of these eight profiles are used in a least-squares refinement of the *d* spacing for that row.

Crystal data

$C_{32}Fe_2H_{28}N_4O_5$, $M=660{\cdot}4$, triclinic, $a=10{\cdot}949$ (11), $b=11{\cdot}064$ (11), $c=13{\cdot}812$ (14) Å, $\alpha=112{\cdot}10$ (10), $\beta=106{\cdot}85$ (10), $\gamma=77{\cdot}41$ (10)°. $U=1473$ Å^3, $D_m=1{\cdot}49$ g cm^{-3} (by flotation), $Z=2$, $D_c=1{\cdot}49$ g cm^{-3}, $F(000)=680$, $\mu=10{\cdot}6$ cm^{-1} for Mo $K\alpha$ radiation ($\lambda=0{\cdot}7107$ Å). Space group $P\bar{1}$ (C_i^1).

Intensity measurements

Intensities were collected from a crystal having dimensions $0{\cdot}20\times0{\cdot}17\times0{\cdot}17$ mm with a Philips PW1100 diffractometer and Mo $K\alpha$ radiation. A unique data set was collected out to 2θ (Mo $K\alpha$) = 40°. The intensities of 2720 independent reflexions were measured; of these, 1958 obeyed the condition $F_o^2>3\sigma(F_o^2)$, and only these were used in subsequent calculations. Three standard reflexions, monitored at two-hourly intervals, showed no significant variations in intensity.

The data were collected by the θ–2θ scan technique with a symmetric scan range of $\pm0{\cdot}8°$ in 2θ from the calculated scattering angle. The scan rate was 0·05° per sec. The Mo $K\alpha$ radiation was monochromated with a flat graphite monochromator crystal and no reflexion was sufficiently strong to require the insertion of an attenuation filter.

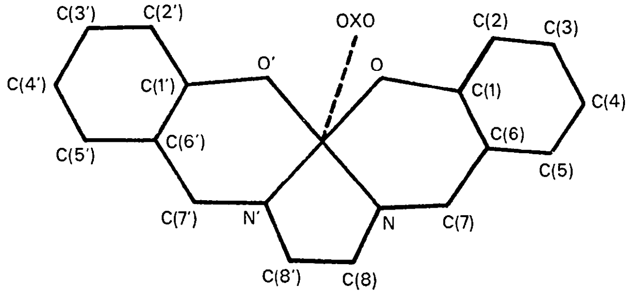

Fig. 2. [Fe(salen)]$_2$O. Labelling of the atoms for one half of the dimer. An *A* or *B* suffix is added to the labels in Table 2 to distinguish between the two chemically equivalent salen ligands.

The intensities were processed with a program written specifically for the PW1100 diffractometer (Hornstra & Stubbe, 1972). The background-corrected intensities were assigned standard deviations according to the formula

$$\sigma(I)=[\mathrm{CT}+(t_c/t_b)^2\,(B_1+B_2)+(pI)^2]^{1/2}$$

where CT is the total integrated peak count obtained in a scan time t_c, B_1 and B_2 are background counts each obtained in time t_b, and

$$I=\mathrm{CT}-(t_c/t_b)\,(B_1+B_2)\,.$$

The value of p was selected as 0·04 to prevent unduly high weight being given to strong reflexions. The values of I and $\sigma(I)$ were then corrected for Lorentz and polarization effects in the normal fashion.

Neither extinction nor absorption corrections were applied. For the crystal used, μR varies between 0·21 and 0·18 and the estimated maximum effect on the intensities caused by neglect of absorption is less than 4%.

Structure solution and refinement

The structure was solved by standard vector methods. In the least-squares calculations discussed here, the function $\sum w(|F_o|-|F_c|)^2$ was minimized, where $|F_o|$ and $|F_c|$ are the observed and calculated structure amplitudes and w is the weight of each individual reflexion.

A zero-moment test (Howells, Phillips & Rogers, 1950) was performed on the intensities and the result (see Fig. 1) strongly indicates a centrosymmetric structure. This was confirmed by the subsequent structure solution. Assuming space group $P\bar{1}$ and an Fe–Fe distance of 3·4 Å, the three-dimensional Patterson map was solved in a straightforward manner for the two iron atom positions. A structure-factor calculation based on these initial iron atom coordinates gave $R_1=40{\cdot}4$ and $R_2=50{\cdot}2$ where

$$R_1=\sum\,||F_o|-|F_c||/\sum\,|F_o|$$

and

$$R_2=(\,\sum w(|F_o|-|F_c|)^2/\,\sum w|F_o|^2)^{1/2}.$$

The first three-dimensional difference synthesis based on the calculated phases and observed structure amplitudes gave approximate coordinates for five of the nine oxygen and nitrogen atoms. A second difference

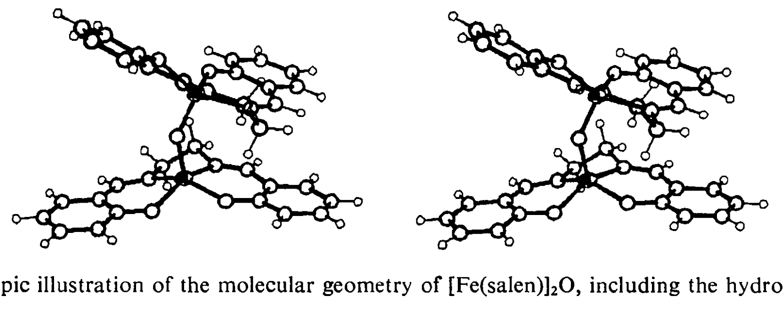

Fig. 3. Stereoscopic illustration of the molecular geometry of [Fe(salen)]$_2$O, including the hydrogen atoms.

synthesis gave the positions of all the remaining non-hydrogen atoms. Several cycles of full-matrix least-squares refinement, varying alternately the positional parameters, then isotropic temperature factors of all atoms, gave $R_1 = 0{\cdot}073$, $R_2 = 0{\cdot}086$. To allow the simultaneous refinement of all parameters with the limited

Table 4. *Observed and calculated structure factors, F_o and F_c are* × 10

Reflexions which do not obey the condition $F_o^2 > 3\sigma(F_o^2)$ are indicated by an asterisk.

H	L	FO	FC
<<<<< K = -10 >>>>>			
1	3	21	22*
1	4	0	-5*
1	5	45	5*
<<<<< K = -9 >>>>>			
1	-1	135	105
2	-1	77	80*
1	0	117	-149
2	0	97	-89
1	1	99	125
2	1	93	84
3	1	250	241
1	2	19	51*
2	2	48	-56*
3	2	268	-270
1	3	49	48*
2	3	57	2*
3	3	312	325
1	4	63	29*
2	4	35	18*
3	4	234	-227
1	5	26	8*
2	5	68	-67*
3	5	297	311
1	6	115	-137
2	6	71	77*
1	7	156	133
2	7	82	-49*
1	8	84	-109*
<<<<< K = -8 >>>>>			
1	-4	64	-33*
1	-3	87	-74
2	-3	40	-13*
3	-3	374	-364
1	-2	160	190
2	-2	34	-68*
3	-2	203	202
4	-2	26	64*
1	-1	56	94*
2	-1	35	-18*
3	-1	53	22*
4	-1	108	-131
1	0	47	-41*
2	0	178	-192
3	0	106	129
4	0	37	78*
1	1	74	-99*
2	1	344	355
3	1	163	-192
4	1	108	-153
5	1	36	-32*
1	2	33	39*
2	2	378	-318
3	2	42	64*
4	2	175	188
5	2	8	-9*
1	3	28	26*
2	3	342	312
3	3	58	67*
4	3	154	-167
1	4	82	-78
2	4	352	-369
3	4	128	-161
4	4	78	102*
1	5	73	79*
2	5	234	265
3	5	42	15*
4	5	187	-125
1	6	67	125*
2	6	47	-49*
3	6	21	-6*
4	6	48	80*
1	7	63	-19*
2	7	87	125
3	7	268	279
1	8	98	-150*
2	8	29	3*
3	8	356	-341
1	9	8	-35*
2	9	26	-5*
1	10	93	59
<<<<< K = -7 >>>>>			
1	-5	46	35*
2	-5	135	-179
3	-5	37	-12*
1	-4	198	-158
2	-4	151	207
3	-4	78	-29*
4	-4	123	-113
1	-3	37	12*
2	-3	371	-403
3	-3	76	-62*
4	-3	66	193*
5	-3	82	-98*
1	-2	154	145
2	-2	79	192*
3	-2	292	302
4	-2	111	-121
5	-2	159	156
1	-1	148	-138
2	-1	42	-1*
3	-1	459	-448
4	-1	123	117
5	-1	67	-76*
1	0	62	75*
2	0	83	-83
3	0	485	474
4	0	129	-152
5	0	4	74*
6	0	87	-74*
1	1	139	114
2	1	52	-22*
3	1	374	-359
4	1	34	-6*
5	1	42	-33*
6	1	34	5*
1	2	271	-255
2	2	64	72*
3	2	272	257
4	2	8	-78*
5	2	33	54*
6	2	41	19*
1	3	243	233
2	3	22	42*
3	3	485	-395
4	3	50	-76*
5	3	74	-82*
6	3	126	-132
1	4	158	-136
2	4	141	176
3	4	501	480
4	4	138	164
5	4	86	195*
1	5	46	-67*
2	5	79	-95*
3	5	498	-592
4	5	113	-111
5	5	198	-172
1	6	82	86*
2	6	8	-76*
3	6	298	294
4	6	50	95*
5	6	54	61*
1	7	92	-37
2	7	339	364
3	7	123	-88
4	7	50	-68*
1	8	59	35*
2	8	333	-363
3	8	62	49*
4	8	142	133
1	9	233	206
2	9	92	104
3	9	0	-13*
1	10	137	-89
2	10	214	-178
1	11	38	-24*
<<<<< K = -6 >>>>>			
1	-7	42	-48*
2	-7	103	65
1	-6	41	58*
2	-6	11	-72*
3	-6	66	-57*
4	-6	29	9*
1	-5	142	-140
2	-5	54	-96*
3	-5	236	236
4	-5	62	60*
5	-5	212	213
1	-4	133	153
2	-4	59	35*
3	-4	254	-246
4	-4	42	-74*
5	-4	132	-130
6	-4	69	-81*
1	-3	247	-253
2	-3	319	-298
3	-3	302	337
4	-3	84	-117
5	-3	142	158
6	-3	86	84*
1	-2	146	134
2	-2	488	507
3	-2	310	-374
4	-2	114	-96
5	-2	139	145
6	-2	368	-385
1	-1	145	-154
2	-1	484	-449
3	-1	117	84
4	-1	224	276
5	-1	213	-183
6	-1	352	356
7	-1	45	-10*
1	0	168	181
2	0	559	573
3	0	190	-213
4	0	46	-10*
5	0	48	-37*
6	0	203	-196
7	0	45	-33*
1	1	339	-307
2	1	595	-606
3	1	96	100
4	1	37	12*
5	1	36	-5*
6	1	233	228
7	1	59	-12*
1	2	48	49*
2	2	984	973
3	2	24	58*
4	2	37	23*
5	2	36	29*
6	2	269	-237
7	2	47	-39*
1	3	285	259
2	3	619	-645
3	3	173	-186
4	3	82	103
5	3	161	148
6	3	421	432
1	4	165	-173
2	4	962	924
3	4	138	87
4	4	163	-215
5	4	149	-138
6	4	238	-293
1	5	152	171
2	5	539	-532
3	5	279	-298
4	5	32	-82*
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5	7	237	-218
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3	8	259	269
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3	10	64	77*
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2	11	101	47
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5	-7	81	-37*
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3	-6	244	209
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5	-6	45	-90*
6	-6	289	274
1	-5	37	-69*
2	-5	452	400
3	-5	176	157
4	-5	212	183
5	-5	178	172
6	-5	197	-211
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2	-4	598	-572
3	-4	94	-113
4	-4	105	-116
5	-4	76	-82*
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7	-4	89	97
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7	-3	82	-85*
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6	-2	237	213
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3	1	197	217
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6	6	226	-227
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4	7	253	-242
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6	7	258	260
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3	8	33	18*
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5	8	49	17*
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3	9	338	-329
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3	10	117	176
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2	11	251	-213
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5	-8	166	188
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3	-7	35	-62*
4	-7	95	66*
5	-7	246	-231
6	-7	130	-136
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3	-6	75	37*
4	-6	48	-19*
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6	-6	142	144
7	-6	33	-21*
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3	-5	58	-70*
4	-5	113	116
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6	-5	161	-159
7	-5	49	49*
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3	-4	64	94*
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5	-4	211	211
6	-4	142	156
7	-4	32	44*
8	-4	57	45*
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2	-3	338	333
3	-3	29	-64*
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5	-3	299	-322
6	-3	143	-121
7	-3	38	-14*
8	-3	211	-222
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6	-2	379	389
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2	-1	851	825
3	-1	237	216
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6	1	293	-303
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6	3	153	-127
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6	5	160	-153
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6	7	104	-115
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3	8	23	-2*
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3	9	69	62*
4	9	75	-67*
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5	-9	181	157
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2	-8	194	211
3	-8	109	-49
4	-8	95	108
5	-8	183	-127
6	-8	118	-78
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2	-7	255	-236
3	-7	41	39*
4	-7	380	-339
5	-7	257	-227
6	-7	83	-22
7	-7	46	-49*
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2	-6	251	272
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5	-6	220	259
6	-6	84	-82
7	-6	74	-70*
8	-6	198	-201
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2	-5	362	-387
3	-5	74	65
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7	-5	77	59*
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6	-4	78	-59*
7	-4	43	-75*
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6	-3	69	74*
7	-3	152	-154
8	-3	126	127
9	-3	232	239
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5	-2	642	622
6	-2	111	-119
7	-2	306	298
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3	-1	244	-239
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6	0	48	56*
7	0	357	326
8	0	150	133
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6	1	137	-138
7	1	271	-259
8	1	181	-185
9	1	231	234
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3	2	273	265
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7	2	122	115
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8	3	114	-127
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7	4	71	-64*
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3	5	101	99
4	5	412	407
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7	5	74	67*
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2	6	421	-451
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7	6	55	17*
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2	7	222	252
3	7	21	1*
4	7	419	399
5	7	221	-200
6	7	39	52*
1	8	248	-242
2	8	227	-210
3	8	59	-59*
4	8	185	-183
5	8	101	-125
6	8	232	228
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2	9	166	170
3	9	81	39*
4	9	82	67*
5	9	172	134
1	10	121	-127
2	10	221	-230
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4	10	208	-205
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2	11	112	151
3	11	34	-2*
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3	-10	73	74*
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2	-9	85	-97
3	-9	101	-107
4	-9	105	-82
5	-9	185	191
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7	-9	36	55*
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2	-8	56	7*
3	-8	224	216
4	-8	141	141
5	-8	247	-229
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7	-8	179	-178
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2	-7	26	15*
3	-7	58	-51*
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5	-7	278	273
6	-7	149	149
7	-7	232	185
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6	-6	141	-161
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5	-1	189	187
6	-1	26	245
7	-1	61	9*
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6	0	263	-269
7	0	121	108
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3	1	219	-202
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6	4	86	-97
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8	4	98	-96
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7	5	230	-230
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3	7	180	75
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5	8	197	213
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3	9	181	189
4	9	39	-77*
5	9	323	-329
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6	-8	35	-9*
7	-8	78	-100*
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3	-7	123	-115
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7	-7	182	195
8	-7	208	-196
9	-7	171	-145
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6	-6	81	-99
7	-6	252	-251
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6	-4	168	-144
7	-4	535	-512
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8	-2	44	46*
9	-2	139	140
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6	3	218	-218
7	3	251	252
8	3	220	214
9	3	126	-129
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3	4	542	521
4	4	488	493
5	4	212	-211
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7	4	192	-197
8	4	212	-276
1	5	312	-305
2	5	235	-238
3	5	175	-183
4	5	462	-476
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5	7	72	-61*
6	7	74	13*
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2	8	36	41*
3	8	209	216
4	8	386	401
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6	8	45	-46*
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2	9	39	62*
3	9	108	101
4	9	217	-253
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2	11	104	133
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3	-11	238	210
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6	-11	124	86
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6	-10	88	-89
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2	-9	65	73*
3	-9	326	329
4	-9	66	33*
5	-9	237	-251
6	-9	45	-14*
7	-9	290	-274
8	-9	78	-98*
1	-8	435	-440
2	-8	31	4*
3	-8	318	-319
4	-8	48	-34*
5	-8	225	239
6	-8	51	-36*
7	-8	282	274
8	-8	118	112
9	-8	65	-49*
1	-7	904	917
2	-7	98	-108
3	-7	322	318
4	-7	311	302
5	-7	142	-149
6	-7	87	80
7	-7	243	-245
8	-7	177	-195
9	-7	49	98*
1	-6	429	-459
2	-6	219	195
3	-6	261	-261
4	-6	336	-346
5	-6	166	165
6	-6	95	-111
7	-6	256	259
8	-6	148	169
9	-6	96	-83
1	-5	404	394
2	-5	215	-199
3	-5	346	342
4	-5	615	591
5	-5	331	-322
6	-5	498	474
7	-5	300	-296
8	-5	131	-163
9	-5	89	95*
10	-5	144	-148
1	-4	379	-372
2	-4	214	-201
3	-4	178	-194
4	-4	677	-670
5	-4	415	392
6	-4	316	-317
7	-4	289	301
8	-4	493	491
9	-4	44	-19*
10	-4	189	189
1	-3	178	202
2	-3	36	1*
3	-3	426	429
4	-3	356	367
5	-3	78	-48
6	-3	153	-172
7	-3	193	-180
8	-3	559	-558
9	-3	29	34*
10	-3	191	-194
2	-2	1238	-1220
3	-2	524	-521
4	-2	431	-441
5	-2	46	-14*
6	-2	16	39*
7	-2	25	2*
8	-2	432	429
9	-2	45	35*
10	-2	188	180
3	-1	28	-44*
4	-1	129	175
5	-1	3	17*
6	-1	39	-11*
7	-1	169	159
8	-1	494	-490
9	-1	74	-60*
10	-1	191	-171
3	0	170	184
4	0	457	-456
5	0	65	-61*
6	0	120	109
7	0	356	-373
8	0	409	419
9	0	38	48*
10	0	138	148
2	1	1280	1266
3	1	633	-635
4	1	359	371
5	1	271	257
6	1	316	326
7	1	379	372
8	1	36	-48*
9	1	104	-116
1	2	212	212
2	2	0	-21*
3	2	254	252
4	2	592	-576
5	2	379	-352
6	2	476	-463
7	2	275	-282
8	2	111	95
9	2	19	55*
0	3	707	-656
1	3	217	-248
2	3	151	148
3	3	408	-416
4	3	505	498
5	3	142	169
6	3	88	67
7	3	283	284
8	3	129	-158
9	3	19	-29*
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1	4	456	444
2	4	226	221
3	4	331	326
4	4	252	-266
5	4	171	-161
6	4	40	-55*
7	4	258	-266
8	4	15	41*
0	5	465	-468
1	5	307	-319
2	5	318	-382
3	5	333	-336
4	5	261	257
5	5	99	107
6	5	20	28*
7	5	288	298
8	5	41	-38*
0	6	121	115
1	6	491	505
2	6	8	47*
3	6	427	438
4	6	69	-52*
5	6	196	-198
6	6	81	67
7	6	262	-278
0	7	113	88
1	7	495	-414
2	7	35	1*
3	7	316	-319
4	7	93	-59*
5	7	158	165
6	7	36	24*
0	8	85	-68
1	8	304	318
2	8	38	-76*
3	8	304	278
4	8	88	-68
5	8	128	-87
6	8	48	-43*
0	9	31	18*
1	9	104	-75
2	9	241	255
3	9	92	-126
4	9	48	34*
5	9	30	2*
0	10	248	-269
1	10	39	9*
2	10	216	-237
3	10	247	221
0	11	173	143
1	11	115	-77
2	11	32	48*
<<<<< K = 1 >>>>>			
0	-12	181	-98
1	-12	182	115
2	-12	48	71*
3	-12	36	41*
4	-12	208	188
5	-12	20	37*
0	-11	289	165
1	-11	41	12*
2	-11	139	117
3	-11	147	-134
4	-11	38	29*
5	-11	279	-264
6	-11	56	13*
7	-11	26	27*
0	-10	489	-482
1	-10	98	62
2	-10	275	-293
3	-10	87	85
4	-10	161	136
5	-10	47	47*
6	-10	208	210
7	-10	113	-98
8	-10	181	-177
0	-9	342	379
1	-9	60	-84*
2	-9	169	174
3	-9	164	166
4	-9	341	-337
5	-9	116	-112
6	-9	361	-369
7	-9	116	-91
8	-9	176	178
9	-9	119	-96
0	-8	642	-687
1	-8	155	148
2	-8	100	-86
3	-8	50	24*
4	-8	563	571
5	-8	220	248
6	-8	289	290
7	-8	113	129
8	-8	158	-170
9	-8	80	63*
0	-7	296	330
1	-7	69	-59*
2	-7	105	-84
3	-7	416	404
4	-7	414	-414
5	-7	48	-46*
6	-7	242	-253
7	-7	219	-233
8	-7	227	213
9	-7	124	-105
0	-6	261	-275
1	-6	176	-146
2	-6	56	-60*
3	-6	557	-561
4	-6	421	424
5	-6	216	232
6	-6	260	267
7	-6	350	346
8	-6	175	-156
9	-6	0	28*
10	-6	58	-55*
0	-5	606	627
1	-5	59	-52*
2	-5	33	-11*
3	-5	558	566
4	-5	537	-531
5	-5	41	-25*
6	-5	445	-434
7	-5	263	-262
8	-5	151	151
9	-5	97	53
10	-5	158	161
0	-4	404	-425
1	-4	355	-380
2	-4	71	-66
3	-4	585	-608
4	-4	382	377
5	-4	77	68
6	-4	295	276
7	-4	461	472
8	-4	209	-198
9	-4	35	-15*
10	-4	139	-126
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1	-3	21	-27*
2	-3	213	206
3	-3	633	634
4	-3	136	148
5	-3	54	-38*
6	-3	137	-141
7	-3	578	-576
8	-3	57	13*
9	-3	79	66*
10	-3	74	-14*
2	-2	158	-167
3	-2	1689	-1628
4	-2	145	-117
5	-2	46	39*
6	-2	28	12*
7	-2	544	536
8	-2	108	119
9	-2	47	-88*
10	-2	177	163
3	-1	1371	1396
4	-1	45	67*
5	-1	60	-78*
6	-1	218	218
7	-1	462	-476
8	-1	212	-217
9	-1	65	52*
10	-1	185	-191
2	0	181	178
3	0	1428	-1424
4	0	183	181
5	0	20	1*
6	0	387	-291
7	0	322	314
8	0	181	177
9	0	78	-38*
10	0	133	125
2	1	359	-377
3	1	562	585
4	1	291	286
5	1	49	-63*
6	1	544	513
7	1	212	-283
8	1	148	-166
9	1	53	-63*
0	2	376	368
1	2	31	-35*
2	2	42	28*
3	2	536	-531
4	2	617	-612
5	2	78	96
6	2	356	-393
7	2	248	241
8	2	203	191
9	2	127	121
0	3	415	-418
1	3	235	208
2	3	66	-87
3	3	485	496
4	3	451	444
5	3	107	-116
6	3	297	308
7	3	119	-138
8	3	148	-146
9	3	48	-83*
0	4	603	615
1	4	186	182
2	4	99	108
3	4	496	-498
4	4	448	-437
5	4	63	69*
6	4	267	-284
7	4	88	95
8	4	147	141
0	5	296	-317
1	5	193	145
2	5	28	-17*
3	5	181	184
4	5	460	462
5	5	188	-185
6	5	329	342
7	5	29	48*
8	5	97	-95
0	6	184	214
1	6	154	155
2	6	133	111
3	6	39	32*
4	6	549	-546
5	6	44	98*

Table 4 (*cont.*)

H	L	FO	FC
6	6	253	-250
7	6	46	-88*
0	7	610	-564
1	7	99	-112
2	7	272	-270
3	7	88	57
4	7	191	159
5	7	164	-175
6	7	41	-37*
0	8	395	403
1	8	88	106
2	8	339	317
3	8	135	95
4	8	45	-24*
5	8	145	173
0	9	330	-332
1	9	54	13*
2	9	168	-141
3	9	32	-9*
4	9	173	166
0	10	308	327
1	10	84	-43
2	10	59	24*
3	10	106	88
0	11	141	-128
1	11	144	-169
<<<<< K = 2 >>>>>			
0	-12	204	228
1	-12	191	232
2	-12	126	123
3	-12	288	306
4	-12	38	74*
5	-12	15	-37*
6	-12	36	-11*
0	-11	299	-302
1	-11	79	-111*
2	-11	89	-80
3	-11	233	-251
4	-11	209	-231
5	-11	91	94
6	-11	20	3*
7	-11	249	232
0	-10	81	89
1	-10	52	4*
2	-10	86	-116
3	-10	504	539
4	-10	87	115
5	-10	57	-20*
6	-10	82	-70
7	-10	264	-259
8	-10	63	-51*
0	-9	3	51*
1	-9	58	24*
2	-9	161	174
3	-9	526	-529
4	-9	112	-84
5	-9	94	87
6	-9	61	-59*
7	-9	316	318
8	-9	56	44*
9	-9	12	11*
0	-8	11	-124
1	-8	47	-81*
2	-8	54	-84*
3	-8	373	387
4	-8	78	74
5	-8	89	-87
6	-8	157	144
7	-8	315	-320
8	-8	125	-123
9	-8	6	-54*
0	-7	151	154
1	-7	71	174
2	-7	84	87
3	-7	361	-361
4	-7	157	-156
5	-7	106	-95
6	-7	373	-368
7	-7	338	347
8	-7	181	164
9	-7	50	31*
10	-7	193	135
0	-6	298	-301
1	-6	7	-54
2	-6	111	-106
3	-6	497	517
4	-6	269	278
5	-6	123	106
6	-6	461	451
7	-6	431	-427
8	-6	171	-150
9	-6	16	-171
10	-6	228	-232
0	-5	426	451
1	-5	295	-295
2	-5	234	245
3	-5	111	-138
4	-5	319	-310
5	-5	261	-233
6	-5	78	-255
7	-5	368	350
8	-5	77	81
9	-5	235	233
10	-5	237	238
0	-4	517	-521
1	-4	29	41*
2	-4	32	[illegible]
3	-4	172	175
4	-4	581	617
5	-4	376	374
6	-4	641	633
7	-4	23	-227
8	-4	266	-277
9	-4	57	-52*
10	-4	286	-295
0	-3	497	514
1	-3	477	-445
2	-3	65	628
3	-3	1124	1124
4	-3	181	159
5	-3	444	444
6	-3	304	-312
7	-3	32	19*
8	-3	379	373
9	-3	7	-33*
10	-3	288	297
0	-2	617	-642
1	-2	1234	-1251
2	-2	141	-146
3	-2	1475	-1712
4	-2	214	254
5	-2	46	44*
6	-2	465	477
7	-2	197	167
8	-2	201	[illegible]
9	-2	[illegible]	[illegible]
10	-2	192	[illegible]
2	-1	[illegible]	[illegible]
3	-1	1682	-1717
4	-1	28	-17*
5	-1	397	-384
6	-1	425	[illegible]
7	-1	325	-311
8	-1	91	[illegible]
9	-1	285	-275
10	-1	182	179
0	0	661	-679
1	0	1266	[illegible]
2	0	1334	[illegible]
3	0	992	976
4	0	284	267
5	0	[illegible]	-434
6	0	75	718
7	0	423	423
8	0	82	-78
9	0	179	194
10	0	150	-172
0	1	527	536
1	1	514	471
2	1	42	-6*
3	1	292	283
4	1	597	-671
5	1	370	356
6	1	468	-466
7	1	447	-451
8	1	127	125
9	1	46	-35*
0	2	480	-491
1	2	40	-66*
2	2	203	-218
3	2	153	-175
4	2	106	117
5	2	191	-182
6	2	293	294
7	2	384	383
8	2	103	-94
9	2	82	89*
0	3	466	472
1	3	227	234
2	3	141	134
3	3	406	526
4	3	211	-196
5	3	178	181
6	3	171	-167
7	3	333	-345
8	3	21	78*
0	4	278	-301
1	4	101	93
2	4	82	-48*
3	4	344	-342
4	4	45	-26*
5	4	41	78*
6	4	36	13*
7	4	293	290
8	4	40	-22*
0	5	332	333
1	5	197	-196
2	5	39	48*
3	5	349	362
4	5	42	-78*
5	5	60	-6*
6	5	40	0*
7	5	200	-203
0	6	128	-124
1	6	103	131
2	6	50	-41*
3	6	545	-548
4	6	36	61*
5	6	0	71*
6	6	31	-14*
7	6	272	252
0	7	73	93*
1	7	135	-143
2	7	96	172
3	7	419	436
4	7	151	-172
5	7	117	-112
6	7	0	-13*
0	8	43	48*
1	8	72	62*
2	8	61	51*
3	8	254	-297
4	8	79	91*
5	8	34	-24*
0	9	209	-204
1	9	45	5*
2	9	55	-18*
3	9	271	293
4	9	60	43*
0	10	215	216
1	10	156	-148
2	10	165	179
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2	-12	234	312
3	-12	63	-39*
4	-12	213	-227
5	-12	38	24*
6	-12	157	-164
0	-11	178	-181
1	-11	252	241
2	-11	277	-278
3	-11	80	36
4	-11	112	150
5	-11	151	145
6	-11	124	115
7	-11	299	249
8	-11	107	-91
0	-10	148	154
1	-10	152	-178
2	-10	465	413
3	-10	49	54*
4	-10	155	-191
5	-10	134	-173
6	-10	366	-362
7	-10	59	-42*
8	-10	62	-29*
0	-9	314	-311
1	-9	156	148
2	-9	557	-552
3	-9	61	-3*
4	-9	156	182
5	-9	32	-14*
6	-9	286	305
7	-9	70	72*
8	-9	61	-41*
9	-9	109	174
0	-8	384	360
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2	-8	515	499
3	-8	200	194
4	-8	123	-127
5	-8	92	174
6	-8	367	-348
7	-8	205	-231
8	-8	33	17*
9	-8	193	-197
0	-7	91	-117
1	-7	220	-235
2	-7	78	14
3	-7	508	-481
4	-7	237	228
5	-7	245	-232
6	-7	451	448
7	-7	323	316
8	-7	29	-39*
9	-7	284	260
10	-7	143	-149
0	-6	211	-189
1	-6	82	-83
2	-6	147	149
3	-6	266	287
4	-6	76	64
5	-6	202	193
6	-6	372	-364
7	-6	351	-314
8	-6	37	45*
9	-6	231	-213
10	-6	16	81*
0	-5	265	-247
1	-5	60	15*
2	-5	242	-237
3	-5	348	-358
4	-5	19	-4*
5	-5	551	-496
6	-5	0	51*
7	-5	158	151
8	-5	52	-37*
9	-5	179	182
10	-5	21	-23*
0	-4	451	459
1	-4	421	398
2	-4	116	-117
3	-4	512	568
4	-4	486	475
5	-4	449	456
6	-4	346	338
7	-4	344	-353
8	-4	34	59*
9	-4	224	-199
10	-4	19	2*
0	-3	527	-518
1	-3	305	-308
2	-3	13	16*
3	-3	418	-449
4	-3	257	-269
5	-3	502	-501
6	-3	100	-93
7	-3	317	300
8	-3	46	47*
9	-3	185	160
10	-3	95	156
0	-2	473	469
1	-2	249	218
2	-2	257	-246
3	-2	914	984
4	-2	151	-147
5	-2	588	631
6	-2	122	-105
7	-2	381	-377
8	-2	39	-48*
9	-2	169	-180
10	-2	125	-124
0	-1	158	163
1	-1	258	-225
2	-1	180	-152
3	-1	495	-516
4	-1	230	241
5	-1	337	-320
6	-1	97	78
7	-1	165	158
8	-1	38	32*
9	-1	159	156
10	-1	165	166
0	0	611	-580
1	0	295	305
2	0	67	48
3	0	528	574
4	0	327	-314
5	0	635	627
6	0	179	[illegible]
7	0	146	-139
8	0	43	[illegible]
9	0	235	-231
0	1	519	524
1	1	396	-375
2	1	63	29*
3	1	458	-465
4	1	0	18*
5	1	144	-133
6	1	512	-514
7	1	321	297
8	1	77	-42*
9	1	162	167
0	2	445	-439
1	2	67	-74
2	2	324	-339
3	2	279	275
4	2	78	97
5	2	147	141
6	2	475	495
7	2	147	-137
8	2	59	-25*
9	2	75	-79*
0	3	74	81
1	3	99	106
2	3	47	19*
3	3	517	-492
4	3	217	-195
5	3	124	-115
6	3	314	-318
7	3	35	51*
8	3	57	5*
0	4	264	227
1	4	67	40*
2	4	175	-177
3	4	178	178
4	4	39	68*
5	4	65	-47*
6	4	315	324
7	4	38	-25*
8	4	25	-24*
0	5	252	224
1	5	193	191
2	5	627	[illegible]
3	5	33	35*
4	5	118	-122
5	5	130	-152
6	5	254	-253
7	5	167	193
0	6	319	-315
1	6	151	-142
2	6	517	-548
3	6	31	-28*
4	6	81	14*
5	6	168	-138
6	6	183	-193
0	7	184	194
1	7	110	132
2	7	429	418
3	7	41	38*
4	7	116	-123
5	7	42	-14*
0	8	77	-47*
1	8	227	-225
2	8	226	-242
3	8	111	-111
4	8	119	85
0	9	116	107
1	9	17	-29*
2	9	256	278
3	9	79	77*
0	10	176	-150
<<<<< K = 4 >>>>>			
0	-12	134	-117
1	-12	137	171
2	-12	82	-52*
3	-12	129	-149
4	-12	79	78*
5	-12	153	-145
6	-12	88	-41
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7	-9	239	-244
8	-9	149	152
9	-9	159	-149
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4	-8	218	-226
5	-8	222	-224
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7	-8	191	201
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9	-8	117	107
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8	-7	252	258
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7	-6	89	82
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8	-3	58	67*
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7	-2	234	-225
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9	-2	271	-211
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7	-1	249	259
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3	0	154	152
4	0	44	-52*
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6	0	333	-351
7	0	41	32*
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5	1	222	-226
6	1	271	249
7	1	147	142
8	1	121	112
9	1	193	213
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3	2	115	134
4	2	228	-234
5	2	183	168
6	2	117	-130
7	2	193	-191
8	2	117	-113
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7	3	185	199
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6	4	131	156
7	4	193	-239
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2	5	272	-248
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3	6	198	169
4	6	21	38*
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6	6	42	25*
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3	7	82	-67*
4	7	33	-37*
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5	-12	40	-47*
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3	-11	44	-56*
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5	-11	69	68*
6	-11	277	-270
7	-11	41	-43*
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4	-10	186	-188
5	-10	93	-89
6	-10	266	270
7	-10	37	26*
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6	-9	283	-272
7	-9	84	-84
8	-9	54	-67*
9	-9	245	-247
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2	-8	242	-232
3	-8	68	-33*
4	-8	57	49*
5	-8	351	-324
6	-8	406	379
7	-8	44	61*
8	-8	94	78
9	-8	191	198
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1	-7	435	-413
2	-7	227	247
3	-7	463	468
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5	-7	416	431
6	-7	226	-202
7	-7	111	-86
8	-7	120	133
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4	-6	293	-289
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3	-5	431	411
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7	-5	265	-234
8	-5	44	84*
9	-5	333	-334
10	-5	64	3*
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2	-4	362	-353
3	-4	395	-419
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9	-4	363	375
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3	-3	131	126
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8	-3	94	-125
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10	-3	68	-101*
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3	-2	225	-239
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7	-2	145	132
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4	-1	98	-103
5	-1	400	435
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1	0	289	302
2	0	647	651
3	0	347	-394
4	0	272	289
5	0	338	-356
6	0	231	-199
7	0	41	-53*
8	0	42	-84*
9	0	171	161
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4	1	26	-22*
5	1	332	341
6	1	475	463
7	1	43	-68*
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0	2	118	124
1	2	205	209
2	2	334	347
3	2	221	-202
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5	2	128	-91
6	2	272	-250
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8	2	87	-73
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2	3	338	-302
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6	3	219	212
7	3	4	-3*
0	4	97	-119
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2	4	321	320
3	4	94	113
4	4	234	208
5	4	29	6*
6	4	201	-208
7	4	51	13*
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1	5	129	-139
2	5	410	-414
3	5	37	-24*
4	5	257	-261
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5	6	38	3*
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2	7	23	-229
3	7	108	-117
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1	8	92	[illegible]
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5	-12	215	221
6	-12	22	-17*
0	-11	118	122
1	-11	242	256
2	-11	78	59*
3	-11	127	-127
4	-11	79	112*
5	-11	264	-267
6	-11	91	89
7	-11	78	-57*
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2	-10	99	-120
3	-10	44	28*
4	-10	143	-169
5	-10	115	227
6	-10	3	-8*
7	-10	65	51*
8	-10	[illegible]	69
0	-9	86	81
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2	-9	98	[illegible]
3	-9	158	-149
4	-9	187	155
5	-9	189	-196
6	-9	163	-166
7	-9	53	[illegible]
8	-9	194	-191
9	-9	137	147
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1	-8	297	[illegible]
2	-8	217	[illegible]
3	-8	117	[illegible]
4	-8	[illegible]	[illegible]
5	-8	56	154
6	-8	338	329
7	-8	125	-116
8	-8	193	212
9	-8	144	-127
0	-7	92	-94
1	-7	182	189
2	-7	147	147
3	-7	65	67*
4	-7	165	[illegible]
5	-7	[illegible]	[illegible]
6	-7	456	-463
7	-7	231	221
8	-7	[illegible]	[illegible]
9	-7	132	132
0	-6	52	[illegible]
1	-6	266	-254
2	-6	37	[illegible]
3	-6	44	[illegible]
4	-6	145	-121
5	-6	236	[illegible]
6	-6	39	38
7	-6	63	[illegible]
8	-6	23	229
9	-6	157	161
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2	-5	415	[illegible]
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4	-5	351	338
5	-5	87	-89
6	-5	416	-437
7	-5	41	-64*
8	-5	385	[illegible]
9	-5	137	-151
0	-4	176	[illegible]
1	-4	41	[illegible]
2	-4	42	[illegible]
3	-4	237	[illegible]
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5	-4	39	-65*
6	-4	438	439
7	-4	63	-41*
8	-4	43	447
9	-4	42	-27*
0	-3	32	-31
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2	-3	147	[illegible]
3	-3	66	[illegible]
4	-3	194	187
5	-3	[illegible]	[illegible]
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7	-3	54	38*
8	-3	326	[illegible]
9	-3	34	-27*
0	-2	364	[illegible]
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2	-2	284	-284
3	-2	291	[illegible]
4	-2	351	[illegible]
5	-2	214	[illegible]
6	-2	325	[illegible]
7	-2	116	131
8	-2	125	[illegible]
9	-2	265	[illegible]
0	-1	189	[illegible]
1	-1	439	-40
2	-1	[illegible]	[illegible]
3	-1	19	[illegible]
4	-1	188	[illegible]
5	-1	367	[illegible]
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7	-1	218	-209
8	-1	126	-142
9	-1	211	-192
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1	0	221	225
2	0	291	-294
3	0	58	-43*
4	0	47	-34*
5	0	429	-430
6	0	278	271
7	0	65	51*
8	0	188	193
0	1	109	-112
1	1	368	-352
2	1	342	333
3	1	49	-78*
4	1	235	218
5	1	438	437
6	1	51	-68*
7	1	42	5*
8	1	86	-45
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2	2	229	-227
3	2	91	-86
4	2	114	-118
5	2	381	-303
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7	2	63	-76*
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1	3	303	-301
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3	3	108	94
4	3	67	31*
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6	3	60	103*
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1	4	345	339
2	4	73	-65*
3	4	30	76*
4	4	92	-82
5	4	186	-166
6	4	62	-65*
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1	5	312	-305
2	5	63	63*
3	5	57	60*
4	5	73	-39*
5	5	158	159
0	6	139	-162
1	6	243	247
2	6	69	53*
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0	7	69	74*
1	7	291	-290
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3	-12	48	56*
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5	-12	48	-48*
0	-11	155	120
1	-11	24	76*
2	-11	232	-218
3	-11	52	-44*
4	-11	251	-238
5	-11	0	45*
6	-11	136	150
7	-11	16	-16*
0	-10	83	82*
1	-10	311	-294
2	-10	325	294
3	-10	39	-2*
4	-10	209	199
5	-10	41	17*
6	-10	143	-140
7	-10	0	[illegible]
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1	-9	298	311
2	-9	132	-128
3	-9	111	-90
4	-9	288	-306
5	-9	196	-210
6	-9	57	49*
7	-9	110	-99
8	-9	159	128
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1	-8	194	-245
2	-8	91	87
3	-8	121	148
4	-8	390	[illegible]
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6	-8	156	-133
7	-8	199	212
8	-8	128	-99
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1	-7	410	414
2	-7	198	-213
3	-7	116	-92
4	-7	319	-334
5	-7	497	-475
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7	-7	91	-94
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9	-7	275	268
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3	-6	75	[illegible]
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7	-6	69	79*
8	-6	41	76*
9	-6	293	-314
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3	-5	178	181
4	-5	131	-144
5	-5	312	-297
6	-5	55	66*
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5	-4	298	318
6	-4	48	43*
7	-4	161	132
8	-4	76	87*
9	-4	296	-298
0	-3	67	89*
1	-3	388	392
2	-3	33	35*
3	-3	218	203
4	-3	184	189
5	-3	243	-247
6	-3	48	-63*
7	-3	164	-153
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3	-2	199	-205
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3	-1	88	-101
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5	-1	376	-392
6	-1	154	-134
7	-1	177	-179
8	-1	211	-188
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1	0	395	-389
2	0	172	-193
3	0	112	113
4	0	417	-441
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6	0	68	86*
7	0	77	60*
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2	1	178	196
3	1	137	-153
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3	2	109	107
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5	2	45	-33*
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2	3	264	267
3	3	42	-2*
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3	5	81	-95*
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6	-11	39	29*
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2	-10	242	264
3	-10	68	61*
4	-10	182	174
5	-10	195	-198
6	-10	46	-38*
7	-10	163	-148
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1	-9	112	-143
2	-9	243	-239
3	-9	64	-40*
4	-9	233	-233
5	-9	173	178
6	-9	57	70*
7	-9	186	200
0	-8	282	-313
1	-8	96	98
2	-8	57	41*
3	-8	169	141
4	-8	226	200
5	-8	139	-158
6	-8	142	-154
7	-8	143	-138
8	-8	257	-247
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7	-7	50	-24*
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5	-6	51	-39*
6	-6	44	-74*
7	-6	24	33*
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6	-5	157	178
7	-5	34	10*
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6	-4	130	-140
7	-4	11	21*
8	-4	187	-201
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6	-3	37	29*
7	-3	18	-19*
8	-3	183	175
0	-2	250	-271
1	-2	150	-145
2	-2	124	114
3	-2	111	-112
4	-2	438	441
5	-2	148	147
6	-2	45	-73*
7	-2	133	176
0	-1	343	343
1	-1	151	148
2	-1	114	-118
3	-1	129	111
4	-1	240	-225
5	-1	241	-245
6	-1	111	148
7	-1	239	-262
0	0	318	-289
1	0	205	-208
2	0	0	8*
3	0	166	-144
4	0	152	145
5	0	191	214
6	0	22	-44*
0	1	308	301
1	1	132	146
2	1	57	-65*
3	1	105	98
4	1	193	-195
5	1	189	-171
6	1	32	17*
0	2	226	-254
1	2	89	-94*
2	2	173	161
3	2	78	-73*
4	2	107	114
5	2	243	236
0	3	117	128
1	3	279	293
2	3	173	-187
3	3	64	51*
4	3	88	-84*
0	4	68	-76*
1	4	224	-218
2	4	97	117
<<<<< K = 9 >>>>>			
1	-10	251	258
2	-10	196	-156
3	-10	329	306
4	-10	239	-224
5	-10	155	-151
0	-9	160	-208
1	-9	170	-164
2	-9	98	79
3	-9	112	-109
4	-9	75	99*
5	-9	268	262
6	-9	57	-1*
0	-8	121	97
1	-8	48	-54*
2	-8	36	-11*
3	-8	105	99
4	-8	144	-151
5	-8	250	-243
6	-8	69	-30*
7	-8	177	-188
0	-7	61	38*
1	-7	94	-105
2	-7	80	94*
3	-7	265	-240
4	-7	176	203
5	-7	220	228
6	-7	57	-45*
7	-7	133	132
0	-6	38	9*
1	-6	247	242
2	-6	40	36*
3	-6	287	292
4	-6	95	-93
5	-6	165	-153
6	-6	0	23*
7	-6	114	-99
0	-5	87	-101
1	-5	376	-408
2	-5	56	-38*
3	-5	320	-335
4	-5	48	76*
5	-5	132	85
6	-5	38	-5*
7	-5	91	108
0	-4	44	31*
1	-4	350	343
2	-4	39	8*
3	-4	266	266
4	-4	160	158
5	-4	143	-108
6	-4	51	4*
7	-4	146	-126
0	-3	71	82*
1	-3	362	-372
2	-3	27	15*
3	-3	258	-265
4	-3	185	-193
5	-3	134	148
6	-3	43	61*
0	-2	154	-172
1	-2	300	333
2	-2	66	-46*
3	-2	210	189
4	-2	245	259
5	-2	138	-169
6	-2	9	31*
0	-1	74	53*
1	-1	146	-122
2	-1	24	-63*
3	-1	130	-128
4	-1	217	-229
5	-1	210	288
0	0	46	-33*
1	0	23	21*
2	0	66	-18*
3	0	46	58*
4	0	158	170
5	0	125	-123
0	1	100	98
1	1	0	32*
2	1	70	-104*
3	1	214	-198
4	1	285	-291
0	2	175	-177
1	2	161	161
2	2	116	98
<<<<< K = 10 >>>>>			
1	-8	86	-86*
2	-8	77	50*
3	-8	168	-149
4	-8	171	-149
0	-7	298	-315
1	-7	43	-2*
2	-7	119	-151
3	-7	153	130
4	-7	248	262
5	-7	101	-98
0	-6	388	394
1	-6	0	26*
2	-6	0	17*
3	-6	108	-89
4	-6	292	-298
5	-6	63	83*
0	-5	348	-331
1	-5	0	3*
2	-5	34	27*
3	-5	23	23*
4	-5	242	237
5	-5	28	34*
0	-4	292	255
1	-4	22	47*
2	-4	37	-64*
3	-4	145	129
4	-4	236	-240
5	-4	113	-113
0	-3	387	-288
1	-3	81	-57*
2	-3	58	3*
3	-3	184	-171
4	-3	197	283
0	-2	386	387
1	-2	54	3*
2	-2	54	94*
3	-2	206	198
4	-2	143	-132
0	-1	338	-333
1	-1	54	-27*
2	-1	44	-68*
3	-1	171	-168

computer store available, final refinement was by the block-diagonal least-squares method. Four cycles of refinement, with anisotropic thermal parameters for the two iron atoms, gave $R_1=0{\cdot}059$, $R_2=0{\cdot}072$. The idealized positions of all hydrogen atoms were then calculated assuming the appropriate trigonal or tetrahedral geometry and a C–H distance of 1·05 Å. A final structure factor calculation with the hydrogen atoms included and each given a temperature factor 1·0 Å^2 greater than the B of the bonded carbon atom gave $R_1=0\cdot052$, $R_2=0\cdot062$.

The labelling scheme used is defined in Fig. 2 and the final values of the positional and vibrational parameters are listed in Table 2, with their estimated standard deviations derived from the inverse least-squares matrix. Table 3 lists the idealized hydrogen atom positions and Table 4 the observed and calculated structure factors.

The scattering factors were those tabulated by Ibers (1962) and the major programs used during the refinement were modified versions of *ORFLS* (Busing, Martin & Levy, 1962), the *SF* block-diagonal least-squares program of Shiono (1968) and the Fourier summation program of White (1965). Fig. 3, 4 and 6 were drawn with the program ORTEP (Johnson, 1965). All calculations were performed on the Monash University C.D.C. 3200 computer.

Table 5. *Selected intramolecular distances and angles in* [Fe(salen)]$_2$O*

(*a*) Distances (Å)

	A	*A′*	*B*	*B′*
Fe—oxo	1·78 (1)		1·78 (1)	
Fe—N	2·12 (1)	2·12 (1)	2·11 (1)	2·12 (2)
Fe—O	1·93 (1)	1·92 (1)	1·91 (1)	1·93 (1)
O—C(1)	1·32 (2)	1·33 (2)	1·33 (2)	1·32 (2)
C(1)–C(2)	1·40 (2)	1·40 (2)	1·41 (2)	1·44 (2)
C(1)–C(6)	1·40 (2)	1·41 (2)	1·43 (2)	1·41 (2)
C(2)–C(3)	1·44 (2)	1·40 (2)	1·42 (3)	1·40 (3)
C(3)–C(4)	1·41 (3)	1·39 (3)	1·38 (2)	1·43 (3)
C(4)–C(5)	1·38 (3)	1·42 (3)	1·40 (3)	1·37 (2)
C(5)–C(6)	1·44 (2)	1·44 (2)	1·44 (3)	1·43 (3)
C(6)–C(7)	1·42 (2)	1·44 (2)	1·44 (2)	1·42 (2)
C(7)–N	1·27 (2)	1·28 (2)	1·28 (2)	1·27 (2)
N—C(8)	1·49 (2)	1·50 (2)	1·51 (2)	1·50 (2)
C(8)–C(8′)	1·52 (2)		1·55 (3)	
O—N	2·75 (2)	2·73 (1)	2·73 (1)	2·75 (1)

Discussion of the structure

The crystallographic asymmetric unit contains one discrete [Fe(salen)]$_2$O molecule (Fig. 1) which consists of two Fe(salen) moieties linked by a single oxygen atom bridge. Both iron atoms are five coordinate with approximately square pyramidal geometry. Pertinent intramolecular distances and angles are listed in Table 5 and are in good agreement with the average values for salen reported by Lingafelter & Braun (1966) and Calligaris, Nardin & Randaccio (1972). The packing of the two molecules in the unit cell is illustrated in Fig. 4. There are no unusual intermolecular contacts and, excluding the hydrogen atoms, the shortest intermolecular contact is 3·32 Å between atoms O′*A* and C(8′)*B* $(-x,1-y,1-z)$.* If the calculated hydrogen atom positions are included, the shortest intermolecular contact is then 2·32 Å between atoms HC(5)*B* and HC(5)*B* $(1-x,1-y,2-z)$.

As in [Fe(salen)]$_2$O.2py, [Fe(salen)]$_2$O.CH$_2$Cl$_2$ and Fe(salen)Cl monomer (Gerloch & Mabbs, 1967), the two salen ligands in unsolvated [Fe(salen)]$_2$O have the ‘asymmetric umbrella’ conformation which appears to be preferred in pentacoordinate species. In this arrangement the ethylenediamine bridge has a *gauche* conformation and the two salicylaldimine groups of each ligand are both bent away from the bridging oxygen atom. The degree of bending can conveniently be described (Calligaris, Nardin & Randaccio, 1972) by the angles α and β (Fig. 5) between the coordination plane and the planes defined by the two salicylaldimine residues.

Table 7 lists, for [Fe(salen)]$_2$O.2py, [Fe(salen)]$_2$O.CH$_2$Cl$_2$ and unsolvated [Fe(salen)]$_2$O, the angles α and

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* The symmetry transformation in parentheses is that to be applied to the coordinates listed in Table 2.

Table 5 (*cont.*)

(*b*) Angles (°)

oxo–Fe—N	104·8 (5)	102·5 (5)	100·3 (5)	105·7 (5)
oxo–Fe—O	107·3 (5)	111·8 (5)	111·4 (5)	107·8 (5)
O—Fe—N	85·5 (5)	85·1 (5)	85·6 (5)	85·4 (5)
O—Fe—N′	148·3 (5)	141·8 (5)	141·2 (5)	150·1 (5)
Fe—O—C(1)	128·7 (9)	134·8 (9)	134·9 (9)	130·4 (9)
O—C(1)–C(2)	117·2 (1·5)	117·6 (1·4)	118·0 (1·4)	118·2 (1·4)
C(2)–C(1)–C(6)	121·4 (1·6)	121·0 (1·5)	121·0 (1·5)	119·1 (1·4)
O—C(1)–C(6)	121·3 (1·5)	121·3 (1·4)	121·0 (1·4)	122·6 (1·4)
C(2)–C(1)–C(6)	121·4 (1·6)	121·1 (1·5)	121·0 (1·5)	119·1 (1·4)
C(2)–C(3)–C(4)	117·9 (2·0)	120·8 (1·9)	122·5 (1·8)	119·7 (1·7)
C(3)–C(4)–C(5)	123·2 (2·1)	121·1 (2·0)	119·9 (1·8)	120·7 (1·8)
C(4)–C(5)–C(6)	118·7 (1·9)	118·1 (1·9)	120·0 (1·7)	120·2 (1·7)
C(5)–C(6)–C(1)	119·3 (1·6)	119·3 (1·6)	118·5 (1·5)	120·2 (1·5)
C(5)–C(6)–C(7)	116·4 (1·6)	115·7 (1·6)	116·8 (1·5)	116·3 (1·4)
C(1)–C(6)–C(7)	124·1 (1·6)	124·9 (1·6)	124·7 (1·5)	123·2 (1·4)
C(6)–C(7)–N	125·6 (1·6)	122·4 (1·6)	122·5 (1·5)	126·0 (1·5)
C(7)–N—Fe	123·7 (1·1)	129·9 (1·1)	129·9 (1·1)	124·6 (1·1)
C(7)–N—C(8)	118·7 (1·4)	118·7 (1·4)	118·4 (1·3)	118·2 (1·3)
C(8)–N—Fe	116·9 (9)	111·2 (9)	111·6 (9)	116·8 (9)
N—C(8)–C(8′)	108·8 (1·4)	107·0 (1·4)	106·4 (1·3)	108·1 (1·3)
Fe*A*–oxo–Fe*B*	144·6 (6)			

* To obtain full atom labels (consistent with Table 1) add the appropriate *A*, *A′*, *B* or *B′*.

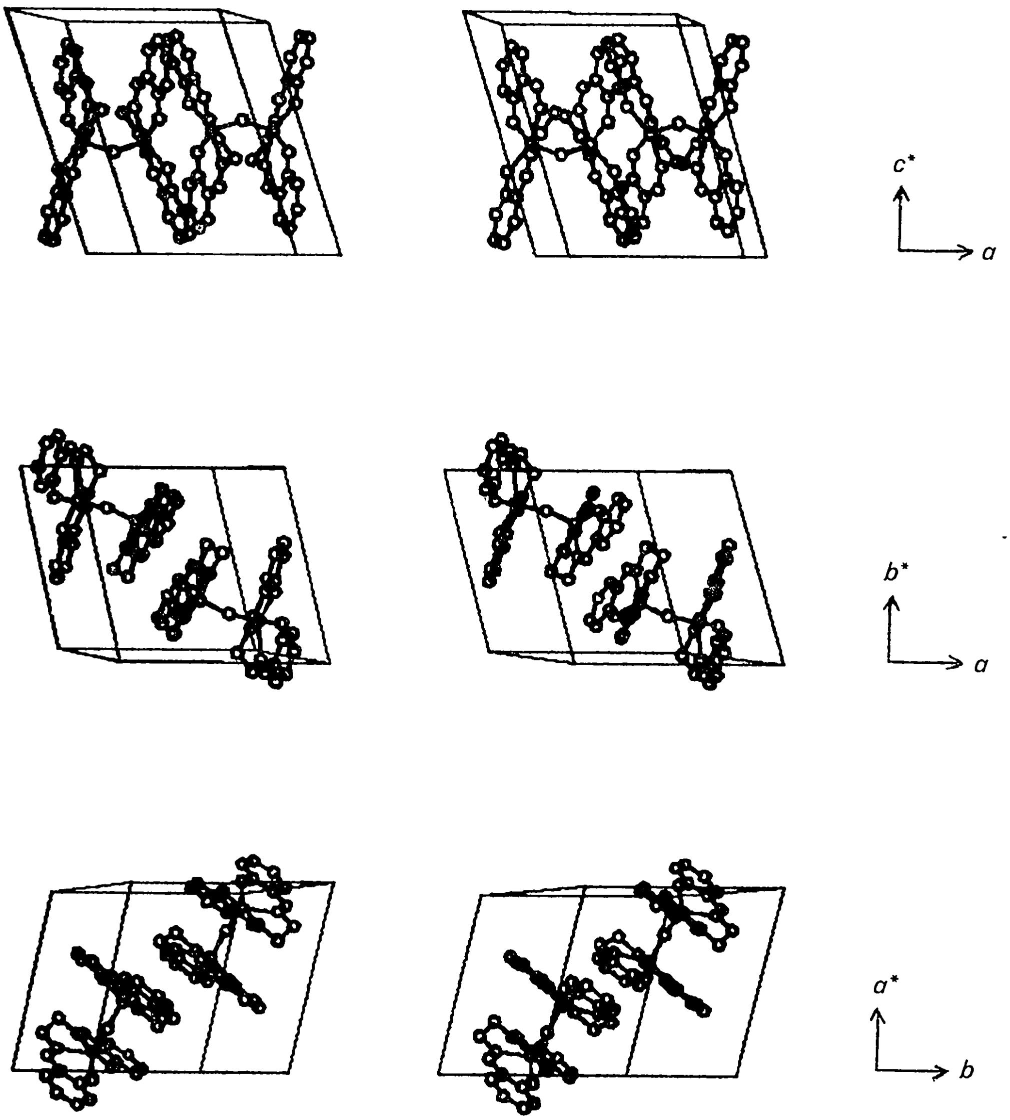

Fig. 4. Contents of the unit cell viewed perpendicular to the *ac**, *ab** and *a*b* planes respectively.

β and the torsional angles N–O–O′–N′ and N–C(8)–C(8′)–N′. This Table also lists, for each salen moiety, the displacements from the coordination plane of the iron atoms and the two ethylene carbon atoms.

An examination of Table 7 reveals certain differences and certain similarities in the ligand conformations of the three structures. The most striking conformational difference is the variation of the angle γ (Fig. 5) which in each salen ligand is the angle between the planes of the two salicylaldimine residues. The γ angles in the two solvated structures are markedly greater than those in the unsolvated structure (*i.e.* the 'umbrella' shape is more pronounced in the solvated structures) and this is best appreciated by reference to Fig. 6 where the three molecules are drawn in similar orientations. However in all three cases the two halves of each dimer have very similar orientations relative to each other. In fact, when the $[Fe(salen)]_2O$ molecules in the pyridinated and unsolvated structures are compared by viewing down the Fe–Fe direction (see Fig. 6), the two are virtually indistinguishable. The differing γ

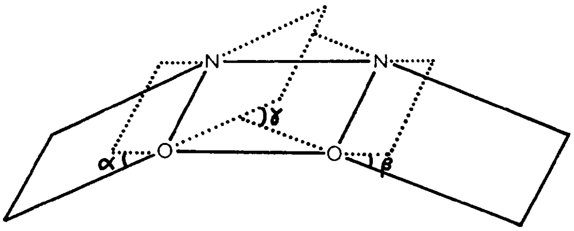

Fig. 5. 'Umbrella' conformation for salen, defining the angles α, β and γ of Table 6.

Table 6. *Equations of least-squares planes for* $[Fe(salen)]_2O$ *and distances of individual atoms* (Å) *from the planes*

X, Y, Z are coordinates in Å referred to an orthogonal system of axes having X along the a axis, Y in the (a,b) plane and Z along the c^* axis. Fractional coordinates x,y,z in the triclinic system are related to X,Y,Z by the matrix equation.

$$\begin{bmatrix} 10\cdot949 & 2\cdot412 & -4\cdot004 \\ 0 & 10\cdot798 & -4\cdot430 \\ 0 & 0 & 12\cdot455 \end{bmatrix} \begin{bmatrix} x \\ y \\ z \end{bmatrix} = \begin{bmatrix} X \\ Y \\ Z \end{bmatrix}$$

(I) Plane through O*A*, C(1)*A*, C(2)*A*, C(3)*A*, C(4)*A*, C(5)*A*, C(6)*A*, C(7)*A*, N*A*

$$0\cdot9150X+0\cdot3341Y-0\cdot2261Z+2\cdot2676=0$$

O*A*	−0·01 (1)	C(3)*A*	−0·01 (2)	C(6)*A*	0·03 (2)
C(1)*A*	0·02 (2)	C(4)*A*	0·01 (2)	C(7)*A*	−0·05 (2)
C(2)*A*	−0·02 (2)	C(5)*A*	0·01 (2)	N*A*	0·02 (2)

(II) Plane through O′*A*, C(1′)*A*, C(2′)*A*, C(3′)*A*, C(4′)*A*, C(5′)*A*, C(6′)*A*, C(7′)*A*, N′*A*

$$0\cdot9091X+0\cdot4127Y-0\cdot0566Z+0\cdot8593=0$$

O′*A*	−0·06 (1)	C(3′)*A*	0·06 (2)	C(6′)*A*	−0·04 (2)
C(1′)*A*	−0·03 (2)	C(4′)*A*	0·01 (2)	C(7′)*A*	−0·01 (2)
C(2′)*A*	0·04 (2)	C(5′)*A*	−0·06 (2)	N′*A*	0·09 (1)

(III) Plane through O*A*, N*A*, O′*A*, N′*A*

$$0\cdot8869X+0\cdot3541Y-0\cdot2966Z+2\cdot5899=0$$

O*A*	0·05 (1)	Fe–*A*	0·581 (2)
N*A*	−0·06 (1)	C(8)*A*	−0·31 (2)
O′*A*	−0·05 (1)	C(8′)*A*	0·31 (2)
N′*A*	0·06 (1)		

(IV) Plane through O*B*, C(1)*B*, C(2)*B*, C(3)*B*, C(4)*B*, C(5)*B*, C(6)*B*, C(7)*B*, N*B*

$$-0\cdot8870X-0\cdot4508Y-0\cdot1004Z+3\cdot8497=0$$

O*B*	−0·07 (1)	C(3)*B*	0·05 (2)	C(6)*B*	−0·02 (2)
C(1)*B*	−0·03 (2)	C(4)*B*	−0·02 (2)	C(7)*B*	−0·01 (2)
C(2)*B*	0·05 (2)	C(5)*B*	−0·03 (2)	C(8)*B*	0·08 (1)

(V) Plane through O′*B*, C(1′)*B*, C(2′)*B*, C(3′)*B*, C(4′)*B*, C(5′)*B*, C(6′)*B*, C(7′)*B*, N′*B*

$$-0\cdot8808X-0\cdot3199Y-0\cdot3489Z+5\cdot4960=0$$

O′*B*	0·02 (1)	C(3′)*B*	−0·05 (2)	C(6′)*B*	0·05 (2)
C(1′)*B*	0·04 (2)	C(4′)*B*	0·00 (2)	C(7′)*B*	−0·05 (2)
C(2′)*B*	−0·03 (2)	C(5′)*B*	0·04 (2)	N′*B*	−0·02 (1)

(VI) Plane through O*B*, N*B*, O′*B*, N′*B*

$$-0\cdot8663X-0\cdot3608Y-0\cdot3456Z+5\cdot5060=0$$

O*B*	−0·08 (1)	Fe–*B*	0·572 (2)
N*B*	0·08 (1)	C(8)*B*	0·31 (2)
O′*B*	0·08 (1)	C(8′)*B*	−0·36 (2)
N′*B*	−0·08 (1)		

angles also seem to have little effect on the displacements of the iron atoms from their respective coordination planes. These displacements are remarkably constant (see Table 7), ranging between 0·55 and 0·58 Å, and are very similar to the values listed in Table 1 for the other LFe–O–FeL structures studied to date. They may also be compared with the iron atom displacements of 0·475 Å in α-chlorohaemin (Koenig, 1965), 0·455 Å in methoxyironmesoporphyrin IX-dimethylester (Hoard, Hamor, Hamor & Caughey, 1965) and 0·38 Å in chloroirontetraphenylporphine (Hoard, Cohen & Glick, 1967).

The Fe–O–Fe bridge in unsolvated $[Fe(salen)]_2O$ is symmetrical within experimental error, the Fe–O (bridge) bond length being 1·78 (1) Å and typical of the short Fe–O (bridge) distances observed in the other oxo-bridged iron(III) structures which have so far been studied (Table 1). However, although the Fe–O–Fe angles in $[Fe(salen)]_2O.2py$, $[Fe(salen)]_2O.CH_2Cl_2$ and unsolvated $[Fe(salen)]_2O$ are very similar with an average of 142°, they are 20–30° smaller than the Fe–O–Fe angles in $[Fe(HEDTA)]_2O^{2-}$, $[Fe(TPP)]_2O$, [Fe(*N*-n-propyl-sal)$_2$]$_2$O and [Fe(*N*-*p*-chlorophenyl-sal)$_2$]$_2$O (Table 1). It is difficult to account for this large angular difference. Intermolecular (crystal packing) forces do not seem to be an important factor since the Fe–O–Fe geometries in $[Fe(salen)]_2O.2py$, $[Fe(salen)]_2O.CH_2Cl_2$ and unsolvated $[Fe(salen)]_2O$ are virtually identical. The smaller Fe–O–Fe angle in $[Fe(salen)]_2O$ could perhaps be interpreted in terms of a smaller degree of π-bonding with the oxygen atom. Yet if, as suggested by Coggon, McPhail, Mabbs & McLachlan (1971), the relative lengths of the Fe–O (bridge) bonds are taken as a measure of the degree of multiple bonding, then this must be the same in all cases. Perhaps the best explanation (Coggon *et al.*, 1971) is to attribute the difference to intramolecular forces and assume that the angle at the oxygen atom is readily distorted whereas the Fe–O bond lengths are not.

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Table 7. *Conformational data* for* $[Fe(salen)]_2O$

	NOON (°)	*d* (Å)	α (°)	β (°)	γ (°)	d_1 (Å)	d_2 Å)	φ (°)
$[Fe(salen)]_2O.2py$†	15·7	0·570	19·7	21·4	41·0	−0·42	0·36	40·3
	7·9	0·549	9·9	15·8	24·1	−0·46	0·32	44·8
$[Fe(salen)]_2O.CH_2Cl_2$‡	8·0	0·570	11·28	23·57	34·3	−0·22	0·40	35·2
	15·0	0·550	11·92	14·67	26·4	−0·35	0·47	43·6
$[Fe(salen)]_2O$§	4·5	0·581	4·50	14·25	10·7	−0·31	0·31	39·8
	6·3	0·572	2·50	15·02	16·2	−0·37	0·31	41·2

* For each Fe(salen) moiety, NOON and φ are the torsional angles N–O–O′–N′ and N–C(8)–C(8′)–N′ respectively and d, d_1, d_2 are the displacements from the coordination plane of atoms Fe, C(8) and C(8′) respectively. The angles α, β and γ are defined in Fig. 5.

† Data from Calligaris, Nardin & Randaccio (1972).

‡ Calculated from the coordinates given by Coggon, McPhail, Mabbs & McLachlan (1971).

§ This work.

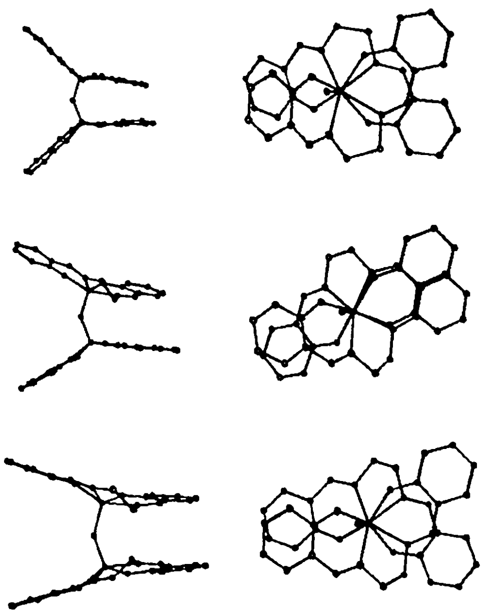

Fig. 6. (*a*) $[Fe(salen)]_2O.2py$ (Gerloch, McKenzie & Towl, 1969), (*b*) $[Fe(salen)]_2O.CH_2Cl_2$ (Coggon, McPhail, Mabbs & McLachlan, 1971), and (*c*) unsolvated $[Fe(salen)]_2O$ (this work). In each case, the diagram on the left is drawn with the Fe–O–Fe plane parallel to the plane of the paper and that on the right is drawn looking down the Fe–Fe direction.

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A Neutron-Diffraction Study of Anhydrous Uranium Tetrachloride

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A neutron diffraction powder study of UCl_4 was carried out, and confirmed the structure derived in an earlier X-ray diffraction powder study. The space group is $I4_1/amd$ (D_{4h}^{19}, No. 141). The X-ray positional parameters for Cl were found to need revision and are now: $y_{\rm Cl}=0{\cdot}3125$ (5) and $z_{\rm Cl}=0{\cdot}9261$ (5). The two sets of U–Cl distances in the coordination dodecahedron were found to be more nearly equal: U–Cl = 2·869 (3) (4 ×) and U–Cl = 2·638 (4) Å (4 ×). The angles these bonds make with the $\bar{4}$ axis are 32·7 (1) and 77·9 (1)°.

Introduction

UCl_4, $ThCl_4$, $PaCl_4$ and $NpCl_4$ are said to be isostructural (Brown & Jones, 1967; Elson, Fried, Sellers & Zachariasen, 1950; Fried & Davidson, 1948; Mooney, 1949). The latter author found UCl_4 to be body-centred tetragonal with the space group $I4_1/amd$ (D_{4h}^{19} No. 141) and the unit-cell dimensions $a=8{\cdot}296$ (9) and