ACTA CHEMICA SCANDINAVICA 24 (1970) 2916–2928

Studies on Flavin Derivatives

The Crystal Structure of 1,10-Ethylene-7,8-dimethylisoalloxazinium Iodide Monohydrate

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The crystal structure of 1,10-ethylene-7,8-dimethylisoalloxazinium iodide monohydrate, $C_{14}H_{13}N_4O_2I \cdot H_2O$, has been determined and refined from three-dimensional X-ray data. The unit cell is monoclinic with the constants $a = 11.417$, $b = 10.788$, and $c = 13.812$ Å, $\beta = 114.78°$. The space group is $P2_1/c$; there is one formula unit in the asymmetric unit and all atoms occupy fourfold positions. Planar cations form sheets between iodide ions. Two N—H···O hydrogen bonds with a length of 2.88 Å, related by a centre of symmetry are found in the structure.

A comparison between bond lengths found in the structure and bond lengths predicted from Pariser-Parr-Pople calculation is made.

Derivatives of isoalloxazine play an important role in many enzymes showing red-ox activity. Thus isoalloxazine forms a part of the coenzymes FAD, flavin adenosine dinucleotide, and FMN, flavin mononucleotide, which function as coenzymes in the oxidation of amino acids, amines, carboxylic acids, reduced nicotinamide nucleotides *etc.* Structural problems raised by these activities are of different types, for instance interaction with proteins and catalytic functions.

A structural investigation of 1,10-ethylene-7,8-dimethylisoalloxazinium iodide monohydrate, $C_{14}H_{13}N_4O_2I \cdot H_2O$, has been undertaken as part of a research program on flavin compounds. The primary object of this structure determination was to study the influence of an ethylene bridge between N(1) and N(10) (see Figs. 1) on the isoalloxazinium ring system.

EXPERIMENTAL

Red single crystals, suitable for X-ray diffraction studies, were kindly supplied by F. Müller.* (Found: C 40.5; H 3.57; N 13.4. Calc. for $C_{14}H_{13}N_4O_2I \cdot H_2O$: C 40.7; H 3.41; N 13.6).

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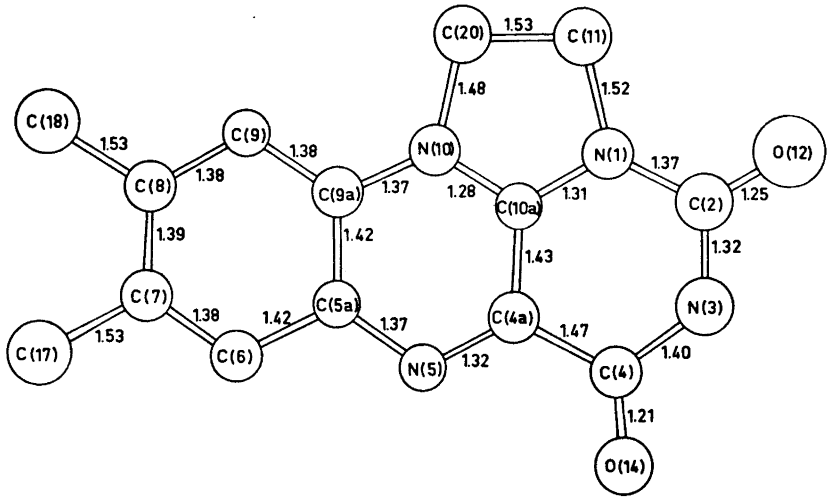

Fig. 1 a. Bond lengths (in Å) in the 1,10-ethylene-7,8-dimethylisoalloxazinium ion.

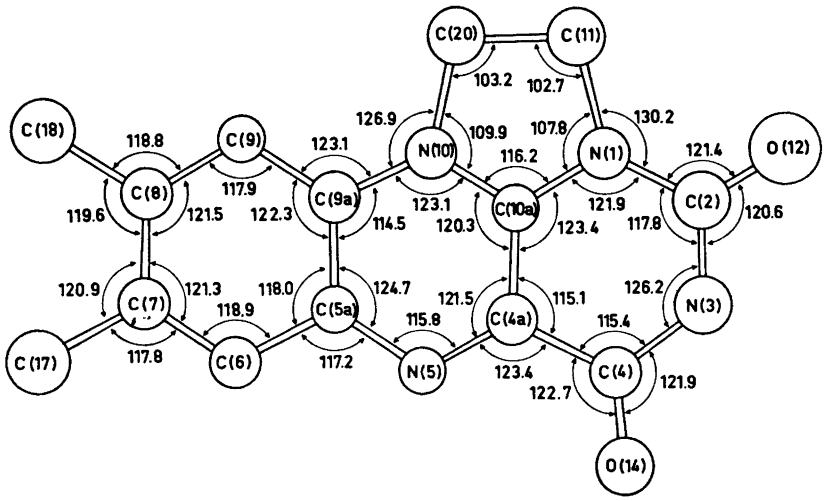

Fig. 1 b. Bond angles in the 1,10-ethylene-7,8-dimethylisoalloxazinium ion.

Preliminary Weissenberg and rotation photographs indicated monoclinic symmetry. Systematic absences occurred for $(h0l)$ reflections with l odd and for $(0k0)$ reflections with k odd; hence, the space group $P2_1/c$ was assumed.

Powder photographs were taken in a Guinier-Hägg focusing camera with Cu$K\alpha_1$ radiation ($\lambda = 1.54056$ Å) and with potassium chloride ($a = 6.2930$ Å) as an internal standard. The least-squares refined [1] unit cell dimensions obtained are shown in Table 1.

A crystal which measured $0.006 \cdot 0.07 \cdot 0.16$ mm^3 was used for the collection of X-ray diffraction data. The crystal was mounted on a Siemens AED goniostat with the long

Table 1. Crystal data.

The estimated standard deviations are given in parentheses and refer to the last decimal place of the respective value.

Lattice constants	$a=11.417(4)$ Å
	$b=10.788(3)$ Å
	$c=13.812(4)$ Å
	$\beta=114.78(2)°$
Cell volume	$V=1544.4$ Å^3
Density (X-ray)	1.781 g/cm^3
Molecules per unit cell	$Z=4$
Space group	$P2_1/c$

edge, which was parallel to b, as phi axis. Ni-filtered CuK radiation was used, and the intensities were collected by the $\theta-2\theta$ scanning technique. The 2251 independent reflections up to a limit of $2\theta=120°$ for CuK radiation were measured twice, with a scan range of 3°. 1166 reflections with $(I_t+I_b)^{\frac{1}{2}}/(I_t-I_b)$ less than 0.25, where I_t and I_b are the total and background counts, were used in the structure determination. The observed intensities were corrected for absorption ($\mu=170$ cm^{-1}) and Lorentz-polarisation factors by the use of a program written by the authors.[2] The unobserved intensities were omitted from all calculations.

STRUCTURE DETERMINATION AND REFINEMENT

The iodide ion position was deduced from the Patterson function and refined by least-squares treatment. A final R index = 0.26 ($R=\sum||kF_o|-|F_c||/\sum|kF_o|$) was obtained. The signs of the structure factors calculated for the iodide ion only were used to calculate three-dimensional Fourier electron density syntheses. From these, approximate parameters for some of the light atoms were derived. Determination of the unknown part of the structure, except the hydrogen atoms, was carried out by iterative Fourier calculations.

Table 2. Weight analysis obtained in the final cycle of the least-squares refinement of 1,10-ethylene-7,8-dimethyl-isoalloxazinium iodide monohydrate. The averages $w(|F_o|-|F_c|)^2=\overline{w\Delta^2}$ are normalized.

Interval sin θ	Number of independent reflections	$\overline{w\Delta^2}$	Interval F_{obs}	Number of independent reflections	$\overline{w\Delta^2}$
0.0 −0.409	197	1.342	0.0− 19.2	116	0.760
0.409−0.515	170	0.609	19.2− 22.0	115	0.603
0.515−0.589	167	0.816	22.0− 25.3	115	1.026
0.589−0.648	134	0.654	25.3− 29.0	115	0.765
0.648−0.699	139	1.260	29.0− 33.5	116	0.598
0.699−0.742	112	0.842	33.5− 39.5	115	1.096
0.742−0.781	89	0.738	39.5− 47.8	115	1.415
0.781−0.817	79	1.909	47.8− 59.7	115	0.663
0.817−0.850	46	1.051	59.7− 79.0	117	1.753
0.850−0.880	23	1.307	79.0−556.7	117	1.308

Table 3. Observed and calculated structure factors. Reflections marked with an asterisk were assigned zero weight in the least-squares refinement.

H	K	L	|FO|	FC	H	K	L	|FC|	FC	H	K	L	FC	FC	H	K	L	|FO|	FC
0	2	0	210.4	-222.3	4	1	1	64.7	-57.3	1	8	3	43.7	41.0	1	6	5	40.2	39.1
0	4	0	47.9	45.2	4	2	1	55.6	-57.0	2	1	3	100.6	-73.2	1	7	5	50.7	-49.6
0	6	0	33.2	29.9	4	3	1	66.4	103.8	2	3	3	72.4	74.9	1	8	5	24.2	21.8
0	8	0	63.4	-65.6	4	4	1	56.1	61.4	2	4	3	31.4	-33.0	1	9	5	16.7	17.0
0	10	0	19.7	14.4	4	5	1	79.0	-88.7	2	5	3	58.3	-98.7	2	2	5	64.5	-68.9
0	12	0	23.9	-19.4	4	6	1	17.7	19.9	2	6	3	36.9	33.6	2	3	5	21.2	-20.0
1	0	0	80.2	-79.7	4	7	1	24.1	38.7	2	7	3	70.2	69.4	2	4	5	80.5	82.4
1	1	0	173.6	-179.6	4	8	1	24.2	-38.8	2	8	3	29.0	-26.6	2	5	5	21.0	21.8
1	2	0	47.6	47.5	5	2	1	67.9	63.3	2	9	3	20.2	-17.4	2	6	5	67.9	-66.8
1	3	0	88.8	86.0	5	4	1	77.6	-86.8	3	2	3	29.2	31.0	2	7	5	17.5	-20.1
1	4	0	40.4	-31.6	5	6	1	35.4	50.6	3	3	3	74.1	-74.7	2	8	5	32.3	28.2
1	7	0	53.3	-49.5	6	1	1	39.2	37.3	3	4	3	71.2	-74.6	3	1	5	42.3	-44.3
1	9	0	53.8	55.0	6	3	1	36.8	-38.1	3	5	3	52.5	51.6	3	2	5	35.0	33.6
1	11	0	35.3	-36.3	6	4	1	26.4	26.9	3	6	3	55.0	55.1	3	3	5	77.5	80.3
2	0	0	189.0	-195.3	6	5	1	54.2	59.6	3	8	3	42.6	-41.6	3	4	5	36.5	37.9
2	1	0	63.5	58.8	6	7	1	30.0	-39.5	4	2	3	40.9	-41.4	3	5	5	70.3	-70.8
2	2	0	96.4	100.5	7	1	1	15.8	-16.4	4	4	3	45.1	44.4	3	6	5	20.6	20.1
2	4	0	68.2	-65.3	7	2	1	31.9	-32.8	4	5	3	42.0	40.7	3	7	5	33.9	34.2
2	5	0	22.2	20.0	7	3	1	26.2	25.2	4	6	3	52.4	-50.3	3	8	5	34.2	-33.6
2	7	0	38.5	35.3	7	4	1	35.1	36.5	4	7	3	77.9	-76.0	3	10	5	38.5*	0.1
2	8	0	42.9	40.1	7	5	1	18.2	-21.8	4	8	3	19.8	20.7	4	2	5	51.1	49.9
2	10	0	36.1	-36.5	7	6	1	28.8	-34.6	4	9	3	23.5	24.7	4	4	5	65.6	-68.4
2	11	0	19.9	18.9	8	1	1	33.0	-29.4	5	3	3	70.8	66.1	4	6	5	40.1	39.3
3	0	0	108.3	107.0	8	4	1	27.4	-29.5	5	4	3	26.6	28.7	5	1	5	28.2	26.9
3	1	0	113.6	105.4	8	6	1	22.7	25.3	5	5	3	49.0	-48.8	5	3	5	27.1	-31.3
3	2	0	124.8	-118.4	0	0	2	27.4*	-6.0	5	6	3	16.8	-19.9	5	4	5	24.5	23.8
3	3	0	57.4	-59.8	0	1	2	22.7	-17.0	5	7	3	26.5	28.8	5	5	5	46.9	45.8
3	4	0	21.7	25.3	0	2	2	85.0	83.3	6	2	3	24.0	26.3	5	6	5	20.4	-17.3
3	7	0	24.3	25.8	0	3	2	141.6	-131.4	6	3	3	23.8	22.1	5	7	5	30.7	-30.9
3	8	0	25.4	-24.6	0	4	2	22.4	-23.4	6	4	3	45.9	-46.8	6	2	5	25.3	-25.4
3	9	0	29.5	-32.7	0	7	2	24.1	22.6	6	6	3	38.1	38.6	6	3	5	23.8	20.9
3	10	0	30.2	26.9	0	8	2	29.1	27.2	6	8	3	20.4	-18.7	6	4	5	25.9	27.7
3	11	0	20.9	23.5	0	9	2	46.6	-48.6	7	3	3	30.5	-32.0	6	5	5	19.8	-19.8
4	0	0	39.4	44.2	0	10	2	28.9	-32.1	7	5	3	32.3	29.3	6	6	5	28.2	-26.5
4	1	0	107.9	-110.1	0	11	2	40.6	44.8	7	7	3	29.7	-25.5	7	1	5	21.0	-19.9
4	2	0	93.4	-80.2	1	0	2	142.4	141.7	8	1	3	20.2	-16.9	7	4	5	25.5	-24.9
4	3	0	92.7	89.0	1	2	2	151.2	-154.3	8	4	3	56.6	50.5	7	6	5	23.8	20.8
4	6	0	18.4	17.1	1	3	2	16.2	-18.9	8	6	3	30.0	-29.2	0	0	6	106.9	103.0
4	10	0	20.6	20.3	1	5	2	20.0	17.5	9	3	3	17.5	17.6	0	2	6	103.1	-109.3
4	11	0	33.8	-31.3	1	6	2	35.2	35.8	9	5	3	23.9	-23.0	0	4	6	20.3	17.7
5	0	0	76.5	-79.3	1	7	2	20.9	21.7	0	0	4	97.4	-91.8	0	6	6	24.6	24.5
5	1	0	43.6	-44.7	1	8	2	40.0	-42.0	0	1	4	135.7	-143.7	0	8	6	33.8	-33.3
5	2	0	114.6	107.1	1	10	2	40.7	41.6	0	2	4	46.7	45.5	0	10	6	43.9	34.0
5	4	0	29.1	-29.2	2	0	2	56.0	57.4	0	3	4	72.5	71.7	1	0	6	21.4	25.1
5	7	0	33.1	-32.5	2	1	2	171.1	-172.9	0	4	4	30.5	-31.2	1	1	6	112.2	-116.4
5	9	0	18.0	21.7	2	2	2	66.3	-63.6	0	7	4	42.2	-40.2	1	2	6	23.5	-28.5
5	10	0	33.5	-27.3	2	3	2	90.2	95.5	0	9	4	46.2	48.0	1	3	6	68.3	65.1
6	0	0	16.1	-18.7	2	7	2	25.2	-38.4	1	0	4	134.0	-142.8	1	7	6	30.8	-30.9
6	1	0	78.2	79.6	2	9	2	45.2	51.7	1	1	4	58.9	60.8	1	9	6	35.8	40.9
6	2	0	18.5	13.2	2	11	2	24.0	-28.9	1	2	4	73.3	78.2	2	0	6	95.6	-103.3
6	3	0	61.1	-55.4	3	0	2	146.7	-146.1	1	4	4	49.1	-48.9	2	1	6	20.4	17.7
6	5	0	31.9	-27.2	3	2	2	80.2	80.5	1	5	4	17.6	19.5	2	2	6	65.5	63.5
7	0	0	56.1	53.0	3	3	2	35.5	-34.0	1	7	4	30.4	29.8	2	3	6	21.9	-24.8
7	2	0	39.1	-38.3	3	4	2	48.4	-50.2	1	8	4	35.9	34.6	2	4	6	32.6	-34.9
7	4	0	17.6	20.4	3	8	2	48.7	57.6	1	9	4	19.9	-18.8	2	8	6	42.3	42.9
7	8	0	23.5	-23.4	3	10	2	27.9	-38.2	2	0	4	89.2	89.9	3	0	6	45.6	49.8
8	0	0	16.3	-19.9	4	0	2	73.8	61.5	2	1	4	77.1	77.7	3	1	6	53.4	56.7
8	1	0	58.3	-59.1	4	1	2	73.7	77.6	2	2	4	94.4	-97.8	3	3	6	25.7	-24.2
8	2	0	15.7	16.1	4	2	2	15.3	14.7	2	3	4	46.3	-47.0	3	6	6	17.9	19.2
8	3	0	21.4	19.6	4	3	2	29.6	-30.9	2	4	4	24.1	22.8	3	7	6	29.1	29.7
9	1	0	71.1	62.8	4	6	2	24.0	25.0	2	7	4	19.9	21.1	3	8	6	37.8	-38.6
9	4	0	17.9	-14.0	4	7	2	32.2	41.5	2	8	4	22.9	-22.4	3	9	6	40.1	-41.7
10	0	0	27.1	24.7	4	8	2	36.4	-47.2	2	9	4	25.9	-28.5	4	0	6	65.8	67.6
10	1	0	18.2	10.7	4	9	2	45.3	-54.3	2	10	4	26.1	25.2	4	1	6	41.7	-41.6
10	2	0	19.5	-16.8	5	0	2	106.1	105.9	3	0	4	35.0	36.0	4	2	6	41.6	-41.4
0	1	1	19.7	8.7	5	1	2	52.0	-53.3	3	1	4	58.0	-55.8	4	4	6	19.3	17.9
0	2	1	104.5	-107.3	5	2	2	60.3	-59.9	3	2	4	60.2	-58.0	4	7	6	24.5	-21.9
0	3	1	102.5	109.6	5	4	2	20.6	26.8	3	3	4	74.9	73.8	4	8	6	16.6	-13.8
0	4	1	64.3	63.1	5	7	2	27.3	-28.4	4	0	4	71.4	-69.3	5	0	6	47.1	-43.8
0	5	1	136.0	-125.8	5	8	2	20.9	-20.6	4	1	4	27.3	-29.3	5	1	6	24.5	-23.3
0	6	1	62.2	-66.9	6	0	2	50.3	-55.3	4	2	4	92.1	91.3	5	2	6	35.4	33.7
0	7	1	42.1	39.8	6	1	2	33.8	-32.8	4	4	4	24.0	-23.6	5	3	6	22.0	21.0
0	8	1	58.4	52.5	6	2	2	38.7	43.5	4	7	4	28.3	-29.3	5	4	6	17.2	-14.7
0	9	1	16.6	-18.2	6	3	2	29.2	29.4	5	1	4	64.6	66.9	5	8	6	21.5	17.6
1	2	1	86.0	85.1	6	4	2	18.2	-19.4	5	3	4	50.6	-48.2	6	0	6	20.0	-15.0
1	3	1	129.8	120.3	6	8	2	16.8	22.3	5	9	4	26.7	-23.6	6	1	6	46.9	43.4
1	4	1	71.9	-70.9	6	9	2	16.2	18.4	6	0	4	47.8	45.8	6	2	6	17.8	16.3
1	5	1	77.7	-79.1	7	0	2	23.9	-22.9	6	2	4	33.8	-32.4	6	3	6	16.6	18.0
1	6	1	82.5	81.4	7	1	2	59.6	56.9	6	8	4	21.5	-20.2	6	7	6	17.9	15.5
1	7	1	53.8	52.8	7	2	2	27.6	26.3	7	1	4	46.0	-46.5	7	1	6	23.9	19.2
1	8	1	18.1	-21.5	7	3	2	15.3	-20.5	7	2	4	19.5	16.4	7	2	6	22.9	-21.6
1	9	1	18.8	-23.3	7	7	2	26.0	22.4	7	3	4	17.9	14.7	8	0	6	22.6	19.8
2	1	1	21.3	20.3	8	0	2	18.8	-9.9	8	1	4	58.6	53.9	8	1	6	22.6	-22.4
2	2	1	48.1	49.0	8	1	2	35.0	29.2	9	0	4	21.4	21.7	1	1	7	40.8	-42.6
2	3	1	82.0	-82.7	8	2	2	26.8	-28.1	5	2	4	16.3	-14.0	1	3	7	52.0	51.5
2	4	1	70.5	-68.7	9	0	2	30.7	26.3	0	1	5	18.8	-20.4	1	4	7	31.7	-28.6
2	5	1	86.5	86.2	9	1	2	25.0	-28.0	0	2	5	61.5	65.0	1	5	7	68.2	-66.5
2	6	1	54.3	56.3	9	2	2	19.1	-13.6	0	3	5	78.6	81.4	1	6	7	32.9	27.9
2	7	1	59.2	-57.4	0	1	3	31.0	35.8	0	4	5	61.2	-63.5	1	7	7	48.7	47.9
2	8	1	32.4	-31.1	0	3	3	114.2	-112.1	0	5	5	59.6	-56.9	1	8	7	20.8	-22.8
2	9	1	20.8	16.4	0	4	3	20.7	-23.9	0	6	5	63.2	68.3	2	2	7	23.0	21.8
3	2	1	90.2	-90.7	0	5	3	136.6	136.7	0	7	5	40.2	40.0	2	3	7	49.5	-50.5
3	3	1	23.7	-30.5	0	6	3	48.3	48.7	0	8	5	22.3	-22.9	2	4	7	46.8	-51.5
3	4	1	101.9	102.0	0	7	3	76.9	-72.9	1	1	5	16.2	16.8	2	5	7	39.8	39.8
3	5	1	31.0	32.9	0	9	3	23.8	23.4	1	2	5	31.9	32.5	2	6	7	34.0	36.4
3	6	1	77.6	-80.9	1	2	3	83.1	-89.5	1	3	5	71.0	-71.5	2	8	7	24.0	-28.1
3	7	1	24.0	-29.6	1	4	3	123.0	121.7	1	4	5	47.9	-46.9	3	2	7	25.1	-26.7
3	8	1	33.5	33.4	1	6	3	75.4	-75.5	1	5	5	74.7	73.1	3	4	7	36.5	36.6

Table 3. Continued.

H	K	L	|FO|	FC	H	K	L	|FO|	FC	H	K	L	|FO|	FC	H	K	L	|FO|	FC
3	5	7	27.3	27.2	0	2	12	30.4	-33.5	-4	10	2	42.1	-39.5	-2	4	4	19.0	-16.4
3	6	7	35.3	-36.8	1	1	12	36.5	-37.5	-5	0	2	67.4	51.2	-2	6	4	15.3	12.2
3	7	7	46.9	-53.9	1	2	12	16.5	-15.5	-5	1	2	87.0	-85.2	-2	7	4	45.5	45.6
4	3	7	45.1	44.0	1	3	12	27.3	27.8	-5	2	2	38.0	25.0	-2	9	4	43.7	-49.2
4	4	7	15.8	17.8	2	0	12	31.7	-30.1	-5	3	2	33.2	33.5	-3	0	4	161.7	-165.1
4	5	7	35.1	-35.8	2	2	12	32.4	35.3	-5	6	2	23.8	23.6	-3	1	4	40.4	-42.1
4	7	7	20.0	23.6	3	1	12	25.2	23.7	-5	7	2	52.0	-45.9	-3	2	4	90.5	94.0
5	2	7	20.9	19.2	0	4	13	25.4	27.5	-5	8	2	56.8	-47.3	-3	4	4	58.9	-58.8
5	4	7	30.5	-34.6	1	3	13	24.8	24.6	-5	9	2	56.3	55.7	-3	5	4	18.0	-14.9
5	6	7	23.3	28.0	0	0	14	23.6	-28.3	-6	0	2	128.4	120.6	-3	7	4	29.2	-29.3
6	3	7	28.4	-23.2	-1	1	1	42.7	35.6	-6	1	2	50.1	51.8	-3	8	4	37.3	36.6
6	5	7	22.0	21.3	-1	2	1	24.3	-23.6	-6	2	2	63.9	-65.2	-3	10	4	32.6	-34.2
7	4	7	37.6	33.9	-1	3	1	117.6	-116.7	-6	4	2	27.5	29.7	-4	0	4	89.7	84.6
0	0	8	85.4	-82.5	-1	4	1	28.0	31.3	-6	7	2	28.8	29.2	-4	1	4	98.3	-97.2
0	1	8	47.8	49.1	-1	5	1	150.9	142.9	-6	8	2	24.1	-23.9	-4	2	4	95.3	-93.2
0	2	8	49.9	49.9	-1	6	1	63.0	-61.6	-6	10	2	20.6	19.1	-4	3	4	56.4	55.9
0	3	8	25.3	-18.1	-1	7	1	63.7	-76.2	-7	0	2	46.7	-52.8	-4	4	4	18.8	20.2
0	4	8	25.7	-27.3	-1	9	1	27.8	24.9	-7	1	2	40.0	38.5	-4	7	4	26.1	-25.0
0	7	8	18.1	18.8	-2	2	1	105.6	101.4	-7	2	2	41.3	42.3	-4	8	4	22.1	-22.1
0	8	8	23.5	23.6	-2	4	1	151.2	-137.0	-7	3	2	31.1	-31.5	-4	9	4	28.7	29.6
1	0	8	61.7	59.6	-2	6	1	81.1	79.2	-7	4	2	20.4	-18.7	-4	10	4	26.3	22.7
1	1	8	43.1	43.9	-2	8	1	45.0	-45.6	-7	6	2	17.5	-13.5	-5	0	4	42.8	46.9
1	2	8	58.3	-61.5	-3	1	1	90.5	-87.0	-7	8	2	23.7	22.3	-5	1	4	91.1	91.1
1	3	8	25.3	-27.6	-3	3	1	85.9	85.7	-7	9	2	22.7	-20.4	-5	2	4	87.2	-79.0
1	4	8	20.1	16.7	-3	4	1	31.6	27.7	-8	0	2	27.1	-26.9	-5	3	4	74.8	-76.6
1	7	8	16.2	14.1	-3	5	1	116.0	-112.0	-8	1	2	64.3	-58.0	-5	10	4	18.0	20.7
1	8	8	18.8	-16.3	-3	6	1	31.2	-30.8	-8	2	2	33.4	32.4	-6	0	4	70.1	-71.9
1	9	8	38.8	-19.6	-3	7	1	72.2	77.8	-8	7	2	30.4	-24.6	-6	1	4	42.7	45.1
2	0	8	20.2	23.7	-3	8	1	27.7	23.7	-9	0	2	24.2	-13.8	-6	2	4	91.0	91.6
2	1	8	67.9	-65.3	-4	2	1	40.9	-38.5	-9	1	2	41.2	-34.7	-6	4	4	29.6	-27.9
2	2	8	33.0	-32.8	-4	3	1	85.5	-84.9	-9	2	2	27.5	-26.9	-6	7	4	27.2	27.9
2	3	8	49.9	48.8	-4	4	1	80.6	82.9	-10	0	2	33.2	27.9	-6	9	4	21.0	-21.9
2	8	8	26.5*	1.4	-4	5	1	53.4	51.2	-10	1	2	24.3	27.5	-6	10	4	30.5	-24.3
2	9	8	54.3*	9.2	-4	6	1	66.9	-66.5	-10	2	2	28.9	-17.1	-7	1	4	71.3	-70.8
3	0	8	53.5	-50.3	-4	8	1	49.1	47.7	-1	2	3	112.8	113.9	-7	2	4	20.9	18.1
3	2	8	60.9	61.8	-5	2	1	49.5	49.7	-1	3	3	83.6	95.1	-7	3	4	56.6	54.1
3	7	8	20.0	-21.8	-5	4	1	41.9	-42.0	-1	4	3	69.1	-64.8	-7	9	4	26.8	25.1
4	1	8	44.3	44.0	-5	5	1	52.3	49.0	-1	5	3	109.1	-105.9	-8	0	4	45.3	48.6
4	3	8	29.0	-30.8	-5	6	1	56.2	54.0	-1	6	3	68.3	71.6	-8	2	4	34.9	-36.6
5	0	8	31.9	30.6	-5	7	1	81.1	-87.0	-1	7	3	31.6	32.2	-8	8	4	21.4	-21.6
5	2	8	23.5	-21.4	-5	9	1	30.1	29.6	-1	8	3	54.2	-49.0	-9	1	4	56.5	58.9
6	1	8	31.4	-30.2	-6	2	1	22.2	22.1	-1	11	3	41.8*	-4.6	-9	3	4	22.0	-19.8
7	1	8	39.9	35.7	-6	3	1	85.9	75.0	-2	2	3	70.8	-71.3	-10	0	4	17.3	-18.2
0	1	9	15.6	13.2	-6	4	1	33.1	-35.4	-2	3	3	127.8	116.0	-10	1	4	63.1	-58.3
0	3	9	50.4	-49.1	-6	5	1	51.1	-52.7	-2	4	3	59.3	59.4	-10	4	4	16.1	-15.0
0	4	9	26.2	-25.4	-6	6	1	23.3	25.5	-2	5	3	87.8	-82.9	-11	0	4	22.8	22.1
0	5	9	50.2	45.7	-6	7	1	30.2	28.1	-2	6	3	71.6	-71.3	-11	1	4	17.5	-13.1
0	6	9	28.7	20.6	-6	8	1	19.5	-18.6	-2	7	3	55.4	54.8	-11	3	4	15.7	10.4
0	7	9	32.6	-33.5	-7	2	1	28.1	-28.6	-2	9	3	22.4	-25.2	-1	2	5	59.0	-63.5
1	2	9	39.5	-40.9	-7	3	1	36.6	31.2	-3	1	3	23.5	22.9	-1	3	5	79.0	81.1
1	4	9	52.2	52.5	-7	4	1	52.0	51.2	-3	2	3	50.1	-51.6	-1	4	5	54.7	52.0
1	6	9	41.5	-43.7	-7	5	1	16.6	-15.6	-3	3	3	71.7	-71.9	-1	5	5	102.5	-100.9
2	1	9	24.6	-26.6	-7	6	1	41.2	-42.1	-3	4	3	72.2	71.6	-1	6	5	46.0	-48.7
2	3	9	46.9	48.8	-7	8	1	21.3	19.4	-3	5	3	79.8	77.8	-1	7	5	38.4	37.2
2	4	9	42.3	18.2	-8	3	1	34.9	-35.0	-3	6	3	58.9	-59.8	-1	8	5	46.1	42.2
2	5	9	46.1	-44.8	-8	5	1	35.5	32.6	-3	7	3	52.5	-50.8	-2	1	5	25.7	25.9
2	6	9	18.1	17.6	-8	7	1	30.9	-28.4	-3	8	3	32.9	33.0	-2	2	5	28.2	-26.9
2	7	9	23.6	23.8	-8	9	1	15.5	12.4	-4	2	3	83.6	83.2	-2	3	5	86.5	-86.2
3	2	9	30.5	31.8	-9	1	1	22.3	-16.8	-4	3	3	31.8	-36.4	-2	4	5	36.4	32.9
3	4	9	44.9	-43.0	-9	4	1	62.9	-58.0	-4	4	3	91.6	-92.5	-2	5	5	108.6	108.9
3	6	9	24.7	23.4	-9	6	1	32.0	32.7	-4	5	3	35.3	37.9	-2	6	5	49.6	-53.9
4	1	9	21.4	15.9	-9	8	1	19.0	-16.2	-4	6	3	72.5	75.2	-2	7	5	58.8	-61.0
4	3	9	21.7	-20.4	-10	3	1	20.5	21.3	-4	7	3	26.1	-31.8	-2	9	5	22.5	20.3
4	4	9	16.8	16.4	-10	5	1	28.5	-26.4	-4	8	3	35.2	-31.8	-3	2	5	77.6	75.9
4	5	9	28.1	27.8	-11	4	1	15.4	13.8	-4	9	3	20.4	18.7	-3	4	5	116.2	-112.3
5	2	9	17.6	-14.5	-1	0	2	30.3*	-5.1	-5	1	3	55.5	-50.2	-3	6	5	68.4	66.2
0	1	10	63.7	-65.2	-1	1	2	21.3	17.0	-5	2	3	73.2	61.3	-3	8	5	39.0	-39.7
0	3	10	38.6	36.5	-1	2	2	86.0	85.3	-5	3	3	100.3	98.4	-4	1	5	59.9	-63.2
0	7	10	19.4	-20.0	-1	3	2	137.4	123.9	-5	4	3	70.2	-67.3	-4	3	5	74.4	73.8
0	8	10	44.9*	1.1	-1	4	2	20.1	-20.8	-5	5	3	88.8	-85.2	-4	4	5	18.7	16.0
1	0	10	57.0	-58.9	-1	7	2	25.0	-22.8	-5	7	3	31.7	35.1	-4	5	5	91.8	-94.3
1	2	10	41.6	39.0	-1	8	2	30.5	29.1	-5	8	3	36.8	34.9	-4	6	5	21.0	-19.5
1	4	10	17.7	-19.0	-1	9	2	42.2	47.0	-6	2	3	68.1	-60.3	-4	7	5	63.0	66.4
1	8	10	36.4	26.2	-1	10	2	36.2	-35.5	-6	4	3	85.9	84.4	-4	8	5	18.5	15.0
2	0	10	26.4	30.7	-1	11	2	46.1	-42.2	-6	6	3	49.1	-50.2	-4	9	5	16.6	-18.7
2	1	10	30.6	32.9	-2	0	2	137.9	143.3	-6	8	3	20.3	21.5	-5	2	5	40.6	-39.1
2	7	10	18.3	17.3	-2	1	2	22.6	-29.8	-7	1	3	38.5	37.7	-5	3	5	65.9	-66.5
3	0	10	33.9	35.1	-2	2	2	147.8	-151.6	-7	3	3	36.5	-37.9	-5	4	5	66.4	68.9
3	1	10	25.9	-25.5	-2	3	2	35.0	33.5	-7	4	3	24.6	-22.5	-5	5	5	37.6	37.6
3	2	10	25.6	-23.3	-2	5	2	20.0	-20.2	-7	5	3	60.7	59.8	-5	6	5	59.6	-62.9
4	0	10	29.5	-27.5	-2	6	2	36.5	37.3	-7	7	3	38.8	-40.4	-5	8	5	38.7	41.8
4	2	10	19.8	20.8	-2	7	2	21.3	-21.5	-8	2	3	34.2	33.1	-6	2	5	42.2	43.1
5	1	10	29.0	26.7	-2	8	2	44.8	-41.8	-8	3	3	21.5	23.3	-6	4	5	32.4	-32.0
0	1	11	17.7	-20.9	-2	9	2	23.2	21.1	-8	4	3	38.8	-37.6	-6	5	5	48.6	46.3
0	3	11	25.9	28.8	-2	10	2	44.8	47.4	-8	6	3	37.9	36.4	-6	6	5	43.7	44.5
0	4	11	20.1	-19.1	-3	1	2	187.4	179.6	-9	1	3	40.1	-32.5	-6	7	5	74.2	-76.4
0	5	11	35.7	-36.2	-3	0	2	69.7	71.1	-9	3	3	17.1	12.1	-6	9	5	26.5	27.9
0	6	11	18.7	18.8	-3	2	2	79.0	-77.7	-9	4	3	27.4	28.4	-7	2	5	22.6	21.4
0	7	11	26.8	26.7	-3	3	2	97.6	-100.5	-9	6	3	24.0	-24.4	-7	3	5	62.1	60.9
1	3	11	28.3	-30.2	-3	7	2	36.1	37.7	-1	0	4	556.5	539.3	-7	4	5	31.0	-33.2
1	4	11	25.2	-29.1	-3	9	2	54.5	-52.2	-1	2	4	164.4	-172.8	-7	5	5	43.5	-44.3
1	5	11	26.3	24.8	-3	11	2	31.1	28.4	-1	4	4	37.5	37.5	-7	6	5	26.2	25.5
1	6	11	18.8	15.5	-4	0	2	147.6	-152.8	-1	6	4	24.2	25.8	-7	7	5	21.7	21.9
2	4	11	25.4	24.0	-4	2	2	74.8	76.1	-1	8	4	55.5	-57.4	-8	2	5	23.6	-25.2
2	6	11	23.8	-21.8	-4	3	2	34.7	29.4	-2	0	4	31.4	-26.7	-8	3	5	31.9	30.9
3	3	11	18.7	23.7	-4	4	2	54.6	-51.6	-2	1	4	146.5	149.9	-8	4	5	40.1	44.3
0	0	12	34.2	33.5	-4	6	2	17.0	-12.6	-2	2	4	28.8	28.6	-9	3	5	29.3	-30.1
0	1	12	18.3	20.3	-4	8	2	63.9	61.5	-2	3	4	76.3	-75.4	-9	5	5	26.3	28.7

Table 3. Continued.

H	K	L	|FO|	FC	H	K	L	|FC|	FC	H	K	L	|FC|	FC	H	K	L	|FO|	FC
-9	7	5	25.8	-24.7	-4	2	7	36.1	-39.3	-3	1	9	18.1	16.4	-4	4	11	20.1	20.8
-10	4	5	53.9	-53.3	-4	3	7	53.4	-49.1	-3	2	9	17.8	-19.9	-4	5	11	42.6	-41.6
-10	6	5	27.7	29.0	-4	4	7	55.4	55.4	-3	3	9	54.4	-52.9	-4	6	11	25.0	-23.5
-11	3	5	20.6	20.1	-4	5	7	55.4	54.2	-3	4	9	28.5	27.1	-4	7	11	28.8	28.6
-11	5	5	23.0	-24.2	-4	6	7	48.4	-47.6	-3	5	9	65.6	66.1	-5	2	11	25.5	-24.6
-1	0	6	18.8	-16.1	-4	7	7	32.9	-34.3	-3	6	9	33.8	-37.2	-5	3	11	27.9	-27.6
-1	1	6	23.0	11.5	-4	8	7	26.0	26.7	-3	7	9	39.5	-38.8	-5	4	11	34.2	35.4
-1	2	6	51.3	57.1	-5	2	7	52.8	54.2	-4	2	9	45.7	45.5	-5	5	11	33.1	30.5
-1	3	6	87.2	-89.7	-5	3	7	34.5	-33.0	-4	4	9	68.3	-70.8	-5	6	11	29.7	-29.5
-1	4	6	20.9	-19.7	-5	4	7	62.3	-62.6	-4	6	9	46.1	44.5	-5	7	11	19.6	-18.0
-1	7	6	21.3	19.7	-5	5	7	34.7	33.8	-4	8	9	25.5	-27.2	-5	8	11	16.6	17.2
-1	8	6	16.7	20.1	-5	6	7	54.0	54.1	-5	1	9	30.5	-34.5	-6	2	11	25.0	28.0
-1	9	6	27.7	-41.0	-5	7	7	23.7	-25.8	-5	3	9	51.5	49.9	-6	3	11	27.1	-22.9
-1	10	6	39.9	-22.2	-5	8	7	26.0	-24.3	-5	5	9	62.4	-61.7	-6	4	11	34.7	-33.8
-1	11	6	34.0	37.6	-6	1	7	29.4	-30.8	-5	7	9	41.6	44.5	-6	5	11	24.5	23.5
-2	0	6	26.1	-20.6	-6	2	7	48.1	43.7	-6	2	9	33.7	-31.3	-6	6	11	30.7	31.9
-2	1	6	16.6	-8.2	-6	3	7	67.2	69.1	-6	3	9	37.0	-39.7	-6	8	11	16.5	-15.1
-2	2	6	60.4	64.7	-6	4	7	50.8	-52.0	-6	4	9	44.0	45.9	-7	2	11	20.9	25.3
-2	3	6	73.1	74.5	-6	5	7	83.1	-82.9	-6	5	9	19.3	20.9	-7	3	11	36.0	39.1
-2	4	6	21.1	-17.6	-6	7	7	23.5	25.8	-6	6	9	39.5	-46.8	-7	4	11	29.6	-33.0
-2	7	6	17.4	-19.9	-6	8	7	25.7	25.2	-6	8	9	30.0	28.7	-7	5	11	37.1	-37.1
-2	8	6	19.4	24.4	-7	2	7	46.7	-43.7	-7	2	9	25.2	27.3	-7	7	11	17.8	14.9
-2	9	6	33.6	36.9	-7	4	7	65.5	63.1	-7	4	9	21.3	-19.4	-8	2	11	28.6	-26.6
-2	10	6	27.5	-30.2	-7	6	7	37.8	-38.3	-7	5	9	34.2	34.8	-8	4	11	39.5	37.9
-3	0	6	104.5	103.2	-7	8	7	20.8	17.2	-7	6	9	22.6	28.6	-8	6	11	24.0	-22.8
-3	1	6	37.7	-38.1	-8	1	7	28.3	27.7	-7	7	9	49.0	-54.9	-9	3	11	21.1	-20.2
-3	2	6	88.9	-101.1	-8	3	7	32.3	-30.5	-7	9	9	16.9	19.9	-9	5	11	25.8	28.6
-3	3	6	32.2	34.3	-8	5	7	45.8	46.3	-8	2	9	16.5	16.1	-10	4	11	22.0	-19.1
-3	6	6	27.1	27.4	-8	6	7	19.6*	5.2	-8	3	9	36.2	36.8	-11	1	11	20.3	-18.7
-3	8	6	32.1	-32.6	-8	7	7	34.5	-32.5	-8	4	9	21.6	-24.6	-1	1	12	32.4	31.1
-3	9	6	19.8	19.4	-9	2	7	27.4	26.0	-8	5	9	29.4	-29.2	-1	2	12	26.5	26.9
-3	10	6	31.4	31.2	-9	4	7	30.4	-30.2	-8	6	9	20.1	19.8	-2	0	12	43.1	-43.7
-4	0	6	51.2	53.3	-9	6	7	32.6	30.8	-8	7	9	20.1	13.6	-2	1	12	46.9	-47.1
-4	1	6	119.2	124.3	-10	1	7	30.7	-27.2	-9	2	9	20.3	-18.0	-2	2	12	20.9	20.7
-4	2	6	53.8	-56.0	-10	4	7	21.0	22.2	-9	3	9	21.1	22.8	-2	3	12	21.6	22.8
-4	3	6	70.4	-72.2	-10	6	7	17.4	-18.5	-9	4	9	27.0	30.9	-3	0	12	111.4	123.9
-4	7	6	28.7	30.0	-11	6	7	22.3*	-4.3	-9	5	9	18.1	-13.4	-3	1	12	17.3	-17.5
-4	9	6	40.0	-41.9	-1	0	8	74.2	-68.3	-9	6	9	25.9	-24.3	-3	2	12	51.7	-51.7
-5	C	6	109.0	-115.9	-1	1	8	91.1	-90.3	-10	3	9	21.8	-20.5	-4	1	12	54.4	55.1
-5	2	6	57.7	56.7	-1	2	8	36.2	33.0	-11	4	9	38.5	-38.0	-4	3	12	28.4	-29.8
-5	4	6	33.9	-37.9	-1	3	8	45.7	45.9	-1	0	10	67.1	62.4	-4	7	12	17.8	18.3
-5	8	6	50.6	52.4	-1	4	8	16.3	-20.5	-1	2	10	64.0	-64.4	-5	0	12	58.6	-57.1
-5	10	6	32.8	-32.4	-1	7	8	23.9	-25.5	-1	4	10	21.4	14.9	-5	2	12	41.2	39.4
-6	0	6	22.8	26.9	-1	9	8	65.4	33.2	-1	8	10	18.8	-21.5	-6	0	12	24.2	21.8
-6	1	6	70.5	-72.9	-2	0	8	254.6	267.5	-2	C	10	22.8	-15.7	-6	1	12	38.8	-40.2
-6	2	6	20.0	19.8	-2	1	8	20.7	-22.0	-2	1	10	24.5	16.2	-6	2	12	22.3	-22.4
-6	3	6	25.5	30.9	-2	2	8	106.6	-100.5	-2	2	10	30.8	31.6	-6	3	12	18.8	24.5
-6	7	6	38.5	-39.6	-2	4	8	23.8	23.4	-2	3	10	51.5	-50.2	-7	0	12	27.7	27.4
-6	8	6	35.1	-37.5	-2	8	8	48.8	-39.0	-2	8	10	16.6	11.9	-7	1	12	31.5	32.5
-6	9	6	42.9	45.9	-3	1	8	100.0	99.0	-3	0	10	35.8	-26.8	-7	2	12	32.2	-35.2
-7	0	6	91.0	56.2	-3	3	8	51.8	-52.5	-3	2	10	44.0	41.9	-7	3	12	25.4	-28.6
-7	1	6	34.4	36.6	-3	7	8	31.6	32.1	-3	3	10	35.6	36.2	-8	C	12	28.8	-30.8
-7	2	6	52.7	-52.6	-3	9	8	33.4	-34.6	-3	8	10	18.1	16.5	-8	1	12	19.0	19.0
-7	4	6	21.9	24.0	-4	0	8	111.1	-104.0	-3	9	10	22.0	22.9	-8	2	12	28.6	32.4
-7	7	6	21.7	23.3	-4	1	8	25.2	-24.4	-4	0	10	63.9	60.8	-9	1	12	25.0	-27.0
-7	8	6	21.8	-21.5	-4	2	8	69.7	67.7	-4	1	10	45.9	-30.9	-11	1	12	27.C	29.8
-8	C	6	35.1	-37.7	-4	4	8	37.6	-38.3	-4	2	1C	7C.2	-56.1	-1	3	13	24.7	-27.7
-8	1	6	38.3	37.2	-4	8	8	26.9	26.6	-5	C	1C	33.8	32.3	-1	5	13	26.6	27.6
-8	2	6	29.5	30.6	-5	0	8	51.4	47.7	-5	1	10	71.7	68.7	-2	2	13	17.1	19.8
-8	3	6	27.0	-26.4	-5	1	8	66.3	-68.1	-5	2	10	30.6	-32.0	-2	3	13	19.6	21.5
-8	8	6	24.8	18.0	-5	2	8	47.8	-51.2	-5	3	10	41.2	-40.0	-2	4	13	20.8	-25.7
-8	9	6	18.3	-17.4	-5	3	8	40.9	41.0	-5	7	10	28.6	19.9	-2	6	13	22.4	23.8
-9	0	6	25.2	-24.1	-5	7	8	18.8	-18.7	-5	9	10	38.3	-26.8	-3	3	13	20.5	22.5
-9	1	6	46.2	-45.2	-5	9	8	18.3	21.1	-6	0	10	71.2	-69.9	-3	4	13	21.4	15.6
-9	2	6	27.7	29.7	-6	0	8	41.4	40.6	-6	2	10	38.4	35.1	-3	5	13	31.8	-35.9
-9	4	6	19.2	-10.2	-6	1	8	60.6	58.6	-6	4	10	23.2	-22.0	-4	3	13	27.0	-27.6
-9	7	6	24.3	-21.1	-6	2	8	56.6	-58.0	-6	8	10	35.7	36.1	-4	4	13	19.2	17.4
-10	0	6	21.9	-14.3	-6	3	8	47.4	-49.8	-7	1	10	51.3	-47.9	-4	5	13	32.6	33.6
-10	1	6	35.8	-30.9	-6	4	8	17.5	20.5	-7	3	10	22.8	21.0	-4	6	13	18.1	-21.6
-10	2	6	18.2	-18.7	-7	0	8	56.3	-51.8	-7	7	10	29.2	-28.3	-5	4	13	32.7	-35.9
-10	3	6	18.5	7.3	-7	1	8	32.8	32.9	-7	8	10	19.4	-23.1	-5	6	13	24.9	24.2
-11	0	6	26.6	25.2	-7	2	8	58.0	59.5	-8	0	10	56.5	59.3	-6	1	13	18.2	-16.4
-11	1	6	17.9	22.1	-7	4	8	20.5	-20.2	-8	2	10	36.9	-33.4	-6	3	13	27.C	27.1
-11	2	6	19.9	-17.4	-7	7	8	19.3	19.5	-8	4	1C	18.C	15.0	-6	5	13	29.4	-33.1
-1	1	7	31.2	32.2	-8	1	8	46.8	-48.5	-8	7	1C	17.4	14.9	-7	2	13	19.7	-20.2
-1	3	7	83.7	-81.1	-8	2	8	13.2	17.3	-9	0	10	23.4	-21.0	-7	4	13	22.9	24.9
-1	4	7	15.8	-14.4	-8	3	8	35.1	37.3	-9	1	10	28.8	28.4	-7	6	13	23.0	-28.3
-1	5	7	94.9	97.9	-9	0	8	37.2	35.6	-10	0	10	22.3	-16.4	-8	5	13	22.4	21.0
-1	6	7	24.0	26.9	-9	2	8	28.6	-28.2	-10	1	10	28.7	-27.7	-9	3	13	16.6	17.6
-1	7	7	54.7	-54.3	-9	8	8	16.3	-16.1	-10	2	10	21.8	21.0	-1	1	14	29.6	-31.0
-2	2	7	76.7	81.0	-10	1	8	44.6	46.5	-11	1	10	24.6	-22.0	-1	3	14	17.7	17.2
-2	3	7	51.5	54.9	-10	3	8	19.3	-15.1	-1	2	11	33.5	-31.7	-2	0	14	28.6	31.0
-2	4	7	59.3	-54.1	-11	1	8	44.8	-44.4	-1	4	11	42.0	45.5	-2	1	14	20.0*	3.5
-2	5	7	60.1	-62.4	-1	2	9	38.5	39.C	-1	6	11	34.4	-34.9	-2	2	14	27.5	-32.2
-2	6	7	56.2	58.2	-1	3	9	40.2	44.0	-2	3	11	48.1	-48.6	-3	3	14	20.5	-24.8
-2	7	7	24.3	19.8	-1	4	9	43.3	-44.9	-2	5	11	54.5	57.8	-4	0	14	20.6	-18.2
-2	8	7	34.1	-35.4	-1	5	9	30.7	-32.4	-2	7	11	31.9	-32.8	-4	2	14	19.C	22.0
-3	1	7	18.2	-17.3	-1	6	9	40.1	44.6	-3	2	11	44.5	47.9	-5	C	14	25.3	29.3
-3	2	7	40.4	-41.8	-1	7	9	24.2	23.7	-3	3	11	20.2	24.9	-5	2	14	23.2	-26.9
-3	3	7	76.5	79.3	-2	2	9	30.5	-32.1	-3	4	11	38.9	-34.4	-6	1	14	30.3	32.1
-3	4	7	38.3	38.9	-2	3	9	44.4	46.5	-3	5	11	28.1	-30.8	-6	2	14	16.8	-16.4
-3	5	7	66.2	-65.8	-2	4	9	35.3	33.6	-3	6	11	36.1	36.2	-7	0	14	29.7	-34.1
-3	6	7	47.5	-46.2	-2	5	9	60.6	-64.2	-3	8	11	18.5	-20.6	-7	2	14	16.3	16.8
-3	7	7	45.1	44.5	-2	6	9	31.5	-27.7	-4	2	11	20.6	-20.3	-8	1	14	23.4	-24.6
-3	9	7	18.2	-21.4	-2	7	9	26.7	27.8	-4	3	11	43.6	45.3	-9	0	14	27.5	30.6
-4	1	7	22.0	19.9	-2	8	9	27.3	25.9										

The atomic parameters were refined by the least-squares method [3] using isotropic temperature factors for the light non-hydrogen atoms and an anisotropic temperature factor for the iodide ion. The scattering factor curves used for oxygen, carbon, and nitrogen were those given by Freeman [4] and for the iodide ion that by Cromer and Waber,[5] corrected by the real part of the anomalous dispersion coefficient. Hughes' weighting procedure [6] with $F_{o\,min}=18$ and $h=4$ was applied. The refinement was terminated when all shifts of the parameters in one cycle were less than one tenth of their estimated standard deviations. At this stage the R value was 0.067. A weight analysis obtained in the last cycle is given in Table 2. The observed and calculated structure amplitudes are listed in Table 3.

Table 4. Fractional atomic coordinates and thermal parameters. The estimated standard deviations are given in parentheses and refer to the last decimal places of the respective values.

	x	y	z	B
I	0.2886 (1)	0.2026 (1)	0.3253 (1)	*a*
N(1)	0.2057(14)	0.3298(12)	0.0584(10)	3.7(3)
C(2)	0.0870(19)	0.2764(19)	0.0272(14)	4.4(4)
N(3)	0.0798(15)	0.1546(14)	0.0200(12)	4.6(4)
C(4)	0.1836(18)	0.0727(16)	0.0467(14)	3.7(4)
C(4a)	0.3104(19)	0.1312(16)	0.0774(13)	3.4(4)
N(5)	0.4183(13)	0.0677(11)	0.1015(10)	2.8(3)
C(5a)	0.5280(17)	0.1369(15)	0.1284(12)	2.9(3)
C(6)	0.6452(18)	0.0715(16)	0.1520(14)	3.7(4)
C(7)	0.7585(18)	0.1377(17)	0.1839(14)	4.1(4)
C(8)	0.7596(16)	0.2665(15)	0.1908(13)	3.6(4)
C(9)	0.6476(17)	0.3332(14)	0.1662(13)	3.1(4)
C(9a)	0.5331(18)	0.2680(16)	0.1354(13)	3.5(4)
N(10)	0.4165(14)	0.3239(13)	0.1099(10)	3.6(3)
C(10a)	0.3106(16)	0.2633(14)	0.0831(13)	2.9(3)
C(11)	0.2436(23)	0.4653(21)	0.0662(16)	5.5(5)
O(12)	−0.0133(16)	0.3407(15)	−0.0012(13)	7.0(4)
O(14)	0.1696(13)	−0.0387(12)	0.0476(10)	5.2(3)
C(17)	0.8841(22)	0.0647(19)	0.2141(17)	5.0(5)
C(18)	0.8869(21)	0.3369(18)	0.2221(17)	5.2(5)
C(20)	0.3905(19)	0.4591(19)	0.1054(14)	4.4(4)
O(21)	0.6384(20)	0.2376(20)	0.4042(17)	10.5(6)

[a] For I the anisotropic temperature factor T obtained was
$T=\exp[0.0148\,h^2+0.0088\,k^2+0.0065\,l^2-0.0005\,hk+0.0085\,hl+0.0010\,kl]$

The coordinates and temperature factors obtained are given in Table 4. These parameters were used for the calculation of three-dimensional difference Fourier syntheses. The interpretation of the Fourier maps was severely hampered by the presence of spurious maxima. Therefore, it was not found possible to select peaks for the hydrogen positions with reasonable confidence.

DESCRIPTION AND DISCUSSION OF THE STRUCTURE

In the following discussion, atoms belonging to different asymmetric units are labelled as follows:

Superscript	Coordinates	Superscript	Coordinates
none	(x,y,z)	v	$(2-x,y-\frac{1}{2},\frac{1}{2}-z)$
i	$(\bar{x},\bar{y},\bar{z})$	vi	$(1-x,y-\frac{1}{2},\frac{1}{2}-z)$
ii	$(x,\frac{1}{2}-y,\frac{1}{2}+z)$	vii	$(x-1,\frac{1}{2}-y,z-\frac{1}{2})$
iii	$(1-x,\bar{y},\bar{z})$	viii	$(x,\frac{1}{2}-y,z-\frac{1}{2})$
iv	$(\bar{x},1-y,\bar{z})$	ix	$(1-x,y+\frac{1}{2},\frac{1}{2}-z)$
		x	$(1+x,\frac{1}{2}-y,\frac{1}{2}+z)$

The structure of the 1,10-ethylene-7,8-dimethylisoalloxazinium ion is shown in Figs. 1. The bond lengths and bond angles uncorrected for thermal motion are given in Table 5.

Table 5. Bond lengths (Å), angles (°) and their standard deviations.

N(1) –C(2)	1.37(2)	C(2) –N(1) –C(10a)	121.9(1.5)	C(6) –C(7) –C(8)	121.3(1.7)		
N(1) –C(10a)	1.31(2)	C(2) –N(1) –C(11)	130.2(1.6)	C(6) –C(7) –C(17)	117.8(1.6)		
N(1) –C(11)	1.52(3)	C(10a)–N(1) –C(11)	107.8(1.5)	C(8) –C(7) –C(17)	120.9(1.6)		
C(2) –N(3)	1.32(3)	N(1) –C(2) –N(3)	117.8(1.7)	C(7) –C(8) –C(9)	121.5(1.6)		
C(2) –O(12)	1.25(3)	N(1) –C(2) –O(12)	121.4(1.8)	C(7) –C(8) –C(18)	119.6(1.6)		
N(3) –C(4)	1.40(2)	N(3) –C(2) –O(12)	120.6(1.9)	C(9) –C(8) –C(18)	118.8(1.5)		
C(4) –C(4a)	1.47(3)	C(2) –N(3) –C(4)	126.2(1.7)	C(8) –C(9) –C(9a)	117.9(1.5)		
C(4) –O(14)	1.21(2)	N(3) –C(4) –C(4a)	115.4(1.5)	C(5a) –C(9a) –C(9)	122.3(1.6)		
C(4a)–N(5)	1.32(2)	N(3) –C(4) –O(14)	121.9(1.7)	C(5a) –C(9a) –N(10)	114.5(1.6)		
C(4a)–C(10a)	1.43(2)	C(4a) –C(4) –O(14)	122.7(1.7)	C(9) –C(9a) –N(10)	123.1(1.5)		
N(5) –C(5a)	1.37(2)	C(4) –C(4a)–N(5)	123.4(1.5)	C(9a) –N(10) –C(10a)	123.1(1.4)		
C(5a) –C(6)	1.42(2)	C(4) –C(4a)–C(10a)	115.1(1.6)	C(9a) –N(10) –C(20)	126.9(1.5)		
C(5a) –C(9a)	1.42(2)	N(5) –C(4a)–C(10a)	121.5(1.6)	C(10a)–N(10) –C(20)	109.9(1.4)		
C(6) –C(7)	1.38(3)	C(4a) –N(5) –C(5a)	115.8(1.3)	N(1) –C(10a)–C(4a)	123.4(1.6)		
C(7) –C(8)	1.39(2)	N(5) –C(5a)–C(6)	117.2(1.4)	N(1) –C(10a)–N(10)	116.2(1.4)		
C(7) –C(17)	1.53(3)	N(5) –C(5a)–C(9a)	124.7(1.5)	C(4a) –C(10a)–N(10)	120.3(1.6)		
C(8) –C(9)	1.38(2)	C(6) –C(5a)–C(9a)	118.0(1.6)	N(1) –C(11) –C(20)	102.7(1.6)		
C(8) –C(18)	1.53(3)	C(5a) –C(6) –C(7)	118.9(1.6)	N(10) –C(20) –C(11)	103.2(1.6)		
C(9) –C(9a)	1.38(2)						
C(9a)–N(10)	1.37(2)						
N(10)–C(10a)	1.28(2)						
N(10)–C(20)	1.48(2)						
C(11) –C(20)	1.53(3)						

A comparison with the crystal structure of 1,3,10-trimethylisoalloxazinium iodide [7] and 1,3,7,8,10-pentamethylisoalloxazinium iodide [8] shows that the presence of an ethylene bridge between N(1) and N(10) makes the cation more planar. The largest deviation of any atom from a least-squares plane through all twenty nonhydrogen atoms in the cation is 0.098 Å for O(14) (*cf.* Table 6).

Table 6. Least squares plane through all nonhydrogen atoms in the 1,10-ethylene-7,8-dimethylisoalloxazinium ion. The plane is described using a vector basis (m,n,p) having $m||a^*,n||b$ and $p||c$. Plane equation: $0.1386\ m-0.0261\ n+0.9900\ p=0.0035$.

Atom	Deviation [a]	Atom	Deviation [a]
N(1)	24	N(10)	34
C(2)	4	C(10a)	34
N(3)	−37	C(11)	−33
C(4)	9	O(12)	−71
C(4a)	−7	O(14)	98
N(5)	−14	C(17)	−12
C(5a)	−28	C(18)	12
C(6)	−75	C(20)	19
C(7)	−31	[b]I	3436
C(8)	23	[b]I(viii)	−3428
C(9)	38	[b]O(21)	3350
C(9a)	14	[b]O(21)(viii)	−3493

[a] Deviations are given in 10^{-3} Å.
[b] Not included in the least-squares plane.

Owing to the presence of the fused five- and six-membered rings the cation is strained, especially at C(11) and C(20) where, at each site, one bond angle deviates more than 3σ from the normally occurring tetrahedral angle, 109.47°, for sp^3 hybridized carbon. The bonds N(1)−C(11), N(10)−C(20), and C(11)−C(20) on the other hand, are all normal single bonds.

Table 7. Comparison between bond lengths calculated by the use of the Pariser-Parr-Pople approximation and bond lengths observed in the crystal structure.

Bond	Observed	Calculated. The iodide ion simulated	Calculated. The iodide ion not simulated
N(1) −C(2)	1.366 Å	1.393 Å	1.395 Å
N(1) −C(10a)	1.313	1.366	1.370
C(2) −N(3)	1.318	1.367	1.368
C(2) −O(12)	1.253	1.239	1.235
N(3) −C(4)	1.399	1.381	1.382
C(4) −C(4a)	1.469	1.469	1.469
C(4) −O(14)	1.213	1.233	1.230
C(4a) −C(10a)	1.427	1.425	1.420
C(4a) −N(5)	1.324	1.327	1.332
N(5) −C(5a)	1.369	1.361	1.356
C(5a) −C(6)	1.425	1.424	1.426
C(5a) −C(9a)	1.418	1.418	1.423
C(6) −C(7)	1.377	1.383	1.383
C(7) −C(8)	1.393	1.415	1.418
C(8) −C(9)	1.381	1.391	1.393
C(9) −C(9a)	1.384	1.410	1.408
C(9a) −N(10)	1.367	1.386	1.386
N(10)−C(10a)	1.285	1.358	1.361

The eight C–N bonds in the isoalloxazinium ring system, ranging from 1.28 to 1.40 Å, are all significantly shorter than a C–N single bond; 1.47 Å.[9] This indicates the presence of a large number of π-electrons in the oxidized isoalloxazinium ring system, which is also in accordance with the planarity of the cation.[10]

Bond lengths, calculated with the Pariser-Parr-Pople (PPP) method in the SCF-MO-LCAO form are compared with the observed values in Table 7. The calculations were performed on an IBM 360/75 computer by means of a program written and kindly put at our disposal by B. Roos, T. Alm and M. Sundbom.* Semi-empirical parameters used in the PPP approximation are derived by a method suggested by Roos and Skancke.[11] The method was first developed and applied to pure unsaturated hydrocarbons. A series of papers has also dealt with extensions to account for methyl groups,[12] different kinds of nitrogen containing molecules,[13] carbonyl,[14] and methylene groups.[15]

One calculation was made, were the iodide ions in the crystal were simulated by a charge of 0.5 e^- on each side of the cation in the iodide positions. Thereby a small improvement in the agreement between observed and calculated bond lengths was achieved (*cf.* Table 7).

The bond lengths are derived from the following bond order-bond length relations:

$$R_{CC} = 1.517 - 0.180\, p_{CC}$$
$$R_{CN} = 1.458 - 0.180\, p_{CN}$$
$$R_{CO} = 1.365 - 0.180\, p_{CO}$$

where R is the interatomic distance and p the corresponding bond order.

With the exceptions for the bonds N(10)–C(10a), N(1)–C(10a), and C(2)–N(3) the agreement between observed and calculated bond lengths is very good. A least-squares refinement of the crystal structure using anisotropic temperature factors for the iodide ion and 14 light atoms ** and isotropic temperature factors for the remaining atoms, was performed. The largest change of bond length obtained from this refinement was found for the C(2)–N(3) bond which increased from 1.32 Å to 1.35 Å. The larger value is more in accordance with the PPP calculations.

The only significant differences between predicted and observed bond lengths are found in the bonds N(10)–C(10a) and N(1)–C(10a). This indicates that it is not possible to explain the planarity of the cation and the short bond lengths in the five-membered ring without consideration of the σ-electrons. Thus there seems to be no simple relationship between the observed bond lengths N(10)–C(10a), 1.28 Å and N(1)–C(10a), 1.31 Å, and corresponding bond orders.

Two intermolecular hydrogen bonds N(3)···O(14)(i), with a length of 2.88 Å, are related by a centre of symmetry (*cf.* Fig. 2). Thus an eight-membered ring including the two hydrogen atoms is obtained. This symmetric

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** The following light atoms were given anisotropic temperature factors: N(1), C(2), C(4), C(4a), N(5), C(5a), C(7), C(8), C(9a), N(10), C(10a), O(12), O(14), and O(21).

type of hydrogen bonding between pyrimidine rings has also been found in other compounds, for example in 5-ethyl-6-methyluracil [16] and in uric acid.[17]

In a review of the internal valence angle at nitrogen atoms in six-membered rings, Singh [18] has pointed out that this angle is $125\pm3°$ when the nitrogen is carrying an extra-annular hydrogen, and $116\pm3°$ when the nitrogen has no attachment. In the present structure the bond angle C(2)−N(3)−C(4) is $126\pm1.7°$. Furthermore, the distance N(3)···O(14)(i), 2.88 Å, and the bond length C(4)−O(14), 1.21 Å, indicate that the hydrogen bond is of the type N(3)−H···O(14)(i) and not of the enolic form N(3)···H−O(14)(i). This is also in agreement with the conclusion, drawn by Dudley, Ehrenberg, Hemmerich and Müller [9] from spectroscopic measurements, that oxidized flavin appears only in diketo form.

Considering the expected difference in hydrogen bonding energy 12−17 kJ for N−H···O and 17−29 kJ for N···H−O [20] and the possible participation of N(3) in the FMN-apoenzyme interaction in NADPH-dehydrogenase ("old yellow enzyme") [21] this hydrogen bond is of particular interest.

Because of the ethylene bridge between N(1) and N(10) there are only three possible hydrogen-bond receptor sites O(12), O(14), and N(5) in the cation. Only O(14) actually participates in hydrogen bonding. This speaks in favour of the hypothesis, advanced by Trus and Fritchie on the basis of a structure determination of 10-methylisoalloxazine hydrobromide dihydrate,[22] that in oxidized protonated flavins, O(14) is the most basic of the receptor atoms.

The comparatively high temperature factor, 10.5, obtained for the crystal water oxygen, O(21), may partly be an effect of the π-electrons from the benzene groups above and below the water molecule. The water molecule does not form any strong hydrogen bond. The shortest distances from the water oxygen to possible hydrogen receptors in the structure are to O(14), 3.14 Å; N(5), 3.61 Å; and to I^-, 3.69 Å. This last figure may indicate the presence of a weak O(21)−H···I^- bond.[23]

The packing in the unit cell is shown in Fig. 2. The cations form sheets separated in the *c*-direction by iodide ions and crystal water. Intermolecular

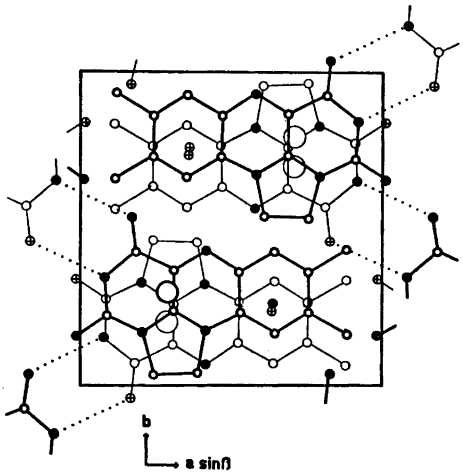

Fig. 2. Projection of 1,10-ethylene-7,8-dimethylisoalloxazinium iodide monohydrate along the *c* axis. Small open circles denote carbon atoms, small filled ones nitrogen atoms and small cross marked circles denote oxygen atoms. The large open circles denote iodide ions.

Table 8. Intermolecular distances less than 3.80 Å between nonhydrogen light atoms. The estimated standard deviations are given in parentheses and refer to the last decimal place of the respective values.

C(2) ···O(14)(i)	3.70(2) Å	O(21)···N(5)(ix)	3.61(2)
N(3) ···N(3)(i)	3.73(2)	···C(5a)	3.64(3)
N(3) ···C(4)(i)	3.69(2)	···C(7)(ii)	3.76(3)
N(3) ···O(14)(i)	2.88(2)	···C(8)	3.76(3)
N(3) ···C(18)(vii)	3.77(3)	···C(8)(ii)	3.60(3)
C(4) ···O(14)(i)	3.70(2)	···C(9)	3.49(3)
C(4) ···C(17)(iii)	3.67(3)	···C(9)(ii)	3.66(3)
N(5) ···C(6)(iii)	3.59(2)	···C(9a)	3.41(3)
C(6) ···C(11)(vi)	3.73(2)	···C(11)(vi)	3.19(3)
C(6) ···C(20)(vi)	3.74(3)	···O(12)(x)	3.72(3)
C(7) ···O(14)(iii)	3.77(2)	···O(14)(vi)	3.14(3)
C(11)···O(12)(iv)	3.18(2)	···C(20)(vi)	3.02(3)
O(12)···O(12)(iv)	3.45(3)		
O(14)···O(14)(i)	3.63(3)		
C(17)···C(18)(v)	3.42(3)		

distances less than 3.8 Å between nonhydrogen light atoms are listed in Table 8. The normal van der Waals distance, 3.40 Å,[24] is shortened to 3.02 Å for C(20)···O(21), to 3.18 Å for C(11)···O(21) and to 3.18 Å for C(11)···O(12)(iv). This may be related to the unusual bond angles, 103.2° and 102.7° at C(20) and C(11), respectively (*cf.* Fig. 1 b).

The position of the iodide ion is similar to the iodide positions in the crystal structures of 1,3,10-trimethylisoalloxazinium iodide [7] and 1,3,7,8,10-pentamethylisoalloxazinium iodide.[8] Data on the coordination are listed in Table 9.

Table 9. Distances (Å) from the iodide ion to N(1), C(4), C(4a), N(5), N(10), C(10a), and O(21). The estimated standard deviations are given in parentheses and refer to the last decimal place of the respective values.

I···N(1)	3.67(1)	I···N(5)	4.20(1)
···N(1)(ii)	3.73(1)	···N(5)(ii)	4.26(1)
···C(4)	3.79(2)	···N(10)	4.04(1)
···C(4)(ii)	4.44(2)	···N(10)(ii)	3.58(1)
···C(4a)	3.62(2)	···C(10a)	3.52(2)
···C(4a)(ii)	3.83(2)	···C(10a)(ii)	3.48(2)
		···O(21)	3.69(2)

Computer programs used for the calculations:

Program name and function. Computer.	Authors.
PIRUM. Indexing of powder photographs and least squares refinement of unit cell parameters.[1] IBM 1800.	P.-E. Werner, Stockholm Sweden.
VIP. Angle settings for three-circle diffractometers.[2] IBM 1800.	R. Norrestam, Stockholm, Sweden.
SIP. Generation of steering paper tape for SIEMENS AED.[2] IBM 360/75.	R. Norrestam, Stockholm, Sweden.
ABS. Calculation of absorption-, extinction-, and Lp-factors.[2] IBM 1800.	P.-E. Werner and M. Leijonmarck, Stockholm, Sweden.

DRF. Fourier summations and structure factor calculations. IBM 360/75.	A. Zalkin, Berkeley, USA. Modified by R. Liminga and J.-O. Lundgren, Upsala, Sweden. Further modified by O. Lindgren, Göteborg and A. G. Nord and B. G. Brandt, Stockholm, Sweden.
LALS. Full matrix least squares refinement of positional and thermal parameters and of scale factors.[3] IBM 360/75.	P. K. Gantzel, R. A. Sparks and K. N. Trueblood, Los Angeles, USA. Modified by A. Zalkin, Berkeley, USA and by J.-O. Lundgren, R. Liminga and C.-I. Brändén, Upsala Sweden, Further modified by O. Lindgren, Göteborg and by B. G. Brandt and A. G. Nord, Stockholm, Sweden.
DISTAN. Calculation of interatomic distances and bond angles with estimated standard deviations, IBM 360/75	A. Zalkin, Berkeley, USA. Modified by A. G. Nord and B. G. Brandt, Stockholm, Sweden.
INERT. Least-squares plane and axes of inertia.[3] IBM 1800.	R. Norrestam, Stockholm, Sweden.
OPSZDO. Pariser-Parr-Pople calculations. IBM 360/75.	T. Alm, B. Roos and M. Sundbom, Stockholm, Sweden.

This research was supported by grants from the *Tri-Centennial Fund of the Bank of Sweden* and from the *Swedish Natural Science Research Council*. The authors are also indebted to Dr. F. Müller for the supply of crystals used in the work. We would also like to express our gratitude to Professor P. Kierkegaard for his active and stimulating interest in this work.

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Received February 27, 1970.