the crystalline sample for this investigation. In addition, we wish to acknowledge the use of the IBM 1620 computer programs of Dr F. R. Ahmed, and the Block Diagonal Least Squares Program by Y. Okaya and D. R. Harris. This work was supported by: USPHS grants GM-09826 from the Institute for General Medical Sciences and NIH-A-3942 from the National Institutes of Health, NSF-GB-429 from the National Science Foundation and a USPHS Traineeship, 2G-718(C2) awarded to one of us (D.J.H.) through the Department of Biophysics, State University of New York at Buffalo.

References

Bello, J. & Haas, D. J. (1965). To be published.
Furnas, T. C. (1957). *Single Crystal Orienter Instruction Manual.* Milwaukee: General Electric Company.
Haas, D. J., Harris, D. R. & Mills, H. H. (1965. *Acta Cryst.* To be published.

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The Crystal and Molecular Structures of *trans*-(1,4), (5,8)-Dimethylene-*cis*, *anti*, *cis*-perhydroanthraquinone

By H. G. Norment*

U.S. Naval Research Laboratory, Washington 25, *D.C., U.S.A.*

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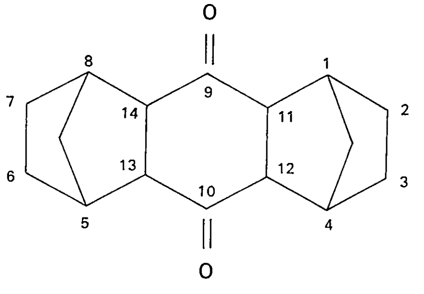
The crystal structure of *trans*-(1,4),(5,8)-dimethylene-*cis,anti,cis*-perhydroanthraquinone, $C_{16}H_{20}O_2$, *Pcab*, $a=10\cdot526$, $b=6\cdot464$, $c=18\cdot307$ Å, $Z=4$, has been solved directly from the normalized structure factor magnitudes with the use of the symbolic addition method. The structure has been refined in three dimensions. Analysis of the anisotropic thermal parameter data indicates that the major thermal vibration or disorder in the crystal is a rigid body molecular displacement in the direction of the crystallographic *b* axis.

Introduction

The compound *trans*-(1,4), (5,8)-*cis,anti,cis*-perhydroanthraquinone was prepared at the University of California, Los Angeles by deVries, Heck, Piccolini & Winstein (1959) as a starting material for the preparation of systems of interest in other investigations. Whereas the bonding configuration has been established by deVries *et al.*, the saturated carbon framework of the molecule allows considerable latitude of steroconfiguration. In order to establish precisely this configuration, the compound has been subjected to X-ray crystal structure analysis.



(I)

The molecular configuration found from the crystal structure analysis is shown in Fig. 1, in which the numbering of the atoms is different from the conventional numbering shown in (I). The numbering shown in Fig. 1 is used hereafter.

Experimental

The sample from which a crystal was selected was synthesized and crystallized successively from ethyl alcohol, acetic acid, and *o*-xylene at the University of California, Los Angeles. It was dried at 110° *in vacuo.* The crystal used to collect all of the diffraction data was a small needle with a cross-section of several tenths of a millimeter. The crystal was mounted on its needle axis which is collinear with the crystallographic *b* axis.

The crystal is orthorhombic, space group *Pcab*, with unit-cell dimensions

$a=10\cdot526\pm0\cdot006$ Å
$b=6\cdot464\pm0\cdot003$
$c=18\cdot307\pm0\cdot012$
$V=1245\cdot7\pm1\cdot3$ Å^3
$d_c=1\cdot302$.
$d_o=1\cdot299$ (by flotation in aqueous sodium iodide).
$Z=4$.

The molecule must possess a center of symmetry since space group *Pcab* has eight equivalent general positi-

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* Present address, Technical Operations Incorporated, Burlington, Massachusetts, U.S.A.

ons. Unit-cell dimensions and errors were calculated from measurements made on twenty pairs of 0*kl* and *hk*0 reflections from two precession photographs (Norment, 1963*a*).

The multiple film equi-inclination Weissenberg method was used to collect intensity data for the *h*0*l*, *h*1*l*, *h*2*l*, and *h*3*l* reflections. Intensities were estimated visually with the use of a set of calibrated reflection spots prepared from the same crystal. A total of 787 independent reflections were observed.

The raw intensity data were punched on IBM cards and input to the IBM 7090 computer for processing by the program of Norment (1962). The data were automatically corrected for Lorentz and polarization effects and scaled to structure factor and normalized structure factor form. Owing to the absence of interpenetrating layers of data, the program found the absolute scale factors independently for each layer. The scaling and normalizing procedures are described by Norment (1963*b*). The observed averages of certain functions of the normalized structure factors E may be compared with theoretical values for centrosymmetric crystals obtained from equation (1·29) of the monograph of Hauptman & Karle (1953):

	Observed	Theoretical
$\langle\lvert E\rvert\rangle$	0·737	0·798
$\langle\lvert E^2-1\rvert\rangle$	1·088	0·968
$\langle E^2\rangle$	1·056	1·000

Phase determination

The crystal structure was solved directly from the normalized structure factor magnitudes by means of the symbolic addition procedure (Karle & Karle, 1963). The main feature of this procedure is the direct implementation of the Σ_2 relation in the monograph by Hauptman & Karle (1953) by means of the assignment of unknown symbols to represent the signs of structure factors. The origin in the crystal was specified by assigning the phases of a proper set of reflections, according to the invariant-seminvariant theory of the monograph; and a probability function derived from equation (3·36) of Hauptman & Karle (1953) and equation (4·12) of Cochran & Woolfson (1955) was used to assess the probability that a phase had been correctly determined. The symbolic addition procedure was applied to the three-dimensional data and led directly to the structure without difficulty.

The data processing program output tape which contained $\mathbf{h}$ and $|E_{\mathbf{h}}|$ for each reflection was passed through a Σ_2 listing program on the IBM 7090 computer (Norment, 1963*a*) which automatically selected the set of reflections for which $|E|\geq 1·5$, arranged the selected set in order of decreasing magnitude of E, and prepared lists of Σ_2 interaction pairs* from the selected set for each reflection in the selected set. There were eighty-nine reflections for which $|E|\geq 1·5$.

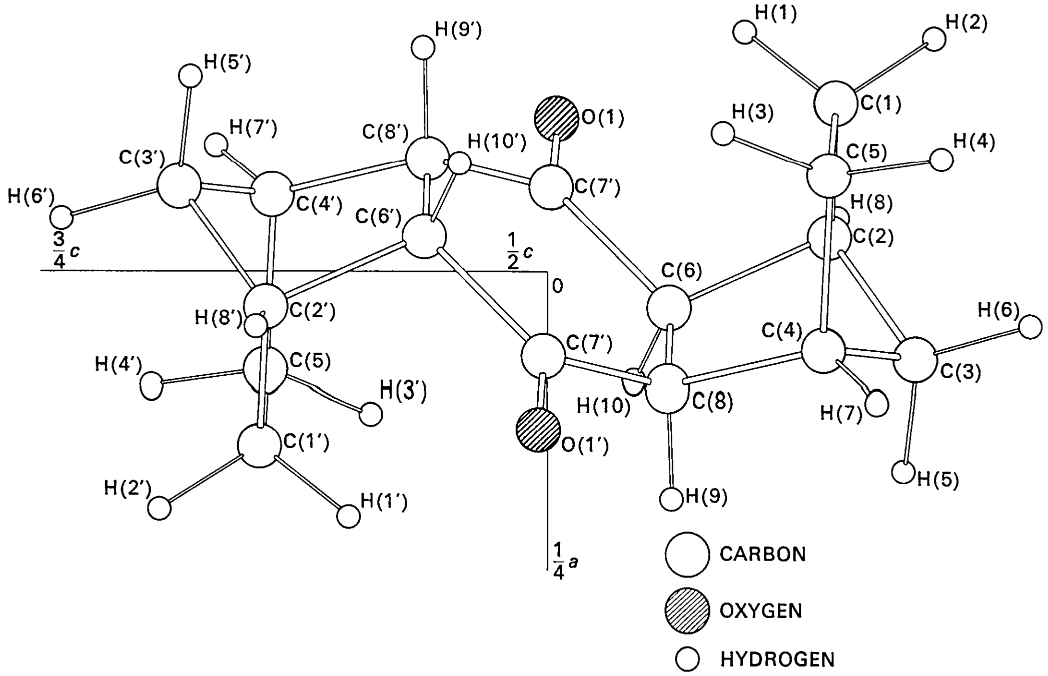

Fig. 1. The molecular configuration of *trans*-(1,4), (5,8)-dimethylene-*cis*, *anti*, *cis*-perhydroanthraquinone.

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* For a reflection defined by its Miller index triple $\mathbf{h}$, a Σ_2 interaction pair is any pair of reflections $\mathbf{h}_i$ and $\mathbf{h}_j$ which satisfy the relation

$$\mathbf{h}=\mathbf{h}_i+\mathbf{h}_j\,.$$

The Σ_2 relation relates the normalized structure factors of all $\mathbf{h}_i$ and $\mathbf{h}_j$ to that of $\mathbf{h}$ *via* the equation

$$sE_{\mathbf{h}}\simeq s\Sigma_{\mathbf{k}}E_{\mathbf{k}}E_{\mathbf{h}-\mathbf{k}}$$

where $\mathbf{k}=\mathbf{h}_i$ and $\mathbf{h}-\mathbf{k}=\mathbf{h}_j$ and s means "the sign of".

These listings were carefully scrutinized and a set of three reflections were selected for arbitrary assignment of phases, following the invariant-seminvariant theory, which satisfied the following requirements:

1. The set of **h** for the three selected reflections is linearly independent modulo 2.
2. The $|E|$ values are large.
3. Each reflection has a relatively long list of Σ_2 interaction pairs which contain among them several interactions among the selected three reflections.

In addition to the phase specifications which fix the origin, two reflections were selected judiciously from the Σ_2 listing so as best to satisfy requirements 2. and 3. and were assigned the symbolic signs a and b.

The five starting reflections are given in Table 1(a). The probability $P_+(E_\mathbf{h})$ that the sign of $E_\mathbf{h}$ is positive is approximately

$$P_+(E_\mathbf{h})=\tfrac{1}{2}+\tfrac{1}{2}\tanh\,(\sigma_3/\sigma_2^{3/2})\,|\,E_\mathbf{h}\,|\,\sum_\mathbf{k} E_\mathbf{k}E_{\mathbf{h}-\mathbf{k}}\,, \quad (3{\cdot}1)$$

where $\sigma_n=\sum_i Z_i^n$ (Hauptman & Karle, 1953; Cochran & Woolfson, 1955). Using the Σ_2 listings and equation (3.1) the set of fifty-one signs listed in Table 1(b) were determined in the order listed. This work was done by hand, care being taken to accept only signs for which the largest probabilities for correctness were obtained. Frequent cross checks and summaries were made by reiterations employing the Σ_2 formula. Certain reflections appearing to have large E magnitudes, which were later found to be actually much smaller, led to frequent inconsistencies. These reflections were detected by the reiteration and eliminated from the early stages of the determination. Signs were determined for one- and two-dimensional reflections as well as three-dimensional ones, but the one- and two-dimensional reflections were not used to determine other signs. Experience has shown that these reflections are involved in erroneous Σ_2 phase indications more frequently than expected.

Since most of the signs in Table 1(b) are functions of the symbolic signs a and/or b it was necessary to determine which of the four assignments for a and b is the correct one. Using only the signs obtained from Tables 1(a) and 1(b), as many sign indications as possible were found for all reflections in the Σ_2 listings for each of the four assignments. Most-probable signs were chosen for as many reflections as possible. Tallies of consistent (assumed correct) indications and inconsistent (assumed incorrect) indications were made and tabulated within bracketed ranges of the product $|E_1E_2E_3|$. These are presented in Table 2 along with the observed percentage consistencies. These may be compared with the theoretical percentage values obtained from equation (3.1). It is clear that even though the case $a=+$, $b=-$ yields the largest number of indications, it is inferior in producing consistent indications. The case $a=-$, $b=-$ was eliminated because by inspection of the data it was apparent that an electron-density Fourier synthesis calculated with signs determined from this assignment would have a large positive peak at the origin. For the remaining two possibilities the assignment $a=-$, $b=+$ yielded considerably more consistency, particularly in the heaviest populated range $5{\cdot}0\le E_1E_2E_3\le 7{\cdot}4$. An additional twenty-one signs were determined on the

Table 1. *Phases determined by the symbolic addition method*

(a) Starting phases

h,k,l	Sign	$\|E\|$
3,3,1	+	4·00
5,2,8	−	2·99
3,1,18	+	2·16
7,3,7	a	3·32
6,2,8	b	2·37

Table 1 (*cont.*)

(b) Phases determined from the starting set

h,k,l	Sign	$\|E\|$	h,k,l	Sign	$\|E\|$
8,1,7	+	2·13	1,3,15	$-ab$	2·22
3,3,15	+	2·68	7,3,15	−	2·12
1,1,15	ab	2·24	8,2,14	$-ab$	2·14
6,2,19	+	2·08	7,3,13	−	2·08
12,1,1	a	1·80	1,1,11	$-a$	1·83
9,2,16	$-a$	3·34	2,1,21	+	1·78
7,2,8	ab	2·23	8,1,3	$-b$	1·76
9,2,2	$-a$	2·16	1,3,1	$-ab$	1·73
6,1,2	a	2·04	2,1,15	$-a$	1·69
1,1,1	ab	1·88	0,0,22	$-a$	6·12
2,2,14	ab	1·87	7,2,18	$-a$	2·67
11,3,6	a	1·86	1,1,21	$-b$	2·35
5,2,6	−	1·84	3,3,7	$-a$	2·00
1,1,9	$-a$	1·78	7,2,6	ab	1·78
2,1,7	+	1·69	3,3,11	$-b$	1·56
12,1,2	$-a$	1·66	4,1,7	$-ab$	1·53
2,1,9	+	1·65	2,0,0	$-ab$	2·29
6,1,17	a	1·64	0,0,6	$-a$	1·70*
0,0,14	+	2·60	2,2,0	ab	1·66
0,0,16	+	2·05	2,2,8	$-b$	1·64
11,3,7	$-a$	2·02	10,2,2	+	3·75*
2,1,13	$-a$	1·98	8,0,12	ab	4·38*
7,3,9	a	1·96	2,0,22	$-b$	3·90*
7,3,1	−	1·92			
3,3,13	+	1·77			
4,2,10	$-a$	1·70			
12,2,8	$-b$	1·66			
8,1,17	$-b$	1·59*			

(c) Additional phases determined on the basis of the assignment $a=-$, $b=+$

h,k,l	Sign	$\|E\|$	h,k,l	Sign	$\|E\|$
7,2,13	−	2·04	1,1,3	+	1·62
10,3,11	+	2·00	7,2,3	+	1·58
3,3,20	+	1·96*	1,3,11	−	2·48
6,1,12	+	1·92	5,2,3	+	1·98
5,2,13	−	1·83	4,1,12	+	1·71
10,3,5	+	1·82	2,3,11	−	1·70
6,1,19	−	1·76	7,1,16	−	1·64*
9,2,4	+	1·75	5,2,9	+	1·53
11,1,4	+	1·75	6,0,0	−	1·53
10,1,3	−	1·65	3,1,6	+	1·52
6,3,1	−	1·64			

* Magnitudes found to be erroneous in the subsequent analysis.

Table 2. *A comparison of consistent and non-consistent phase indications for the four possible assignments for a and b*

X = number of consistent indications
Y = number of inconsistent indications
Z = percentage of consistent indications
The theoretical percentage values were calculated from equation (3·1)

	$\|E_1E_2E_3\|$																				
	≤20			15–19			10–14			7·5–9			5·0–7·4			2·5–4·9			Totals		
	X	Y	Z	X	Y	Z	X	Y	Z	X	Y	Z	X	Y	Z	X	Y	Z	X	Y	Z
$a=+$ $b=+$	5	0	100	13	0	100	44	4	92	43	6	88	61	27	69	12	0	100	178	37	83
$a=+$ $b=-$	4	0	100	14	0	100	44	8	85	44	8	85	75	27	73	16	7	70	197	50	80
$a=-$ $b=+$	5	0	100	13	0	100	42	6	89	41	7	85	80	8	91	9	4	70	190	25	88
$a=-$ $b=-$	5	0	100	12	1	92	48	2	96	41	6	87	69	16	81	13	1	93	188	26	88
Theory		99+				97 to 99			91 to 97			85 to 91			76 to 85			64 to 76			

basis of this assignment. These are listed in Table 1(*c*). A total of seventy-seven signs (10% of the total number of reflections and 88% of the reflections for which $|E| \geq 1·5$) were determined. Of the seventy-seven reflections for which signs were determined, the ensuing analysis showed that four erroneously had been given large $|E|$values. Of the remaining 73 only one sign (2,0,22), $|E| = 3·90$, was found to be incorrect.

The phase determination procedure was handicapped by the fact that the reflections with the second and third largest $|E|$ values, 8,0,12 and 8,0,11, actually should have had zero E values. (The errors were caused by undetected stray punches on the cards input to the data processing program.) The fifth largest $|E|$ value, for reflections 10,2,2, should have been 1·07 instead of 3·75. The determination of the signs for these reflections was very difficult because they interacted relatively infrequently through the Σ_2 relation with other strong reflections. In addition, they were at a considerable disadvantage probability-wise because of their small true E magnitudes and this was manifested by frequent sign inconsistencies among the Σ_2 summands. Of the five incorrect reflections, the sign of one could not be determined, the signs of two were incorrectly determined and the signs of two were determined correctly. These errors were not discovered until the structure was partly refined.

Structure determination

A three-dimensional Fourier map was calculated with the E magnitudes and signs for the 77 reflections as listed in Tables 1(*a*), 1(*b*), and (1*c*), where $a = -$ and $b = +$. The value of using E coefficients in calculating Fourier maps has been described by Karle, Hauptman, Karle & Wing (1958). The eleven highest peaks were selected from the map and these were input to a computer program (Norment, 1963*a*) which interpolates to determine the peak maxima, transforms the atomic coordinates into neighboring symmetrically equivalent positions, and calculates distances and angles between atoms separated by normal intramolecular distances. A structure consistent with the known chemical configuration of the molecule was found in the result. The sixth and eleventh peaks, in order of decreasing peak height, were extraneous. Bond distances for this model are given in Table 3. It is apparent that knowledge of the bonding configuration of the molecule would not have been needed in advance.

Table 3. *Interatomic distances for the crude structure calculated with the 77 directly determined phases*

C(1)–C(2)	1·61 Å	C(2)–C(5)	2·26 Å
C(2)–C(3)	1·49	C(2)–C(7)	2·60
C(3)–C(4)	1·55	C(2)–C(8)	2·63
C(4)–C(5)	1·59	C(3)–C(5)	2·23
C(5)–C(1)	1·38	C(3)–C(6)	2·40
C(2)–C(6)	1·62	C(3)–C(8)	2·45
C(6)–C(8)	1·78	C(4)–C(6)	2·54
C(8)–C(4)	1·55	C(4)–C(7′)	2·55
C(6)–C(7)	1·52	C(5)–C(8)	2·49
C(7)–O(1)	1·20	C(6)–O(1)	2·40
C(8)–C(7′)	1·28	C(6)–C(7′)	2·56
C(1)–C(3)	2·41	C(6)–C(8′)	2·55
C(1)–C(4)	2·53	C(7)–C(8)	2·75
C(1)–C(6)	2·57	O(1)–C(8′)	1·95
C(2)–C(4)	2·34		

Refinement

The structure was refined in three dimensions by least squares on the IBM 7090 computer with the program ORXLS of Busing & Levy (1959*a*). The atomic scattering factors of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) were used for the heavy atoms. For the hydrogen atoms, the

McWeeny (1951) values were used. Four scale factors were refined, one for each of the *b*-axis layers. For the first six cycles, refinement was performed on F^2 with weights of W/F_0^2, where W is the number of intensity readings made. In all subsequent cycles, refinement was performed on F with unit weights. Reflections observed with zero intensity were given zero weights. The first three cycles were done with data for which $\sin\theta/\lambda \leq 0{\cdot}35$; all observed data were used beyond the third cycle. A total of eighteen cycles were run.

Some difficulty was encountered at the beginning of the refinement because of a tendency for the isotropic thermal parameters to become negative. Consequently the first three cycles were done with B fixed at a value of 3 for all atoms.

After the eighth cycle a difference Fourier map was calculated from which seven of the ten hydrogen atoms were located. Atoms H(2), H(8), and H(10) were not found. After the next cycle, which included the seven hydrogen atoms in fixed positions, an additional hydrogen atom, H(2), was found from a difference Fourier map. Two more cycles, with hydrogen atoms in fixed positions, yielded a difference Fourier map from which H(10) was found but H(2) was lost again. (For the purpose of calculating these maps, calculated structure factors for the heavy atoms only were used). Up to this point, cycle 11, isotropic thermal parameters had been used. It had been thought advisable to find the hydrogen atoms before beginning anisotropic thermal parameter refinement. Since this

Table 4. *Final atomic parameters and standard errors*

(*a*) Positional parameters

Atom	x	$\sigma(x)$	y	$\sigma(y)$	z	$\sigma(z)$
C(1)	−0·1441	0·0008	−0·0225	0·0016	0·3571	0·0004
C(2)	−0·0294	0·0008	−0·1753	0·0014	0·3607	0·0004
C(3)	0·0726	0·0008	−0·0519	0·0018	0·3177	0·0005
C(4)	0·0650	0·0007	0·1461	0·0015	0·3639	0·0004
C(5)	−0·0821	0·0008	0·1970	0·0018	0·3609	0·0004
C(6)	0·0291	0·0008	−0·1571	0·0014	0·4394	0·0004
C(7)	−0·0714	0·0008	−0·2072	0·0018	0·4981	0·0004
C(8)	0·0959	0·0006	0·0546	0·0014	0·4409	0·0004
O(1)	−0·1300	0·0007	−0·3681	0·0013	0·4951	0·0003
H(1)	0·189	0·005	0·038	0·008	0·438	0·003
H(2)	0·107	0·007	0·276	0·010	0·337	0·004
H(3)	−0·202	0·006	−0·045	0·009	0·401	0·003
H(4)	−0·045	0·006	−0·376	0·010	0·356	0·003
H(5)	−0·197	0·010	−0·045	0·013	0·309	0·005
H(6)	−0·096	0·006	0·273	0·010	0·305	0·004
H(7)	−0·116	0·007	0·253	0·011	0·412	0·004
H(8)	0·046	0·007	−0·030	0·011	0·261	0·004
H(9)	0·165	0·007	−0·121	0·011	0·323	0·004
H(10)	0·091	0·008	−0·246	0·011	0·456	0·004

(*b*) Thermal parameters

Atom	B	$\sigma(B)$	B_{11}	$\sigma(B_{11})$	B_{22}	$\sigma(B_{22})$	B_{33}	$\sigma(B_{33})$	B_{12}	$\sigma(B_{12})$	B_{13}	$\sigma(B_{13})$	B_{23}	$\sigma(B_{23})$
C(1)			0·0071	0·0008	0·0650	0·0107	0·0012	0·0002	−0·0019	0·0014	−0·0007	0·0004	0·0001	0·0006
C(2)			0·0097	0·0009	0·0373	0·0086	0·0020	0·0002	−0·0002	0·0013	0·0003	0·0004	−0·0002	0·0006
C(3)			0·0066	0·0008	0·0876	0·0113	0·0020	0·0002	0·0035	0·0014	0·0004	0·0004	−0·0016	0·0008
C(4)			0·0055	0·0007	0·0651	0·0096	0·0015	0·0002	−0·0021	0·0011	0·0002	0·0003	0·0011	0·0007
C(5)			0·0082	0·0009	0·0686	0·0111	0·0014	0·0002	0·0033	0·0017	−0·0005	0·0004	0·0005	0·0007
C(6)			0·0075	0·0008	0·0478	0·0098	0·0018	0·0002	0·0026	0·0012	0·0000	0·0004	0·0003	0·0006
C(7)			0·0061	0·0008	0·0622	0·0105	0·0013	0·0002	−0·0009	0·0013	−0·0012	0·0003	0·0012	0·0007
C(8)			0·0035	0·0006	0·0573	0·0103	0·0016	0·0002	−0·0024	0·0010	0·0001	0·0003	0·0005	0·0006
O(1)			0·0097	0·0007	0·0618	0·0097	0·0027	0·0002	−0·0067	0·0012	−0·0007	0·0003	0·0005	0·0006
H(1)	0·9	1·1												
H(2)	2·2	1·6												
H(3)	2·4	1·4												
H(4)	1·4	1·4												
H(5)	6·7	2·4												
H(6)	2·8	1·4												
H(7)	3·6	1·7												
H(8)	4·0	1·9												
H(9)	3·2	1·8												
H(10)	3·6	1·9												

(*c*) Scale factors

Reflection Set	S.F.	σ(S.F.)
*h*0*l*	0·9382	0·0089
*h*1*l*	0·9423	0·0099
*h*2*l*	1·7752	0·0160
*h*3*l*	2·1715	0·0240

Table 5. *Final interatomic bond distances and angles with standard errors*

Bond distances			Angles			Angles		
Atoms	Distance	Standard error	Atoms	Angle	Standard error	Atoms	Angle	Standard error
C(2)–C(1)	1·561 Å	0·012 Å	C(1)–C(2)–C(3)	100·86°	0·69°	C(3)–C(2)–H(8)	124·3°	0·9°
C(3)–C(2)	1·552	0·012	C(2)–C(3)–C(4)	96·48	0·64	C(6)–C(2)–H(8)	100·1	0·9
C(3)–C(4)	1·536	0·013	C(3)–C(4)–C(5)	101·83	0·69	H(6)–C(3)–H(5)	112·5	1·8
C(5)–C(1)	1·563	0·013	C(4)–C(5)–C(1)	102·79	0·75	C(2)–C(3)–H(6)	111·7	1·2
C(5)–C(4)	1·584	0·011	C(5)–C(1)–C(2)	104·43	0·74	C(2)–C(3)–H(5)	111·1	1·3
C(6)–C(2)	1·572	0·010	C(3)–C(2)–C(6)	98·94	0·65	C(4)–C(3)–H(6)	114·0	1·2
C(6)–C(7)	1·543	0·011	C(3)–C(4)–C(8)	99·80	0·71	C(4)–C(3)–H(5)	110·0	1·2
C(7)–O(1)	1·210	0·010	C(2)–C(6)–C(8)	105·14	0·60	C(3)–C(4)–H(7)	112·4	1·1
C(8)–C(4)	1·563	0·010	C(4)–C(8)–C(6)	103·06	0·59	C(5)–C(4)–H(7)	102·9	1·2
C(8)–C(6)	1·539	0·012	C(8)–C(6)–C(7)	119·23	0·68	C(8)–C(4)–H(7)	128·9	1·2
C(7)–C(8′)	1·512	0·012	C(6)–C(7)–O(1)	119·91	0·83	H(4)–C(5)–H(3)	126·7	1·6
C(1)–H(1)	1·02	0·06	C(6)–C(7)–C(8′)	119·59	0·83	C(1)–C(5)–H(4)	107·4	1·0
C(1)–H(2)	1·06	0·09	C(6)–C(8)–C(7′)	121·03	0·65	C(1)–C(5)–H(3)	101·8	1·2
C(2)–H(8)	1·31	0·06	C(2)–C(6)–C(7)	110·70	0·65	C(4)–C(5)–H(4)	104·0	1·1
C(3)–H(5)	1·07	0·07	C(4)–C(8)–C(7′)	112·55	0·74	C(4)–C(5)–H(3)	111·6	1·3
C(3)–H(6)	1·09	0·08	O(1)–C(7)–C(8′)	120·50	0·65	C(4)–C(8)–H(9)	101·8	1·0
C(4)–H(7)	1·07	0·06	H(1)–C(1)–H(2)	108·9	2·0	C(6)–C(8)–H(9)	111·1	1·0
C(5)–H(3)	1·07	0·07	H(1)–C(1)–C(2)	109·8	1·1	C(7′)–C(8)–H(9)	105·8	1·0
C(5)–H(4)	1·14	0·06	H(2)–C(1)–C(2)	110·9	1·6	C(2)–C(6)–H(10)	122·5	1·5
C(6)–H(10)	0·92	0·08	H(1)–C(1)–C(5)	110·0	1·1	C(8)–C(6)–H(10)	102·8	1·5
C(8)–H(9)	0·99	0·05	H(2)–C(1)–C(5)	112·6	1·5	C(7)–C(6)–H(10)	97·2	1·6
			C(1)–C(2)–H(8)	122·0	0·9			

Table 6. *Least-squares planes calculated for the atoms as indicated*

The planes are defined by the equation $m_1x+m_2y+m_3z=d$, where x, y, and z are fractional coordinates and d is the perpendicular distance from the origin to the plane in Å

m_1	m_2	m_3	d	Atoms	Deviation $\times 10^2$(Å)	Standard deviation $\times 10^2$(Å)
−0·5174	−0·07227	18·284	6·616	C(1)	−1·08	1·82
				C(2)	0·73	
				C(4)	−0·72	
				C(5)	1·06	
8·904	−2·618	−6·349	−2·104	C(2)	1·03	2·63
				C(6)	−1·55	
				C(8)	1·54	
				C(4)	−1·03	
7·611	−2·633	10·215	5·107	C(6)	1·71	2·45
				C(8)	−1·75	
				C(7′)	1·73	
				C(6′)	−1·71	
				C(8′)	1·75	
				C(7)	−1·73	
7·522	−2·731	10·207	5·103	C(6)	3·04	3·14
				C(8)	−3·09	
				C(7′)	−0·93	
				C(6′)	−3·04	
				C(8′)	3·09	
				C(7)	0·93	
				O(1)	−2·24	
				O(1′)	2·24	
−7·382	2·881	−10·185	−5·144	C(6)	−0·02	0·06
				C(7)	0·05	
				O(1)	−0·02	
				C(8′)	−0·02	

could not be accomplished, two cycles of refinement with the eight hydrogen atoms in fixed positions were done, with the use of anisotropic thermal parameters for the heavy atoms. A difference Fourier map now yielded all ten hydrogen atoms. These were refined *via* a difference Fourier map following another least-squares cycle of refinement on the heavy atoms. This was followed by two additional heavy atom refinement cycles and then two least-squares cycles on the hydrogen atoms, including isotropic thermal parameters, with heavy atoms fixed.

In the course of the refinement twenty-two reflection intensity errors, mostly caused by indexing mistakes, were corrected. In addition, the observed 022 reflection intensity was found to be grossly in error. The error in intensity estimation was caused by severe distortion of the diffraction spot on the *h*2*l* layer Weissenberg photographs from which the intensity was measured. The 006 reflection intensity also was erroneous but the reason for this was not apparent. Neither of these reflections were included in the final stages of refinement nor are they included in the *R* index computation results given below.

The final *R* index is 0·103 for reflections observed with non-zero intensities. Including all of the observed data it is 0·160. Final atomic parameters and errors are listed in Table 4. The observed and calculated structure factors are listed in Table 9.

Discussion

Final intramolecular bond distances and angles, with standard errors, are listed in Table 5. These were calculated on the IBM 7090 computer using the program ORXFE of Busing & Levy (1959*b*). The closest intermolecular distance, 3·064 Å, is between O(3) and H(7) where H(7) belongs to a molecule related to the basic one by the transformation

$$x \rightarrow x$$
$$y \rightarrow y-\tfrac{1}{2}$$
$$z \rightarrow \tfrac{1}{2}-z\,.$$

The closest intermolecular distance between heavy atoms is 3·851 Å between C(5) and C(4) where C(4) is transformed by

$$x \rightarrow x-\tfrac{1}{2}$$
$$y \rightarrow \tfrac{1}{2}-y$$
$$z \rightarrow z\,.$$

The long C(2)–H(8) distance, 1·31 Å, is just barely greater than the expected distance plus three times the standard error. It is reminiscent of a similar difficulty found by Donohue & Marsh (1962) in the refinement of *N*-acetylglycine, where the peculiar hydrogen position was found to be sensitive to several sets of relatively inaccurately measured structure factors which in turn were sensitive to the locations of hydrogen atoms which were bonded to a freely rotating methyl group. The exact nature of the difficulty has not been found in this case but most likely could be traced to a similar cause.

This structure may be compared with the work of Ferguson, Fritchie, Robertson & Sim (1961) and Brueckner, Hamor, Robertson & Sim (1962) on substituted camphanes. The camphanes have bridged ring systems identical to that formed by C(1), C(2), C(3), C(4), C(5), C(6), and C(8) of this structure. The camphane structures were not refined extensively; however, the work of Ferguson, *et al.* on (+)-10-bromo-2-chloro-2-nitrosocamphane is of particular interest because there are two molecules in the asymmetric unit and average bond distances can be calculated. These agree only fairly well with the distances reported herein but the overall average carbon–carbon bonded distances for the two camphane molecules agrees exactly with the average taken over the analoggous part of the structure reported here, *viz.* 1·56 Å.

Least-squares planes for several sets of atoms in the molecule were calculated on the IBM 7090 (Norment, 1963*a*) by the method of Shomaker, Waser, Marsh & Bergman (1959). These are given in Table 6. The six membered central ring is very nearly planar, and the carbonyl oxygen atoms very nearly lie in this same plane. Table 7 lists angles between various least-squares planes and lines in the molecule.

Table 7. *Angles between least-squares planes and lines which are defined by the atoms as indicated*

Plane or line	Plane or line	Angle
C(1)C(2)C(4)C(5)	C(2)C(3)C(4)	125° 8′
C(1)C(2)C(4)C(5)	C(2)C(6)C(8)C(4)	112 33
C(1)C(2)C(4)C(5)	C(6)C(8)C(7′)C(6′)C(8′)C(7)	58 15
C(2)C(3)C(4)	C(2)C(6)C(8)C(4)	122 19
C(2)C(3)C(4)	C(6)C(8)C(7′)C(6′)C(8′)C(7)	175 53
C(2)C(6)C(8)C(4)	C(6)C(8)C(7′)C(6′)C(8′)C(7)	125 40
C(7)C(7′)	C(6)C(8′)	2 10
C(7)C(7′)	C(2)C(4)	3 45
C(7)C(7′)	C(1)C(5)	4 46
C(7)C(7′)	O(1)C(7)	1 53
C(1)C(2)C(4)C(5)	C(7)C(7′)	0 40
C(2)C(3)C(4)	C(7)C(7′)	2 53
C(2)C(6)C(8)C(4)	C(7)C(7′)	3 3
C(8)C(6′)	C(7)C(7′)	89 37
C(8)C(6′)	C(6)C(8)	90 34
C(8)C(6′)	C(2)C(4)	91 50
C(8)C(6′)	C(1)C(5)	93 14
C(8)C(6′)	O(1)C(7)	0 2
C(8)C(6′)	C(1)C(2)C(4)C(5)	121 45
C(8)C(6′)	C(2)C(3)C(4)	176 33
C(8)C(6′)	C(2)C(6)C(8)C(4)	125 47

Table 8 gives the r.m.s. atomic vibrations for the heavy atoms in the directions of the individual vibration ellipsoid axes (Busing & Levy, 1959*b*). It also lists the angles between the ellipsoid axes and an orthogonal set of vectors, $\overrightarrow{\text{axis 1}}$, $\overrightarrow{\text{axis 2}}$, and $\overrightarrow{\text{axis 3}}$. These are defined as:

$$\overrightarrow{\text{axis 1}} = \overrightarrow{[\text{C}(7),\ \text{C}(7')]}$$

$$\overrightarrow{\text{axis 2}} = \overrightarrow{\text{axis 1}} \times \overrightarrow{[\text{C}(8),\ \text{C}(6')]}$$

$$\overrightarrow{\text{axis 3}} = \overrightarrow{\text{axis 1}} \times \overrightarrow{\text{axis 2}}\,.$$

Table 8. *R.M.S. thermal vibrations in the directions of the individual atomic ellipsoid axes and the angles between the major ellipsoid axes and axis* 1, *axis* 2, *and axis* 3 (*see text*)

Atom		R.M.S. thermal vibration along the ellipsoid axes 1. Minor 2. Mean 3. Major	Angle between major ellipsoid axis and: 1. Axis 1 2. Axis 2 3. Axis 3	Angle between mean ellipsoid axis and: 1. Axis 1 2. Axis 2 3. Axis 3	Angle between minor ellipsoid axis and: 1. Axis 1 2. Axis 2 3. Axis 3
C(1)	1	0·14±0·01	33± 3	121± 3	79± 4
	2	0·20±0·01	117± 2	118± 7	41± 6
	3	0·37±0·03	107± 2	135± 8	129± 8
C(2)	1	0·18±0·01	31±11	121±11	92± 6
	2	0·23±0·01	117±10	141±10	65± 9
	3	0·28±0·03	105± 6	110±10	155± 8
C(3)	1	0·17±0·01	25± 2	112± 4	101± 7
	2	0·20±0·01	113± 2	157± 2	90±16
	3	0·43±0·03	100± 2	86±18	169± 8
C(4)	1	0·16±0·01	33± 2	120± 7	102±14
	2	0·18±0·01	116± 2	147±16	72±25
	3	0·37±0·03	110± 2	100±26	158±13
C(5)	1	0·15±0·01	23± 4	112± 4	85± 4
	2	0·21±0·01	109± 3	127± 8	44± 7
	3	0·38±0·03	103± 3	134± 8	133± 8
C(6)	1	0·18±0·01	21± 5	111± 5	90± 8
	2	0·20±0·01	107± 4	140±17	55±18
	3	0·32±0·03	102± 3	123±19	145±18
C(7)	1	0·12±0·01	32± 3	62± 3	76± 4
	2	0·20±0·01	114± 2	73± 6	30± 4
	3	0·36±0·03	109± 3	34± 6	116± 6
C(8)	1	0·14±0·01	34± 2	95±10	56± 3
	2	0·17±0·01	117± 2	132±14	54±15
	3	0·35±0·03	109± 2	42±16	54±16
O(1)	1	0·20±0·01	45± 4	56± 8	64± 9
	2	0·23±0·01	125± 3	85±12	35± 3
	3	0·37±0·02	115± 3	34± 9	112±12

Note that the angles between the major ellipsoid axes and $\overrightarrow{\text{axis 1}}$, $\overrightarrow{\text{axis 2}}$, and $\overrightarrow{\text{axis 3}}$ are strikingly similar for all nine atoms. The average angles for the eight carbon atoms are 29°, 114°, and 106°. The vector defined by these angles makes an angle of 1° 13′ with the crystallographic *b* axis. Therefore it is apparent that the predominant thermal motion and/or disorder is a rigid-body molecular displacement parallel to the crystallographic *b* axis.

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References

Berghuis, J., Haanappel, IJ. M., Potters, M., Loopstra, B. O., MacGillavry, C. H. & Veenendaal, A. L. (1955). *Acta Cryst.* **8**, 478.

Brueckner, D. A., Hamor, T. A., Robertson, J. M. & Sim, G. A. (1962). *J. Chem. Soc.* p. 799.

Busing, W. R. & Levy, H. A. (1959*a*). *A Crystallographic Least Squares Refinement Program for the IBM* 704. Oak Ridge Natl. Lab. Central File No. 59-4-37.

Busing, W. R. & Levy, H. A. (1959*b*). *A Crystallographic Function and Error Program for the IBM* 704. Oak Ridge Natl. Lab. Central File No. 59-12-3.

Cochran, W. & Woolfson, M. M. (1955). *Acta Cryst.* **8**, 1.

Donohue, J. & Marsh, R. E. (1962). *Acta Cryst.* **15**, 1941.

Ferguson, G., Fritchie, C. J., Robertson, J. M. & Sim, G. A. (1961). *J. Chem. Soc.* p. 1976.

Hauptman, H. & Karle, J. (1953). *Solution of the Phase Problem. I. The Centrosymmetric Crystal.* ACA Monograph No. 3. Brooklyn, New York: Polycrystal Book Service.

Karle, I. L., Hauptman, H., Karle, J. & Wing, A. B. (1958). *Acta Cryst.* **11**, 257.

Karle, I. L. & Karle, J. (1963). *Acta Cryst.* **16**, 969.

McWeeny, R. (1951). *Acta Cryst.* **4**, 513.

Norment, H. G. (1962). *An X-Ray Diffraction Data Reduction Program for the IBM* 704 *and* 7090. U.S. Naval Research Laboratory Report 5739.

Norment, H. G. (1963*a*), *A Collection of FORTRAN Programs for Crystal Structure Analysis,* U.S. Naval Research Laboratory Report 5885.

Norment, H. G. (1963*b*). *Encyclopedia of X- and Gamma Radiation.* G.L.Clark, Editor. p.77. New York: Reinhold.

Shomaker, V., Waser, J., Marsh, R. E. & Bergman, G. (1959). *Acta Cryst.* **12**, 600.

DeVries, L., Heck, R., Piccolini, R. & Winstein, S. (1959). *Chem. & Ind.* p. 1416.

Table 9. *Final observed and calculated structure factors*

h	k	l	kF_o	F_c
0	0	4	59.53	-61.15
0	0	8	49.01	52.04
0	0	10	41.70	-38.22
0	0	12	8.83	8.28
0	0	14	73.02	72.46
0	0	16	51.91	51.55
0	0	18	-0.	-4.39
0	0	20	22.51	20.21
0	0	22	33.80	30.52
0	1	2	4.61	1.98
0	1	4	20.89	-20.72
0	1	6	36.53	-40.04
0	1	8	8.85	-7.26
0	1	10	13.84	10.48
0	1	12	11.55	-10.11
0	1	14	8.22	-6.57
0	1	16	-0.	4.04
0	1	16	-0.	4.04
0	1	18	7.51	-6.35
0	1	20	11.46	-11.44
0	1	22	6.09	-5.50
0	2	4	4.29	-4.17
0	2	6	22.16	-22.14
0	2	8	39.83	-41.44
0	2	10	11.50	-9.00
0	2	12	2.91	-2.32
0	2	14	18.27	-17.32
0	2	16	4.57	-4.23
0	2	18	-0.	-0.37
0	2	20	4.51	-2.78
0	2	22	5.82	-5.81
0	3	2	16.13	-15.54
0	3	4	26.11	27.05
0	3	6	7.73	6.26
0	3	8	3.95	-3.42
0	3	10	-0.	1.13
0	3	12	8.35	-8.77
0	3	14	3.77	-5.22
0	3	16	-0.	-2.39
0	3	18	-0.	-0.71
0	3	20	5.84	5.49
1	1	1	91.83	-94.52
1	1	2	-0.	0.20
1	1	3	72.24	80.40
1	1	4	23.23	24.67
1	1	5	19.47	19.75
1	1	6	37.55	42.68
1	1	7	-0.	1.75
1	1	8	5.16	-2.26
1	1	9	40.30	43.03
1	1	10	17.21	-14.90
1	1	11	31.75	31.48
1	1	12	8.88	8.06
1	1	13	-0.	-1.67
1	1	14	7.34	-5.98
1	1	15	23.94	-22.18
1	1	16	10.24	-9.32
1	1	17	5.43	-4.09
1	1	18	11.92	10.65
1	1	19	8.72	6.54
1	1	20	8.04	8.24
1	1	21	12.45	-11.92
1	1	22	-0.	0.16
1	1	23	-0.	1.31
1	2	0	11.78	11.38
1	2	1	11.82	11.31
1	2	2	13.98	12.92
1	2	3	7.42	-5.40
1	2	4	29.68	26.70
1	2	5	6.90	6.99
1	2	6	20.71	21.97
1	2	7	11.20	11.71
1	2	8	8.26	7.68
1	2	9	18.99	-18.71
1	2	10	16.78	15.68
1	2	11	8.78	-9.28
1	2	12	12.59	12.03
1	2	13	4.32	3.19
1	2	14	-0.	-1.19
1	2	15	7.76	-7.97
1	2	16	4.57	2.55
1	2	17	3.18	-3.68
1	2	18	3.05	3.34
1	2	19	3.49	2.38
1	2	20	-0.	-0.73
1	2	21	-0.	1.20
1	2	22	-0.	0.11
1	3	1	23.08	21.03
1	3	2	6.26	-5.73
1	3	3	15.00	-15.54
1	3	4	3.03	2.71
1	3	5	20.49	-19.67
1	3	6	6.06	5.45
1	3	7	16.95	15.57
1	3	8	4.00	-3.74
1	3	9	-0.	-2.24
1	3	10	6.09	-5.53
1	3	11	20.19	-20.49
1	3	12	-0.	0.21
1	3	13	5.00	5.71
1	3	14	2.67	-2.78
1	3	15	13.98	14.92
1	3	16	3.71	-4.36
1	3	17	2.52	-1.69
1	3	18	-0.	0.82
1	3	19	2.64	-2.85
1	3	20	2.31	0.61
1	3	21	4.86	4.90
2	0	0	163.59	175.25
2	0	1	-0.	-0.30
2	0	2	28.35	23.85
2	0	3	10.59	-4.68
2	0	4	39.02	-37.99
2	0	5	61.17	-66.17
2	0	6	33.56	31.89
2	0	7	32.72	-33.02
2	0	8	13.12	9.10
2	0	9	48.36	49.32
2	0	10	33.95	-31.25
2	0	11	21.65	18.11
2	0	12	9.08	-7.35
2	0	13	-0.	1.33
2	0	14	31.23	27.64
2	0	15	34.99	37.22
2	0	16	25.07	25.86
2	0	17	7.40	8.03
2	0	18	-0.	-2.58
2	0	19	17.30	-16.65
2	0	20	-0.	4.95
2	0	21	-0.	-0.21
2	0	22	14.73	12.60
2	0	23	-0.	4.47
2	1	1	58.65	58.48
2	1	2	14.23	-12.90
2	1	3	57.17	56.68
2	1	4	20.82	-20.81
2	1	5	37.60	40.26
2	1	6	23.60	-20.81
2	1	7	46.21	52.29
2	1	8	6.57	-3.96
2	1	9	34.96	34.79
2	1	10	12.78	9.47
2	1	11	15.71	16.67
2	1	12	4.82	3.77
2	1	13	25.60	22.16
2	1	14	-0.	2.00
2	1	15	17.48	17.08
2	1	16	-0.	2.54
2	1	17	9.42	10.57
2	1	18	-0.	-4.31
2	1	19	11.40	10.60
2	1	20	7.86	-6.87
2	1	21	9.20	7.28
2	1	22	4.04	-4.13
2	1	23	-0.	5.92
2	2	0	47.57	-46.18
2	2	1	17.79	-14.95
2	2	2	18.71	-16.94
2	2	3	7.49	-6.25
2	2	4	23.90	21.09
2	2	5	14.97	-14.99
2	2	6	12.18	-13.26
2	2	7	12.98	-10.83
2	2	8	22.59	-22.90
2	2	9	11.57	11.96
2	2	10	9.62	9.72
2	2	11	-0.	1.58
2	2	12	5.68	3.84
2	2	13	4.37	0.76
2	2	14	17.61	-19.22
2	2	15	10.38	9.17
2	2	16	5.58	-3.62
2	2	17	5.45	-4.58
2	2	18	4.25	4.07
2	2	19	6.98	-5.92
2	2	20	5.33	-4.80
2	2	21	-0.	-0.54
2	2	22	5.31	-5.76
2	3	1	27.51	26.13
2	3	2	-0.	-0.18
2	3	3	6.41	5.89
2	3	4	7.74	6.94
2	3	5	10.22	-8.30
2	3	6	3.42	-2.15
2	3	7	4.92	4.77
2	3	8	2.17	0.28
2	3	9	-0.	-0.86
2	3	10	4.23	3.80
2	3	11	13.53	-13.23
2	3	12	3.66	-2.50
2	3	13	4.68	5.08
2	3	14	-0.	0.28
2	3	15	7.93	9.10
2	3	16	-0.	2.19
2	3	17	4.31	-3.05
2	3	18	-0.	-0.65
2	3	19	-0.	0.90
2	3	20	-0.	1.66
3	1	1	21.77	-15.77
3	1	2	39.13	39.55
3	1	3	36.40	-33.48
3	1	4	15.72	13.54
3	1	5	23.23	-21.55
3	1	6	40.75	39.62
3	1	7	9.34	-4.78
3	1	8	-0.	2.22
3	1	9	14.84	-12.86
3	1	10	20.09	-17.84
3	1	11	11.66	-13.21
3	1	12	8.65	4.21
3	1	13	5.18	-2.29
3	1	14	14.24	-12.67
3	1	15	7.69	-5.20
3	1	16	12.26	-8.08
3	1	17	10.06	-10.35
3	1	18	15.24	14.13
3	1	19	5.89	-1.60
3	1	20	7.53	5.51
3	1	21	4.63	1.30
3	1	22	-0.	3.79
3	2	0	-0.	5.59
3	2	1	8.06	7.60
3	2	2	2.69	2.27
3	2	3	20.33	19.11
3	2	4	20.12	19.29
3	2	5	20.07	19.40
3	2	6	10.61	9.18
3	2	7	10.73	9.61
3	2	8	10.90	-8.54
3	2	9	-0.	1.46
3	2	10	12.38	12.75
3	2	11	4.15	-4.65
3	2	12	9.80	10.77
3	2	13	6.02	-5.81
3	2	14	8.07	-8.66
3	2	15	-0.	-1.96
3	2	16	-0.	-0.37
3	2	17	4.38	4.55
3	2	18	3.58	2.67
3	2	19	5.71	6.04
3	2	20	-0.	-2.95
3	2	21	4.69	4.98
3	2	22	2.43	-2.47
3	3	1	46.38	44.41
3	3	2	6.06	5.12
3	3	3	-0.	-0.89
3	3	4	8.27	-7.94
3	3	5	-0.	-0.21
3	3	6	-0.	-0.35
3	3	7	18.89	19.19
3	3	8	-0.	0.45
3	3	9	-0.	-1.59
3	3	10	3.50	-3.28
3	3	11	11.92	-11.17
3	3	12	2.62	0.51
3	3	13	12.04	12.61
3	3	14	-0.	-1.24
3	3	15	15.92	17.45
3	3	16	-0.	-0.94
3	3	17	-0.	1.02
3	3	18	-0.	-0.13
3	3	19	-0.	1.45
3	3	20	-0.	-2.66
4	0	0	87.58	-81.11
4	0	1	40.47	-36.41
4	0	2	40.62	-37.28
4	0	3	25.03	-20.57
4	0	4	23.86	-22.89
4	0	5	26.65	-20.51
4	0	6	48.76	-50.15
4	0	7	23.81	-22.26
4	0	8	28.66	-26.07
4	0	9	25.71	17.93
4	0	10	13.73	-12.13
4	0	11	12.79	12.59
4	0	12	18.24	-15.84
4	0	13	10.10	8.26
4	0	14	15.10	-17.47
4	0	15	19.61	19.97
4	0	16	10.45	-9.68
4	0	17	-0.	-3.63
4	0	18	-0.	-2.72
4	0	19	7.80	-6.63
4	0	20	-0.	-8.96
4	0	21	-0.	0.50
4	0	22	-0.	-9.07
4	1	1	15.00	14.35
4	1	2	42.88	-40.34
4	1	3	10.51	7.57
4	1	4	10.88	-8.12
4	1	5	9.60	7.26
4	1	6	11.62	8.21
4	1	7	31.58	30.78
4	1	8	7.50	-5.71
4	1	9	20.87	19.19
4	1	10	-0.	1.43
4	1	11	-0.	1.50
4	1	12	21.16	20.72
4	1	13	9.24	9.78
4	1	14	12.78	12.41
4	1	15	9.50	9.45
4	1	17	-0.	2.34
4	1	18	-0.	-1.81
4	1	19	-0.	3.60
4	1	20	-0.	1.86
4	1	21	5.93	5.13
4	1	22	3.01	-3.44
4	2	0	23.04	-22.29
4	2	1	33.17	-33.31
4	2	2	-0.	-1.53
4	2	3	7.12	-6.67
4	2	4	17.59	16.77
4	2	5	20.27	19.42
4	2	6	-0.	1.01
4	2	7	8.97	-7.33
4	2	8	9.63	8.39
4	2	9	6.78	-7.35
4	2	10	18.33	19.79
4	2	11	-0.	2.80
4	2	12	5.99	5.79
4	2	13	5.69	5.63
4	2	14	7.79	-8.00
4	2	15	-0.	-1.78
4	2	16	4.44	-3.33
4	2	17	8.11	-8.66
4	2	18	3.43	1.98
4	2	19	4.39	4.21
4	2	20	-0.	-2.58
4	2	21	-0.	0.98
4	3	1	3.71	-0.73
4	3	2	3.76	4.70
4	3	3	2.86	-3.39
4	3	4	11.64	-11.59
4	3	5	13.08	-12.52
4	3	6	7.68	-7.63
4	3	7	-0.	-1.90
4	3	8	4.24	4.49
4	3	9	4.72	-5.26
4	3	10	4.87	4.76
4	3	11	12.78	-14.34
4	3	12	4.71	4.09
4	3	13	-0.	-0.77
4	3	14	5.05	5.48
4	3	15	2.59	1.83
4	3	16	5.36	5.53
4	3	17	5.39	-4.94
4	3	18	-0.	0.41
4	3	19	-0.	-1.12
4	3	20	2.57	-3.01
5	1	1	10.37	9.12
5	1	2	35.13	35.21
5	1	3	20.33	-19.05
5	1	4	13.45	-11.42
5	1	5	4.15	-3.84
5	1	6	-0.	-3.55
5	1	7	7.10	-5.89
5	1	8	9.91	10.41
5	1	9	19.85	-18.09
5	1	10	7.08	-4.74
5	1	11	9.01	-7.72
5	1	12	12.54	-12.18
5	1	13	5.42	-2.68
5	1	14	10.25	-9.33
5	1	15	7.65	4.79
5	1	16	-0.	3.60
5	1	17	-0.	4.08
5	1	18	-0.	2.20
5	1	19	-0.	-1.45
5	1	20	7.80	-6.92
5	1	21	-0.	3.75
5	2	0	21.77	-20.28
5	2	1	19.65	18.07
5	2	2	19.69	-17.70
5	2	3	26.24	27.10
5	2	4	9.05	-8.95
5	2	5	4.98	3.23
5	2	6	22.00	-23.79
5	2	7	3.93	1.67
5	2	8	32.91	-33.55
5	2	9	16.16	15.28
5	2	10	10.17	-11.23
5	2	11	6.02	-5.46
5	2	12	4.53	-4.54
5	2	13	15.90	-17.50
5	2	14	11.49	-13.18
5	2	15	3.15	2.56
5	2	16	4.28	-4.10
5	2	17	8.77	8.44
5	2	18	-0.	1.16
5	2	19	4.00	1.05
5	2	20	-0.	-3.33
5	2	21	1.57	3.60
5	3	1	10.34	9.71
5	3	2	9.71	7.84
5	3	3	3.94	3.21
5	3	4	13.36	-14.27
5	3	5	-0.	2.33
5	3	6	7.49	-8.81
5	3	7	10.20	-9.47
5	3	8	8.70	8.57
5	3	9	11.13	-10.81
5	3	10	6.59	6.52
5	3	11	-0.	-0.16
5	3	12	-0.	0.73
5	3	13	-0.	-0.23
5	3	14	4.94	5.39
5	3	15	-0.	1.39
5	3	16	7.17	7.66
5	3	17	4.66	4.61
5	3	18	-0.	-2.13
5	3	19	1.96	2.16
5	3	20	3.38	-5.10
6	0	0	56.14	-49.02
6	0	1	18.10	-14.54
6	0	2	11.03	-6.19
6	0	3	11.59	-7.50
6	0	4	-0.	0.78
6	0	5	-0.	-1.15
6	0	6	19.99	-19.39
6	0	7	9.12	-6.47
6	0	8	-0.	0.60
6	0	9	-0.	-2.36
6	0	10	-0.	-1.79
6	0	11	-0.	2.76
6	0	12	-0.	-2.54
6	0	13	-0.	4.20
6	0	14	-0.	-5.92
6	0	15	-0.	0.07
6	0	16	14.31	-17.30
6	0	17	-0.	-6.51
6	0	18	-0.	-3.10
6	0	19	-0.	0.79
6	1	1	10.08	-8.66
6	1	2	38.02	-35.15
6	1	3	25.53	-25.72
6	1	4	-0.	-1.27
6	1	5	13.57	-12.94
6	1	6	16.16	17.03
6	1	7	14.89	14.94
6	1	8	12.27	-12.59
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6	1	10	9.88	-10.10
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6	1	12	18.70	22.15
6	1	13	5.45	-1.58
6	1	14	10.84	11.06
6	1	15	-0.	1.46
6	1	16	11.57	-11.78
6	1	17	10.02	-9.23
6	1	18	-0.	-1.27
6	1	19	8.63	-6.73
6	1	20	5.30	6.15
6	2	0	7.34	6.15
6	2	1	11.53	-9.39
6	2	2	-0.	0.86
6	2	3	4.86	4.47
6	2	4	6.71	-7.04
6	2	5	16.75	18.11
6	2	6	12.53	13.31
6	2	7	-0.	-1.26
6	2	8	24.05	26.66
6	2	9	11.16	-11.89
6	2	10	-0.	0.54
6	2	11	-0.	-0.83
6	2	12	-0.	-0.09
6	2	13	-0.	-1.24
6	2	14	10.08	12.50
6	2	15	7.28	-8.23
6	2	16	4.93	-3.27
6	2	17	4.54	-3.78
6	2	18	5.48	-6.23
6	2	19	6.35	6.02
6	2	20	3.40	4.73
6	3	1	13.86	-14.77
6	3	2	-0.	-0.65
6	3	3	10.34	-9.07
6	3	4	11.08	-11.19
6	3	5	8.86	-9.00
6	3	6	4.50	-3.17
6	3	7	2.59	-3.71
6	3	8	-0.	-0.40
6	3	9	-0.	-1.91
6	3	10	-0.	-0.58
6	3	11	6.12	-4.15
6	3	12	7.83	6.54
6	3	13	3.64	-3.73
6	3	14	-0.	2.72
6	3	15	5.05	-5.46
6	3	16	-0.	-0.07
6	3	17	4.49	-5.02
6	3	18	-0.	0.75
6	3	19	2.09	-2.55
7	1	1	21.90	21.28
7	1	2	6.76	-4.94
7	1	3	8.40	6.16
7	1	4	6.93	-5.49
7	1	5	18.36	16.63
7	1	6	-0.	-4.26
7	1	7	-0.	1.04
7	1	8	-0.	-3.30
7	1	9	7.59	2.49
7	1	10	-0.	-0.75
7	1	11	13.44	13.61
7	1	12	-0.	0.40
7	1	13	-0.	-1.52
7	1	14	-0.	1.29
7	1	15	10.51	6.55
7	1	16	-0.	-0.51
7	1	17	12.73	12.71
7	1	18	-0.	-2.72
7	1	19	-0.	-0.87
7	2	0	2.93	-3.19
7	2	1	12.64	11.41
7	2	2	5.24	4.38
7	2	3	16.62	15.32
7	2	4	5.76	5.37
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7	2	11	6.56	-7.14
7	2	12	-0.	-0.78
7	2	13	14.65	-18.81
7	2	14	5.14	-4.38
7	2	15	3.42	3.46
7	2	16	5.61	4.00
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7	2	19	4.02	-5.46
7	3	1	14.59	-14.91
7	3	2	6.16	-6.70
7	3	3	-0.	-1.28
7	3	4	8.16	-8.50
7	3	5	5.01	-5.10
7	3	6	5.05	-6.35
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7	3	13	10.48	-10.96
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7	3	16	3.25	3.13
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7	3	18	2.02	-2.29
8	0	0	21.79	-16.45
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8	0	4	13.69	13.16
8	0	5	10.13	-9.55
8	0	6	10.24	-8.96
8	0	7	-0.	7.04
8	0	8	-0.	-1.46
8	0	9	10.50	7.90
8	0	10	-0.	0.07
8	0	11	-0.	-4.11
8	0	12	-0.	2.64
8	0	13	-0.	-4.63
8	0	14	-0.	-1.19
8	0	15	-0.	0.88
8	1	1	5.16	-1.78
8	1	2	15.19	-11.72
8	1	3	20.81	-18.99
8	1	4	-0.	-0.06
8	1	5	-0.	-1.48
8	1	6	12.65	11.89
8	1	7	21.60	19.69
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8	1	9	9.50	6.96
8	1	10	10.21	-9.74
8	1	11	5.36	-6.69
8	1	12	9.84	10.67
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8	1	18	-0.	0.59
8	2	0	9.42	8.39
8	2	1	10.92	11.15
8	2	2	7.91	7.53
8	2	3	10.17	10.80
8	2	4	-0.	-1.25
8	2	5	-0.	0.99
8	2	6	8.09	7.94
8	2	7	5.74	5.89
8	2	8	12.45	14.69
8	2	9	-0.	0.04
8	2	10	4.48	-3.51
8	2	11	7.80	-8.57
8	2	12	-0.	0.92
8	2	13	9.47	-10.58
8	2	14	11.43	12.00
8	2	15	3.01	-3.47
8	2	16	-0.	0.80
8	2	17	-0.	3.13
8	3	1	8.21	-7.18
8	3	2	-0.	0.92
8	3	3	3.78	-2.16
8	3	4	2.68	-3.40
8	3	5	4.73	4.50
8	3	6	2.67	1.74
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8	3	9	3.66	5.19
8	3	10	3.56	-4.46
8	3	11	5.66	6.47
8	3	12	-0.	1.11
8	3	13	-0.	0.77
8	3	14	3.44	-2.91
8	3	15	3.81	-3.64
8	3	16	2.55	-3.76
8	3	17	1.15	-1.82
9	1	1	7.67	5.72
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9	1	3	7.69	-7.52
9	1	4	7.70	6.74
9	1	5	7.72	7.55
9	1	6	5.45	2.57
9	1	7	13.37	13.28
9	1	8	9.37	-8.58
9	1	9	7.50	6.63
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9	1	14	-0.	4.63
9	1	15	-0.	3.67
9	1	16	3.94	-2.87
9	1	17	-0.	-2.12
9	2	0	11.55	12.26
9	2	1	-0.	0.12
9	2	2	18.63	20.12
9	2	3	-0.	-0.23
9	2	4	14.67	15.27
9	2	5	8.40	-8.15
9	2	6	4.53	-3.61
9	2	7	5.63	-6.74
9	2	8	4.42	-2.79
9	2	9	5.42	4.79
9	2	10	6.44	3.71
9	2	11	-0.	0.07
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9	3	6	4.88	-6.33
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9	3	9	-0.	-0.14
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9	3	11	-0.	1.26
9	3	12	4.64	4.68
9	3	13	3.90	-3.23
9	3	14	2.70	2.48
10	0	0	10.46	-6.18
10	0	1	12.24	12.33
10	0	2	7.38	5.29
10	0	3	-0.	0.14
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10	0	5	13.10	-13.31
10	0	6	13.92	-14.22
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10	0	10	-0.	-1.72
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10	1	14	-0.	-0.49
10	1	15	-0.	-4.32
10	2	0	4.45	4.30
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10	2	2	7.95	8.63
10	2	3	3.12	2.19
10	2	4	5.90	6.20
10	2	5	-0.	-0.68
10	2	6	3.67	-3.49
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10	2	8	4.92	-5.01
10	2	9	-0.	-1.82
10	2	10	-0.	0.65
10	2	11	4.20	-3.34
10	2	12	2.72	1.77
10	2	13	-0.	-0.63
10	2	14	-0.	0.37
10	3	1	3.03	2.50
10	3	2	3.02	4.08
10	3	3	5.29	4.42
10	3	4	4.18	4.06
10	3	5	8.92	8.84
10	3	6	4.00	3.67
10	3	7	3.87	3.62
10	3	8	-0.	0.68
10	3	9	3.51	4.39
10	3	10	3.27	-3.49
10	3	11	7.27	9.38
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11	1	1	8.68	-7.29
11	1	2	-0.	-2.28
11	1	3	11.37	-8.14
11	1	4	11.21	10.76
11	1	5	-0.	-3.30
11	1	6	-0.	3.81
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11	1	11	-0.	-3.78
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11	2	9	4.70	-1.68
11	2	10	-0.	0.30
11	3	1	-0.	0.82
11	3	2	-0.	4.11
11	3	3	-0.	0.64
11	3	4	2.50	-2.33
11	3	5	3.42	3.01
11	3	6	7.27	-7.40
11	3	7	7.57	7.90
11	3	8	3.88	2.58
11	3	9	4.41	5.62
12	0	0	7.28	7.11
12	0	1	-0.	1.88
12	0	2	-0.	-1.25
12	0	3	12.28	-8.09
12	0	4	-0.	-2.86
12	0	6	-0.	2.30
12	0	7	-0.	-4.58
12	1	1	9.74	-7.29
12	1	2	8.95	8.02
12	1	3	7.15	-5.02
12	1	4	-0.	2.68
12	1	5	-0.	-2.27
12	1	6	-0.	-1.62
12	1	7	-0.	-5.73
12	1	8	-0.	4.56
12	1	9	-0.	-5.17
12	1	10	-0.	3.26
12	2	0	-0.	1.09
12	2	1	4.01	-3.27
12	2	2	-0.	-1.14
12	2	3	4.87	-5.24
12	2	4	-0.	-1.13
12	2	5	2.56	1.92
12	2	6	3.41	-2.97
12	2	7	-0.	1.75
12	2	8	3.79	-7.07