

The Crystal Structure of *N,N*-Diphenylacetamide

BY W. R. KRIGBAUM, R. -J. ROE*, AND J. D. WOODS†

Department of Chemistry, Duke University, Durham, North Carolina

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The crystal structure of *N,N*-diphenylacetamide was determined by X-ray diffraction using a counter detector. The unit cell is orthorhombic with $a = 11.0103 \text{ \AA}$, $b = 12.0754 \text{ \AA}$, and $c = 17.2944 \text{ \AA}$ and space group *Pbca*. The eight molecules occupy general positions in the unit cell. Coordinates and anisotropic temperature factors were refined by differential Fourier syntheses to $R = 0.059$ for 2136 observed structure amplitudes. The amido group is planar, and the two benzene rings are inclined by 61.75° (ring nearest oxygen) and 77.25° from the amido plane.

Unit cell and space group

Prismatic crystals of high quality were grown by slow evaporation of a saturated n-hexane solution. The unit-cell dimensions were determined from diffractometer measurements by the extrapolation procedure developed independently by Nelson & Riley (1945) and Taylor & Sinclair (1945). The dimensions were:

$$\begin{aligned} a &= 11.0103 \pm 0.0004 \text{ \AA} \\ b &= 12.0754 \pm 0.0004 \\ c &= 17.2944 \pm 0.0002 \end{aligned}$$

We are indebted to Professor John C. Morrow of the Chemistry Department, University of North Carolina, for two density determinations yielding $1.23 \pm 0.02 \text{ g.cm}^{-3}$. The theoretical density, assuming eight molecules in the unit cell, is 1.22 g.cm^{-3} . Extinctions were observed for $hk0$ with h odd, $0kl$ with k odd, and $h0l$ with l odd, which corresponds to the space group *Pbca* (D_{2h}^{10}).

Experimental

All diffraction measurements were performed with a General Electric diffractometer equipped with single-crystal orienter, scintillation counter, and pulse-height analyzer. Corrections for counting loss were determined in the following manner. Klug & Alexander (1954) give a relation between the observed counting rate n and the true counting rate N :

$$n/N = 1 - n\tau \quad (1)$$

where τ is the characteristic time constant of the detector system. If we consider two reflections giving different observed counting rates, n_1 and n_2 , the following approximation can be derived:

$$\frac{n_1}{n_2} \simeq \frac{N_1}{N_2} [1 - (n_1 - n_2)\tau] \quad (2)$$

* Present address: Bell Telephone Laboratories, Inc. Murray Hill, N. J., U.S.A.

† Present address: R. J. Reynolds Tobacco Co., Winston Salem, N. C., U.S.A.

Values of n_1 and n_2 were measured with varying numbers of foils attenuating the diffracted beam, and these data were plotted as (n_1/n_2) versus $(n_1 - n_2)$. The value of τ , 4.13×10^{-6} sec, was determined from the ratio of the initial slope and intercept. A linear behavior was observed up to 8×10^4 counts.sec⁻¹, so calibrated metal foils were used to ensure that the counting rate did not exceed this value during collection of the intensity data.

The initial set of intensity measurements were performed with a crystal having approximate dimensions of $0.6 \times 0.5 \times 0.5$ mm. The strategy proposed by Alexander & Smith (1961) was utilized to expedite the data collection process. Both the peak intensity and integrated intensity (2θ scan) were carefully measured for a number of selected reflections. The ratio of integrated to peak intensity was found to be independent of χ and φ , and to vary in a continuous manner with 2θ . The background level was minimized by the use of a nickel β filter and careful adjustment of the pulse height selector, and under these conditions the background could also be represented satisfactorily as a function of the single variable, 2θ . The actual data collection was accomplished by setting χ , φ , and 2θ at their precalculated values, adjusting the latter two angles to maximize the intensity, and measuring for a period of 20 seconds with stationary crystal and counter. In this manner an intensity measurement required about three minutes, and data for the 2136 reflections having $2\theta \leq 140^\circ$ could be collected in two weeks. During this time the intensity of a reference peak was remeasured every 30 minutes to ascertain that the crystal alignment remained unchanged. A computer program corrected the observed peak intensities for counting loss and background radiation, converted to integrated intensity, and applied the Lorentz-polarization correction.

During the course of the refinement it became apparent that some of the observed structure factors corresponding to low 2θ values were seriously in error owing to primary extinction. A second, smaller crystal was therefore quenched several times in liquid nitrogen to increase its mosaic character, and the intensities of all reflections having $2\theta \leq 40^\circ$ were remeasured using this crystal.

Trial structure

The geometry of the benzene rings, and the planarity of the R-C-N portion of the amide grouping, could be assumed at the outset. The studies of acetamide by

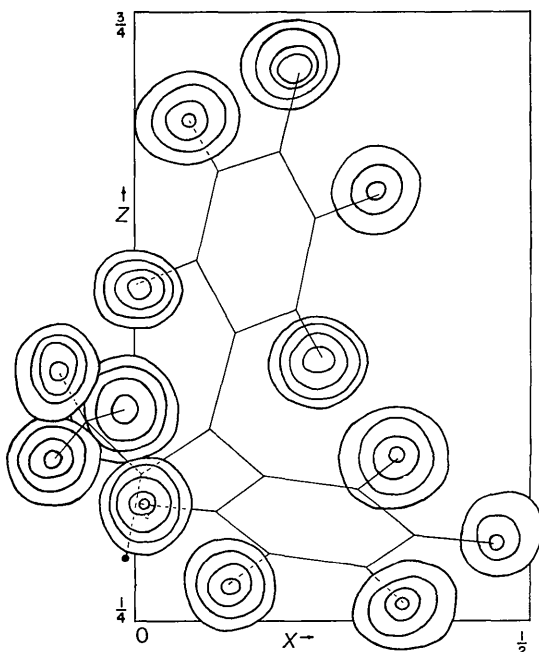


Fig. 1. Difference electron density map showing the hydrogen atoms projected upon the x - z plane. Contours are drawn every $0.1 \text{ e.}\text{\AA}^{-2}$.

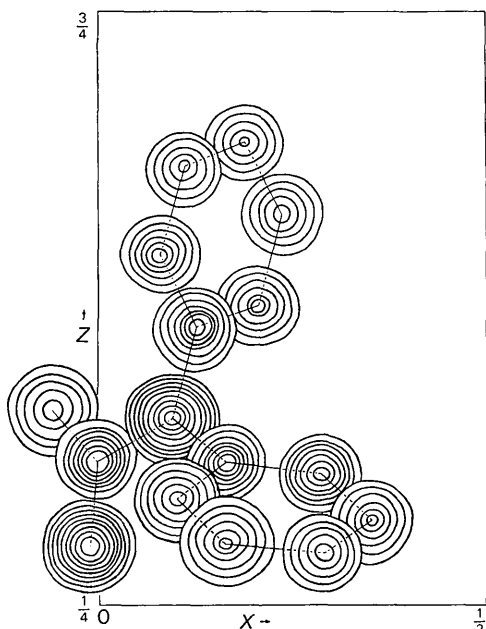
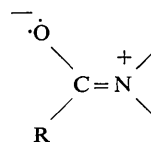


Fig. 2. Composite electron density map of N,N -diphenylacetamide. Contours are drawn every $\text{e.}\text{\AA}^{-3}$, beginning at $2 \text{ e.}\text{\AA}^{-3}$.

Senti & Harker (1940), of formamide by Ladell & Post (1954), and of acetanilide by Brown & Corbridge (1954) and Brown (1966) all indicate considerable contribution from the resonance form:



since in these compounds the C=O bond distance is longer than the usual double bond, while C-N is shorter than a normal single bond. In each of the above monosubstituted amides the bonding about nitrogen is planar. However, the work of Pederson & Pederson (1965) on N -methylacetanilide had not been reported, so that we could not exclude pyramidal bonding about nitrogen for a disubstituted amide. Thus, the position and orientation of the molecule in the unit cell, the orientation of the two benzene rings, and the bonding about nitrogen were the unknown structural parameters.

The three-dimensional Patterson function was first calculated using a grid spacing of $1/50$ of the unit-cell dimensions. This revealed little detail owing to the large number of nearly equivalent atoms present. The resolution could be considerably improved by sharpening about carbon, using an isotropic temperature factor of 2.0. The Harker sections (Harker, 1936), and the corresponding implication diagrams (Buerger, 1959), were constructed; however, these were of little assistance due to the large number of prominent non-Harker peaks. We therefore reexamined the sharpened Patterson maps, and eventually deduced a suitable trial structure giving $R=0.637$ for 606 low order reflections through identification of some 80 Patterson vectors.

Structure refinement

All calculations were performed on the Duke University Computing Laboratory IBM 7072 machine. A three-dimensional Fourier synthesis program written specifically for space group $Pbca$ was used for the initial refinement. During this process, which reduced R to 0.304 for 894 reflections, it was discovered that the tilt of one benzene ring, as well as the positions of the methyl carbon and oxygen, were reversed in the trial structure. The remainder of the refinement was performed with a three-dimensional differential synthesis program written by Dr Shiono, and provided through the kindness of Professor Jeffrey, Crystallography Laboratory, University of Pittsburgh. The coordinates of the ten benzene hydrogen atoms were inserted, assuming a C-H bond distance of 1.07 \AA , and all 2136 observed structure factors were included in the remainder of the refinement. When R reached 0.194 it became evident, through an initial decrease of R with increasing $\sin \theta$, that some of the low order reflections were affected by primary extinction. When the inten-

Table 1. *Observed and calculated structure amplitudes*

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL						
2	4	5	3.5	3.5	1	16	13.2	13.0	1	7	13	12.9	13.1	13.1	2	2	10	19.4	40.9	2	9	5	8.8	8.3	
4	152.3	152.3	152.3	152.3	1	18	34.1	34.0	1	7	14	7.9	7.9	7.9	2	2	11	3.9	3.9	2	9	6	3.8	4.7	
6	80.5	79.6	79.6	79.6	1	20	3.5	2.6	1	7	15	19.2	19.2	19.2	2	2	12	32.2	34.0	2	9	7	7.0	6.9	
8	14.9	13.4	13.4	13.4	1	1	17.1	16.5	1	7	16	5.7	6.3	6.3	2	2	13	15.5	16.0	2	9	8	1.0	12.0	
10	52.9	52.9	52.9	52.9	1	2	77.3	77.3	1	7	17	2.6	1.7	1.7	2	2	14	3.0	3.0	2	9	9	19.0	18.9	
12	27.7	28.6	28.6	28.6	1	1	3	9.5	8.6	1	1	18	6.1	6.1	2	2	15	5.7	6.2	2	9	10	12.6	12.4	
14	32.1	34.4	34.4	34.4	1	1	4	17.0	14.9	1	1	19	9.7	9.3	2	2	16	2.9	4.1	2	9	11	6.0	5.7	
16	12.7	13.1	13.1	13.1	1	1	5	10.6	12.1	1	1	20	2.3	1.5	2	2	17	16.4	16.7	2	9	12	11.6	11.6	
18	26.1	26.8	26.8	26.8	1	1	6	2.8	2.8	1	1	21	4.5	4.5	2	2	18	6.2	6.1	2	9	13	3.3	3.2	
20	7.2	7.0	7.0	7.0	1	1	7	21.3	21.3	1	1	22	1.4	1.4	2	2	19	6.6	6.3	2	9	14	1.1	1.1	
2	182.0	181.6	181.6	181.6	1	1	8	50.0	47.9	1	1	23	10.8	10.8	2	2	20	5.6	5.3	2	9	15	2.4	2.5	
2	1	53.3	52.7	52.7	1	1	9	15.3	14.0	1	1	24	7.6	7.6	2	2	21	127.3	131.9	2	9	16	8	1.4	
2	2	134.2	134.1	134.1	1	1	10	6.2	5.6	1	1	25	8.7	8.7	2	2	22	4.0	4.8	2	10	17	20.1	20.9	
2	3	26.3	23.0	23.0	1	1	11	15.4	15.9	1	1	26	3.5	4.3	2	2	23	2	46.1	45.9	2	10	1	1.2	1.7
2	4	72.7	76.0	76.0	1	1	12	19.5	18.9	1	1	27	10.8	10.8	2	2	24	15.8	16.6	2	10	2	9.0	9.1	
2	5	102.1	110.2	110.2	1	1	13	1.5	2.8	1	1	28	22.4	22.4	2	2	25	19.2	19.2	2	10	3	4.1	4.4	
2	6	11.3	5.6	5.6	1	1	14	10.6	10.8	1	1	29	26.5	27.2	2	2	26	5.9	6.3	2	10	4	9.5	10.0	
2	7	3.2	1.5	1.5	1	1	15	5.8	5.9	1	1	30	6.8	6.6	2	2	27	17.0	16.3	2	10	5	3.4	3.6	
2	8	81.3	86.1	86.1	1	1	16	26.4	26.0	1	1	31	7.9	8.0	2	2	28	32.5	33.2	2	10	6	6.0	5.6	
2	9	24.8	26.0	26.0	1	1	17	2.2	2.2	1	1	32	1.4	1.4	2	2	29	5.0	4.4	2	10	7	2.6	2.7	
2	10	12.7	8.7	8.7	1	1	18	1.5	1.1	1	1	33	1.4	1.6	2	2	30	8.5	7.4	2	10	8	13.9	13.9	
2	11	32.1	32.3	32.3	1	1	19	5.2	5.8	1	1	34	6.3	6.8	2	2	31	31.1	30.6	2	10	9	7.6	7.6	
2	12	20.7	22.9	22.9	1	1	20	2	3.1	2	1	35	2.4	2.4	2	2	32	3.6	2.2	2	10	10	4.9	4.6	
2	13	8.9	8.7	8.7	1	2	1	2.7	2.9	1	1	36	12.1	13.0	2	2	33	2.9	2.8	2	10	11	4.3	3.4	
2	14	8.5	8.7	8.7	1	2	2	65.7	66.1	1	1	37	10.8	10.8	2	2	34	2.2	2.0	2	10	12	5.9	6.1	
2	15	10.0	11.2	11.2	1	1	3	92.2	95.8	1	1	38	19.8	19.8	2	2	35	7.9	7.9	2	10	13	3.1	3.3	
2	16	12.1	11.5	11.5	1	2	4	92.3	97.4	1	1	39	18.1	18.1	2	2	36	10.1	10.9	2	10	15	2	7	7
2	17	9.9	9.5	9.5	1	2	5	33.4	32.2	1	1	40	13.2	12.7	2	2	37	1.8	1.4	2	11	1	18.4	18.2	
2	18	14.3	13.8	13.8	1	2	6	52.3	52.1	1	1	41	5.7	6.4	2	2	38	9.7	9.7	2	11	1	8.0	12.5	
2	19	3.3	2.9	2.9	1	2	7	13.5	13.2	1	1	42	7.3	7.3	2	2	39	5.9	5.9	2	11	2	4.1	4.4	
2	20	1.1	0.9	0.9	1	2	8	12.6	12.4	1	1	43	1.0	1.1	2	2	40	1.7	1.5	2	11	3	6.8	5.7	
4	4	43.3	43.7	43.7	1	2	9	26.1	26.1	1	1	44	9.9	1.0	2	2	41	20	1.7	1.5	2	11	3	6.8	5.7
4	5	26.2	26.9	26.9	1	2	10	2.4	2.4	1	1	45	1.9	1.7	2	2	42	6.8	6.7	2	11	4	1.3	1.5	
4	6	67.9	64.0	64.0	1	2	11	23.6	23.2	1	1	46	2.1	2.1	2	2	43	9.5	9.9	2	11	5	2.1	1.9	
4	7	3.7	1.5	1.5	1	2	12	1.9	2.0	1	1	47	4.8	5.4	2	2	44	24.6	22.2	2	11	6	0.6	0.6	
4	8	30.2	30.4	30.4	1	2	13	12.2	13.1	1	1	48	0.6	0.6	2	2	45	17.7	17.6	2	11	8	4.7	4.4	
4	9	2.5	3.4	3.4	1	2	14	25.0	24.2	1	1	49	6.6	6.7	2	2	46	26.6	26.6	2	11	8	4.7	4.4	
4	10	15.0	15.1	15.1	1	2	15	7.6	7.5	1	1	50	6.0	6.0	2	2	47	46.1	43.8	2	11	10	11.7	11.8	
4	11	56.6	56.1	56.1	1	2	16	1.5	1.3	1	1	51	0.7	1.3	2	2	48	9.6	10.8	2	11	11	5.3	5.2	
4	12	16.4	16.4	16.4	1	2	17	6.4	6.9	1	1	52	1.7	1.9	2	2	49	13.8	13.8	2	11	12	7.8	7.8	
4	13	2.9	2.7	2.7	1	2	18	6.5	6.9	1	1	53	19.4	19.8	2	2	50	16.2	16.5	2	11	13	0.9	1.1	
4	14	41.8	43.4	43.4	1	2	19	5.6	6.1	1	1	54	12.4	13.0	2	2	51	25.8	25.7	2	12	1	17.5	18.1	
4	15	9	9	9	1	2	20	2.0	2.0	1	1	55	4	2.7	4.0	2	2	52	2.0	1.7	2	12	1	8.5	8.5
4	16	30.0	32.0	32.0	1	3	1	24.1	24.1	1	1	56	19.0	19.0	2	2	53	18.3	18.3	2	12	2	3.7	3.7	
4	17	11.0	11.5	11.5	1	3	2	8.6	8.7	1	1	57	16.0	16.6	2	2	54	1.7	1.7	2	12	3	8.2	8.4	
4	18	6.6	6.5	6.5	1	3	3	48.2	48.5	1	1	58	7.0	7.0	2	2	55	27.4	29.4	2	12	4	6.2	6.5	
4	19	20.5	22.0	22.0	1	3	4	85.1	85.1	1	1	59	18.1	18.6	2	2	56	34.1	34.1	2	12	5	4.8	4.8	
4	20	6.8	6.1	6.1	1	3	5	26.7	25.1	1	1	60	6.5	6.5	2	2	57	1.8	1.8	2	12	6	5.8	5.4	
4	21	12.0	12.0	12.0	1	3	6	44.8	45.2	1	1	61	6.2	6.8	2	2	58	11.1	11.4	2	12	7	2.9	3.2	
4	22	16.8	16.8	16.8	1	3	7	11.6	11.6	1	1	62	3.8	3.8	2	2	59	11.8	11.8	2	12	8	7.2	6.3	
4	23	11.8	11.5	11.5	1	3	8	28.9	27.1	1	1	63	6.0	5.8	2	2	60	5.5	5.1	2	12	9	6.3	6.3	
4	24	12.4	12.4	12.4	1	3	9	10.5	8.7	1	1	64	0.5	0.4	2	2	61	9.4	9.4	2	12	10	0.7	1.1	
4	25	1.7	1.7	1.7	1	3	10	5.5	1.4	1	1	65	1.0	1.0	2	2	62	20	25.4	2	12	11	7.9	6.9	
6	1	48.8	47.1	47.1	1	3	11	15.1	15.3	1	1	66	1.1	1.1	2	2	63	4.3	4.5	2	13	1	1.2	1.2	
6	2	3.9	3.0	3.0	1	3	12	29.0	30.6	1	1	67	1.1	1.1	2	2	64	5.1	4.3	2	13	2	6.5	6.6	
6	3	8.2	7.3	7.3	1	3	13	8.7	9.5	1	1	68	1.8	1.8	2	2	65	29.4	28.6	2	13	3	7.9	7.5	
6	4	1.1	1.1	1.1	1	3	14	9	3.1	1	1	69	8.0	7.7	2	2	66	31.8	31.8	2	13	3	5.0	4.3	
6	5	6.7	6.4	6.4	1	3	15	14.1	13.9	1	1	70	2.4	2.4	2	2	67	16.0	16.0	2	13	4	4.8	4.8	
6	6	11.3	10.1	10.1	1	3	16	16.6	16.4	1	1	71	5.1	11.0	2	2	68	20.3	20.4	2	13	5	8.1	8.4	
6	7	13.3	13.3	13.3	1	3	17	8.3	8.8	1	1	72	3.8	4.0	2	2	69	16.8	15.1	2	13	6	5.8	5.5	
6	8	24.1	24.3	24.3	1	3	18	6.5	6.8	1	1	73	2.7	2.7	2	2	70	7.4	7.9	2	13	6	6.8	6.8	
6	9	1.3	1.5	1.5	1	3	19	2.1	2.3	1	1	74	5.2	5.1	2	2	71	9.6	8.9	2	13	8	2.4	2.3	
6	10	15.5	15.5	15.5	1	3	20	2.8	3.2	1	1	75	1.9	1.9	2	2	72	17.5	17.0	2	13	9	1.1	1.7	
6	11	38.2	40.6	40.6	1	3	21	2.2	2.2	1	1	76	1.7	1.7	2	2	73	3.2	3.2	2	14	1	3.2	3.3	
6	12	31.5	33.0	33.0	1	3	22	6.0	6.0	1	1	77	1.3	1.3											

Table 1 (cont.)

H	K	L	F0BS	FCAL	H	K	L	F0BS	FCAL	H	K	L	F0BS	FCAL	H	K	L	F0BS	FCAL
3 3 16	7.2	6.6	-	3 11 11	3.6	3.1	-	4 6 1	32.4	33.6	-	5 1 9	2.4	2.6	5 9 3	9.8	9.2		
3 3 17	4.1	2.3	-	3 11 12	1.0	4.4	-	4 6 2	12.3	12.3	-	5 1 10	18.4	18.6	5 9 4	15.0	15.0	-	-
3 3 18	4.2	4.6	-	3 11 13	5.9	5.9	-	4 6 3	9.6	2.2	-	5 1 11	4.5	4.5	5 9 5	4.9	5.1	-	-
3 3 19	6.1	6.2	-	3 12 1	3.5	3.5	-	4 6 4	4.4	4.4	-	5 1 12	2.7	2.7	5 9 6	7.5	7.5	-	-
3 3 20	2.0	2.8	-	3 12 2	5.3	4.2	-	4 6 5	12.5	11.4	-	5 1 13	6.1	5.9	5 9 7	16.9	16.7	-	-
3 3 4	1	22.2	22.6	3 12 3	5.1	5.2	-	4 6 6	14.4	14.7	-	5 1 14	38.7	40.5	5 9 8	4.5	4.5	-	-
3 3 2	13.5	14.5	7.3	3 12 4	7.3	7.0	-	4 6 7	7.4	15.6	-	5 1 15	4.2	4.3	5 9 9	8.4	9.2	-	-
3 3 4	3	40.3	39.1	3 12 5	4.3	4.0	-	4 6 8	11.7	10.7	-	5 1 16	9.8	9.8	5 9 10	2.0	2.0	-	-
3 3 4	4	38.1	37.1	3 12 6	2.6	2.0	-	4 6 9	18.4	17.7	-	5 1 17	3.0	2.3	5 9 11	3.3	3.5	-	-
3 3 4	6	19.1	13.7	3 12 7	2.6	2.1	-	4 6 10	8.6	8.1	-	5 1 18	4.1	3.7	5 9 12	6.5	6.3	-	-
3 3 4	6	21.6	21.1	3 12 8	3.4	3.6	-	4 6 11	7.0	6.7	-	5 1 19	4.8	4.9	5 9 13	10.1	10.4	-	-
3 3 4	7	16.6	14.6	3 12 9	.5	.4	-	4 6 12	20.0	18.7	-	5 2 1	3.5	3.5	5 9 14	2.4	2.4	-	-
3 3 4	8	2.2	1.2	3 12 10	8.5	8.1	-	4 6 13	6.0	5.8	-	5 2 2	14.2	12.8	5 10 1	7.3	7.8	-	-
3 3 4	9	15.8	16.4	3 12 11	8.5	8.1	-	4 6 14	4.7	4.5	-	5 2 3	15.4	14.2	5 10 2	4.0	4.3	-	-
3 3 10	3.1	3.9	-	3 13 1	5.5	5.6	-	4 6 15	8.9	8.5	-	5 2 4	17.3	17.8	5 10 3	6.9	6.9	-	-
3 3 11	2.0	2.7	-	3 13 2	4.8	4.0	-	4 6 16	4.5	4.7	-	5 2 5	3.3	3.3	5 10 4	2.0	1.9	-	-
3 3 12	7.4	7.7	-	3 13 3	4.1	3.8	-	4 6 17	1.4	2.1	-	5 2 6	17.5	17.1	5 10 5	7.4	7.8	-	-
3 3 13	15.2	16.0	-	3 13 4	2.1	1.9	-	4 6 18	3.0	3.0	-	5 2 7	11.0	11.3	5 10 6	7.5	8.0	-	-
3 3 14	23.3	24.2	5.2	3 13 5	6.1	5.2	-	4 6 19	10.9	12.2	-	5 2 8	6.9	5.7	5 10 7	2.0	2.4	-	-
3 3 15	2.0	2.0	-	3 13 6	1.7	2.4	-	4 7 1	10.0	8.7	-	5 2 9	.2	.1	5 10 8	3.4	3.2	-	-
3 3 16	4.9	5.2	-	3 13 7	2.5	2.3	-	4 7 2	3.0	2.9	-	5 2 10	11.2	11.8	5 10 9	1.6	1.8	-	-
3 3 17	4.6	3.8	-	3 13 8	.9	1.1	-	4 7 3	3.8	3.3	-	5 2 11	.5	.4	5 10 10	6.2	6.6	-	-
3 3 18	4.6	3.5	-	3 14 1	3.5	4.1	-	4 7 4	4.4	4.1	-	5 2 12	6.0	5.9	5 10 11	9.4	10.3	-	-
3 3 19	1.8	1.8	-	3 14 2	1.1	1.1	-	4 7 5	6.6	7.5	-	5 2 13	1.3	1.5	5 10 12	1.1	1.1	-	-
3 3 20	4.9	5.2	-	3 14 3	1.4	1.4	-	4 7 6	.7	.7	-	5 2 14	2.2	1.7	5 10 13	2.3	2.5	-	-
3 3 5	2	24.7	24.0	4	27.1	27.0	-	4 7 7	6.8	4.4	-	5 2 15	7.5	6.8	5 11 1	4.2	4.0	-	-
3 3 5	3	33.	36.4	4	26.7	27.4	-	4 7 8	15.5	14.6	-	5 2 16	2.1	1.7	5 11 2	17.9	18.1	-	-
3 3 5	4	38.4	38.8	4	16.7	15.8	-	4 7 9	10.3	10.3	-	5 2 17	9.0	9.2	5 11 3	6.4	5.4	-	-
3 3 5	5	17.4	17.3	4	6	16.8	-	4 7 10	16.7	5.6	-	5 2 18	1.7	1.4	5 11 4	1.6	1.8	-	-
3 3 5	6	4.6	5.5	4	8	16.5	-	4 7 11	1.2	1.2	-	5 2 19	6.0	5.4	5 11 5	1.3	1.3	-	-
3 3 5	8	3.8	4.3	4	10	5.2	-	4 7 12	1.9	.8	-	5 3 1	54.3	56.3	5 11 6	2.0	2.3	-	-
3 3 5	9	2.2	1.9	4	12	19.8	-	4 7 13	12.3	12.2	-	5 3 2	26.0	28.3	5 11 7	2.1	2.4	-	-
3 3 5	11	6.4	6.3	4	14	24.6	-	4 7 14	1.2	1.2	-	5 3 3	16.7	16.0	5 11 8	4.8	4.5	-	-
3 3 5	12	19.2	19.5	4	16	5.0	-	4 7 15	.4	.9	-	5 3 4	32.5	31.5	5 11 9	1.4	1.4	-	-
3 3 5	14	26.1	26.5	4	18	11.5	-	4 7 16	2.7	2.8	-	5 3 5	7.5	6.6	5 11 10	5.7	5.5	-	-
3 3 5	15	9.1	9.1	4	20	4.9	-	4 7 17	4.8	4.8	-	5 3 6	3.1	3.1	5 11 11	4.7	4.7	-	-
3 3 16	7	4.8	-	4	1	25.1	-	4 8 1	21.4	3.6	-	5 3 7	25.5	26.4	5 12 1	2.7	3.1	-	-
3 3 17	5.1	4.8	-	4	1	2.3	-	4 8 2	2.3	10.5	-	5 3 8	11.1	10.8	5 12 2	6.0	5.4	-	-
3 3 18	1.8	2.0	-	4	1	1.6	-	4 8 3	10.6	10.5	-	5 3 9	11.6	14.8	5 12 3	6.1	6.5	-	-
3 3 19	4.1	4.0	-	4	1	1.6	-	4 8 4	5.0	4.4	-	5 3 10	2.7	3.0	5 12 4	4.2	4.3	-	-
3 3 6	1	4.2	3.2	4	1	5.5	-	4 8 5	10.0	10.0	-	5 3 11	8.8	9.9	5 12 5	1.3	1.3	-	-
3 3 6	2	34.5	32.6	4	1	7.3	-	4 8 6	3.3	4.2	-	5 3 12	7.2	7.2	5 12 6	.8	.1	-	-
3 3 6	3	31.2	29.9	4	1	8	-	4 8 7	19.2	19.7	-	5 3 13	4.7	4.8	5 12 7	3.8	3.7	-	-
3 3 6	4	9	9.4	4	1	10	-	4 8 8	14.4	14.7	-	5 3 14	2.9	3.1	5 12 8	3.7	4.3	-	-
3 3 6	5	8.3	9.4	4	1	10	-	4 8 9	10.3	8.5	-	5 3 15	5.3	5.3	5 12 9	3.5	3.5	-	-
3 3 6	6	9.2	9.3	4	1	11	-	4 8 10	6.2	8.2	-	5 3 16	1.1	.9	5 12 10	1.4	1.6	-	-
3 3 6	7	7.5	7.5	4	1	12	-	4 8 11	.5	.7	-	5 3 17	.5	.7	5 12 11	1.6	1.5	-	-
3 3 6	8	6.0	6.0	4	1	12	-	4 8 12	4.7	4.4	-	5 3 18	1.2	1.2	5 12 12	1.9	1.7	-	-
3 3 6	9	6.1	6.0	4	1	13	-	4 8 13	2.3	2.9	-	5 3 19	3.4	3.4	5 12 13	10.7	10.7	-	-
3 3 6	10	2.1	1.9	4	1	14	-	4 8 14	2.3	2.9	-	5 4 1	2.4	2.6	6	2	63.1	-	-
3 3 6	11	16.0	15.0	4	1	15	-	4 8 15	1.5	1.6	-	5 4 2	22.1	22.8	6	4	2.6	-	-
3 3 6	12	1.9	1.0	4	1	16	-	4 8 16	3.2	3.2	-	5 4 3	18.9	19.1	6	6	51.9	-	-
3 3 6	13	14.9	15.0	4	1	17	-	4 8 17	7.8	14.4	-	5 4 4	2.2	2.0	6	10	8.6	-	-
3 3 6	14	11.8	11.7	4	1	18	-	4 9 1	3.7	4.2	-	5 4 5	15.6	14.1	6	12	32.2	-	-
3 3 6	15	5.5	4.6	4	1	19	-	4 9 2	10.0	9.8	-	5 4 6	15.7	15.7	6	16	14.5	-	-
3 3 6	16	1.1	1.2	4	2	2	-	4 9 3	2.9	3.2	-	5 4 7	2.2	2.0	6	10	8.6	-	-
3 3 6	17	3.3	3.5	4	2	1	-	4 9 4	2.9	3.2	-	5 4 8	13.6	14.1	6	12	32.2	-	-
3 3 7	1	11.5	11.1	4	2	2	-	4 9 5	12.1	12.2	-	5 4 9	8.4	9.7	6	16	23.4	-	-
3 3 7	2	9.8	7.7	4	2	1	-	4 9 6	5.5	5.5	-	5 4 10	8.4	9.7	6	16	23.4	-	-
3 3 7	3	1.5	3.4	4	2	2	-	4 9 7	6.9	8.0	-	5 4 11	3.7	3.1	6	18	12.6	-	-
3 3 7	4	8.3	8.1	4	2	3	-	4 9 8	7.8	7.5	-	5 4 12	3.1	3.1	6	18	12.6	-	-
3 3 7	5	7.2	7.7	4	2	4	-	4 9 9	11.1	11.1	-	5 4 13	2.2	2.8	6	1	16.0	-	-
3 3 7	6	3.5	3.0	4	2	5	-	4 9 10	3.2	4.4	-	5 4 14	2.1	2.9	6	1	2.9	-	-
3 3 7	7	8.5	8.2	4	2	6	-	4 9 11	33.2	33.2	-	5 4 15	5.7	5.8	6	13	14.6	-	-
3 3 7	8	7.9	8.9	4	2	7	-	4 9 12	10.8	10.1	-	5 4 16	1.1	1.3	6	1	4.7	-	-
3 3 7	9	12.9	13.1	4	2	8	-	4 9 13	3.6	3.8	-	5 4 17	4.5	4.5	6	1	5.0	-	-
3 3 7	10	10.5	10.3	4	2	9	-	4 9 14	2.6	2.6	-	5 4 18	1.3	1.3	6	1	17.9	-	-
3 3 7	11	2.2	1.7	4	2	10	-	4 9 15	3.0	2.9	-	5 4 19	2.5	2.5	6	1	10.4	-	-
3 3 7	12	4.0	3.3	4	2	11	-	4 10 1	16.5	16.9	-	5 4 20	7.0	7.6	6	1	7.8	-	-
3 3 7	13	1.5	1.2	4	2	12	-	4 10 2	8.1	3.9	-	5 4 21	7.2	7.0	6	1	8.4	-	-
3 3 7	14	2.2	1.6	4	2	13	-	4 10 3	4.5	4.5	-	5 4 22	4.7	4.8	6	1	11.9	-	-
3 3 7	15	6.4	6.1	4	2	14	-	4 10 4	7.7	8.7	-	5 4 23	1.1	1.1	6	1	2.5	-	-
3 3 7	16	7.0	7.1	4	2	15	-	4 10 5	6.2	6.7	-	5 4 24	6.0	6.9	6	1	2.7	-	-
3 3 7	17	6.0	6.4	4	2	16	-	4 10 6	3.5	3.9	-	5 4 25	9.5	10.1	6	1	2.8	-	-
3 3 7	18	1.1	1.2	4	2	17	-	4 10 7	3.5	3.9	-	5 4 26	9.5	10.1	6	1	2.8	-	-
3 3 7	19	3.3	3.5	4	2	18	-	4 10 8	5.3	5.3	-	5 4 27	15.0	16.0	6	1	1.6	-	-

Table 1 (cont.)

H K L	FOBS	FCAL	H K L	FOBS	FCAL	H K L	FOBS	FCAL	H K L	FOBS	FCAL
6 5 1	30.4	30.6	7 1 11	.5	.-	7 10 6	5.8	5.6	8 7 12	3.4	3.6
6 5 2	7.0	7.0	7 1 12	6.2	6.4	7 10 6	.5	.3	8 7 13	1.2	1.3
6 5 3	25.5	25.5	7 1 13	5.0	5.6	7 10 9	3.0	2.3	8 8 1	1.0	.7
6 5 4	16.8	16.8	7 1 14	16.7	16.9	7 10 9	1.3	1.1	8 8 2	9.2	9.0
6 5 5	4.9	4.7	7 1 15	11.9	11.8	7 11 1	1.3	1.1	8 8 3	.5	.5
6 5 6	9.6	9.2	7 1 16	13.9	13.3	7 11 2	1.9	1.9	8 8 4	6.5	7.1
6 5 7	1.6	1.9	7 1 17	7.4	7.7	7 11 3	1.6	1.4	8 8 5	2.9	3.2
6 5 8	9.0	9.5	7 2 2	8.6	8.4	7 11 4	.3	.3	8 8 6	3.2	3.9
6 5 9	12.6	12.6	7 2 3	1.1	1.0	7 11 5	4.7	4.7	8 8 6	3.2	3.2
6 5 10	18.1	17.8	7 2 4	8.7	8.2	7 11 6	.7	.8	8 8 8	2.9	2.3
6 5 11	5.1	5.8	7 2 5	6.7	5.8	7 11 8	2.5	3.1	8 8 11	5.8	7.0
6 5 12	12.4	12.7	7 2 6	2.1	1.9	7 12 2	1.2	1.2	8 8 12	.5	.3
6 5 13	.8	.9	7 2 7	14.5	15.2	7 12 3	2.7	2.4	8 9 1	2.2	1.9
6 5 14	3.3	.7	7 2 8	14.3	14.5	7 12 4	6.3	6.3	8 9 1	4.4	4.2
6 5 15	3.8	4.0	7 2 9	1.8	.3	7 12 4	1.2	1.2	8 9 2	.4	.1
6 5 16	1.3	1.0	7 2 10	5.7	5.0	7 12 5	.8	.8	8 9 3	9.2	8.9
6 5 17	4.2	3.5	7 2 11	3.4	4.3	8 2	4.8	5.9	8 9 4	5.8	5.4
6 5 18	13.5	12.2	7 2 12	15.0	14.9	8 4	4	1.1	8 9 5	.6	.3
6 5 19	3.0	4.0	7 2 13	1.1	.4	8 6	8.2	9.7	8 9 6	4.7	5.5
6 5 20	22.2	22.2	7 2 14	4.7	4.3	8 8	4.5	4.2	8 9 7	8.8	8.7
6 6 2	4.6	3.0	7 2 15	4.7	3.9	8 10	7.2	7.1	8 9 8	2.0	1.4
6 6 3	2.4	1.2	7 2 16	12.1	12.0	8 12	2.5	2.5	8 9 9	4.5	5.1
6 6 4	16.1	16.5	7 2 17	1.4	1.3	8 14	2.2	1.8	8 9 10	12.5	12.2
6 6 5	11.8	12.7	7 3 1	11.2	11.2	8 16	6.3	6.2	10 10	1.6	2.4
6 6 6	5.3	4.2	7 3 2	21.6	23.6	8 1	29.8	29.1	10 11	1.2	1.5
6 6 7	3.6	4.0	7 3 3	5.0	4.3	8 1	2	2.1	10 12	2.2	2.3
6 6 8	10.8	9.1	7 3 4	17.4	17.7	8 1	1	8.3	10 13	4.3	4.3
6 6 9	7.9	9.1	7 3 5	5.9	22.9	8 1	3	3.2	10 14	4.2	4.4
6 6 10	3.7	3.2	7 3 6	22.7	22.8	8 1	3	3.2	10 14	1.7	1.8
6 6 11	8.9	8.9	7 3 7	25.4	25.5	8 1	6	9.3	10 16	1.8	1.8
6 6 12	1.2	.4	7 3 8	8.4	8.0	8 1	7	4.6	10 17	4.2	4.3
6 6 13	.8	.1	7 3 9	8.2	2.6	8 1	8	19.3	10 18	2.6	3.0
6 6 14	4.9	5.3	7 3 10	2.7	2.6	8 1	9	2.6	10 11	2.3	2.3
6 6 15	6.3	6.0	7 3 11	4.5	4.7	8 1	5	3.1	10 11	4	4.2
6 6 16	.8	.6	7 3 12	8.7	8.9	8 1	10	23.7	10 11	2	3.8
6 6 17	23.0	24.4	7 3 13	18.7	18.9	8 1	11	16.9	10 11	3	5.3
6 6 18	13.6	13.6	7 3 14	4.3	4.4	8 1	12	16.6	10 11	4	5.2
6 6 19	12.4	7.1	7 3 15	2.1	2.0	8 1	14	12.3	10 11	5	5.5
6 6 20	7.9	7.1	7 3 16	5.2	5.0	8 1	15	10.3	10 11	6	1.3
6 7 2	7.5	7.6	7 3 17	2.2	2.2	8 1	16	2.2	10 11	7	3.7
6 7 3	7.1	7.4	7 4 1	9.8	10.1	8 2	2	20.9	9 4	7	7.4
6 7 4	7.5	7.6	7 4 2	14.4	14.5	8 2	1	4.2	9 6	6	7
6 7 5	7.1	7.3	7 4 3	7.2	7.2	8 2	2	4.2	9 8	19.7	19.1
6 7 6	5.0	4.8	7 4 4	7.1	7.6	8 2	4	4.8	9 8	19.7	14.3
6 7 7	8.2	7.3	7 4 5	11.1	11.3	8 2	5	15.7	9 12	1.6	1.6
6 7 8	5.0	4.8	7 4 6	2.3	2.7	8 2	6	3.0	9 14	3.0	3.5
6 7 9	4.6	4.4	7 4 7	1.7	1.4	8 2	7	2.0	9 1	1	4.9
6 7 10	15.5	16.8	7 4 8	6.0	6.1	8 2	9	16.4	9 1	3	4.9
6 7 11	10.7	11.3	7 4 9	6.0	6.1	8 2	9	16.4	9 1	3	1.1
6 7 12	1.5	.3	7 4 10	5.2	4.6	8 2	3	8.4	9 1	4	9.2
6 7 13	8.0	7.3	7 4 11	4.6	5.4	8 2	10	19.4	9 1	5	4.9
6 7 14	2.6	2.3	7 4 12	5.5	4.7	8 2	11	11.3	9 1	6	15.2
6 7 15	1.1	2.4	7 4 13	1.7	2.1	8 2	12	8.3	9 1	7	1.8
6 7 16	10.4	10.0	7 4 14	8.0	7.3	8 2	13	8.3	9 1	8	1.4
6 8 1	7.9	8.0	7 4 15	1.5	1.7	8 2	14	4.2	9 1	8	11.9
6 8 2	7.4	6.1	7 4 16	6.0	6.5	8 2	15	5	9 1	10	8
6 8 3	3.9	4.2	7 4 17	1.6	1.5	8 2	16	4.7	9 1	11	9.0
6 8 4	1.9	2.3	7 5 1	20.4	21.2	8 3	1	.6	9 1	12	7.8
6 8 5	14.1	14.1	7 5 2	10.0	10.5	8 3	1	10.2	9 1	14	7.5
6 8 6	6.4	6.9	7 5 3	8.7	9.5	8 3	4	5.3	9 1	15	1.2
6 8 7	1.2	1.3	7 5 4	15.8	15.5	8 3	4	18.7	9 2	1	12.7
6 8 8	1.9	1.9	7 5 5	17.7	17.4	8 3	6	21.3	9 2	1	5.4
6 8 9	8.3	8.4	7 5 6	3.6	3.4	8 3	7	2.0	9 2	4	6.3
6 8 10	8.3	8.4	7 5 7	4.3	4.3	8 3	8	4.8	9 2	5	5.5
6 8 11	2.9	2.6	7 5 8	7.2	8.4	8 3	9	23.0	9 2	5	12.2
6 8 12	9.8	9.8	7 5 9	1.2	1.3	8 3	2	9.7	9 2	6	1.6
6 8 13	4.8	4.6	7 5 10	1.2	1.3	8 3	2	9.7	9 2	7	8.4
6 8 14	2.9	2.6	7 5 11	3.1	3.1	8 3	10	7.2	9 2	8	15.7
6 9 1	3.8	2.4	7 5 12	5.9	5.9	8 3	11	5.9	9 2	8	6.9
6 9 2	2.8	2.4	7 5 13	1.3	1.6	8 3	12	8.8	9 2	10	14.7
6 9 3	7.2	7.9	7 5 14	4.4	4.7	8 3	13	9.4	9 2	11	5.0
6 9 4	5.9	6.1	7 5 15	.7	1.1	8 3	14	2.2	9 2	12	5.9
6 9 5	5.9	6.1	7 5 16	2.1	1.1	8 3	16	2.2	9 2	13	6.0
6 9 6	3.4	3.1	7 5 17	2.7	1.1	8 3	16	2.2	9 2	14	5.9
6 9 7	5.2	6.2	7 6 1	21.4	21.8	8 4	1	.5	9 2	14	7.7
6 9 8	2.4	2.6	7 6 2	20.5	20.1	8 4	2	22.4	9 2	15	3.8
6 9 9	2.4	2.6	7 6 3	14.2	14.2	8 4	2	22.4	9 2	15	15.7
6 9 10	2.7	2.0	7 6 4	1.8	.9	8 4	2	11.8	9 3	3	9.0
6 9 11	1.8	1.2	7 6 5	17.5	18.4	8 4	3	5.5	9 3	4	14.4
6 9 12	1.5	2.0	7 6 6	3.0	3.0	8 4	4	1.0	9 3	5	3.6
6 9 13	8.2	7.8	7 6 7	7.9	5.2	8 4	5	19.0	9 3	5	3.7
6 9 14	9.9	10.0	7 6 8	5.1	5.5	8 4	6	6.5	9 3	7	21.0
6 9 15	2.6	6.5	7 6 9	1.5	1.5	8 4	7	2.0	9 3	8	9.7
6 9 16	3.9	4.8	7 6 10	4.0	4.7	8 4	7	2.0	9 3	10	1.0
6 9 17	6.8	6.5	7 6 11	7.3	7.3	8 4	9	7.6	9 3	2	10.8
6 9 18	2.7	2.5	7 6 12	2.9	4.7	8 4	10	9.9	9 3	10	6.5
6 9 19	6.0	6.0	7 6 13	4.8	4.8	8 4	11	5.6	9 3	11	9.4
6 9 20	4.0	5.1	7 6 14	4.5	4.8	8 4	12	4.1	9 3	12	4.5
6 10 1	.5	.2	7 6 15	1.5	1.6	8 4	13	.7	9 3	13	3.3
6 10 2	8.0	8.0	7 7 1	39.6	42.0	8 4	14	3.6	9 3	14	3.4
6 10 3	6.3	5.0	7 7 2	19.1	20.3	8 4	15	4.4	9 3	15	3.2
6 10 4	7.1	7.4	7 7 3	20.7	21.0	8 5	1	33.6	9 4	1	6.5
6 10 5	4.3	3.7	7 7 4	10.4	10.8	8 5	1	20.5	9 4	2	9.6
6 10 6	8.9	9.0	7 7 5	7.2	8.4	8 5	2	18.2	9 4	3	3.9
6 10 7	1.9	1.8	7 7 6	15.5	16.2	8 5	3	5.8	9 4	4	6.6
6 10 8	3.5	4.4	7 7 7	4.0	4.1	8 5	4	23.8	9 4	5	5.5
6 10 9	4.5	4.3	7 7 8	2.9	3.4	8 5	5	4.0	9 4	6	4.8
6 10 10	4.6	4.3	7 7 9	9.2	8.4	8 5	6	6.6	9 4	7	4.2
6 10 11	5.6	5.7	7 7 10	1.8	1.4	8 5	7	14.5	9 4	8	4.8
6 10 12	4.5	5.3	7 7 11	.6	.4	8 5	8	5.3	9 4	9	4.8
6 10 13	4.5	5.3	7 7 12	5.5	5.7	8 5	9	5.6	9 4	10	4.2
6 10 14	2.4	2.7	7 7 13	.8	.9	8 5	10	5.6	9 4	11	5.0
6 10 15	1.4	1.9	7 7 14	1.4	.9	8 5	11	3.3	9 4	12	5.4
6 10 16	2.8	3.3	7 7 15	4.1	4.0	8 5	12	5.6	9 4	13	3.2
6 11 1	.9	.6	7 7 16	9.2	9.2	8 5	13	5.9	9 4	14	1.6
6 11 2	1.1	1.4	7 7 17	6.2	6.1	8 5	14	.6	9 5	1	10.5
6 11 3	3.0	2.8	7 7 18	9.7	9.3	8 5	15	4.5	9 5	2	33.3
6 11 4	3.2	3.2	7 7 19	1.4	1.3	8 5	16	27.6	9 5	3	13.6
6 11 5	2.3	2.8	7 7 20	1.4	1.3	8 6	1	17.8	9 5	4	8.1
6 11 6	4.4	5.0	7 7 21	7.6	7.8	8 6	2	4.0	9 5	5	10.1
6 11 7	3.4	3.4	7 7 22	4.8	4.8	8 6	3	12.3	9 5	6	4.7
6 11 8	.6	.5	7 7 23	1.9	2.4	8 6	4	5.8	9 5	7	4.7
6 11 9	1.0	1.1	7 7 24	2.5	2.4	8 6	5	4.2	9 5	8	3.7
6 11 10	1.9	1.9	7 7 25	9.6	9.6	8 6	6	13.6	9 5	9	2.0
6 11 11	6.4	5.9	7 7 26	9.4	9.4	8 6	6	13.6	9 5	10	1.4</

Table 1 (*cont.*)

H	K	L	F0BS	FCAL	H	K	L	F0BS	FCAL	H	K	L	F0BS	FCAL	H	K	L	F0BS	FCAL	H	K	L	F0BS	FCAL
10	8	4	7.4	7.3	11	2	2	.4	.5-	11	4	8	5.4	5.7	12	4	12.3	12.5-	12.5-	12	3	7	5.1	5.1-
10	8	5	4.7	5.0-	11	2	3	2.1	1.6	11	4	9	1.3	1.6-	12	6	8.0	8.0	8.0	12	3	8	8.1	8.0
10	8	6	2.3	2.3	11	2	4	5.5	4.8-	11	4	10	7.5	7.5	12	8	7.4	7.0-	7.0-	12	4	1	2.1	2.3
10	8	7	5.1	5.0	11	2	5	1.1	1.6-	11	5	1	1.0	1.2-	12	1	8.9	9.1-	9.1-	12	4	1	6.4	6.3
10	8	8	3.4	3.3	11	2	6	.8	.7-	11	5	2	2.5	2.6	12	1	1	.6	.6	12	4	2	1.9	2.1-
10	9	10.2	10.4-	10.4-	11	2	7	3.3	3.2	11	5	3	10.2	10.1	12	1	2	1.2	1.2-	12	4	3	2.5	2.4
10	9	1	.7	.6-	11	2	8	4.4	4.6-	11	5	3	3.4	1.1	12	1	3	4.5	4.9-	12	4	4	3.8	3.9-
10	9	2	4.9	5.0-	11	2	9	4.9	4.9	11	5	6	1.6	1.9-	12	1	4	10.9	10.7	12	4	5	2.1	2.0-
10	9	3	2.7	3.3-	11	2	10	.7	.6	11	5	7	3.0	2.8-	12	1	5	5.0	5.0-	12	4	6	1.3	1.2-
10	9	4	6.4	5.7-	11	2	11	.6	1.7	11	5	8	2.4	3.0-	12	1	6	1.9	1.2	12	4	7	4.1	3.2
10	9	5	4.2	3.3-	11	3	1	5.0	5.0-	11	5	9	1.1	.5	12	1	7	8.2	8.1	12	5	1	5.1	4.5-
11	2	2	2.8	2.2	11	3	2	.9	1.3-	11	6	1	3.9	3.7-	12	1	8	4.7	4.9-	12	5	2	.5	.1-
11	4	13.7	13.3	13.3	11	3	3	5.5	6.4-	11	6	2	.4	.4	12	1	9	2.1	2.5	12	5	3	3.3	3.5
11	6	.7	.7	.7	11	3	4	2.8	2.8-	11	6	3	1.2	4.1	12	2	2	3.5	4.0-	12	5	4	6.3	6.0
11	8	1.4	1.6	1.6	11	3	5	4.4	4.6	11	6	4	6.4	5.1-	12	2	2	2.7	2.8	12	5	5	5.2	5.1
11	10	2.7	3.0-	3.0-	11	3	6	2.8	3.5	11	6	5	.7	.3-	12	2	3	.7	.6-	12	6	1	1.5	1.4-
11	12	6.3	6.4-	6.4-	11	3	7	1.6	1.1	11	6	6	4.8	5.0	12	2	4	7.1	7.3	12	6	1	2.8	2.7-
11	1	1	7.6	8.1	11	3	8	13.9	14.0	11	6	8	.2	.7-	12	2	5	.6	.6-	12	6	2	3.4	3.5
11	1	2	12.4	13.0	11	3	9	1.0	1.3	11	7	1	.9	.8-	12	2	6	8.9	8.6	12	6	3	1.0	1.3-
11	1	3	5.4	5.9-	11	3	10	.6	.6-	11	7	2	1.4	1.2-	12	2	7	1.0	1.0-	13	2	3	3.8	4.4
11	1	4	4.4	5.3-	11	3	11	3.2	3.0-	11	7	3	5.4	6.0-	12	2	8	2.3	2.2	13	4	5	5.0	4.7
11	1	5	1.3	1.6	11	4	1	5.7	6.1-	11	7	5	1.7	2.4-	12	3	1	4.3	3.7-	13	1	1	.4	.9-
11	1	6	8.3	8.8-	11	4	2	1.1	1.3-	11	7	6	3.3	3.3-	12	3	2	2.1	2.1	13	1	2	5.3	5.1
11	1	8	8.9	8.8-	11	4	3	4.9	4.5-	11	8	1	2.5	2.8	12	3	2	4.2	4.1	13	1	3	3.1	3.1
11	1	9	3.0	2.9-	11	4	4	1.2	1.7-	11	8	2	2.1	2.9	12	3	3	4.8	4.4	13	1	4	2.1	2.1-
11	1	10	4.6	4.6-	11	4	5	3.8	1.9-	11	8	3	.2	.7	12	3	4	4.0	3.9-	13	2	2	.4	.1
11	1	11	3.0	2.9	11	4	6	2.8	3.0-	12	1	1	8.8	9.2	12	3	5	1.6	1.1-	13	2	3	2.9	2.7-
11	2	1	9.8	10.5	11	4	7	5.1	5.1-	12	2	2	5.5	5.1-	12	3	6	8.1	7.4-	13	3	1	1.5	1.3-
																								4.8-

Table 2. Atomic coordinates* and temperature factors

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> ₁₁	<i>B</i> ₂₂	<i>B</i> ₃₃	<i>B</i> ₁₂	<i>B</i> ₂₃	<i>B</i> ₁₃
N	0.0947	0.2625	0.4071	3.522	3.699	2.589	-0.690	0.301	-0.367
O	-0.0134	0.2215	0.3005	4.996	4.694	3.209	-0.636	-0.230	-1.035
C	0.0067	0.2048	0.3687	3.206	3.175	3.028	-0.054	-0.169	-0.364
C(M)	-0.0633	0.1200	0.4141	3.730	3.753	4.498	-0.927	0.214	-0.391
C(1)	0.1277	0.2390	0.4861	3.453	3.779	2.516	-0.828	0.488	-0.364
C(2)	0.2053	0.1526	0.5032	4.714	4.395	4.245	-0.314	0.969	-0.783
C(3)	0.2344	0.1319	0.5804	5.800	6.043	5.676	-1.534	2.809	-2.113
C(4)	0.1854	0.1968	0.6387	6.794	8.549	3.129	-3.494	1.884	-1.256
C(5)	0.1078	0.2826	0.6206	6.589	8.279	2.885	-2.041	-0.342	0.026
C(6)	0.0800	0.3044	0.5446	4.394	5.789	3.115	-0.655	0.439	0.015
C(1')	0.1605	0.3483	0.3682	3.412	3.447	2.186	-0.205	-0.010	-0.288
C(2')	0.1000	0.4394	0.3389	4.444	3.769	3.503	0.271	0.195	-0.375
C(3')	0.1639	0.5221	0.3011	6.503	3.709	3.618	-0.363	0.646	-0.499
C(4')	0.2885	0.5140	0.2929	6.196	4.937	3.724	-2.000	0.644	0.127
C(5')	0.3490	0.4224	0.3215	4.042	5.629	4.185	-1.255	0.495	0.285
C(6')	0.2850	0.3397	0.3591	3.659	4.456	3.720	0.056	0.556	-0.184
H(1M)†	-0.098	0.155	0.456						
H(2M)	-0.105	0.090	0.380						
H(3M)	-0.016	0.059	0.426						
H(2)	0.243	0.102	0.458						
H(3)	0.294	0.065	0.594						
H(4)	0.208	0.180	0.698						
H(5)	0.070	0.333	0.666						
H(6)	0.020	0.371	0.531						
H(2')	0.003	0.446	0.346						
H(3')	0.117	0.593	0.279						
H(4')	0.338	0.578	0.264						
H(5')	0.446	0.416	0.315						
H(6')	0.332	0.269	0.382						

* The coordinates are expressed as fractions of the unit cell edges.

† The temperature factors for hydrogen could not be determined. During the final refinements they were assigned the same temperature factors as the carbon atoms to which they were bonded.

sities of reflections having $\sin \theta \leq 0.40$ were remeasured with the second crystal, *R* became 0.169 after one cycle of refinement. A test of computed absorption corrections for 50 randomly selected reflections indicated an average error of 3% in the structure factors, which we felt could be neglected. Anisotropic temperature factors were then introduced, which reduced *R* to 0.113 after two cycles of refinement. A difference Fourier synthesis, shown in slab projection down the *y* axis in Fig. 1, clearly showed all thirteen hydrogen atoms, thus locating the three methyl hydrogens. Six of the most intense reflections, 020, 212, 220, 022, 004, and 104

still exhibited extinction effects, so these structure factors were assigned the calculated values, giving *R* = 0.106. Correction of some bookkeeping errors, and five further cycles of refinement, led to the final value, *R* = 0.059, for the 2136 observed reflections. The observed and calculated structure amplitudes appear in Table 1. During the last two cycles the shifts in both coordinates and temperature factors were so small that further refinement appeared unwarranted. Fig. 2 shows a composite of electron density sections taken through the atomic centers, and projected upon the *xy* plane.

Attempts were made to refine the hydrogen atom positions, both by differential and by difference differential syntheses. These were unsuccessful, despite the fact that all of the hydrogen atoms appear to be clearly resolved in the difference electron density projection shown in Fig. 1.

Description of the structure

This structure is of interest because it bears some similarity to the peptide portion of proteins, yet some differences might be anticipated owing to the presence of the two bulky phenyl groups, and the absence of any possibility for hydrogen bonding. Table 2 gives the atomic coordinates and temperature factors, while the standard deviations in atomic positions, bond angles and bond distances, as calculated according to the method of Cruickshank (1949), appear in Table 3. The intramolecular bond distances and angles are shown in Fig. 3.

Table 3.

(a) Estimated standard deviations in bond distances			
N—C	0.0028 Å	C(5)—C(6)	0.0045 Å
C—C(<i>M</i>)	0.0034	C(6)—C(1)	0.0035
C—O	0.0028	C(1')—C(2')	0.0031
N—C(1)	0.0029	C(2')—C(3')	0.0038
N—C(1')	0.0027	C(3')—C(4')	0.0041
C(1)—C(2)	0.0035	C(4')—C(5')	0.0040
C(2)—C(3)	0.0042	C(5')—C(6')	0.0037
C(3)—C(4)	0.0048	C(6')—C(1')	0.0031
C(4)—C(5)	0.0051		
(b) Estimated standard deviations in bond angles			
N—C—C(<i>M</i>)	0.20°	C(4)—C(5)—C(6)	0.34°
O—C—C(<i>M</i>)	0.20	C(5)—C(6)—C(1)	0.28
N—C—O	0.20	C(6)—C(1)—C(2)	0.22
C—N—C(1)	0.20	C(1')—C(2')—C(3')	0.24
C—N—C(1')	0.20	C(2')—C(3')—C(4')	0.25
C(1)—N—C(1')	0.20	C(3')—C(4')—C(5')	0.27
C(1)—C(2)—C(3)	0.25	C(4')—C(5')—C(6')	0.25
C(2)—C(3)—C(4)	0.30	C(5')—C(6')—C(1')	0.22
C(3)—C(4)—C(5)	0.34	C(6')—C(1')—C(2')	0.20
(c) Estimated standard deviations in atomic positions			
N	0.0018 Å	C(5)	0.0036 Å
O	0.0018	C(6)	0.0027
C	0.0021	C(1')	0.0020
C(<i>M</i>)	0.0027	C(2')	0.0024
C(1)	0.0022	C(3')	0.0029
C(2)	0.0027	C(4')	0.0029
C(3)	0.0032	C(5')	0.0028
C(4)	0.0036	C(6')	0.0024
(d) Root-mean-square estimated standard deviations in electron density and its derivatives			
Electron density	0.053 Å ⁻³		
<i>A_h</i>	0.150 Å ⁻⁴		
<i>A_k</i>	0.143 Å ⁻⁴		
<i>A_i</i>	0.146 Å ⁻⁴		
<i>A_{h_k}</i>	0.351 Å ⁻⁵		
<i>A_{k_i}</i>	0.389 Å ⁻⁵		
<i>A_{l_h}</i>	0.384 Å ⁻⁵		
<i>A_{h_h}</i>	0.654 Å ⁻⁵		
<i>A_{k_k}</i>	0.654 Å ⁻⁵		
<i>A_{l_l}</i>	0.639 Å ⁻⁵		

The acetyl portion for *N,N*-diphenylacetamide is quite planar. The least-square plane for the atoms N, O, C, and C(*M*), expressed in terms of fractional coordinates, is:

$$0.6726x - 0.6976y - 0.2471z = -3.2496$$

and the displacements from this plane are:

$$N + 0.0004; O + 0.0005; C - 0.0013; C(M) = 0.0003 \text{ \AA}.$$

The two benzene rings of *N,N*-diphenylacetamide are also planar to within the experimental uncertainty. The equation for the least-square plane for the primed ring (nearest oxygen) is:

$$0.1308x + 0.4749y + 0.8702z = 7.7658,$$

with displacements:

$$C(1') + 0.0044; C(2') - 0.0011; C(3') - 0.0039; \\ C(4') + 0.0056; C(5') - 0.0023; C(6') - 0.0027 \text{ \AA},$$

while the equation of the unprimed ring carbon atoms is:

$$0.7821x + 0.1619y - 0.0670z = 2.3290$$

with displacements:

$$C(1) - 0.0028; C(2) - 0.0021; C(3) + 0.0035; \\ C(4) 0.0000; C(5) - 0.0050; C(6) + 0.0064 \text{ \AA}.$$

As shown in Fig. 3, the structure of the primed ring is considerably more regular, the maximum variation in bond distance being 0.003 Å for this ring, and

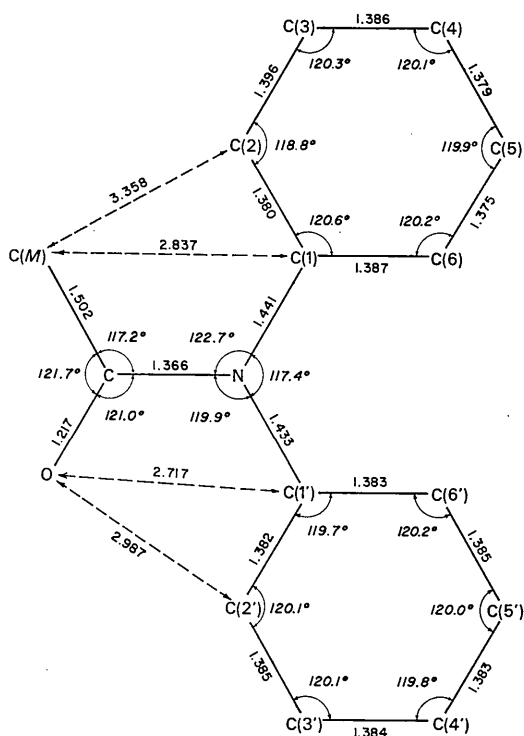


Fig. 3. Inter-molecular bond distances and angles in *N,N*-diphenylacetamide.

0.021 Å for the unprimed ring. The structural parameters obtained for diphenylacetamide agree quite well with the corresponding portions of the acetanilide structure originally given by Brown & Corbridge (1954), and recently refined by Brown (1966). As might be expected, the latter refinement has substantially improved the agreement between the two structures. For example, the acetyl group became more nearly planar (in agreement with the present structure), the average aromatic bond distances for the two structures now differ by only 0.002 Å, and the average difference for the other bonds was reduced from 0.021 Å to 0.011 Å. In only one case did the agreement of bond distances become poorer. Refinement reduced the C(1)–N bond length of acetanilide from 1.426 to 1.413 Å, as compared with 1.441 Å for C(1)–N and 1.433 Å for C(1')–N in diphenylacetamide.

The bond angle of 118.8° at C(2) is one disturbing irregularity in the diphenylacetamide structure. Since the estimated standard deviation for this bond is only 0.25°, the departure from the expected 120° appears to be statistically significant. On the other hand, similar divergences are found in the refined acetanilide structure, in which the aromatic bond angles range from 119.0° to 121.7°.

In acetanilide the π electron interaction draws the benzene ring to within 17.6° of the amide plane, reducing the distance corresponding to C(2')–O in Fig. 3 to 2.87 Å. This results in considerable strain, as shown by the displacement of that nitrogen atom 0.046 Å from the mean plane of the aromatic carbon atoms, and by the expansion of the bond angle corresponding to CNC(1') to 127.6°. On the other hand, Pederson & Pederson (1965) report that the benzene ring of *N*-methylacetanilide occupies the *exo* position *trans* to oxygen, and is orthogonal to the amido plane. This implies that the π electrons of a benzene ring in the *exo* position are not favorably disposed for interaction, and leads to the prediction that the primed ring of *N,N*-diphenylacetamide should lie close to the amido plane, while the unprimed ring should be orthogonal to it. However, this is not the case. The observed angles, 61.75° (primed ring) and 77.25° are both probably as small as the rather severe steric requirements will permit. The equation of the least-squares plane fitted to the atoms N, O, C(*M*), C(1), C(4), C(1'), C(4') is:

$$0.6777x - 0.6727y - 0.2970z = -3.5089.$$

The displacements from this plane,

$$\begin{aligned} \text{N} &-0.0076; \text{O} +0.0061; \text{C} +0.0016; \text{C}(\text{M}) -0.0653; \\ \text{C}(1) &+0.0244; \text{C}(4) +0.0125; \text{C}(1') -0.0139; \\ \text{C}(4') &-0.0178 \text{ \AA}; \end{aligned}$$

suggest that there is some torsional distortion of the molecule about the C–N bond which reduces the interactions of C(*M*) and C(2), and O and C(2'). The fact that the methyl group is not rotating is further evidence for a strong interaction of C(*M*) and C(2).

The temperature coefficients shown in Table 3 indicate that the thermal motion in the vicinity of the *para* carbon atoms is large and anisotropic, as might be expected for such an angular structure. The shortest intermolecular distances are listed in Table 4. These are mainly of the expected magnitude, with the exception of the rather small H(5)–H(4') distance of 2.248 Å.

Table 4. Intermolecular distances

(a) Distances less than 4.0 Å involving atoms other than hydrogen

N—C(4')	6*	3.815 Å
O—C(3)	2	3.884
O—C(4)	2	3.616
O—C(4)	7	3.687
O—C(5)	7	3.386
O—C(3')	3	3.410
O—C(4')	6	3.525
O—C(5')	8	3.554
O—C(6')	8	3.819
C—C(3)	2	3.695
C—C(4)	2	3.734
C—C(4')	6	3.480
C—C(5')	6	3.849
C(<i>M</i>)—C(2)	5	3.915
C(<i>M</i>)—C(3)	2	3.735
C(<i>M</i>)—C(3)	5	3.579
C(<i>M</i>)—C(4)	2	3.658
C(<i>M</i>)—C(5)	2	3.854
C(<i>M</i>)—C(4')	6	3.897
C(<i>M</i>)—C(5')	6	3.718
C(4)—C(2')	7	3.946
C(4)—C(3')	7	3.864
C(4)—C(4')	7	3.858
C(4)—C(5')	7	3.913
C(4)—C(6')	7	3.990
C(5)—C(5')	2	3.905
C(5)—C(6')	2	3.865
C(2')—C(5')	8	3.920

(b) Distances less than 3.0 Å involving hydrogen atoms

O—H(5)	7	2.586 Å
O—H(3')	3	2.367
O—H(4')	6	2.670
C—H(4')	6	2.923
C(4')—H(4)	7	2.994
C(5')—H(4)	7	2.915
C(6')—H(4)	7	2.921
H(2 <i>M</i>)—H(3)	5	2.835
H(2 <i>M</i>)—H(3')	3	2.753
H(2 <i>M</i>)—H(5')	6	2.957
H(3 <i>M</i>)—H(3 <i>M</i>)	5	2.950
H(3 <i>M</i>)—H(5')	6	2.694
H(3)—H(2')	2	2.528
H(5)—H(2')	5	2.795
H(5)—H(3')	5	2.438
H(5)—H(4')	4	2.248
H(2')—H(5')	8	2.876
H(3')—H(6')	6	2.829

* Symmetry code

1. x, y, z
2. $\frac{1}{2} + x, \frac{1}{2} - y, \bar{z}$
3. $\bar{x}, \frac{1}{2} + y, \frac{1}{2} - z$
4. $\frac{1}{2} - x, \bar{y}, \frac{1}{2} + z$
5. $\bar{x}, \bar{y}, \bar{z}$
6. $\frac{1}{2} - x, \frac{1}{2} + y, z$
7. $x, \frac{1}{2} - y, \frac{1}{2} + z$
8. $\frac{1}{2} + x, y, \frac{1}{2} - z$

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Hydrogen Atom Positions in Manganous Formate Dihydrate and a Rerefinement of Copper Formate Dihydrate

BY M. I. KAY, I. ALMODOVAR AND S. F. KAPLAN

Puerto Rico Nuclear Center, Mayaguez, Puerto Rico*

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Three zones of neutron diffraction data were obtained for $\text{Mn}(\text{HCO}_2)_2 \cdot 2\text{H}_2\text{O}$ and the positions of the hydrogen atoms determined. The hydrogen bonding scheme proposed by Osaki, Nakai and Watanabe was found to be correct. The crystal structure of copper formate dihydrate was rerefined by means of the method of least squares. Some changes in bond distances were noted.

Introduction

Manganous formate dihydrate, $\text{Mn}(\text{HCO}_2)_2 \cdot 2\text{H}_2\text{O}$, is representative of a large number of more or less isostructural formate dihydrates having bivalent cations of similar ionic radius. The space group of these compounds is $P2_1/c$. There are four formula weights per unit cell with two independent metal ions in two kinds of twofold special positions and two independent formate ions and water molecules in the fourfold general positions. The structures of the Mg, Zn, and Mn salts have been determined by Osaki, Nakai & Watanabe (1963, 1964; hereafter referred to as ONW), the Ni salt by Krogman & Mattes (1963), and the Cu salt by Bukowska-Strzyzewska (1965; BS). The Cd and Fe salts (Hoy, Barros, Barros & Friedberg, 1965) are also known to be of similar structure. Since these compounds have important magnetic properties (Abe & Matsura, 1964; Abe, Morigaki, Matsura, Torii & Yamagata, 1964; Abe & Torii, 1965; Hoy *et al.*, 1965), and Cohen, Friedberg & Wagner (1964), it was decided to determine the hydrogen positions in at least one of them to permit a better correlation of structure with other properties, particularly proton magnetic resonance spectra. The chemical binding, and its relationship to different degrees of hydration of the copper salt, is also of interest in its own right.

The structure of copper formate tetrahydrate was determined by Kiriya, Ibamoto & Matsuo (1954) and refined with neutron data by Okada, Kay, Cromer & Almodovar (1966), who found layers of copper formate alternating with layers of water containing disordered hydrogen atoms. The dihydrate structures are characterized by (100) layers of metal formate similar to the (001) layers of the $P2_1/a$ tetrahydrate, with the metal atoms at 0, 0, 0 and $0, \frac{1}{2}, \frac{1}{2}$. In the second layer near $x = \frac{1}{2}$, however, there is a second set of metal ions at $\frac{1}{2}, \frac{1}{2}, 0$ and $\frac{1}{2}, 0, \frac{1}{2}$, connected to the first set by a formate group and also connected to four water oxygen atoms which complete a coordination octahedron. That is, the metal ions of the first set at $x = 0$ are each surrounded by an octahedron of six formate oxygens while those of the second set at $x = \frac{1}{2}$ are each surrounded by an octahedron of two formate oxygen atoms and four water oxygen atoms. The metal atoms and formate groups thus form a three-dimensional network, in contrast to the two-dimensional system found in the tetrahydrate. All metal atoms are on symmetry centers. A stereoscopic view of the structure is given in Fig. 1.

The present work is concerned with the determination of the hydrogen positions in manganous formate dihydrate and a rerefinement of the structure of copper formate dihydrate. BS found large differences in the C–O distances in the latter compound which she ascribed to differences in Cu–O distances which in turn cause one C–O bond to have considerably more double-bond character than the other. This effect was not ob-

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