INTRA VS. INTERMOLECULAR HYDROGEN BONDING OF 2-BENZOYLCYCLOHEXANECARBOXYLIC ACID

By

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Thesis Approved:

Thesis Adviser

Dean of the Graduate College

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CHAPTER I

INTRODUCTION

Metal hydride reduction of the cis- and trans-2-benzoylcyclohexanecarboxylic acids provides the corresponding hydroxy acids with high selectivity for obedience of the Cram rule.[1] Platinum oxide-catalyzed hydrogenation, however, favors formation of the anti-Cram product but with less selectivity. Intramolecular association of functional groups, carboxylate anion and ketone carbonyl carbon, during metal-hydride reduction and hydrogen bonding of carboxyl group to ketone carbonyl oxygen during catalytic hydrogenation is assumed to control the product stereochemistry during reduction. The determination of exact conformation of compounds in the crystalline state is readily accomplished using X-ray structural analysis. The question arises as to how crystal forces influence the conformation of these flexible molecules and whether the solid-state conformation differs from that in solution.

The objective of this research is to determine the solid-state structure of (±)-cis-2-benzoylcyclohexanecarboxylic acid (6), to investigate the possibility of intramolecular interaction of functional groups in the solid state, and to make comparison of the results of NMR and IR studies in the liquid state.

CHAPTER II

HISTORICAL

Latimer and Rodebush were the first to recognize the existence of hydrogen bonding in 1920.[2] They also were the first to introduce the term "hydrogen bond", but credit for introducing the hydrogen bond concept should be given to Maurice L. Huggins.[3] As he stated,

> It is my prejudiced belief that, except for electron-pair bonding between atoms and coulombic attractions and repulsions between ions, the most structural principle in chemistry and biology is that of hydrogen bonding. Hydrogen bonding is also of great importance in physics, crystallography, minerology, geology, meteorology, and various other 'ologies.' I therefore take pride in the fact that I was the first to introduce the hydrogen bond concept: the idea that a hydrogen atom can be bonded at the same time to two other atoms.

Lewis credits Huggins for the idea of hydrogen bonding by writing

> It seems to me that the most important addition to my theory of valance lies in the suggestion of what has become known as the hydrogen bond. The idea was first suggested by Dr. M. L. Huggins and was also advanced by Latimer and Rodebush, who showed the great value of the idea in their paper to which reference has already been made.[4]

Initially the hydrogen bond between two like atoms was assumed to be symmetrical. The reason for this assumption was the small shift in infrared band frequencies when a compound containing an hydroxyl group passes from a gaseous state to the liquid or solid state. Bernal and Fowler[5] came to the conclusion that the hydrogen bonds are unsymmetrical in such substances as water. They suggested that water remains in part

a hydrogen bonded structure similar to that of ice. As more and more hydrogen bonds are broken, the water molecules may arrange themselves in a manner approximating close packed spheres, which would result in a significant increase in density. There are instances, however, in which the hydrogen bonds between like bridgehead atoms are definitely symmetrical, with the hydrogen atom in the middle of the bridge.

Some general limitations of definitions of hydrogen bonding were introduced by Bernal.[5]

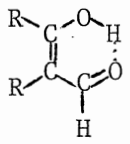
1. The presence of a hydrogen, or a deuterium or tritium atom on or near a line joining two other atoms.
2. The atoms involved are almost exclusively those of fluorine, oxygen and nitrogen.
3. The strength of the hydrogen bond is determined by another atom, usually covalently or ionically linked to the oxygen (nitrogen, fluorine) which we may call the proton-activator.
4. The hydrogen bond is made to an atom, which may be called a proton-acceptor, which may or may not itself have also a hydrogen atom attached to it that is either oxygen, NH, or OH.
5. A hydrogen bond may be relatively free, that is be at a state of energy minimum with respect to the bond itself, or it may be constrained by molecular or crystal geometry.

Hydrogen bonds can be formed in two ways, either within molecules forming intramolecular hydrogen bonds or between molecules forming a hydrogen bond between two molecules of the same or different substances. The

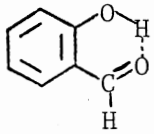
structures which result are different, and the properties of the individual compounds are dependent on the formation of an intramolecular or intermolecular bond. Compounds that form intermolecular hydrogen bonds have high freezing and boiling points because of the strong intermolecular forces. This effect is reduced or lost if the hydrogen bonding groups are so placed in the molecule as to permit intramolecular hydrogen bonding.[6]

An intramolecular hydrogen bond can form only when one proton donor and one proton acceptor site on the same molecule are in a favorable spatial configuration, that is, the distance between the hydrogen of the donor group and the acceptor site is between 1.4 and 2.5 Å, and the angular orientation of the acceptor site does not deviate greatly from the bond axis of the donor group.[7]

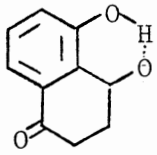
Studies of the intramolecular hydrogen bonds between hydroxyl groups and electronegative atoms in other groups furnish evidence regarding the limitations of such bonds. Using three different compounds, the ring produced by the intramolecular hydrogen bonding is, in all three types, six membered; also it contains either two conjugated double bonds or one bond conjugated with a benzene ring.[8]



1a

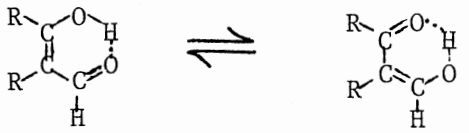


2



3

Such a distribution of bonds weakens the attraction of the hydroxyl oxygen for its hydrogen, at the same time strengthening the attraction of the oxygen on the other side for this hydrogen. In resonance terminology, the ring containing the bridge is stabilized because of hybridization of forms 1a and 1b.



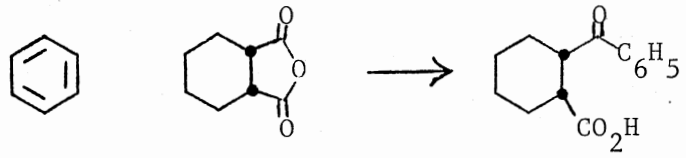
1a 1b

Another important factor of intramolecular hydrogen bonding is the increased rigidity caused by a double bond or benzene ring, making the single bonds on each side necessarily coplaner. The decrease of stability of an intramolecular hydrogen bond may be caused not only by the lack of rigidity and reduced possibility of resonance, but also by restricted rotation around single bonds hindered by the attractions and repulsions of neighboring non-bonded atoms.[7]

Many reactions in chemistry depend on the way in which hydrogen bonding occurs. Intramolecular hydrogen bonding generally makes the reduction of a reactive group easier since the hydrogen atom or double bond is less mobile. In the reduction of 2-cis-benzoylcyclohexanecarboxylic acid, different hydride reducing selectivity was observed. This seems due to the fact that there is a control by intramolecular association of functional groups during reduction through hydrogen bonding in catalytic hydrogenation.[9]

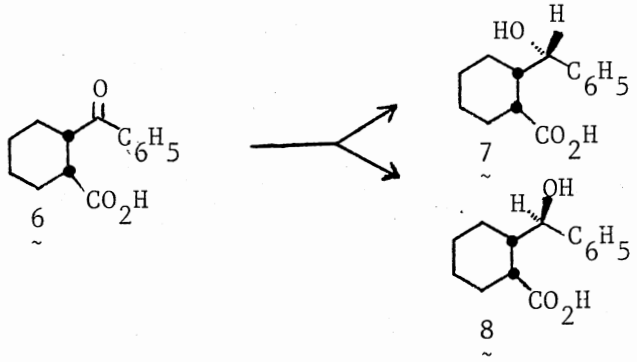
2-Benzoylcyclohexanecarboxylic acid (6) was easily synthesized by the Friedel-Crafts reaction of cyclohexane-1,2-dicarboxylic anhydride

with benzene as reported by Fieser and Novello,[10] and later by Scribner and Miller.[11] The Scribner and Miller procedure was easily reproduced, and pure cis-keto acid was isolated in 97% yield from a large scale Friedel-Crafts reaction of cis-hexahydrophthalic anhydride (5) with benzene (4) in the presence of aluminum chloride.



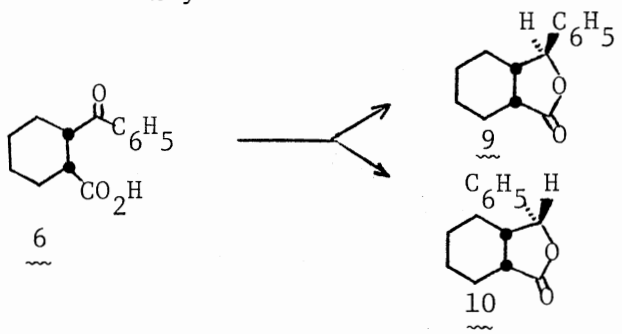
4 5 6

Reduction of the keto acid 6 to hydroxy acids was carried out with sodium borohydride; however, the reaction was extremely slow. Even after three days of stirring the substrates with the reagent in ethanol, isopropyl alcohol, or methanol, the product was a mixture of keto acids and hydroxy acids. Because of the slow reduction of the keto acid using this method, epimerization occurred and none of the two desired hydroxy acids 7 and 8 could be isolated in a reasonable purity.



From a search for a more powerful reducing reagent which would attack the keto function leaving the carboxyl group untouched, lithium triethylborohydride (Super-Hydride) appeared promising.[12] Reduction of cis-keto acid (6) with Super-Hydride afforded a mixture of cis-lactones 9 and 10

in a 94:6 ratio and 82% yield.



No hydroxy acid could be isolated from the reaction mixture.[13] The Fieser-Novello[10] preparation of (7) and (8) with zinc-alkali reduction of keto acid (6) to a hydroxy acid with mp 145-147 °C, could not be accomplished. Direct reduction of the cis-keto acid 6 to cis-2-benzylcyclohexane carboxylic acid (11) in ethyl acetate using Pd/C with less than 1% epimerization was successfully carried out. However, the Clemmensen reduction yielded only the more stable trans-desoxy acid (12).

The greatest hydride reducing selectivity was observed with lithium trialkylborohydrides and lithium tri-t-butoxyaluminohydride. The product ratio of a given pair of γ-lactones is controlled by intramolecular association of functional groups during reduction, through hydrogen bonding in catalytic hydrogenation, or through interaction of carboxylate anion and ketone carbonyl groups during metal hydride reduction.[13]

CHAPTER III

RESULTS AND DISCUSSION

The final comparison between the observed and calculated crystal structure factors is given in Table I (pages 20-29). Table II (page 30) lists atomic coordinates. Table III (page 31) lists the bond angles (°) and distances (Å). Tables IV and V (pages 32, 33) contain the Anisotropic and Isotropic Thermal Parameters of (6), respectively.

Figures 1, 2, and 3 (pages 35, 36, and 37) show the molecular structure of (±)-cis-2-benzoylcyclohexanecarboxylic acid (6). Figure 4 (page 38) shows the same molecule with bond distances and Figure 5 (page 39) shows the dimer of (6). Projection View of Unit Cell (a x b x 1/2c) is shown in Figure 6 (page 40).

As shown in Figure 1, carbons 1-6 are numbered on the benzene ring starting with the substituted carbon. The benzylic position becomes carbon 7 and the carbonyl of the carboxyl group is numbered 14. Carbons 8-13 are numbered on the cyclohexane ring, the carbon bearing the carboxyl substituent being 13.

Many studies of derivatives of cyclohexane show solid-state conformation exclusively of the chair type. No substituted cyclohexanes are free from strain, the closest approach between substituents being between those in adjacent axial and equatorial positions.[14,15] The bond length

and bond angles of the cyclohexane ring show an average value of 1.525Å and 111.3Å, which is in agreement with cyclohexane compounds found in the literature.[16]

The bond length and bond angles in the benzene ring show a very slight variation of hexagonal symmetry. The average value for the C-C distance in the ring is 1.376Å, which is in agreement with the literature values for the C-C distance in the aromatic compounds of 1.395Å.[16]

The fact that the C(14) - O(3) bond (1.226Å) is shorter than the C(14 - O(2) bond (1.317Å) supports the conclusion that the carboxyl proton is attached to O(3) and not O(2). This conclusion is also in agreement with the fact that the angle O(3) - C(14) - C(13), 112.9°, is smaller than O(2) - C(14) - C(13), 114.2°. The angle O(2) - C(14) - O(3) is 112.9°. The benzoyl oxygen points away from the carboxyl group and the bond length (1.212Å) indicates that there is no hydrogen bonding.

The eight molecules of (±)-2-benzoylcyclohexanecarbocylic acid exist in pairs bonded together with two intermolecular hydrogen bonds involving the carboxyl groups, [O2 - H1, 1.00(4)Å; H1 - O3', 1.65(4)Å]. The ketone carbonyl oxygen atom, O1, is not involved in hydrogen bonding and does not show the intramolecular hydrogen bonding postulated for the molecule in solution. The cyclohexane ring exists in chair form with the benzoyl substituent in an axial arrangement and the carboxylic substituent in an equatorial orientation as predicted by conformational theory.[15] All angles and distances are normal. There are no other significant intermolecular contact distances.

The proton-decoupled ^{13}C NMR spectra shows the acid having twelve lines, of which six appear in the aliphatic range. Interconvertible con-

formations with two substituents being axial-equatorial to each other contribute to the ^{13}C spectrum of the molecule. The effect of a carboxyl substituent on ^{13}C chemical shifts of cyclohexane ring has been studied, and the chiral shift parameters for this substituent are known.[17,18]

A carboxyl ^{13}C substituent in an axial position deshields the α position by 11.7 ppm, and the β position by 0.2 ppm, but shields the γ carbons by 4.4 ppm and δ carbon by 1.0 ppm. When the carboxyl group takes an equatorial position, it has a deshielding effect on both the α carbon (16.2 ppm) and an equal shielding effect on β, γ, and δ carbons (1.6 ppm). The effect of the benzoyl substituent can also be estimated from the ^{13}C chemical shift values for an acetyl substituted cyclohexane.[19] Correcting for the effect of substituting a phenyl group for methyl[20,21] α- and β-deshielding would be in the order of 22-25 for α, and 1-2 for β carbons, and a shielding of almost the same magnitude as for the carboxyl substituent for γ- and δ-carbons. Based on these literature data, the assignment of ^{13}C chemical shifts were made.[13]

In the FT-Infrared spectra, 2-benzoylcyclohexanecarboxylic acid (6) shows strong absorptions at 2850 cm^{-1} and 1685 cm^{-1}. One of the most characteristic features of carboxylic acids is the broad O - H absorption from 3400 - 2400 cm^{-1}. Strong hydrogen bonding present in the dimer is responsible for the absorption at 2850 cm^{-1}. Since the absorption is very broad, it will hide the C-H stretching vibration which occurs in the same region. Only when carboxylic acids are in very dilute solutions or in the vapor phase, can they exist as acid monomers.[20] The monomers absorb at about 1760 cm^{-1} because of an electron withdrawing effect. The strong hydrogen bonding weakens the C=O bonds, thus lowering the C=O absorption frequency. The appearance of the carbonyl stretching peak at

1700 cm^{-1} and a broad hydroxyl peak, with λ_{max} at 2850 cm^{-1} indicate that the acid is largely in the hydrogen-bonded, dimeric form. Intramolecular hydrogen bonding would have caused a greater shift of the carbonyl group to a lower frequency than the intermolecular bonding did. Since no measured shift was seen, the presence of intramolecular bonding could not be established.

Conjugation of the carbonyl group with an aryl ring shifts the normal C=O stretching band to a lower frequency which for 6 is 1685 cm^{-1}.

In NMR measurements, the limiting "observation" time is determined by the relaxation times of the proton spin orientations in the magnetic field which is about 10^{-3} sec and even longer, whereas in infrared measurements the observation time is determined not only by the vibrational frequencies of the hydrogen bond, but also by the acceptor group itself. Because of the weakness of the hydrogen bond and the large number of collisions taking place in molecular systems even at low concentrations, many hydrogen bonds are continuously breaking as new hydrogen bonds are forming. Thus, the time in which a hydrogen bond is broken and reformed is so short that it can only be observed as an average which will depend on both the number and kind of donor species present. This makes IR spectrosopy the more useful tool in hydrogen bonding studies of solutions, particularly in studies of self-associated systems. The presence of two concentration independent OH absorption bands in the IR spectrum is in general considered to be characteristic of intramolecular hydrogen bonding. Of course, the use of both NMR and FT-IR methods should provide a more complete picture of a hydrogen bonded system than either method alone.

The X-ray structural analysis shows that the compound in the crys-

talline state has a different conformation as compared to that in solution. In the crystal, the benzoyl group is shown to be axial to the carbonyl group. The changes in conformation are essentially due to hydrogen bonding when intra- and intermolecular hydrogen bonds compete with one another. It seems obvious that this is an example in which the crystal structure is not consistent with the most stable conformation in solution.

In this research, evidence has been presented regarding the existence and nature of the hydrogen bonding in 2-benzoylcyclohexanecarboxylic acid as it affects the structure in the solid state.

CHAPTER IV

EXPERIMENTAL

All melting and boiling points are uncorrected. Fourier transform infrared spectra were recorded on a Fourier transform infrared spectrometer Digilab FTS-20 C. Proton NMR spectra were determined at 100.1 MHz on a Varian XL-100A using tetramethylsilane as internal standard in $CDCl_3$ or DMSO-d_6 solvent. The ^{13}C NMR spectra were obtained at 25.2 MHz in the FT mode on a Varian XL-100A interfaced with a 12 K Nikolet 1080 computer system. Chemical shifts are reported in ppm using tetramethylsilane as an internal standard.

cis-2-Benzoylcyclohexanecarboxylic Acid (6).

A 775 g (5 mol) sample of cis-hexahydrophthalic anhydride and 6 L of benzene were mechanically stirred in a 12-L flask at 0-5 °C. To the resulting solution was added 1343 g (10.1 mol) $AlCl_3$ during a 2 h period. The temperature was maintained below 10 °C during the addition. The yellow mixture was stirred for forty min, the cooling bath was drained, and the flask contents were then warmed to 40-50 °C by adding hot water to the tub surrounding the flask. The reaction mixture was decomposed by pouring onto 5 Kg of ice mixed with 2 L of concentrated hydrochloric acid. The resulting mixture was transferred to a 22-L separatory funnel and 4 L of ether were added. The organic layer was separated, washed with

water, dried ($MgSO_4$), and concentrated to 5 L. The keto acid crystallized upon cooling, and 815 g were collected by filtration. Evaporation of solvent from mother liquor gave another 315 g of the keto-acid. The overall yield was 1130 g (97%); mp 138-139 °C (lit.[11] mp 138.5-140.9 °C: FT-IR (CCl_4) cm^{-1} 2850 (CO_2H), 1700 and 1685 (COO; ^1H NMR ($CDCl_3$) δ 7.9-7.2 (m, 5, ArH), 3.9 (m, 1), 2.7 (m, 1), 2.301.3 (m, 8); ^{13}C NMR ($CDCl_3$) ppm 202.02 (CO), 180.31 (CO_2H), 136.36, 132.30, 128.31, (x2), 127.98 (x2), 44.06, 42.70, 27.58, 24.40, 22.38.

Crystal Data.

Colorless crystals of (±)-cis-2-benzoylcyclohexanecarboxylic acid (6) were obtained by slow evaporation of a solution of the compound in acetic acid at room temperature.

A crystal of approximate dimensions 0.5 x 0.3 x 0.2 mm was mounted on a Syntex ^3P diffractometer having graphite monochromated molybdenum K_α radiation (λ = 0.71069 monochromator angle of 6.1 °).

A preliminary rotation diffraction pattern was obtained using a Polaroid casette and showed sharp, strong diffraction indicative of a crystal sufficiently ordered for high-resolution single-crystal analysis. [Each diffraction spot is the result of the condition $N\lambda=2d\sin\theta$ being satisfied (where λ = the wavelength of incident radiation, d = the distance between diffracting planes and θ is the angle of incidence) by the crystal at a particulr physical orientation]. During normal alignment procedures, fifteen independent reflections ($15°<2\theta<30°$) were used for determination of accurate unit cell dimensions (a - 17.103(6), b = 6.758(2), c = 22.149(7)Å, and β = 101.20(3)°). The data showed systematic absences hkl, h + k - 2n + 1, h01 1 = 2n + 1 (h = 2n +1), and 0k0

$k = 2n + 1$ which were consistent with the centrosymmetric space group C2/c. The calculated density (cell volume of 2511(2)$\mathring{A}^3$) with 8 molecules per unit cell (MW 232.28) is 1.22 g cm^{-3}.

Data (3293 independent reflections were measured) of which 1285 reflections were observed [$1 > 3.0\ \sigma(I)$] where I is the observed structure amplitude and $\sigma(I)$ is its corresponding estimated standard deviation after the background was subtracted and the intensity corrected for Lorentz and polarization factors. The scan mode was $0\text{-}2\theta$ with $2\theta_{max}$ = 116°. Three high angle reflections in the crystal were chosen as standards. Intensities of these standards were remeasured after every 97 reflections. The net intensities of these standards declines less than 5% over the whole data collection period.

The linear absorption coefficient for this compound with Mo K_α radiation is $\mu = 1.00\ cm^{-1}$, a very low value, so absorption corrections were deemed unnecessary.

After normalization of the structure factors and phasing using the triplet relationships (MULTAN80),[23] the resulting density distributions were plotted in maps of sections through the unit cell. This preliminary map was interpreted in terms of a postulated molecular structure.

Using full matrix least-squares refinement of scale factor, positional and anisotropic thermal parameters, an R-factor of 6.8% was obtained. The R-factor is the magnitude of the disagreement between the data calculated from the assumed model and the actual measured data.

$$R = \frac{\Sigma(||F_{obs}| - |F_{cal}|)|}{\Sigma|F_{obs}|} \times 100$$

Structure factors (F_{cal}) are calculated from the assumed atomic coordinates and compared to the observed structure factors (F_{obs}) derived from the measured diffraction intensities. After determining hydrogen positions from a difference Fourier synthesis, a final refinement cycle was run with the hydrogen atoms included with isotropic thermal parameters leading to a satisfactory R-factor of 4%.

Ortep, a Fortran Thermal-Ellipsoid Plot Program for Crystal Structure Illustrations, written by Carroll Johnson of Oak Ridge National Laboratory, was used to draw structures of the molecule in a projected three-dimensional view in various orientations. The program reads in x,y,z coordinates for atoms, cell parameters and symmetry information and allows visual evaluation of its structural aspects.

BIBLIOGRAPHY

1. Cram, H.; Elhafez, S. J. Am. Chem. Soc. 1952, 74, 5828.

2. Latimer, W. M.; Rodebush, W. H. J. Am. Chem. Soc. 1920, 42, 1419.

3. Huggins, M. L. Angewandte Chemie, International Edition 1971, 10, 147-208.

4. Lewis, G. N. "Valence and the Structure of Atoms and Molecules," Chemical Catalog, New York, 1923.

5. Bernal, J. D.; Fowler, R. H. J. Chem. Phys. 1933, 1, 515.

6. Pimentel, G. C.; McClellan, A. L. "The Hydrogen Bond," Freeman and Co., San Francisco, 1960, pp. 8-9.

7. Huggins, M. L. J. Org. Chem. 1936, 1, 435.

8. Hendricks, A.: Wulf, B.; Liddle, D. J. Am. Chem. Soc. 1936, 58, 548.

9. Pourahmady, N.; Eisenbraun, E. J. J. Org. Chem. 1982 (In press).

10. Fieser, L. F.; Novello, G. F. J. Am. Chem. Soc. 1942, 64, 802.

11. Scribner, J. D.; Miller, J. A. J. Chem. Soc. 1965, 5377.

12. Brown, H. C.; Kim, S. C.; Krishnamurthy, S. J. Org. Chem. 1980, 45, 1.

13. Pourahmady, N.; Ph.D. Thesis, Department of Chemistry, Oklahoma State University, 1981.

14. Barton, D. M. R. J. Chem. Soc. 1953, 1027.

15. Nyburg, S. C. "X-Ray Analysis of Organic Structure," Academic Press, 1961, pp. 234-235.

16 International Tables for X-ray Crystallography (1962), Vol. III, p. 276, Birmingham: Kynoch Press.

17. Gordon, M.; Grover, S. H.; Stothers, J. B. Can. J. Chem. 1973, 51, 2092.

18. (a) Peters, J. A.; Van der Toorn, J. M.; Van Bekkum, H. Tetrahedron 1975, 31, 2272; (b) Peters, J. A.; Baas, J. M. A.; Van der

Graaf, B.; Van Der Toorn, J. M.; Van Bekkum, H. Tetrahderon 1978, 34, 3313.

19. Phek, T.; Lippmaa, E. Org. Magn, Reson. 1971, 3, 679.

20. Wehrli, F. W.; Wirthlin, T. "Interpretation of Carbon-13 NMR Spectra," Heyden, Philadelphia, 1978, pp. 22-23.

21. Levy, G. C.; Lichter, R. L.; Nelson, G. L. "Carbon-13 Nuclear Magnetic Resonance Spectroscopy," (2nd Ed.), Wiley-Interscience, New York, 1980, pp. 50-101.

22. Pavia, D. L.; Lampman, G. M.; Kriz, G. S. Jr., "Introduction to Spectroscopy," W. B. Saunders Company, 1979, p. 47.

23. MULTAN 80 Main, P.; Fiske, S. J.; Hull, S. E.; Lessinger, L.; Germain, G.; Declera, J. P.; Wolfson, M. M., University of York, York, England, 1980.

APPENDIX

TABLE I.

A COMPARISON OF OBSERVED AND CALCULATED STRUCTURE FACTORS

-17,1,L		
0	69	83
1	20	47
2	50	43
3	20	59
4	18	1
5	20	16
6	20	13
7	50	42
8	59	59
9	30	18
10	59	46
11	96	91
12	64	55
13	20	9
14	42	12
15	57	47
16	64	32
17	72	76
18	22	23
19	62	31
20	42	9

-17,3,L		
0	42	50
1	50	43
2	94	92
3	35	15
4	50	43
5	20	14
6	79	62
7	54	30
8	94	85
9	20	51
10	45	77
11	20	47
12	20	5
13	57	43
14	20	9
15	47	45
16	40	16
17	47	38
18	57	51
19	22	12
20	57	41

-17,5,L		
0	22	42
1	22	23
2	32	38
3	22	17
4	22	24
5	22	29
6	22	3
7	32	30
8	72	78
9	59	15
10	22	21
11	52	25
12	22	8
13	22	18
14	62	61
15	54	34
16	32	40
17	72	63
18	22	18
19	22	11
20	22	5

-17,7,L		
0	22	5
1	25	7
2	35	2
3	35	6
4	22	4
5	64	21
6	59	5
7	22	8
8	22	14
9	22	26
10	25	3
11	22	17
12	22	8
13	22	25
14	25	12
15	25	25
16	62	25
17	25	14
18	25	7
19	35	1
20	57	9

-16,0,L		
0	18	12
2	118	108
4	47	56
6	131	142
8	121	117
10	86	83
12	168	161
14	266	281
16	74	83
18	37	17
20	20	5

-16,2,L		
0	86	81
1	74	83.
2	18	13
3	25	12
4	42	4
5	18	12
6	30	3
7	91	84
8	59	50
9	57	44
10	18	45
11	64	53
12	32	20
13	47	41
14	50	51
15	116	110
16	50	62
17	64	54
18	27	23
19	20	48
20	20	1

-16,4,L		
0	76	67
1	32	5
2	37	21
3	20	3
4	20	13
5	74	67
6	50	44
7	37	57
8	61	88
9	35	42
10	20	2
11	30	32
12	40	31
13	20	8
14	45	3
15	20	3
16	40	5
17	20	26
18	42	14
19	20	3
20	20	20

-16,6,L		
0	37	3
1	96	90
2	99	84
3	52	36
4	20	21
5	20	1
6	37	25
7	47	25
8	20	29
9	20	29
10	20	3
11	50	36
12	52	60
13	54	17
14	118	102
15	42	34
16	50	37
17	32	21
18	22	23
19	35	19
20	30	11

-16,8,L		
0	42	4
1	22	4
2	22	6
3	52	6
4	22	1
5	22	3
6	22	4
7	22	9
8	67	16
9	22	9
10	22	9
11	54	2
12	22	3
13	47	7
14	50	37
15	22	7
16	25	41
17	22	5
18	22	10
19	22	2
20	22	11

-15,1,L		
0	156	165
1	212	214
2	16	36
3	64	70
4	178	162
5	67	64
6	42	46

-15,1,L		
7	18	6
8	74	70
9	42	8
10	141	160
11	121	119
12	81	73
13	52	23
14	279	271
15	84	77
16	111	105
17	40	44
18	47	53
19	59	43
20	57	52

-15,3,L		
0	158	149
1	116	121
2	74	47
3	18	24
4	45	26
5	18	24
6	67	51
7	47	36
8	136	138
9	40	21
10	18	1
11	32	34
12	32	19
13	16	27
14	20	25
15	20	15
16	89	79
17	20	12
18	91	88
19	20	15
20	20	15

-15,5,L		
0	20	39
1	50	64
2	20	17
3	54	47
4	57	51
5	47	29
6	32	8
7	20	8
8	20	22
9	20	26
10	27	62
11	20	7
12	57	35
13	37	12
14	52	12
15	20	16
16	52	1
17	45	31
18	20	5
19	40	25
20	54	18

-15,7,L		
0	54	45
1	59	35
2	35	3
3	30	2
4	42	1
5	22	40
6	30	43
7	20	14
8	22	1
9	42	36
10	52	39
11	22	26
12	22	39
13	22	13
14	67	52
15	47	39
16	37	59
17	22	23
18	22	35
19	22	15
20	22	13

-14,0,L		
0	95	93
2	30	13
4	116	112
6	18	16
8	94	104
10	108	99
12	52	52
14	173	169
16	101	99
18	150	154
20	27	35

-14,2,L		
0	18	3
1	158	161
2	18	13
3	37	48
4	57	50
5	35	19
6	32	42
7	62	58
8	202	213
9	96	86
10	18	18
11	232	227
12	50	35
13	202	193
14	52	52
15	18	20
16	52	52
17	52	29
18	35	15
19	79	73
20	64	73

-14,4,L		
0	207	219
1	72	83
2	18	5
3	27	18
4	59	54
5	59	64
6	42	50
7	32	16
8	207	216
9	96	91
10	131	157
11	18	31
12	108	112
13	57	96
14	116	98
15	40	63

TABLE I. (Continued)

16	99	100
17	20	1
18	62	47
19	20	2
20	20	45

-14,6,L

0	59	66
1	20	34
2	47	25
3	27	10
4	20	8
5	20	9
6	32	6
7	67	41
8	54	23
9	20	21
10	35	35
11	20	9
12	42	24
13	20	8
14	22	1
15	20	12
16	52	4
17	32	21
18	22	8
19	35	20
20	22	3

-14,8,L

0	22	27
1	22	6
2	22	12
3	22	31
4	22	38
5	47	5
6	22	27
7	22	6
8	22	2
9	22	20
10	22	9
11	67	31
12	22	4
13	32	40
14	59	42
15	40	20
16	22	10
17	25	18
18	52	13
19	47	1
20	45	4

-13,1,L

0	116	118
1	18	5
2	133	133
3	30	2
4	232	220
5	47	36
6	45	24
7	18	0
8	126	122
9	104	89
10	57	68
11	18	1
12	108	92
13	108	129
14	316	326
15	40	40
16	18	14
17	106	104
18	18	7
19	59	41
20	32	25

-13,3,L

0	45	23
1	168	172
2	350	356
3	96	121
4	141	149
5	101	86
6	138	129
7	35	30
8	155	149
9	32	22
10	47	9
11	85	83
12	168	188
13	18	22
14	54	28
15	72	64
16	111	101
17	59	62
18	20	7
19	72	62
20	54	54

-13,5,L

0	20	34
1	35	32
2	18	17
3	42	22
4	20	23
5	64	56
6	18	8
7	76	68
8	104	110
9	20	11
10	72	56
11	52	17
12	35	29
13	57	56
14	69	55
15	27	3
16	47	22
17	20	26
18	20	19
19	20	2
20	57	31

-13,7,L

0	22	31
1	81	79
2	133	125
3	67	53
4	20	9
5	40	12
6	69	34
7	20	35
8	20	2
9	62	56
10	20	6
11	40	37
12	32	39
13	59	9
14	91	68
15	74	15
16	45	48
17	22	24
18	52	35
19	22	9
20	64	9

-12,0,L

0	69	80
2	269	260
4	158	163
6	215	203
8	111	113
10	116	119
12	197	190
14	316	315
16	173	175
18	311	312
20	165	158

-12,2,L

0	126	121
1	454	456
2	79	65
3	116	98
4	197	191
5	18	17
6	121	128
7	136	128
8	111	104
9	296	301
10	76	72
11	155	143
12	72	59
13	86	96
14	237	243
15	18	22
16	47	56
17	18	2
18	32	25
19	27	12
20	64	64

-12,4,L

0	45	59
1	18	26
2	32	13
3	108	115
4	192	192
5	111	117
6	254	249
7	54	42
8	42	1
9	86	97
10	89	98
11	45	8
12	76	71
13	72	88
14	108	117
15	145	131
16	84	78
17	20	6
18	126	109
19	59	42
20	20	5

-12,6,L

0	50	17
1	32	42
2	50	47
3	111	94
4	54	47
5	42	5
6	47	59
7	20	35
8	30	12
9	47	48
10	69	54
11	20	42
12	57	62
13	101	75
14	54	45
15	59	37
16	79	88
17	57	7
18	42	55
19	20	31
20	22	17

-12,8,L

0	67	73
1	59	59
2	40	28
3	22	4
4	32	19
5	69	37
6	69	59
7	37	8
8	50	33
9	74	54
10	22	39
11	22	19
12	22	4
13	22	3
14	22	40
15	22	22
16	62	69
17	22	15
18	69	44
19	52	2
20	45	3

-11,1,L

0	84	74
1	50	41
2	392	382
3	111	97
4	156	161
5	123	119
6	276	278
7	50	29
8	52	43
9	15	44
10	104	119
11	197	197
12	244	252
14	89	78
15	133	154
16	207	218
17	37	22
18	365	367
19	101	102
20	79	76
	138	136

-11,3,L

0	76	68
1	35	68
2	18	3
3	66	77
4	50	73
5	96	94
6	121	113
7	121	124
8	279	283
9	50	23
10	37	44
11	18	7
12	86	89
13	138	135
14	18	22
15	18	12
16	40	35
17	76	61
18	99	90
19	74	82
20	35	40

-11,5,L

0	205	218
1	27	36
2	81	81
3	64	78
4	57	64
5	94	79
6	72	69
7	86	93
8	136	140
9	91	92
10	113	111
11	72	76
12	160	172
13	165	182
14	57	56
15	104	114
16	40	40
17	54	36
18	20	14
19	20	28
20	20	38

-11,7,L

0	54	40
1	50	47
2	20	18
3	32	7
4	20	3
5	20	8
6	30	31
7	59	65
8	20	4
9	20	0
10	20	20
11	20	32
12	69	60
13	47	6
14	59	69
15	22	14
16	77	43
17	37	22
18	22	3
19	22	15
20	22	20

-10,0,L

0	42	52
2	831	845
4	254	249
6	663	654
8	91	80
10	89	75
12	333	326
14	446	457
16	505	515
18	108	98
20	18	22

-10,2,L

0	222	225
1	264	260
2	111	110
3	15	14
4	279	269
5	444	445
6	141	150
7	99	96
8	72	70
9	15	8
10	212	216
11	249	251

TABLE I. (Continued)

12	99	87
13	205	213
14	121	124
15	288	284
16	96	94
17	313	305
18	69	87
19	18	8
20	20	50

-10,4,L

0	333	340
1	350	345
2	471	472
3	266	282
4	202	203
5	99	87
6	101	102
7	35	13
8	116	120
9	18	16
10	195	187
11	205	202
12	232	220
13	175	171
14	18	8
15	79	68
16	200	180
17	20	13
18	42	51
19	20	53
20	20	0

-10,6,L

0	18	27
1	89	70
2	79	69
3	40	10
4	42	22
5	42	63
6	35	43
7	47	47
8	47	39
9	40	35
10	20	52
11	76	66
12	20	5
13	99	75
14	67	53
15	20	37
16	101	95
17	118	125
18	20	28
19	20	20
20	67	50

-10,8,L

0	62	63
1	67	73
2	94	91
3	42	25
4	62	6
5	20	36
6	22	35
7	57	45
8	20	19
9	22	13
10	22	19
11	69	46
12	22	14
13	20	0
14	37	13
15	47	45
16	47	23
17	50	50
18	35	26
19	14	19
20	69	6

-9,1,L

0	414	399
1	22	7
2	619	618
3	335	329
4	81	75
5	136	131
6	313	306
7	50	45
8	79	69
9	254	265
10	222	208
11	128	125
12	178	180
13	293	305
14	190	192
15	18	4
16	116	126
17	27	44
18	259	264
19	170	173
20	163	159

-9,3,L

0	51	69
1	108	113
2	227	227
3	37	20
4	150	144
5	50	35
6	50	35
7	168	169
8	42	50
9	18	24
10	281	292
11	74	74
12	123	136
13	64	54
14	143	145
15	111	116
16	259	275
17	18	19
18	76	75
19	57	64
20	67	36

-9,5,L

0	84	72
1	59	47
2	50	68
3	113	107
4	148	160
5	141	128
6	165	169
7	101	88
8	18	28
9	202	196
10	52	52
11	47	53
12	101	106
13	200	179
14	133	124
15	72	48
16	20	34
17	76	72
18	54	39
19	59	37
20	20	5

-9,7,L

0	20	5
1	20	24
2	74	52
3	20	11
4	47	38
5	59	6
6	72	55
7	20	1
8	72	51
9	20	1
10	20	0
11	20	4
12	20	9
13	20	5
14	50	32
15	22	35
16	59	49
17	22	28
18	89	89
19	22	50
20	22	22

-8,0,L

0	461	464
2	801	790
4	920	931
6	412	403
8	47	54
10	259	253
12	350	357
14	185	180
16	18	10
18	185	185
20	85	89

-8,2,L

0	165	157
1	631	646
2	15	2
3	434	438
4	121	105
5	256	246
6	370	359
7	50	37
8	15	9
9	486	493
10	27	29
11	192	182
12	40	40
13	133	148
14	148	156
15	89	81
16	360	370
17	18	21
18	64	74
19	94	97
20	27	16

-8,4,L

0	113	113
1	69	70
2	94	86
3	27	28
4	291	290
5	99	82
6	54	53
7	18	3
8	84	77
9	123	137
10	52	49
11	72	61
12	143	148
13	104	83
14	215	202
15	20	16
16	222	232
17	50	40
18	79	93
19	20	21
20	45	38

-8,6,L

0	180	178
1	141	142
2	190	191
3	42	54
4	84	88
5	62	64
6	50	28
7	50	54
8	104	106
9	200	208
10	20	60
11	37	45
12	192	189
13	89	81
14	20	6
15	20	47
16	20	33
17	20	36
18	64	61
19	59	65
20	72	50

-8,8,L

0	22	32
1	22	35
2	40	0
3	20	11
4	47	32
5	30	9
6	50	20
7	32	39
8	69	50
9	37	55
10	22	27
11	20	17
12	37	18
13	22	11
14	59	28
15	35	3
16	42	27
17	22	3
18	47	7
19	54	20
20	22	1

-7,1,L

0	358	355
1	256	246
2	335	346
3	340	333
4	13	18
5	853	878
6	192	186
7	182	180
8	325	325
9	353	355
10	306	298
11	345	342
12	76	92
13	255	255
14	94	90
15	402	403
16	187	195
17	143	166
18	91	82
19	35	24
20	64	80

-7,3,L

0	414	412
1	62	74
2	256	247
3	15	9
4	99	90
5	304	304
6	15	12
7	59	66
8	74	92
9	227	218
10	254	256
11	131	112
12	111	88
13	47	45
14	150	157
15	190	203
16	145	160
17	153	163
18	76	81
19	27	49
20	148	167

-7,5,L

0	284	287
1	69	62
2	279	276
3	145	147
4	86	84
5	205	210
6	64	78
7	47	43
8	67	77
9	86	85
10	148	160
11	79	91
12	121	123
13	133	141
14	20	17
15	148	134
16	20	17
17	128	117
18	20	18
19	20	6
20	20	21

-7,7,L

0	20	43
1	76	83
2	57	20
3	18	4
4	35	2
5	45	31
6	50	78
7	50	8
8	54	25
9	20	10
10	35	30
11	45	3
12	59	62
13	37	36
14	20	2
15	20	28
16	69	70
17	72	65
18	74	41
19	22	1
20	74	71

TABLE I. (Continued)

-6,0,L

0	735	724
2	705	712
4	648	645
6	13	0
8	910	898
10	136	132
12	254	252
14	219	220
16	168	170
18	18	1
20	59	84

-6,2,-

0	153	146
1	530	522
2	222	228
3	131	121
4	284	281
5	266	252
6	318	304
7	355	335
8	143	154
9	91	104
10	288	280
11	375	377
12	15	4
13	195	181
14	86	90
15	121	130
16	360	375
17	215	209
18	94	91
19	269	272
20	84	63

-6,4,L

0	158	153
1	133	133
2	274	272
3	15	1
4	217	220
5	175	173
6	182	170
7	35	8
8	150	148
9	138	144
10	276	272
11	30	49
12	89	94
13	81	82
14	67	77
15	42	25
16	286	301
17	18	19
18	18	39
19	37	9
20	37	27

-6,6,L

0	148	152
1	136	133
2	62	72
3	72	78
4	84	90
5	45	50
6	94	110
7	173	165
8	52	68
9	50	8
10	45	44
11	111	107
12	18	4
13	45	18
14	59	56
15	64	54
16	40	59
17	121	117
18	20	4
19	37	34
20	22	30

-6,8,L

0	35	21
1	54	24
2	59	53
3	20	27
4	20	7
5	30	5
6	20	16
7	50	42
8	20	14
9	20	13
10	59	29
11	20	23
12	20	9
13	30	13
14	20	15
15	52	14
16	47	31
17	20	18
18	22	52
19	42	38
20	40	10

-5,1,L

0	200	197
1	1326	1338
2	173	167
3	335	343
4	13	16
5	110	109
6	72	55
7	94	93
8	13	12
9	545	537
10	192	191
11	158	152
12	15	42
13	254	247
14	47	53
15	150	151
16	104	95
17	18	14
18	40	56
19	89	93
20	37	26

-5,3,L

0	353	355
1	259	264
2	104	104
3	84	95
4	565	567
5	72	63
6	375	344
7	173	162
8	148	142
9	202	183
10	293	288
11	15	20
12	18	4
13	89	94
14	419	431
15	42	13
16	680	688
17	27	25
18	18	16
19	18	4
20	42	47

-5,5,L

0	195	190
1	464	462
2	217	208
3	32	18
4	145	149
5	45	19
6	30	28
7	45	37
8	74	67
9	104	109
10	45	11
11	96	97
12	205	214
13	74	67
14	138	141
15	145	141
16	123	135
17	20	28
18	72	39
19	18	6
20	27	15

-5,7,L

0	123	121
1	64	62
2	116	116
3	79	87
4	57	73
5	47	3
6	57	4
7	42	55
8	18	8
9	20	43
10	20	3
11	20	25
12	54	56
13	45	32
14	30	16
15	20	11
16	20	28
17	37	12
18	42	22
19	52	41
20	22	19

-4,0,L

0	1699	1687
2	461	463
4	599	591
6	885	899
8	1181	1175
10	915	886
12	205	181
14	377	379
16	74	78
18	106	101
20	62	61

-4,2,L

0	30	43
1	663	666
2	360	349
3	165	157
4	168	178
5	54	58
6	328	312
7	96	86
8	173	173
9	621	624
10	72	82
11	170	156
12	141	145
13	207	195
14	180	184
15	89	87
16	141	135
17	138	129
18	18	21
19	18	21
20	18	27

-4,4,L

0	136	127
1	25	19
2	461	462
3	52	43
4	145	145
5	131	132
6	301	288
7	106	87
8	165	168
9	32	67
10	84	98
11	42	23
12	99	105
13	86	63
14	303	303
15	18	9
16	232	225
17	95	103
18	113	119
19	111	113
20	18	24

-4,6,L

0	330	338
1	155	154
2	163	157
3	18	15
4	37	17
5	32	24
6	62	58
7	50	37
8	72	51
9	190	199
10	180	185
11	72	62
12	74	69
13	18	9
14	20	2
15	79	71
16	42	45
17	59	46
18	20	19
19	20	44
20	54	36

-4,8,L

0	113	120
1	32	9
2	32	9
3	42	0
4	40	31
5	20	18
6	20	14
7	30	32
8	42	22
9	69	72
10	59	64
11	47	13
12	86	60
13	20	29
14	20	6
15	22	13
16	42	2
17	47	12
18	54	45
19	22	13
20	54	9

-3,1,L

1	826	846
2	565	568
3	22	1
4	414	413
5	136	131
6	121	130
7	288	291
8	358	340
9	444	430
10	587	584
11	215	202
12	540	527
13	205	197
14	175	172
15	210	204
16	67	70
17	57	55
18	136	131
19	18	21
20	37	52

-3,3,L

0	656	643
1	431	422
2	323	308
3	293	299
4	444	443
5	57	41
6	86	92
7	323	309
8	417	415
9	15	11
10	155	157
11	42	10
12	121	104
13	138	147
14	18	21
15	54	51
16	247	249
17	136	124
18	57	58
19	76	82
20	45	40

-3,5,L

0	254	261
1	18	11
2	18	13
3	74	62
4	166	176
5	32	20
6	18	4
7	106	99
8	18	25
9	18	0
10	190	191
11	45	3
12	57	57
13	101	93
14	18	34
15	76	77

TABLE I. (Continued)

16	18	51
17	20	7
18	18	1
19	32	3
20	40	26

-3,7,L

0	261	266
1	79	76
2	62	55
3	35	5
4	61	67
5	47	18
6	10	9
7	18	19
8	67	45
9	20	22
10	66	111
11	54	65
12	94	88
13	64	52
14	52	28
15	20	21
16	40	9
17	64	38
18	47	50
19	40	3
20	20	10

-2,0,L

0	661	657
6	173	176
8	1117	1108
10	345	338
12	641	635
14	350	335
16	377	390
18	18	9
20	57	56

-2,2,L

0	819	813
1	1235	1203
2	187	188
3	464	467
4	333	334
5	113	115
6	431	432
7	626	619
8	390	381
9	74	65
10	392	402
11	72	63
12	40	54
13	455	473
14	200	206
15	121	112
16	422	434
17	18	52
18	95	97
19	67	54
20	32	27

-2,4,L

0	301	308
1	94	92
2	116	126
3	269	275
4	461	455
5	116	104
6	404	387
7	165	171
8	370	381
9	113	116
10	131	135
11	18	22
12	62	60
13	37	44
14	212	213
15	20	10
16	515	516
17	79	74
18	145	172
19	18	4
20	30	54

-2,6,L

0	89	84
1	345	334
2	202	196
3	215	215
4	69	59
5	35	34
6	54	9
7	321	316
8	180	189
9	18	11
10	18	35
11	106	90
12	121	127
13	158	150
14	45	15
15	20	31
16	20	3
17	67	58
18	20	43
19	30	28
20	47	14

-2,8,L

0	32	20
1	20	29
2	59	60
3	20	11
4	32	9
5	20	28
6	20	22
7	37	47
8	74	76
9	20	11
10	59	46
11	20	23
12	20	3
13	40	4
14	52	38
15	22	19
16	32	31
17	22	38
18	42	17
19	22	4
20	47	16

1,5,L

13	50	37
14	40	40

-1,1,L

1	555	582
2	550	563
3	414	421
4	747	781
5	229	232
6	619	616
7	136	124
8	126	142
9	409	414
10	390	383
11	646	623
12	264	245
13	106	116
14	266	255
15	91	98
16	274	272
17	76	83
18	145	135
19	18	39
20	40	19

-1,3,L

0	279	275
1	382	386
2	168	164
3	187	181
4	170	148
5	126	126
6	779	770
7	37	42
8	15	10
9	59	48
10	165	165
11	30	30
12	27	15
13	89	106
14	441	454
15	45	67
16	340	351
17	113	115
18	187	181
19	50	60
20	18	31

-1,5,L

0	232	234
1	200	205
2	330	326
3	301	291
4	67	46
5	57	37
6	67	53
7	222	213
8	27	47
9	217	218
10	148	148
11	197	204
12	27	47
13	72	98
14	101	113
15	101	95
16	141	137
17	106	104
18	106	105
19	20	5
20	20	26

-1,7,L

0	131	143
1	20	34
2	69	81
3	18	22
4	65	67
5	18	4
6	32	39
7	20	0
8	20	38
9	57	11
10	30	24
11	20	15
12	32	26
13	47	4
14	20	15
15	37	49
16	20	41
17	69	63
18	22	36
19	22	34
20	22	16

0,0,L

2	338	358
4	454	457
6	1065	1066
8	95	105
10	1896	1856
12	730	703
14	399	400
16	35	34
18	259	265
20	131	115

0,2,L

0	390	391
1	831	841
2	30	21
3	372	371
4	328	316
5	865	864
6	20	3
7	390	393
8	180	194
9	335	341
10	279	285
11	266	276
12	96	99
13	15	33
14	301	312
15	577	587
16	20	32
17	377	369
18	18	4
19	91	105
20	18	18

0,4,L

0	732	746
1	42	11
2	37	42
3	350	355
4	145	145
5	306	302
6	276	280
7	35	23
8	15	24
9	153	147
10	74	79
11	40	28
12	291	299
13	303	310
14	308	321
15	234	226
16	50	35
17	126	129
18	123	129
19	64	67
20	104	98

0,6,L

0	619	612
1	145	150
2	108	112
3	18	32
4	64	79
5	360	371
6	50	54
7	18	7
8	18	3
9	64	80
10	205	216
11	86	85
12	123	118
13	20	30
14	32	52
15	210	210
16	45	19
17	74	63
18	45	40
19	20	3
20	45	24

0,8,L

0	200	211
1	121	94
2	145	143
3	20	0
4	20	27
5	91	81
6	81	78
7	32	35
8	20	6
9	67	60
10	59	94
11	40	19
12	141	125
13	22	23
14	52	37
15	54	62
16	22	43
17	40	43
18	64	42
19	35	26
20	22	18

1,1,L

1	335	347
2	27	49
3	1640	1650
4	1627	1667
5	939	940
6	971	979
7	360	352
8	567	552
9	81	69
10	308	304
11	121	118
12	18	53
13	22	102
14	25	204
15	25	180
16	25	97
17	192	205
18	22	83
19	18	21
20	18	14

1,3,L

0	276	275
1	537	538
2	165	146
3	247	228
4	572	567
5	301	288
6	577	560
7	54	44
8	94	104

TABLE I. (Continued)

9	138	149
10	15	15
11	150	151
12	239	242
13	57	48
14	64	55
15	20	38
16	567	560
17	111	121
18	113	121
19	18	11
20	108	103

1,5,L

0	234	233
1	76	65
2	116	114
3	131	138
4	165	186
5	170	185
6	185	171
7	215	214
8	47	65
9	148	153
10	79	76
11	18	37
12	37	5
15	76	48
16	113	115
17	52	54
18	96	110
19	81	72
20	116	99

1,7,L

0	148	143
1	50	39
2	59	49
3	165	153
4	108	111
5	20	34
6	18	10
7	74	62
8	42	31
9	67	43
10	101	92
11	20	4
12	20	28
13	94	94
14	37	2
15	32	43
16	22	2
17	20	3
18	45	64
19	35	39
20	22	1

2,0,L

0	668	666
2	1053	1052
4	466	476
6	32	28
8	592	576
10	47	46
12	47	40
14	210	213
16	271	265
18	18	2
20	18	24

2,2,L

0	809	813
1	853	856
2	501	500
3	101	96
4	505	500
5	1161	1164
6	555	555
10	234	235
11	611	629
12	234	231
17	84	129
18	20	20
19	20	73
20	25	94

2,4,L

0	316	307
1	429	438
2	316	317
3	227	229
4	15	46
5	15	26
6	328	333
7	123	119
8	212	205
9	54	22
10	340	359
11	207	228
12	247	250
13	118	118
14	69	74
15	18	51
16	113	116
17	57	59
18	175	185
19	84	92
20	153	145

2,6,L

0	84	84
1	18	28
2	227	238
3	254	269
4	45	58
5	27	5
6	18	22
7	168	171
8	45	30
9	113	116
10	74	67
11	79	81
12	18	25
13	96	103
14	42	50
15	20	17
16	81	63
17	45	8
18	99	110
19	47	29
20	20	3

2,8,L

0	20	20
1	67	48
2	79	85
3	20	8
4	153	148
5	20	42
6	74	54
7	96	83
8	76	75
10	101	97
11	57	46
12	20	38
13	22	14
14	123	109
15	69	46
16	94	77
17	22	35
18	42	17
19	47	3
20	22	25

3,1,L

1	1013	1051
2	219	225
3	570	587
4	483	479
9	210	207
10	224	222
11	133	133
12	264	265
13	15	11
14	27	50
15	328	333
16	20	16
17	362	354
18	192	198
19	76	77
20	18	22

3,3,L

0	644	644
1	375	360
2	67	50
3	259	253
4	791	774
5	249	236
6	57	45
7	182	173
8	104	96
9	343	337
10	74	47
11	123	130
12	288	292
13	150	161
14	619	627
15	143	159
16	18	15
17	94	101
18	69	72
19	128	131
20	76	64

3,5,L

0	259	261
1	27	24
2	155	177
3	160	146
4	57	72
5	180	179
6	18	5
7	153	145
8	18	23
9	57	58
10	67	63
11	54	44
12	18	34
13	62	54
14	37	60
15	175	175
16	30	12
17	165	162
18	74	73
19	37	23
20	96	86

3,7,L

0	251	266
1	20	7
2	99	119
3	61	69
4	18	26
5	45	70
6	18	21
7	72	71
8	45	15
9	50	20
10	20	48
11	40	28
12	138	129
13	91	86
14	20	19
15	20	21
16	40	25
17	35	32
18	22	10
19	22	17
20	22	15

4,0,L

0	1682	1686
2	1908	1885
4	79	79
6	1016	1019
8	404	395
10	118	128
12	505	513
14	185	192
16	76	59
18	150	156
20	113	104

4,2,L

0	13	43
1	261	284
2	865	860
3	1253	1272
4	45	52
5	1292	1288
6	372	367
7	431	412
8	215	223
9	35	38
10	27	38
11	180	191
12	353	342
13	219	217
14	76	65
15	249	255
16	200	199
17	456	451
18	96	119
19	18	24
20	67	77

4,4,L

0	131	128
1	59	61
2	143	146
3	229	219
4	143	139
5	15	16
6	35	57
7	293	289
8	187	192
9	35	13
10	18	34
11	25	27
12	32	1
13	32	31
14	57	57
15	25	23
16	18	50
17	202	207
18	99	96
19	20	4
20	145	136

4,6,L

0	325	338
1	143	146
2	143	147
3	25	1
4	18	9
5	118	144
6	106	96
7	108	101
8	42	14
9	52	38
10	59	71
11	57	58
12	94	91
13	20	32
14	47	16
15	20	1
16	20	21
17	136	147
18	116	114
19	22	50
20	79	59

4,8,L

0	111	120
1	20	33
2	153	148
3	84	94
4	67	61
5	72	55
6	72	89
7	22	26
8	47	75
9	22	5
10	52	51
11	62	26
12	106	101
13	22	31
14	22	35
15	62	29
16	22	46
17	50	22
18	67	47
19	22	1
20	22	11

5,1,L

0	207	196
1	1159	1185
2	306	302
3	582	579
4	791	783
5	579	577
6	86	82
7	145	138
8	318	309
9	50	45
10	195	197
11	496	493
12	192	192
13	18	7
14	385	388
15	178	185
16	150	167
17	57	72
18	234	244
19	81	80
20	35	49

TABLE I. (Continued)

5,3,L

0	345	355
1	227	225
2	478	463
3	131	123
4	15	15
5	89	95
6	325	320
7	178	193
8	57	55
9	141	143
10	123	124
11	192	200
12	239	224
13	30	26
14	47	24
15	42	11
16	47	61
17	18	36
18	59	51
19	57	45
20	62	38

5,5,L

0	185	189
1	143	156
2	18	2
3	79	89
4	18	18
5	150	133
6	64	75
7	67	66
8	79	63
9	118	126
10	35	42
11	64	77
12	118	129
13	18	16
14	18	17
15	96	91
16	20	8
17	79	64
18	175	169
19	91	90
20	54	64

5,7,L

0	131	121
1	69	60
2	20	12
3	50	50
4	84	83
5	89	64
6	96	94
7	32	39
8	20	9
9	20	26
10	86	91
11	64	60
12	20	4
13	20	25
14	94	93
15	74	59
16	104	92
17	22	7
18	84	62
19	22	4
20	37	2

6,0,L

0	735	726
2	145	134
4	661	657
6	72	84
8	468	455
10	419	422
12	259	256
14	25	6
16	306	311
18	449	458
20	20	5

6,2,L

0	143	147
1	747	749
2	483	483
3	434	441
4	360	356
5	15	19
6	74	48
7	106	91
8	217	217
9	195	207
10	54	45
11	202	212
12	84	86
13	42	18
14	187	193
15	86	94
16	86	96
17	37	29
18	123	125
19	45	28
20	47	43

6,4,L

0	153	154
1	133	140
2	256	250
3	185	195
4	121	127
5	279	269
6	54	52
7	165	164
8	67	64
9	101	122
10	178	177
11	32	3
12	37	34
13	30	10
14	89	90
15	18	56
16	30	74
17	67	64
18	57	31
19	40	44
20	52	44

6,6,L

0	138	152
1	123	119
2	18	40
3	30	36
4	62	49
5	18	2
6	18	33
7	72	74
8	18	29
9	67	84
10	18	20
11	32	43
12	57	47
13	57	63
14	20	2
15	20	27
16	74	74
17	20	14
18	111	108
19	20	22
20	47	9

6,8,L

0	30	21
1	35	46
2	20	41
3	20	30
4	192	168
5	20	5
6	86	83
7	47	25
8	20	32
9	20	0
10	54	52
11	20	31
12	22	24
13	22	9
14	91	86
15	37	13
16	89	90
17	22	11
18	57	16
19	42	16
20	22	10

7,1,L

0	350	355
1	37	14
2	385	376
3	259	241
4	62	63
5	367	353
6	104	104
7	178	173
8	35	14
9	237	243
10	32	45
11	106	104
12	207	204
13	18	29
14	104	92
15	42	47
16	96	100
17	195	192
18	50	6
19	20	9
20	20	6

7,3,L

0	402	412
1	145	148
2	488	497
3	25	35
4	101	105
5	251	250
6	180	184
7	67	80
8	94	85
9	121	135
10	158	157
11	101	101
12	180	197
13	27	26
14	101	105
15	18	26
16	54	12
17	18	4
18	64	35
19	20	13
20	72	82

7,5,L

0	281	287
1	79	83
2	170	165
3	116	115
4	40	23
5	111	118
6	59	58
7	128	112
8	74	83
9	57	61
10	18	35
11	47	44
12	40	50
13	64	30
14	30	49
15	40	6
16	84	64
17	138	142
18	32	2
19	52	6
20	61	78

7,7,L

0	52	43
1	27	18
2	62	65
3	57	44
4	89	97
5	62	26
6	72	59
7	20	27
8	20	41
9	20	1
10	20	17
11	42	10
12	54	60
13	20	18
14	64	45
15	54	10
16	22	12
17	42	5
18	22	27
19	47	11
20	22	9

8,0,L

0	471	464
2	42	43
4	308	316
6	552	539
8	414	408
10	197	200
12	35	34
14	158	146
16	219	213
18	293	298
20	42	49

8,2,L

0	165	157
1	611	620
2	293	306
3	375	373
4	116	118
5	187	192
6	126	127
7	202	199
8	141	144
9	185	174
10	150	163
11	348	353
12	89	86
13	27	25
14	18	16
15	62	46
16	50	45
17	89	90
18	101	95
19	47	48
20	20	0

8,4,L

0	106	113
1	160	173
2	202	214
3	18	9
4	126	121
5	165	159
6	72	58
7	35	46
8	61	97
9	40	35
10	37	38
11	96	110
12	113	141
13	40	8
14	94	87
15	121	123
16	99	79
17	64	57
18	32	29
19	54	36
20	42	41

8,6,L

0	175	178
0	173	178
1	106	114
2	67	62
3	52	31
4	54	35
5	47	39
6	118	112
7	30	21
8	47	46
9	47	29
10	20	3
11	18	25
12	72	62
13	20	8
14	57	21
15	20	9
16	57	38
17	20	7
18	84	89
19	22	5
20	22	41

8,8,L

0	40	32
1	74	55
2	74	72
3	37	25
4	42	23
5	20	30
6	108	93
7	37	9
8	57	64
9	22	19
10	59	23
11	57	31
12	22	26
13	50	0
14	50	11
15	22	23
16	67	49

TABLE I. (Continued)

17	22	9
18	54	31
19	22	7
20	22	16

9,1,L

0	409	399
1	412	400
2	247	253
3	237	230
4	217	219
5	50	70
6	340	339
7	50	66
8	227	222
9	15	14
10	18	18
11	104	89
12	52	41
13	64	41
14	18	40
15	18	21
16	288	296
17	20	39
18	256	248
19	20	30
20	20	58

9,3,L

0	67	69
1	101	101
2	254	257
3	182	192
4	42	55
5	79	90
6	47	27
7	25	1
8	30	1
9	18	26
10	18	30
11	18	12
12	72	74
13	54	65
14	101	120
15	32	9
16	69	52
17	37	18
18	20	4
19	42	12
20	35	22

9,5,L

0	69	72
1	242	263
2	35	43
3	62	38
4	37	23
5	27	17
6	18	3
7	35	15
8	62	39
9	18	1
10	40	62
11	64	79
12	35	36
13	37	25
14	20	1
15	27	32
16	40	8
17	20	5
18	108	101
19	42	2
20	22	23

9,7,L

0	20	5
1	67	51
2	20	43
3	20	28
4	91	87
5	59	52
6	74	92
7	20	11
8	40	49
9	20	4
10	20	14
11	50	26
12	40	22
13	20	14
14	37	37
15	57	6
16	96	96
17	22	11
18	91	71
19	22	7
20	22	25

10,0,L

0	15	52
2	340	347
4	370	381
6	62	65
8	126	110
10	57	47
12	72	59
14	47	18
16	269	264
18	81	85
20	62	67

10,2,L

0	227	224
1	318	328
2	335	355
3	380	384
4	207	202
5	15	6
6	37	6
7	64	65
8	15	12
9	153	150
10	47	45
11	86	93
12	108	113
13	108	111
14	72	76
15	121	118
16	52	15
17	18	10
18	30	41
19	50	52
20	52	0

10,4,L

0	323	340
1	141	132
2	375	375
3	67	79
4	62	66
5	18	7
6	72	83
7	54	53
8	99	115
9	84	93
10	111	109
11	64	75
12	76	95
13	72	81
14	59	46
15	20	1
16	40	26
17	20	25
18	20	49
19	42	4
20	52	27

10,6,L

0	18	27
1	18	4
2	18	46
3	79	66
4	62	84
5	20	7
6	30	37
7	45	42
8	37	35
9	89	71
10	54	42
11	20	32
12	40	11
13	20	26
14	54	34
15	69	66
16	96	105
17	45	7
18	22	35
19	35	2
20	50	2

10,8,L

0	67	63
1	72	73
2	59	62
3	57	31
4	89	86
5	37	8
6	57	48
7	22	25
8	54	41
9	59	2
10	50	48
11	72	44
12	47	40
13	22	13
14	74	45
15	22	4
16	47	34
17	22	12
18	22	8
19	22	16
20	22	1

11,1,L

0	76	74
1	101	103
2	15	14
3	170	174
4	64	67
5	150	152
6	136	143
7	89	84
8	69	87
9	96	82
10	32	32
11	76	87
12	30	12
13	37	24
14	20	14
15	72	56
16	35	10
17	67	32
18	37	75
19	108	107
20	52	15

11,3,L

0	74	68
1	148	154
2	730	748
3	173	182
4	178	179
5	18	35
6	18	5
7	111	102
8	32	25
9	30	15
10	57	59
11	37	45
12	219	241
13	30	13
14	101	103
15	20	41
16	59	8
17	57	49
18	30	3
19	37	48
20	37	44

11,5,L

0	202	218
1	18	8
2	143	168
3	27	51
4	37	58
5	47	54
6	40	5
7	69	49
8	18	5
9	18	21
10	47	14
11	52	43
12	64	66
13	20	14
14	20	30
15	20	21
16	22	51
17	22	15
18	59	37
19	74	46
20	22	26

11,7,L

0	45	40
1	57	35
2	20	25
3	45	29
4	35	16
5	76	54
6	20	22
9	35	23
10	22	22
11	22	38
12	42	1
13	22	10
14	22	17
15	54	49
16	22	13
17	59	8
18	22	25
19	22	13
20	22	4

12,0,L

0	76	80
2	301	303
4	153	151
6	175	166
8	390	386
10	18	39
12	47	1
14	118	120
16	131	145
18	247	244
20	22	50

12,2,L

0	113	121
1	264	266
2	185	199
3	138	146
4	153	167
5	170	166
6	18	22
7	40	25
8	121	124
9	168	178
10	18	39
11	57	60
12	116	100
13	40	57
14	72	59
15	76	72
16	20	26
17	20	17
18	47	58
19	54	21
20	20	39

12,4,L

0	52	58
1	76	92
2	59	35
3	45	30
4	20	52
5	113	107
6	76	93
7	40	39
8	27	46
9	18	2
10	20	7
11	20	35
12	54	56
13	20	13
14	57	55
15	20	36
16	47	36
17	20	9
18	20	27
19	59	16
20	20	26

12,6,L

0	20	17
1	20	9
2	20	14
3	47	30
4	20	18
5	20	7
6	76	66
7	59	27
8	84	92
9	20	31
10	20	10
11	42	27
12	20	30

TABLE I. (Continued)

12,6,L

13	50	47
14	57	40
15	22	10
16	22	37
17	22	9
18	96	91
19	22	0
20	22	17

12,8,L

0	79	73
1	22	49
2	50	53
3	57	30
4	22	20
5	22	2
6	59	61
7	22	7
8	67	68
9	40	53
10	52	23
11	22	8
12	22	14
13	22	10
14	22	0
15	37	10
16	22	32
17	22	0
18	40	28
19	37	4
20	25	17

13,1,L

0	104	118
1	148	156
2	141	152
3	296	298
4	18	23
5	45	46
6	27	42
7	18	38
8	170	171
9	18	25
10	47	62
11	42	52
12	99	92
13	118	117
14	20	22
15	113	92
16	96	104
17	20	30
18	94	92
19	54	44
20	20	12

13,3,L

0	27	23
1	18	20
2	192	198
3	170	163
4	155	156
5	59	73
6	57	12
7	18	34
8	18	55
9	18	39
10	18	11
11	18	20
12	18	17
13	54	40
14	52	50
15	20	23
16	45	11
17	20	10
18	57	48
19	52	27
20	22	37

13,5,L

0	20	34
1	32	3
2	86	110
3	96	89
4	84	56
5	91	84
6	74	90
7	20	13
8	81	67
9	45	2
10	20	4
11	47	10
12	52	0
13	22	54
14	20	6
15	104	90
16	121	96
17	40	26
18	22	63
19	45	13
20	22	14

13,7,L

0	30	31
1	108	99
2	40	53
3	62	22
4	20	38
5	20	2
6	72	69
7	22	16
8	20	7
9	42	1
10	40	33
11	22	39
12	22	45
13	22	16
14	45	18
15	45	14
16	64	55
17	22	1
18	22	17
19	22	3
20	22	6

14,0,L

0	104	90
2	32	5
4	136	127
6	256	252
8	69	64
10	18	6
12	30	5
14	50	58
16	180	177
17	22	0
18	20	2
20	22	31

14,2,L

0	50	3
1	269	267
2	392	407
3	182	172
4	54	54
5	42	45
6	18	29
7	180	179
8	69	70
9	18	3
10	18	0
11	18	17
12	106	108
13	52	23
14	42	45
15	20	47
16	20	16
17	67	73
18	22	14
19	111	96
20	64	4

14,4,L

0	205	219
1	113	121
2	301	321
3	32	18
4	76	94
5	18	7
6	20	22
7	42	33
8	32	11
9	52	48
10	20	30
11	52	12
12	91	93
13	20	20
14	20	45
15	20	7
16	20	3
17	22	17
18	22	14
19	37	47
20	57	22

14,6,L

0	62	66
1	118	109
2	47	44
3	20	13
4	20	19
5	67	63
6	79	76
7	40	43
8	20	14
9	76	32
10	22	18
11	47	56
12	22	12
13	22	1
14	40	8
15	50	59
16	74	73
17	22	26
18	59	5
19	50	16
20	42	4

14,8,L

0	22	27
1	22	35
2	22	29
3	45	7
4	69	49
5	40	1
6	22	28
7	54	34
8	22	19
9	22	12
10	22	10
11	22	3
12	22	5
13	22	12
14	22	23
15	22	5
16	40	18
17	62	15
18	22	5
19	22	21
20	37	7

15,1,L

0	168	165
1	40	21
2	59	70
3	35	2
4	18	14
5	121	109
6	72	58
7	18	27
8	192	203
9	20	5
10	69	66
11	18	23
12	18	4
13	20	19
14	42	66
15	20	35
16	37	15
17	22	12
18	113	82
19	79	61
20	22	10

15,3,L

0	143	148
1	18	10
2	121	129
3	108	101
4	84	89
5	30	47
6	18	30
7	18	11
8	67	41
9	20	28
10	20	27
11	20	10
12	52	32
13	20	13
14	20	5
15	20	17
16	40	24
17	20	6
18	22	19
19	64	10
20	22	27

15,5,L

0	20	39
1	74	63
2	20	22
3	20	32
4	50	61
5	128	114
6	50	9
7	42	19
8	20	36
9	20	1
10	20	17
11	37	23
12	40	20
13	47	44
14	59	41
15	22	31
16	22	4
17	22	8
18	52	48
19	59	26
20	57	18

15,7,L

0	35	45
1	54	35
2	45	36
3	47	35
4	20	2
5	22	2
6	22	30
7	22	3
8	79	46
9	22	11
10	69	11
11	22	9
12	22	8
13	67	5
14	22	24
15	22	9
16	22	9
17	22	1
18	57	16
19	45	14
20	22	12

16,0,L

0	18	12
2	148	154
4	141	155
6	50	51
8	155	144
10	18	18
12	86	76
14	104	103
16	50	41
18	45	57
20	22	5

16,2,L

0	79	81
1	96	97
2	59	43
3	138	136
4	32	12
5	45	27
6	101	101
7	81	70
8	20	14
9	18	11
10	20	11
11	35	50
12	62	26
13	20	36
14	42	22
15	67	29
16	59	50
17	45	14
18	22	28
19	57	1
20	54	48

16,4,L

0	67	67
1	40	25
2	20	35
3	37	37
4	69	62
5	86	104
6	111	100

TABLE I. (Continued)

7	40	47
8	20	6
9	42	13
10	40	17
11	54	28
12	45	18
13	62	31
14	52	10
15	64	50
16	86	74
17	47	35
18	69	42
19	22	3
20	22	46

16,6,L

0	20	3
1	64	69
2	20	45
3	20	27
4	20	19
5	20	11
6	45	48
7	22	3
8	45	38
9	52	37
10	20	1
11	45	21
12	47	12
13	22	2
14	22	5
15	22	13
16	45	36
17	22	7
18	74	27
19	54	16
20	45	15

16,8,L

0	22	4
1	54	19
2	42	18
3	50	6
4	22	7
5	22	5
6	22	18
7	22	3
8	22	6
9	40	6
10	22	10
11	52	14
12	22	1
13	47	4
14	22	3
15	40	1
16	57	1
17	22	7
18	22	4
19	35	6
20	57	17

17,1,L

0	91	83
1	40	52
2	99	100
3	76	79
4	76	50
5	72	84
6	106	129
7	20	30
8	20	43
9	35	9
10	30	49
11	45	35
12	20	3
13	20	6
14	20	16
15	45	37
16	62	77
17	22	4
18	22	4
19	22	30
20	22	5

17,3,L

0	18	50
1	35	20
2	121	127
3	20	3
4	74	82
5	20	33
6	20	23
7	20	42
8	32	43
9	20	5
10	64	42
11	20	29
12	20	18
13	20	21
14	57	40
15	22	30
16	22	4
17	22	11
18	54	5
19	22	2
20	22	57

17,5,L

0	20	42
1	20	57
2	20	3
3	81	71
4	37	40
5	20	33
6	69	58
7	54	26
8	52	33
9	20	20
10	20	11
11	20	14
12	22	11
13	45	24
14	22	5
15	22	26
16	22	44
17	37	12
18	40	29
19	22	13
20	47	18

17,7,L

0	20	5
1	20	8
2	37	18
3	22	9
4	32	54
5	22	12
6	54	34
7	40	6
8	22	7
9	22	7
10	45	1
11	22	16
12	22	4
13	22	10
14	37	16
15	22	4
16	35	23
17	22	6
18	35	13
19	22	13
20	22	2

18,0,L

0	74	65
2	67	47
4	116	116
6	91	96
8	47	20
10	50	21
12	32	22
14	101	52
16	22	40
18	22	0
20	22	13

18,2,L

0	136	109
1	45	59
2	76	85
3	35	37
4	128	115
5	20	11
6	37	27
7	20	14
8	20	47
9	20	29
10	37	28
11	20	4
12	20	0
13	20	3
14	22	46
15	22	6
16	35	13
17	22	0
18	30	0
19	59	39
20	22	5

18,4,L

0	20	46
1	59	0
2	40	33
3	20	35
4	20	54
5	54	48
6	42	42
7	20	22
8	20	25
9	20	7
10	20	17
11	20	13
12	50	2
13	35	12
14	35	17
15	22	14
16	22	11
17	42	10
18	22	11
19	22	10
20	45	10

18,6,L

0	37	9
1	20	21
2	40	26
3	35	34
4	20	28
5	50	13
6	20	20
7	32	26
8	22	0
9	20	12
10	22	16
11	22	3
12	22	4
13	22	4
14	22	26
15	22	3
16	22	15
17	22	9
18	37	0
19	25	11
20	22	13

18,8,L

0	37	32
1	22	6
2	54	41
3	22	6
4	22	23
5	22	8
6	22	5
7	22	12
8	37	3
9	22	3
10	22	7
11	22	1
12	22	9
13	40	5
14	50	8
15	22	7
16	22	2
17	25	2
18	25	7
19	42	13
20	45	6

19,1,L

0	37	29
1	64	51
2	20	9
3	89	59
4	64	70
5	20	30
6	47	22
7	20	14
8	40	5
9	30	21
10	45	29
11	20	12
12	45	4
13	20	15
14	54	51
15	20	12
16	20	9
17	22	16
18	22	3
19	22	12
20	47	29

19,3,L

0	91	95
1	47	42
2	74	55
3	35	8
4	20	10
5	37	12
6	72	79
7	20	14
8	47	25
9	20	0
10	22	35
11	47	30
12	20	12
13	22	19
14	35	10
15	54	4
16	22	39
17	22	0
18	22	23
19	22	3
20	74	44

19,5,L

0	20	26
1	20	32
2	20	1
3	67	59
4	22	12
5	22	17
6	20	21
7	22	4
8	42	37
9	20	3
10	22	22
11	22	8
12	67	9
13	35	21
14	45	1
15	40	3
16	22	17
17	22	9
18	22	23
19	22	2
20	52	23

19,7,L

0	22	5
1	47	1
2	57	13
3	22	4
4	22	7
5	22	0
6	22	6
7	22	7
8	22	8
9	22	10
10	40	1
11	64	1
12	22	0
13	22	6
14	52	11
15	22	3
16	25	7
17	25	11
18	25	2
19	25	13
20	25	11

TABLE II.

ATOMIC COORDINATES

Atom	x(σ(x))	y(σ(y))	z(σ(z))	U_{eq}*
C1	0.3584(1)	-0.0012(4)	0.2372(1)	41
C2	0.3096(2)	0.1309(5)	0.2604(1)	55
C3	0.3013(2)	0.1175(6)	0.3210(2)	70
C4	0.3414(2)	-0.0238(6)	0.3589(2)	69
C5	0.3890(2)	-0.1558(6)	0.3365(2)	68
C6	0.3970(2)	-0.1467(5)	0.2760(1)	51
C7	0.3704(1)	0.0029(4)	0.1720(1)	41
C8	0.3469(2)	0.1867(4)	0.1333(1)	49
C9	0.3952(2)	0.3688(5)	0.1603(2)	67
C10	0.4802(2)	0.3589(6)	0.1511(2)	74
C11	0.4837(3)	0.3433(6)	0.0832(2)	81
C12	0.4396(2)	0.1600(6)	0.0552(2)	69
C13	0.3542(2)	0.1547(5)	0.0656(1)	57
C14	0.3098(2)	-0.0260(5)	0.0377(1)	59
O1	0.3982(1)	-0.1400(3)	0.1507(1)	61
O2	0.2398(1)	-0.0509(4)	0.0530(1)	76
O3	0.3351(1)	-0.1357(4)	0.0018(1)	81

H1	0.285(2)	0.329(7)	0.469(2)	H91	0.394(2)	0.381(5)	0.207(2)
H2	0.282(2)	0.230(4)	0.235(1)	H92	0.369(2)	0.482(5)	0.139(1)
H3	0.271(2)	0.201(5)	0.338(1)	H101	0.508(2)	0.239(5)	0.171(1)
H4	0.335(2)	-.029(4)	0.402(1)	H102	0.506(2)	0.477(5)	0.167(1)
H5	0.413(2)	-.253(5)	0.360(1)	H111	0.537(2)	0.340(5)	0.076(1)
H6	0.430(1)	-.236(4)	0.259(1)	H112	0.457(2)	0.467(5)	0.060(2)
H8	0.289(2)	0.205(4)	0.135(1)	H121	0.436(2)	0.160(5)	0.012(2)
H13	0.325(2)	0.265(4)	0.044(1)	H122	0.467(2)	0.040(4)	0.074(1)

U_{eq} is 1/3 the trace of the diagonalized anisotropic matrix in units of $Å^2 \times 10^3$

TABLE III.

BOND ANGLES (°) AND DISTANCES (Å)

C1 - C2	1.387(4)	C1 - C2 - C3	120.1(3)
C1 - C6	1.386(4)	C2 - C3 - C4	120.6(4)
C2 - C3	1.380(5)	C3 - C4 - C5	119.8(4)
C3 - C4	1.365(6)	C4 - C5 - C6	120.3(4)
C4 - C5	1.364(6)	C5 - C6 - C1	120.7(3)
C5 - C6	1.374(5)	C2 - C1 - C6	118.4(2)
C1 - C7	1.498(3)	C6 - C1 - C7	118.2(2)
C7 - C8	1.518(4)	C2 - C1 - C7	123.4(2)
C7 - O1	1.212(3)	C1 - C7 - C8	119.2(2)
C8 - C9	1.537(4)	C1 - C7 - O1	119.9(2)
C8 - C13	1.544(3)	C8 - C7 - O1	120.9(2)
C9 - C10	1.508(5)	C7 - C8 - C9	111.8(2)
C10 - C11	1.520(6)	C7 - C8 - C13	111.8(2)
C11 - C12	1.519(6)	C9 - C8 - C13	110.6(3)
C12 - C13	1.523(5)	C8 - C9 - C10	111.8(3)
C13 - C14	1.506(4)	C9 - C10 - C11	111.2(3)
C14 - O2	1.317(4)	C10 - C11 - C12	110.4(4)
C14 - O3	1.226(4)	C11 - C12 - C13	111.9(3)
O2 - H1	1.00(4)	C12 - C13 - C8	113.9(2)
H1 - O3'	1.65(4)	C12 - C13 - C14	112.3(3)
		C8 - C13 - C14	112.5(2)
		C13 - C14 - O2	114.2(3)
		C13 - C14 - O3	122.9(3)
		O2 - C14 - O3	122.9(3)
		C14 - O2 - H1	107(2)
		O2 - H1 - O3'	173(4)

TABLE IV.

ANISOTROPIC THERMAL PARAMETERS FOR $C_{14}H_{16}O_3$

Atom	U11	U22	U33	U12	U13	U23
C1	44(1)	39(1)	41(2)	-3(1)	5(1)	-4(1)
C2	60(2)	54(2)	50(2)	10(2)	7(1)	-3(2)
C3	72(2)	76(2)	62(3)	18(2)	22(2)	-14(2)
C4	73(2)	93(3)	42(2)	5(2)	14(2)	-7(2)
C5	73(2)	80(2)	50(2)	19(2)	10(2)	12(2)
C6	54(2)	55(2)	44(2)	9(2)	12(1)	-2(2)
C7	47(1)	34(1)	43(2)	-6(1)	3(1)	-5(1)
C8	59(2)	38(2)	50(2)	0(1)	5(1)	2(1)
C9	93(3)	34(1)	74(3)	-11(2)	13(2)	0(2)
C10	94(3)	53(2)	74(3)	-31(2)	5(2)	-7(2)
C11	84(3)	72(3)	87(3)	-31(2)	20(2)	6(2)
C12	76(2)	76(3)	54(2)	-22(2)	13(2)	3(2)
C13	69(2)	55(2)	47(2)	-8(2)	1(2)	11(2)
C14	64(2)	73(2)	40(2)	-16(2)	0(1)	6(2)
01	95(2)	38(1)	50(1)	5(1)	23(1)	-3(1)
02	66(2)	92(2)	71(2)	-21(1)	8(1)	-13(1)
03	94(2)	98(2)	52(1)	-35(1)	22(1)	-20(1)

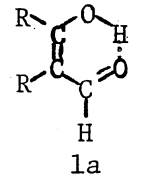
TABLE V.

ISOTROPIC THERMAL PARAMETERS FOR HYDROGEN ATOMS OF $C_{14}H_{16}O_3$

Isotropic Thermal Parameters for Hydrogen Atoms of $C_{14}H_{16}O_3$

H1	149(18)
H2	61(9)
H3	76(11)
H4	75(10)
H5	82(12)
H6	52(8)
H8	52(9)
H13	59(8)
H91	97(12)
H92	74(10)
H101	80(11)
H102	89(10)
H111	83(11)
H112	98(12)
H121	89(12)
H122	66(10)

Anisotropic thermal parameters of the form $\exp[-2\pi^2(U11h^2a^{*2}+U22k^2b^{*2}+U33l^2c^{*2}+2U12hka^*b^* + 2U13hla^*c^* + 2U23klb^*c^*)]$



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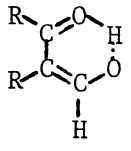

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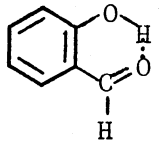

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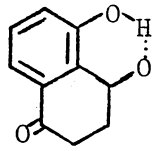

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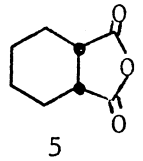

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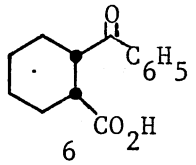

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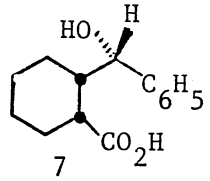

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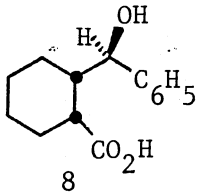

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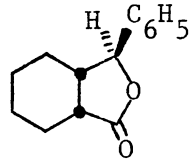

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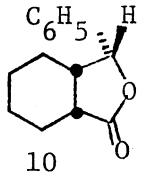

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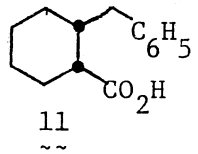

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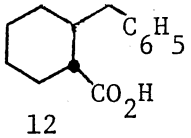

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Figure 1. Glossary of Structures

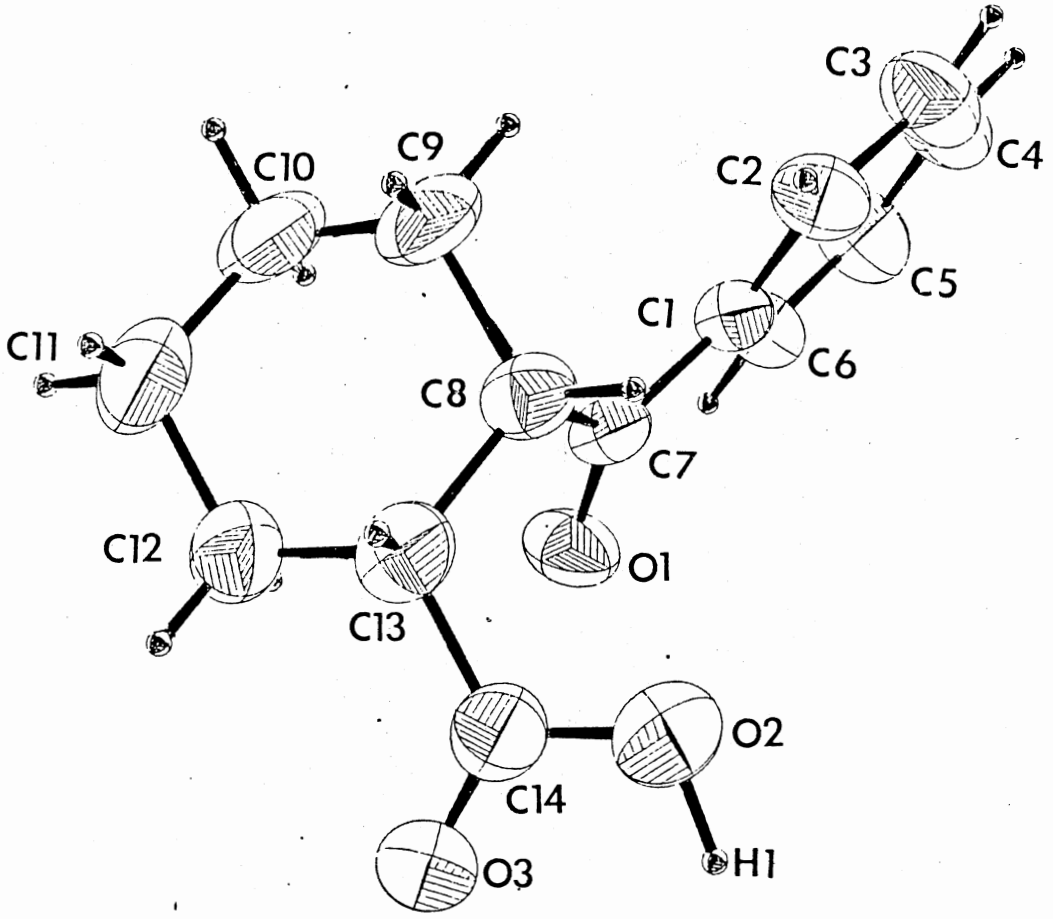

Figure 2. 2-Benzoylcyclohexanecarboxylic Acid.

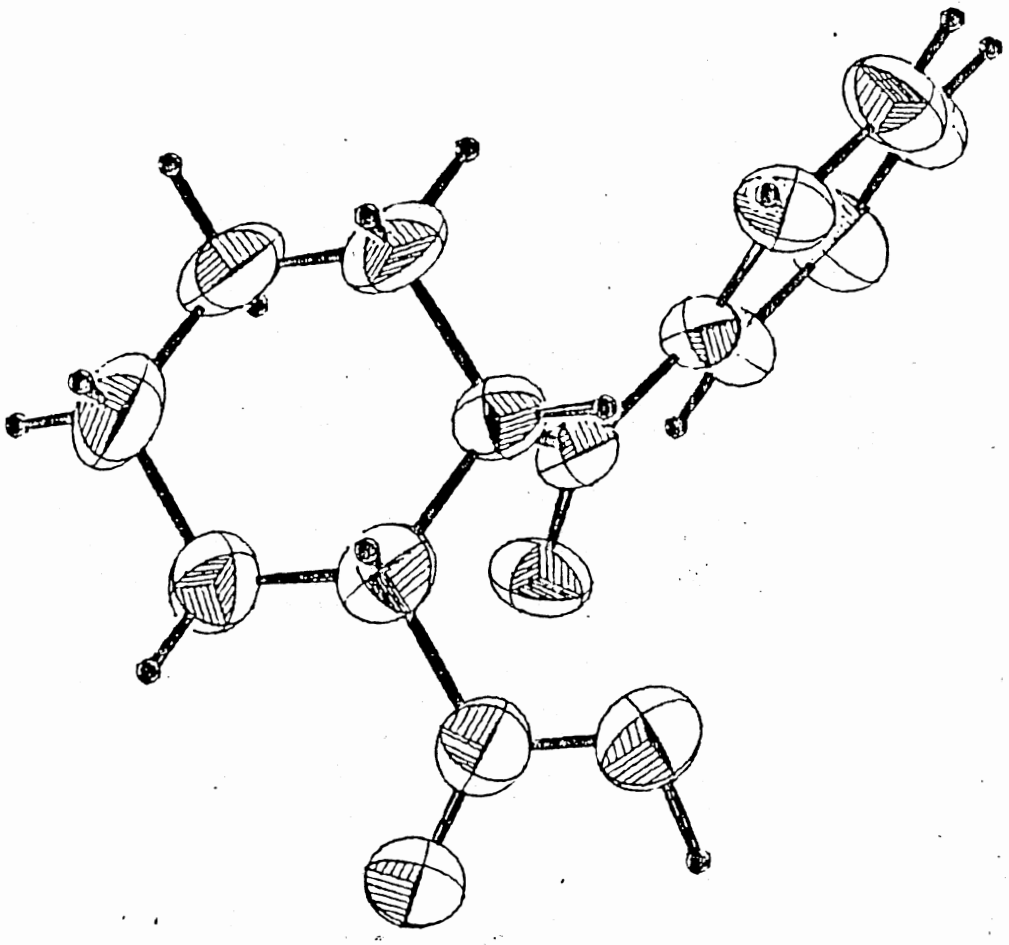

Figure 3. 2-Benzoylcyclohexanecarboxylic Acid.

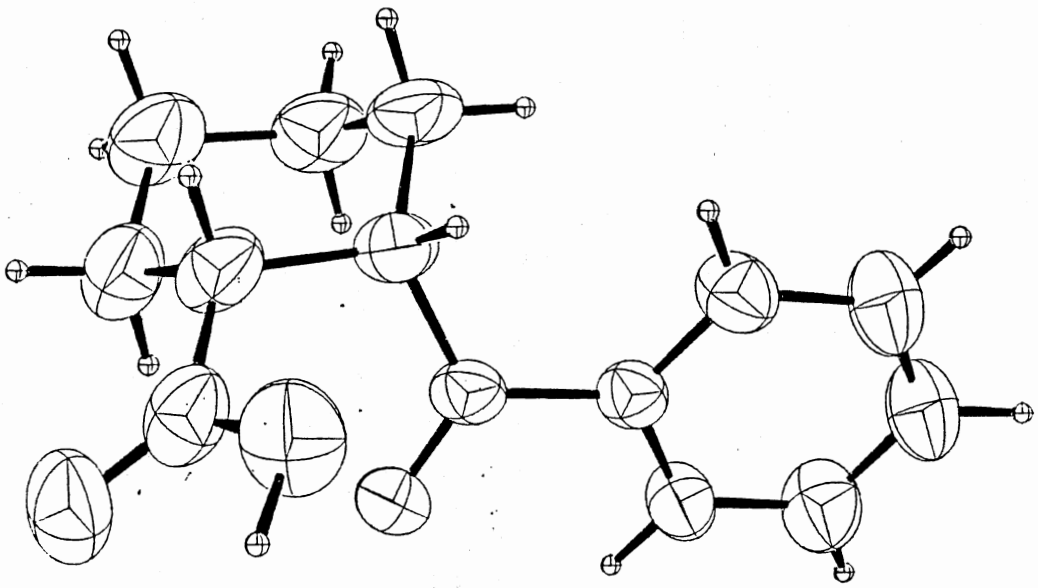

Figure 4. 2-Benzoylcyclohexanecarboxylic Acid.

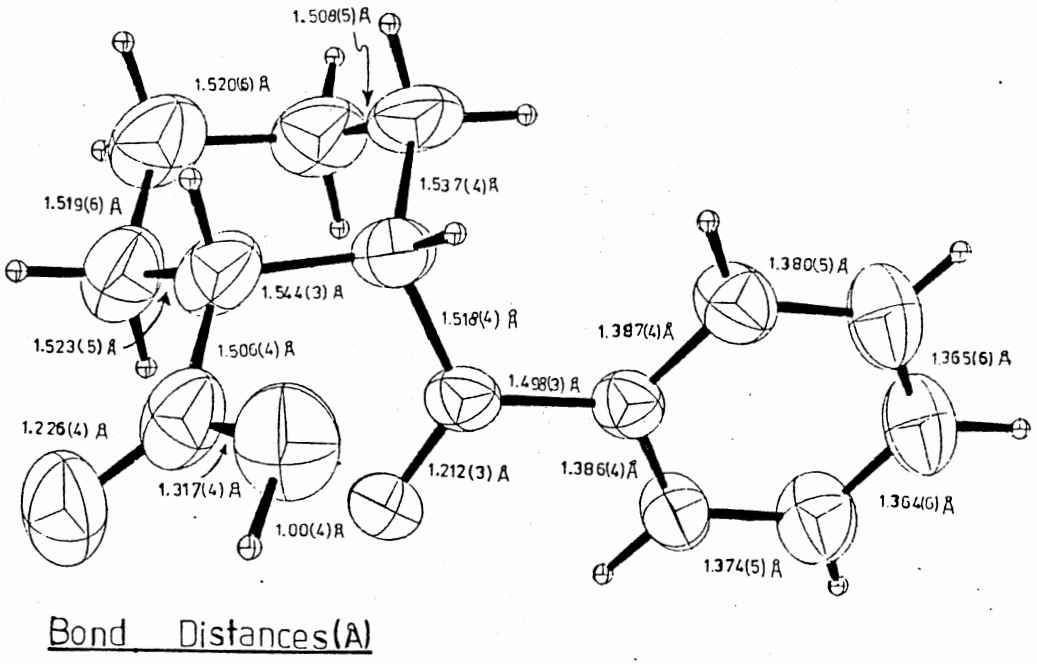

Figure 5. 2-Benzoylcyclohexacarboxylic Acid with Bond Distances.

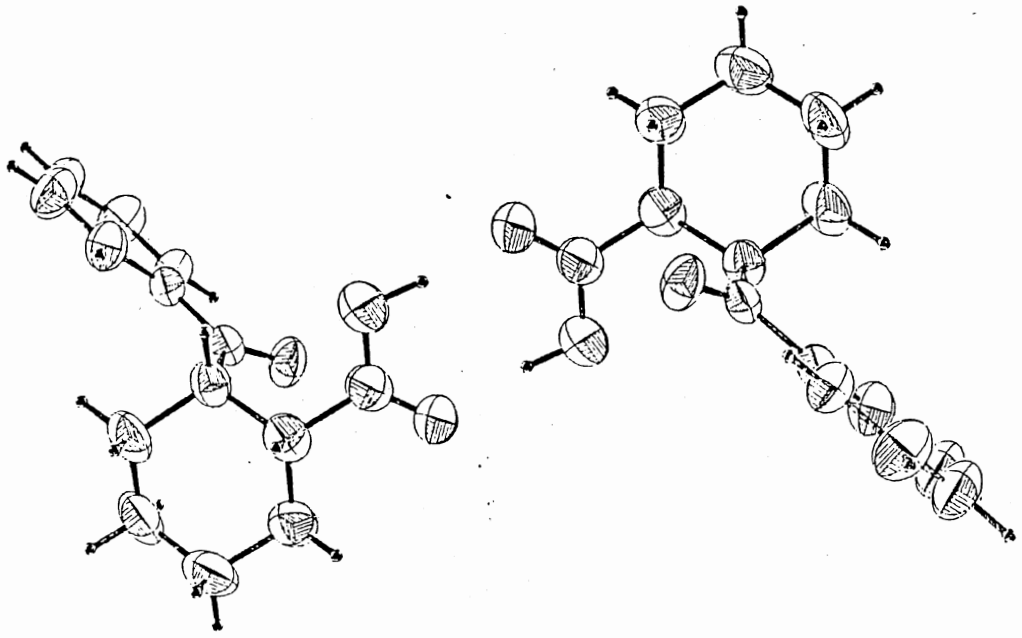

Figure 6. Dimers of 2-Benzoylcyclohexanecarboxylic Acid.

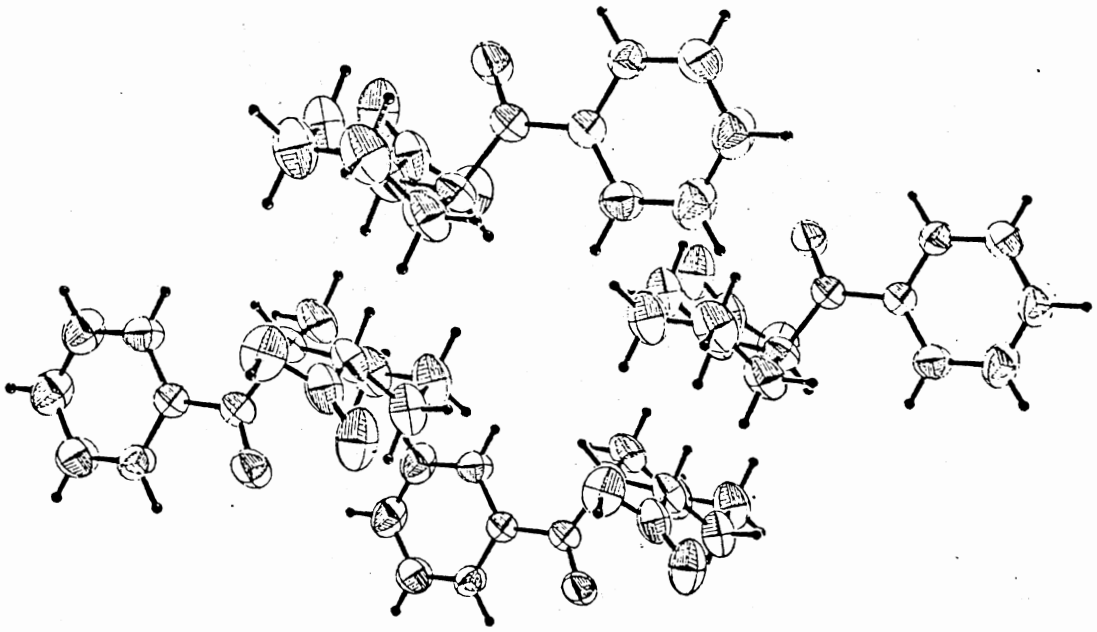

Figure 7. Projection View of Unit Cell (a x b x 1/2 c).

VITA

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