Title

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Ferrichrome-A Tetrahydrate. Determination of Crystal and Molecular Structure[1]

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The crystal and molecular structure of ferrichrome-A tetrahydrate, $C_{41}H_{58}N_9O_{20}Fe \cdot 4H_2O$, has been determined with single-crystal X-ray diffraction techniques including use of anomalous dispersion to establish absolute configuration. The crystals are monoclinic, space group $P2_1$, with two molecules per unit cell with dimensions a = 11.02, b = 13.26, and c = 18.22 Å., and β = 99.48°. The molecule contains a hexapeptide ring with the sequence of amino acid residues -Orn-Orn-Orn-Ser-Ser-Gly- and with a trans conformation at each peptide link. The iron atom is bound by three hydroxamate rings in the configuration of a left-handed propeller. Two hydrogen bonds are found within the molecule. Disorder is present in some of the side chains and in one of the four water molecules.

Ferrichrome-A is a metabolic product of the smut fungus Ustilago sphaerogena, and it is related in structure to several substances which are growth factors for certain micro-organisms.[2-4] The molecule (Fig. 1) contains a ferric atom bound by three hydroxamate rings; it includes a hexapeptide ring made up of one glycine, two serine, and three ornithine amino acid residues; and it has three trans (β-methyl) glutaconic acid residues. This molecular structure (except for the hydrogen bonding) has been established by chemical methods[5-7] and is fully confirmed by the X-ray crystallographic study described in this paper.

Ferrichrome-A crystallizes from water as the tetrahydrate.[5] We studied these crystals to confirm the chemical results, to determine those details of structure not obtainable by chemical methods, and to seek clues to the structures of proteins. We also made a preliminary study of ferrichrome,[8] a closely related substance which can be crystallized from methanol, but did not determine the structure.

Experimental

Many batches of ferrichrome-A crystals, grown from water, were provided us by Prof. J. B. Neilands. The diffraction data were obtained from a dark red and opaque crystal with dimensions approximately 0.1 × 0.1 × 0.2 mm. The crystal was glued to a thin glass fiber and handled throughout the course of the experiment in air. After 20 to 30 hours of exposure to X-rays over a period of two months it showed no deterioration.

The data were taken on a General Electric XRD-5 unit equipped with a goniostat, a pulse height discriminator, and a scintillation counter. Iron Kα radiation was used, and cell dimensions were based on $\lambda(\alpha_1) = 1.93597$, $\lambda(\alpha_2) = 1.93991$ Å. The tube was operated at 40 kv. and 20 ma.

The diffracted beam was filtered through a Mn metal film that absorbed 75% of the Kβ and 25% of the Kα radiation.

The background was plotted as a function of θ, the Bragg angle. Where the background was larger due to streaking along a central lattice row, a minimum background was sought on the small-angle side of the reflection (2θ scan); otherwise, background was taken from the plot. Above $2\theta = 50^\circ$ the background was about 2 counts/sec.

Each independent reflection with $\sin\theta/\lambda < 0.51$ ($2\theta < 165^\circ$) was measured. Of these 3115 reflections, 56 were recorded as zero, while the others ranged from 1 count/sec. to 17,000 counts/sec. Each reflection was counted for 10 sec. using a stationary counter and stationary crystal technique.

The data were corrected for the effects of α_1-α_2 splitting on the basis of a curve derived from measurements of a set of strong reflections with both Kα and unfiltered Kβ radiation. No such correction was necessary up to $2\theta = 100^\circ$. Correction for the Lorentz-polarization factor was made by the standard formula.[9]

For copper Kα radiation ($\lambda = 1.54$ Å.) the out-of-phase amplitude for Fe is $\Delta f'' = 3.4$ electrons. If $k \neq 0$, the intensities of $hk\ell$ and $h\bar{k}\ell$ are different in general because of this phase shift. Our crystal was set with the b axis parallel to the phi axis, and the settings chi, phi and theta are the same for $hk\ell$ and $h\bar{k}\ell$ except that the sign of chi is negative. We found that our General Electric goniostat could be operated as far as $\chi = -20^\circ$, even though the scale extends only to -10°, by counting revolutions of the setting wheel. We measured intensities of 71 pairs of reflections with copper Kα radiation and found 40 with considerable intensity differences.

Humidity Effects. The cell dimensions were observed to expand in an anisotropic manner with changes in the humidity. Between 30% and 40% relative humidity the a, b and c axes expanded 0.08% (0.008 Å.), 0.17% (0.022 Å.) and 0.03% (0.005 Å.) respectively. The changes in dimensions occured rapidly and reversibly. The room was not controlled for humidity, and the changes were sufficient to cause the goniostat settings to be off. Three sets of goniostat settings were computed which covered a low, medium and high range of humidity. By using the appropriate set, the intensities could be accurately and rapidly measured. The relative humidity ranged from a low of 24% to a high of 47% during the measurements.

Computations. The calculations were performed on an IBM-7044 computer equipped with a 32K memory. Least-squares refinements were made with a modified version of the Sparks, Gantzell and Trueblood program (unpublished). As modified to work under the Fortran IV-IBSYS system, this full-matrix program could only handle ~~121~~ 161 parameters. Since over 300 parameters were being adjusted, the refinement was done piecemeal in blocks of ~~121~~ 161 parameters or less at a time, each block requiring about 70 minutes per cycle. The function minimized was $\sum w(|F_o| - |F_c|)^2 / \sum w|F_o|^2$. The weighting factors w were taken as unity. Programs of our own design (unpublished) were used for data processing, Fourier functions, and distances and angles.

The "unreliability index" referred to in this paper is defined as

$$R = \sum ||F_o| - |F_c|| / \sum |F_o|.$$

Crystal Data for Ferrichrome. A tiny crystal of ferrichrome[8] about 0.1 mm. long and a few hundredths mm. in diameter was studied by the Weissenberg technique with Cr Kα radiation (λ = 2.291 Å.). The orthorhombic cell has dimensions a = 16.1, b = 29.6 and c = 8.95 Å with four molecules per cell. Absent reflections correspond to space group $P2_12_12_1$. The

density is estimated to be 1.40 ± 0.05 g./ml. by flotation in a mixture of ethylene dichloride and chloroform. This density with the above cell dimensions indicates a molecular weight of about 900, whereas the analysis[5] indicates 741. The discrepancy may result from methanol incorporated in the crystals, but removed by drying prior to analysis; if so, it corresponds to five molecules of methanol per molecule of ferrichrome. Because better crystals of ferrichrome-A became available, no further work was done on ferrichrome.

Unit Cell and Space Group of Ferrichrome-A Tetrahydrate. The crystals are monoclinic with the cell dimensions (at 22° and 36% relative humidity): a = 11.02 ± 0.03 Å., b = 13.26 ± 0.03 Å., c = 18.22 ± 0.01 Å., β = 99.48 ± 0.08°. The precision of our measurements is considerably better than the errors indicated, which include the variations in cell dimensions due to the fluctuations in humidity during the data-taking period.

The cell volume and measured density (1.45 ± 0.06 g./ml.)[2] dictate two molecules per unit cell. The density calculated from the X-ray data is 1.42 ± 0.01 g./ml. Reflections of the type $0k0$ are observed only if $k = 2n$. The space group $P2_1$ is the only one consistent with this extinction rule in the observed point group (2). It lacks reflection and inversion symmetry as required for an optically active substance. The two-fold general position: (x, y, z; $-x$, $\frac{1}{2} + y$, $-z$) accommodates one complete molecule in the asymmetric unit. Because this space group is polar, the origin must be defined with respect to the b direction; the y parameter of Fe was set at zero for this purpose.

Structure Determination

The structure was derived by Fourier methods combined with least-square calculations in which atoms were judged by the behavior of their individual

isotropic thermal parameters. A three-dimensional Patterson function was calculated and studied. Three large peaks appeared: the origin peak and two peaks in the Harker section at $z = 1/2$. The Harker peaks were about 12% and 10% of the height of the origin peak. We gambled on the assumption that the largest of the two Harker peaks was the Fe-Fe vector, though it was too large to be a single peak. A least-squares calculation using all the data and the Fe atom alone yielded $R = 0.54$. Six oxygen atoms forming the anticipated octahedron about Fe were derived from the Patterson function and tested by least squares. The Fe and four of these oxygens refined to $R = 0.51$. An electron density Fourier was calculated, and the next seven biggest peaks were included in the next series of refinements. About half of these did not refine and were removed. Another Fourier and least-squares procedure similar to the above one was tried.

We were very uncertain of our progress and then calculated a Patterson superposition of the origin peak on the "Fe-Fe" peak. From this pattern the three hydroxamate rings at the iron atom and a scattering of other atoms were guessed at, and the procedure was continued. At this point we had 27 atoms in the refinement, but could not recognize any structure besides the hydroxamate system.

Dispersion Effect with Copper Radiation. Slow progress stimulated us to make the measurements with copper radiation to get more direct evidence of the iron coordinates. Inspection of the results showed immediately that the large effects were associated with large values of $\cos 2\pi(\frac{h}{24} + \frac{k}{4} + \frac{\ell}{8})$, corresponding to $x \sim 0.04$, $z \sim 0.12$ for Fe in agreement with our choice from the Patterson function.

Because the refinement began to improve, we made no use of the phase information which can be extracted from these data, but we used

them to check the absolute configuration of the structure and as a confirmation of the correctness of our structure determination. After the entire structure is known, one can calculate the magnitude of the effect for any pair of reflections. In Table I we list the intensity difference as a percentage of the average of the two intensities for the 26 cases in which the absolute difference was largest. The agreement is good, and the sign of the effect is correct in each case, as it is also for the next 14 pairs in order of decreasing effect. For the remaining 31 pairs, the effects are small or negligible, and the sign is wrong in 12 cases. If the absolute configuration were reversed, the numerical effects would be the same, but all the signs would be reversed.

Final Solution. Confident of the Fe position, we continued the procedure of adding new peaks from the Fourier, refining them by least squares, deleting those whose temperature factors became too large, calculating a new Fourier and repeating the procedure. Occasionally atoms were included where we thought they might be required to link up the developing molecule. About half the atoms we added each time did not refine, but the list of acceptable atoms continued to grow. Finally the hexapeptide ring did resolve, and the process continued till the methyl glutaconic acid side chains developed, and the molecule was fully determined.

Difficulties were encountered in resolving the structure of one seryl hydroxyl group, one terminal carboxyl group, the central portion of a methyl glutaconic acid side chain, and one of the water molecules. Disorder was invoked to explain the difficulties. The disordered atoms were split into fractional atoms in separate locations. Seven of the 75 heavy atoms (hydrogen excluded) were given two locations each, and one water molecule was split into four quarter-atoms. This increased the

Table I. Dispersion effect for $hk\ell$-$h\bar{k}\ell$ pairs with CuKα radiation.

Values of $200(|F_+|^2 - |F_-|^2)/(|F_+|^2 + |F_-|^2)$

$hk\ell$	Obsd.	Calcd.	$hk\ell$	Obsd.	Calcd.
210	-11	-11	0.3.11	-38	-47
015	+27	+32	014	+1	+1
$31\bar{2}$	-14	-16	0.1.11	+18	+13
$31\bar{1}$	-3	-1	0.1.14	+48	+47
312	-44	-55	420	-10	-23
016	-130	-121	$62\bar{2}$	-10	-11
410	+36	+38	634	-7	-3
027	-69	-81	028	-3	-2
0.1.10	-34	-41	019	+44	+37
510	+8	+19	0.3.14	+75	+93
610	-41	-48	$93\bar{6}$	-50	-59
614	+55	+60	0.2.11	-48	-64
520	-8	-8	029	-10	-21

the number of atomic positions refined to 85. Attempts to treat the disordered atoms with anisotropic thermal parameters (rather than splitting them into fractional atoms) were not successful. It is interesting that a similar kind of disorder was encountered by Alden, Stout, Kraut and High.[10]

An electron-density difference function (all atoms except hydrogen subtracted out) gave peaks in approximately the positions expected for 42 of the 68 hydrogen atoms. These hydrogen peaks were found only for parts of the molecule not involved in disorder. None of the hydrogen atoms of water could be found even when anisotropic thermal parameters were used for oxygen. The difference function showed many other peaks similar in height to the hydrogen peaks, or smaller, which may result from errors in the data or from anisotropic motion of other atoms. These 42 hydrogen atoms were included in the final calculations, but the resulting thermal parameters and bond lengths indicate that the resulting positions are not accurate enough to justify reporting them here.

In the final calculations, with anisotropic thermal parameters only for iron, R was 0.090 for the 3115 reflections. Shifts of coordinates of (non-hydrogen) atoms not involved in disorder were less than the estimated standard deviations by factors that in most cases exceeded 10. Further refinement might lead to a slightly better fit, especially with a larger computer which could refine all the atoms simultaneously. But our lack of progress in the later cycles convinced us that the structure is refined as far as is justified for the present experimental data.

Results

The atomic coordinates and isotropic thermal parameters are shown in Table II. As the full matrix was too large to fit into the computer

Table II. Atomic Parameters in Ferrichrome-A Tetrahydrate[a]

Atom[b]	x/a	y/b	z/c	B(Å^2)
Fe	0.0444	0.0	0.126	3.0[c]
O1ORN1	-0.238	0.453	-0.507	4.6
O2ORN1	-0.204	0.462	-0.186	4.0
N1ORN1	-0.171	0.319	-0.330	3.6
N2ORN1	0.280	0.043	0.204	3.7
C1ORN1	-0.183	0.402	-0.453	3.6
C2ORN1	-0.249	0.372	-0.390	3.5
C3ORN1	-0.311	0.461	-0.360	3.8
C4ORN1	-0.393	0.434	-0.304	4.0
C5ORN1	0.400	0.014	0.246	4.1
O1ORN2	-0.235	0.164	-0.365	4.3
O2ORN2	0.008	0.359	-0.119	3.3
N1ORN2	0.021	0.214	-0.230	3.3
N2ORN2	-0.026	0.310	-0.059	3.2
C1ORN2	-0.176	0.222	-0.319	3.3
C2ORN2	-0.106	0.179	-0.247	3.2
C3ORN2	-0.180	0.199	-0.183	3.1
C4ORN2	-0.127	0.149	-0.108	4.3
C5ORN2	-0.014	0.199	-0.063	3.8
O1ORN3	0.081	0.107	-0.310	4.1
O2ORN3	-0.041	0.009	0.214	3.7
N1ORN3	0.303	0.188	-0.304	3.4
N2ORN3	-0.164	0.026	0.194	3.9

C1ORN3	0.106	-0.176	-0.266	2.9
C2ORN3	0.238	0.212	-0.242	3.3
C3ORN3	0.257	0.324	-0.218	3.8
C4ORN3	0.221	0.398	-0.280	3.7
C5ORN3	-0.235	0.009	0.253	4.8
C1SER1	0.416	0.308	-0.439	5.1
C2SER1'	0.455	0.172	0.397	5.0 [d]
C2SER1"	0.400	0.315	0.394	7.8 [d]
N SER1	0.186	0.304	0.457	4.5
C1SER1	0.369	0.230	0.535	3.5
C2SER1	0.280	0.228	0.463	3.8
C3SER1	0.355	0.239	0.397	6.5
C1SER2	0.126	0.257	-0.436	4.6
O2SER2	-0.122	0.026	0.446	6.4
N SER2	-0.068	0.370	-0.450	3.7
C1SER2	0.110	0.311	0.508	3.8
C2SER2	0.006	0.387	0.491	3.8
C3SER2	0.048	0.492	0.487	4.9
O GLY	0.489	0.155	-0.232	5.6
N GLY	0.388	0.133	-0.432	4.5
C1GLY	0.419	0.159	-0.294	4.0
C2GLY	0.474	0.130	-0.362	4.2
C1NGA1	0.135	0.133	0.133	3.8
O2NGA1'	0.463	0.404	0.297	7.0 [e]
O2NGA1"	-0.419	0.289	0.227	9.6 [e]

O3MGA1'	-0.390	0.392	0.221	10.4 [e]
O3MGA1"	-0.430	0.445	0.261	11.2 [e]
C1MGA1	0.243	0.132	0.175	3.5
C2MGA1	0.321	0.221	0.190	4.1
C3MGA1	0.316	0.302	0.149	4.5
C4MGA1	0.402	0.393	0.171	7.1
C5MGA1	-0.495	0.381	0.229	9.3
C6MGA1	0.231	0.319	0.076	6.6
O1MGA2	-0.077	0.462	-0.023	3.8
O2MGA2	0.076	0.414	0.229	5.9
O3MGA2	0.027	0.304	0.309	7.5
C1MGA2	-0.066	0.368	-0.009	3.6
C2MGA2	-0.082	0.325	0.063	4.2
C3MGA2	-0.119	0.370	0.119	4.0
C4MGA2	-0.106	0.313	0.192	4.5
C5MGA2	0.002	0.346	0.247	4.8
C6MGA2	-0.178	0.472	0.117	6.0
O1MGA3	-0.121	0.066	0.081	4.3
O2MGA3	-0.491	0.110	-0.093	7.7
O3MGA3	0.349	0.189	-0.067	9.8
C1MGA3	-0.199	0.059	0.124	5.2
C2MGA3'	-0.321	0.114	0.108	4.4 [f]
C2MGA3"	-0.336	0.029	0.081	4.7 [f]
C3MGA3'	-0.413	0.064	0.063	4.8 [f]
C3MGA3"	-0.411	0.106	0.058	3.4 [f]

C4MGA3'	0.453	0.085	0.030	5.2 [f]
C4MGA3"	0.465	0.130	0.040	3.4 [f]
C5MGA3	0.443	0.141	-0.046	6.5
C6MGA3'	0.403	0.459	-0.041	8.2 [f]
C6MGA3"	-0.380	0.214	0.074	7.9 [f]
W1	0.249	0.467	0.334	6.2
W2	0.030	0.105	0.352	7.4
W3	-0.227	0.447	0.342	6.3
W4'	-0.312	0.163	0.474	4.3 [g]
W4"	-0.347	0.216	0.441	10.0 [g]
W4'"	0.399	0.497	0.502	5.8 [g]
W4""	-0.370	0.023	0.451	5.2 [g]

[a] Not including hydrogen atoms. [b] Identification is by a six letter code. ORN stands for ornithyl, SER for seryl, GLY for glycyl, MGA for β-methyl glutaconic acid, W for water oxygen, Fe for iron, O for oxygen, N for nitrogen, and C for carbon. [c] The Fe temperature factor is an average value of the following anisotropic set: B11 = 3.20, B22 = 3.67, B33 = 2.16, B12 = -0.11, B13 = -0.18, and B23 = 0.08 $Å^2$. [d] Disordered hydroxyl in a seryl group. Each was refined as half an oxygen atom. [e] Disordered oxygens on the carboxyl of a methyl glutaconic acid chain. The singly primed atoms form one conformation, the doubly primed atoms for the second. Each was refined as half an oxygen atom. [f] Disordered central portion of a methyl glutaconic acid chain. Singly primed atoms form one conformation and were each refined as 0.6 of an atom. Doubly primed atoms form the minor conformation and were each refined as 0.4 of an atom. [g] Disordered water molecule. Each was refined as a quarter oxygen atom.

memory, standard deviations of the parameters could not be calculated properly. From calculations based on as much of the matrix as could fit into the memory, the standard deviations of atomic coordinates are estimated to be 0.0001 for Fe and 0.001 for C, N and O atoms which are not disordered. A projection of the molecule is shown in Fig. 2.

Tables III, IV and V itemize the distances and angles as designated in Fig. 3. The standard deviations of these distances are estimated to be about 0.02 Å. and of the angles about 1°.

There are two intra-molecular hydrogen bonds, indicated as dotted lines in Fig. 1 and 2. One is across the hexapeptide ring between the carbonyl oxygen of a seryl group (O1SER2) and the nitrogen of an ornithyl group (N1ORN3). The second hydrogen bond is between the two ends of an ornithyl group (N1ORN2) to (O2ORN2). The two hydrogen-bond distances are 2.98 Å. and 2.80 Å. respectively. Several other oxygen atoms are hydrogen bonded to the four water molecules which in turn are hydrogen bonded to oxygens in adjacent ferrichrome-A molecules. There is disorder in these bonds, which involve atoms in alternate sites.

The anomalous-dispersion experiment enabled us to determine the absolute configuration of the molecule. The five asymmetric amino acid residues are all found to have the expected L configuration. The three rings at the iron atom have the shape of a left-handed propeller as shown in Fig. 4.

At the suggestion of Dr. J. Kraut we calculated the angular twists of adjacent peptides in the hexapeptide ring, in order to compare some actual values of these angles with those postulated by Ramachandran, Ramakrishnan and Sasisekharan.[11] These authors calculated the range of twisting allowed in terms of two angles ϕ and ϕ' which represent

Table III. Distances and Angles in the Three Hydroxamate Rings about Fe in Ferrichrome-A

	RING 1	RING 2	RING 3	AVE.
a	1.97 Å	1.96 Å	2.00 Å	1.98 Å
b	2.02 Å	2.03 Å	2.06 Å	2.04 Å
c	1.37 Å	1.39 Å	1.37 Å	1.38 Å
d	1.30 Å	1.28 Å	1.27 Å	1.28 Å
e	1.32 Å	1.31 Å	1.33 Å	1.32 Å
f	2.81 Å	2.79 Å	2.80 Å	2.80 Å
g	2.83 Å	2.79 Å	2.79 Å	2.80 Å
α	78°	79°	78°	78°
β	113°	112°	111°	112°
γ	115°	113°	112°	113°
δ	117°	116°	116°	116°
ϵ	116°	118°	119°	118°

Table IV. Distances and Angles in the Six Amino Acids in Ferrichrome-A

		GLY	SER1	SER2	ORN1	ORN2	ORN3	AVE.
N -C1	h	1.41 Å	1.35 Å	1.33 Å	1.30 Å	1.33 Å	1.32 Å	1.34 Å
C2-N1	i	1.47 Å	1.44 Å	1.47 Å	1.45 Å	1.46 Å	1.47 Å	1.46 Å
C1-C2	j	1.52 Å	1.51 Å	1.52 Å	1.51 Å	1.52 Å	1.53 Å	1.52 Å
C1=O1	k	1.25 Å	1.22 Å	1.23 Å	1.27 Å	1.24 Å	1.22 Å	1.24 Å
C2-C3	ℓ		1.57 Å	1.47 Å	1.51 Å	1.54 Å	1.56 Å	1.53 Å
C3-O2	m		(x)	1.42 Å				
C3-C4	n				1.51 Å	1.55 Å	1.50 Å	1.52 Å
C4-C5	o				1.51 Å	1.53 Å	1.54 Å	1.53 Å
C5-N2	p				1.47 Å	1.48 Å	1.46 Å	1.47 Å
N -C1-O1	ζ	125°	126°	121°	123°	122°	121°	123°
N -C1-C2	η	117°	113°	116°	117°	119°	117°	117°
O1-C1-C2	θ	117°	121°	123°	120°	119°	122°	120°
C1-C2-N1	ι	115°	114°	104°	113°	113°	106°	111°
C2-N1-C	K	115°	121°	124°	124°	120°	122°	121°

C1-C2-C3	λ	108°	114°	112°	109°	117°	112°
N1-C2-C3	μ	111°	114°	111°	114°	111°	112°
C2-C3-O2	ν	(x)	113°				
C2-C3-C4	ξ			114°	115°	114°	114°
C3-C4-C5	o			114°	117°	113°	115°
C4-C5-N2	π			114°	113°	110°	112°
C5-N2-O2	ρ			113°	113°	114°	113°
C -N2-C5	σ			130°	131°	130°	130°

x Disordered serine oxygen. The corresponding two distances and angles (π and ν) are 1.12 Å, 1.42 Å, 117°, and 114°.

Table V. Distances and Angles in the Three Glutaconic Acid Side Chains in Ferrichrome-A

ATOMS*	CODE*	MGA1	MGA2	MGA3	AVE.
Fe-O1	b	2.02 Å	2.03 Å	2.06 Å	2.03 Å
O1-C1	d	1.30 Å	1.28 Å	1.27 Å	1.28 Å
C1-N2(ORN)	e	1.32 Å	1.31 Å	1.33 Å	1.32 Å
C1-C2	q	1.47 Å	1.47 Å	(y)	1.47 Å
C2-C3	r	1.30 Å	1.30 Å	(y)	1.30 Å
C3-C4	s	1.54 Å	1.51 Å	(y)	1.53 Å
C3-C6	t	1.51 Å	1.50 Å	(y)	1.51 Å
C4-C5	u	(xx)	1.50 Å	(y)	1.50 Å
C5-O2	v	(xx)	1.29 Å	1.26 Å	1.28 Å
C5-O3	w	(xx)	1.25 Å	1.22 Å	1.24 Å
Fe-O1-C1	γ	115°	113°	113°	114°
O1-C1-N2(ORN)	ϵ	116°	118°	118°	117°
O1-C1-C2	τ	123°	122°	(y)	123°
C2-C1-N2(ORN)	υ	121°	120°	(y)	121°
C1-C2-C3	ϕ	127°	129°	(y)	128°
C2-C3-C6	χ	126°	125°	(y)	126°
C2-C3-C4	ψ	122°	118°	(y)	120°
C6-C3-C4	ω	112°	117°	(y)	115°
C3-C4-C5	ω''	119°	113°	(y)	116°
C4-C5-O2	ω'	(xx)	120°	(y)	120°
C4-C5-O3	ω'''	(xx)	121°	(y)	121°
O2-C5-O3	ω''''	(xx)	120°	121°	121°

xx Disordered carboxyl group.

y Disordered C2, C3, C4, and C6 atoms.

* Labelled according to figure 3.

right-handed rotations of the peptide NCO planes as viewed from the alpha carbon. The observed angles are listed in Table VI. The three

Table VI. Peptide Twist Angles in Ferrichrome-A

Peptides	ϕ	ϕ'
GLY-ORN3-ORN2	35°	200° [a]
ORN3-ORN2-ORN1	103°	311°
ORN2-ORN1-SER2	76°	5° [a]
ORN1-SER2-SER1	17°	174°
SER2-SER1-GLY	123°	131°
SER1-GLY-ORN3	262°	358° [a]

[a]Angles outside the "permissible" limits.

values that lie outside the limits suggested by Ramachandran, et al. involve either glycine or hydrogen bonding. These values are not very far outside the outer limits, and an extension of the ϕ' limits by about 20° would include all of these values.

A list of the observed and calculated structure factors is shown in Table VII.

Acknowledgment. We wish to than Professor J. B. Neilands for making available to us this interesting substance and for his efforts in providing us with a suitable single crystal without which the work could not have been done. We also thank Dr. T. N. Margulis and Dr. M. P. Klein who separately brought this problem to our attention and stimulated our interest.

Table VII. Observed and Calculated Structure Factors

(Table, in three parts, to be reproduced photographically.)

H,K= 0, 0

L	FOBS	FCAL
1	19	76
2	486	657
3	872	932
4	232	293
5	657	620
6	283	282
7	597	576
8	167	153
9	745	760
10	147	136
11	258	233
12	368	363
13	71	87
14	31	18
15	80	76
16	431	423
17	199	156
18	40	9

H,K= 0, 1

L	FOBS	FCAL
1	580	659
2	946	1007
3	745	681
4	777	766
5	631	570
6	166	153
7	78	95
8	256	223
9	160	170
10	356	324
11	382	330
12	325	281
13	325	301
14	246	256
15	159	166
16	269	246
17	77	90
18	164	177

H,K= 0, 2

L	FOBS	FCAL
-0	181	170
1	740	821
2	220	235
3	509	473
4	615	621
5	389	332
6	671	647
7	257	180
8	702	648
9	330	300
10	246	197
11	188	160
12	165	166
13	393	390
14	119	88
15	107	121
16	28	66
17	50	42
18	190	186

H,K= 0, 3

L	FOBS	FCAL
1	1865	2017
2	1020	981
3	518	461
4	189	164
5	614	591
6	314	316
7	301	317
8	451	458
9	132	150
10	232	210
11	317	296
12	144	126
13	196	198
14	186	144
15	177	155
16	124	96
17	269	253

H,K= 0, 4

L	FOBS	FCAL
-0	547	522
1	383	357
2	567	572
3	721	732
4	404	397
5	253	197
6	310	297
7	108	72
8	365	333
9	220	239
10	85	40
11	159	154
12	77	79
13	142	137
14	104	90
15	249	256
16	104	97
17	133	126

H,K= 0, 5

L	FOBS	FCAL
1	783	760
2	329	315
3	170	150
4	556	535
5	162	150
6	45	31
7	276	268
8	380	359
9	67	59
10	408	368
11	154	145
12	77	90
13	118	117
14	162	160
15	39	26
16	60	46
17	55	68

H,K= 0, 6

L	FOBS	FCAL
-0	1082	1054
1	278	284
2	251	275
3	503	492
4	416	398
5	156	172
6	457	453
7	29	28
8	339	344
9	135	130
10	114	101
11	217	206
12	160	167
13	256	237
14	88	74
15	190	177
16	213	204

H,K= 0, 7

L	FOBS	FCAL
1	431	432
2	382	361
3	239	230
4	235	249
5	204	226
6	381	380
7	137	107
8	160	141
9	264	248
10	256	264
11	119	109
12	136	134
13	40	36
14	78	83
15	137	115

H,K= 0, 8

L	FOBS	FCAL
-0	160	128
1	188	172
2	187	189
3	442	422
4	287	299
5	98	103
6	205	209
7	399	383
8	394	385
9	256	268
10	31	30
11	83	74
12	205	229
13	116	111
14	139	121

H,K= 0, 9

L	FOBS	FCAL
1	247	230
2	88	68
3	233	210
4	44	53
5	89	52
6	166	149
7	199	193
8	69	82
9	196	187
10	0	33
11	82	67
12	151	152
13	91	128

H,K= 0, 10

L	FOBS	FCAL
-0	276	262
1	77	68
2	150	157
3	190	169
4	76	66
5	163	169
6	30	31
7	111	113
8	190	203
9	62	80
10	78	74
11	131	138
12	53	42

H,K= 0, 11

L	FOBS	FCAL
1	74	70
2	52	52
3	67	70
4	114	133
5	104	86
6	167	131
7	112	121
8	107	122
9	113	133
10	148	177

H,K= 0, 12

L	FOBS	FCAL
-0	152	166
1	39	30
2	110	130
3	86	76
4	53	21
5	109	94
6	130	141
7	80	76
8	124	110

H,K= 0, 13

L	FOBS	FCAL
1	68	74
2	108	111
3	47	39
4	27	25
5	97	82

H,K= 1, 0

L	FOBS	FCAL
-18	182	163
-17	26	49
-16	492	444
-15	86	89
-14	31	20
-13	63	38
-12	134	152
-11	121	109
-10	55	61
-9	789	774
-8	463	513
-7	449	406
-6	833	831
-5	1779	1770
-4	494	535
-3	554	480
-2	152	116
-1	725	735
-0	1232	1487
1	310	239
2	172	411
3	909	974
4	191	253
5	313	290
6	29	20
7	152	159
8	278	295
9	377	377
10	114	108
11	362	368
12	210	213
13	351	335
14	44	25
15	368	362
16	195	195
17	92	74
18	26	30

H,K= 1, 1

L	FOBS	FCAL
-18	163	177
-17	58	49
-16	0	23
-15	135	121
-14	44	74
-13	310	290
-12	319	323
-11	309	299
-10	228	233
-9	192	172
-8	180	185
-7	368	357
-6	669	633
-5	531	532
-4	327	336
-3	599	639
-2	708	659
-1	640	696
-0	611	781
1	698	646
2	490	496
3	665	663
4	759	667
5	280	251
6	417	403
7	429	409
8	447	377
9	225	197
10	387	406
11	100	94
12	99	75
13	352	353
14	168	153
15	83	60
16	216	201
17	84	111

H,K= 1, 2

L	FOBS	FCAL
-18	105	94
-17	96	79
-16	205	156
-15	156	164
-14	175	161
-13	223	212
-12	344	340
-11	146	154
-10	306	318
-9	343	323
-8	362	357
-7	307	300
-6	476	465
-5	430	440
-4	766	823
-3	1010	1060
-2	510	581
-1	288	327
-0	942	975
1	57	57
2	1249	1312
3	474	506
4	395	395
5	442	471
6	136	93
7	415	432
8	267	303
9	337	347
10	65	50
11	126	125
12	357	338
13	70	79
14	159	159
15	77	75
16	54	91
17	87	61

H,K= 1, 3

L	FOBS	FCAL
-18	64	96
-17	111	80
-16	126	120
-15	129	115
-14	215	221
-13	89	61
-12	196	193
-11	143	106
-10	148	147
-9	131	126
-8	402	340
-7	387	370
-6	370	343
-5	264	211
-4	140	139
-3	315	261
-2	697	735
-1	232	230
-0	931	978
1	749	765
2	874	868
3	671	657
4	400	401
5	79	54
6	432	439
7	339	374
8	520	509
9	94	85
10	251	235
11	371	362
12	0	21
13	152	171
14	61	89
15	258	229
16	145	122
17	103	105

H,K= 1, 4

L	FOBS	FCAL
-17	51	60
-16	92	75
-15	125	113
-14	30	28
-13	121	131
-12	328	316
-11	132	119
-10	159	160
-9	75	69
-8	243	203
-7	220	173
-6	194	148
-5	258	200
-4	330	328
-3	480	452
-2	324	323
-1	571	567
-0	695	733
1	375	325
2	220	176
3	281	277
4	661	636
5	607	589
6	406	389
7	131	111
8	447	422
9	310	293
10	106	102
11	286	285
12	166	190
13	69	100
14	240	223
15	78	70
16	44	53
17	120	118

H,K= 1, 5

L	FOBS	FCAL
-17	94	84
-16	71	87
-15	127	118
-14	200	169
-13	75	48
-12	144	122
-11	310	323
-10	218	213
-9	244	243
-8	232	238
-7	27	13
-6	36	10
-5	412	431
-4	136	152
-3	125	143
-2	621	602
-1	555	580
-0	428	443
1	432	443
2	121	136
3	318	297
4	148	86
5	420	437
6	343	381
7	108	99
8	221	218
9	110	111
10	347	381
11	236	223
12	145	147
13	228	214
14	209	212
15	53	23
16	32	29

H,K= 1, 6

L	FOBS	FCAL
-16	151	145
-15	217	208
-14	126	104
-13	137	143
-12	148	158
-11	194	183
-10	54	75
-9	205	207
-8	208	178
-7	29	23
-6	163	209
-5	587	601
-4	177	181
-3	215	213
-2	107	115
-1	632	600
-0	609	579
1	481	485
2	356	348
3	605	603
4	466	451
5	95	52
6	440	404
7	242	232
8	295	285
9	230	221
10	130	98
11	62	37
12	187	214
13	171	172
14	86	65
15	190	195
16	127	141

H,K= 1, 7

L	FOBS	FCAL
-15	132	136
-14	95	109
-13	341	324
-12	30	27
-11	228	250
-10	349	351
-9	266	252
-8	31	37
-7	282	311
-6	123	77
-5	151	177
-4	263	272
-3	229	210
-2	228	173
-1	277	283
-0	389	345
1	702	735
2	346	315
3	40	18
4	407	378
5	148	141
6	404	364
7	321	318
8	44	44
9	173	170
10	325	325
11	68	82
12	77	50
13	214	207
14	115	115
15	61	53

H,K= 1, 8

L	FOBS	FCAL
-15	45	71
-14	66	61
-13	118	103
-12	309	309
-11	84	115
-10	81	82
-9	315	308
-8	305	257
-7	365	352
-6	63	60
-5	177	171
-4	247	201
-3	210	190
-2	294	310
-1	122	129
-0	292	310
1	42	72
2	108	78
3	404	392
4	297	307
5	82	72
6	157	148
7	430	426
8	210	196
9	268	250
10	100	99
11	163	186
12	159	149
13	87	69
14	101	83

H,K= 1, 9

L	FOBS	FCAL
-13	165	193
-12	189	202
-11	39	28
-10	141	133
-9	52	51
-8	206	180
-7	161	132
-6	54	71
-5	134	149
-4	114	96
-3	255	272
-2	104	82
-1	104	119
-0	162	181
1	232	219
2	255	228
3	54	38
4	246	250
5	89	69
6	232	239
7	157	134
8	125	126
9	218	218
10	149	128
11	106	94
12	42	44
13	59	68

H,K= 1, 10

L	FOBS	FCAL
-12	97	136
-11	204	217
-10	37	36
-9	56	86
-8	50	42
-7	137	107
-6	152	150
-5	111	96
-4	128	140
-3	70	106
-2	70	96
-1	77	83
-0	196	230
1	54	38
2	283	301
3	155	170
4	92	113
5	143	126
6	213	242
7	128	151
8	139	133
9	66	65
10	68	35
11	116	138
12	56	93

H,K= 1, 11

L	FOBS	FCAL
-10	70	76
-9	42	64
-8	64	77
-7	142	160
-6	123	94
-5	50	34
-4	163	173
-3	130	144
-2	95	104
-1	43	79
-0	195	230
1	85	109
2	123	138
3	227	237
4	120	120
5	358	362
6	100	87
7	65	56
8	116	92
9	51	46
10	50	59

H,K= 1, 12

L	FOBS	FCAL
-8	27	25
-7	98	110
-6	121	116
-5	133	159
-4	46	23
-3	66	66
-2	86	76
-1	117	124
-0	87	78
1	91	107
2	77	96
3	133	132
4	158	172
5	67	52
6	47	66
7	137	169
8	104	110

H,K= 1, 13

L	FOBS	FCAL
-5	72	87
-4	33	45
-3	138	141
-2	72	74
-1	55	69
-0	0	32
1	44	69
2	100	125
3	44	63
4	30	34

H,K= 2, 0

L	FOBS	FCAL
-18	154	162
-17	137	143
-16	134	114
-15	175	188
-14	198	195
-13	55	63
-12	239	224
-11	540	566
-10	245	233
-9	666	638
-8	235	210
-7	498	445
-6	1106	1052
-5	146	179
-4	704	737
-3	716	763
-2	536	526
-1	864	889
-0	523	516
1	815	801
2	358	300
3	328	239
4	257	179
5	671	637
6	358	373
7	48	73
8	573	598
9	122	124
10	352	348
11	450	447
12	126	174
13	108	79
14	117	112
15	28	42
16	150	141
17	124	92

H,K= 2, 1

L	FOBS	FCAL
-18	103	83
-17	91	64
-16	64	52
-15	203	187
-14	125	168
-13	100	85
-12	193	164
-11	258	262
-10	540	508
-9	322	324
-8	275	280
-7	402	381
-6	774	798
-5	303	312
-4	1036	1005
-3	1147	1107
-2	652	623
-1	918	1028
-0	1031	1167
1	159	163
2	977	987
3	995	980
4	377	438
5	906	869
6	316	264
7	369	365
8	543	487
9	373	381
10	320	338
11	69	53
12	45	80
13	247	240
14	256	247
15	150	116
16	139	144
17	72	80

H,K= 2, 2

L	FOBS	FCAL
-18	64	56
-17	51	36
-16	398	365
-15	95	77
-14	203	187
-13	114	86
-12	197	221
-11	79	52
-10	234	237
-9	434	374
-8	567	588
-7	490	478
-6	425	370
-5	495	533
-4	409	405
-3	977	991
-2	619	672
-1	357	331
-0	102	125
1	688	717
2	351	295
3	559	510
4	457	442
5	448	405
6	113	124
7	554	528
8	197	239
9	64	72
10	67	42
11	140	182
12	303	264
13	115	131
14	203	175
15	28	45
16	44	29
17	138	107

H,K= 2, 3

L	FOBS	FCAL
-18	99	122
-17	210	190
-16	28	41
-15	118	119
-14	44	55
-13	272	250
-12	144	106
-11	259	260
-10	385	398
-9	417	391
-8	288	295
-7	261	225
-6	115	107
-5	645	628
-4	243	278
-3	432	482
-2	426	447
-1	447	433
-0	131	66
1	750	778
2	791	752
3	329	268
4	389	379
5	367	379
6	334	308
7	197	209
8	88	90
9	41	96
10	544	493
11	242	206
12	251	263
13	81	95
14	110	136
15	129	116
16	24	26
17	60	77

H,K= 2, 4

L	FOBS	FCAL
-17	79	70
-16	160	137
-15	76	72
-14	91	72
-13	159	185
-12	317	329
-11	44	46
-10	0	23
-9	95	54
-8	355	365
-7	375	384
-6	344	334
-5	616	603
-4	376	298
-3	402	411
-2	531	508
-1	707	762
-0	709	738
1	510	505
2	571	554
3	357	365
4	750	759
5	298	287
6	458	462
7	165	153
8	91	103
9	160	119
10	104	128
11	340	324
12	120	103
13	125	95
14	156	146
15	126	120
16	109	96

H,K= 2, 5

L	FOBS	FCAL
-17	82	70
-16	129	118
-15	154	153
-14	93	122
-13	81	82
-12	83	53
-11	260	257
-10	194	183
-9	67	108
-8	236	229
-7	317	286
-6	202	201
-5	200	220
-4	219	229
-3	367	355
-2	378	408
-1	96	102
-0	188	236
1	902	841
2	447	426
3	429	439
4	178	184
5	235	190
6	612	604
7	116	83
8	268	229
9	108	89
10	148	151
11	83	107
12	92	104
13	230	221
14	155	137
15	173	163
16	40	22

H,K= 2, 6

L	FOBS	FCAL
-16	69	70
-15	132	84
-14	135	130
-13	146	162
-12	134	141
-11	137	117
-10	197	188
-9	44	35
-8	294	310
-7	313	313
-6	456	495
-5	333	284
-4	160	207
-3	402	402
-2	254	228
-1	444	456
-0	250	244
1	277	230
2	324	329
3	573	604
4	313	318
5	40	47
6	309	261
7	260	240
8	235	240
9	230	203
10	175	164
11	230	198
12	310	308
13	97	88
14	58	30
15	154	149

H,K= 2, 7

L	FOBS	FCAL
-15	168	175
-14	115	121
-13	248	219
-12	133	124
-11	273	265
-10	160	166
-9	63	65
-8	253	258
-7	323	292
-6	302	330
-5	125	124
-4	126	115
-3	289	314
-2	379	356
-1	171	189
-0	615	608
1	323	320
2	347	349
3	112	98
4	187	205
5	459	453
6	138	144
7	70	69
8	173	178
9	242	247
10	303	277
11	178	185
12	85	73
13	179	160
14	103	136

H,K= 2, 8

L	FOBS	FCAL
-15	51	53
-14	33	14
-13	176	153
-12	138	130
-11	59	75
-10	114	95
-9	385	386
-8	255	241
-7	271	289
-6	99	88
-5	328	317
-4	166	181
-3	86	58
-2	191	206
-1	147	150
-0	409	456
1	181	183
2	238	249
3	417	420
4	168	161
5	31	26
6	354	328
7	63	85
8	200	205
9	122	124
10	29	58
11	202	195
12	164	155
13	56	75

H,K= 2, 9

L	FOBS	FCAL
-13	108	130
-12	158	132
-11	87	108
-10	160	175
-9	52	67
-8	126	153
-7	218	230
-6	63	69
-5	154	150
-4	200	198
-3	63	58
-2	248	235
-1	206	240
-0	157	160
1	218	255
2	229	217
3	134	129
4	363	344
5	290	274
6	107	96
7	53	59
8	146	114
9	125	137
10	289	277
11	85	101
12	43	66

H,K= 2, 10

L	FOBS	FCAL
-12	67	60
-11	162	147
-10	98	96
-9	48	23
-8	57	64
-7	219	234
-6	176	166
-5	97	114
-4	177	191
-3	219	255
-2	54	47
-1	76	64
-0	231	247
1	31	36
2	69	56
3	213	245
4	178	214
5	104	85
6	204	214
7	90	99
8	129	129
9	70	103
10	89	47
11	70	75

H,K= 2, 11

L	FOBS	FCAL
-10	29	36
-9	24	44
-8	109	116
-7	108	96
-6	56	54
-5	80	117
-4	29	18
-3	176	202
-2	148	154
-1	151	162
-0	94	101
1	66	48
2	134	137
3	87	80
4	248	256
5	210	200
6	85	84
7	57	59
8	92	93
9	56	39

H,K= 2, 12

L	FOBS	FCAL
-8	53	56
-7	48	43
-6	75	73
-5	134	122
-4	103	82
-3	151	175
-2	54	67
-1	47	62
-0	147	171
1	60	60
2	167	189
3	112	104
4	119	104
5	58	84
6	96	125
7	111	119

H,K= 2, 13

L	FOBS	FCAL
-4	53	45
-3	50	81
-2	83	90
-1	77	114
-0	71	103
1	69	97
2	62	83
3	60	40

H,K= 3, 0

L	FOBS	FCAL
-18	119	126
-17	215	190
-16	40	28
-15	100	72
-14	98	98
-13	289	282
-12	310	309
-11	100	93
-10	530	490
-9	534	500
-8	534	515
-7	718	734
-6	395	379
-5	418	433
-4	538	504
-3	546	528
-2	613	559
-1	135	78
-0	118	100
1	186	181
2	89	39
3	470	458
4	298	264
5	227	259
6	314	277
7	499	491
8	56	61
9	614	569
10	336	357
11	509	485
12	54	62
13	75	69
14	193	212
15	38	60
16	125	97

H,K= 3, 1

L	FOBS	FCAL
-18	213	191
-17	124	116
-16	183	170
-15	150	170
-14	69	38

-13	362	348
-12	362	350
-11	280	304
-10	488	463
-9	115	82
-8	653	660
-7	1184	1146
-6	108	125
-5	341	365
-4	487	480
-3	662	708
-2	815	767
-1	1269	1241
-0	398	391
1	1034	969
2	309	313
3	882	815
4	394	431
5	539	529
6	206	204
7	297	287
8	63	88
9	444	441
10	69	86
11	189	160
12	156	165
13	207	212
14	135	120
15	140	120
16	23	44

H,K= 3, 2

L	FOBS	FCAL
-18	97	99
-17	62	91
-16	48	23
-15	89	63
-14	138	136
-13	184	217
-12	70	49
-11	75	84
-10	134	122
-9	695	681
-8	592	586
-7	446	461
-6	378	317
-5	218	197
-4	226	223
-3	437	472
-2	387	365
-1	884	834
-0	369	387
1	517	553
2	343	383
3	374	368
4	304	304
5	222	222
6	553	591
7	323	361
8	401	376
9	159	159
10	149	150
11	234	224
12	120	113
13	96	87
14	190	168
15	151	143
16	102	110

H,K= 3, 3

L	FOBS	FCAL
-18	64	75
-17	144	134
-16	94	95
-15	130	129
-14	216	225
-13	77	78
-12	331	338
-11	196	218
-10	347	356
-9	139	148
-8	489	474
-7	572	559
-6	196	190
-5	277	292
-4	542	547
-3	109	90
-2	298	349
-1	162	133
-0	542	527
1	313	315
2	363	322
3	200	178
4	41	49
5	465	472
6	471	448
7	438	404
8	507	513
9	210	213
10	347	322
11	236	220
12	127	145
13	126	156
14	246	232
15	36	59
16	125	131

H,K= 3, 4

L	FOBS	FCAL
-17	216	220
-16	175	141
-15	69	67
-14	124	123
-13	175	141
-12	335	333
-11	104	90
-10	126	108
-9	221	232
-8	580	569
-7	413	452
-6	262	271
-5	337	342
-4	183	204
-3	158	155
-2	537	555
-1	417	410
-0	535	545
1	342	324
2	309	323
3	237	252
4	440	436
5	400	333
6	111	90
7	319	280
8	74	88
9	171	153
10	130	128
11	207	192
12	140	139
13	82	67
14	127	130
15	172	183
16	34	36

H,K= 3, 5

L	FOBS	FCAL
-17	83	69
-16	43	54
-15	236	241
-14	87	79
-13	91	103
-12	139	129
-11	141	146
-10	200	185
-9	360	351
-8	196	219
-7	164	179
-6	134	159
-5	70	54
-4	362	387
-3	566	548
-2	464	450
-1	516	553
-0	136	153
1	603	599
2	221	198
3	177	189
4	571	558
5	119	84
6	263	272
7	160	146
8	285	287
9	192	163
10	63	38
11	97	63
12	89	88
13	115	139
14	172	166
15	89	76

H,K= 3, 6

L	FOBS	FCAL
-16	126	135
-15	89	93
-14	68	70
-13	29	32
-12	209	217
-11	245	233
-10	141	167
-9	201	174
-8	190	201
-7	60	50
-6	335	358
-5	218	246
-4	141	157
-3	449	439
-2	382	380
-1	309	292
-0	351	392
1	275	265
2	307	278
3	189	168
4	174	147
5	268	270
6	114	114
7	420	394
8	63	52
9	89	91
10	141	177
11	239	225
12	90	83
13	46	38
14	128	135

H,K= 3, 7

L	FOBS	FCAL
-15	31	30
-14	58	81
-13	221	220
-12	97	99
-11	211	189
-10	321	342
-9	197	225
-8	307	311
-7	395	360
-6	44	39
-5	68	96
-4	186	224
-3	360	338
-2	257	258
-1	243	211
-0	515	515
1	347	358
2	382	391
3	424	444
4	92	82
5	254	241
6	220	245
7	145	151
8	232	223
9	299	299
10	127	98
11	114	93
12	96	89
13	153	149
14	41	37

H,K= 3, 8

L	FOBS	FCAL
-14	158	164
-13	154	167
-12	96	96
-11	91	103
-10	85	60
-9	257	271
-8	231	221
-7	105	110
-6	105	72
-5	104	137
-4	104	101
-3	82	98
-2	321	319
-1	141	132
-0	294	291
1	120	120
2	280	245
3	317	362
4	144	130
5	55	43
6	301	292
7	111	88
8	219	196
9	59	23
10	182	158
11	53	39
12	198	182
13	66	57

H,K= 3, 9

L	FOBS	FCAL
-13	46	69
-12	86	86
-11	114	127
-10	126	106
-9	166	186
-8	148	129
-7	144	162
-6	251	270
-5	230	257
-4	189	212
-3	141	178
-2	141	148
-1	114	79
-0	77	81
1	202	185
2	144	131
3	31	29
4	31	51
5	312	325
6	86	52
7	59	64
8	103	112
9	210	226
10	140	139
11	75	84
12	56	65

H,K= 3, 10

L	FOBS	FCAL
-12	54	48
-11	118	143
-10	26	10
-9	60	69
-8	69	77
-7	243	248
-6	115	102
-5	113	97
-4	180	170
-3	162	168
-2	87	80
-1	355	331
-0	179	194
1	0	43
2	81	88
3	68	63
4	143	149
5	175	178
6	175	189
7	176	176
8	45	39
9	59	46
10	44	20

H,K= 3, 11

L	FOBS	FCAL
-10	53	74
-9	98	110
-8	25	20
-7	78	66
-6	38	41
-5	68	58
-4	28	44
-3	99	90
-2	213	243
-1	64	79
-0	86	125
1	99	88
2	49	50
3	79	82
4	216	205
5	135	138
6	57	47
7	102	102
8	40	57

H,K= 3, 12

L	FOBS	FCAL
-7	57	84
-6	31	33
-5	106	122
-4	143	154
-3	108	126
-2	57	93
-1	167	166
-0	89	84
1	72	60
2	98	98
3	90	89
4	45	38
5	136	146
6	45	58

H,K= 3, 13

L	FOBS	FCAL
-3	95	65
-2	82	76
-1	90	127
-0	83	83
1	76	53

H,K= 4, 0

L	FOBS	FCAL
-18	142	153
-17	219	239
-16	100	95
-15	42	58
-14	119	123
-13	276	291
-12	109	84
-11	174	142
-10	283	290
-9	593	575
-8	682	646
-7	1057	1019
-6	327	302
-5	32	36
-4	175	156
-3	493	497
-2	307	310
-1	301	297
-0	288	312
1	114	141
2	808	798
3	40	66
4	113	89
5	36	27
6	341	349
7	344	334
8	316	337
9	423	432
10	267	251
11	203	230
12	156	164
13	194	185
14	185	201
15	55	22
16	16	16

H,K= 4, 1

L	FOBS	FCAL
-18	115	95
-17	141	124
-16	149	159
-15	282	263
-14	225	217
-13	129	138
-12	338	322
-11	131	164
-10	290	240
-9	168	183
-8	262	229
-7	945	915
-6	1070	986
-5	410	409
-4	138	123
-3	388	336
-2	262	253
-1	289	283
-0	289	316
1	468	478
2	1322	1278
3	73	60
4	709	684
5	356	356
6	186	165
7	269	216
8	288	264
9	44	79
10	161	170
11	269	237
12	209	190
13	231	220
14	102	106
15	0	17
16	120	135

H,K= 4, 2

L	FOBS	FCAL
-18	103	121
-17	54	67
-16	109	76
-15	164	177
-14	218	238
-13	315	326
-12	63	66
-11	184	160
-10	350	331
-9	303	321
-8	138	121
-7	145	137
-6	329	333
-5	614	565
-4	144	154
-3	158	159
-2	242	255
-1	178	165
-0	357	374
1	733	723
2	274	214
3	537	530
4	410	388
5	562	515
6	448	437
7	282	275
8	214	201
9	207	197
10	89	120
11	251	249
12	43	50
13	186	181
14	228	203
15	166	156

H,K= 4, 3

L	FOBS	FCAL
-17	84	81
-16	142	152
-15	40	15
-14	90	62
-13	93	73
-12	105	89
-11	298	310
-10	255	234
-9	66	73
-8	612	574
-7	512	508
-6	413	429
-5	61	55
-4	393	387
-3	318	320
-2	256	241
-1	329	328
-0	136	111
1	340	302
2	276	286
3	582	564
4	297	315
5	283	263
6	183	175
7	42	35
8	302	296
9	253	254
10	83	112
11	233	230
12	250	225
13	241	257
14	37	30
15	91	107

H,K= 4, 4

L	FOBS	FCAL
-17	144	142
-16	76	70
-15	55	46
-14	144	152
-13	97	95
-12	174	161
-11	45	32
-10	140	151
-9	327	355
-8	179	177
-7	341	342
-6	405	398
-5	583	606
-4	188	159
-3	334	295
-2	128	135
-1	708	687
-0	274	267
1	268	300
2	620	582
3	518	475
4	232	202
5	154	146
6	398	392
7	254	252
8	77	79
9	170	175
10	143	138
11	325	338
12	116	100
13	82	58
14	190	200
15	175	183

H,K= 4, 5

L	FOBS	FCAL
-16	60	88
-15	87	82
-14	49	68
-13	140	128
-12	190	161
-11	169	177
-10	100	122
-9	192	202
-8	75	58
-7	210	220
-6	41	48
-5	393	391
-4	259	220
-3	499	506
-2	183	182
-1	257	253
-0	612	601
1	127	149
2	527	526
3	195	188
4	211	167
5	196	187
6	199	148
7	117	62
8	187	194
9	205	200
10	173	169
11	180	163
12	226	222
13	193	191
14	171	161

H,K= 4, 6

L	FOBS	FCAL
-16	98	107
-15	94	96
-14	47	41
-13	49	44
-12	108	103
-11	87	86
-10	172	167
-9	216	195
-8	157	179
-7	239	236
-6	648	459
-5	84	97
-4	196	168
-3	208	244
-2	146	127
-1	145	154
-0	243	227
1	142	133
2	306	301
3	149	157
4	281	304
5	135	155
6	281	255
7	141	122
8	244	213
9	153	141
10	221	196
11	288	253
12	172	150
13	135	130
14	63	79

H,K= 4, 7

L	FOBS	FCAL
-15	92	104
-14	61	83
-13	238	223
-12	28	34
-11	60	64
-10	189	184
-9	199	215
-8	223	200
-7	383	357
-6	279	284
-5	108	66
-4	336	345
-3	216	215
-2	280	279
-1	68	84
-0	0	40
1	369	356
2	276	252
3	231	196
4	113	105
5	184	189
6	100	141
7	103	95
8	224	230
9	0	20
10	186	158
11	38	48
12	34	30
13	30	54

H,K= 4, 8

L	FOBS	FCAL
-14	87	125
-13	64	46
-12	133	135
-11	141	125
-10	141	140
-9	276	288
-8	134	132
-7	44	40
-6	160	197
-5	126	127
-4	32	13
-3	104	126
-2	104	122
-1	220	217
-0	191	212
1	70	95
2	0	80
3	350	347
4	44	42
5	44	53
6	144	150
7	43	59
8	145	122
9	112	119
10	52	39
11	63	55
12	35	15

H,K= 4, 9

L	FOBS	FCAL
-13	37	69
-12	40	22
-11	118	140
-10	55	78
-9	28	9
-8	66	49
-7	144	154
-6	250	250
-5	130	129
-4	183	174
-3	162	145
-2	62	51
-1	99	94
-0	218	231
1	54	58
2	172	166
3	151	167
4	43	45
5	249	290
6	41	16
7	217	195
8	60	42
9	182	157
10	38	54
11	21	47

H,K= 4, 10

L	FOBS	FCAL
-11	56	59
-10	121	145
-9	93	110
-8	66	53
-7	74	76
-6	218	215
-5	180	192
-4	66	68
-3	99	119
-2	204	212
-1	165	159
-0	90	74
1	152	199
2	129	129
3	77	66
4	171	177
5	48	37
6	152	162
7	217	214
8	33	51
9	67	61

H,K= 4, 11

L	FOBS	FCAL
-9	106	157
-8	39	41
-7	133	142
-6	100	95
-5	157	172
-4	165	190
-3	55	50
-2	62	65
-1	48	56
-0	39	43
1	175	207
2	76	89
3	142	119
4	44	113
5	124	118
6	88	97
7	47	20

H,K= 4, 12

L	FOBS	FCAL
-6	86	116
-5	91	94
-4	109	143
-3	56	45
-2	99	113
-1	94	79
-0	0	44
1	56	88
2	106	127
3	28	42
4	50	64

H,K= 5, 0

L	FOBS	FCAL
-17	272	224
-16	171	182
-15	40	31
-14	153	154
-13	193	175
-12	137	132
-11	77	81
-10	160	139
-9	42	43
-8	91	119
-7	117	153
-6	121	131
-5	85	115
-4	74	58
-3	760	756
-2	124	169
-1	76	65
-0	345	364
1	230	277
2	212	246
3	893	828
4	54	45
5	28	34
6	221	228
7	63	24
8	83	77
9	173	168
10	93	93
11	215	208
12	108	111
13	27	12
14	0	55
15	91	105

H,K= 5, 1

L	FOBS	FCAL
-17	22	28
-16	170	167
-15	272	266
-14	234	205
-13	354	345
-12	63	72
-11	63	53
-10	115	117
-9	131	140
-8	71	69
-7	332	320
-6	318	243
-5	189	181
-4	322	304
-3	361	328
-2	232	217
-1	179	142
-0	617	534
1	593	548
2	891	862
3	388	381
4	242	244
5	64	86
6	30	18
7	212	206
8	177	165
9	232	215
10	108	56
11	96	69
12	111	143
13	181	164
14	86	97
15	77	72

H,K= 5, 2

L	FOBS	FCAL
-17	88	95
-16	100	115
-15	105	89
-14	148	154
-13	147	174
-12	104	120
-11	214	199
-10	503	475
-9	117	90
-8	163	149
-7	69	65
-6	208	162
-5	527	533
-4	81	112
-3	331	321
-2	252	243
-1	437	433
-0	573	525
1	440	417
2	651	629
3	371	378
4	163	132
5	354	376
6	253	258
7	180	215
8	189	173
9	204	191
10	252	241
11	170	162
12	121	80
13	127	90
14	156	142

H,K= 5, 3

L	FOBS	FCAL
-17	53	54
-16	115	112
-15	160	156
-14	109	110
-13	238	238
-12	93	117
-11	199	191
-10	109	110
-9	254	253
-8	85	81
-7	197	198
-6	260	247
-5	339	354
-4	200	208
-3	164	155
-2	116	108
-1	311	264
-0	310	317
1	243	196
2	27	18
3	227	237
4	618	607
5	249	206
6	86	112
7	234	249
8	244	245
9	153	170
10	43	28
11	0	19
12	205	231
13	99	120
14	82	72

H,K= 5, 4

L	FOBS	FCAL
-16	51	65
-15	37	32
-14	298	294
-13	130	107
-12	31	32
-11	54	47
-10	331	355
-9	269	258
-8	215	223
-7	156	138
-6	611	619
-5	248	252
-4	190	180
-3	269	282
-2	72	71
-1	341	301
-0	215	208
1	185	203
2	544	544
3	382	369
4	94	63
5	53	49
6	377	388
7	167	137
8	172	204
9	160	163
10	160	151
11	226	207
12	76	66
13	219	199
14	77	104

H,K= 5, 5

L	FOBS	FCAL
-16	101	110
-15	66	83
-14	184	172
-13	244	213
-12	245	219
-11	53	67
-10	239	220
-9	89	85
-8	163	156
-7	320	310
-6	133	162
-5	220	231
-4	195	211
-3	396	411
-2	171	154
-1	305	313
-0	100	114
1	163	135
2	166	155
3	475	488
4	160	170
5	219	231
6	144	128
7	218	179
8	298	279
9	180	152
10	109	59
11	196	197
12	81	114
13	37	44

H,K= 5, 6

L	FOBS	FCAL
-15	110	98
-14	167	149
-13	132	123
-12	29	17
-11	120	113
-10	69	50
-9	185	170
-8	164	189
-7	44	61
-6	242	243
-5	208	217
-4	141	133
-3	268	253
-2	166	145
-1	245	250
-0	68	71
1	119	137
2	44	40
3	348	322
4	186	170
5	223	239
6	125	124
7	107	105
8	263	273
9	88	81
10	68	31
11	119	138
12	79	43

H,K= 5, 7

L	FOBS	FCAL
-14	49	58
-13	105	105
-12	157	142
-11	70	65
-10	30	41
-9	81	83
-8	178	184
-7	373	379
-6	71	64
-5	0	2
-4	194	200
-3	54	74
-2	63	66
-1	144	135
-0	89	88
1	218	251
2	216	212
3	223	237
4	172	210
5	146	177
6	153	178
7	141	143
8	167	135
9	292	274
10	122	123
11	69	77
12	113	126

H,K= 5, 8

L	FOBS	FCAL
-13	51	42
-12	152	129
-11	38	50
-10	164	168
-9	157	151
-8	127	131
-7	61	77
-6	292	285
-5	98	109
-4	77	94
-3	54	40
-2	63	86
-1	140	118
-0	63	67
1	153	160
2	197	208
3	299	304
4	241	255
5	113	96
6	214	198
7	142	64
8	119	137
9	183	181
10	67	72
11	128	124

H,K= 5, 9

L	FOBS	FCAL
-12	74	104
-11	112	135
-10	85	68
-9	151	159
-8	101	79
-7	163	180
-6	72	87
-5	112	150
-4	242	292
-3	53	37
-2	31	15
-1	75	76
-0	129	126
1	80	117
2	67	101
3	42	78
4	159	143
5	69	74
6	113	139
7	64	52
8	111	92
9	149	138

H,K= 5, 10

L	FOBS	FCAL
-10	116	159
-9	96	97
-8	0	10
-7	146	164
-6	183	209
-5	56	72
-4	147	177
-3	153	135
-2	221	223
-1	118	160
-0	95	103
1	102	85
2	115	130
3	216	197
4	141	136
5	151	183
6	104	45
7	55	46
8	32	28

H,K= 5, 11

L	FOBS	FCAL
-8	61	102
-7	55	72
-6	51	70
-5	203	235
-4	191	222
-3	36	33
-2	77	102
-1	68	88
-0	63	107
1	252	254
2	78	93
3	94	104
4	178	172
5	48	37

H,K= 5, 12

L	FOBS	FCAL
-4	69	66
-3	59	95
-2	55	63
-1	74	116
-0	38	38
1	61	51

H,K= 6, 0

L	FOBS	FCAL
-17	178	209
-16	24	33
-15	81	87
-14	103	91
-13	85	71
-12	44	43
-11	44	19
-10	32	22
-9	117	106
-8	0	4
-7	0	50
-6	133	155
-5	260	265
-4	274	289
-3	67	52
-2	55	36
-1	447	428
-0	278	267
1	40	42
2	29	44
3	478	466
4	406	416
5	0	21
6	269	246
7	122	134
8	77	79

9	87	79
10	60	37
11	63	60
12	53	62
13	46	33

H,K= 6, 1

L	FOBS	FCAL
-17	68	69
-16	191	170
-15	54	61
-14	176	177
-13	67	72
-12	31	21
-11	31	64
-10	63	36
-9	272	270
-8	126	165
-7	99	97
-6	0	15
-5	128	139
-4	280	323
-3	114	102
-2	379	389
-1	62	50
-0	282	269
1	183	181
2	322	344
3	186	190
4	253	258
5	433	410
6	294	288
7	273	262
8	221	207
9	206	205
10	51	52
11	126	105
12	133	112
13	45	64

H,K= 6, 2

L	FOBS	FCAL
-16	111	101
-15	132	149
-14	106	77
-13	51	27
-12	126	138
-11	232	240
-10	122	125
-9	224	210
-8	31	35
-7	238	239
-6	432	449
-5	41	61
-4	350	355
-3	268	234
-2	429	411
-1	49	19
-0	367	393
1	220	210
2	412	412
3	322	314
4	193	184
5	357	348
6	181	169
7	178	170
8	54	42
9	193	196
10	199	203
11	136	94
12	68	36
13	53	42

H,K= 6, 3

L	FOBS	FCAL
-16	120	126
-15	44	43
-14	194	185
-13	120	109
-12	213	219
-11	0	27
-10	151	133
-9	89	96
-8	328	296
-7	157	151
-6	68	112
-5	252	260
-4	321	319
-3	0	16
-2	158	145
-1	192	202
-0	312	330
1	121	146
2	172	171
3	61	87
4	551	553
5	183	181
6	167	161
7	358	365
8	53	26
9	109	101
10	76	59
11	174	163
12	213	189
13	51	65

H,K= 6, 4

L	FOBS	FCAL
-16	69	58
-15	72	77
-14	141	140
-13	64	63
-12	59	53
-11	150	177
-10	326	319
-9	141	147
-8	63	77
-7	83	88
-6	309	313
-5	86	92
-4	74	92
-3	458	477
-2	191	217
-1	90	71
-0	100	86
1	132	143
2	276	280
3	173	168
4	104	68
5	373	366
6	386	360
7	227	226
8	516	485
9	114	111
10	84	107
11	82	60
12	88	71

H,K= 6, 5

L	FOBS	FCAL
-15	47	45
-14	108	116
-13	191	183
-12	149	142
-11	184	189
-10	150	144
-9	116	106
-8	269	286
-7	192	201
-6	272	277
-5	121	122
-4	224	228
-3	264	242
-2	274	251
-1	358	363
-0	128	106
1	227	225
2	308	289
3	318	315
4	105	116
5	172	134
6	131	123
7	110	105
8	318	315
9	182	157
10	248	239
11	200	183
12	19	56

H,K= 6, 6

L	FOBS	FCAL
-14	129	155
-13	92	90
-12	78	63
-11	179	177
-10	198	188
-9	209	208
-8	115	98
-7	125	139
-6	109	121
-5	170	174
-4	207	203
-3	144	138
-2	54	64
-1	283	284
-0	216	201
1	122	88
2	192	216
3	154	169
4	62	81
5	31	33
6	288	323
7	42	12
8	222	200
9	159	140
10	50	53
11	164	160

H,K= 6, 7

L	FOBS	FCAL
-14	62	104
-13	73	88
-12	114	108
-11	27	33
-10	160	142
-9	192	203
-8	60	77
-7	137	136
-6	119	134
-5	54	75
-4	272	284
-3	198	203
-2	44	26
-1	94	106
-0	109	103
1	125	93
2	172	207
3	31	30
4	129	134
5	127	107
6	123	135
7	297	286
8	59	58
9	153	129
10	74	61

H,K= 6, 8

L	FOBS	FCAL
-12	36	45
-11	60	77
-10	206	190
-9	198	224
-8	117	139
-7	220	234
-6	166	163
-5	186	196
-4	61	79
-3	86	79
-2	97	112
-1	0	29
-0	61	44
1	53	24
2	242	235
3	178	172
4	116	138
5	157	139
6	86	86
7	77	62
8	71	68
9	158	148

H,K= 6, 9

L	FOBS	FCAL
-11	71	87
-10	76	111
-9	136	141
-8	78	105
-7	94	116
-6	139	141
-5	172	174
-4	95	98
-3	58	71
-2	41	80
-1	119	101
-0	104	91
1	0	39
2	98	92
3	135	134
4	170	178
5	0	26
6	87	110
7	135	136
8	105	120

H,K= 6, 10

L	FOBS	FCAL
-9	33	21
-8	103	140
-7	40	34
-6	96	108
-5	105	128
-4	26	33
-3	160	186
-2	184	198
-1	88	84
-0	83	77
1	82	92
2	36	49
3	136	119
4	56	80
5	117	120
6	159	198

H,K= 6, 11

L	FOBS	FCAL
-6	44	45
-5	89	129
-4	120	169
-3	30	47
-2	48	73
-1	82	106
-0	36	42
1	96	134
2	39	37

H,K= 7, 0

L	FOBS	FCAL
-16	114	97
-15	81	81
-14	154	145
-13	142	169
-12	30	5
-11	150	177
-10	69	44
-9	32	24
-8	109	105
-7	268	306
-6	382	380
-5	44	27
-4	105	62
-3	272	275
-2	0	0
-1	222	235
-0	61	61
1	247	268
2	44	62
3	365	383
4	32	27
5	164	150
6	231	204
7	162	168
8	90	67
9	91	63
10	67	43
11	44	46
12	75	51

H,K= 7, 1

L	FOBS	FCAL
-16	98	91
-15	72	66
-14	65	21
-13	28	11
-12	126	124
-11	140	136
-10	222	255
-9	274	282
-8	448	403
-7	109	106
-6	213	210
-5	102	101
-4	307	310
-3	146	160
-2	221	234
-1	74	82
-0	198	179
1	206	235
2	54	95
3	89	75
4	264	253
5	70	60
6	112	85
7	75	71
8	112	115
9	90	96
10	181	173
11	90	97
12	145	154

H,K= 7, 2

L	FOBS	FCAL
-16	76	98
-15	106	104
-14	69	63
-13	89	93
-12	83	71
-11	192	184
-10	44	23
-9	210	201
-8	89	69
-7	406	398
-6	236	238
-5	88	82
-4	265	244
-3	232	238
-2	126	120
-1	134	141
-0	294	273
1	269	247
2	159	160
3	134	140
4	63	81
5	201	197
6	69	99
7	114	112
8	236	204
9	303	278

H,K= 7, 3

L	FOBS	FCAL
-15	95	62
-14	127	122
-13	145	148
-12	138	135
-11	95	64
-10	0	35
-9	215	209
-8	31	56
-7	314	323
-6	44	45
-5	355	342
-4	321	313
-3	82	60
-2	240	226
-1	347	341
-0	121	146
1	99	85
2	169	211
3	402	416
4	280	270
5	128	148
6	97	58
7	440	374
8	153	131
9	138	143
10	131	112
11	63	43

H,K= 7, 4

L	FOBS	FCAL
-15	145	169
-14	92	97
-13	153	156
-12	63	85
-11	195	192
-10	90	92
-9	247	235
-8	186	171
-7	160	135
-6	218	225
-5	223	219
-4	229	220
-3	302	305
-2	104	98
-1	99	100
-0	206	215
1	296	299
2	216	211
3	130	118
4	239	214
5	344	332
6	68	93
7	178	140
8	187	203
9	70	89
10	104	100
11	69	56

H,K= 7, 5

L	FOBS	FCAL
-14	88	108
-13	66	74
-12	146	151
-11	178	156
-10	242	250
-9	60	91
-8	216	225
-7	93	71
-6	88	61
-5	332	338
-4	137	141
-3	100	109
-2	173	160
-1	170	168
-0	173	167
1	54	52
2	271	250
3	69	56
4	355	359
5	30	29
6	0	36
7	240	233
8	129	123
9	181	156
10	48	52

H,K= 7, 6

L	FOBS	FCAL
-13	36	46
-12	75	103
-11	27	26
-10	172	180
-9	58	72
-8	51	76
-7	145	180
-6	165	168
-5	148	135
-4	103	86
-3	120	112
-2	31	18
-1	31	28
-0	88	108
1	102	115
2	258	253
3	43	41
4	42	57
5	92	50
6	232	202
7	147	125
8	134	94
9	58	62

H,K= 7, 7

L	FOBS	FCAL
-12	61	71
-11	73	68
-10	90	105
-9	98	97
-8	102	113
-7	243	262
-6	189	195
-5	52	43
-4	198	216
-3	61	69
-2	30	35
-1	96	70
-0	95	92
1	73	94
2	145	127
3	148	142
4	102	133
5	190	201
6	218	187
7	74	86
8	143	118

H,K= 7, 8

L	FOBS	FCAL
-11	40	38
-10	145	166
-9	61	63
-8	82	105
-7	115	129
-6	196	191
-5	75	85
-4	0	4
-3	29	23
-2	57	51
-1	86	103
-0	107	116
1	141	135
2	121	120
3	125	124
4	91	66
5	76	60
6	290	293
7	76	80

H,K= 7, 9

L	FOBS	FCAL
-9	54	67
-8	84	105
-7	121	137
-6	84	101
-5	73	105
-4	74	84
-3	37	55
-2	84	90
-1	121	135
-0	52	28
1	45	29
2	91	94
3	134	112
4	102	67
5	19	46

H,K= 7, 10

L	FOBS	FCAL
-6	18	40
-5	63	77
-4	48	69
-3	96	118
-2	145	161
-1	82	85
-0	74	41
1	157	159
2	44	66
3	35	54

H,K= 8, 0

L	FOBS	FCAL
-15	143	154
-14	149	138
-13	26	5
-12	126	100
-11	29	42
-10	84	103
-9	101	89
-8	102	100
-7	227	202
-6	540	532
-5	189	228
-4	312	267
-3	32	26
-2	55	45
-1	0	21
-0	109	112
1	315	324
2	70	77
3	120	122
4	81	112
5	96	92
6	0	3
7	153	137
8	169	152
9	152	156
10	37	42

H,K= 8, 1

L	FOBS	FCAL
-15	40	51
-14	51	48
-13	81	65
-12	73	81
-11	0	33
-10	42	39
-9	337	375
-8	102	90
-7	187	197
-6	113	135
-5	339	323
-4	291	252
-3	63	71
-2	55	63
-1	164	163
-0	95	71
1	130	163
2	153	149
3	155	188
4	119	122
5	105	145
6	128	129
7	268	252
8	269	233
9	36	28
10	118	138

H,K= 8, 2

L	FOBS	FCAL
-14	80	72
-13	67	63
-12	130	150
-11	214	213
-10	190	204
-9	193	198
-8	231	248
-7	221	222
-6	153	168
-5	196	219
-4	32	52
-3	202	183
-2	126	134
-1	63	67
-0	206	202
1	228	225
2	0	27
3	97	70
4	61	74
5	112	129
6	276	256
7	222	202
8	187	176
9	189	174
10	212	208

H,K= 8, 3

L	FOBS	FCAL
-13	116	121
-12	84	79
-11	110	120
-10	170	180
-9	96	114
-8	208	212
-7	61	40
-6	254	242
-5	81	79
-4	256	269
-3	320	321
-2	193	204
-1	233	252
-0	197	199
1	210	209
2	61	64
3	52	42
4	186	206
5	70	75
6	107	128
7	52	51
8	153	157
9	71	85

H,K= 8, 5

L	FOBS	FCAL
-13	50	57
-12	39	43
-11	72	80
-10	27	38
-9	84	76
-8	174	168
-7	51	53
-6	112	117
-5	245	257
-4	74	63
-3	91	105
-2	114	146
-1	195	195
-0	61	71
1	43	59
2	59	39
3	50	55
4	153	184
5	0	22
6	141	110
7	213	183
8	128	105

H,K= 8, 6

L	FOBS	FCAL
-12	29	28
-11	44	38
-10	90	104
-9	108	85
-8	139	175
-7	159	151
-6	146	143
-5	87	116
-4	106	142
-3	110	99
-2	110	119
-1	0	29
-0	71	67
1	58	49
2	260	254
3	0	28
4	85	71
5	109	130
6	110	89
7	66	40

H,K= 8, 7

L	FOBS	FCAL
-11	0	13
-10	50	29
-9	62	66
-8	0	31
-7	101	86
-6	76	65
-5	113	97
-4	104	113
-3	40	53
-2	63	51
-1	112	106
-0	104	107
1	47	23
2	107	113
3	58	81
4	186	142
5	39	33
6	57	74

H,K= 8, 8

L	FOBS	FCAL
-9	65	104
-8	21	19
-7	0	22
-6	131	143
-5	62	81
-4	81	89
-3	0	29
-2	77	86
-1	44	54
-0	44	54
1	97	93
2	101	94
3	44	66
4	33	41

H,K= 8, 9

L	FOBS	FCAL
-6	81	118
-5	57	94
-4	50	76
-3	0	31
-2	121	139
-1	78	55
-0	87	88
1	87	89
2	90	111

H,K= 9, 0

L	FOBS	FCAL
-13	0	153
-12	41	25
-11	150	136
-10	27	50
-9	338	324
-8	273	282
-7	118	136
-6	256	268
-5	182	268
-4	336	359
-3	255	242
-2	106	60
-1	0	19
-0	30	19
1	0	8
2	0	24
3	108	124
4	266	228
5	106	115
6	45	39
7	34	26
8	100	73

H,K= 9, 1

L	FOBS	FCAL
-13	104	112
-12	52	54
-11	44	64
-10	121	108
-9	196	207
-8	144	136
-7	169	181
-6	30	40
-5	236	194
-4	175	149
-3	172	192
-2	129	105
-1	177	195
-0	109	119
1	52	92
2	42	38
3	115	106
4	156	136
5	0	13
6	26	26
7	53	66
8	143	147

H,K= 9, 2

L	FOBS	FCAL
-13	67	101
-12	59	66
-11	154	171
-10	65	72
-9	73	87
-8	191	200
-7	136	124
-6	196	172
-5	112	123
-4	30	40
-3	257	255
-2	171	177
-1	202	197
-0	194	194
1	162	171
2	0	24
3	117	121
4	68	84
5	113	121
6	158	168
7	50	34
8	82	84

H,K= 9, 3

L	FOBS	FCAL
-12	88	80
-11	121	126
-10	238	257
-9	47	81
-8	28	41
-7	75	85
-6	196	191
-5	339	345
-4	228	215
-3	136	141
-2	186	184
-1	139	140
-0	72	71
1	41	62
2	64	96
3	125	131
4	27	36
5	124	109
6	64	63
7	87	92

H,K= 9, 4

L	FOBS	FCAL
-12	96	129
-11	100	97
-10	24	35
-9	81	58
-8	163	198
-7	151	157
-6	155	161
-5	76	92
-4	111	116
-3	166	172
-2	171	178
-1	246	249
-0	169	177
1	0	4
2	39	49
3	71	80
4	93	79
5	236	222
6	84	79
7	40	48

H,K= 9, 5

L	FOBS	FCAL
-11	28	40
-10	71	75
-9	110	117
-8	44	75
-7	83	85
-6	27	43
-5	99	106
-4	200	213
-3	88	136
-2	247	254
-1	147	150
-0	78	44
1	184	187
2	88	74
3	123	130
4	139	105
5	75	66
6	29	19

H,K= 9, 6

L	FOBS	FCAL
-10	46	95
-9	71	79
-8	110	142
-7	34	26
-6	152	139
-5	126	157
-4	90	84
-3	177	164
-2	79	69
-1	87	85
-0	51	56
1	146	156
2	85	97
3	68	49
4	40	49
5	26	26

H,K= 9, 7

L	FOBS	FCAL
-8	22	7
-7	47	67
-6	30	32
-5	71	85
-4	107	132
-3	33	52
-2	89	89
-1	134	145
-0	45	56
1	57	56
2	120	114
3	61	56

H,K= 9, 8

L	FOBS	FCAL
-4	22	40
-3	0	79
-2	80	88
-1	39	6

H,K= 10, 0

L	FOBS	FCAL
-11	46	48
-10	56	90
-9	0	11
-8	72	74
-7	82	78
-6	0	5
-5	220	236
-4	156	201
-3	73	54
-2	0	22
-1	134	129
-0	27	50
1	27	10
2	131	133
3	43	55
4	144	108
5	144	131

H,K= 10, 1

L	FOBS	FCAL
-11	22	16
-10	97	105
-9	52	23
-8	51	95
-7	97	131
-6	0	15
-5	0	17
-4	164	155
-3	67	57
-2	95	105
-1	119	130
-0	123	137
1	95	99
2	162	149
3	69	83
4	0	13
5	33	29

H,K= 10, 2

L	FOBS	FCAL
-10	72	107
-9	100	120
-8	88	93
-7	77	70
-6	190	213
-5	75	77
-4	38	65
-3	138	147
-2	132	127
-1	60	49
-0	142	145
1	93	123
2	91	74
3	111	135
4	104	125
5	139	146

H,K= 10, 3

L	FOBS	FCAL
-10	47	37
-9	74	86
-8	40	24
-7	136	170
-6	36	51
-5	134	166
-4	26	60
-3	126	115
-2	222	232
-1	178	181
-0	112	110
1	56	51
2	101	107
3	124	99
4	95	87

H,K= 10, 4

L	FOBS	FCAL
-9	62	81
-8	114	141
-7	39	45
-6	33	43
-5	95	115
-4	75	68
-3	207	205
-2	138	133
-1	90	72
-0	135	133
1	46	56
2	96	118
3	41	65

H,K= 10, 5

L	FOBS	FCAL
-7	67	84
-6	88	95
-5	31	53
-4	22	37
-3	39	33
-2	78	97
-1	106	131
-0	89	79
1	67	67
2	110	136

H,K= 10, 6

L	FOBS	FCAL
-5	53	91
-4	90	124
-3	104	129
-2	41	62
-1	69	92

H,K= 11, 0

L	FOBS	FCAL
-7	20	36
-6	56	74
-5	72	96
-4	152	193
-3	0	13
-2	121	104
-1	95	91
-0	117	108
1	52	44

H,K= 11, 1

L	FOBS	FCAL
-6	72	85
-5	62	88
-4	44	38
-3	20	16
-2	92	118
-1	97	112
-0	48	64

H,K= 11, 2

L	FOBS	FCAL
-5	55	60
-4	18	53
-3	45	67
-2	0	17
-1	58	49

References

(1) Work done under the auspices of the U. S. Atomic Energy Commission.

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(8) Ferrichrome, $C_{27}H_{42}N_9O_{12}Fe$, differs from ferrichrome-A (Fig. 1) in that H replaces each $HOCH_2$- (i. e., residues of glycine instead of serine) and $-CH_3$ replaces each $-CHC(CH_3)CH_2COOH$ (i. e., residues of acetic acid instead of methylglutaconic acid).[5]

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Figure Captions

Figure 1. Ferrichrome-A

Figure 2. Projection of ferrichrome-A molecule down the cryallographic b axis.

Figure 3. Schematic molecular formulae of the subgroups in ferrichrome-A, to be used in identifying the distances and angles in Tables III, IV, and V.

Figure 4. Absolute configuration about the iron atom in ferrichrome-A.

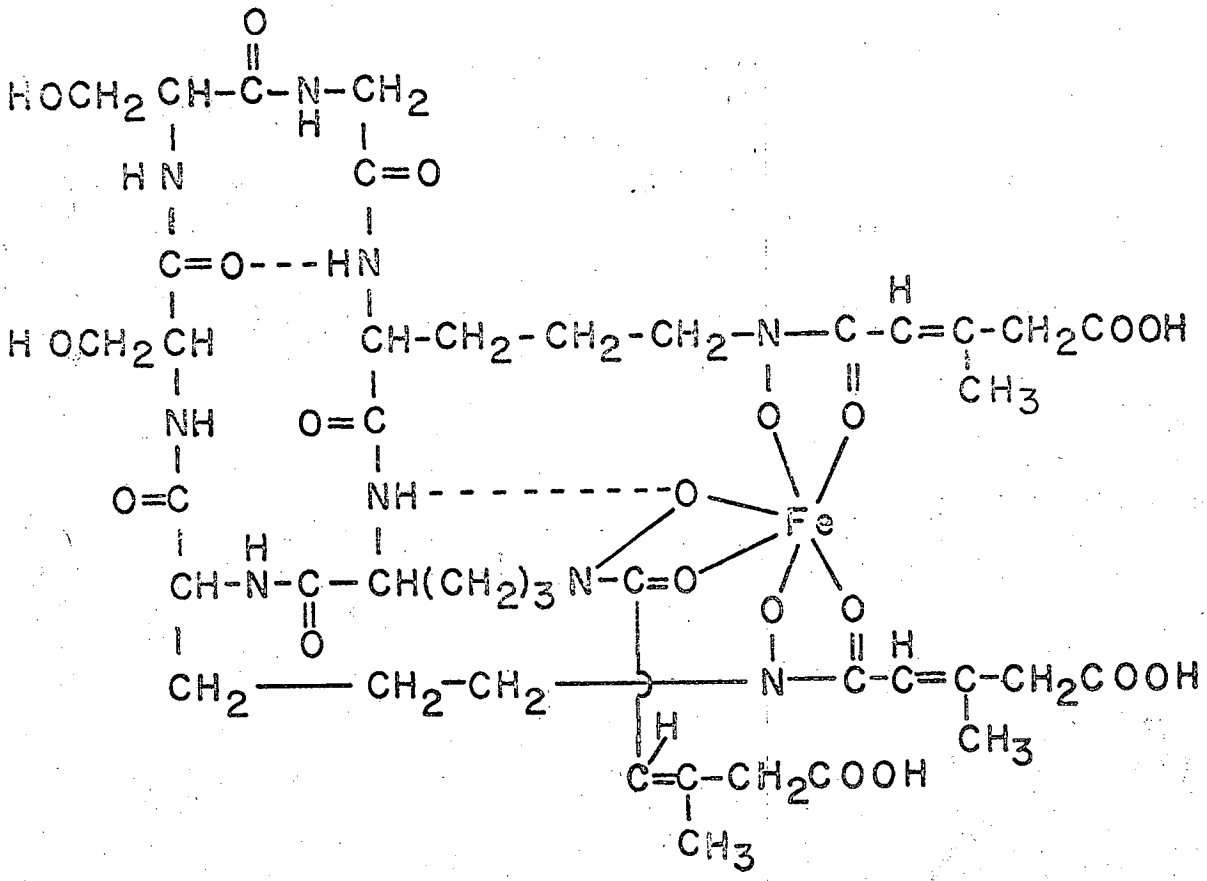


Fig. 1

Fig 1 Zalkin, Forrester & Templeton

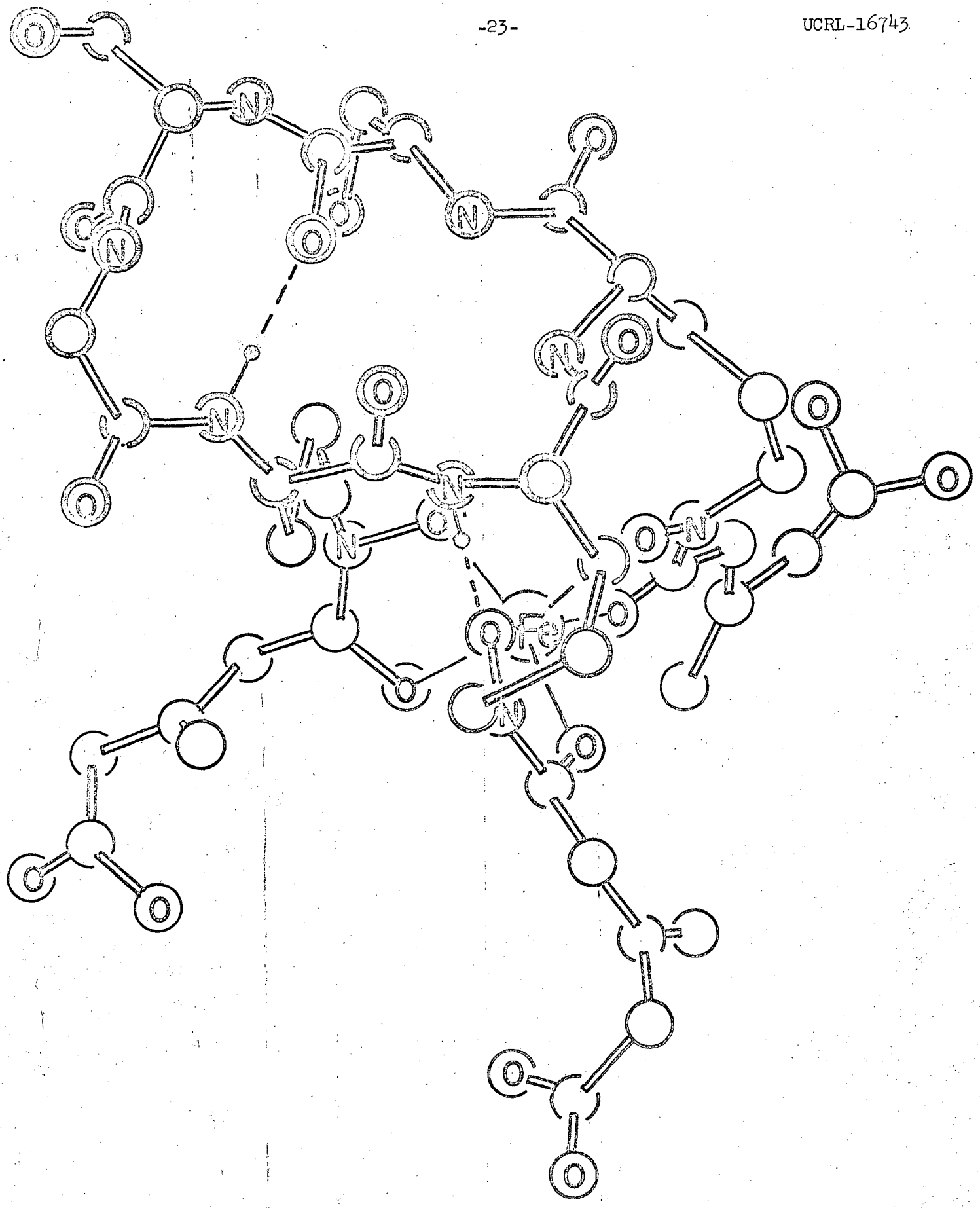
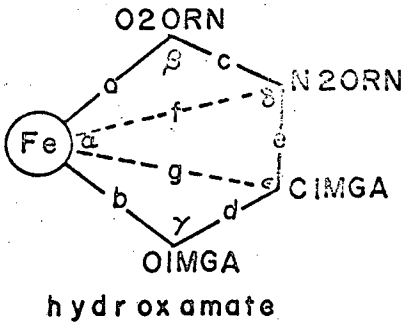
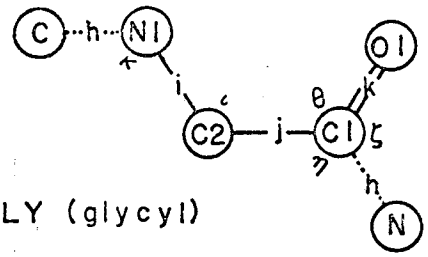


Fig. 2

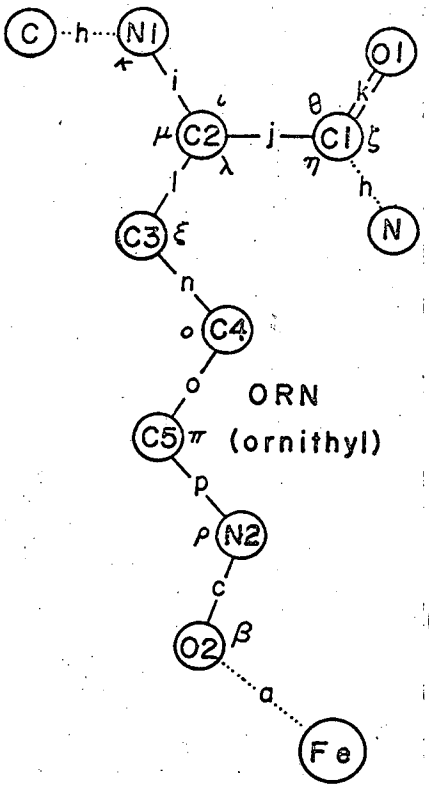
Ferrichrysin Tentative Formula



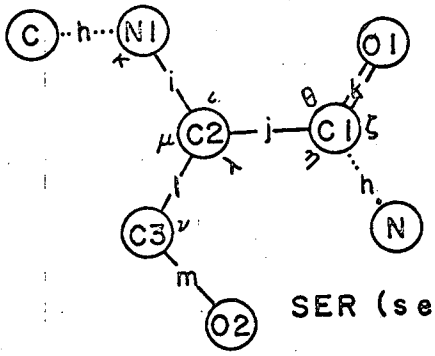
hydroxamate



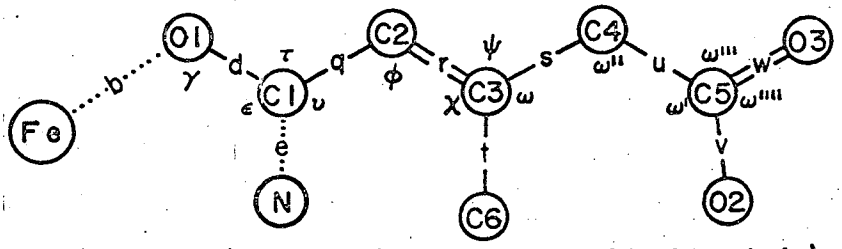
GLY (glycyl)



ORN (ornithyl)



SER (seryl)



MGA(trans(β-methyl)glutaconic acid side chain)

MUB-6463

Fig. 3

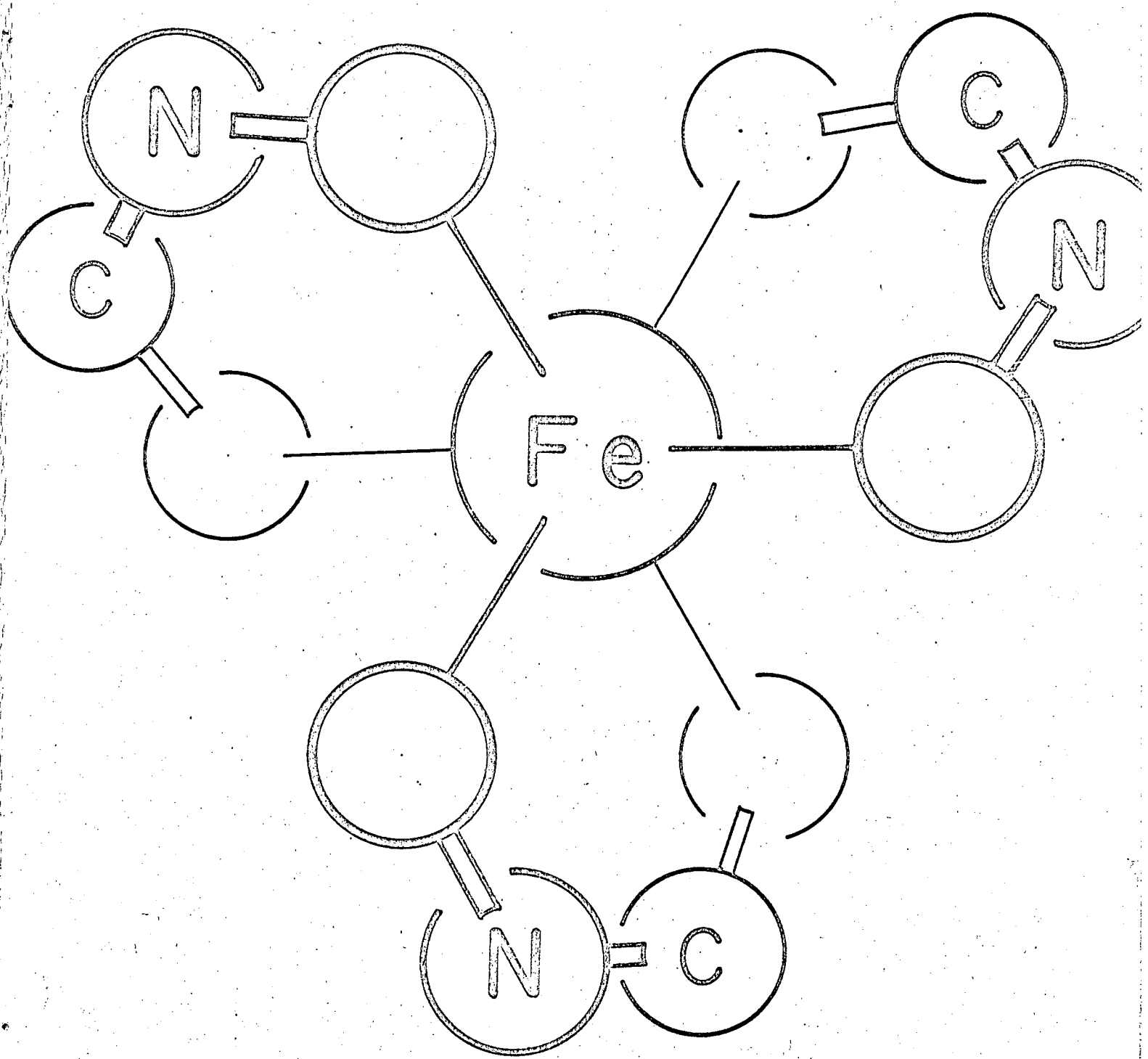


Fig. 4

Fig 4 ZALKIN, FORRESTER, & TEMPLETON.