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Carbon Compounds of the Transition Metals, VI;*
The Reaction Product of Azobenzene with Iron Carbonyls: the Structure of μ-N, N'-dehydrosemidinatobis (tricarbonyliron).

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The structure of μ-N,N'-dehydrosemidinatobis (tricarbonyliron) has been determined in the solid at room temperature. The red crystals are triclinic with $a = 8.59 \pm 0.02$, $b = 13.92 \pm 0.03$, $c = 7.85 \pm 0.02$ *Å*, $\alpha = 92°14' \pm 15'$, $\beta = 93°24' \pm 15'$ *and* $\gamma = 90°24' \pm 15'$. *Two* $Fe(CO)_3$ *units are bridged by the o-semidine skeleton which is formed during synthesis of the complex from azobenzene and iron carbonyls. In the iron-nitrogen tetrahedron so formed, the Fe-Fe distance is 2.37 Å and the Fe-N distance 2.00 Å. The nitrogen atoms are no longer bonded together in the complex.*

Introduction

The preparation of a number of complexes from iron carbonyls and organic molecules, which contain at least one nitrogen atom linked by a double bond to either carbon or nitrogen, has been reported[1] and their structures have been shown to correspond to at least three different types.[2,3] The reaction with azobenzene yields a complex of stoicheiometry $C_6H_5NNC_6H_5.Fe_2(CO)_6$ in contrast to the complexes of azobenzene with platinum, palladium[4] and nickel[5] in which there is a loss of one hydrogen atom per azobenzene/metal unit. The iron complex is particularly interesting since reduction by lithium aluminium hydride yields *o*-semidine whereas the product was hydrazobenzene for the platinum and palladium complexes whilst the nickel complex yielded azobenzene. The present investigation was undertaken to determine whether the ligand is present in the azobenzene or the *o*-semidine form and also the mode of attachment of the organo-ligand. We show that the *o*-semidine skeleton is already preformed in the complex, Fig. 1.

Experimental Section

Crystal data. $C_{18}H_{10}N_2O_6Fe_2$ (M=462.0), m.p. 163-164°, triclinic, $a = 8.59 \pm 0.02$, $b = 13.92 \pm 0.03$, $c = 7.85 \pm 0.02$ Å, $\alpha = 92°14' \pm 15'$, $\beta = 93°24' \pm 15'$, $\gamma = 90°24' \pm 15'$, $U = 937$ Å^3, $D_m = 1.65$ g.cm.$^{-3}$ by flotation in aq. $AgNO_3$, $Z = 2$, $D_c = 1.655$ g.cm.$^{-3}$, space-group $P1$ (C_i^1, No. 2). For Mo-K_α radiation ($\lambda = 0.7107$ Å), $\mu = 16.45$ cm.$^{-1}$.

The complex, which occurs as dark red crystals, was kindly supplied by Professor Pauson and was recrystallised from petroleum ether (60/80) at 0°. The unit-cell dimensions were determined by the precession method. The spec-group was assumed to be centrosymmetric, $P1$, and this choice is justified by the refinement of the structure.

Collection of Intensity Data. The crystal used was of approximate dimensions 0.3×0.2×0.2 mm. Intensities were estimated visually from the following precession photographs taken with molybdenum radiation; *nkl* and *hkn*, = 0, 1,2. Of the 1749 independent reflexions thus accessible, 236 were too weak to be measured. After Lorentz and polarisation corrections had been applied, the inter-level scale factors were evaluated by the method of Hamilton, Rollett and Sparks.[6] No correction was made for absorption.

Structure Determination and Refinement. The metal atom coordinates were obtained from the three-dimensional Patterson function and two successive Fourier syntheses of the approximations to the electron density, based on the phases derived from the partially derived structures, were sufficient to reveal the complete molecule except for hydrogen atoms. In these Fourier summations we used a weighting function similar to that proposed by Woolfson.[7] The structure, assigned arbitrary values for the isotropic temperature factors of 3 Å^2 for Fe and 4 Å^2 for the light atoms, gave an initial value of 26% for $R\ (=\Sigma||F_o|-|F_c||/\Sigma|F_o|)$ which decreased rapidly and smoothly during the course of full-matrix least-squares refinement to a final value of 7.9% for the 1513 observed reflexions. A difference synthesis showed all the hydrogen atoms expected for the structure, and these have been included

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(*) Part V in this series: O. S. Mills and J. P. Nice, *J. Organometallic Chem.*, in the press.

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(3) D. Bright and O. S. Mills, *Chem. Comm.*, 245 (1967).

(4) A. C. Cope and R. W. Siekman, *J. Amer. Chem. Soc.*, *87*, 3272 (1965).

(5) J. P. Kleiman and M. Dubeck, *J. Amer. Chem. Soc.*, *85*, 1544 (1963).

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in the final structure-factor calculations. For the purposes of the least-squares refinement, the iron atoms were treated anisotropically and the light atoms isotropically. The hydrogen atom parameters were not refined.

The function minimised was $\Sigma w(|F_o|-|F_c|)^2$ where $w = 1/(1+F_o^2/12F_{min}^2)$. In the case of the unobserved reflexions, contributions were made to the least squares totals only when $|F_c|>F_{min}$; in these cases ΔF was set equal to $\pm F_{min}-F_c$ and a weight of 0.5 applied. This arbitrarily adjusted weight brought the average value of $w\Delta F^2$ for the unobserved reflexions into line with the corresponding values for groups of observed reflexions arranged according to increasing value of F_o. In the final cycle of refinement, the maximum coordinate shift was less than 12% of the estimated standard deviation for the shift.

All calculations were performed on the Atlas Computer installed in the University of Manchester and made use of programmes written by members of our group.[8] Scattering factors used were those listed in International Tables.[9]

Table I gives the final atomic parameters and Table II the relevant molecular geometry. In Table III we list the intermolecular contacts which are less than 3.5 Å.

Table I. Atomic parameters

Atom	x/a	x/b	z/c	B(Å)
Fe 1	0.2250(2)	0.3728(1)	0.3168(2)	
Fe 2	0.4064(2)	0.2905(1)	0.1493(2)	
C 1	0.5448(14)	0.3784(8)	0.0959(17)	2.99(21)
O 1	0.6367(13)	0.4344(7)	0.0693(15)	4.93(21)
C 2	0.4445(18)	0.2055(10)	−0.0255(22)	4.34(28)
O 2	0.4688(15)	0.1498(8)	−0.1289(18)	6.56(28)
C 3	0.5422(15)	0.2507(8)	0.3104(18)	3.20(22)
O 3	0.6321(13)	0.2296(7)	0.4156(15)	4.82(21)
C 4	0.0230(17)	0.3988(9)	0.3472(24)	3.95(26)
O 4	−0.1035(15)	0.4146(8)	0.3656(20)	6.22(27)
C 5	0.2986(15)	0.4913(8)	0.3325(18)	3.44(23)
O 5	0.3472(13)	0.5698(7)	0.3425(15)	5.29(23)
C 6	0.2866(16)	0.3603(8)	0.5334(19)	3.34(23)
O 6	0.3343(14)	0.3527(7)	0.6738(16)	5.33(23)
C 7	0.0974(13)	0.2985(7)	0.0058(16)	2.58(19)
C 8	−0.0116(15)	0.3085(8)	−0.1288(19)	3.48(23)
C 9	−0.1263(18)	0.2366(9)	−0.1534(21)	4.34(28)
C 10	−0.1266(17)	0.1589(9)	−0.0536(20)	3.97(26)
C 11	−0.0146(15)	0.1495(8)	0.0857(19)	3.49(23)
C 12	0.0977(12)	0.2214(6)	0.1076(15)	2.21(17)
C 13	0.2243(14)	0.1517(7)	0.3581(17)	2.72(19)
C 14	0.3113(15)	0.0701(8)	0.3223(18)	3.11(21)
C 15	0.3091(16)	−0.0059(8)	0.4275(19)	3.74(25)
C 16	0.2314(16)	−0.0026(8)	0.5764(19)	3.65(24)
C 17	0.1468(17)	0.0796(9)	0.6144(20)	4.19(27)
C 18	0.1407(16)	0.1553(8)	0.5062(19)	3.75(25)
N 1	0.2207(12)	0.3643(6)	0.0644(14)	2.63(16)
N 2	0.2175(10)	0.2314(5)	0.2463(12)	1.95(14)

The quantities enclosed in brackets are the estimated standard deviations estimated from the inverted least-squares matrix of the final cycle of refinement and are in units of the last place of decimals given. The anisotropic temperature coefficients for the iron atoms are

Fe1 $b_{11}= 0.00887(23)$ $b_{22}= 0.00319(7)$ $b_{33}= 0.00800(28)$
$b_{12}= -0.00039(10)$ $b_{13}= -0.00062(42)$ $b_{23}= -0.00037(12)$

Fe2 $b_{11}= 0.00709(20)$ $b_{22}= 0.00366(8)$ $b_{33}= 0.00806(28)$
$b_{12}= -0.00043(10)$ $b_{13}= 0.00009(38)$ $b_{23}= 0.00080(13)$

where expression for the temperature factor is

$$\exp-(b_{11}h^2+b_{22}k^2+b_{33}l^2+2b_{12}hk+2b_{13}hl+2b_{23}kl)$$

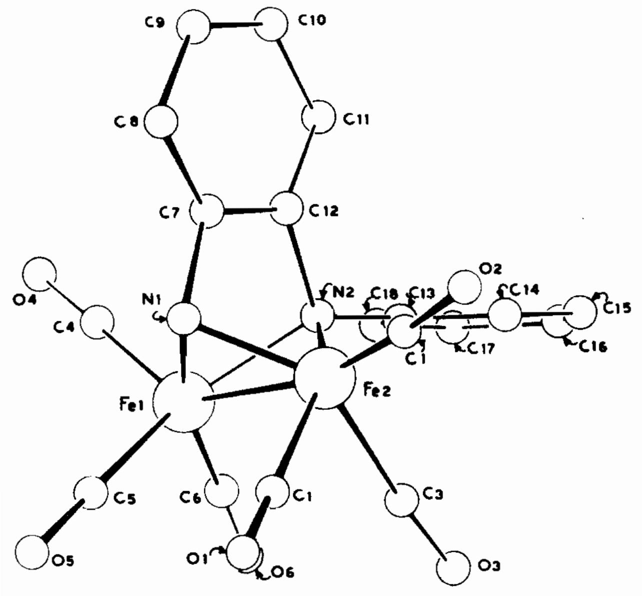
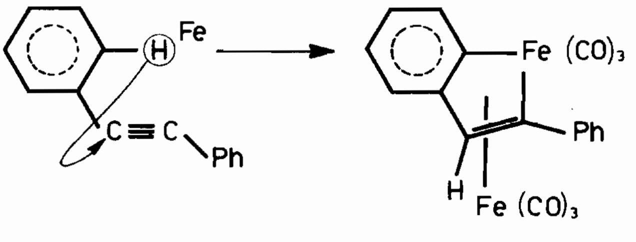


Figure 1. Molecular configuration and numbering scheme.

Results and Discussion

The crystal structure consists of discrete molecules of the kind shown in Fig. 1. The molecule already contains the *o*-semidine skeleton which bridges symmetrically, via the nitrogen atoms, the two iron atoms. It is thus μ-N,N′-dehydrosemidinatobis(tricarbonyliron). It may alternatively be considered as a heterocyclic substituted benzvalene which emphasizes the presence of the bicyclo [2,1,1]-system. It follows that at some stage of the complex formation rupture of the —N=N— bond occurs with accompanying transfer of a proton from the *ortho* position of one of the phenyl rings to the nitrogen atom attached to that ring. Examples are already known of related migrations in complexes which involve the $Fe_2(CO)_6$ unit. However in these cases the complex so formed contains a five-membered heterocycle π-bonded, at least in part, to a $Fe(CO)_3$ fragment. Thus diphenylacetylene yields[10] the phenyl derivative of tricarbonyl(ferraindenyl)-π-(tricarbonyliron).[11]



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(8) We thank Professor Kilburn and the staff of the Computing Machine Laboratory for the excellent service received. The programmes used were written by a number of co-workers. We thank Drs. L. I. Hodgson, F. S. Stephens, A. D Redhouse and J. P. Nice for their considerable help.

(9) International Tables for X-ray Crystallography, vol. III, Kynoch Press (1962).

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(11) M. Van Meerssche, P. Piret, J. Meunier-Piret and Y. Degreve, *Bull. Soc. Chim. Belg.*, *73*, 824 (1964).

Table II. Molecular geometry

Intramolecular distances with estimated standard deviations

	Å		(Å)
Fe1-Fe2	2.372(2)	C6-O6	1.162(17)
Fe1-C4	1.803(15)	C7-C8	1.381(17)
Fe1-C5	1.760(12)	C7-C12	1.363(15)
Fe1-C6	1.766(15)	C7-N1	1.438(14)
Fe1-N1	1.979(11)	C8-C9	1.399(18)
Fe1-N2	2.024(7)	C9-C10	1.359(19)
Fe2-C1	1.779(12)	C10-C11	1.424(19)
Fe2-C2	1.822(16)	C11-C12	1.383(15)
Fe2-C3	1.777(14)	C12-N2	1.455(14)
Fe2-N1	1.999(9)	C13-C14	1.393(15)
Fe2-N2	2.017(9)	C13-C18	1.401(18)
C1-O1	1.139(15)	C13-N2	1.441(14)
C2-O2	1.131(17)	C14-C15	1.368(18)
C3-O3	1.145(14)	C15-C16	1.380(20)
C4-O4	1.127(18)	C16-C17	1.392(18)
C 5-O5	1.165(14)	C17-C18	1.378(19)

Intramolecular angles with their estimated standard deviations

Fe2-Fe1-N1	53.8(3)	C9-C10-C11	121.4(12)
Fe2-Fe1-N2	53.9(3)	C10-C11-C12	116.0(12)
C4-Fe1-C5	98.4(6)	C7-C12-C11	122.0(11)
C4-Fe1-C6	97.8(8)	C7-C12-N2	111.4(9)
C5-Fe1-C6	88.6(6)	C11-C12-N2	126.4(11)
N1-Fe1-N2	73.0(4)	C14-C13-C18	118.8(11)
Fe1-Fe2-N1	53.0(3)	C14-C13-N2	121.6(11)
Fe1-Fe2-N2	54.2(2)	C18-C13-N2	119.7(9)
C1-Fe2-C2	95.8(6)	C13-C14-C15	119.8(13)
C1-Fe2-C3	89.2(6)	C14-C15-C16	122.2(12)
C2-Fe2-C3	100.4(6)	C15-C16-C17	118.0(12)
N1-Fe2-N2	72.8(4)	C16-C17-C18	120.9(15)
Fe2-C1-O1	176.8(3)	C13-C18-C17	120.1(12)
Fe2-C2-O2	177.1(14)	Fe1-N1-Fe2	73.2(4)
Fe2-C3-O3	176.6(10)	Fe1-N1-C7	107.5(8)
Fe1-C4-O4	179.6(16)	Fe2-N1-C7	109.5(6)
Fe1-C5-O5	179.8(13)	Fe1-N2-Fe2	71.9(3)
Fe1-C6-O6	176.8(15)	Fe1-N2-C12	105.8(5)
C8-C7-C12	122.3(10)	Fe1-N2-C13	126.7(7)
C8-C7-N1	127.8(10)	Fe2-N2-C12	107.0(7)
C12-C7-N1	109.9(10)	Fe2-N2-C13	123.9(7)
C7-C8-C9	116.7(12)	C12-N2-C13	114.0(8)
C8-C9-C10	121.5(14)		

Best least-squares planes through atomic positions.

In the following equations the quantities X, Y, Z refer to orthogonal coordinates obtained by the transformation

$$\begin{vmatrix} X \\ Y \\ Z \end{vmatrix} = \begin{vmatrix} a & b.\cos\gamma & c.\cos\beta \\ O & b & -c.\sin\beta.\cos\alpha^* \\ O & O & c.\sin\beta.\sin\alpha^* \end{vmatrix} \begin{vmatrix} x/a \\ y/b \\ z/c \end{vmatrix}$$

(i) Plane defined by C7, C8, C9, C10, C11, N1 and N2

$$0.6452X - 0.4717Y - 0.6010Z + 1.492 = O$$

Distances of atoms from plane (Å),

C7	0.025	C8	0.009	C9	−0.031	C10	−0.002
C11	0.004	C12	0.043	N1	−0.013	N2	−0.034

(ii) Plane defined by C13, C14, C15, C16, C17 and C18

$$-0.7697X - 0.3822Y - 0.5114Z + 3.542 = O$$

Distances of atoms from plane (Å),

C13	0.002	C14	−0.020	C15	0.022
C16	−0.006	C17	−0.012	C18	0.013

(iii) Direction cosine of Fe1 to Fe2 vector with respect to the orthogonal axes

0.6939, −0.4610, −0.5532

(iv) Angle between the normals to the *o*-diamine skeleton, plane(i), and the phenyl group, plane(ii), =89°20′.

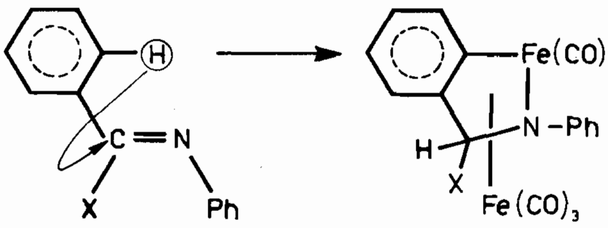
(c) Angle between the normal to the *o*-diamine skeleton, plane(i), and the Fe1-Fe2 vector, =3°58′.

Table III. Intermolecular contacts less than 3.5 Å

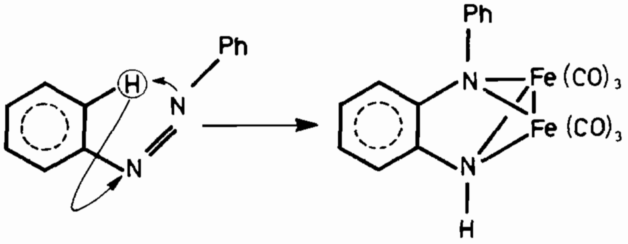
	(Å)		(Å)
C1 .... O1(I)	3.31	O3 .... O4(II)	3.47
O1 .... O1(I)	3.15	O3 .... C16(V)	3.38
O1 .... O4(II)	3.15	C4 .... O4(VI)	3.41
O1 .... C5(I)	3.43	O4 .... C5(VI)	3.23
O1 .... O5(I)	3.24	O4 .... O5(IV)	3.20
O1 .... N1(I)	3.28	O5 .... C6(IV)	3.36
C2 .... O6(III)	3.29	O5 .... O6(IV)	2.95
O2 .... O6(III)	3.45	O6 .... C8(VII)	3.49
O2 .... C10(II)	3.49	O6 .... N1(VII)	3.27
O2 .... C17(III)	3.44	C15 ... C15(V)	3.41
O3 .... O5(IV)	3.31		

The Roman numerals contained in parenthese indicate that the contact is made to an atom related, by one of the operations given below, to the one whose coordinates (x, y, z) are tabulated in Table 1. I: 1—x, 1—y, —z; II: 1+x, y, z; III: x, y, z—1; IV: 1—x, 1—y, 1—z; V: 1—x, —y, 1—z; VI: —x, 1—y, 1—z; VII: x, y, 1+z.

A related pathway is possible for the Schiff base complexes.[1,2]



Such a path is presumeably not the one involved in the azobenzene reaction since there remains no evidence for ring-metal bonding. The presence of the metal carbonyl must however promote the migration of the second nitrogen to the *ortho* position.



Although no symmetry is required of the molecule by spacegroup considerations, the molecule possesses idealised *m* symmetry. Further, the portion of the molecule which excludes the phenyl substituent has almost *mm*2 (C_{2v}) symmetry. Thus the two $Fe(CO)_3$ groups, which are equivalent within this degree of idealisation, are eclipsed. A 36-electron configuration for the iron atoms can be rationalised on the assumption that each nitrogen acts as a three-electron donor and that a metal-metal bond occurs. Indeed the Fe-Fe distance of 2.37 Å is considerably shorter than previously reported values, eg. $Fe_2(CO)_9$[12] (2.46 Å), $(C_5H_5)_2(CO)_4Fe_2$[13] (2.49 Å), $(CH_3COH)_2(CO)_6Fe_2$[14] (2.49 Å,

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(12) H. M. Powell and R. V. G. Ewens, *J. Chem. Soc.*, 286 (1939).
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(14) A. A. Hock and O. S. Mills, *Acta Cryst.*, *14*, 139 (1961).

Table 4 Observed and calculated structure factors

The four columns in each group contain the values of *k*, *l*, F_o, F_c reading from left to right. Each group is headed by the common value of *h*.

11 *k* *l*

-5	1	10.1	9.9
-4	1	10.4	-9.2
-4	2	4.6	-4.8
-3	2	8.8	12.0
-2	1	12.1	13.5
0	0	14.6	-14.9
1	0	0.0	-2.4
1	1	12.7	12.6
2	0	7.5	7.9
3	0	7.4	-9.6
4	-1	11.9	-10.9
4	-2	9.5	-8.5
3	-2	13.7	14.6
2	-1	11.0	11.5
2	-2	5.2	-5.9
1	-1	9.5	-10.0
1	-2	7.4	6.6
0	-1	7.0	7.6
0	-2	9.2	8.6
-1	-1	9.6	10.7
-1	-2	5.3	-4.4
-2	-1	7.6	-5.4
-2	-2	9.7	-9.6

10 *k* *l*

-8	2	10.0	10.5
-7	0	0.0	-0.6
-7	1	9.6	11.2
-6	0	8.8	8.4
-6	2	7.6	-9.5
-5	0	11.9	-12.9
-5	1	0.0	-4.3
-5	2	14.3	12.5
-4	0	9.0	9.3
-4	1	15.2	16.0
-3	0	13.3	11.2
-3	1	8.2	-9.3
-3	2	13.0	-11.2
-2	0	20.2	-20.5
-2	1	8.2	-6.6
-2	2	9.5	6.9
-1	0	9.1	10.4
-1	1	18.4	12.0
0	0	10.7	11.9
0	1	11.6	-9.8
1	0	15.2	-15.6
1	1	0.0	0.5
2	0	0.0	2.5
3	0	0.0	3.8
3	2	15.5	13.3
5	0	7.8	-6.2
5	1	10.2	-10.5
6	0	7.7	-5.5
7	0	12.3	13.0
6	-2	6.0	-1.1
5	-2	11.0	-6.1
4	-1	0.0	0.6
4	-2	13.7	13.1
3	-1	8.2	8.6
3	-2	9.2	-7.4
2	-1	14.2	-14.0
2	-2	7.8	-8.0
1	-1	5.7	5.0
1	-2	17.7	17.5
0	-1	22.7	24.3
0	-2	0.0	-2.0
-1	-1	16.8	-17.2
-1	-2	13.8	-14.5
-2	-1	15.4	-15.7
-3	-1	11.6	11.2
-3	-2	11.3	11.2
-5	-2	7.7	-4.3

9 *k* *l*

-10	0	5.2	-7.0
-10	2	17.8	15.0
-9	1	12.6	12.4
-8	0	7.6	6.4
-8	1	15.2	14.2
-8	2	15.4	-14.7
-7	0	12.0	-17.6
-7	1	11.6	-11.6
-7	2	17.5	17.5
-6	0	13.9	10.5
-6	1	20.1	20.8
-5	0	10.0	9.6
-5	1	14.2	-13.9
-5	2	0.0	-3.7
-4	0	21.4	-20.6
-4	1	0.0	-0.5
-3	0	12.2	13.5
-3	1	11.5	10.1
-2	0	0.0	4.2
-2	1	11.5	-10.1
-2	2	0.0	0.9
-1	0	20.9	-22.1
-1	1	0.0	-1.4
-1	2	15.4	-13.1
0	0	22.0	21.4
0	1	12.8	9.5
0	2	0.0	4.4
1	0	13.0	11.4
1	1	17.5	8.8
1	2	10.7	8.6
2	0	7.5	-6.3
2	1	8.1	-6.5
2	2	16.5	-15.2
3	0	9.7	-8.2
3	1	18.8	-14.6
3	2	14.0	6.3
4	0	7.9	-5.6
4	1	16.8	18.0
4	2	7.8	8.6
5	0	25.2	25.6
6	0	11.2	-8.5
7	0	9.0	-9.4
7	2	15.6	13.5
8	0	17.6	17.6
9	0	5.4	-4.9
10	0	7.4	5.4
11	-2	10.0	-7.7
10	-2	10.8	8.1
9	-1	13.8	13.8
9	-2	7.7	7.2
8	-2	12.9	-11.9
7	-1	19.5	-17.3
6	-1	11.6	11.6
6	-2	11.3	9.2
4	-1	11.5	-10.6
4	-2	0.0	-1.3
3	-2	7.9	-5.7
2	-1	0.0	-2.7
2	-2	15.0	14.8
1	-1	18.4	17.2
1	-2	0.0	-1.5
0	-1	15.7	-14.3
0	-2	12.3	-15.2
-1	-1	18.4	-16.0
-1	-2	26.4	25.6
-2	-1	15.8	15.1
-2	-2	0.0	1.4
-3	-1	16.6	-14.9
-3	-2	10.6	-11.1
-4	-2	15.1	16.4
-5	-1	17.1	16.7
-5	-2	0.0	-1.6
-6	-1	8.2	-7.8

8 *k* *l*

-12	2	14.2	14.1
-11	1	3.8	7.8
-11	2	14.2	-13.8
-10	0	0.0	-2.9
-10	1	18.2	-16.5
-10	2	11.0	-8.3
-9	0	15.9	-15.6
-9	1	0.0	6.0
-9	2	17.5	17.3
-8	1	18.2	15.0
-8	2	9.6	-7.2
-7	0	7.9	9.7
-7	1	10.1	-12.2
-6	0	25.7	-25.8
-5	0	20.9	21.5
-5	1	11.3	6.6
-5	2	0.0	2.5
-4	0	0.5	3.5
-4	1	11.2	-10.3
-3	0	10.5	-11.7
-3	1	0.0	-5.0
-3	2	11.3	-8.7
-2	0	0.0	-4.2
-2	2	14.4	12.8
-1	0	0.0	2.8
-1	1	0.0	2.4
-1	2	0.0	8.8
0	0	13.5	9.8
0	1	0.0	0.9
0	2	19.1	-20.0
1	0	0.0	-2.7
1	1	0.0	-6.2
1	2	15.9	14.5
2	0	0.0	-5.3
2	1	21.8	22.3
2	2	11.7	9.5
3	0	6.7	10.1
3	1	7.9	-4.3
3	2	26.1	-26.6
4	0	17.3	-16.9
5	0	15.7	-14.5
6	0	25.0	25.0
7	0	3.2	-2.4
7	2	0.0	-1.4
8	0	13.2	-12.5
9	0	15.1	15.5
10	0	7.8	-2.6
11	0	0.0	-3.3
11	2	7.7	-6.3
12	2	11.6	15.0
12	-2	9.1	-9.0
11	-1	6.0	-7.1
11	-2	0.0	5.2
10	-1	13.5	15.2
10	-2	7.9	5.9
9	-2	23.3	-26.3
8	-1	17.9	-19.8
7	-1	21.2	21.8
7	-2	23.1	20.5
6	-1	11.4	13.5
6	-2	12.3	-11.2
5	-1	25.4	-27.5
4	-1	7.5	9.1
4	-2	0.0	0.6
3	-1	9.6	9.8
3	-2	0.0	1.5
2	-1	0.0	-2.6
2	-2	0.0	-0.7
1	-1	0.0	0.7
1	-2	14.3	-13.9
0	-1	0.0	2.0
0	-2	24.2	23.7
-1	-1	0.0	4.3
-2	-1	10.7	-9.6
-2	-2	25.2	-22.3
-3	-1	0.0	-2.3
-3	-2	19.1	17.2
-4	-1	27.6	29.0
-4	-2	11.8	-9.6
-5	-1	19.8	-17.2
-5	-2	26.6	-25.5
-6	-1	8.0	-5.4
-6	-2	19.5	17.8
-7	-1	21.6	21.3
-7	-2	0.0	3.2
-8	-1	10.2	-7.7

7 *k* *l*

-14	2	13.4	14.1
-13	1	13.6	13.4
-12	1	13.4	-10.4
-11	0	15.6	-16.2
-11	1	0.0	-0.8
-11	2	17.9	9.2
-10	0	17.8	16.6
-10	1	8.2	6.7
-10	2	7.9	-9.2
-9	0	0.0	-6.2
-9	1	18.2	-15.9
-8	0	22.9	-24.5
-8	1	12.6	11.2
-8	2	12.1	12.4
-7	0	17.2	17.4
-7	1	15.8	15.2
-6	0	17.9	13.2
-6	1	11.0	-11.0
-5	0	19.2	-23.6
-5	1	0.0	0.5
-5	2	0.0	-4.8
-4	0	0.0	0.3
-4	1	0.0	-5.5
-4	2	7.1	7.6
-3	0	9.2	7.5
-3	1	11.2	11.0
-3	2	0.0	1.8
-2	0	9.5	11.5
-2	1	16.0	-16.6
-2	2	21.1	-21.4
-1	0	8.3	-10.0
-1	1	27.3	-23.4
-1	2	20.3	21.9
0	0	[illegible]	-6.1
0	1	13.9	14.7
0	2	37.4	33.8
1	0	36.3	37.2
1	1	11.0	12.6
1	2	23.7	-23.9
2	0	25.6	-25.8
2	1	48.1	-40.5
2	2	0.0	-0.1
3	0	19.7	-19.7
3	1	19.1	18.9
3	2	0.0	4.6
4	0	31.3	33.5
5	0	25.3	-26.1
6	0	0.0	-5.4
7	0	14.2	15.1
8	0	0.0	-4.8
9	0	7.4	-7.3
9	2	14.6	-12.9
10	0	0.0	-3.1
10	1	0.0	-1.2
10	2	18.8	19.1
11	0	7.8	9.1
11	1	17.9	12.8
11	2	9.6	-6.4
12	0	7.3	5.4
12	1	19.8	-19.9
12	2	8.4	-6.9
13	0	13.3	-14.4
13	2	16.4	16.7
14	0	9.6	10.2
14	1	14.2	14.9
14	-1	10.5	-10.0
11	-2	12.4	10.2
10	-2	25.8	-25.0
9	-1	16.0	-16.5
9	-2	[illegible]	5.1
8	-1	12.1	11.0
8	-2	21.5	21.1
7	-1	14.3	13.0
7	-2	24.1	-21.2
6	-1	26.2	-27.5
6	-2	7.5	5.0
5	-1	19.2	22.7
5	-2	10.2	11.5
4	-1	23.1	22.2
4	-2	14.5	-17.0
3	-1	22.7	-23.3
3	-2	0.0	0.1
2	-1	8.6	9.5
2	-2	4.8	5.0
1	-1	6.6	8.2
1	-2	4.8	4.3
0	-1	13.3	-11.8
0	-2	14.8	-12.9
-1	-1	0.0	2.0
-1	-2	11.2	-12.5
-2	-1	16.1	12.0
-2	-2	13.3	14.1
-3	-1	11.8	9.2
-3	-2	8.1	-6.1
-4	-1	17.0	-16.0
-4	-2	32.5	-38.9
-5	-1	10.5	-8.5
-5	-2	25.8	28.0
-6	-1	26.5	26.9
-6	-2	10.3	5.4
-7	-1	9.6	-8.1
-7	-2	20.5	-18.8
-8	-1	19.4	-17.3
-8	-2	7.6	7.8
-9	-1	23.6	22.7
-10	-1	10.0	-11.4

6 *k* *l*

-15	2	7.4	-8.5
-14	1	13.7	-13.9
-14	2	9.7	-7.5
-13	0	7.3	-9.0
-13	2	14.7	14.5
-12	0	19.3	19.6
-12	1	14.2	14.1
-11	0	0.0	0.6
-11	1	18.2	-19.7
-11	2	6.2	-9.9
-10	0	22.6	-24.7
-10	1	0.0	-3.1
-10	2	7.7	-7.4
-9	0	22.7	23.5
-9	1	17.7	15.5
-8	0	0.0	7.2
-8	1	15.5	-12.1
-8	2	0.0	4.3
-7	0	23.5	-22.9
-7	1	0.0	0.7
-6	0	16.7	15.1
-6	1	10.4	9.5
-5	0	0.0	-6.6
-5	1	12.4	11.0
-5	2	0.0	5.1
-4	0	0.0	-2.0
-4	1	0.0	2.1
-4	2	34.9	-38.3
-3	0	22.2	-23.2
-3	1	26.2	-30.8
-3	2	29.7	30.0
-2	0	6.8	-2.9
-2	1	13.8	[illegible]
-2	2	11.9	4.5
-1	0	11.7	11.0
-1	1	15.3	-13.5
-1	2	45.5	-48.0
0	0	24.8	-28.7
0	1	43.5	-46.6
0	2	19.2	16.2
1	0	0.0	-7.1
1	1	49.5	50.2
1	2	24.5	24.0
2	0	52.1	53.7
2	1	0.0	-3.6
2	2	18.7	-13.6
3	0	21.9	-25.6
4	0	13.3	-13.0
5	0	19.1	19.0
6	0	16.3	-14.3
7	0	13.7	13.5
8	0	15.7	-14.2
8	2	20.5	21.1
9	0	0.0	-0.8
9	1	12.5	12.7
9	2	16.5	-16.8
10	0	0.0	5.8
10	1	13.0	-13.0
11	0	17.5	-10.3
11	1	0.0	4.8
11	2	10.0	10.0
12	0	19.1	19.4
12	1	16.6	11.9
12	2	7.5	-6.8
13	0	0.0	1.0
13	1	16.5	-12.3
14	0	15.1	-16.0
15	0	12.5	12.5
15	-1	10.6	-10.8
14	-1	15.0	-17.0
14	-2	10.9	10.3
13	-1	17.2	17.6
12	-1	8.2	-8.7
11	-1	0.0	-2.5
11	-2	9.6	-6.5
10	-2	10.8	8.7
9	-1	7.9	-7.9
9	-2	15.4	14.8
8	-1	14.2	12.8
8	-2	11.1	-13.2
7	-1	15.7	-7.5
7	-2	11.6	-4.4
6	-1	7.4	8.7
6	-2	11.3	13.5
5	-1	23.4	25.2
5	-2	26.4	-30.2
4	-1	50.4	-55.3
4	-2	8.1	-7.0
3	-1	25.2	21.3
3	-2	32.5	32.5
2	-1	25.7	25.7
2	-2	19.0	-15.9
1	-1	21.4	-24.3
1	-2	11.4	16.6
0	-1	20.8	16.5
0	-2	0.0	2.8
-1	-1	17.6	21.4
-1	-2	0.0	-2.8
-2	-1	21.5	-22.7
-2	-2	3.4	2.2
-3	-1	7.0	0.9
-3	-2	19.4	-18.2
-4	-1	33.1	-31.6
-4	-2	33.9	36.7
-5	-1	10.0	16.3
-5	-2	0.0	-1.8
-6	-1	7.4	-4.4
-6	-2	19.8	-20.6
-7	-1	7.6	-6.9
-7	-2	25.2	25.6
-8	-1	25.8	25.7
-8	-2	10.4	9.4
-9	-1	9.7	-6.9
-9	-2	25.2	-25.6
-10	-1	21.5	-21.1
-10	-2	7.7	8.0
-11	-1	15.3	14.4

5 *k* *l*

-16	1	9.7	-7.7
-15	0	10.2	-8.2
-15	1	11.2	11.4
-15	2	10.6	12.2
-14	0	13.1	13.0
-14	1	12.8	12.1
-14	2	12.4	-12.3
-13	1	20.3	-20.5
-12	0	17.8	-19.1
-12	1	14.1	13.8
-12	2	9.6	5.5
-11	0	17.9	17.5
-11	1	8.1	4.7
-11	2	5.4	-5.4
-10	0	0.0	5.0
-10	1	17.2	-8.8
-10	2	5.1	-7.8
-9	0	10.2	-10.6
-9	2	22.5	-21.3
-8	0	10.5	7.6
-8	1	0.0	-3.5
-8	2	22.2	23.2
-7	0	10.0	4.2
-7	1	25.6	25.9
-7	2	0.0	-4.0
-6	0	14.6	10.4
-6	1	17.9	-10.0
-6	2	34.5	-35.1
-5	0	16.4	-11.9
-5	1	30.9	-29.8
-5	2	35.2	35.0
-4	0	26.9	-27.4
-4	1	31.8	34.8
-4	2	5.5	-6.3
-3	0	37.2	38.1
-3	1	17.8	-16.9
-3	2	39.2	-43.1
-2	0	53.4	-55.2
-2	1	0.0	-5.4
-2	2	20.7	21.1
-1	0	15.7	13.6
-1	1	55.5	54.0
-1	2	16.0	14.4
0	0	38.2	31.8
0	1	0.0	-5.9
0	2	0.0	-6.1
1	0	53.8	-52.5
1	1	38.4	-37.3
1	2	0.0	-0.8
2	0	8.1	-7.0
2	1	17.3	15.0
2	2	7.0	-4.6
3	0	24.9	24.0
4	0	14.2	11.1
5	0	7.5	6.7
6	0	14.5	-13.5
7	0	21.3	20.7
7	1	24.3	25.4
7	2	34.7	-35.5
8	0	0.0	3.1
8	1	34.4	-32.9
8	2	0.0	-5.5
9	0	22.5	-24.7
9	1	0.0	0.3
9	2	15.2	18.9
10	0	10.4	12.4
10	1	16.1	16.6
10	2	10.5	-11.7
11	0	7.7	3.4
11	1	10.5	-9.3
12	0	24.0	-23.3
12	1	5.8	5.0
13	0	18.8	19.2
13	2	0.0	0.6
14	0	0.0	-2.5
14	1	8.2	-7.3
15	0	14.0	-13.1
15	-1	7.9	-7.4
15	-2	15.0	15.9
14	-1	23.2	23.7
13	-1	12.0	-14.4
13	-2	20.6	-21.0
12	-1	18.6	-17.7
12	-2	16.0	12.5
11	-1	27.8	27.5
11	-2	0.0	1.7
10	-1	9.2	-9.3
10	-2	11.1	-10.1
9	-1	9.8	8.2
8	-1	15.7	12.3
8	-2	0.0	0.0
7	-1	16.1	-11.7
7	-2	31.8	19.1
6	-1	13.9	12.9
6	-2	17.8	-17.0
5	-1	21.6	-31.0
5	-2	6.0	-4.7
4	-1	0.0	0.0
4	-2	12.0	46.7
3	-1	31.3	31.1
3	-2	22.4	-23.0
2	-1	43.5	-41.3
2	-2	23.3	22.1
1	-1	10.2	15.1
1	-2	26.4	26.3
0	-1	39.4	38.6
0	-2	49.6	-41.5
-1	-1	62.0	-65.3
-1	-2	10.0	-7.2
-2	-1	30.7	30.4
-2	-2	27.7	25.9
-3	-1	32.2	32.2
-3	-2	7.6	-6.1
-4	-1	24.4	-23.2
-4	-2	15.9	16.0
-5	-1	0.0	4.3
-5	-2	21.3	-22.8
-6	-1	7.0	-5.4
-6	-2	23.4	22.7
-7	-1	16.0	15.7
-7	-2	13.6	12.0
-8	-1	11.6	-10.3
-8	-2	37.1	-43.9
-9	-1	23.1	-24.8
-9	-2	22.8	25.3
-10	-1	24.7	21.7
-10	-2	10.6	11.4
-11	-1	8.1	-5.9
-11	-2	10.9	-10.7
-12	-1	17.4	-11.5
-12	-2	14.0	15.8
-13	-1	14.9	17.2

4 *k* *l*

-17	2	7.0	6.7
-16	0	13.1	15.0
-16	2	7.6	-6.6
-15	1	15.1	-14.2
-14	0	15.1	-16.5
-14	1	11.6	11.8
-14	2	5.6	7.2
-13	0	16.2	19.5
-13	1	5.8	5.7
-12	1	11.4	-11.5
-12	2	0.0	3.7
-11	0	0.0	-2.3
-10	0	0.0	-2.5
-10	1	15.2	-14.4
-10	2	25.5	26.5
-9	0	5.1	-4.5
-9	1	26.7	26.3
-9	2	3.8	5.4
-8	0	23.0	21.5
-8	1	11.4	-11.5
-8	2	32.0	-33.4
-7	0	32.4	-31.5
-7	1	37.0	-32.6
-7	2	26.1	25.2
-6	0	34.9	34.0
-6	1	46.6	50.0
-6	2	11.0	8.8
-5	0	37.8	37.0
-5	1	22.3	-21.1
-5	2	42.6	-43.1
-4	0	42.5	-40.5
-4	1	46.7	-50.8
-4	2	27.7	30.2
-3	0	17.7	19.5
-3	1	19.8	-3.1
-3	2	6.1	4.5
-2	0	35.5	34.9
-2	1	30.1	-33.5
-2	2	17.9	16.7
-1	0	44.3	-44.9
-1	1	18.5	-14.6
-1	2	9.8	-10.7
0	0	7.1	-4.4
0	1	10.6	7.9
0	2	16.0	-10.3
1	0	39.1	37.0
1	1	0.0	-5.2
1	2	40.6	40.1
2	1	7.5	7.5
2	2	0.0	-0.8
3	0	16.7	14.3
4	0	49.2	-44.4
5	0	25.2	25.8
6	0	14.6	12.7
6	1	43.2	-47.2
6	2	14.5	-14.9
7	0	34.2	-35.5
7	1	20.5	19.5
7	2	37.5	39.8
8	0	57.2	60.0
8	1	18.1	13.4
8	2	16.5	-16.3
9	0	12.9	-9.0
9	1	31.0	-32.8
9	2	9.7	-7.8
10	0	18.0	-20.3
10	1	0.0	1.5
10	2	13.6	8.4
11	0	19.9	22.3
11	1	9.8	9.0
12	0	0.0	-1.0
12	2	8.7	7.9
13	0	9.5	-8.2
13	1	0.0	-3.3
13	2	16.2	-15.1
14	0	0.0	5.4
14	1	10.0	-10.0
14	2	9.4	7.7
15	0	0.0	4.0
15	2	3.2	10.3
16	2	10.1	10.1
16	-2	7.1	7.8
15	-1	17.0	12.0
14	-1	8.7	-7.9
14	-2	23.6	-20.4
13	-1	12.9	-9.3
13	-2	17.2	15.3
12	-1	26.8	28.9
11	-1	20.4	-21.5
11	-2	21.5	-23.3
10	-1	22.8	-26.3
10	-2	11.4	12.1
9	-1	34.3	39.0
9	-2	18.4	16.2
8	-1	21.4	-20.8
8	-2	10.1	-11.2
7	-1	24.6	-23.8
7	-2	13.2	-13.1
6	-1	27.7	27.5
6	-2	23.5	21.7
5	-1	11.8	-13.2
5	-2	22.7	26.6
4	-1	0.0	0.5
4	-2	48.4	-51.2
3	-1	28.4	-28.7
3	-2	8.3	-6.9
2	-1	24.4	21.7
2	-2	54.0	54.7
1	-1	59.7	57.2
0	-1	43.5	-42.9
0	-2	36.2	-33.5
-1	-1	10.0	8.9
-2	-1	47.3	49.5
-2	-2	43.1	-13.9
-3	-1	60.8	-66.7
-3	-2	0.0	7.8
-4	-1	0.0	6.4
-4	-2	0.0	0.3
-5	-1	24.9	24.5
-5	-2	7.3	7.9
-6	-1	24.9	-32.1
-6	-2	0.0	1.2
-7	-1	0.0	1.8
-7	-2	17.1	-20.0
-8	-1	0.0	1.1
-8	-2	17.9	21.5
-9	-1	0.0	-4.4
-9	-2	6.9	4.9
-10	-1	0.0	4.8
-10	-2	23.8	-26.4
-11	-1	13.5	-11.9

-11	-2	18.9	21.3
-12	-1	24.3	22.6
-12	-2	0.0	-0.9
-13	-1	0.0	-3.1
-13	-2	21.3	-23.3
-14	-1	12.4	-12.4
-14	-2	12.5	11.2
-15	-1	15.7	15.3
-17	-1	9.1	-9.9

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-17	0	7.1	-6.6
-17	1	14.2	-13.8
-15	0	15.8	16.4
-13	0	10.6	-11.1
-13	1	0.0	-1.1
-13	2	14.0	-13.6
-12	0	6.8	4.9
-12	1	8.0	-6.8
-12	2	18.3	17.0
-11	0	12.0	11.9
-11	1	19.6	18.6
-11	2	0.0	-3.7
-10	0	18.3	16.0
-10	1	27.3	-25.3
-10	2	22.0	-19.3
-9	0	32.7	-32.3
-9	1	27.6	-27.6
-9	2	45.5	46.9
-8	0	0.0	2.3
-8	1	36.5	38.3
-8	2	15.0	13.4
-7	0	45.2	47.5
-7	1	20.0	-17.4
-7	2	19.9	-19.8
-6	0	68.1	-64.2
-6	1	30.0	-30.8
-6	2	14.7	13.7
-5	0	7.9	9.3
-5	1	45.4	47.9
-4	0	67.9	63.0
-4	1	32.6	-34.4
-3	0	44.9	-46.8
-3	1	41.3	-41.3
-2	0	74.4	69.5
-2	1	51.3	51.2
-1	0	39.3	-37.5
-1	1	18.7	18.3
0	0	0.0	4.6
0	1	17.3	16.4
1	0	9.3	-9.5
1	1	49.6	-48.0
2	0	16.1	-15.4
3	0	86.1	79.2
4	0	25.6	-23.2
4	1	67.4	-66.9
5	0	46.9	-46.1
5	1	5.9	6.3
5	2	47.5	45.1
6	0	56.5	61.0
6	1	23.3	23.0
6	2	14.3	-15.2
7	0	17.6	-17.9
7	1	30.8	-29.0
7	2	0.0	-0.8
8	0	47.0	-47.9
8	1	14.4	11.5
8	2	6.3	4.3
9	0	51.5	53.1
9	1	12.7	12.3
9	2	6.8	5.3
10	0	0.0	0.9
10	1	15.6	-12.9
10	2	11.9	-10.6
11	0	24.4	-23.5
11	1	7.9	-6.5
12	0	7.2	5.4
12	1	8.8	8.0
12	2	10.6	11.4
13	0	7.7	-5.7
13	1	19.0	17.4
14	0	14.9	13.0
14	2	23.0	-22.4
15	0	8.8	-8.4
15	1	12.4	-14.3
15	2	12.5	10.7
16	1	14.8	14.7
17	0	13.5	14.4
17	1	9.1	-7.2
17	2	10.1	-8.0
15	-1	0.0	3.0
15	-2	7.9	-8.4
14	-2	5.6	7.4
13	-1	14.4	13.2
13	-2	5.5	6.7
12	-1	8.0	-8.3
12	-2	9.6	-8.8
11	-1	20.5	-22.0
10	-1	26.5	28.6
10	-2	12.3	9.6
9	-1	14.7	-10.4
9	-2	18.0	-13.7
8	-1	41.7	-42.8
8	-2	37.8	37.4
7	-1	62.7	66.4
7	-2	15.6	14.8
6	-1	12.2	-12.9
6	-2	44.3	-46.1
5	-1	42.8	-46.4
5	-2	16.9	-17.2
4	-1	24.4	24.8
3	-1	25.5	24.2
2	-1	47.6	46.0
1	-1	38.3	-38.0
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-1	-1	79.1	77.8
-2	-1	82.1	-81.5
-3	-1	10.1	-9.8
-5	-1	64.3	-66.9
-5	-2	9.3	6.4
-6	-1	14.1	13.0
-6	-2	12.3	10.9
-7	-1	39.6	43.7
-7	-2	10.1	-9.8
-8	-1	36.7	-40.9
-8	-2	6.2	-3.6
-9	-1	0.0	1.2
-9	-2	16.3	-17.1
-10	-1	10.7	10.0
-10	-2	7.1	4.9
-11	-1	15.7	14.3
-11	-2	13.8	14.1
-12	-1	0.0	1.4
-12	-2	25.3	-30.1
-13	-1	19.3	-20.2
-13	-2	13.8	15.9
-14	-1	15.8	13.4
-14	-2	12.0	10.3
-15	-2	17.9	-18.5
-16	-1	18.9	-17.7
-17	-1	9.5	8.8

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-17	0	7.2	5.7
-16	0	7.2	-6.6
-15	0	5.5	0.7
-15	2	5.6	-5.0
-14	1	0.0	1.3
-14	2	16.0	16.1
-13	1	18.9	17.4
-13	2	9.8	-9.6
-12	0	0.0	1.0
-12	1	0.0	-3.2
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-11	1	0.0	2.4
-11	2	20.5	24.5
-10	0	27.3	27.5
-10	1	43.8	47.4
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-9	0	15.3	12.2
-9	1	41.6	-44.6
-9	2	26.4	-25.2
-9	3	6.5	7.0
-9	4	0.0	-5.3
-9	5	0.0	7.7
-8	0	55.6	-63.4
-8	1	21.7	-22.3
-8	2	27.3	26.5
-8	3	7.5	5.9
-8	4	22.5	23.0
-8	5	19.3	22.6
-8	6	0.0	-3.1
-8	7	17.8	-18.3
-7	0	29.0	30.3
-7	1	45.0	48.0
-7	2	11.6	-12.5
-7	3	8.0	5.1
-7	4	7.6	-6.3
-7	5	30.4	-33.1
-7	6	0.0	-4.0
-7	7	13.7	12.4
-6	0	23.0	21.6
-6	1	0.0	-0.9
-6	2	0.0	2.0
-6	3	21.0	-24.7
-6	4	45.8	-52.0
-6	5	9.1	8.3
-6	6	0.0	4.5
-6	7	7.2	-4.0
-5	0	35.0	-28.4
-5	1	17.1	-12.9
-5	2	21.6	-23.2
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-5	5	16.4	12.4
-5	6	15.2	-16.1
-5	7	10.1	-10.3
-5	8	0.0	-1.5
-5	9	7.3	-8.6
-4	0	14.2	-11.8
-4	1	11.6	11.1
-4	3	58.6	62.9
-4	4	17.9	17.0
-4	5	24.6	-22.9
-4	6	8.1	5.5
-4	7	8.7	7.6
-4	8	9.0	-5.4
-4	9	10.4	10.7
-3	0	7.3	-5.7
-3	1	34.7	34.0
-3	3	17.2	-13.4
-3	4	65.6	-66.8
-3	5	20.7	19.2
-3	6	23.9	24.5
-3	7	15.4	-13.2
-3	8	27.3	23.9
-3	9	0.0	2.6
-2	0	52.0	-50.6
-2	3	31.0	-31.2
-2	4	43.7	49.6
-2	5	28.9	29.0
-2	6	0.0	-2.4
-2	7	0.0	3.4
-2	8	0.0	1.8
-2	9	23.7	-25.5
-1	0	9.1	9.6
-1	4	21.8	16.5
-1	5	19.3	-16.7
-1	6	12.4	-8.5
-1	7	19.5	-20.9
-1	8	16.5	-13.8
-1	9	10.4	10.3
0	0	45.5	-42.0
0	4	57.1	-53.8
0	5	17.0	-17.5
0	6	14.9	-12.0
0	7	0.0	6.5
0	8	30.7	29.2
1	0	29.6	26.8
1	4	16.4	15.3
1	5	5.7	-2.8
1	6	29.6	27.0
1	7	22.1	21.5
1	8	17.2	-13.9
1	9	15.1	-16.1
2	0	64.1	-53.2
2	4	18.2	13.8
2	5	6.4	5.6
2	6	0.0	3.7
2	7	28.1	-31.9
2	8	15.1	-14.7
2	9	10.4	10.7
3	0	73.3	-68.4
3	3	11.5	8.1
3	4	10.5	10.7
3	5	0.0	-2.3
3	6	30.5	-30.1
3	7	0.0	4.1
3	8	30.7	30.0
4	0	81.8	73.7
4	1	8.8	9.3
4	3	11.6	8.2
4	4	0.0	-1.0
4	5	44.2	-44.2
4	6	30.6	28.7
4	7	22.8	20.3
4	8	16.7	-14.1
5	0	56.1	-50.3
5	1	64.7	-66.8
5	2	21.1	-18.3
5	3	23.5	-20.6
5	4	14.0	-14.1
5	5	42.8	40.4
5	6	23.8	23.6
5	7	24.0	-24.1
5	8	0.0	-3.1
6	0	44.3	-47.7
6	1	37.1	36.2
6	2	19.6	17.6
6	3	0.0	7.1
6	4	40.3	42.2
6	5	9.3	7.4
6	6	23.4	-23.7
6	7	0.0	2.0
6	8	11.7	9.3
7	0	60.6	67.4
7	1	36.5	34.4
7	2	13.9	-11.7
7	3	35.8	35.3
7	4	30.1	-30.8
7	5	43.8	-43.8
8	0	18.2	-16.6
8	1	19.0	-16.5
8	2	0.0	3.4
8	3	32.5	-32.0
8	4	25.7	-24.6
8	5	25.8	27.4
8	6	0.0	-3.2
8	7	0.0	-4.6
9	0	18.8	-18.2
9	1	0.0	-3.7
9	2	27.3	-27.3
9	3	19.8	16.8
9	4	29.6	31.4
9	5	7.2	-5.8
9	6	10.5	7.1
10	0	0.0	3.9
10	1	0.0	4.5
10	2	27.8	26.4
10	3	23.3	25.4
10	4	23.1	-23.1
10	5	0.0	-4.6
10	6	0.0	7.0
10	7	10.4	-12.2
11	0	0.0	-9.7
11	1	17.1	17.6
11	3	24.6	-26.9
11	5	14.0	11.3
12	0	0.0	5.9
12	1	17.7	-19.5
12	2	23.1	-25.0
12	3	13.8	15.5
12	4	19.6	19.6
12	6	10.3	8.8
13	0	13.7	-11.1
13	1	8.2	-4.7
13	2	24.1	23.6
13	3	7.5	7.4
13	4	16.4	-15.9
14	0	16.3	-15.0
14	1	33.2	35.2
14	2	8.5	-6.8
14	3	20.4	-22.0
15	0	16.6	17.3
15	2	16.3	-15.9
15	3	9.0	10.1
16	0	15.4	-15.8
17	1	8.8	8.4
17	-1	15.5	-15.5
16	-1	8.1	7.7
15	-1	5.8	3.4
14	-2	5.6	5.6
13	-1	19.2	20.0
12	-1	0.0	-5.4
12	-2	9.5	10.8
11	-1	0.0	3.9
10	-1	10.3	-13.0
10	-2	33.5	-33.9
9	-1	18.0	-16.0
9	-2	31.0	31.5
8	-1	65.2	69.7
7	-1	25.4	-23.8
7	-2	50.4	-49.1
6	-1	16.7	-14.7
5	-1	73.5	76.5
4	-1	21.5	-21.4
3	-1	51.2	-53.0
2	-1	10.2	8.6
1	-1	31.8	29.4
-1	-1	11.8	-13.3
-3	-1	49.3	52.2
-4	-1	35.3	-37.5
-5	-1	17.3	17.5
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-6	-2	41.5	-41.0
-7	-1	37.7	-41.6
-7	-2	18.7	-16.7
-8	-1	10.5	11.4
-8	-2	9.0	5.0
-9	-1	29.7	28.3
-9	-2	0.0	-6.6
-10	-1	24.5	-23.3
-10	-2	9.4	10.4
-11	-1	10.9	3.5
-11	-2	16.8	-19.6
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-12	-2	8.9	9.9
-13	-1	11.2	9.5
-13	-2	14.6	15.9
-14	-1	0.0	7.3
-14	-2	11.7	-10.7
-15	-1	15.4	-15.3
-16	-1	5.7	5.0
-16	-2	9.6	6.3
-17	-2	12.9	-10.8
-18	-1	8.6	-7.9

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-18	0	6.7	-3.7
-17	2	9.7	-9.4
-17	3	11.9	12.3
-16	0	8.6	-8.5
-16	2	15.1	16.0
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-4	0	19.2	19.2
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-4	2	57.2	-59.0
-4	3	14.7	-19.0
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-4	5	23.7	25.6
-4	6	12.7	10.2
-4	7	18.7	18.4
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-4	9	18.3	-20.5
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-2	6	21.6	-20.5
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-2	8	29.4	30.2
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5	7	0.0	4.8
6	0	29.5	-28.3
6	1	0.0	-2.5
6	2	16.1	14.0
6	3	59.2	-62.2
6	4	0.0	-8.5
6	5	37.6	36.9
6	9	15.0	-13.0
7	0	7.2	-4.9
7	1	6.1	-3.6
7	2	0.0	-4.1
7	3	31.0	29.1
7	4	51.6	54.4
7	5	10.7	-8.0
7	6	13.3	-11.0
7	7	7.8	-3.1
7	8	16.5	-16.0
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8	1	13.4	-13.5
8	2	34.8	33.2
8	3	25.0	23.8
8	4	25.9	-27.1
8	5	11.7	-11.5
8	6	9.4	-7.5
8	7	11.0	-11.6
9	0	18.2	-16.7
9	1	44.9	43.6
9	2	27.8	-26.3
9	3	46.4	-46.5
9	4	0.0	2.0
9	5	9.3	9.6
9	6	0.0	-0.3
9	7	26.8	27.1
9	8	11.9	11.2
10	0	35.9	37.1
10	1	38.9	-37.7
10	2	30.1	-29.6
10	3	27.4	26.5
10	4	0.0	2.5
10	5	0.0	-1.9
0	2	6.8	6.6
0	3	42.8	45.3
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0	5	33.1	34.2
0	6	0.0	5.2
0	7	35.1	-38.6
1	2	50.6	48.3
1	3	26.4	-22.7
1	4	13.5	-10.2
1	5	16.1	-17.0
1	6	46.4	-48.4
1	7	0.0	5.2
1	8	19.9	18.6
2	2	60.6	-62.0
2	3	0.0	-4.7
2	4	14.7	-12.1
2	5	20.8	-18.8
2	6	41.2	38.7
2	7	26.1	26.7
2	8	17.7	-14.2
3	0	33.8	-33.2
3	1	25.1	-25.2
3	2	8.5	7.3
3	3	15.9	-11.3
3	4	10.9	8.0
3	5	56.7	60.0
3	6	11.3	10.5
3	7	17.5	-21.7
4	1	63.9	62.6
4	2	11.5	-8.6
4	3	6.7	-5.1
4	4	53.8	54.9
4	5	30.7	-29.4
4	6	28.8	-30.8
4	8	12.3	9.4
5	0	83.5	83.2
5	1	14.1	-10.9
5	2	0.0	0.7
5	3	35.4	34.2
5	4	50.7	-49.0
5	5	41.4	-38.4
10	6	23.0	21.7
10	8	12.5	-15.0
11	0	31.2	-33.0
11	1	17.9	-17.4
11	2	28.3	29.7
11	3	7.6	7.7
11	4	7.7	-8.2
11	6	12.6	-11.3
11	7	16.7	-18.5
12	0	15.8	14.5
12	1	25.8	28.9
12	3	12.3	-11.4
13	0	29.0	34.8
13	1	14.9	-15.6
13	2	12.4	-10.7
13	3	11.1	9.4
13	6	13.9	15.4
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14	1	8.9	-5.7
14	2	16.5	14.5
14	5	10.6	10.2
14	6	9.8	-10.2
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15	1	13.8	10.8
15	5	9.9	-9.2
16	0	7.7	8.3
16	1	9.4	-6.0
17	0	10.1	-9.8
18	-1	12.5	-11.4
18	-2	7.1	-5.6
17	-2	15.4	15.0
16	-1	15.7	14.5
16	-2	9.6	-8.1
16	-3	10.2	-7.9
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15	-5	10.5	8.7
14	-1	9.8	5.6
14	-2	11.1	10.7
14	-3	6.6	-4.0
14	-4	15.0	13.6
14	-6	15.7	-15.6
13	-1	19.8	22.4
13	-3	6.5	7.1
13	-4	6.5	-4.6
13	-5	17.8	-21.4
12	-1	11.9	-12.8
12	-2	8.4	-5.8
12	-3	16.7	-15.0
12	-4	18.3	-18.7
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11	-4	33.8	35.5
11	-5	17.6	15.7
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10	-1	13.5	-11.4
10	-2	11.7	7.9
10	-3	41.1	43.4
10	-4	18.0	-18.0
10	-5	28.3	-26.5
9	-1	24.5	23.1
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9	-3	46.2	-49.3
9	-4	34.0	-35.4
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2	-6	26.8	23.7
2	-7	22.2	23.0
2	-8	22.2	-18.9
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1	-3	29.7	-24.7
1	-4	12.7	10.5
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1	-6	9.3	-6.4
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1	-8	15.0	-13.3
1	-9	9.3	-7.2
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0	-5	30.8	-31.7
0	-6	27.6	-27.0
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-15	-3	12.2	-10.9
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-17	-3	12.7	14.2
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0 k l			
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-16	0	7.6	4.8
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-15	0	22.9	-24.5
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-3	1	17.9	-16.8
-3	2	42.4	-45.3
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-3	5	8.4	-6.6
-3	6	27.9	29.0
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-3	8	24.1	-22.9
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-1	4	33.3	33.8
-1	5	0.0	-0.7
-1	6	42.1	-42.4
-1	7	0.0	10.4
-1	8	18.3	14.2
0	2	13.6	-11.8
0	3	46.6	45.2
0	4	68.3	-66.9
0	5	42.0	-39.4
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0	7	20.9	15.7
1	2	38.4	35.6
1	3	53.9	-53.4
1	4	7.1	-6.3
1	5	51.1	54.0
1	6	10.2	-7.5
1	7	9.5	-6.0
2	1	19.7	-14.6
2	2	29.5	28.2
2	3	72.3	70.3
2	4	39.9	40.7
2	5	11.9	-11.5
2	6	25.4	-23.7
2	7	0.0	-2.5
2	8	0.0	-3.8
2	9	0.0	-0.9
2	10	13.6	11.9
3	0	61.8	55.3
3	1	17.3	-15.9
3	2	0.0	-6.8
3	3	9.9	5.9
3	4	58.2	-63.7
3	5	14.2	-14.4
3	6	17.4	16.0
3	7	7.9	8.3
3	8	0.0	1.5
3	9	16.7	15.6
4	0	38.7	-39.9
4	1	0.0	5.7
4	2	38.7	-33.4
4	3	47.4	-46.3
4	4	13.6	11.7
4	5	51.3	54.8
4	6	0.0	-3.1
4	7	0.0	5.6
4	8	0.0	2.0
4	9	11.7	-11.3
5	0	40.1	38.6
5	1	51.8	-48.6
5	2	18.4	-13.8
5	3	41.4	40.0
5	4	34.6	34.9
5	5	17.5	-20.3
5	6	0.0	-11.3
5	7	0.0	-7.0
5	8	14.8	-18.7
6	0	7.0	-6.4
6	1	15.7	-15.0
6	2	68.5	66.4
6	3	0.0	1.0
6	4	35.9	-35.5
6	5	15.5	-12.3
6	6	0.0	4.6
6	7	0.0	-3.4
6	8	12.6	11.4
6	9	16.3	16.5
7	0	7.0	5.6
7	1	58.8	62.2
7	2	0.0	-1.1
7	3	40.6	-44.1
7	4	0.0	3.6
7	5	18.0	16.5
7	7	15.3	14.9
7	8	11.4	8.4
7	9	14.1	-15.3
8	0	30.4	30.8
8	1	45.2	-47.2
8	2	25.3	-25.3
8	3	15.2	15.5
8	4	14.7	13.0
8	5	0.0	0.7
8	6	0.0	3.3
8	7	8.2	-6.7
8	8	19.8	-21.7
9	0	28.4	-28.5
9	1	19.8	-17.2
9	2	38.1	41.1
9	3	0.0	1.8
9	4	15.5	-14.6
9	5	7.9	10.0
9	6	24.1	-23.1
9	7	6.8	-5.6
9	8	11.0	10.8
10	0	8.7	4.0
10	1	40.5	39.1
10	2	0.0	-1.9
10	3	17.3	-17.9
10	4	8.9	7.4
10	5	11.0	-9.8
10	7	25.2	29.0
11	0	16.8	14.2
11	1	29.5	-30.4
11	2	13.4	-11.8
11	5	10.2	8.0
11	6	17.8	17.5
11	8	13.9	-14.7
12	0	21.7	-23.8
12	1	11.4	-10.2
12	4	7.7	7.3
12	5	11.8	11.3
12	6	12.2	-12.2
12	7	10.6	-11.2
13	0	26.6	29.9
13	1	19.1	20.7
13	2	10.3	9.5
13	5	16.3	-15.2
14	0	26.6	29.4
14	1	15.4	-12.6
14	4	17.0	-18.9
14	6	10.4	9.9
15	0	21.5	-24.5
15	1	8.2	-7.4
15	2	9.4	-6.7
15	4	9.9	10.4
15	5	11.3	12.5
16	0	7.8	4.8
16	3	15.2	13.6
17	2	9.3	6.9
18	2	9.8	-8.4

$(C_6H_5C)_2(CO)_6Fe_2$[11] (2.51 Å) and $(C_2H_5S)_2(CO)_6Fe_2$[15] (2.54 Å). All of these examples contain carbon bridges save for the last which contains a sulphur bridge. The shortening which we observe seems to be associated with the nitrogen bridge since we have found two other examples in which the metal separation is also less than the smallest value given above viz. $[(CH_3C_6H_4)_2CN]_2\text{-}(CO)_6Fe_2$[3] (2.40 Å) and $CH_3C_6H_4\text{-}NCH_2C_6H_4(CO)_6Fe_2$[2] (2.43 Å). This shortening is not due to a smaller metal to bridging-atom distance, indeed the reverse is the case, but due to a smaller angle subtended at the nitrogen atom than at the carbon atom. It could be argued that the angle found, 73°, is a result of the lengths Fe-N and Fe-Fe. The Fe-N distances, mean 2.00 Å, do not vary significantly between themselves and are consistent with a single-bond, in contrast with the shorter value, mean 1.67 Å, found in nitrosyl complexes eg. $(C_2H_5S)_2(NO)_4Fe_2$[16] where multiple-bond character occurs. It is, however, then difficult to formulate reasons for the shorter Fe-Fe distance. A small positive charge on the Fe atoms would cause a shortening, which should also be reflected in the Fe-N distance, but it is unlikely that the associated negative charge would be localised on the nitrogen atoms and although the carbonyl groups present could well accept such a negative charge it is not clear why the same arrangement should not then apply to the carbon-bridged species. It is noted that similar small angles are subtended in sulphur complexes eg $(C_2H_5S)_2\text{-}(CO)_6Fe_2$[15] (68.3°) and $(C_2H_5S)_2(NO)_4Fe_2$[16] (73.7°) but that these complexes do not have these short Fe-Fe distances. It is also clear that the bridged nitrogen arrangement is a preferred state since the N-C-C angles subtended at the ring are reduced from the sp^2 value of 120° to the observed value of 110° which corresponds to a shorter N-N distance in the complex than the free ligand. The N-N distance, 2.4 Å, is long enough to exclude the possibility of a nitrogen-nitrogen bond in the ligand.

The mean Fe-C and C-O distances, 1.78 and 1.14 Å respectively, compare well with mean distances reported e.g. 1.75 and 1.12 Å,[13] 1.81 and 1.15 Å,[15] 1.78 and 1.14 Å[14] and 1.77 and 1.15.[11] The C-N distances, mean 1.44 Å, compare very well with those recently reported for azobenzene[17] (1.45 Å) and *p*-azotoluene[18] (1.43 Å). The ring C-C distances yield an average value of 1.39 Å. Individual Fe-C-O angles do not deviate significantly from linearity.

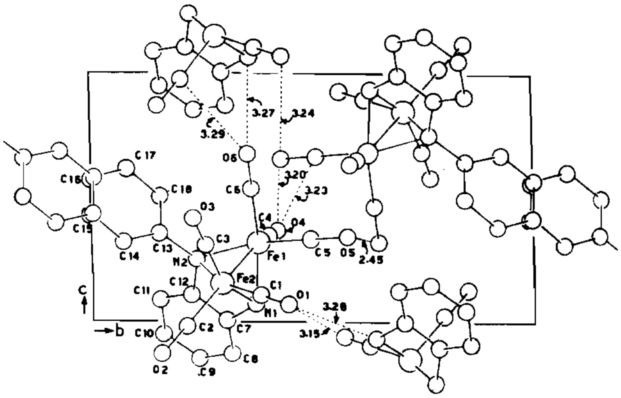

Figure 2. View of the structure along the *a* axis. The shortest non-bonding distances are shown.

The packing of the molecules in the unit cell is shown in projection in Fig. 2. The absence of very short intermolecular distances, as shown in Table III, indicates that the separations between the molecules correspond to van der Waals contacts.

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