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APPLICATION OF A NOVEL SUPERPOSITION TECHNIQUE
TO THE STRUCTURE OF AN ORGANOPHOSPHORUS INSECTICIDE
AND AN ORGANOMETALLIC COMPOUND

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Based on a M.S. thesis submitted to Iowa State University

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Application of a novel superposition technique
to the structure of an organophosphorus insecticide
and an organometallic compound

by

Debra Elaine Beckman

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Application of a novel superposition technique
to the structure of an organophosphorus insecticide
and an organometallic compound

Debra Elaine Beckman

Under the supervision of R. A. Jacobson
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The structures of O-O-dimethyl-O-(3,5,6-trichloro-2-pyridyl) phosphorothioate (Dowco 214) and dicarbonylbis-(η -cyclopentadienyl)- μ -carbonyl- μ -thiocarbonyldiiron have been solved by single crystal x-ray diffraction and use of a modified Patterson superposition technique that uses two multiple vectors to define a structural parallelogram. This method results in a simpler and more accurate shift vector position determination and a general improvement in map clarity.

Dowco 214 crystallizes in the space group $P\bar{1}$ with $a = 11.598(2)$ Å, $b = 13.619(3)$ Å, $c = 8.281(1)$ Å, $\alpha = 94.65(1)^\circ$, $\beta = 94.87(2)^\circ$, $\gamma = 79.97(2)^\circ$ and four molecules per cell (two per asymmetric unit). A CNDO II calculation was performed and partial charge densities assigned. The molecule contains distances between positively charged centers that correspond well to the reported anionic-esteratic distance

(a possible reaction variable) in AChE. Additional reaction variables are discussed.

$\text{Cp}_2\text{Fe}_2(\text{CO})_3\text{CS}$ crystallizes in the space group $P2_1/c$ with $a = 14.508(8) \text{ \AA}$, $b = 13.618(5) \text{ \AA}$, $c = 15.193(7) \text{ \AA}$, $\beta = 110.50(6)^\circ$ and eight molecules per unit cell (two per asymmetric unit). The compound contains both a carbonyl and thiocarbonyl bridge and π -bonded cyclopentadienyl rings that are cis to one another. The iron-iron bond length is intermediate to that of its carbonyl and thiocarbonyl analogs.

A MODIFIED PATTERSON SUPERPOSITION TECHNIQUE USING MULTIPLE VECTORS

Introduction

In recent years much crystallographic emphasis has been placed on direct methods, such as Multan,¹ to obtain trial structures when the "heavy-atom" Patterson analysis is inapplicable. In general these techniques are dependent on knowledge of the space group and frequently prove ineffective in analyses involving crystals of low symmetry.

The Patterson superposition technique²⁻⁴ has been one of the techniques investigators have turned to when direct methods have not been successful. Here specific information regarding the space group is not a necessity and fragments of molecules can be easily recognized in the course of the application of the superposition technique. However the deconvolution of N images (N being the number of atoms in the unit cell) contained in the Patterson usually requires the selection of a series of shift vectors of low multiplicity, all of which must be in the same image of the structure. This then is the major source of difficulty with the Patterson superposition method. As the number of atoms in a unit cell increases, the complexity of the Patterson increases as N^2 and the vectors of

low multiplicity become harder to find and are more readily perturbed by the larger surrounding peaks. Presented in this section is a modification of the basic Patterson superposition technique that uses vectors of higher multiplicity and can lead to a substantial reduction in the number of extraneous accidental peaks on the final superposition map.

Theory

In the point atom approximation the Patterson function can be represented as the vector set $\{\vec{A}_j - \vec{A}_1\}$, in which i and j can assume any value from 1 to N (where N is the number of atoms in the unit cell) and $\vec{A}_j$ is the vector from the origin to atom j . Ideally, if one were able to choose a single vector, $\vec{A}_4 - \vec{A}_1$ (cf. Figure 1) to use as a shift vector only two images of the structure would result. If one lets

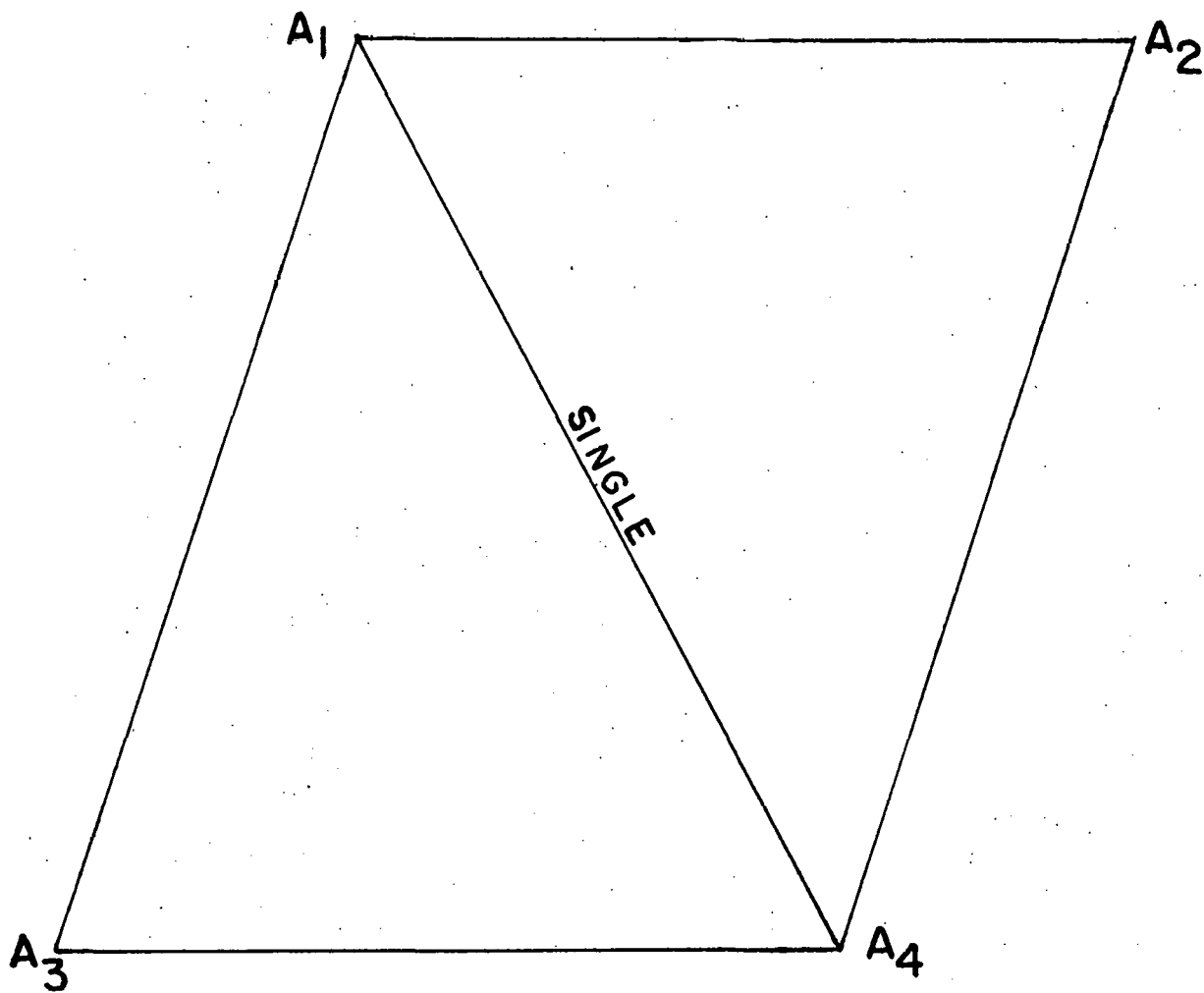
$$\text{Set I} \equiv \{\vec{A}_j - \vec{A}_1\}, \quad \begin{matrix} i=1, \dots, N \\ j=1, \dots, N \end{matrix}$$

then

$$\{\text{Set I}\} \cap \{\text{Set I} + \vec{A}_4 - \vec{A}_1\} = \{\vec{A}_j - \vec{A}_1\}, \{\vec{A}_4 - \vec{A}_1\}.$$

One of these sets is related to the other by the center of symmetry which is located halfway along the shift vector. The selection of a single shift vector is often difficult,

Figure 1. Parallelogram illustrating available
shift vectors



particularly in structures containing a large number of atoms. Also, the resulting map contains a substantial number of accidental extraneous peaks due to the finite width of the peak.

If one selected a vector of double or greater multiplicity for a shift vector, e.g. $\vec{A}_2 - \vec{A}_1 \equiv \vec{A}_4 - \vec{A}_3$, then the superposition procedure should again select that subset corresponding to the junction of the shifted and unshifted Pattersons, this time containing at least four images of the structure.

$$\begin{aligned} & \{\text{Set I}\} \cap \{\text{Set I} + \begin{matrix} \vec{A}_2 - \vec{A}_1 \\ \vec{A}_4 - \vec{A}_3 \end{matrix}\} \\ &= \{\vec{A}_j - \vec{A}_1\}, \{\vec{A}_2 - \vec{A}_1\}, \{\vec{A}_j - \vec{A}_3\}, \{\vec{A}_4 - \vec{A}_1\}. \\ & \text{Set II} \end{aligned}$$

In a conventional superposition procedure additional (one or more) shift vectors would have to be carefully chosen (since all must be in the same image) to further separate these images.

The occurrence of a multiple vector in the Patterson signifies that a parallelogram must exist in the structure (Figure 1), and therefore permits an alternate approach. A shift of Set II by the negative of the original shift vector should bring the head of the related

vector into coincidence with its tail and hence point out the best possibilities for the other side of the parallelogram (i.e., $\vec{A}_3 - \vec{A}_1$ above). Note that this should be at least a vector of multiplicity two in the original Patterson.

Once this vector has been singled out, it is a simple matter to obtain the mathematical equivalent of that which would have been obtained by using the diagonal of the parallelogram, $\vec{A}_4 - \vec{A}_1$ (a single vector in this example), by carrying out the superposition procedure using Set II on Set II with a shift corresponding to the other leg of the parallelogram, $\vec{A}_3 - \vec{A}_1 \equiv \vec{A}_4 - \vec{A}_2$.

Thus

$$\begin{aligned} \{\text{Set II}\} \cap \{\text{Set II} + \begin{matrix} \vec{A}_3 - \vec{A}_1 \\ \vec{A}_4 - \vec{A}_2 \end{matrix}\} & \quad \begin{matrix} i=1, \dots, N \\ j=1, \dots, N \end{matrix} \\ &= \{\vec{A}_j - \vec{A}_1\}, \{\vec{A}_4 - \vec{A}_1\}. \\ & \quad \text{Set III} \end{aligned}$$

There are two distinct advantages to this approach. First, the use of the multiple vectors allows a much more accurate definition of the position of the vector corresponding to the diagonal of the parallelogram. Second, since Set III is obtained by superposition of two maps containing $\sim 4N$ peaks each, instead of two maps containing

$\sim N^2$ peaks each, the number of extraneous peaks is significantly reduced yielding a result which is much easier to interpret. Set III would be equivalent to doing three ordinary minimum function superpositions using both sides of the parallelogram plus the diagonal.

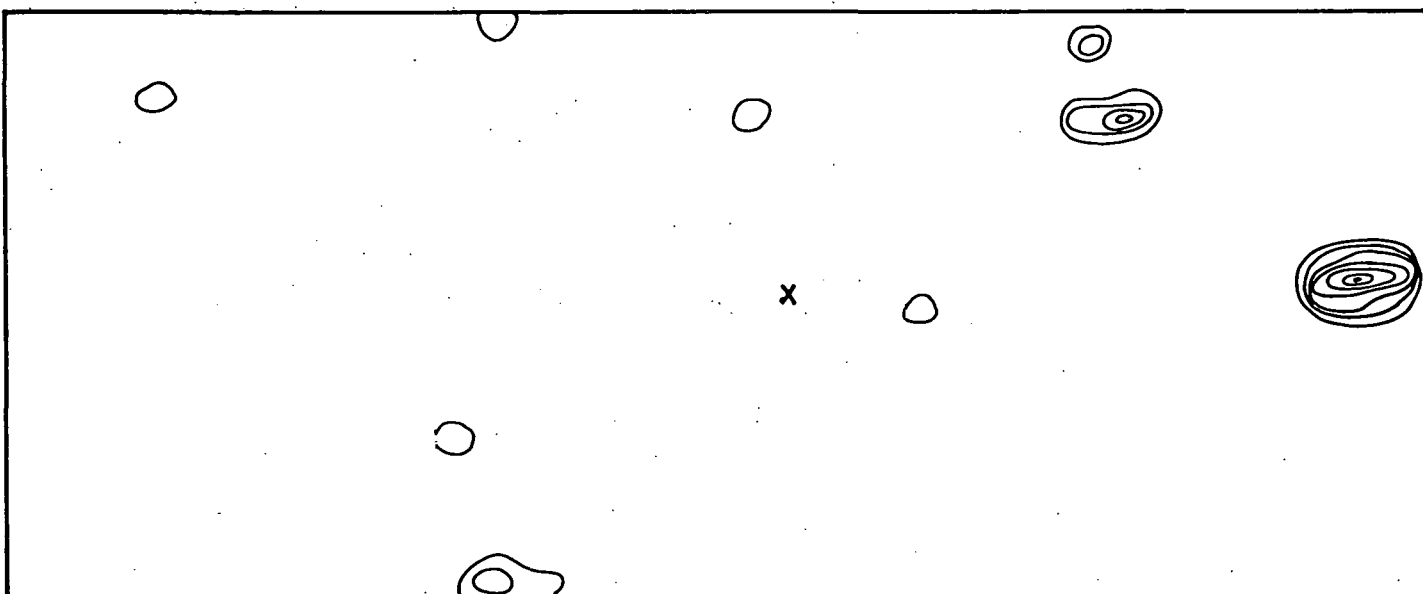
Experimental Results

To explore the applicability of this modification of the conventional Patterson superposition procedure, it was tested on an organophosphorus insecticide, O,O-dimethyl-O-(3,5,6-trichloro-2-pyridyl) phosphorothioate, discussed later, that crystallizes in the low symmetry space group $P\bar{1}$ and contains 64 non-hydrogen atoms in the unit cell. Both direct methods and standard Patterson superposition methods had failed to yield any reasonable trial structure. A simple change in an existing PL/I superposition procedure⁵ was made and a trial structure determination was carried out using the modified superposition approach described above. The resulting map readily identified the positions of all the larger atoms in the structure and the correct placement of the origin. (The midpoint of the diagonal vector coincided with the crystallographic center of symmetry.) Examination of the map revealed that of those 67 peaks greater than 70 in height on an arbitrary scale (with 435 as maximum) 74% of these

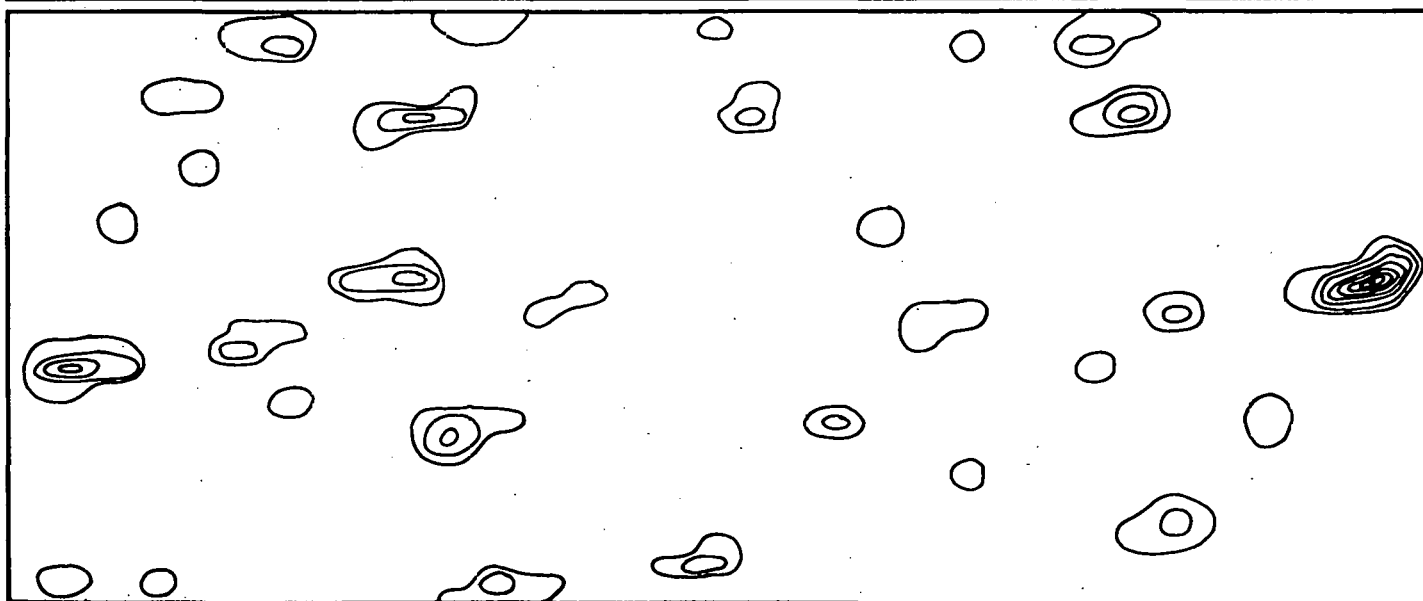
corresponded to atoms in the structure (50 of the 64 atoms in the structure). A standard superposition using the same single vector (the diagonal) yielded 300 peaks over 70 in height on the same scale and only 21% of these corresponded to atoms in the structure. Layer 25 (of 32) of both maps are provided in Figure 2 to demonstrate the general map "clean-up" that results from this modified superposition technique.

As a second test case $\text{Cp}_2\text{Fe}_2(\text{CO})_3\text{CS}$ (dicarbonylbis(η -cyclopentadienyl)- μ -carbonyl- μ -thiocarbonyldiiron), another structure that had not previously been determined and is discussed later in this thesis, was used. It contains 160 non-hydrogen atoms in a unit cell of $\text{P2}_1/\text{c}$ symmetry, i.e. 40 non-hydrogen atoms per asymmetric unit. Again a peak corresponding to a multiple vector was chosen from the sharpened Patterson map and a second shift-vector from the backshifted map. In this case the vector corresponding to the diagonal across the parallelogram did not contain the true inversion center and two images resulted. An additional vector was readily selected that belonged to one of these images and one additional superposition (by the selected vector) yielded a map in which 87% of the peaks greater than 100 in height on an arbitrary scale corresponded to actual atomic positions.

Figure 2. Contour map of layer 25 of 32 for Dowco 214 depicting (A) a modified superposition and (B) a conventional superposition. On (A) all contours are at actual atomic positions and the X indicates an actual atomic position where no contour existed.



A



B

Summary

The structures that were solved demonstrate that this modified Patterson superposition using multiple vectors is a viable method for obtaining a good trial structure and is especially applicable to those structures where size and/or low symmetry inhibits solution by other conventional techniques. The method also involves a relatively minimal amount of computer time.

THE CRYSTAL AND MOLECULAR STRUCTURE OF
DOWCO 214, AN ORGANOPHOSPHORUS INSECTICIDE

Introduction

Structural studies of various organophosphorus (OP) insecticides, with similar formulations but widely differing activities/toxicities, have been undertaken in this laboratory⁶⁻¹⁵ via single crystal x-ray diffraction techniques. These studies are part of a program to generate the necessary data base to correlate toxicity of insecticides, particularly of the organophosphorus type, and their structural and electronic features through generation of a body of precise structural parameters. Accurate structural determination of the relatively small OP insecticide molecules allows inferences to be drawn concerning the three-dimensional structure of the active sites on the complex acetylcholinesterase (AChE) molecule. The effectiveness of the insecticide appears to depend on a favorable combination of gross topological features, relative charge densities on the sites that bond to the enzyme, and the esteratic-anionic site separations within a range that best accommodate the various AChE enzymes.¹⁶⁻²⁰ It is hoped that such structural information will aid in the construction of more specific insecticides conforming

to the most favorable configuration to interact with the target AChE molecule.

The toxic mode of these organophosphorus insecticides is believed to be inactivation of acetylcholinesterase via phosphorylation of an active enzyme site. The AChE molecule is thus unable to hydrolyze the natural substrate, acetylcholine (ACh), that serves as an electrical impulse transmitter across the nerve synapse. The inhibition of AChE may be represented by the reaction



where X is the "leaving group" on the ester and EH is the uninhibited AChE.¹⁶ Acetylcholine and OP insecticides have many chemical and structural similarities that aid the phosphorylation. The phosphate ester corresponds to the acetyl group of ACh that binds to the esteratic site of AChE while some portion of the leaving group acts like the quaternary nitrogen of ACh and reacts with the anionic site.¹⁸ The subsequent build up of ACh in the system leads to autotoxicosis of the insect.

Among the crystal structures recently completed in this laboratory have been that of chlorpyrifos and its methyl oxon homolog, fospirate (the fourth and fifth references cited above) with LD₅₀'s of 163 and 869 mg/kg,²¹ respectively. Continuing in this vein, we decided to carry

out a crystal structure analysis of O-O-dimethyl-O-(3,5,6-trichloro-2-pyridyl) phosphorothioate, hereafter referred to as Dowco 214, the methyl homolog of chlorpyrifos. It has a much lower mammalian toxicity ($LD_{50} = 1500 \text{ mg/kg}$)²² than either chlorpyrifos or fospirate and yet is more effective against adult mosquitos.

Experimental Section

Crystal Data Samples of Dowco 214 were kindly supplied by D. W. Osborne of the Dow Chemical Company. A rectangular crystal of approximate dimensions 0.4 x 0.4 x 0.6 mm was mounted on the end of a glass fiber with Elmers Glue-All and attached to a standard goniometer head. Three preliminary ω -oscillation photographs, taken at various χ and ϕ settings on a four-circle diffractometer, provided the coordinates of eleven independent reflections which were input into an automatic indexing program.²³ The resulting reduced cell and reduced cell scalars indicated triclinic symmetry. Observed layer line spacings on subsequent axial ω -oscillation photographs were equal within experimental error to those predicted by the indexing program.

Lattice constants were obtained by a least-squares refinement of the precise $\pm 2\theta$ ($|2\theta| > 25^\circ$) measurements of

25 strong independent reflections at 27°C using graphite monochromated Mo K α radiation ($\lambda = 0.70954$), yielding $a = 11.598(2)$ Å, $b = 13.619(3)$ Å, $c = 8.281(1)$ Å, $\alpha = 94.65(1)^\circ$, $\beta = 94.87(2)^\circ$, and $\gamma = 79.97(2)^\circ$.

Collection and Reduction of X-ray Intensity Data

Data were collected at 27°C on an automated four-circle diffractometer described previously.²⁴ All data within a 2θ sphere of 50° (5356 reflections) in the hkl , $hk\bar{l}$, $h\bar{k}l$ and $h\bar{k}\bar{l}$ octants were measured using an ω -stepscan technique.

As a general check on electronic and crystal stability, the intensities of three standard reflections were remeasured every 75 reflections. These standard reflections were not observed to vary significantly throughout the data collection period. A Howells, Phillips and Rogers (1950) test²⁵ indicated a center of symmetry and the space group was thus assumed to be $P\bar{1}$.

The intensity data were corrected for Lorentz and polarization effects but, since $\mu = 9.87 \text{ cm}^{-1}$, absorption corrections were not deemed necessary (maximum and minimum transmission factors were 0.66 and 0.54, respectively).

The variance in each intensity was calculated by

$$\sigma^2 = C_T + k_t C_B + (0.03 C_T)^2 + (0.03 C_B)^2,$$

where C_T and C_B represent the total and background counts, k_t is a counting time constant and the factor of 0.03

represents an estimate of non-statistical errors. The estimated deviations were calculated by the finite difference method.²⁶ Equivalent data were averaged and 3810 reflections with $|F_o| > 3.0\sigma(F_o)$ were deemed observed and retained for use in subsequent structure analysis and refinement steps. These data are listed in Appendix A of this thesis.

Solution and Refinement

The presence of four molecules per unit cell in the space group P_1 meant that the structural parameters for two molecules per asymmetric unit had to be determined. Conventional Patterson and direct method procedures¹ were tried but failed to yield any reasonable solution. Therefore we felt this would be a good test case for the recently developed Patterson superposition method which is described in the previous section of this thesis and involves the use of two multiple vector peaks to define a structural parallelogram. This method readily revealed the positions of the nearly tetrahedral thiophosphate groups and the bridging ring carbon in each molecule. The remaining atoms were found by successive factor²⁷ and electron density map calculations.²⁸ The ring hydrogen atom on each molecule was put in at a position of 1.05 Å from the corresponding

ring carbon and its isotropic temperature factor was fixed at $4.5 \text{ \AA}^2$. Parameters of all 32 non-hydrogen atoms were refined via a full-matrix least-squares procedure²⁷ minimizing the function $\Sigma \omega(|F_o| - |F_c|)^2$, where $\omega = 1/\sigma_F^2$. This refinement converged to a conventional crystallographic residual index of $R = \Sigma ||F_o| - |F_c|| / \Sigma |F_o| = 0.078$. Scattering factors used were those of Hanson *et al.*²⁹ for the non-hydrogen atoms and those of Stewart *et al.*³⁰ for the hydrogen atoms. The final positional and thermal parameters are listed in Tables 1 and 2, respectively.

Description of Structure and Discussion

Selected interatomic distances and angles³¹ are listed in Tables 3 and 4, respectively, and are in good agreement with distances and angles in previously reported insecticides, particularly those of fospirate⁸ and chlorpyrifos.¹¹ Selected least-squares planes are listed in Table 5. Table 6 contains the listing of selected dihedral and torsional angles for both molecule A and B. A view of molecule B depicting 50% probability ellipsoids³² is provided in Figure 3. Both of the molecules, A and B, can be seen in the unit cell drawing (Figure 4). The pyridoxyl group is again found to be essentially planar (*cf.* Figure 3), the greatest deviation from the least-squares plane

Table 1. Final atomic positional^a parameters for Dowco 214

Atom	<u>Molecule A</u>		
	x	y	z
C21	1.0656(1) ^b	0.4265(1)	0.1637(2)
C22	1.1834(1)	0.3299(1)	0.7801(2)
C23	0.9213(2)	0.2921(1)	0.7956(2)
S	0.6263(1)	0.4695(1)	0.3827(2)
P	0.7036(1)	0.3736(1)	0.2318(2)
O1	0.8434(3)	0.3706(3)	0.2271(4)
O2	0.6683(4)	0.3842(3)	0.0474(5)
O3	0.6883(4)	0.2647(3)	0.2549(5)
N	0.8914(4)	0.3357(3)	0.4940(5)
C1	0.9224(4)	0.3647(4)	0.3596(6)
C2	1.0329(5)	0.3874(4)	0.3427(6)
C3	1.1137(5)	0.3780(4)	0.4737(7)
C4	1.0827(5)	0.3457(4)	0.6169(7)
C5	0.9689(5)	0.3270(4)	0.6215(7)
C6	0.6663(7)	0.4772(5)	-0.0306(8)
C7	0.7393(7)	0.1787(5)	0.1452(9)
H	1.1992	0.3942	0.4657

^aThe positional parameters for all atoms are represented in fractional unit cell coordinates. Positions for methyl hydrogens are not given.

^bIn this and succeeding tables, estimated standard deviations are given in parentheses for the least significant figures and include the error in the lattice constants. No standard deviation is given for the unrefined hydrogens.

Atom	<u>Molecule B</u>		
	x	y	z
C1	0.3812(2)	0.2679(2)	0.1610(2)
C2	0.5310(2)	0.2680(1)	0.7926(2)
C3	0.4263(2)	0.0680(2)	0.8075(2)
S	0.0677(2)	0.1074(2)	0.3619(3)
P	0.1869(1)	0.0312(1)	0.2394(2)
O1	0.2987(4)	0.0858(4)	0.2301(5)
O2	0.1610(4)	0.0099(3)	0.0529(5)
O3	0.2362(4)	-0.0763(3)	0.2844(6)
N	0.3637(5)	0.0856(4)	0.5016(6)
C1	0.3532(5)	0.1288(5)	0.3636(7)
C2	0.3983(5)	0.2157(5)	0.3442(7)
C3	0.4540(5)	0.2575(4)	0.4776(7)
C4	0.4630(5)	0.2142(4)	0.6240(7)
C5	0.4171(5)	0.1275(5)	0.6290(7)
C6	0.1095(7)	0.0904(6)	-0.0504(10)
C7	0.2310(7)	-0.1122(6)	0.4453(9)
H	0.4884	0.3244	0.4685

Table 2. Final thermal^a parameters ($\times 10^3$) for Dowco 214

Atom	<u>Molecule A</u>					
	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C1	8.3(2)	10.4(1)	13.4(3)	-4.0(1)	2.4(1)	2.0(1)
C2	7.6(1)	10.3(1)	15.0(3)	-2.5(1)	-3.3(2)	1.1(2)
C3	10.2(2)	10.1(1)	12.9(3)	-3.7(1)	0.2(2)	3.8(2)
S	7.4(1)	6.4(1)	20.1(3)	-1.3(1)	3.0(2)	-1.6(1)
P	5.1(1)	4.9(1)	12.2(2)	-1.8(1)	-0.1(1)	0.4(1)
O1	4.8(3)	8.3(3)	10.5(6)	-2.0(2)	-0.1(3)	0.5(3)
O2	8.2(4)	6.4(3)	13.9(7)	-2.9(3)	-3.2(4)	2.0(3)
O3	9.0(4)	5.1(3)	15.7(7)	-2.3(3)	1.0(4)	1.2(3)
N	5.9(4)	5.6(3)	10.8(7)	-2.0(3)	0.8(4)	0.7(4)
C1	4.4(4)	5.0(3)	11.5(9)	-1.5(3)	0.4(5)	-0.3(4)
C2	5.6(4)	5.3(4)	10.4(8)	-1.6(3)	1.3(5)	-0.3(4)
C3	5.4(4)	5.6(4)	12.2(9)	-1.8(3)	1.8(5)	-0.5(5)
C4	6.2(5)	5.1(4)	12.9(10)	-1.1(3)	-1.4(5)	-0.9(5)
C5	6.1(5)	5.3(4)	12.8(9)	-1.4(3)	1.6(5)	1.3(5)
C6	12.0(7)	7.2(5)	17.0(12)	-3.7(5)	-2.1(8)	4.4(6)
C7	12.9(8)	5.3(4)	17.2(12)	-1.5(5)	0.7(8)	-1.8(6)
H	4.5					

^aThe β_{ij} are defined by: $T = \exp\{-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})\}$. For all hydrogen atoms an isotropic thermal parameter of 4.5 was assigned. Non-hydrogen thermal parameters are ($\times 10^3$).

Table 2 (Continued)

Atom	<u>Molecule B</u>					
	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C&1	13.7(2)	10.2(2)	15.0(3)	-5.1(1)	-1.5(2)	3.1(2)
C&2	10.2(2)	7.6(1)	15.3(2)	-3.0(1)	-2.0(2)	-2.1(1)
C&3	15.6(2)	8.6(1)	15.3(3)	-4.3(1)	-0.6(2)	2.6(2)
S	8.4(2)	7.4(1)	28.1(5)	-0.7(1)	4.0(2)	-2.9(2)
P	6.3(1)	4.4(1)	15.5(3)	-1.9(1)	-0.6(1)	-0.5(1)
O1	8.7(4)	10.0(4)	15.2(7)	-6.0(3)	0.4(4)	-1.3(4)
O2	10.6(4)	6.0(3)	16.2(8)	-2.6(3)	-3.4(5)	-0.2(4)
O3	12.9(5)	5.4(3)	19.6(9)	0.3(3)	3.0(5)	1.3(4)
N	8.7(5)	6.8(4)	14.8(9)	-3.3(3)	1.1(5)	-0.3(5)
C1	6.1(5)	7.7(5)	12.2(10)	-2.8(4)	1.0(5)	-1.8(5)
C2	6.3(5)	6.7(4)	14.0(10)	-1.8(4)	0.7(6)	0.1(5)
C3	5.9(5)	5.8(4)	15.9(11)	-2.2(4)	1.0(6)	-0.2(5)
C4	6.2(5)	5.7(4)	15.0(11)	-1.6(3)	0.3(6)	-1.3(5)
C5	7.7(5)	6.0(4)	13.8(10)	-1.9(4)	1.1(6)	0.1(5)
C6	13.3(8)	8.7(6)	22.8(16)	-3.3(6)	-5.7(9)	4.9(8)
C7	13.4(8)	8.6(6)	18.8(14)	-4.2(6)	0.3(8)	3.8(7)
H	4.5					

Table 3. Selected interatomic distances (Å) for Dowco 214

	Molecule A	Molecule B
S-P	1.894(2)	1.893(2)
P-O2	1.559(4)	1.561(4)
O2-C6	1.462(7)	1.458(8)
P-O3	1.553(4)	1.540(5)
O3-C7	1.492(8)	1.467(8)
P-O1	1.618(4)	1.613(4)
O1-C1	1.367(6)	1.367(7)
C1-C2	1.390(7)	1.401(9)
C2-C3	1.371(7)	1.366(8)
C3-C4	1.393(8)	1.379(8)
C4-C5	1.392(8)	1.383(8)
C5-N	1.325(7)	1.317(7)
N-C1	1.309(7)	1.315(8)
C2-C&1	1.708(5)	1.708(6)
C3-H	1.062(4)	1.066(5)
C4-C&2	1.711(5)	1.719(6)
C5-C&3	1.717(6)	1.730(6)
P-C3	5.010(5)	4.929(6)
P-C4	5.185(6)	5.136(6)
P-C5	4.271(6)	4.281(6)
P-H	5.954(2)	5.855(2)
C6-S	3.504(7)	3.472(9)
C7-S	4.318(7)	3.339(8)
N-C6	5.203(8)	5.223(9)
N-C7	3.934(8)	3.311(9)

Table 4. Selected bond angles ($^{\circ}$) for Dowco 214

	Molecule A	Molecule B
S-P-O2	118.2(2)	119.1(2)
P-O2-C6	122.5(4)	121.0(4)
S-P-O3	118.5(2)	118.2(2)
P-O3-C7	122.0(4)	123.8(5)
S-P-O1	113.5(2)	113.8(2)
P-O1-C1	125.1(3)	123.1(4)
O1-P-O2	98.2(2)	97.6(2)
O1-P-O3	105.7(2)	106.4(3)
O2-P-O3	102.7(2)	98.7(3)
O1-C1-C2	118.0(5)	117.8(6)
O1-C1-N	118.5(4)	118.9(6)
C1-C2-C3	118.0(5)	117.4(6)
C2-C3-C4	119.2(4)	119.8(6)
C3-C4-C5	117.9(5)	118.0(5)
C4-C5-N	122.5(5)	123.1(6)
C5-N-C1	118.3(5)	118.8(5)
C1-C2-C21	120.5(4)	121.8(5)
C3-C2-C21	121.5(4)	120.7(5)
C2-C3-H	120.4(5)	119.8(6)
C4-C3-H	120.4(5)	120.4(5)
C3-C4-C23	120.1(4)	119.8(5)
C5-C4-C23	122.0(5)	122.2(5)
C4-C5-C24	121.0(4)	120.8(5)
N-C5-C24	116.5(4)	116.0(5)
N-C1-C2	123.5(5)	123.3(5)

Table 5. Least-squares planes^a for Dowco 214
(Molecules A and B)

Planes defined by all eleven pyridoxyl members

$$A: (-0.17643)x + (0.93478)y + (0.30831)z = 3.30518$$

$$B: (0.81300)x + (-0.53098)y + (-0.23890)z = 1.85636$$

<u>Atom</u>	<u>Distance from plane (Å)</u>	
	<u>A</u>	<u>B</u>
C1	-0.0013	-0.0113
O1	-0.0540	-0.0006
C2	0.0102	0.0036
C11	0.0323	-0.0127
C3	0.0106	0.0133
H3	0.0149	0.0139
C4	-0.0087	-0.0018
C12	-0.0589	-0.0184
C5	0.0058	0.0066
C13	0.0525	0.0000
N	-0.0033	0.0075

Planes defined by P, S, O1 and C1

$$A: (0.28317)x + (0.85463)y + (-0.43522)z = 6.05112$$

$$B: (0.12403)x + (-0.77212)y + (0.62324)z = 1.07282$$

<u>Atom</u>	<u>Distance from plane (Å)</u>	
	<u>A</u>	<u>B</u>
P	-0.1942	0.1917
S	0.0737	-0.0760
O1	0.2482	-0.2454
C1	-0.1278	0.1298

^aPlanes are defined by $c_1X + c_2Y + c_3Z - d = 0$,
where X, Y, and Z are Cartesian coordinates which are
related to the triclinic cell coordinates (x,y,z) by
the transformations:
 $X = x \sin \gamma + z \{(\cos \beta - \cos \alpha \cos \gamma) / \sin \gamma\} = x_a + z \cos \beta$
 $Y = x \cos \gamma + y_b + z \cos \alpha = y_b$
 $Z = z \{1 - \cos^2 \alpha - \cos^2 \beta - \cos^2 \gamma + 2 \cos \alpha \cos \beta \cos \gamma\}^{1/2} / \sin \gamma = z \sin \beta$.

Table 5 (Continued)

Planes defined by P, S, O2 and C6

A: $(0.96531)x + (0.19056)y + (0.17848)z = 9.63156$

B: $(0.77352)x + (0.62421)y + (0.10963)z = 1.78990$

<u>Atom</u>	<u>Distance from plane (Å)</u>	
	<u>A</u>	<u>B</u>
P	0.2152	0.2082
S	-0.0817	-0.0815
O2	-0.2545	-0.2424
C6	0.1211	0.1157

Planes defined by P, S, O3 and C7

A: $(0.76851)x + (-0.16043)y + (0.61938)z = 7.2179$

B: $(0.78218)x + (0.41571)y + (0.46408)z = 2.55462$

<u>Atom</u>	<u>Distance from plane (Å)</u>	
	<u>A</u>	<u>B</u>
P	0.0085	0.1002
S	-0.0107	-0.0420
O3	0.0169	-0.1166
C7	-0.0147	0.0584

Table 6. Configurational similarities of molecules A and B for Dowco 214

<u>Plane</u>		<u>Defined by</u>	
1		C1-C5, N	
2		O1, O2, O3	
3		P, O1, C1	
4		S, P, O1	

<u>Plane</u>		<u>Dihedral Angle ($^{\circ}$)</u>	
		<u>A</u>	<u>B</u>
1	2	26.94	16.78
1	3	16.52	36.23
2	3	43.19	47.40
3	4	50.89	48.01

		<u>Torsional Angle ($^{\circ}$)</u>	
		<u>A</u>	<u>B</u>
P-O1-C1-C2		-164.48	-144.66
C1-O1-P-S		50.68	48.01
C1-O1-P-O2		177.86	174.39
C1-O1-P-O3		-76.25	-84.11
S-P-O2-O6		52.17	49.41
S-P-O3-O7		177.87	-23.33

Figure 3. View of Dowco 214 molecule B with partial charge densities obtained from CNDO II molecular orbital calculations

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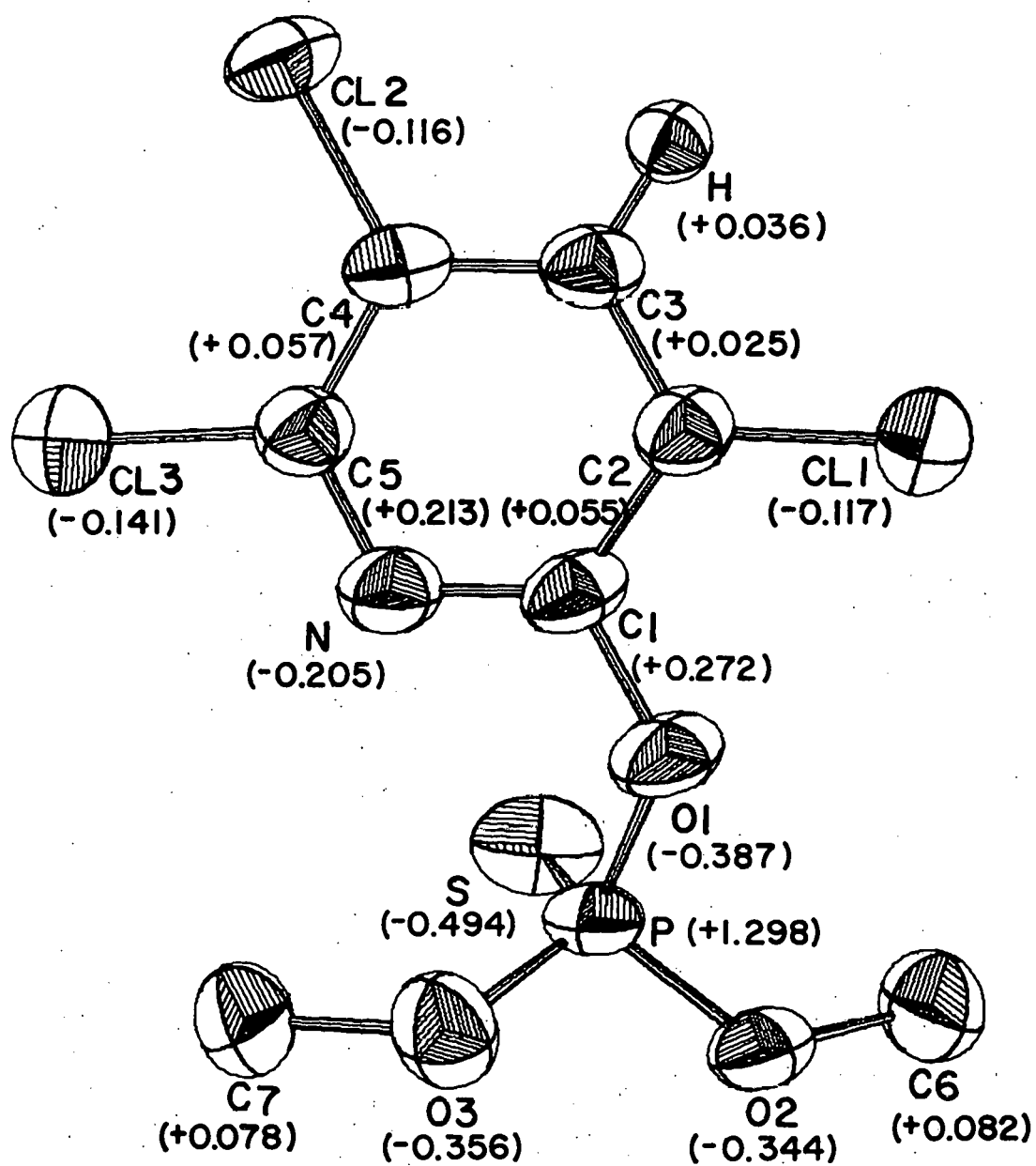
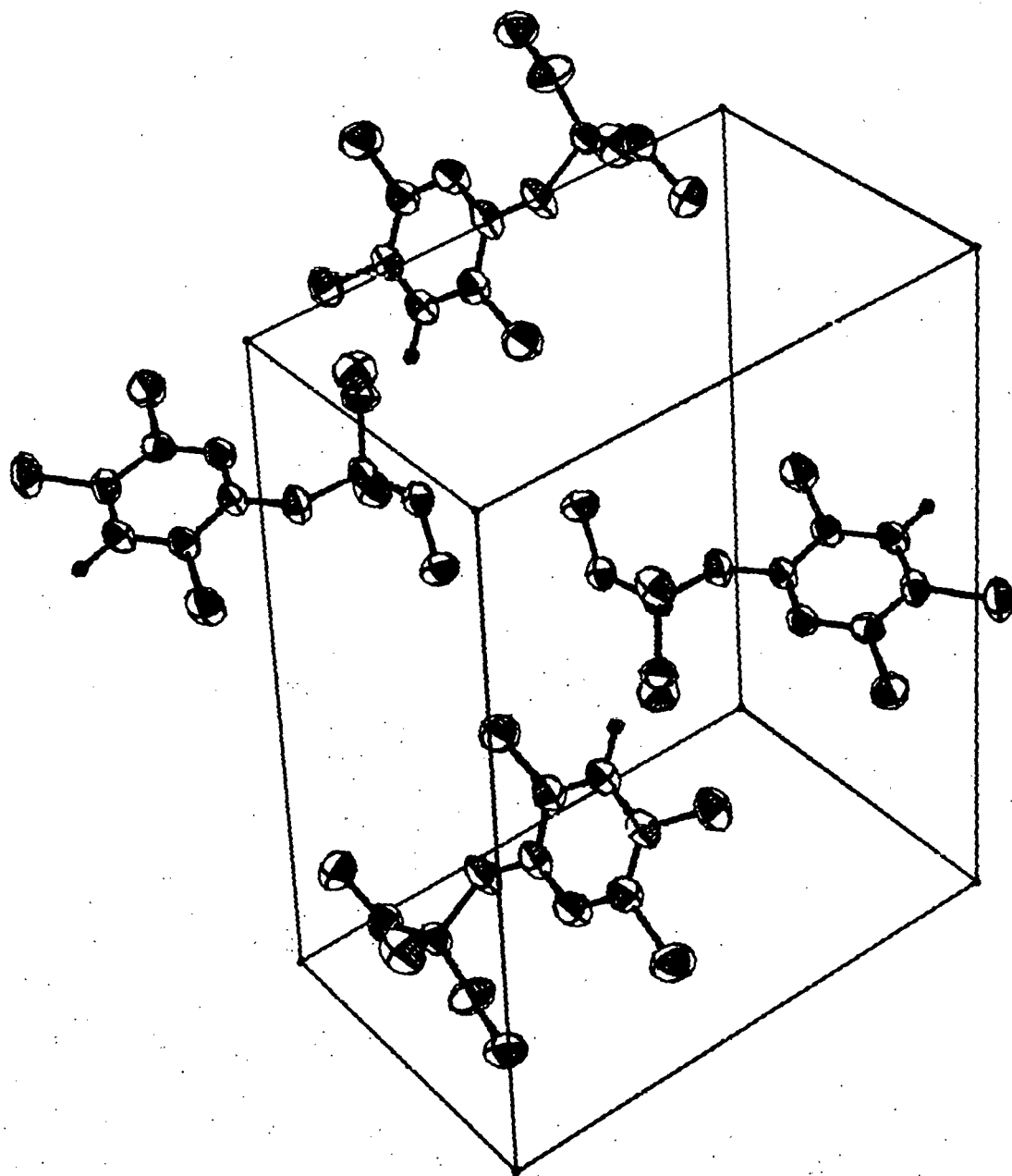


Figure 4. Unit cell of Dowco 214



(cf. Table 6) defined by the six-membered ring, the three attached chlorines and O1 being .0589 Å for C12 (molecule A). The shortest intermolecular distance, 2.91 Å, occurs between H2(B) and S(A). This and other intermolecular distances indicate that the packing in the Dowco 214 crystal is primarily van der Waals in nature. This is also evident by a comparison of the distances in the two symmetry unrelated molecules in the asymmetric unit, since packing environments are certainly different. Examination of these distances (cf. Table 3) show only slight variations of 4σ or less. The same is true of most of the angles, with only angles showing any appreciable deviation being those around the methoxy oxygens. This is not unexpected as there should be a very low barrier to rotation about this phosphorus-oxygen bond.

The suggested bonding formulation involving a weak overlap of the p_z orbital on O1 with the ring system is again substantiated by the interatomic distances found, and also by a CNDO II molecular orbital calculation of the Pople and Beveridge³³ type. As in the case of previous OP insecticides whose structures have been analyzed, the Cl-O1 bond in Dowco 214 is significantly shorter than the other methoxy C-O bonds and also the P-O1 bond is (by > 10σ) the longest of the three P-O bonds. Thus phosphorylation³⁴

should be enhanced. Dowco 214, like the other pyridoxyl OP insecticides chlorpyrifos and fospirate, displays a Cl-O1 bond length ~4σ less than that of the phenoxy OP insecticide; this is possibly the result of replacement of a ring carbon by that of the more electronegative nitrogen.

The tetrahedral geometry about the phosphorus atom is distorted such that the S=P-O angles are greater than the O-P-O angles. Dowco 214 exhibits internal ring angles identical within standard deviations found for chlorpyrifos and fospirate, the angles involving nitrogen as the terminal atom being greater than 120° and all other internal ring angles being less than 120° (cf. Table 3). As discussed in prior papers the orientation of the ring group relative to the P=S (or P=O) may be a reaction variable for OP insecticide interaction with AChE. The angle between the normal to the ring and the P=S vector is 26.9° in molecule A and 16.8° in molecule B, both of which are in the range observed for the more toxic OP insecticides previously studied.

If one considers a plane that is perpendicular to the pyridoxyl ring and contains the Cl-O1 bond, it is seen (Figure 1) that as in the case of other insecticides (ronnel,⁶ crufomate,⁹ ronnel oxon,¹⁰ fospirate⁸ and

chlorpyrifos¹¹), the phosphorus atom and the C21 atom are on opposite sides of this plane. The position of the phosphorus atom in Dowco 214 is apparently dictated by a barrier to rotation about the C1-O1 bond caused by interactions between the phosphorus atom and C21 or N. Since there is no substituent on the nitrogen atom, the thiophosphate group can to some extent rotate toward the nitrogen thus avoiding close contact with C21; the P-O1-C1-C2 torsional angle is 164.5° in molecule A and 144.7° in molecule B.

Organophosphorus insecticides cause autotoxicosis through inhibition of acetylcholinesterase (AChE). It has been reported that the acetylcholine molecule, in its proper configuration to interact with bovine erythrocyte AChE, has an active site separation of $4.7 \text{ \AA}$, while fly head AChE shows an anionic-esteratic center distance of up to $5.7 \text{ \AA}$.³⁵ Thus compatibility of charges at appropriate distances must be considered when choosing probable active sites on the OP insecticide. As a first approximation to the OP active sites only two atoms will be considered in accordance with Krupka's³⁶ active site model of AChE (however also see O'Brien³⁷). Without any further knowledge of the structure of the AChE, steric interactions around the active site can only be inferred.

The results of a CNDO II molecular orbital calculation³³ on Dowco 214 show that there are, in addition to the phosphorus, three probable positive centers that could be involved in enzyme bonding in insects (cf. Figure 3 and Table 3). These are C(3), C(4) and H which are respectively at 4.93, 5.14 and 5.85 Å from the phosphorus in the molecule B and 5.01, 5.18 and 5.95 Å from the phosphorus in molecule A. These are within the insect range 5.0 to 5.5 Å³⁵ and/or 4.5-5.9 Å³⁷ and yet outside the mammalian site separation range, 4.3-4.7 Å.^{35,38} It may also be the case that there is a region of $\delta(+)$ charge between C3 and its hydrogen that could also give a distance appropriate for AChE interaction. The P-C(5) distance of 4.28 Å could accommodate mammalian AChE and may help account for Dowco 214's toxic effects.

THE CRYSTAL AND MOLECULAR STRUCTURE OF
 $(C_5H_5)_2Fe_2(CO)_3CS$, DICARBONYLBIS(η -CYCLOPENTADIENYL)- μ -
 CARBONYL- μ -THIOCARBONYLDIIRON,
 AN ORGANOMETALLIC COMPOUND

Introduction

To date only a few complexes containing thiocarbonyl bridges have been reported,³⁹⁻⁴³ all demonstrating the preference of the CS group for a bridging over a terminal position. We decided to undertake an x-ray crystal structure analysis of the dicarbonylbis(η -cyclopentadienyl)- μ -carbonyl- μ -thiocarbonyldiiron, $Cp_2Fe_2(CO)_3CS$, complex, which contains both a thiocarbonyl and carbonyl bridge in the same molecule, to determine its structural characteristics and any differences and/or similarities to the carbonyl⁴⁴ and thiocarbonyl³⁹ analogs. Of particular interest are the deviations, if any, in the iron-iron and iron-bridging carbon distances when the number of bridging thiocarbonyls is changed. The increasing number of structurally characterized metal-metal bonded dimers is an indication of the current interest in metal cluster compounds, with dimers being considered the simplest building blocks for larger clusters. The thiocarbonyl ligand described in this paper is a unique new bridging ligand that may lead to many novel clusters.

Experimental Section

Crystal Data $\text{Cp}_2\text{Fe}_2(\text{CO})_3\text{CS}$, M.W. = 369.99,
 monoclinic $\text{P2}_1/\text{c}$, $a = 14.508(8) \text{ \AA}$, $b = 13.618(5) \text{ \AA}$,
 $c = 15.193(7) \text{ \AA}$, $\beta = 110.50(6)^\circ$, $V = 2811.56 \text{ \AA}^3$,
 $\rho_c = 1.753 \text{ g/cm}^3$, $Z = 8$, $\mu = 22.6 \text{ cm}^{-1}$ for Mo $\text{K}\alpha$.

Crystals of the title compound, whose preparation is discussed elsewhere,⁴³ were obtained from M. H. Quick and R. J. Angelici. A rectangular prismatic crystal of approximate dimensions 0.4 mm x 0.3 mm x 0.2 mm was selected, mounted on a glass fiber with Elmers Glue-All and attached to a standard goniometer head. Four preliminary ω -oscillation photographs, taken at various χ and ϕ settings on a four-circle diffractometer, provided the coordinates of ten independent reflections which were input into an automatic indexing program.²³ The resulting reduced cell and reduced cell scalars indicated 2/m (monoclinic) symmetry. Observed layer line spacings on subsequent axial ω -oscillation photographs were equal within experimental error to those predicted by the indexing program, as well as confirming the symmetry.

Lattice parameters (reported above) were obtained by a least-squares refinement of the precise $\pm 2\theta$ ($|2\theta| > 25^\circ$) measurements of 14 strong independent reflections at 27 C using graphite-monochromated Mo $\text{K}\alpha$ radiation ($\lambda = 0.70954 \text{ \AA}$).

Collection and Reduction of X-ray Intensity Data

Data were collected at 27 C° on an automated four-circle diffractometer described previously by Rohrbaugh and Jacobson.²⁴ All data within a 2 θ sphere of 50° (11,386 reflections) in the hkl , $hk\bar{l}$, $h\bar{k}l$, and $h\bar{k}\bar{l}$ octants were measured using an ω -stepscan technique.

As a general check on electronic and crystal stability, the intensities of five standard reflections were measured every 75 reflections. These standard reflections were not observed to vary, nor the crystal to decompose throughout the entire process of data collection. The space group was uniquely determined to be $P2_1/c$ by systematic absences in the data occurring when $l = 2n+1$ for the $h0l$ reflections and $k = 2n+1$ for the $0k0$ reflections.

The intensity data were corrected for Lorentz and polarization effects. Since $\mu = 22.6 \text{ cm}^{-1}$ ($0.51 < \text{transmission factor} < 0.64$), no absorption correction was considered necessary. The variance in each intensity was calculated by

$$\sigma_2 = C_T + k_t C_B + (0.03 C_T)^2 + (0.03 C_B)^2,$$

where C_T and C_B represent the total and background counts, k_t is a counting time constant and the factor of 0.03 represents an estimate of non-statistical errors. The

estimated deviations were calculated by the finite difference method.²⁶ Equivalent data were then averaged and 3658 reflections with $|F_o| > 3\sigma(F_o)$ were retained for use in subsequent structure analysis and refinement steps. During the latter stages of refinement fifteen large reflections that suffered from secondary extinction effects were removed from the data set. These data are listed in Appendix B of this thesis.

Solution and Refinement

Since the cell parameters and calculated density indicated the presence of eight molecules per unit cell, the structural parameters for two molecules (one asymmetric unit) had to be determined. Due to the number of atoms in the asymmetric unit, the title compound appeared to be a good test case for the Patterson superposition technique which involves the use of two multiple vector peaks to define a structural parallelogram, as discussed earlier. This new method readily revealed the positions of the four irons, the sulfurs and one bridging carbon. The remaining atoms were found by successive structure factor⁴⁵ and electron density map calculations.²⁸ (The ring carbons had to be located using a sharpened electron density map.) Ring hydrogens were put in at

positions 1.05 Å from the corresponding ring carbons and their isotropic temperature factors were fixed at 4.5 Å². The parameters of all 40 non-hydrogen atoms were refined via a block diagonal least-squares refinement,⁴⁵ minimizing the function $(|F_o| - |F_c|)^2$ where $\omega = 1/\sigma_F^2$. A final full-matrix least-squares refinement was done 20 atoms (one molecule) at a time to keep the computing requirements more manageable. This refinement converged to a conventional crystallographic residual, $R = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|$, of 0.070. The scattering factors used for non-hydrogen atoms were those of Hanson et al.²⁹ modified for the real and imaginary parts of anomalous dispersion.⁴⁶ The scattering factor used for hydrogen was that of Stewart et al.³⁰ The final positional and thermal parameters are listed in Tables 7 and 8, respectively.

Description of Structure and Discussion

Computer-generated perspective drawings³² of an individual Cp₂Fe₂(CO)₃CS molecule (A) and of the unit cell containing all eight molecules are provided in Figures 5 and 6, respectively. Tables 9, 10 and 11 list selected interatomic distances and angles.

The compound contains a bridging thiocarbonyl group and again demonstrates the preference, as compared to CO,

Table 7. Final positional parameters^a for
(C₅H₅)₂Fe₂(CO)₃CS

Molecule A			
Atom	x	y	z
Fe1	0.51391(7) ^b	0.02768(8)	0.31244(8)
Fe2	0.37171(7)	-0.00637(8)	0.16466(7)
S	0.5180(2)	0.1630(2)	0.1368(2)
O1	0.4352(5)	0.1927(5)	0.3815(5)
O2	0.2465(4)	0.1545(4)	0.1742(4)
O3	0.3540(4)	-0.0848(4)	0.3363(4)
C1	0.4659(6)	0.1290(6)	0.3532(5)
C2	0.2958(5)	0.0893(6)	0.1714(5)
C3	0.3919(5)	-0.0409(6)	0.2917(5)
C4	0.4768(5)	0.0839(5)	0.1913(5)
C1R1	0.6554(9)	-0.0068(14)	0.3092(11)
C2R1	0.6048(8)	-0.1002(10)	0.3222(10)
C3R1	0.5908(7)	-0.0888(9)	0.4030(8)
C4R1	0.6265(8)	0.0087(10)	0.4433(9)
C5R1	0.6640(8)	0.0528(10)	0.3800(14)
H1R1	0.6547	-0.0736	0.2779
H2R1	0.6130	-0.0433	0.2738
H3R1	0.5814	-0.0280	0.4461
H4R1	0.6094	-0.0589	0.4744
H5R1	0.6974	0.1218	0.3897
C1R2	0.3504(20)	-0.0349(10)	0.0228(8)
C2R2	0.2643(11)	-0.0579(15)	0.0450(12)
C3R2	0.2927(12)	-0.1326(11)	0.1041(13)
C4R2	0.3873(12)	-0.1530(9)	0.1288(10)
C5R2	0.4219(9)	-0.0957(12)	0.0777(9)
H1R2	0.3461	0.0243	-0.0284
H2R2	0.1891	-0.0362	0.0259
H3R2	0.2487	0.0243	0.1357
H4R2	0.4326	-0.2059	0.1832
H5R2	0.4955	-0.0944	0.0781

^aThe positional parameters for all atoms are represented in fractional unit cell coordinates.

^bIn this and succeeding tables estimated standard deviations are given in parentheses for the least significant figures. No standard deviation is given for the unrefined hydrogens.

<u>Molecule B</u>			
Atom	x	y	z
Fe1	0.07270(7)	0.76860(8)	0.25347(7)
Fe2	-0.06600(7)	0.72910(8)	0.10433(7)
S	-0.1335(2)	0.8748(2)	0.2407(2)
O1	0.1229(4)	0.9599(4)	0.1998(4)
O2	-0.0619(5)	0.9064(5)	0.0004(4)
O3	0.1292(4)	0.6755(5)	0.1076(4)
C1	0.1037(5)	0.8839(6)	0.2203(5)
C2	-0.0623(5)	0.8349(6)	0.0412(5)
C3	0.0724(6)	0.7101(6)	0.1384(5)
C4	-0.0608(6)	0.8077(6)	0.2073(5)
C1R1	0.0737(10)	0.7458(14)	0.3907(7)
C2R1	0.1572(12)	0.7882(8)	0.3943(7)
C3R1	0.2084(7)	0.7278(11)	0.3543(8)
C4R1	0.1552(11)	0.6454(8)	0.3251(7)
C5R1	0.0691(10)	0.6541(10)	0.3451(8)
H1R1	0.0196	0.7751	0.4174
H2R1	0.1806	0.8598	0.4220
H3R1	0.2773	0.7431	0.3459
H4R1	0.1759	0.5835	0.2936
H5R1	0.0099	0.6028	0.3291
C1R2	-0.0991(8)	0.5985(8)	0.0223(7)
C2R2	-0.0914(8)	0.5752(7)	0.1122(8)
C3R2	-0.1623(8)	0.6282(8)	0.1343(7)
C4R2	-0.2140(6)	0.6880(7)	0.0539(10)
C5R2	-0.1713(8)	0.6663(8)	-0.0149(7)
H1R2	-0.0588	0.5637	-0.0164
H2R2	-0.0358	0.5271	0.1604
H3R2	-0.1772	0.6240	0.1980
H4R2	-0.2750	0.7335	0.0470
H5R2	-0.1865	0.7159	-0.0788

Table 8. Final thermal^a parameters ($\times 10^3$) for $(C_5H_5)_2Fe_2(CO)_3CS$

Atom	Molecule A					
	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Fe1	4.38(6)	5.84(7)	5.93(6)	-0.40(5)	1.19(5)	0.58(5)
Fe2	4.96(6)	5.71(7)	5.48(6)	0.10(5)	1.66(5)	-1.18(5)
S	11.9(2)	7.1(2)	8.8(2)	-0.7(1)	6.0(2)	1.0(1)
O1	11.4(5)	7.7(4)	10.3(5)	-2.4(4)	5.2(4)	-3.3(4)
O2	8.8(4)	8.7(4)	7.6(4)	3.7(4)	2.1(3)	0.7(3)
O3	7.3(4)	8.1(4)	8.8(4)	-1.6(3)	2.7(3)	1.8(3)
C1	7.1(5)	6.2(5)	6.1(5)	-2.2(4)	2.2(4)	0.8(4)
C2	5.6(5)	6.5(5)	4.9(4)	1.6(4)	1.0(3)	0.1(4)
C3	5.8(5)	5.5(5)	6.5(5)	0.1(4)	1.3(4)	0.3(4)
C4	6.2(5)	4.8(4)	6.9(5)	0.6(4)	2.8(4)	-0.4(4)
C1R1	5.7(7)	18.8(17)	16.3(13)	4.7(9)	2.9(8)	4.8(12)
C2R1	7.4(7)	11.5(10)	12.4(10)	3.8(7)	1.7(7)	1.5(8)
C3R1	5.7(6)	11.5(10)	9.9(7)	1.9(6)	-0.2(5)	2.2(7)
C4R1	7.3(7)	14.2(11)	11.4(9)	-0.2(7)	-1.6(6)	0.3(8)
C5R1	5.2(7)	9.4(10)	19.8(16)	-2.0(6)	-0.2(9)	2.4(10)
C1R2	30.0(30)	9.7(9)	5.0(6)	1.3(14)	4.5(12)	-2.3(6)
C2R2	12.5(12)	20.8(18)	14.4(13)	9.3(13)	-4.4(9)	-12.8(12)
C3R2	10.6(10)	13.9(11)	20.0(15)	-7.5(9)	9.2(11)	-12.9(11)
C4R2	16.7(13)	8.3(8)	13.2(11)	1.7(8)	6.5(10)	-2.6(8)
C5R2	11.2(10)	13.3(12)	8.8(8)	-2.9(9)	6.6(8)	-4.7(8)

^aThe β_{ij} are defined by: $T = \exp\{-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})\}$. For all hydrogen atoms an isotropic thermal parameter of 4.5 was assigned.

Table 8 (Continued)

Atom	<u>Molecule B</u>					
	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Fe1	4.82(6)	5.07(6)	3.84(5)	-0.11(5)	1.35(5)	0.22(5)
Fe2	4.69(6)	5.02(7)	4.35(6)	-0.52(5)	1.33(5)	0.32(5)
S	7.4(2)	8.0(2)	9.5(2)	1.6(1)	5.0(1)	-0.3(1)
O1	7.2(4)	5.8(4)	8.5(4)	-1.7(3)	3.1(3)	-0.4(3)
O2	9.2(5)	7.7(4)	7.8(4)	-0.1(4)	1.4(4)	3.0(4)
O3	7.3(4)	9.0(4)	6.6(3)	1.6(3)	3.7(3)	-0.4(3)
C1	4.6(4)	6.1(5)	4.5(4)	-0.1(4)	1.1(3)	-0.7(4)
C2	5.4(5)	6.8(6)	4.3(4)	0.0(4)	1.1(3)	0.4(4)
C3	6.0(5)	5.6(5)	4.5(4)	-0.4(4)	1.1(4)	1.0(3)
C4	5.8(5)	5.5(5)	5.1(4)	-1.0(4)	2.3(4)	0.5(4)
C1R1	10.4(9)	20.4(17)	4.7(6)	4.0(11)	4.0(6)	3.6(8)
C2R1	15.5(13)	10.0(9)	3.8(5)	0.4(9)	-0.8(7)	-0.3(5)
C3R1	6.9(7)	13.6(12)	7.4(7)	-0.8(7)	1.0(5)	3.9(7)
C4R1	12.7(11)	7.7(7)	6.3(6)	3.6(7)	2.3(7)	1.9(5)
C5R1	11.0(10)	11.6(10)	5.8(6)	-4.5(8)	-1.4(6)	3.7(6)
C1R2	10.5(8)	7.5(7)	7.7(7)	-3.6(6)	3.1(6)	-2.1(6)
C2R2	10.3(8)	5.8(6)	9.2(7)	-2.2(6)	2.9(6)	0.2(5)
C3R2	8.9(7)	8.2(7)	8.6(7)	-3.8(6)	4.3(6)	-0.7(6)
C4R2	3.7(5)	7.1(7)	17.1(11)	-1.8(5)	1.7(6)	-2.4(7)
C5R2	8.0(7)	9.0(8)	7.3(6)	-2.7(6)	0.4(6)	-0.1(6)

Figure 5. View of $(C_5H_5)_2Fe_2(CO)_3CS$ molecule A

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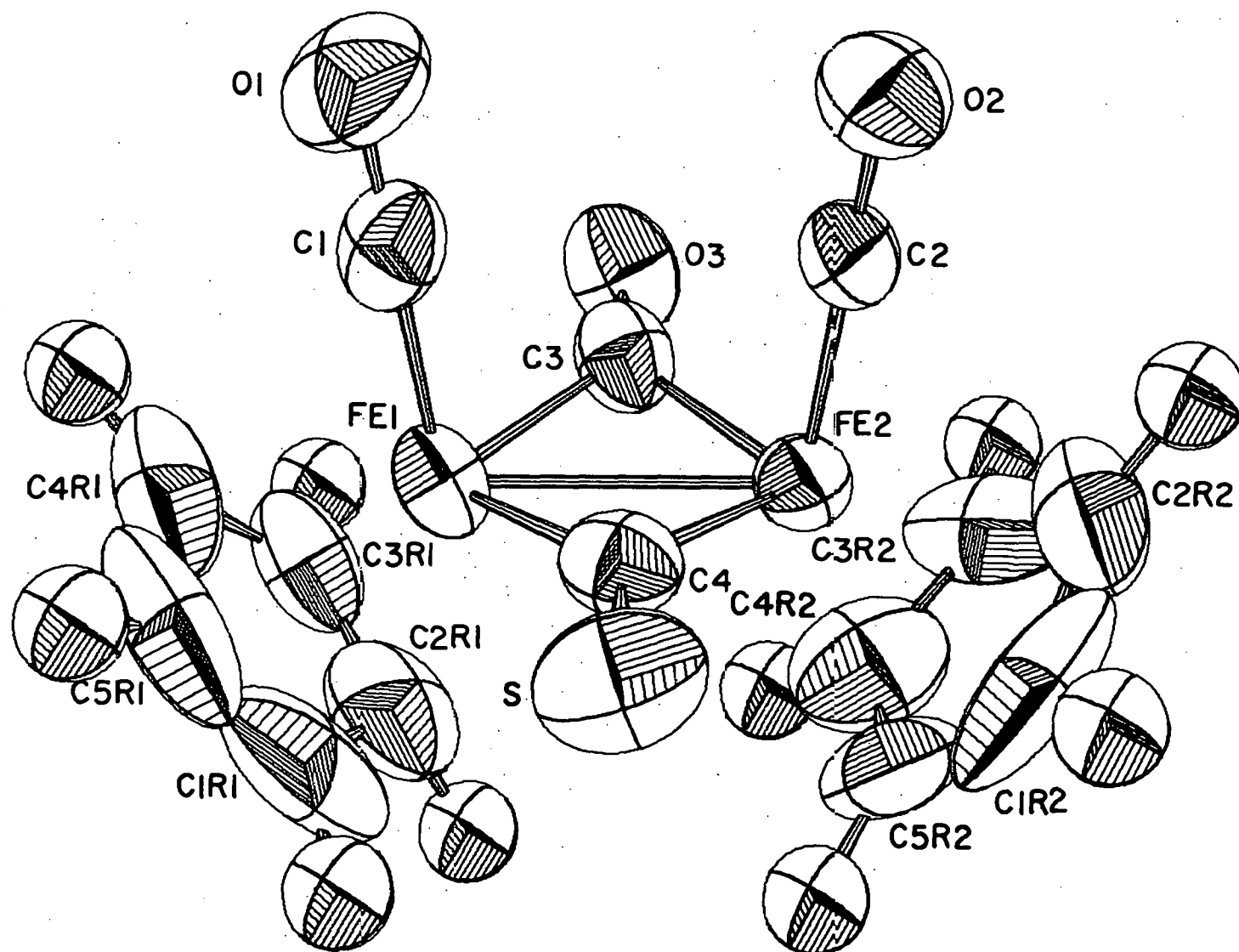


Figure 6. Unit cell stereograph of $(C_5H_5)_2Fe_2(CO)_3CS$

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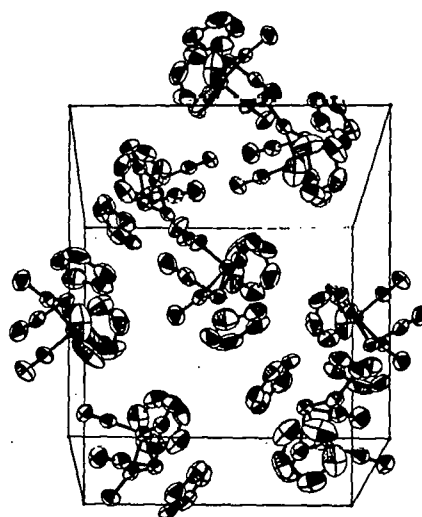
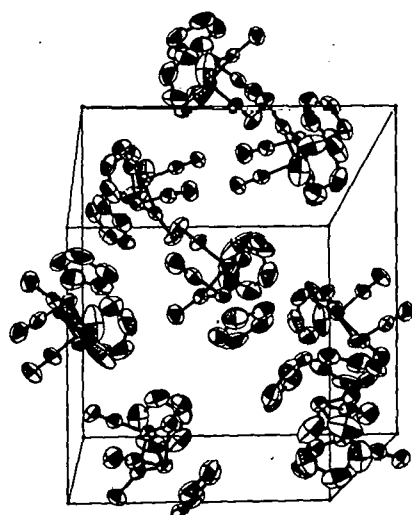


Table 9. Selected bonding interatomic distances (Å)
for $(C_5H_5)_2Fe_2(CO)_3CS$

	Molecule A	Molecule B
Fe1-Fe2	2.504(3)	2.506(2)
Fe1-C1	1.754(9)	1.754(8)
Fe2-C2	1.732(8)	1.742(8)
Fe1-C4	1.889(8)	1.890(8)
Fe2-C4	1.889(8)	1.875(8)
Fe1-C3	1.927(8)	1.920(8)
Fe2-C3	1.906(9)	1.907(8)
Fe1-cg1 ^a	1.752	1.752
Fe2-cg2	1.749	1.750
C1-O1	1.127(12)	1.143(10)
C2-O2	1.150(10)	1.155(11)
C3-O3	1.175(11)	1.179(11)
C4-S	1.596(9)	1.606(9)
C1R1-C2R1	1.515(22)	1.326(23)
C2R1-C3R1	1.321(21)	1.384(21)
C3R1-C4R1	1.482(18)	1.345(18)
C4R1-C5R1	1.395(25)	1.391(22)
C5R1-C1R1	1.319(26)	1.418(22)
C1R2-C2R2	1.441(35)	1.368(17)
C2R2-C3R2	1.322(25)	1.390(17)
C3R2-C4R2	1.319(23)	1.441(16)
C4R2-C5R2	1.317(23)	1.421(20)
C5R2-C1R2	1.368(23)	1.362(15)

^aCenter of gravity of the ring.

Table 10. Selected non-bonding^a interatomic distances (Å)
for $(C_5H_5)_2Fe_2(CO)_3CS$

O1A-H3R1A ^{II}	2.68	O1B-H5R1B ^{VI}	2.66
Sa-H3R1A ^{III}	2.97	O1B-H3R2B ^{VI}	2.68
SA-H3R1B ^{III}	3.09	SB-H4R1B ^{VI}	2.92
SA-H4R2A ^{IV}	3.13	SB-H2R2B ^{VI}	3.16
O4A-H4R1A ^{II}	2.39	O4B-H3R2A ^{II}	2.58
O4A-H3R1B ^I	2.62	O4B-H5R1A ^{IV}	2.60
O2A-H5R2B ^V	2.25	O2B-H2R2A ^{VII}	2.49
O2A-H3R2B ^{VI}	2.51		

^aContacts are between the first atom at x, y, z and the second at the equivalent position denoted by the Roman Numerals:

I	x	y	z
II	1-x	y	1-z
III	1-x	.5+y	.5-z
IV	1-x	-.5+y	.5-z
V	-x	-y	-z
VI	-x	.5+y	.5-z
VII	-x	1.5+y	-z

Table 11. Selected interatomic angles ($^{\circ}$) for
 $(C_5H_5)_2Fe_2(CO)_3CS$

	Molecule A	Molecule B
O3-C3-Fe1	137.8(6)	138.4(6)
O3-C3-Fe2	140.5(6)	139.7(6)
S-C4-Fe1	138.4(4)	138.0(4)
S-C4-Fe2	138.5(4)	138.5(4)
O3-C3-C4	173.8(7)	175.4(7)
S-C4-C3	174.2(5)	173.1(5)
C3-Fe1-Fe2	48.9(2)	48.9(2)
C3-Fe2-Fe1	49.6(2)	49.3(2)
C4-Fe1-Fe2	48.5(2)	48.0(2)
C4-Fe2-Fe1	48.5(2)	48.5(2)
C3-Fe1-C4	96.0(3)	95.9(3)
C3-Fe2-C4	96.7(3)	96.9(3)
Fe1-C1-O1	178.1(8)	178.5(9)
Fe2-C2-O2	177.9(8)	177.9(9)
C1-Fe1-C4	90.7(4)	88.6(3)
C1-Fe1-C3	89.1(4)	91.7(4)
C2-Fe2-C4	88.8(4)	89.3(4)
C2-Fe2-C3	90.1(4)	91.7(4)
C1-Fe1-Fe2	99.1(2)	98.0(2)
C2-Fe2-Fe1	98.6(2)	98.6(2)
Fe1-C3-Fe2	81.6(4)	81.8(4)
Fe1-C4-Fe2	83.0(3)	83.5(4)
C1-Fe1-cg1	125.1	124.7
C2-Fe2-cg2	125.5	124.9
Fe2-Fe1-cg1	135.7	137.2
Fe1-Fe2-cg2	135.9	136.5
C3-Fe1-cg1	121.9	121.1
C3-Fe2-cg2	121.6	120.6
C4-Fe2-cg1	124.4	125.3
C1R1-C2R1-C3R1	105.3(12)	110.3(12)
C2R1-C3R1-C4R1	110.0(12)	107.9(12)
C3R1-C4R1-C5R1	105.4(12)	108.2(11)
C4R1-C5R1-C1R1	110.5(13)	106.9(12)
C5R1-C1R1-C2R1	108.7(15)	106.8(14)
C1R2-C2R2-C3R2	104.1(15)	108.2(9)
C2R2-C3R2-C4R2	113.5(17)	107.4(11)
C3R2-C4R2-C5R2	106.4(13)	106.0(9)
C4R2-C5R2-C1R2	104.7(15)	107.8(10)
C5R2-C1R2-C2R2	110.9(16)	110.7(11)

of the CS group for the bridging rather than terminal position. Quick⁴³ suggests the bridging position should be preferred since the formation of the additional (second) Fe-CS bond compensates for any loss in C-S π -bonding. The carbonyl π -bond should be stronger than the thiocarbonyl π -bond and thus the CO should have less tendency to occupy the bridging position. Distances to the bridging carbons from the irons in the title compound appear to be quite similar to its carbonyl⁴⁴ and thiocarbonyl³⁹ analogs. The iron to terminal carbonyl-carbon distances, 1.745 Å (ave.), are uniformly shorter than the bridging distances, 1.91 Å (ave.), (cf. Table 9). The carbon-sulfur distance of 1.60(1) Å (ave.) is significantly longer than that found for terminal C=S groups (1.51-1.54 Å).⁴⁷

Other distances and angles in $\text{Cp}_2\text{Fe}_2(\text{CO})_3\text{CS}$ are strikingly similar to those in its analogs except for the Fe(1)-Fe(2) distance, 2.505 Å (ave.), which is intermediate to that of $\text{cis-}[\text{CpFe}(\text{CO})_2]_2$,⁴⁴ 2.531 Å, and that in $\text{cis-Cp}_2\text{Fe}_2(\text{CO})_2(\text{CS})_2$,³⁹ 2.482 Å. This supports any theory proposing enhancement of the bonding between the two halves of the molecule by the substitution of bridging thiocarbonyls for carbonyls.

The cyclopentadienyl rings are essentially planar, the greatest deviation from least-squares planarity being 0.032 Å, and assume cis orientation in the molecule (cf. Table 12). The C-C ring distances range from 1.319-1.515 Å, averaging 1.38 Å; the iron to ring carbon distances range from 2.06-2.14 Å, with the average distance being 2.11 Å. Such a variation is not unexpected considering the large thermal parameters and general difficulty encountered locating and refining the ring carbons and is probably indicative of considerable freedom of motion.

The bridging groups in the molecule are bent away from the cyclopentadienyl rings via a folding along the Fe-Fe bond. The dihedral angle between the least-squares planes of Fe1-C3-O3-Fe2 and Fe1-C4-S-Fe2 is 14.45° in molecule A and 12.93° in molecule B, the difference likely due to packing forces. Additional dihedral and torsional angles of interest are included in Table 13.

The packing seems to be van der Waals in nature with few short approaches between terminal oxygen or sulfurs and hydrogens (Table 10). Furthermore these short distances seem to have little bearing on the structure as evidenced by the close correspondence between the structural features of the two independent molecules in the asymmetric unit and by similarities to its analogs.

Table 12. Selected least squares planes^a for
 $(C_5H_5)_2Fe_2(CO)_3CS$ (Molecules A and B)

Planes defined by Fe1, Fe2, C3, O3, C4, S

A: $(0.60210)x + (-0.77433)y + (-0.19461)z = 2.14646$

B: $(-0.33724)x + (-0.84827)y + (0.40827)z = 4.08255$

Atom	Distance from plane (Å)	
	A	B
Fe1	0.1846	0.1611
Fe2	0.1842	0.1630
C3	-0.0338	-0.0353
O3	-0.1678	-0.1488
C4	-0.0092	0.0121
S	-0.1579	-0.1522

^aPlanes are defined by $c_1X + c_2Y + c_3Z - d = 0$,
 where X, Y, and Z are Cartesian coordinates which are
 related to the triclinic cell coordinates (x,y,z) by
 the transformations:

$$X = x \sin \gamma + z \{ (\cos \beta - \cos \alpha \cos \gamma) / \sin \gamma \} = x a + z c \cos \beta$$

$$Y = x \cos \gamma + y b + z c \cos \alpha = y b$$

$$Z = z \{ (1 - \cos^2 \alpha - \cos^2 \beta - \cos^2 \gamma + 2 \cos \alpha \cos \beta \cos \gamma)^{1/2} / \sin \gamma \} = z c \sin \beta$$

Table 13. Configurational similarities of Molecules A and B for $(C_5H_5)_2Fe_2(CO)_3CS$

<u>Plane</u>		<u>Defined by</u>	
1		Fe1, Fe2, C3, O3	
2		Fe1, Fe2, C4, S	
3		C1R1-C5R1	
4		C1R2-C5R2	
5		Fe1, Fe2, C3, C4	

<u>Plane</u>		<u>Dihedral Angle (°)</u>	
		<u>A</u>	<u>B</u>
1	2	14.45	12.93
3	5	47.50	47.78
4	5	47.65	47.65

		<u>Torsional Angle (°)</u>	
		<u>A</u>	<u>B</u>
C1, Fe1, Fe2, C2		2.03	0.05
O3, C3, Fe1, C1		-80.17	84.38
O3, C3, Fe2, C2		82.92	-83.62
S, C4, Fe1, C1		82.24	-78.30
S, C4, Fe2, C2		-81.29	78.15

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And finally, my parents for their love and support through the good and statistical thermodynamics.

APPENDIX A: OBSERVED AND CALCULATED STRUCTURE
FACTORS FOR DOWCO 214

				-5	2	40	38	-3	1	35	-34
				-5	3	8	-10	-3	2	34	-29
				-5	4	4	5	-3	4	26	23
				-5	5	7	-9	-3	5	23	22
K = -16				-4	1	33	32	-3	6	15	-15
H	L	F0	FC	-4	2	14	15	-2	1	17	12
-4	1	4	-7	-4	3	5	-12	-2	2	22	21
-3	1	18	17	-4	4	26	-25	-2	3	6	-9
-2	2	9	11	-4	5	5	6	-2	4	14	-12
-1	1	21	-19	-3	1	6	-11	-2	5	23	-19
				-3	2	8	-12	-2	6	18	16
K = -15				-3	3	18	-15	-1	1	25	-22
H	L	F0	FC	-3	5	7	12	-1	2	21	20
-7	1	3	-5	-2	1	5	-5	-1	5	6	-6
-7	2	8	-9	-2	2	4	-1	0	2	8	11
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8	0	14	11	-1	4	24	-24	8	4	6	-10
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9	0	23	27	0	2	13	12	10	0	4	-9
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9	2	20	-22	1	0	6	-4	10	2	3	4
9	4	7	10	1	1	29	26	11	0	6	-11
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10	1	8	12	1	4	41	-38				
10	2	24	-28	1	5	7	10				
10	3	13	-16	1	6	5	9				
11	1	6	11	2	0	50	-46				
11	2	17	18	2	1	31	28				
12	0	13	20	2	2	16	17				
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				2	6	4	4				
				3	0	44	44				
				3	1	11	10				
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				3	3	45	-44				
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				4	0	15	15				
				4	1	58	56				
				4	2	55	-46				
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				4	4	17	17				
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H	L	FC	FC					H	L	FC	FC
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-7	2	33	-32					-6	2	10	-12
-7	3	11	16					-6	3	16	19
-6	2	11	-12					-5	1	3	0
-6	4	20	20					-5	2	15	-16
-5	0	18	-17					-5	4	7	8
-5	1	10	-12					-4	0	19	17
-5	2	8	8					-4	1	4	6
-5	3	24	23					-4	2	16	18
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-4	0	16	-16					-3	1	12	-9
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-4	2	16	-15					-3	3	9	12
								-3	4	6	-9
								-2	0	18	18
								-2	1	4	-2
								-2	2	3	-4
								-2	3	25	-24
								-2	4	24	22

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-1	0	20	19	10	0	6	7	8	1	18	-19
-1	1	9	-12	10	1	14	-15	8	2	3	0
-1	2	13	15					9	0	27	31
-1	3	18	24	K = 13							
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-1	5	7	13	-5	1	17	-18	H	L	FO	FC
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0	1	18	-17	-4	2	5	-8	-4	1	7	-9
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2	4	7	-9	1	1	26	22	2	0	7	6
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6	0	50	-46	4	0	14	-14	7	1	10	-11
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6	2	34	35	4	3	15	15	8	0	8	9
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6	5	12	-16	5	2	9	10				
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8	1	11	17	6	4	25	-25	1	3	6	7
8	2	7	-13	7	1	29	32	2	0	22	20
9	0	10	13	7	2	5	-6	2	2	10	-12
9	1	5	-4	7	3	20	-23	2	3	20	-21

3	0	11	12
4	0	7	-10
4	1	3	-5
4	2	3	0
5	0	15	17
5	1	17	-17
5	2	4	7
6	0	7	9
6	1	18	15

K = 16

H	L	FC	FC
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4	0	13	16

APPENDIX B: OBSERVED AND CALCULATED STRUCTURE
FACTORS FOR $(C_5H_5)_2Fe_2(CO)_3CS$

L = 16				3 13	5	7	8	2	130	125
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				4 4	13	-14	8	7	4	-4
L = 17				4 5	16	-16	8	8	8	8
H	K	FC	FC	4 6	8	7	8	9	16	-15
-8	4	3	2	4 7	8	-9	8	10	11	11
-7	2	3	4	4 8	37	-37	8	11	6	-6
-5	3	4	4	4 9	4	5	8	13	3	-4
				4 10	20	-20	9	0	83	-80
				4 12	4	-5	9	2	9	9
L = 18				4 13	5	-7	9	3	21	20
H	K	FC	FC	4 14	9	-10	9	4	50	-47
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1	6	103	-104	5 1	30	-27	9	7	34	30
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1	14	4	4	5 7	15	-16	10	1	12	-11
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2	3	79	-83	5 10	22	21	10	5	7	-7
2	4	59	62	5 12	5	7	10	7	7	-8
2	5	7	8	5 14	7	10	10	8	3	-4
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2	7	7	-8	6 1	13	-13	10	11	13	-13
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2	10	6	-6	6 4	25	-25	11	3	3	3
2	11	7	-8	6 6	64	66	11	4	15	15
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3	1	10	9	7 2	13	13	12	1	4	6
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3	3	28	-29	7 4	112	-112	12	3	2	3
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3	6	33	-34	7 8	23	-24	12	6	18	-18
3	7	42	38	7 11	2	-1	12	7	21	-18
3	8	13	12	7 12	14	-14	12	9	5	-8
3	9	32	29	8 0	11	8	12	10	9	-12
3	10	22	21	8 1	27	-25	13	0	25	22
3	12	19	17							

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13	4	18	16	-12	9	14	-15	-6	5	63	57
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[illegible]