

Fig. 2. Projection of the Fe environment onto the cyclopentadienyl plane.

isotropic thermal parameters, while bond distances and angles may be found in Table 2. Fig. 1 displays a view of the molecule with the atom numbering and Fig. 2 shows a projection of the Fe-atom environment onto the cyclopentadienyl plane.

Related literature. Structures of two other related cyclopentadienyl-Fe^{II} derivatives have been published (Sanz-Aparicio, Martínez-Carrera & García-Blanco, 1986*a,b*). For other dithiophosphate ester ligands in a monodentate configuration see Molloy, Hossain, van

der Helm, Zuckerman & Haiduc (1979) and Shetty & Fernando (1970).

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The Structure of 3,5-Dinitroisoxazole*

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Abstract. C₃HN₃O₅, $M_r = 159.06$, $P\bar{1}$, $a = 9.858$ (3), $b = 10.382$ (3), $c = 11.675$ (2) Å, $\alpha = 115.92$ (2), $\beta = 111.37$ (2), $\gamma = 60.91$ (3)°, $V = 921.39$ Å³, $Z = 6$, $D_x = 1.720$ Mg m⁻³, $\lambda(\text{Mo } K\alpha_1) = 0.70927$ Å, $\mu = 0.16$ mm⁻¹, $F(000) = 480$, room temperature, final $R = 0.044$ for 1215 observed reflections with $I > 2\sigma(I)$ out of 2411 independent reflections. There are three molecules in the asymmetric unit. The packing is very inefficient as the planes of these three molecules are almost normal to each other. The geometries of the

three molecules are very similar. Rings are planar within 0.005 Å. Possible C–H...O hydrogen bonds (3.35 Å) are present.

Experimental. Title compound prepared by method of Golod, Novatskii & Bagal (1973). Crystals for X-ray diffraction grown by sublimation. Selected crystal ca 0.43 × 0.19 × 0.11 mm. CAD-4 diffractometer, θ - 2θ scan. Scan range $1 + 0.34 \tan \theta$ °, scan speed 1.6 to 5.5° min⁻¹. Background first and last 1/6 of scan. Graphite-monochromated Mo $K\alpha$ radiation. Unit cell, 25 reflections $7 < \theta < 20$ °. No absorption corrections. $(\sin \theta)/\lambda$ max. = 0.540 Å⁻¹. Index range $-10 \leq h \leq 10$, $-11 \leq k \leq 11$, $-12 \leq l \leq 12$; 4812 reflections

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Table 1. Final least-squares parameters for 3,5-dinitroisoxazole

Positional parameters for C, N, O $\times 10^4$, for H $\times 10^3$, equivalent isotropic $U \times 10^3$. $U = \frac{1}{3} \sum U_{ii}$.

	x	y	z	$U(\text{\AA}^2)$
Molecule 1				
C(1)	0293 (6)	7182 (7)	5096 (5)	6.0 (7)
C(2)	0464 (6)	8408 (7)	6172 (6)	5.8 (7)
C(3)	1711 (6)	7661 (6)	6930 (5)	4.9 (6)
N(1)	1319 (5)	5828 (5)	5164 (4)	5.3 (5)
N(2)	-918 (7)	7250 (7)	3923 (5)	8.5 (8)
N(3)	2465 (6)	8242 (6)	8242 (4)	6.6 (6)
O(1)	2263 (4)	6135 (4)	6384 (4)	7.5 (5)
O(2)	-889 (6)	6042 (6)	3096 (4)	11.9 (7)
O(3)	-1815 (5)	8502 (6)	3874 (4)	11.1 (7)
O(4)	1952 (5)	9633 (3)	8733 (4)	8.9 (5)
O(5)	3547 (5)	7307 (5)	8768 (4)	8.2 (5)
H	-13 (6)	949 (6)	631 (5)	9.2 (19)
Molecule 2				
C(1)	4983 (6)	7712 (7)	1864 (4)	5.3 (6)
C(2)	4883 (6)	6578 (7)	2089 (5)	4.5 (6)
C(3)	3783 (6)	7414 (6)	2826 (5)	5.0 (6)
N(1)	4065 (5)	9107 (5)	2414 (4)	6.3 (5)
N(2)	5950 (5)	7523 (7)	1066 (4)	6.9 (6)
N(3)	3028 (6)	6952 (6)	3347 (4)	6.7 (6)
O(1)	3255 (4)	8925 (4)	3045 (3)	7.6 (5)
O(2)	5946 (5)	8683 (5)	1067 (4)	11.0 (6)
O(3)	6658 (4)	6184 (5)	455 (4)	8.6 (5)
O(4)	3572 (5)	5589 (5)	3211 (4)	10.5 (6)
O(5)	1921 (5)	7955 (4)	3873 (4)	9.0 (5)
H	543 (5)	553 (5)	181 (4)	6.3 (16)
Molecule 3				
C(1)	826 (7)	2678 (6)	1025 (5)	5.5 (7)
C(2)	2288 (7)	2757 (6)	1494 (6)	6.2 (7)
C(3)	2670 (6)	2561 (5)	2620 (5)	5.2 (7)
N(1)	351 (5)	2472 (5)	1791 (5)	7.4 (6)
N(2)	-252 (8)	2828 (7)	-215 (6)	8.4 (9)
N(3)	4109 (6)	2442 (5)	3618 (5)	6.9 (6)
O(1)	1565 (5)	2396 (4)	2855 (4)	8.6 (5)
O(2)	-1341 (6)	2486 (7)	-511 (5)	13.9 (8)
O(3)	85 (7)	3279 (7)	-818 (5)	12.2 (8)
O(4)	5178 (5)	2403 (5)	3325 (5)	11.8 (7)
O(5)	4079 (5)	2386 (5)	4618 (4)	10.6 (6)
H	286 (5)	285 (4)	113 (4)	4.4 (15)

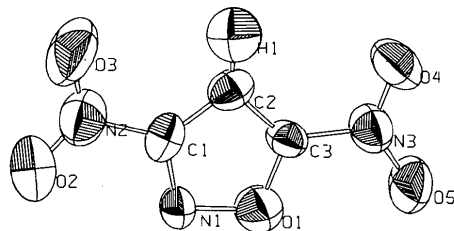


Fig. 1. ORTEP (Johnson, 1965) drawing of molecule 1 to show atom-numbering scheme.

measured and averaged to yield 2411 unique reflections of which 1214 were observed with $I > 2\sigma(I)$, $R_F = 0.018$. Standard reflections $1\bar{3}\bar{2}$ and $1\bar{2}\bar{1}$ showed no significant variation. Least squares minimized $\sum w(\Delta F)^2$ with $w = [\sigma_c^2(F) + 0.03F^2]^{-1}$, $\sigma_c^2(F)$ based on counting statistics. Structure solved by *MULTAN* (Germain, Main & Woolfson, 1971). Scale factor, positional parameters, anisotropic thermal parameters for C, N, O, and isotropic thermal parameters for H

Table 2. Bond distances (\AA) and angles ($^\circ$) and possible C—H...O bonds (\AA , $^\circ$) in 3,5-dinitroisoxazole

	Molecule 1	Molecule 2	Molecule 3
C(1)—C(2)	1.373 (6)	1.366 (6)	1.373 (7)
C(1)—N(1)	1.289 (6)	1.291 (5)	1.279 (6)
C(1)—N(2)	1.454 (7)	1.457 (6)	1.471 (8)
C(2)—C(3)	1.332 (6)	1.334 (6)	1.308 (6)
C(2)—H(1)	0.95 (5)	0.91 (4)	0.88 (3)
C(3)—N(3)	1.435 (6)	1.445 (6)	1.461 (7)
C(3)—O(1)	1.320 (5)	1.323 (5)	1.316 (5)
N(1)—O(1)	1.386 (4)	1.383 (4)	1.375 (5)
N(2)—O(2)	1.197 (5)	1.202 (5)	1.180 (6)
N(2)—O(3)	1.177 (5)	1.217 (5)	1.198 (6)
N(3)—O(4)	1.203 (5)	1.202 (5)	1.199 (5)
N(3)—O(5)	1.222 (5)	1.215 (4)	1.205 (5)
Angles ($^\circ$)			
C(2)—C(1)—N(1)	115.0 (5)	114.3 (5)	114.5 (5)
C(2)—C(1)—N(2)	127.4 (6)	128.0 (6)	128.5 (6)
N(1)—C(1)—N(2)	117.6 (5)	117.7 (5)	117.0 (6)
C(1)—C(2)—C(3)	100.9 (5)	101.6 (5)	101.4 (5)
C(1)—C(2)—H(1)	128.6 (30)	129.2 (26)	130.5 (28)
C(3)—C(2)—H(1)	130.5 (31)	129.2 (26)	128.0 (28)
C(2)—C(3)—N(3)	130.5 (5)	131.5 (5)	131.4 (6)
C(2)—C(3)—O(1)	113.0 (5)	112.6 (5)	112.8 (5)
N(3)—C(3)—O(1)	116.4 (5)	115.9 (5)	115.7 (6)
C(1)—N(1)—O(1)	104.2 (4)	104.9 (4)	104.1 (4)
C(1)—N(2)—O(2)	117.4 (6)	117.4 (5)	116.9 (7)
C(1)—N(2)—O(3)	116.1 (6)	115.0 (5)	115.9 (7)
O(2)—N(2)—O(3)	126.4 (7)	127.6 (6)	127.2 (8)
C(3)—N(3)—O(4)	116.0 (5)	115.6 (6)	115.0 (5)
C(3)—N(3)—O(5)	118.5 (5)	118.0 (5)	116.6 (6)
O(4)—N(3)—O(5)	125.6 (5)	126.4 (5)	128.4 (6)
C(3)—O(1)—N(1)	106.8 (4)	106.6 (4)	107.1 (4)
Hydrogen bonds (\AA, $^\circ$)			
$d(\text{C}\cdots\text{O})$ (\AA)		$d(\text{H}\cdots\text{O})$ (\AA)	$\angle \text{C—H}\cdots\text{O}$ ($^\circ$)
Molecule 1...2			
C(2)—H(1)...O(5)	3.35 (1)	2.42 (5)	167 (4)
Molecule 2...1			
C(2)—H(1)...O(5)	3.35 (1)	2.45 (4)	167 (3)

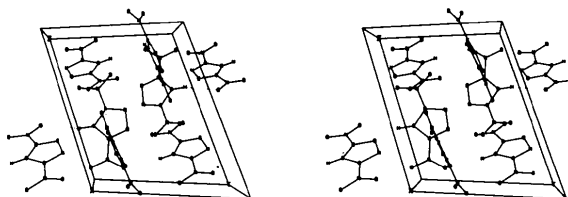


Fig. 2. Stereo drawing of unit cell plus two molecules. The possible C(2)—H(1)...O(5) hydrogen bonds are indicated as dotted lines. The origin is at the lower left rear.

were refined. Final $R = 0.044$, $wR = 0.039$, $S = 1.5$. Max. $\Delta/\sigma = 0.04$. Final ΔF Fourier synthesis $-0.28 < \Delta\rho < 0.22 \text{ e \AA}^{-3}$. Scattering factors f (RHF for C, N, O and SDS for H), f' , f'' from *International Tables for X-ray Crystallography* (1974). Calculations on CDC-7600 using the Los Alamos Crystal Structure System developed primarily by A. C. Larson.*

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44027 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Molecule 1, showing the atom-numbering scheme is in Fig. 1. Final parameters are given in Table 1. Bond lengths and angles are given in Table 2. The ring bonds are shorter than in 5-phenyl-3-isoxazolol (Biagini, Cannas & Marongiu, 1969) and in *N*-(5-methyl-3-isoxazolyl)sulfanilamide (Bettinetti, Giordano, La Manna, Giuseppetti & Tadini, 1982). Nitro-group parameters are in the range of values found in other compounds. A stereo drawing of the unit cell is shown in Fig. 2. Nitro groups are twisted out of the ring plane by 1.9 and 3.8° for molecule 1, 7.2 and 6.5° for molecule 2, and 12.0 and 8.1° for molecule 3 (e.s.d. ~0.3°). Possible C—H...O hydrogen bonds are given in Table 2. The observed density is lower than the prediction of 1.86 Mg m⁻³ by Stine's (1981) method or 1.92 Mg m⁻³ by Cromer & Ryan (1985).

Related literature. Sutor (1962) discussed the existence of C—H...O hydrogen bonds. Cromer, Coburn, Ryan & Wasserman (1986) note the possibility of this type bond in 1-methyl-2,3,4,5-tetranitropyrrrole.

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Dimethyl(thio)l(trimethylsilyl)amino]phosphorane

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Abstract. C₅H₁₆NPSSi, *M*_r = 181.31, monoclinic, *P*2₁/*n*, *a* = 6.306 (1), *b* = 10.128 (6), *c* = 16.929 (6) Å, β = 96.14 (5)°, *V* = 1075.13 (19) Å³, *Z* = 4, *D*_x = 1.12 g cm⁻³, λ(Mo *K*α) = 0.71073 Å, μ = 4.85 cm⁻¹, *F*(000) = 392, *T* = 295 K. Final *R* = 0.064 for 1016 observed unique reflections. The structure shows that the compound has a distorted trigonal-planar geometry around the central N atom and the N—H and P=S bonds are in *trans* positions with respect to the N—P bond. The S—P—N—Si and S—P—N—H torsion angles are 176 (3) and -18.5 (6)°. The deviation of the H atom from the P—N—Si plane is 0.23 (5) Å. The bond distances, S=P, P—N, Si—N, av. P—C, and av. Si—C are 1.952 (3), 1.634 (6), 1.741 (7), 1.819 (10), and 1.855 (10) Å, respectively.

Experimental. The title compound was synthesized quantitatively by the hydrolysis of (Me₃Si)₂NP(=S)Me₂, which is one of the unexpected by-

products of the reaction between (Me₃Si)₂NP(Me)₂ and N≡SF₃ (Hosmane & Maldar, 1987, unpublished results). Large well formed colorless crystals of the title compound were grown by sublimation onto a glass surface. A single crystal (0.3 × 0.3 × 0.2 mm) was coated with an epoxy resin and mounted on an Enraf–Nonius CAD-4F diffractometer. The unit-cell parameters were determined by least-squares fit of 15 reflections in the range 10 ≤ 2θ ≤ 24°, and the space group *P*2₁/*n* was assigned on the basis of systematic absences (0*k*0, *k* odd, *h*0*l*, *h* + *l* odd). A total of 2013 independent reflections collected in the range 2 ≤ 2θ ≤ 50° (*hkl* range *h* 0→7, *k* 0→11, *l* -20→20) using graphite-monochromated Mo *K*α radiation and the θ/2θ scan mode, 1016 observed reflections with *I* > 3σ(*I*), σ(*I*) from counting statistics; three standard reflections remeasured after every 100 reflections did not show any significant change in intensity during data collection. Lorentz–polarization corrections applied,