

Fig. 1. The structure of the [Fe(tpp)(Him)₂]⁺ cation and the [Cu(mnt)₂]⁻ anion (50% probability thermal ellipsoids). H atoms have been omitted for clarity.

hydrogen bonded to the coordinated imidazole; in the other, O(1) and C(30) are subject to unresolvable disorder, which gives rise to high thermal parameters for those two atoms.

A number of related compounds have been studied: (tba)[Cu(mnt)₂] (Forrester, Zalkin & Templeton, 1964); [Fe(tpp)(Him)₂]Cl (Collins, Countryman & Hoard, 1972); [Fe(oep)(2-MeHim)₂]ClO₄ (Geiger, Lee & Scheidt, 1984; oep = 2,3,7,8,12,13,17,18-octaethylporphinate); [Fe(tpp)(2-MeHim)₂]ClO₄ (Kirner & Hoard, 1978); {K(cryptand[2.2.2])}-[Fe(tpp)(4-MeHim)₂] (Quinn, Strouse & Valentine, 1983); [Fe(tpp)(1-Meim)₂] (Steffen, Chun & Hoard, 1978); [Fe(tpp)(Hbim)₂]ClO₄ (Levan & Strouse, 1983; Hbim = benzimidazole). The metric parameters for the title compound suggest that Fe is low spin (Scheidt & Reed, 1981). The relationship

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Methyl 2-[(2,6-Dimethoxyphenyl)ethynyl]-3-methoxybenzoate

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Abstract. C₁₉H₁₈O₅, *M_r* = 326.4, orthorhombic, *Pna*2₁, *a* = 19.071 (2), *b* = 7.7090 (11), *c* = 11.3771 (12) Å, *V* = 1672.6 (6) Å³, *Z* = 4, *D_x* =

between imidazole orientation and Fe spin state has been discussed (Scheidt & Chipman, 1986).

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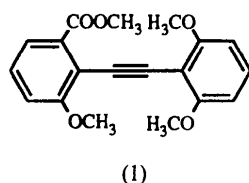
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1.296 g cm⁻³ at 298 (1) K, λ(Cu Kα) = 1.54184 Å, μ = 7.36 cm⁻¹, *F*(000) = 688, 1811 unique data measured, final *R* = 0.031 for 1745 reflections with *I* > 3.0σ(*I*). Maximum deviations from planarity of the two aromatic rings are 0.0149 (13) Å for the ring

bearing the two methoxy substituents, and 0.0060 (13) Å for the other ring. The dihedral angle between the rings is 112.81 (6)°. The methoxy substituent on the ring also bearing the ester is nearly coplanar with the benzenoid ring, with C—C—O—C torsion angle of -0.9 (2)°. The methoxy group on the adjacent ring nearest the ester is twisted the farthest from coplanarity with the benzenoid ring, with C—C—O—C torsion angle of -19.7 (3)°. The third methoxy group is nearly coplanar with the benzenoid ring with C—C—O—C torsion angle of 7.8 (2)°. The triple-bond distance is 1.195 (2) Å, and bond angles at the two ethynylic C atoms are 175.0 (1) and 177.7 (1)°.

Experimental. A colorless crystal of (1) was isolated by evaporation from ethyl acetate from the crude



reaction product of 2-ethynyl-1,3-dimethoxybenzene, methyl 3-methoxy-2-(trifluoromethylsulfonyl)benzoate, bis(triphenylphosphine)palladium(II) chloride, and triethylamine in *N,N*-dimethylformamide. Crystal size 0.33 × 0.50 × 0.50 mm, space group from systematic absences $0kl$ with $k+l$ odd and $h0l$ with h odd, and successful refinement of a noncentrosymmetric model. Cell dimensions from setting angles of 25 reflections having $20 < \theta < 30^\circ$. Data collection on Enraf-Nonius CAD-4 diffractometer, Cu $K\alpha$ radiation, graphite monochromator, ω - 2θ scans designed for $I = 50\sigma(I)$, subject to max. scan time = 120 s, scan rates varied 0.46–16.5° min⁻¹. Two octants of data with $4 < 2\theta < 150^\circ$, $-23 \leq h \leq 23$, $0 \leq k \leq 9$, $0 \leq l \leq 14$ measured. Data corrected for background, Lorentz and polarization effects. Three standard reflections 200, 020, 004 varied randomly, no decay correction was applied. Absorption corrections were based on ψ scans, with relative transmission coefficients ranging from 0.9636 to 0.9987. 3894 total data were collected, and redundant data merged, $R_{\text{int}} = 0.017$, to yield 1811 unique data, 1745 observed with $I > 3\sigma(I)$. Structure solved by direct methods, using *MULTAN82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), refined by full-matrix least squares based upon F with weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$ using Enraf-Nonius *SDP/VAX* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974). Non-H atoms refined anisotropically; H atoms located by ΔF synthesis. Methyl H atoms fixed with C—H 0.95 Å and $B_{\text{iso}} = 1.3B_{\text{eq}}$ for the methyl C atom.

Table 1. Fractional atomic coordinates and equivalent isotropic temperature factors

$$B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	B_{eq} (Å ²)
O1	0.60132 (6)	0.1591 (1)	0	4.36 (2)
O2	0.54287 (7)	0.6758 (2)	-0.1899 (1)	5.38 (3)
O3	0.70736 (6)	0.6110 (2)	0.2275 (1)	4.42 (2)
O4	0.8356 (1)	0.5701 (3)	-0.2342 (2)	8.61 (4)
O5	0.73528 (7)	0.7149 (2)	-0.2181 (1)	5.10 (2)
C1	0.55431 (7)	0.2401 (2)	-0.0702 (1)	3.63 (2)
C2	0.49580 (9)	0.1605 (2)	-0.1207 (2)	4.84 (3)
C3	0.45326 (8)	0.2557 (3)	-0.1945 (2)	5.47 (4)
C4	0.46632 (8)	0.4268 (3)	-0.2204 (2)	5.16 (3)
C5	0.52438 (8)	0.5075 (2)	-0.1694 (2)	3.97 (2)
C6	0.56818 (6)	0.4165 (2)	-0.0921 (1)	3.22 (2)
C7	0.62911 (6)	0.4961 (2)	-0.0427 (1)	3.03 (2)
C8	0.68138 (7)	0.5608 (2)	-0.0047 (1)	3.08 (2)
C9	0.74737 (7)	0.6300 (2)	0.0334 (1)	3.18 (2)
C10	0.76083 (7)	0.6518 (2)	0.1545 (2)	3.66 (2)
C11	0.82610 (9)	0.7093 (3)	0.1925 (2)	4.93 (3)
C12	0.87833 (9)	0.7433 (3)	0.1122 (3)	5.54 (4)
C13	0.86670 (8)	0.7223 (2)	-0.0069 (2)	5.08 (4)
C14	0.80122 (7)	0.6672 (2)	-0.0467 (2)	3.79 (2)
C15	0.79246 (9)	0.6425 (2)	-0.1761 (2)	4.47 (3)
C16	0.5963 (1)	-0.0251 (2)	0.0130 (2)	5.79 (4)
C17	0.5159 (1)	0.7556 (3)	-0.2918 (2)	6.60 (4)
C18	0.7194 (1)	0.6261 (3)	0.3514 (2)	5.19 (3)
C19	0.7240 (2)	0.6938 (3)	-0.3431 (2)	6.39 (5)

Table 2. Bond distances (Å) and angles (°)

O1	C1	1.353 (2)	C4	C5	1.396 (2)		
O1	C16	1.430 (2)	C5	C6	1.401 (2)		
O2	C5	1.364 (2)	C6	C7	1.429 (2)		
O2	C17	1.409 (3)	C7	C8	1.195 (2)		
O3	C10	1.352 (2)	C8	C9	1.434 (2)		
O3	C18	1.432 (2)	C9	C10	1.412 (2)		
O4	C15	1.194 (3)	C9	C14	1.402 (2)		
O5	C15	1.315 (2)	C10	C11	1.390 (2)		
O5	C19	1.447 (3)	C11	C12	1.377 (3)		
C1	C2	1.397 (2)	C12	C13	1.383 (4)		
C1	C6	1.408 (2)	C13	C14	1.395 (2)		
C2	C3	1.379 (3)	C14	C15	1.494 (3)		
C3	C4	1.374 (3)					
C1	O1	C16	118.4 (1)	C7	C8	C9	175.0 (1)
C5	O2	C17	117.5 (2)	C8	C9	C10	119.9 (1)
C10	O3	C18	117.7 (1)	C8	C9	C14	121.5 (1)
C15	O5	C19	115.6 (2)	C10	C9	C14	118.5 (1)
O1	C1	C2	124.7 (1)	O3	C10	C9	115.8 (1)
O1	C1	C6	115.2 (1)	O3	C10	C11	124.0 (2)
C2	C1	C6	120.1 (1)	C9	C10	C11	120.3 (1)
C1	C2	C3	119.1 (2)	C10	C11	C12	120.2 (2)
C2	C3	C4	122.3 (2)	C11	C12	C13	120.8 (2)
C3	C4	C5	118.9 (2)	C12	C13	C14	119.8 (2)
O2	C5	C4	123.9 (2)	C9	C14	C13	120.5 (2)
O2	C5	C6	115.4 (1)	C9	C14	C15	122.2 (1)
C4	C5	C6	120.7 (2)	C13	C14	C15	117.3 (2)
C1	C6	C5	118.9 (1)	O4	C15	O5	124.7 (2)
C1	C6	C7	119.9 (1)	O4	C15	C14	121.9 (2)
C5	C6	C7	121.1 (1)	O5	C15	C14	113.4 (1)
C6	C7	C8	177.7 (1)				

Other H atoms were refined isotropically. Final $R = 0.031$ (0.033 for all data), $wR = 0.045$, $S = 2.859$ for 241 variables. Max. shift $< 0.01\sigma$ in the final cycle, max. residual density 0.18 (3), min. -0.11 (3) e Å⁻³, extinction coefficient $g = 7.1 (3) \times 10^{-6}$, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c .

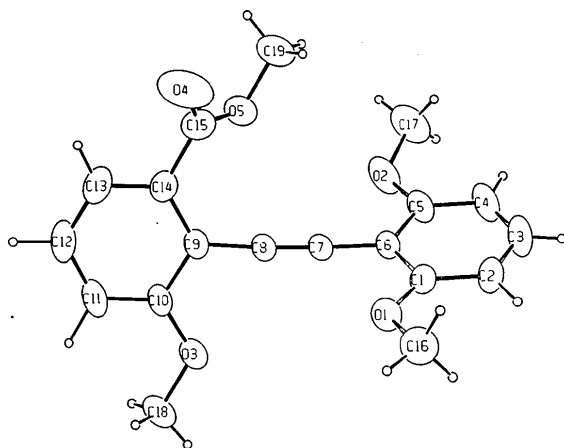


Fig. 1. ORTEP (Johnson, 1965) drawing of the title compound, with thermal ellipsoids drawn at the 50% probability level.

Coordinates* and equivalent isotropic thermal parameters are given in Table 1; bond distances and angles are given in Table 2. The molecule is illustrated in Fig. 1.

Related literature. Structure of 2-ethynyl-1,3-dimethoxybenzene: Evans, Horn, Fronczek, Gandour & Watkins (1990). Structure of 1,2-bis(9-anthryl)acetylene: Becker, Skelton & White (1985). Structure of bis(*m*-chlorophenyl)acetylene: Espiritu & White (1977). Structure of diphenylacetylene: Mavridis & Moustakali-Mavridis (1977). Structure of *p*-butyl-*p*'-methoxydiphenylacetylene: Cotrait (1977).

* Tables of H-atom positional parameters, least-squares planes, anisotropic thermal parameters, and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52329 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 1-Chloro-3,3,5,5-tetraphenyl-1,3,5,2,4,6-selenadiphosphatriazine

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Abstract. C₂₄H₂₀ClN₃P₂Se, *M_r* = 526.8, triclinic, *P* $\bar{1}$, *a* = 9.759 (1), *b* = 11.046 (4), *c* = 13.332 (2) Å, α = 65.50 (2), β = 64.89 (1), γ = 74.96 (2)°, *V* = 1178.1 (4) Å³, *Z* = 2, *D_x* = 1.49 g cm⁻³, $\lambda(\text{Mo } K\alpha)$ = 0.71073 Å, μ = 18.4 cm⁻¹, *F*(000) = 532, *T* = 293 K, *R* = 0.036 for 2048 reflections with *F_o*² > 3σ(*F_o*²). In the SeNPNP ring the P₂N₃ linkage is planar within 0.053 (2) Å and the Se atom is 0.499 (1) Å out of the

plane on the same side as the Cl atom. The bond distances of the Se—N—P—N sequence average 1.730 (9), 1.637 (5) and 1.584 (4) Å and the ring angles at Se and P are 111.5 and 117.8 (2)°, respectively.

Experimental. Title compound (I) prepared by the reaction of [ClPh₂PNPPh₂Cl]⁺.Cl⁻ with Me₃-