

# ORGANOMETALLICS

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Crystal data for : C<sub>20</sub>H<sub>24</sub>P<sub>2</sub>S<sub>2</sub>

Crystal data are presented in Table 1. Data collection : for this compound was collected on a STOE IPDS diffractometer, the scan type used is  $\omega/2\theta$  a post-refinement of cell constants were performed by using a setting of 2000 reflections in the  $2\theta$  range  $2.9^\circ < \theta < 48.4^\circ$

Corrections for Lorentz and polarisation effects were applied on the data. Structure solution : The structure was solved by Direct Methods using the package SIR92 (1). Structure Refinement : Refinement was performed on F using the Crystals program adapted on a PC (2). Anisotropic thermal parameters were assigned to C, P, S atoms, while H atoms were included in the process of refinement as the fixed contributors with (C-H=0.96Å) and U[iso] fixed 20% higher than those of the carbons to which they were connected.

Supplementary material available : Final atomic coordinates are given in Table S2, bond lengths and angles in Table S3, fractional atomic coordinates and U[iso] for H atoms Table S4, anisotropic thermal parameters Table S5 with Structures factors tables Table S6.

Further details of the Crystal Structure Investigation are available on request from the Director of the Cambridge Crystallographic Data Centre, 12 Union Road, GB-Cambridge CB21EZ (UK), on quoting the full journal citation.

References

1 Altomare, A., Cascarano, G., Giacovazzo, G., Guagliardi, A., Burla, M. C., Polidori, G., Camalli, M. (1994) SIR92 - a program for automatic solution of crystal structures by direct methods. J. Appl. Cryst. (27), 435.

2 Watkin, D. J., Prout, C. K., Carruthers, R. J., Betteridge, P. (1996) CRYSTALS Issue 10. Chemical Crystallography Laboratory, OXFORD, UK.

**Crystal data**

Chemical formula	$C_{20}H_{24}P_2S_2$
Molecular weight	390.5
$\rho_{\text{calcd}}$ ( $\text{g}\cdot\text{cm}^{-3}$ )	1.27
$\mu$ ( $\text{cm}^{-1}$ )	4.04
Crystal System	Monoclinic
Space group	$P 2_1/c$
a ( $\text{\AA}$ )	11.371(2)
b ( $\text{\AA}$ )	10.663(2)
c ( $\text{\AA}$ )	16.842(4)
$\beta$ ( $^\circ$ )	90.15(2)
V ( $\text{\AA}^3$ )	2042.1
Z	4
No of used reflections for cell post-refinement	2000

**Data collection**

Radiation type	MoK $\alpha$
Wavelength ( $\text{\AA}$ )	0.71073
Tube power (kW)	1.50
Collimator size (mm)	0.5
Temperature (K)	293
Detector distance (mm)	80.0
2 $\theta$ range ( $^\circ$ )	2.9 - 48.4
Phi movement mode	Rotation
Phi start ( $^\circ$ )	0.0
Phi end ( $^\circ$ )	250.0
Phi incr ( $^\circ$ )	2.0
No of exposures	125
Measurement duration (h)	13
Irradiation/exposures (min)	2.00
No of measured reflections	15975
No of merged reflections	15497
Merging R value	0.040
No of syst.absent reflections	478
No of independent reflections	3211
No of observed reflections	2412
Observation criterion	$[I > 3\sigma(I)]$

## Refinement

Refinement on	F
Hydrogen atoms	calculated and not refined
R <sup>a</sup>	0.047
Rw <sup>b</sup>	0.048
G.O.F (S) <sup>c</sup>	1.00
Weighting scheme	1
No of reflections used	2412
No of parameters used	218

$$^a. R = \frac{\sum (||F_o| - |F_c|)}{\sum |F_o|}$$

$$^b. R_w = \left[ \frac{\sum w (||F_o| - |F_c|)^2}{\sum (|F_o|)^2} \right]^{1/2}$$

$$^c. \text{Goodness of Fit} = \frac{\sum (|F_o - F_c|)^2}{(N_{\text{obs}} - N_{\text{parameters}})^{1/2}}$$

Table S2 : Fractional atomic coordinates and Equivalent thermal parameters.

Estimated standard deviations are in parentheses.

Atom	x/a	y/b	z/c	*U <sub>eq</sub> , .Å <sup>2</sup>
P(1)	0.25981(9)	0.1112(1)	0.61124(5)	0.0504
P(2)	0.26138(8)	0.13391(9)	0.81001(5)	0.0462
S(1)	0.1120(1)	0.1951(1)	0.58776(7)	0.0739
S(2)	0.3731(1)	0.0602(1)	0.88527(7)	0.0699
C(1)	0.3853(4)	0.1833(4)	0.5632(2)	0.0653
C(2)	0.4075(5)	0.2976(5)	0.6151(3)	0.0786
C(3)	0.3958(3)	0.2576(4)	0.7035(2)	0.0574
C(4)	0.3339(3)	0.3518(4)	0.7555(2)	0.0517
C(5)	0.3507(4)	0.4807(4)	0.7509(3)	0.0654
C(6)	0.2919(4)	0.5583(4)	0.8024(3)	0.0753
C(7)	0.2174(5)	0.5102(4)	0.8590(3)	0.0748
C(8)	0.2007(4)	0.3820(4)	0.8652(2)	0.0640
C(9)	0.2589(3)	0.3027(3)	0.8131(2)	0.0491
C(10)	0.3287(3)	0.1301(3)	0.7100(2)	0.0466
C(11)	0.2609(3)	-0.0537(4)	0.5885(2)	0.0469
C(12)	0.3616(4)	-0.1245(4)	0.5978(2)	0.0651
C(13)	0.3625(5)	-0.2506(5)	0.5771(3)	0.0849
C(14)	0.2640(6)	-0.3047(5)	0.5465(3)	0.0908
C(15)	0.1643(5)	-0.2374(5)	0.5364(3)	0.0779
C(16)	0.1610(3)	-0.1117(4)	0.5572(2)	0.0619
C(20)	0.1134(3)	0.0639(4)	0.8210(2)	0.0549
C(21)	0.0170(4)	0.1482(6)	0.7861(3)	0.0800
C(22)	0.0887(4)	0.0479(4)	0.9097(2)	0.0629
C(23)	0.1135(6)	-0.0626(6)	0.7817(4)	0.0950

$$*U_{eq} = [U_{11} * U_{22} * U_{33}]^{1/3}$$

Table S3 : Interatomic distances (Å) and bond angles (°) e.s.d.'s in parentheses refers to the last significant digit .

P(1) - S(1)	1.944(2)	C(5) - C(6)	1.373(6)
P(1) - C(1)	1.813(4)	C(6) - C(7)	1.376(6)
P(1) - C(10)	1.848(3)	C(7) - C(8)	1.384(6)
P(1) - C(11)	1.800(4)	C(8) - C(9)	1.389(5)
P(2) - S(2)	1.957(1)	C(11) - C(12)	1.380(5)
P(2) - C(9)	1.801(4)	C(11) - C(16)	1.395(5)
P(2) - C(10)	1.852(3)	C(12) - C(13)	1.389(6)
P(2) - C(20)	1.850(4)	C(13) - C(14)	1.360(7)
C(1) - C(2)	1.520(6)	C(14) - C(15)	1.352(7)
C(2) - C(3)	1.554(6)	C(15) - C(16)	1.386(6)
C(3) - C(4)	1.509(5)	C(20) - C(21)	1.533(6)
C(3) - C(10)	1.563(5)	C(20) - C(22)	1.530(5)
C(4) - C(5)	1.390(5)	C(20) - C(23)	1.503(6)
C(4) - C(9)	1.394(5)		
S(1) - P(1) - C(1)	113.3(2)	C(6) - C(7) - C(8)	120.4(4)
S(1) - P(1) - C(10)	119.8(1)	C(7) - C(8) - C(9)	119.2(4)
C(1) - P(1) - C(10)	91.3(2)	P(2) - C(9) - C(4)	110.2(3)
S(1) - P(1) - C(11)	114.4(1)	P(2) - C(9) - C(8)	129.4(3)
C(1) - P(1) - C(11)	108.3(2)	C(4) - C(9) - C(8)	120.3(4)
C(10) - P(1) - C(11)	107.2(2)	P(1) - C(10) - P(2)	130.2(2)
S(2) - P(2) - C(9)	113.2(1)	P(1) - C(10) - C(3)	103.7(2)
S(2) - P(2) - C(10)	108.1(1)	P(2) - C(10) - C(3)	104.4(2)
C(9) - P(2) - C(10)	93.1(2)	P(1) - C(11) - C(12)	121.1(3)
S(2) - P(2) - C(20)	111.2(1)	P(1) - C(11) - C(16)	120.5(3)
C(9) - P(2) - C(20)	112.7(2)	C(12) - C(11) - C(16)	118.4(4)
C(10) - P(2) - C(20)	117.4(2)	C(11) - C(12) - C(13)	120.5(4)
P(1) - C(1) - C(2)	102.3(3)	C(12) - C(13) - C(14)	119.9(5)
C(1) - C(2) - C(3)	108.4(4)	C(13) - C(14) - C(15)	120.7(5)
C(2) - C(3) - C(4)	114.5(4)	C(14) - C(15) - C(16)	120.4(4)
C(2) - C(3) - C(10)	110.4(3)	C(11) - C(16) - C(15)	120.1(4)
C(4) - C(3) - C(10)	108.0(3)	P(2) - C(20) - C(21)	112.0(3)
C(3) - C(4) - C(5)	124.1(4)	P(2) - C(20) - C(22)	108.2(3)
C(3) - C(4) - C(9)	116.2(3)	C(21) - C(20) - C(22)	107.8(3)
C(5) - C(4) - C(9)	119.7(4)	P(2) - C(20) - C(23)	108.5(3)
C(4) - C(5) - C(6)	119.5(4)	C(21) - C(20) - C(23)	111.0(4)
C(5) - C(6) - C(7)	121.0(4)	C(22) - C(20) - C(23)	109.3(4)

Table S4 : Fractional atomic coordinates and Isotropic thermal parameters for H atoms. Estimated standard deviations are in parentheses.

Atom	x/a	y/b	z/c	U[iso], .Å <sup>2</sup>
H(11)	0.4519	0.1278	0.5630	0.0854
H(12)	0.3669	0.2071	0.5095	0.0854
H(21)	0.4854	0.3290	0.6056	0.1080
H(22)	0.3510	0.3615	0.6029	0.1080
H(31)	0.4746	0.2536	0.7240	0.0723
H(51)	0.4030	0.5155	0.7118	0.0800
H(61)	0.3028	0.6476	0.7989	0.0906
H(71)	0.1767	0.5658	0.8942	0.0939
H(81)	0.1495	0.3485	0.9053	0.0778
H(101)	0.3748	0.0548	0.7137	0.0560
H(121)	0.4316	-0.0864	0.6189	0.0796
H(131)	0.4325	-0.2996	0.5847	0.1042
H(141)	0.2656	-0.3914	0.5315	0.1108
H(151)	0.0956	-0.2775	0.5151	0.0985
H(161)	0.0898	-0.0645	0.5497	0.0745
H(211)	-0.0582	0.1088	0.7929	0.0987
H(212)	0.0174	0.2276	0.8131	0.0987
H(213)	0.0314	0.1611	0.7306	0.0987
H(221)	0.0121	0.0118	0.9167	0.0775
H(222)	0.0913	0.1286	0.9352	0.0775
H(223)	0.1470	-0.0059	0.9329	0.0775
H(231)	0.0364	-0.0992	0.7867	0.1472
H(232)	0.1706	-0.1149	0.8060	0.1472
H(233)	0.1311	-0.0521	0.7262	0.1472

Table S5 : Anisotropic thermal parameters (with e.s.d.'s in parentheses).

They enter the expression for the structure factor in the form :

$$\exp[-2p^2(U_{11}h^2a^2 + U_{22}k^2b^2 + U_{33}l^2c^2 + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$$

Atom	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
P(1)	0.0585(6)	0.0612(6)	0.0372(5)	0.0026(5)	0.0090(4)	0.0013(5)
P(2)	0.0514(5)	0.0531(6)	0.0362(5)	-0.0005(4)	0.0026(4)	-0.0011(5)
S(1)	0.0942(9)	0.110(1)	-0.0511(6)	0.0189(7)	0.0083(6)	0.0435(8)
S(2)	0.0678(7)	0.0956(9)	0.0583(6)	0.0116(6)	-0.0048(5)	0.0197(7)
C(1)	0.0910(3)	0.076(3)	0.053(2)	-0.003(2)	0.030(2)	-0.021(3)
C(2)	0.139(5)	0.081(3)	0.068(3)	-0.010(3)	0.048(3)	-0.041(3)
C(3)	0.061(2)	0.064(3)	0.058(2)	-0.012(2)	0.017(2)	-0.015(2)
C(4)	0.054(2)	0.058(2)	0.047(2)	-0.006(2)	0.001(2)	-0.010(2)
C(5)	0.073(3)	0.061(3)	0.066(3)	-0.005(2)	0.008(2)	-0.015(2)
C(6)	0.102(4)	0.054(3)	0.079(3)	-0.007(2)	0.005(3)	-0.010(3)
C(7)	0.106(4)	0.064(3)	0.070(3)	-0.016(2)	0.016(3)	0.012(3)
C(8)	0.088(3)	0.065(3)	0.048(2)	-0.005(2)	0.013(2)	0.003(2)
C(9)	0.054(2)	0.053(2)	0.042(2)	-0.002(2)	-0.002(2)	-0.003(2)
C(10)	0.054(2)	0.050(2)	0.040(2)	-0.004(2)	0.008(2)	-0.003(2)
C(11)	0.050(2)	0.063(2)	0.034(2)	-0.005(2)	0.003(2)	-0.006(2)
C(12)	0.064(3)	0.071(3)	0.065(3)	-0.015(2)	-0.003(2)	0.005(2)
C(13)	0.098(4)	0.082(4)	0.083(3)	-0.013(3)	-0.001(3)	0.022(3)
C(14)	0.135(5)	0.073(4)	0.080(4)	-0.014(3)	-0.002(3)	-0.013(3)
C(15)	0.104(4)	0.087(4)	0.064(3)	-0.007(3)	-0.005(3)	-0.038(3)
C(16)	0.058(2)	0.087(3)	0.048(2)	0.005(2)	0.001(2)	-0.009(2)
C(20)	0.062(2)	0.069(3)	0.042(2)	-0.002(2)	0.010(2)	-0.016(2)
C(21)	0.059(3)	0.169(5)	0.059(3)	0.032(3)	-0.005(2)	-0.014(3)
C(22)	0.070(3)	0.080(3)	0.049(2)	0.012(2)	0.014(2)	0.000(2)
C(23)	0.157(5)	0.111(4)	0.115(4)	-0.051(4)	0.075(4)	-0.078(4)