Crystal structure report for AQ206a

X-ray crystallographic study
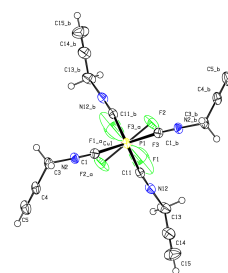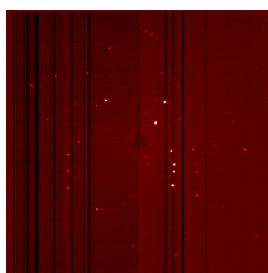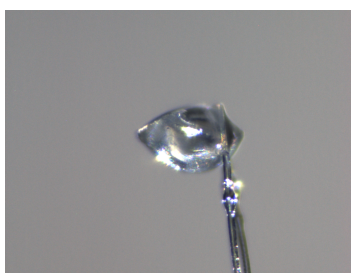
$(C_{16}H_{12}CuN_4 \cdot F_6P)$; $M = 468.81$. A suitable crystal for X-ray diffraction single crystal experiment (colourless prism, dimensions = 0.430 x 0.260 x 0.200 mm) was selected and mounted with a cryoloop on the goniometer head of a D8 Venture diffractometer diffractometer equipped with a (CMOS) PHOTON 100 detector [*], using Mo-Kα radiation ($\lambda = 0.71073$ Å, multilayer monochromator) at $T = 150(2)$ K. Crystal structure has been described in monoclinic symmetry and $C\ 2/c$ (I.T.#15) centric space group. Cell parameters have been refined as follows: $a = 5.4651(6)$, $b = 22.311(2)$, $c = 16.4771(18)$ Å, $\beta = 94.038(4)$ °, $V = 2004.1(4)$ Å^3. Number of formula unit Z is equal to 4 and calculated density d and absorption coefficient μ values are 1.554 g.cm^{-3} and 1.231 mm^{-1} respectively. The structure was solved by dual-space algorithm using the *SHELXT* program [1], and then refined with full-matrix least-squares methods based on F^2 (*SHELXL* program [2]). All non-Hydrogen atoms were refined with anisotropic atomic displacement parameters. H atoms were finally included in their calculated positions and treated as riding on their parent atom with constrained thermal parameters. A final refinement on F^2 with 2290 unique intensities and 128 parameters converged at $\omega R(F^2) = 0.1353$ ($R_F = 0.0513$) for 2070 observed reflections with ($I > 2\sigma$).

1. G. M. Sheldrick, *Acta Cryst.*, **2015**, *A71*, 3-8
2. Sheldrick G.M., *Acta Cryst.*, **2015**, *C71*, 3-8

```
Software                 : BIS V6.2.1/2016-03-01
Number of scans          :        2
Total number of frames [*] :      360
Total length of scans  [*] :   360.00 (deg.)
Rotation speed         [*] :     5.00 sec./deg.
Total exposition time  [*] :        30.0 min.

 [*] fast scan not included

Measurement mode         : shutterless mode

   Scan  Time(s)  Width  DX (mm)  Frames  Theta  Omega    Phi    Chi   T(K)
..............................................................................

  1 Phi      5.0   1.00     34.0     360   0.27 270.94 360.00  23.00 150.00
  2 Fast     1.0   1.00     34.0     180   0.00 360.00   0.00  54.74 150.00
```

... Crystal data ...

Empirical formula	$C_{16}H_{12}CuF_6N_4P$
Extended formula	$C_{16}H_{12}CuN_4, F_6P$
Formula weight	468.81 g/mol
Temperature	150(2) K
Radiation type	Mo-Kα
Wavelength	0.71073 Å
Crystal system, space group	monoclinic, C $2/c$
Unit cell dimensions	a = 5.4651(6) Å
	b = 22.311(2) Å
	c = 16.4771(18) Å
	β = 94.038(4) ˚
Volume	2004.1(4) Å^3
Z, Calculated density	4, 1.554 $g.cm^{-3}$
Absorption coefficient	1.231 mm^{-1}
F(000)	936
Crystal size	0.430 x 0.260 x 0.200 mm
Crystal color	colourless
Crystal description	prism

... Data collection ...

Diffractometer	D8 Venture diffractometer
θ range for data collection	2.206 to 27.502 ˚
$(sin\theta/\lambda)_{max}$ (Å^{-1})	0.650
h$_{min}$, h$_{max}$	-7, 7
k$_{min}$, k$_{max}$	-22, 28
l$_{min}$, l$_{max}$	-21, 21
Reflections collected / unique	13996 / 2290 [^{a}R(int) = 0.0446]
Reflections [$I > 2\sigma$]	2070
Completeness to θ_{max}	0.997
Absorption correction type	multi-scan
Max. and min. transmission	0.782 , 0.682

... Refinement ...

Refinement method	Full-matrix least-squares on F^2
H-atom treatment	H-atom parameters constrained
Data / restraints / parameters	2290 / 0 / 128
^{b}Goodness-of-fit	1.049
Final R indices [$I > 2\sigma$]	$^cR_1$ = 0.0513, $^dwR_2$ = 0.1353
R indices (all data)	$^cR_1$ = 0.0567, $^dwR_2$ = 0.1401
$\Delta\rho_{max}$, $\Delta\rho_{min}$	1.306, -1.165 $\overline{e}$.Å^{-3}

$$^a R_{int} = \frac{\sum |F_o^2 - \langle F_o^2 \rangle|}{\sum F_o^2}$$

$$^b S = \left\{ \frac{\sum [w(F_o^2 - F_c^2)^2]}{n-p} \right\}^{1/2}$$

$$^c R_1 = \frac{\sum |F_o| - |F_c|}{\sum |F_o|}$$

$$^d wR_2 = \left\{ \frac{\sum [w(F_o^2 - F_c^2)^2]}{\sum [w(F_o^2)^2]} \right\}^{1/2}$$

$w = 1./[\sigma(F_o^2) + (aP)^2 + bP]$ with $P = [2F_c^2 + Max(F_o^2, 0)]/3$

Fractional atomic coordinates, site occupancy (%) and equivalent isotropic displacement parameters (Å^2).

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	occ.	U(eq)	adp
Cu1	0.500000	0.62943(2)	0.750000	1	0.02128(19)	Uani
C1	0.7056(6)	0.67677(14)	0.68164(19)	1	0.0243(6)	Uani
N2	0.8439(5)	0.70031(12)	0.64378(16)	1	0.0253(6)	Uani
C3	1.0263(6)	0.72829(16)	0.5968(2)	1	0.0294(7)	Uani
H3A	1.191170	0.721924	0.624272	1	0.035	Uiso
H3B	0.995732	0.772000	0.593700	1	0.035	Uiso
C4	1.0193(6)	0.70330(15)	0.5141(2)	1	0.0290(7)	Uani
C5	1.0138(7)	0.68312(18)	0.4482(2)	1	0.0381(8)	Uani
H5	1.009273	0.666788	0.394880	1	0.046	Uiso
C11	0.2656(6)	0.57779(14)	0.68743(19)	1	0.0244(6)	Uani
N12	0.1156(5)	0.54764(12)	0.65762(16)	1	0.0266(6)	Uani
C13	-0.0777(8)	0.50961(17)	0.6223(2)	1	0.0366(8)	Uani
H13A	-0.184217	0.496573	0.665135	1	0.044	Uiso
H13B	-0.005203	0.473421	0.598799	1	0.044	Uiso
C14	-0.2244(7)	0.54166(17)	0.5588(2)	1	0.0381(9)	Uani
C15	-0.3454(9)	0.5668(2)	0.5080(3)	1	0.0554(12)	Uani
H15	-0.443422	0.587166	0.466804	1	0.066	Uiso
P1	0.500000	0.37997(5)	0.750000	1	0.0241(3)	Uani
F1	0.3187(9)	0.3292(3)	0.7247(3)	1	0.144(2)	Uani
F2	0.3932(6)	0.38028(13)	0.83765(15)	1	0.0573(8)	Uani
F3	0.3155(13)	0.4276(3)	0.7231(3)	1	0.195(4)	Uani

5

The anisotropic displacement factor exponent takes the form: $-2\pi[h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12}]$

Atom	U11	U22	U33	U23	U13	U12
Cu1	0.0191(3)	0.0239(3)	0.0216(3)	0.000	0.00708(19)	0.000
C1	0.0225(14)	0.0253(15)	0.0253(15)	-0.0003(12)	0.0032(12)	0.0011(12)
N2	0.0238(13)	0.0280(13)	0.0249(13)	0.0039(11)	0.0069(11)	-0.0027(10)
C3	0.0276(16)	0.0340(17)	0.0277(16)	0.0028(13)	0.0101(13)	-0.0105(13)
C4	0.0263(16)	0.0293(16)	0.0328(18)	0.0027(13)	0.0119(13)	-0.0065(12)
C5	0.039(2)	0.0374(19)	0.040(2)	-0.0049(16)	0.0163(16)	-0.0114(16)
C11	0.0259(15)	0.0260(15)	0.0220(14)	0.0001(12)	0.0073(12)	0.0021(12)
N12	0.0315(14)	0.0265(13)	0.0223(12)	-0.0010(10)	0.0062(11)	-0.0035(11)
C13	0.050(2)	0.0316(18)	0.0280(17)	-0.0034(14)	0.0014(15)	-0.0185(16)
C14	0.044(2)	0.0382(19)	0.0319(18)	-0.0049(15)	0.0028(16)	-0.0224(16)
C15	0.056(3)	0.057(3)	0.050(3)	0.007(2)	-0.017(2)	-0.025(2)
P1	0.0341(6)	0.0224(5)	0.0158(5)	0.000	0.0015(4)	0.000
F1	0.127(4)	0.197(5)	0.113(4)	-0.099(4)	0.049(3)	-0.106(4)
F2	0.083(2)	0.0651(17)	0.0259(12)	-0.0040(11)	0.0219(12)	-0.0227(14)
F3	0.279(7)	0.216(5)	0.101(3)	0.096(4)	0.105(4)	0.217(5)

```
Cu1   - C1_#1     = 1.956(3)
Cu1   - C1        = 1.956(3)
Cu1   - C11       = 1.961(3)
Cu1   - C11_#1    = 1.961(3)
C1    - N2        = 1.141(4)
N2    - C3        = 1.446(4)
C3    - C4        = 1.470(5)
C3    - H3A       = 0.9900
C3    - H3B       = 0.9900
C4    - C5        = 1.174(5)
C5    - H5        = 0.9500
C11   - N12       = 1.144(4)
N12   - C13       = 1.446(4)
C13   - C14       = 1.459(6)
C13   - H13A      = 0.9900
C13   - H13B      = 0.9900
C14   - C15       = 1.173(6)
C15   - H15       = 0.9500
P1    - F3        = 1.510(4)
P1    - F3_#1     = 1.510(4)
P1    - F1_#1     = 1.542(4)
P1    - F1        = 1.542(4)
P1    - F2        = 1.595(2)
P1    - F2_#1     = 1.595(2)
```

Symmetry transformations used to generate equivalent atoms:

```
#1    -x, y, -z+1/2          T = [ 1,  0,  1]
```
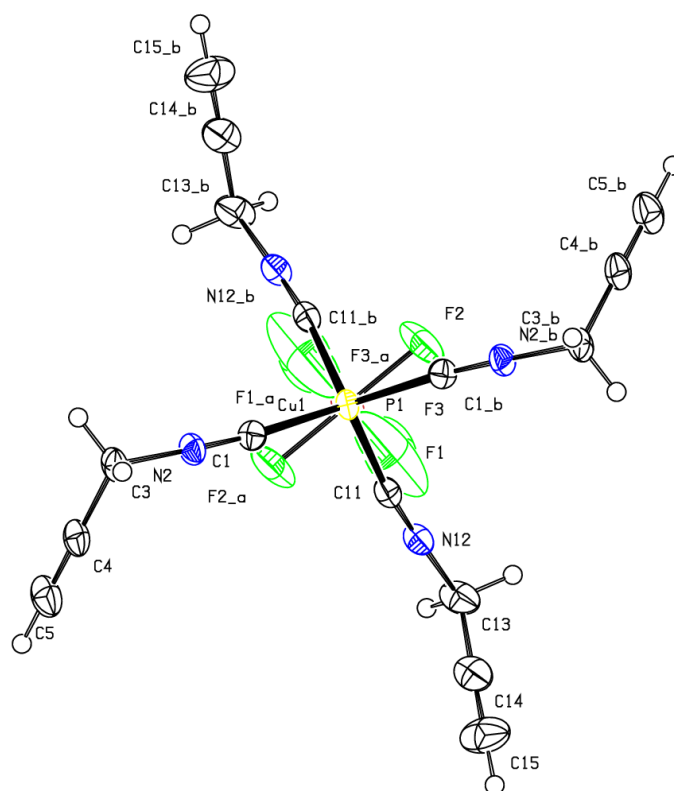
Angles [°]

C1_#1	– Cu1	– C1	=	114.62(19)
C1_#1	– Cu1	– C11	=	103.86(13)
C1	– Cu1	– C11	=	113.27(13)
C1_#1	– Cu1	– C11_#1	=	113.27(13)
C1	– Cu1	– C11_#1	=	103.86(13)
C11	– Cu1	– C11_#1	=	108.04(19)
N2	– C1	– Cu1	=	173.1(3)
C1	– N2	– C3	=	177.7(3)
N2	– C3	– C4	=	111.0(3)
N2	– C3	– H3A	=	109.4
C4	– C3	– H3A	=	109.4
N2	– C3	– H3B	=	109.4
C4	– C3	– H3B	=	109.4
H3A	– C3	– H3B	=	108.0
C5	– C4	– C3	=	179.7(4)
C4	– C5	– H5	=	180.0
N12	– C11	– Cu1	=	173.2(3)
C11	– N12	– C13	=	178.2(3)
N12	– C13	– C14	=	110.5(3)
N12	– C13	– H13A	=	109.6
C14	– C13	– H13A	=	109.6
N12	– C13	– H13B	=	109.6
C14	– C13	– H13B	=	109.6
H13A	– C13	– H13B	=	108.1
C15	– C14	– C13	=	178.9(4)
C14	– C15	– H15	=	180.0
F3	– P1	– F3_#1	=	90.5(7)
F3	– P1	– F1_#1	=	177.4(4)
F3_#1	– P1	– F1_#1	=	92.0(4)
F3	– P1	– F1	=	92.0(4)
F3_#1	– P1	– F1	=	177.4(4)
F1_#1	– P1	– F1	=	85.6(5)
F3	– P1	– F2	=	89.0(2)
F3_#1	– P1	– F2	=	90.63(19)
F1_#1	– P1	– F2	=	91.6(2)
F1	– P1	– F2	=	88.77(19)
F3	– P1	– F2_#1	=	90.63(19)
F3_#1	– P1	– F2_#1	=	89.0(2)
F1_#1	– P1	– F2_#1	=	88.76(19)
F1	– P1	– F2_#1	=	91.6(2)
F2	– P1	– F2_#1	=	179.5(2)

Symmetry transformations used to generate equivalent atoms:

#1   -x, y, -z+1/2   T = [1,   0,   1]