

Electronic supplementary materials *Mendeleev Commun.*, 2012, **22**, 294–296

Synthesis and structural characterization of the first europium(III) pyridylphosphine complex, [Eu(*N,N',N''*-2-Py₃P)(NO₃)₃]

Alexander V. Artem'ev, Nina K. Gusarova, Svetlana F. Malysheva, Olga N. Kazheva, Grigorii G. Alexandrov, Oleg A. Dyachenko and Boris A. Trofimov*

General remarks

Tris(2-pyridyl)phosphine (**1**) was synthesized directly from 2-bromopyridine and red phosphorus according to the recently developed one-pot microwave assistant procedure.¹ Eu(NO₃)₃·6H₂O was used as received. Commercial ethanol (5% water content) was used as a solvent. Diethyl ether was distilled over metal sodium before use. The microanalysis was performed on a Flash EA 1112 Series elemental analyzer. Melting point (uncorrected) was determined in a Kofler micro hot stage apparatus. Fourier transform IR spectrum was run on a Bruker Vertex 70 instrument. UV-VIS electronic spectrum was measured on a Lambda 35 spectrophotometer. The ¹H and ³¹P NMR spectra were recorded on a Bruker AV-400 spectrometer (400.13 and 161.98 MHz, respectively) and referenced to H₃PO₄ (³¹P NMR) as external standard.

X-ray quality crystals of composition [Eu(*N,N',N''*-2-Py₃P)(NO₃)₃]·CH₂Cl₂ were obtained by slow evaporation of solution of complex **2** in a 70:30 ethanol/dichloromethane mixture at room temperature over two weeks. X-ray diffraction analysis of the crystals was performed at 173 K on Bruker SMART APEX-II CCD diffractometer [Mo-K_α radiation, λ(Mo-K_α) = 0.71073 Å, ω-scans]. The structure was solved by direct methods and subsequent Fourier syntheses, and refined by full-matrix least-squares procedures using an anisotropic approximation for all non-H atoms with SHELXTL PLUS 5 program.² The hydrogen atoms were fixed in positions of ideal geometry. An absorption correction was applied using APEX2 program.³

CCDC 869847 contains the supplementary crystallographic data for [Eu(2-Py₃P)(NO₃)₃]·CH₂Cl₂ complex. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, by e-mailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44(0)1223-336033.

References

1. B. A. Trofimov, N. K. Gusarova, A. V. Artem'ev, S. F. Malysheva, N. A. Belogorlova, A. O. Korocheva, O. N. Kazheva, G. G. Alexandrov and O. A. Dyachenko, *Mendeleev Commun.*, 2012, **22**, 187.
2. G. M. Sheldrick, *SHELXTL v. 5.10, Structure Determination Software Suite*, Bruker AXS, Madison, Wisconsin, USA, 1998.
3. *APEX2 Software Package*, Bruker AXS Inc., 5465, East Cheryl Parkway, Madison, Wisconsin, USA, 2005.

Table S1 Crystal data and structure refinement for the complex **2**·CH₂Cl₂

Empirical formula	C ₁₅ H ₁₂ EuN ₆ O ₉ P·CH ₂ Cl ₂
Formula weight	688.16
Temperature (K)	173
Crystal system	Monoclinic
Space group	<i>Cc</i>
Crystal size (mm)	0.05 × 0.11 × 0.25
Unit cell dimensions (Å, °)	
<i>a</i>	10.2858(9)
<i>b</i>	17.856(2)
<i>c</i>	12.829(1)
β	101.214(1)
Volume (Å ³), <i>Z</i>	2311.3(3), 4
Calculated density (g·cm ⁻³)	1.98
Absorption coefficient (mm ⁻¹)	3.075
<i>F</i> (000)	1344
θ range for data collection (°)	2.32 ÷ 27.41
Reflections collected/unique	8911/4969
Data/restraints/parameters	4969/2/316
Reflections [<i>I</i> > 2 σ (<i>I</i>)]	4694
Goodness-of-fit on <i>F</i> ²	0.959
Final <i>R</i> indices [<i>I</i> > 2 σ (<i>I</i>)]	<i>R</i> ₁ = 0.0257; <i>wR</i> ₂ = 0.0567
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0281; <i>wR</i> ₂ = 0.0579

Table S2 Bond lengths (Å) and angles (°) for the complex **2**·CH₂Cl₂

Eu(1)-O(6)	2.452(3)
Eu(1)-O(7)	2.465(3)
Eu(1)-O(3)	2.468(4)
Eu(1)-O(9)	2.464(3)
Eu(1)-O(1)	2.466(3)
Eu(1)-O(4)	2.479(3)
Eu(1)-N(5)	2.514(4)
Eu(1)-N(4)	2.540(4)
Eu(1)-N(6)	2.564(4)
Eu(1)-N(1)	2.889(4)
Eu(1)-N(3)	2.893(4)
Eu(1)-N(2)	2.897(4)
P(1)-C(6)	1.840(4)

P(1)-C(11)	1.845(4)
P(1)-C(5)	1.844(4)
O(1)-N(1)	1.278(5)
O(2)-N(1)	1.220(5)
O(3)-N(1)	1.273(6)
O(4)-N(2)	1.276(5)
O(5)-N(2)	1.211(5)
O(6)-N(2)	1.262(5)
O(7)-N(3)	1.270(5)
O(8)-N(3)	1.203(5)
O(9)-N(3)	1.277(5)
N(4)-C(5)	1.348(6)
N(4)-C(1)	1.354(6)
N(5)-C(10)	1.348(5)
N(5)-C(6)	1.351(5)
N(6)-C(15)	1.339(6)
N(6)-C(11)	1.353(5)
C(1)-C(2)	1.380(7)
C(2)-C(3)	1.372(7)
C(3)-C(4)	1.385(6)
C(4)-C(5)	1.377(6)
C(6)-C(7)	1.379(6)
C(7)-C(8)	1.389(6)
C(8)-C(9)	1.376(7)
C(9)-C(10)	1.372(6)
C(11)-C(12)	1.399(6)
C(12)-C(13)	1.387(7)
C(13)-C(14)	1.370(8)
C(14)-C(15)	1.386(7)
C(16)-Cl(1)	1.763(6)
C(16)-Cl(2)	1.762(6)
O(6)-Eu(1)-O(7)	75.08(12)
O(6)-Eu(1)-O(3)	120.52(12)
O(7)-Eu(1)-O(3)	76.42(12)
O(6)-Eu(1)-O(9)	121.27(11)
O(7)-Eu(1)-O(9)	51.82(11)
O(3)-Eu(1)-O(9)	73.51(12)
O(6)-Eu(1)-O(1)	119.16(12)
O(7)-Eu(1)-O(1)	127.20(11)
O(3)-Eu(1)-O(1)	51.96(11)
O(9)-Eu(1)-O(1)	112.79(11)
O(6)-Eu(1)-O(4)	51.59(11)
O(7)-Eu(1)-O(4)	85.29(13)
O(3)-Eu(1)-O(4)	75.09(13)
O(9)-Eu(1)-O(4)	131.39(11)
O(1)-Eu(1)-O(4)	72.70(12)
O(6)-Eu(1)-N(5)	144.40(11)
O(7)-Eu(1)-N(5)	122.80(14)
O(3)-Eu(1)-N(5)	94.63(13)
O(9)-Eu(1)-N(5)	71.29(12)
O(1)-Eu(1)-N(5)	76.75(11)

O(4)-Eu(1)-N(5)	147.52(12)
O(6)-Eu(1)-N(4)	71.84(12)
O(7)-Eu(1)-N(4)	87.93(13)
O(3)-Eu(1)-N(4)	155.52(16)
O(9)-Eu(1)-N(4)	82.02(12)
O(1)-Eu(1)-N(4)	144.24(12)
O(4)-Eu(1)-N(4)	122.85(13)
N(5)-Eu(1)-N(4)	77.99(12)
O(6)-Eu(1)-N(6)	79.74(13)
O(7)-Eu(1)-N(6)	153.49(12)
O(3)-Eu(1)-N(6)	124.73(13)
O(9)-Eu(1)-N(6)	143.03(11)
O(1)-Eu(1)-N(6)	72.97(11)
O(4)-Eu(1)-N(6)	85.57(11)
N(5)-Eu(1)-N(6)	75.08(12)
N(4)-Eu(1)-N(6)	76.35(13)
O(6)-Eu(1)-N(1)	121.50(11)
O(7)-Eu(1)-N(1)	101.39(12)
O(3)-Eu(1)-N(1)	26.02(12)
O(9)-Eu(1)-N(1)	94.62(11)
O(1)-Eu(1)-N(1)	26.11(11)
O(4)-Eu(1)-N(1)	69.92(11)
N(5)-Eu(1)-N(1)	87.25(11)
N(4)-Eu(1)-N(1)	165.19(11)
N(6)-Eu(1)-N(1)	98.72(12)
O(6)-Eu(1)-N(3)	99.07(12)
O(7)-Eu(1)-N(3)	25.89(12)
O(3)-Eu(1)-N(3)	71.46(12)
O(9)-Eu(1)-N(3)	26.05(11)
O(1)-Eu(1)-N(3)	121.78(11)
O(4)-Eu(1)-N(3)	107.91(12)
N(5)-Eu(1)-N(3)	97.32(12)
N(4)-Eu(1)-N(3)	86.20(13)
N(6)-Eu(1)-N(3)	162.02(12)
N(1)-Eu(1)-N(3)	97.13(12)
O(6)-Eu(1)-N(2)	25.60(11)
O(7)-Eu(1)-N(2)	79.05(12)
O(3)-Eu(1)-N(2)	98.15(13)
O(9)-Eu(1)-N(2)	130.87(11)
O(1)-Eu(1)-N(2)	96.24(11)
O(4)-Eu(1)-N(2)	25.99(11)
N(5)-Eu(1)-N(2)	156.94(12)
N(4)-Eu(1)-N(2)	97.18(12)
N(6)-Eu(1)-N(2)	81.86(11)
N(1)-Eu(1)-N(2)	95.90(11)
N(3)-Eu(1)-N(2)	104.90(11)
C(6)-P(1)-C(11)	99.42(19)
C(6)-P(1)-C(5)	104.21(18)
C(11)-P(1)-C(5)	102.03(19)
N(1)-O(1)-Eu(1)	95.7(2)
N(1)-O(3)-Eu(1)	95.8(3)
N(2)-O(4)-Eu(1)	95.6(2)

N(2)-O(6)-Eu(1)	97.3(2)
N(3)-O(7)-Eu(1)	96.2(2)
N(3)-O(9)-Eu(1)	96.1(3)
O(2)-N(1)-O(3)	122.3(4)
O(2)-N(1)-O(1)	121.8(4)
O(3)-N(1)-O(1)	115.9(4)
O(2)-N(1)-Eu(1)	173.1(3)
O(3)-N(1)-Eu(1)	58.2(2)
O(1)-N(1)-Eu(1)	58.2(2)
O(5)-N(2)-O(6)	121.8(4)
O(5)-N(2)-O(4)	122.7(4)
O(6)-N(2)-O(4)	115.4(4)
O(5)-N(2)-Eu(1)	177.6(3)
O(6)-N(2)-Eu(1)	57.1(2)
O(4)-N(2)-Eu(1)	58.4(2)
O(8)-N(3)-O(7)	123.1(4)
O(8)-N(3)-O(9)	121.5(4)
O(7)-N(3)-O(9)	115.4(4)
O(8)-N(3)-Eu(1)	174.1(4)
O(7)-N(3)-Eu(1)	57.9(2)
O(9)-N(3)-Eu(1)	57.9(2)
C(5)-N(4)-C(1)	117.1(4)
C(5)-N(4)-Eu(1)	127.9(3)
C(1)-N(4)-Eu(1)	115.0(3)
C(10)-N(5)-C(6)	117.9(4)
C(10)-N(5)-Eu(1)	113.9(3)
C(6)-N(5)-Eu(1)	128.1(3)
C(15)-N(6)-C(11)	117.9(4)
C(15)-N(6)-Eu(1)	115.8(3)
C(11)-N(6)-Eu(1)	126.3(3)
N(4)-C(1)-C(2)	123.2(4)
C(1)-C(2)-C(3)	118.9(4)
C(4)-C(3)-C(2)	118.7(4)
C(3)-C(4)-C(5)	119.5(4)
N(4)-C(5)-C(4)	122.6(4)
N(4)-C(5)-P(1)	122.7(3)
C(4)-C(5)-P(1)	114.6(3)
N(5)-C(6)-C(7)	121.7(4)
N(5)-C(6)-P(1)	123.0(3)
C(7)-C(6)-P(1)	115.3(3)
C(8)-C(7)-C(6)	119.5(4)
C(9)-C(8)-C(7)	119.0(4)
C(8)-C(9)-C(10)	118.6(4)
N(5)-C(10)-C(9)	123.3(4)
N(6)-C(11)-C(12)	121.2(4)
N(6)-C(11)-P(1)	123.8(3)
C(12)-C(11)-P(1)	115.1(3)
C(13)-C(12)-C(11)	119.6(4)
C(12)-C(13)-C(14)	118.9(5)
C(15)-C(14)-C(13)	118.6(4)
N(6)-C(15)-C(14)	123.6(4)
Cl(1)-C(16)-Cl(2)	110.9(3)