SUPPORTING INFORMATION

Heterospin molecular complexes of $Cu(hfac)_2$ with pyridyl-substituted nitronyl nitroxides: peculiarities of structure and magnetic properties

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Experimental section

1. Synthetic procedures

1.1. Materials

The reagents bis(hexafluoroacetylacetonato)copper(II) $Cu(hfac)_2$ [1], 2-(4-R-pyridin-3-yl)-4,4,5,5-tetramethyl-4,5-dihydro-1*H*-imidazole-3-oxide-1-oxyls [2–5] were synthesized by known procedures. n-Hexane (>99%, "Component-reaktiv"), n-heptane (>99%, "Component-reaktiv"), acetone (>99.8%, "Ekos-1") was used without any additional purification. Dichloromethane which is stabilized by 0.5% of methanol has been purified and dried according to known protocol [6].

1.2. General procedures for obtaining complexes

Procedure A. A 1 ml acetone solution of $\mathbf{L^R}$ (0.2 mmol) was added in one portion to a 1 ml acetone solution of $Cu(hfac)_2$ (0.2 mmol). Equivalent volume of *n*-hexane was layered on carefully and the flask was exposed to air at 4°C. The precipitate was filtered off, washed with ice-cold n-hexane and dried in air.

Procedure B. To a solution of $Cu(hfac)_2$ (0.2 mmol) in 1 mL of CH_2Cl_2 a solution of $\mathbf{L^R}$ (0.2 mmol) in 2 mL of CH_2Cl_2 was added. A 2 ml of *n*-heptane was layered on carefully and the flask was exposed to air at 4°C. The resulting crystals were filtered off and air dried.

$[Cu(hfac)_2L^{Cl}]_2$. Procedure A. Yield 83%. Found (%): C, 35.4; H, 2.2; N, 5.4; F, 30.8; Cl, 4.9. $C_{44}H_{34}Cl_2Cu_2F_{24}N_6O_{12}$. Calculated (%): C, 35.4; H, 2.3; N, 5.6; F, 30.5; Cl, 4.8.

$[Cu(hfac)_2L^{Br}]_2$. Procedure A. Yield 85%. Found (%): C, 33.4; H, 2.1; N, 5.0; F, 29.0; Br, 10.0. $C_{44}H_{34}Br_2Cu_2F_{24}N_6O_{12}$. Calculated (%): C, 33.4; H, 2.1; N, 5.3; F, 28.8; Br, 10.1.

$[Cu(hfac)_2L^{CF3}]_2$. Procedure A. Yield 78%. Found (%): C, 35.2; H, 2.1; N, 5.2; F, 36.5. $C_{46}H_{34}Cu_2F_{30}N_6O_{12}$. Calculated (%): C, 35.4; H, 2.2; N, 5.4; F, 36.5.

$[Cu(hfac)_2L^{Ph}]_2$. Procedure A. Yield 27%. Found (%): C, 42.6; H, 2.8; N, 5.0; F, 29.0. $C_{56}H_{44}Cu_2F_{24}N_6O_{12}$. Calculated (%): C, 42.6; H, 2.8; N, 5.3; F, 28.9.

$[Cu(hfac)_2L^{Thio}]_2$. Procedure A. Yield 60%. Found (%): C, 38.9; H, 2.4; N, 5.0; S, 4.0; F, 29.0. $C_{52}H_{40}Cu_2F_{24}N_6O_{12}S_2$. Calculated (%): C, 39.3; H, 2.5; N, 5.3; S, 4.0; F, 28.7.

$[Cu(hfac)_2]_4(L^{CP})_4$. Procedure B. Yield 95%. Found (%): C, 41.8; H, 3.2; N, 5.4; F, 29.7. $C_{108}H_{104}Cu_4F_{48}N_{12}O_{24}$. Calculated (%): C, 41.6; H, 3.4; N, 5.4; F, 29.3.

$[Cu(hfac)_2]_4(L^{Cy})_2$. Procedure B. Yield 96%. Found (%): C, 35.5; H, 2.0; N, 3.1; F, 36.4. $C_{76}H_{60}Cu_4F_{48}N_6O_{20}$. Calculated (%): C, 35.9; H, 2.4; N, 3.3; F, 35.9.

$[Cu(hfac)_2L^{OMe}]_2$. Procedure A. Yield 84%. Found (%): C, 37.2; H, 3.0; N, 5.5; F, 30.9. $C_{46}H_{40}Cu_2F_{24}N_6O_{14}$. Calculated (%): C, 37.2; H, 2.7; N, 5.7; F, 30.7.

$[Cu(hfac)_2L^{OEt}]_2$. Procedure A. Yield 79%. Found (%): C, 38.2; H, 2.9; N, 5.6; F, 30.3. $C_{48}H_{44}Cu_2F_{24}N_6O_{12}$. Calculated (%): C, 38.1; H, 2.9; N, 5.6; F, 30.2.

$[Cu(hfac)_2]_4(L^{i\text{-}Pr})_4$. Procedure B. Yield 98%. Found (%): C, 40.2; H, 3.2; N, 5.5; F, 30.7. $C_{50}H_{48}Cu_2F_{24}N_6O_{12}$. Calculated (%): C, 39.8; H, 3.2; N, 5.6; F, 30.2.

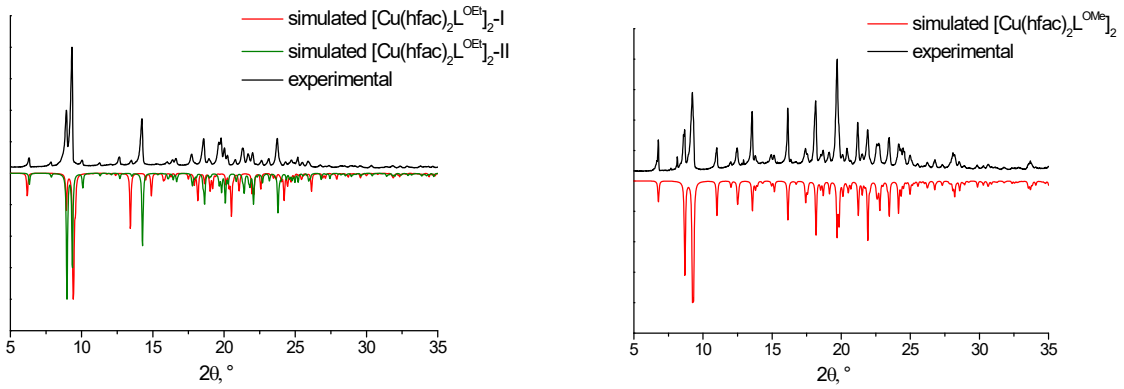
$[Cu(hfac)_2L^{n\text{-}Pr}]_2$. Procedure B. Yield 76%. Found (%): C, 40.2; H, 3.0; N, 5.3; F, 30.5. $C_{50}H_{48}Cu_2F_{24}N_6O_{12}$. Calculated (%): C, 39.8; H, 3.2; N, 5.6; F, 30.2.

2. Characterization

The microanalyses were performed on a CHNS EA-3000 analyzer (HEKAtech), halogens were determined according to Schöniger's flask combustion technique: chlorine and bromine by mercurimetric titration, fluorine by spectrophotometry of its Ln-alizarin-complex on a Cary-50 (Agilent) at the Multi-access Service Center of Vorozhtsov Novosibirsk Institute of Organic Chemistry of Siberian Branch of Russian Academy of Sciences.

2.1. Powder X-ray diffraction

PXRD data were collected on a PowDix 600 (ADANI) diffractometer equipped with a MYTHEN2 R 1D (Dectris) detector at room temperature using Cu Kα radiation at a scanning speed on θ of 0.01°/s. The powders of samples were placed in an aluminum sample holder. The experimental patterns were compared to the simulated ones based on crystal structures obtained by single crystal XRD, which confirmed good phase purity for all of the compounds. The result of phase analysis is entirely consistent with the magnetic measurements and elemental analyses.



a *b*

Fig. S1. Simulated and experimental PXRD patterns of $[Cu(hfac)_2L^{OEt}]_2$ (*a*) and $[Cu(hfac)_2L^{OMe}]_2$ (*b*).

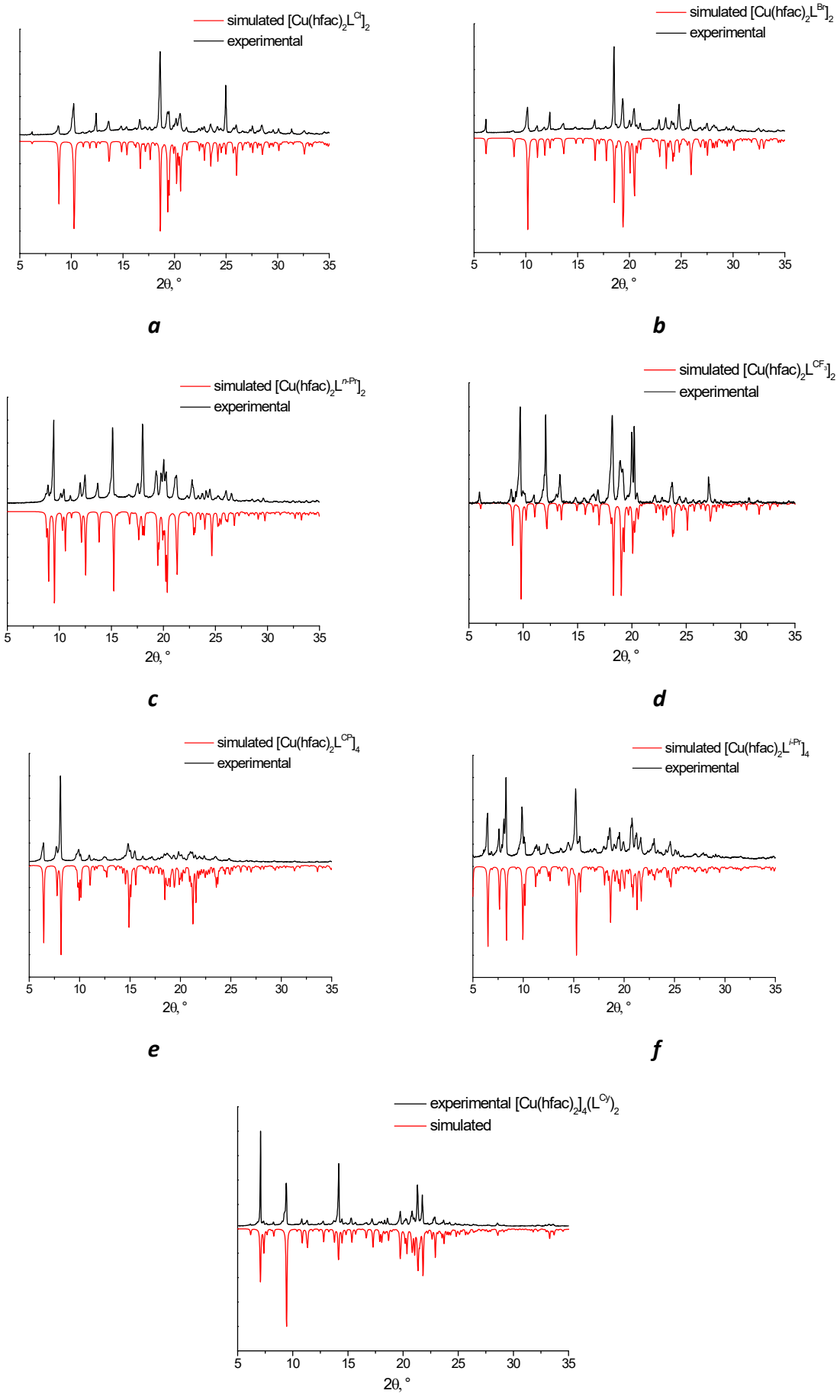


Fig. S2. Simulated and experimental PXRD patterns of $[Cu(hfac)_2L^{Cl}]_2$ (*a*), $[Cu(hfac)_2L^{Br}]_2$ (*b*), $[Cu(hfac)_2L^{n\text{-}Pr}]_2$ (*c*), $[Cu(hfac)_2L^{CF3}]_2$ (*d*), $[Cu(hfac)_2]_4(L^{CP})_4$ (*e*), $[Cu(hfac)_2L^{i\text{-}Pr}]_4$ (*f*), $[Cu(hfac)_2]_4(L^{Cy})_2$ (*g*).

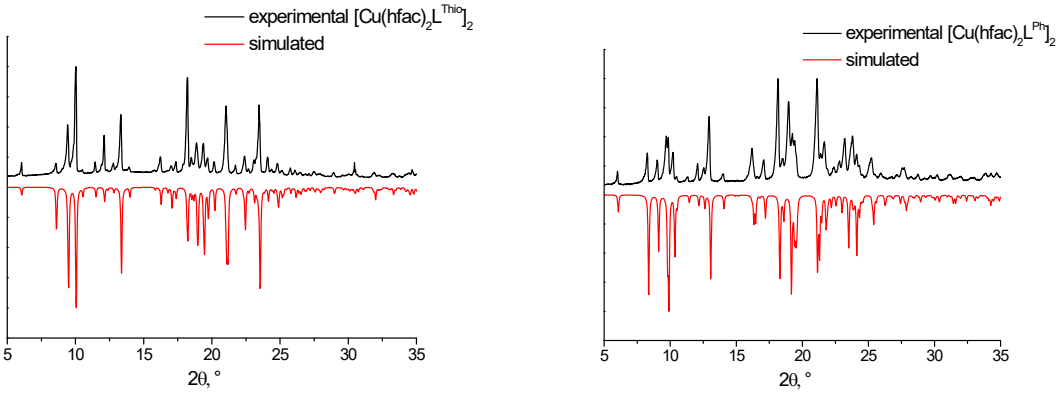


Fig. S3. Simulated and experimental PXRD patterns of $[Cu(hfac)_2L^{Thio}]_2$ (*a*) and $[Cu(hfac)_2L^{Ph}]_2$ (*b*).

2.2. Magnetic measurements

The magnetic susceptibility of the polycrystalline samples was measured with an MPMSXL (Quantum Design) SQUID magnetometer at temperatures of 2–350 K in a magnetic field of 5 kOe. The paramagnetic components of magnetic susceptibility χ were determined with allowance for the diamagnetic contribution evaluated from Pascal's constants. The effective magnetic moment was calculated as $\mu_{eff} = [3k\chi T/(N_A\mu_B^2)]^{1/2} \approx (8\chi T)^{1/2}$, where N_A, μ_B and k — Avogadro's number, Bohr's magneton, and the Boltzmann constant, respectively. Analysis of the experimental data was performed using PHI program [7].

2.3. Single crystal X-ray diffraction

The intensity data for the single crystals of the compounds were collected on a SMART APEX II (Bruker AXS) automated diffractometer with a Helix (Oxford Cryosystems) open flow helium cooler or APEX DUO (Bruker AXS) automated diffractometer with a Cobra (Oxford Cryosystems) open flow nitrogen cooler using the standard procedure (Mo or Cu Kα radiation). The structures were solved by direct methods and refined by the full-matrix least-squares procedure anisotropically for non-hydrogen atoms. The H atoms were calculated geometrically and included in the refinement as riding groups. All calculations were performed using the SHELX programs [8,9]. The crystallographic data and details of experiments are presented in the Supplementary data (Table S1). Complete X-ray diffraction datasets are deposited to the Cambridge Crystallographic Data Center (CCDC Nos are listed in the Table S1).

Table S1. Crystal data and details of experiments for $[Cu(hfac)_2L^R]_2$

Formula	$[Cu(hfac)_2L^{Cl}]_2$	$[Cu(hfac)_2L^{Br}]_2$	$[Cu(hfac)_2L^{OMe}]_2$	$[Cu(hfac)_2L^{OEt}]_2$-I	$[Cu(hfac)_2L^{OEt}]_2$-II
FW	1492.75	1581.66	1483.92	1511.97	1511.97
T, K	296	296	296	296	296
Space group, *Z*	*P*-1, 1	*P*-1, 1	$P2_1/c$, 2	$P2_1/c$, 2	*P*-1, 1
a,	9.8746(3)	9.9004(6)	14.4037(9)	14.2166(6)	10.6583(3)
b,	11.1848(3)	11.1739(7)	14.0717(9)	18.8967(7)	11.4385(4)
c, Å	15.1435(4)	15.2180(9)	16.2483(10)	11.7669(5)	14.7135(4)
α, °	94.536(2)	94.670(3)	90	90	101.619(1)
β, °	105.712(2)	105.300(3)	115.139(4)	101.305(2)	91.641(1)
γ, °	113.245(1)	113.986(3)	90	90	117.014(1)
V, Å^3	1446.28(7)	1449.2(2)	2981.3(3)	3099.8(2)	1550.43(8)
V/*Z*, Å^3	1446	1449	1491	1550	1550
D_{calc}, g/cm^3	1.714	1.812	1.653	1.620	1.619
ϑ_{max}, °	28.385	67.821	28.000	22.998	67.766
I_{hkl} total / uniq /	24794 / 7094	20077 / 5161	25745 / 7130	21323 / 4313	23816 / 5559
R_{int}	0.0421	0.0641	0.0349	0.0635	0.0501
I_{hkl} ($I>2\sigma_I$) / N	4434 / 461	4462 / 452	4775 / 524	2847 / 506	4705 / 526
Goof	0.932	1.086	0.969	0.940	1.031
*R*1 / *wR*2 ($I>2\sigma_I$)	0.0408 / 0.0972	0.0793 / 0.1966	0.0390 / 0.0897	0.0376 / 0.0725	0.0522 / 0.1443
*R*1 / *wR*2 (all data)	0.0708 / 0.1069	0.0856 / 0.2002	0.0708 / 0.1058	0.0675 / 0.0804	0.0604 / 0.1549
CCDC	2160694	2160688	2322572	2160674	2322569

Table S1. Crystal data and details of experiments for $[Cu(hfac)_2L^R]_2$ (continuation)

Formula	$[Cu(hfac)_2L^{n\text{-}Pr}]_2$	$[Cu(hfac)_2L^{i\text{-}Pr}]_4$	$[Cu(hfac)_2]_4L^{Cy}_2$	$[Cu(hfac)_2]_4L^{CP}_4$	$[Cu(hfac)_2L^{CF3}]_2$
FW	1508.02	3016.04	2543.46	3120.19	1559.87
T, K	240	296	296	296	296
Space group, *Z*	*P*-1, 1	*P*-1, 1	*P*-1, 1	*P*-1, 1	*P*-1, 1
a,	10.0433(7)	12.9249(11)	13.560(4)	12.8787(4)	10.1290(6)
b,	11.4576(8)	15.5532(13)	13.632(4)	15.4515(5)	11.3259(7)
c, Å	15.5010(11)	19.6020(17)	15.472(5)	19.9472(7)	15.4203(9)
α, °	70.884(3)	97.260(4)	108.589(6)	69.652(2)	97.324(3)
β, °	83.932(3)	108.948(5)	95.635(6)	71.759(2)	103.008(4)
γ, °	67.474(3)	113.736(4)	108.934(5)	66.608(2)	116.335(3)
V, Å^3	1556.4(2)	3257.8(5)	2498.4(14)	3344.5(2)	1491.5(2)
V/*Z*, Å^3	1556	1629	2498	3344	1491
D_{calc}, g/cm^3	1.609	1.537	1.690	1.549	1.737
ϑ_{max}, °	67.464	24.998	28.432	22.499	22.499
I_{hkl} total / uniq /	21732 / 5537	47304 / 11466	46134 /12483	37517 / 8743	21817 / 3882
R_{int}	0.0315	0.0351	0.0911	0.0572	0.0627
I_{hkl} (I>2σ$_I$) / N	5108 / 569	8120 / 973	6150 / 884	5623 / 1055	2711 / 542
Goof	1.023	1.075	0.889	1.009	0.972
*R*1 / *wR*2 (I>2σ$_I$)	0.0379 / 0.1045	0.0520 / 0.1454	0.0398 / 0.0842	0.0431 / 0.0973	0.0409 / 0.0922
*R*1 / *wR*2 (all I_{hkl})	0.0406 / 0.1076	0.0754 / 0.1635	0.0940 / 0.0980	0.0870 / 0.1201	0.0630 / 0.1024
CCDC	2160678	2322575	2160699	2322576	2160697

Table S1. Crystal data and details of experiments for $[Cu(hfac)_2L^R]_2$ (continuation)

Formula	$[Cu(hfac)_2L^{Ph}]_2$				
FW	1576.04				
Space group, *Z*	*P*-1, 1				
T, K	293	240	200	150	110
a,	10.1383(4)	10.1211(5)	10.1260(2)	10.1299(3)	10.1333(3)
b,	11.9217(5)	11.8936(7)	11.9623(4)	11.8398(3)	11.7570(4)
c, Å	15.8104(6)	15.7645(8)	15.7188(4)	15.6339(4)	15.5832(5)
α, °	94.3542(18)	94.128(4)	93.6043(16)	93.9306(13)	94.2706(15)
β, °	108.3180(16)	108.514(4)	108.7135(11)	108.4411(12)	108.2460(15)
γ, °	113.6971(18)	114.163(4)	115.5974(11)	115.3408(12)	115.1866(14)
V, Å^3	1615.55(11)	1596.45(16)	1580.48(7)	1561.48(7)	1548.26(9)
V/*Z*, Å^3	1615	1596.4	1580.5	1561.5	1548.3
D_{calc}, g/cm^3	1.620	1.639	1.656	1.676	1.690
ϑ_{max}, °	67.932	67.873	67.662	67.543	67.634
I_{hkl} total / uniq	24760 / 5766	15237 / 5684	24018 / 5662	24725 / 5572	21639 / 5522
R_{int}	0.0280	0.0486	0.0301	0.0290	0.0297
I_{hkl} (I>2σ_I) / N	5106 / 559	3660 / 520	5065 / 537	5104 / 510	5032 / 456
Goof	0.972	1.028	1.036	1.062	1.057
*R*1 / *wR*2 (I>2σ_I)	0.0461 / 0.1313	0.0593 / 0.1482	0.0483 / 0.1235	0.0465 / 0.1227	0.0476 / 0.1258
*R*1 / *wR*2 (all I_{hkl})	0.0512 / 0.1376	0.1008 / 0.1724	0.0536 / 0.1282	0.0498 / 0.1256	0.0512 / 0.1289
CCDC	2322584	2160680	2322567	2322583	2322585

Table S1. Crystal data and details of experiments for $[Cu(hfac)_2L^R]_2$ (continuation)

Formula	$[Cu(hfac)_2L^{Thio}]_2$			
FW	1588.10			
T, K	296	220	170	120
Space group, *Z*	*P*-1, 1			
a,	10.3562(6)	10.3383(9)	10.3236(8)	10.3312(5)
b,	11.3841(7)	11.3546(10)	11.3283(9)	11.2769(6)
c, Å	15.6769(9)	15.5573(14)	15.4538(12)	15.3209(7)
α, °	72.469(3)	72.304(5)	72.418(5)	72.939(3)
β, °	71.725(3)	71.704(5)	71.960(5)	72.059(3)
γ, °	67.095(3)	66.374(5)	65.621(4)	64.992(3)
V, Å^3	1582.36(17)	1554.9(3)	1533.2(2)	1511.14(14)
V/*Z*, Å^3	1583	1555	15335	15115
D_{calc}, g/cm^3	1.667	1.696	1.720	1.745
ϑ_{max}, °	24.999	28.388	28.364	28.309
I_{hkl} total / uniq / R_{int}	22623 / 5589 / 0.0495	28424 / 7713 / 0.0690	25909 / 7591 / 0.0800	23775 / 73980 / 0.0848
I_{hkl} (I>2σ$_I$) / N	3964 / 534	4979 / 527	4946 / 484	4777 / 496
Goof	1.063	1.084	1.065	0.986
*R*1 / *wR*2 (I>2σ$_I$)	0.0437 / 0.1140	0.0509 / 0.1281	0.0554 / 0.1354	0.0509 / 0.1144
*R*1 / *wR*2 (all I_{hkl})	0.0645 / 0.1229	0.0853 / 0.1426	0.0924 / 0.1507	0.0901 / 0.1300
CCDC	2322578	2322574	2322580	2322566

3. Selected bond lengths, angles and contacts in dimers

Table S2. Selected bond lengths, contacts and angles (Å, deg) (data are given for binuclear fragment only*)

Formula	T, K	*Cu-O_{NO}	*∠CuON	*(O_{NO}–)Cu–O_{hfac}	*Cu-N	N–O_{Cu} / N–O	∠CN_2O_2–Py	O_{NO}…O_{NO}	Cu…Cu
					I				
$[Cu(hfac)_2L^{OMe}]_2$	296	1.977(2)	124.9(1)	2.008(2)	1.990(1)	1.301(2) / 1.263(2)	57.1	–	5.8108(5)
$[Cu(hfac)_2L^{OEt}]_2$–I	296	1.985(2)	124.0(1)	1.987(2)	2.022(2)	1.300(2) / 1.265(2)	56.1	5.783(3)	5.6724(5)
$[Cu(hfac)_2L^{OEt}]_2$–II	296	1.982(2)	126.5(1)	1.983(2)	2.029(2)	1.297(3) / 1.269(3)	47.7	4.625(8)	5.8497(4)
					II				
$[Cu(hfac)_2L^{CF3}]_2$	296	2.530(2)	154.1(2)	2.225(2)	2.060(2)	1.274(3) / 1.274(3)	80.5	6.276(5)	7.3425(6)
$[Cu(hfac)_2L^{n\text{-}Pr}]_2$	240	2.454(1)	143.4(1)	2.276(2)	2.015(1)	1.278(2) / 1.274(2)	61.7	4.636(2)	6.9038(6)
$[Cu(hfac)_2L^{Cl}]_2$	296	2.501(1)	149.2(1)	2.241(2)	2.058(1)	1.280(2) / 1.271(2)	68.0	6.075(3)	7.1619(5)
$[Cu(hfac)_2L^{Br}]_2$	296	2.510(4)	150.0(4)	2.238(4)	2.054(4)	1.275(6) / 1.268(6)	70.8	4.625(8)	7.204(1)
$[Cu(hfac)_2]_4(L^{i\text{-}Pr})_4$	296	2.507(3)	142.7(2)	2.330(3)	2.002(2)	1.279(3) / 1.279(3)	60.1	3.622(4)	6.3206(8)
$[Cu(hfac)_2]_4(L^{Cy})_2$	296	2.555(2)	138.5(1)	2.256(2)	2.014(2)	1.278(2) / 1.273(3)	52.3	–	6.722(2)
$[Cu(hfac)_2]_4(L^{CP})_4$	296	2.492(2)	140.3(2)	2.322(2)	1.991(3)	1.283(3) / 1.285(3)	58.5	3.467(5)	6.0336(7)
					III				
$[Cu(hfac)_2L^{Thio}]_2$	296	2.249(2)	133.1(1)	2.263(3)	1.996(2)	1.285(3) / 1.267(3)	58.8	5.513(3)	6.3393(5)
	220	2.160(2)	131.0(1)	2.186(3)	1.995(2)	1.292(3) / 1.264(3)	57.7	5.509(4)	6.2536(7)
	170	2.058(2)	128.6(2)	2.060(3)	1.999(2)	1.302(3) / 1.273(3)	56.9	5.497(4)	6.1720(6)
	120	1.990(2)	126.3(2)	2.014(2)	1.999(2)	1.300(3) / 1.268(3)	56.9	5.490(5)	6.1357(6)
$[Cu(hfac)_2L^{Ph}]_2$	296	2.189(1)	131.5(1)	2.227(3)	2.008(2)	1.294(3) / 1.269(3)	54.7	5.432(4)	6.2011(5)
	240	2.135(3)	130.6(3)	2.149(6)	2.003(4)	1.324(5) / 1.270(5)	54.3	5.396(6)	6.159(1)
	200	2.080(2)	128.8(1)	2.076(2)	2.005(2)	1.294(3) / 1.273(3)	53.4	5.389(4)	6.1073(6)
	150	2.018(2)	127.1(1)	2.015(2)	2.005(2)	1.296(3) / 1.268(3)	52.9	5.356(4)	6.0637(4)
	110	1.991(2)	126.0(1)	2.006(2)	2.004(2)	1.301(3) / 1.270(3)	52.6	5.340(3)	6.0419(4)

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