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Supplementary Information

Cationic 2,2’-bipyridine Complexes of Germanium(II) and Tin(II)

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Experimental Details

All reactions were carried out in a MBraun glove box under an N_2 atmosphere. Solvents were dried over CaH_2 or Na/Benzophenone and stored over molecular sieves (3 Å: MeCN, 4 Å: Et_2O, CH_2Cl_2, DMSO) unless otherwise specified. Anhydrous MeCN-d_3 was purchased from Aldrich and stored over molecular sieves for 48 hours prior to use. $GeCl_2$·Dioxane and $SnCl_2$ were purchased from Sigma-Aldrich and used as received. 2,2'-bipyridine was purchased from Sigma-Aldrich and recrystallized from CH_2Cl_2 prior to use. Trimethylsilyl trifluoromethanesulfonate (TMSOTf) was purchased from Sigma-Aldrich and distilled prior to use.

Solution ^{1}H, ^{13}C, ^{19}F and ^{119}Sn NMR spectra were collected at room temperature on a Bruker AVANCE 300 (7.02 T) or Bruker Avance 360 (8.4 T) NMR spectrometers, with relative frequencies and temperatures explicitly given for each compound. Chemical shifts are reported in ppm relative to trace protonated solvent (^{1}H), perdeuterated solvent (^{13}C), or an external reference standard (^{119}Sn, $Sn(CH_4)_4$; ^{19}F, $CFCl_3$). NMR spectra of reaction mixtures were obtained by transferring an aliquot of the bulk solution to a 5 mm NMR tube, which was then capped and sealed with Teflon tape.

Infrared spectra were collected on samples using Nujol mull using a Perkin-Elmer Spectru, FT-IR spectrometer and peaks are reported in wavenumbers (cm^{-1}). Melting points were obtained on samples sealed in glass capillaries under dry nitrogen using an Electrothermal apparatus.

Elemental analyses were performed on selected compounds by Canadian Microanalytical Services Ltd., Delta, British Columbia, Canada.

Unless otherwise stated, crystals for single crystal X-ray diffraction studies were obtained by vapor diffusion at room temperature or from slow diffusion of a layered non-solvent into a saturated solution of the compound at -35°C. Single crystal X-ray diffraction data were collected on a Bruker D8 Apex II diffractometer with graphite-monochromated Mo Kα radiation (λ = 0.71073 Å) at room temperature. Data were collected and integrated using the Bruker SAINT software package.[1] Absorption corrections were performed using the multi-scan technique (SADABS).[2] The structures were solved by intrinsic phasing using SHELXT[3] and refined using all reflections with SHELXL.[4] All non hydrogen atoms were refined anisotropically. Structures were solved and refined using Olex2 package.[5] Crystallographic information files have been deposited in the Cambridge Structural Database (CSD) with numbers 1546731, 1546732, and 1546733 for [(bipy)GeCl][OTf], $[(bipy)_2Ge][OTf]_2$, and$[(bipy)_2Sn][OTf]_2$ respectively. Quantum chemical calculations were carried out using Gaussian 09.[6]

Synthesis of [(bipy)GeCl][OTf] – Method A: $GeCl_2$·dioxane (2 mmol, 0.463g), TMSOTf (2 mmol, 362 μL) and 2,2'-bipyridine (2 mmol, 0.312 g) were combined in 5 mL CH_3CN to obtain a colourless, cloudy mixture which was stirred for 2 hours. Removal of the solvent under reduced pressure gave the product as a fine colourless powder. The powder was washed with CH_2Cl_2 and Et_2O and re-dissolved in a minimum volume of CH_3CN. The resulting solution was filtered, layered with Et_2O, and placed in a freezer at -35°C for 48 hours to obtain colourless crystalline material.

Method B: $GeCl_2$·dioxane (2 mmol, 0.463g) and 2,2'-bipyridine (2 mmol, 0.312 g) were combined in 5 mL CH_2Cl_2 to immediately give a bright yellow suspension, which was stirred for 1 hour. Removal of the solvent under reduced pressure gave a dark yellow powder as the previously characterized neutral adduct. The powder was subsequently re-suspended in CH_3CN, and to this mixture was added TMSOTf (2 mmol, 362 μL), giving a slightly cloudy, colourless mixture after 2 hours. Removal of the solvent gave the title product as a colourless powder. The powder was washed with CH_2Cl_2 and Et_2O and re-dissolved in a minimum volume of CH_3CN.

The resulting solution was filtered, layered with Et_2O, and placed in a freezer at -35°C for 48 hours to obtain colourless crystalline material.

Yield: 0.726 g (88%); ^{1}H NMR, ppm (CD_3CN, 298K, 300.1 MHz) δ = 8.05 (ddd, J = 7.6, 5.6, 1.2 Hz, 2H), 8.55 (td, J = 7.6, 1.5 Hz, 2H), 8.66 (*pseudo dt*, J = 8.1, 1.1 Hz, 2H), 9.12 (ddd, J = 5.6, 1.5, 0.9 Hz, 2H; ^{13}C{^{1}H} NMR, ppm (CD_3CN, 298K, 75.5 MHz): δ = 124.3 (s), 128.9 (s), 144.8 (s), 146.0 (s), 148.8 (s); ^{19}F{^{1}H} NMR, ppm (CD_3CN, 298K, 282.5 MHz) δ = -79.2 (s); Melting point: 205-206°C; IR, Nujol mull, cm^{-1}: 3118, 2921, 2853, 2325, 1621, 1605, 1581, 1576, 1506, 1480, 1466, 1453, 1441, 1377, 1326, 1291, 1276, 1266, 1248, 1219, 1185, 1150, 1109, 1058, 1049, 1039, 1024, 1019, 984, 921, 776, 770, 726, 658, 649, 635, 576, 572;

Synthesis of [(bipy)$_2$Ge][OTf]$_2$ – Method A: $GeCl_2$·dioxane (2 mmol, 0.463g), TMSOTf (4 mmol, 724 μL) and 2,2'-bipyridine (4 mmol, 0.624 g) were combined in 5 mL CH_3CN to obtain a clear, yellow solution which was stirred for 2 hours. Removal of the solvent under reduced pressure gave a fine yellow powder. The powder was washed with CH_2Cl_2 and Et_2O and re-dissolved in a minimum volume of CH_3CN. The resulting solution was filtered, layered with Et_2O, and placed in a freezer at -35°C for 48 hours to obtain yellow crystalline material.

Method B: $GeCl_2$·dioxane (2 mmol, 0.463g) and 2,2'-bipyridine (2 mmol, 0.312 g) were combined in 5 mL CH_2Cl_2 to immediately give a bright yellow suspension, which was stirred for 1 hour. Removal of the solvent under reduced pressure gave a dark yellow powder as the previously characterized neutral adduct. The powder was subsequently re-suspended in CH_3CN, and to this mixture was added TMSOTf (4 mmol, 724 μL), giving first a cloudy, colourless mixture, turning to a clear, yellow solution which was stirred for 2 hours. Removal of the solvent under reduced pressure gave a fine yellow powder. The powder was washed with CH_2Cl_2 and Et_2O and re-dissolved in a minimum volume of CH_3CN. The resulting solution was filtered, layered with Et_2O, and placed in a freezer at -35°C for 48 hours to obtain yellow crystalline material.

Yield: 1.175 g (86%); ^1H NMR, ppm (CD_3CN, 298K, 300.1 MHz) δ = 7.89 (t, broad, J = 6.5 Hz, 2H), 8.48 (td, broad, J = 7.9, 1.2 Hz, 2H), 8.69 (d, broad, J = 8.2 Hz, 2H), 8.86 (d, broad, J = 4.9 Hz, 2H; $^{13}C\{^1H\}$ NMR, ppm (CD_3CN, 298K, 75.5 MHz): δ = 124.5 (s), 128.5 (s), 143.9 (s), 147.4 (s), 159.1 (s); $^{19}F\{^1H\}$ NMR, ppm (CD_3CN, 298K, 282.5 MHz) δ = -79.3 (s); Melting point: 195-196°C; (dec) IR, Nujol mull, cm^{-1}: 3091, 2953, 2921, 2853, 2342, 1610, 1604, 1594, 1583, 1562, 1509, 1462, 1446, 1377, 1317, 1281, 1254, 1234, 1224, 1191, 1152, 1112, 1099, 1088, 1031, 1021, 971, 943, 819, 772, 757, 734, 723, 654, 648, 637, 630, 612, 573; Elemental Analysis including 0.75 molecules of CH_2Cl_2 (Calc/Exp): C (36.59/36.52); H (2.36/2.13); N (7.50/7.53) for crystalline sample with yield of 0.093g

Synthesis of [(bipy)SnCl][OTf] – Method A: $SnCl_2$ (2 mmol, 0.379g), TMSOTf (2 mmol, 362 µL) and 2,2'-bipyridine (2 mmol, 0.312 g) were combined in 5 mL CH_3CN to obtain a colourless suspension which was stirred for 2 hours. Removal of the solvent under reduced pressure gave the product as a fine colourless powder. The powder was washed with CH_2Cl_2 and Et_2O and pumped dry. Due to it's low solubility in CH_2Cl_2 and CH_3CN, crystalline material suitable for x-ray analysis could not be isolated. However, the analysis of the bulk material was consistent with the title compound.

Method B: $SnCl_2$ (2 mmol, 0.379g) and 2,2'-bipyridine (2 mmol, 0.312 g) were combined in 5 mL CH_2Cl_2 to immediately give a bright yellow suspension, which was stirred for 1 hour. Removal of the solvent under reduced pressure gave a dark yellow powder as the previously characterized neutral adduct. The powder was subsequently re-suspended in CH_3CN, and to this mixture was added TMSOTf (2 mmol, 362 µL), giving a colourless suspension after 2 hours.

Removal of the solvent gave the title product as a colourless powder. The powder was washed with CH_2Cl_2 and Et_2O and pumped dry. Due to it’s low solubility in CH_2Cl_2 and CH_3CN, crystalline material suitable for x-ray analysis could not be isolated. However, the analysis of the bulk material was consistent with the title compound. Yield: 0.827 g (90%); ^1H NMR, ppm (CD_3CN/DMSO, 298K, 300.1 MHz) δ = 7.63 (ddd, J = 7.6, 5.3, 1.2 Hz, 2H), 8.11 (td, J = 7.8, 1.8 Hz, 2H), 8.45 (*pseudo dt*, J = 7.9, 1.1 Hz, 2H), 8.91 (ddd, J = 5.3, 1.8, 0.9 Hz, 2H;. $^{13}C\{^1H\}$ NMR, ppm (CD_3CN/DMSO, 298K, 75.5 MHz): δ = 123.3 (s), 126.8 (s), 141.0 (s), 149.3 (s), 153.6 (s); $^{19}F\{^1H\}$ NMR, ppm (CD_3CN, 298K, 282.5 MHz) δ = -79.2 (s); $^{119}Sn\{^1H\}$ NMR, ppm, (CD_3CN/DMSO, 298K, 134.3 MHz) δ = -577.0 (s); Melting point: 211-212°C; IR, Nujol mull, cm^{-1}: 3113, 2921, 2853, 2724, 2364, 1610, 1601, 1576, 1567, 1502, 1463, 1448, 1377, 1324, 1292, 1271, 1250, 1219, 1188, 1160, 1151, 1114, 1108, 1074, 1064, 1044, 1019, 979, 909, 771, 767, 757, 730, 671, 654, 638, 633, 576, 572; Elemental Analysis (Calc/Exp): C (28.76/28.56); H (1.76/1.68); N (6.10/5.98).

Synthesis of [(bipy)$_2$Sn][OTf]$_2$] – Method A: $SnCl_2$ (2 mmol, 0.379g), TMSOTf (4 mmol, 724 µL) and 2,2’-bipyridine (4 mmol, 0.624 g) were combined in 5 mL CH_3CN to obtain a clear, yellow solution which was stirred for 2 hours. Removal of the solvent under reduced pressure gave a fine yellow powder. The powder was washed with CH_2Cl_2 and Et_2O and re-dissolved in a minimum volume of CH_3CN. The resulting solution was filtered, layered with Et_2O, and placed in a freezer at -35°C for 48 hours to obtain yellow crystalline material.

Method B: $SnCl_2$ (2 mmol, 0.379g) and 2,2’-bipyridine (2 mmol, 0.312 g) were combined in 5 mL CH_2Cl_2 to immediately give a bright yellow suspension, which was stirred for 1 hour. Removal of the solvent under reduced pressure gave a dark yellow powder as the previously characterized neutral adduct. The powder was subsequently re-suspended in CH_3CN, and to this mixture was added TMSOTf (4 mmol, 724 μL), giving first a colourless suspension, turning to a clear, yellow solution which was stirred for 2 hours. Removal of the solvent under reduced pressure gave a fine yellow powder. The powder was washed with CH_2Cl_2 and Et_2O and re-dissolved in a minimum volume of CH_3CN. The resulting solution was filtered, layered with Et_2O, and placed in a freezer at -35°C for 48 hours to obtain yellow crystalline material.

Yield: 1.283 g (88%); ^{1}H NMR, ppm (CD_3CN, 298K, 300.1 MHz) δ = 7.79 (ddd, *J* = 7.8, 5.3, 1.2 Hz, 2H), 8.33 (td, *J* = 7.9, 1.6 Hz, 2H), 8.51 (*pseudo dt*, *J* = 8.1, 1.0 Hz, 2H), 8.74 (ddd, *J* = 5.3, 1.8, 0.9 Hz, 2H;. $^{13}C\{^1H\}$ NMR, ppm (CD_3CN, 298K, 75.5 MHz): δ = 125.3 (s), 129.0 (s), 143.7 (s), 149.3 (s), 151.5 (s); $^{19}F\{^1H\}$ NMR, ppm (CD_3CN, 298K, 282.5 MHz) δ = -79.5 (s); $^{119}Sn\{^1H\}$ NMR, ppm, (CD_3CN, 298K, 134.3 MHz) δ = -728.6 (s); Melting point 192°C (dec): IR, Nujol mull, cm^{-1}: 3068, 2924, 2853, 2725, 2258, 1605, 1598, 1578, 1567, 1495, 1467, 1445, 1377, 1321, 1276, 1296, 1276, 1238, 1222, 1158, 1135, 1025, 1013, 1004, 908, 808, 756, 748, 732, 653, 634, 627, 572; Elemental Analysis (Calc/Exp): C (36.24/35.89); H (2.21/2.07); N (7.68/7.51) for crystalline sample with yield of 0.113g.

Crystallographic Details

Data was collected on a Bruker X8 Apex II diffractometer with graphite-monocromated Mo Kα radiation (λ = 0.71073 Å) at room temperature. Data were collected and integrated using the Bruker SAINT software package.[1] Absorption corrections were performed using the multi-scan technique (SADABS).[2] The structures were solved by intrinsic phasing using SHELXT[3] and refined using all reflections with SHELXL.[4] All non hydrogen atoms were refined anisoropically. Structures were solved and refined using Olex2 package.[5]

Table S1: Crystal data and refinement details for **[GeCl(bipy)][OTf]**

Compound	**[GeCl(bipy)][OTf]**
Empirical formula	$C_{11}H_8ClF_3GeN_2O_3S$
CCDC number	1546731
Formula weight	413.29
Temperature (K)	296.15
Crystal system	monoclinic
Space group	$P2_1/n$
a (Å)	7.5779(7)
b (Å)	16.7991(14)
c (Å)	11.0317(10)
α (°)	90
β (°)	93.030(3)
γ (°)	90
Volume (Å^3)	1402.4(2)
Z	4
ρ_{calc} (g/cm^3)	1.957
μ (mm^{-1})	2.571
F(000)	816.0
Crystal size (mm^3)	0.21 × 0.16 × 0.12
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.422 to 60.082
Index ranges	$-10 \le h \le 10$, $-23 \le k \le 21$, $-15 \le l \le 14$
Reflections collected	14581
Independent reflections	4100 [R_{int} = 0.0504, R_{sigma} = 0.0503]
Structure solution method	intrinsic phasing
Refinement method	full-matrix least-squares on F^2 (SHELXL-03)
Absorption correction	multi-scan (SADABS)
Data/restraints/parameters	4100/0/199
Goodness-of-fit on F^2	1.047
Final R indexes [I>=2σ (I)]	R_1 = 0.0320, wR_2 = 0.0715
Final R indexes [all data]	R_1 = 0.0441, wR_2 = 0.0759
Largest diff. peak/hole / e Å^{-3}	0.55/-0.35

Table S2: Crystal data and refinement details for **$[Ge(bipy)_2][OTf]_2$**

Compound	**$[Ge(bipy)_2][OTf]_2$**
Empirical formula	$C_{24}H_{19}F_6GeN_5O_6S_2$
CCDC number	1546732
Formula weight	724.15
Temperature (K)	273.15
Crystal system	triclinic
Space group	P-1
a (Å)	9.6827(5)
b (Å)	12.2345(7)
c (Å)	13.5264(8)
α (°)	105.9780(10)
β (°)	96.5280(10)
γ (°)	109.6380(10)
Volume ($Å^3$)	1412.75(14)
Z	2
ρ_{calc} (g/cm^3)	1.702
μ (mm^{-1})	1.321
F(000)	728.0
Crystal size (mm^3)	0.22 × 0.16 × 0.14
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.216 to 60.156
Index ranges	$-13 \le h \le 13$, $-17 \le k \le 17$, $-19 \le l \le 19$
Reflections collected	30486
Independent reflections	8260 [$R_{int} = 0.0265$, $R_{sigma} = 0.0254$]
Structure solution method	intrinsic phasing
Refinement method	full-matrix least-squares on F^2 (SHELXL-03)
Absorption correction	multi-scan (SADABS)
Data/restraints/parameters	8260/0/398
Goodness-of-fit on F^2	1.044
Final R indexes [$I>=2\sigma(I)$]	$R_1 = 0.0244$, $wR_2 = 0.0613$
Final R indexes [all data]	$R_1 = 0.0280$, $wR_2 = 0.0629$
Largest diff. peak/hole / e $Å^{-3}$	0.47/-0.45

Table S3: Crystal data and refinement details for **$[Sn(bipy)_2][OTf]_2$**

Compound	**$[Sn(bipy)_2][OTf]_2$**
Empirical formula	$C_{25.3}H_{21.5}Cl_2F_6N_{5.2}O_6S_2Sn$
CCDC number	1546733
Formula weight	862.12
Temperature (K)	296.15
Crystal system	monoclinic
Space group	$P2_1/n$
a (Å)	9.3539(4)
b (Å)	19.7280(9)
c (Å)	17.8782(8)
α (°)	90
β (°)	103.627(2)
γ (°)	90
Volume (Å^3)	3206.3(2)
Z	4
ρ_{calc} (g/cm^3)	1.786
μ (mm^{-1})	1.178
F(000)	1711.0
Crystal size (mm^3)	0.41 × 0.38 × 0.14
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.124 to 60.206
Index ranges	$-13 \leq h \leq 13$, $-27 \leq k \leq 24$, $-25 \leq l \leq 24$
Reflections collected	36861
Independent reflections	9384 [R_{int} = 0.0361, R_{sigma} = 0.0376]
Structure solution method	intrinsic phasing
Refinement method	full-matrix least-squares on F^2 (SHELXL-03)
Absorption correction	multi-scan (SADABS)
Data/restraints/parameters	9384/1332/554
Goodness-of-fit on F^2	1.206
Final R indexes [I>=2σ (I)]	R_1 = 0.0567, wR_2 = 0.1135
Final R indexes [all data]	R_1 = 0.0699, wR_2 = 0.1179
Largest diff. peak/hole / e Å^{-3}	1.32/-2.03

Cartesian Coordinates of Optimized Geometries

Geometries computed at the B3LYP/Def2TZVPP level of theory

$[(bipy)_2Ge]^{2+}$
E = -3067.411353

Ge	0	0	-1.09773
N	-0.80637	-1.30335	0.37021
N	0.80637	1.30335	0.37021
N	2.11304	-0.73106	-0.72786
N	-2.11303	0.73106	-0.72786
C	2.14395	1.24258	0.59904
C	2.88453	0.17495	-0.0999
C	-2.14395	-1.24258	0.59904
C	-2.75342	-2.15271	1.45588
H	-3.81431	-2.09123	1.64568
C	-0.07456	-2.25287	0.97909
H	0.98283	-2.25579	0.76581
C	-0.629	-3.18136	1.83871
H	0.00246	-3.92373	2.306
C	0.07456	2.25287	0.9791
H	-0.98283	2.25578	0.76581
C	-2.88453	-0.17494	-0.0999
C	1.99524	3.13088	2.08071
H	2.46501	3.83941	2.75015
C	0.62899	3.18136	1.83871
H	-0.00247	3.92372	2.306
C	-1.99524	-3.13088	2.08071
H	-2.46501	-3.83941	2.75015
C	4.05191	-1.86786	-1.51936
H	4.47382	-2.68372	-2.08945
C	4.27296	0.09538	-0.15768
H	4.89479	0.83457	0.32488
C	-2.67758	1.71717	-1.43479
H	-2.006	2.39722	-1.94536
C	2.75342	2.15271	1.45588
H	3.81431	2.09123	1.64568
C	2.67758	-1.71717	-1.43479
H	2.006	-2.39722	-1.94536
C	-4.27296	-0.09538	-0.15768
H	-4.89479	-0.83456	0.32489
C	-4.0519	1.86786	-1.51937
H	-4.47382	2.68372	-2.08945

$[(bipy)_2Sn]^{2+}$
E = -1204.766271

Sn	0	0	-1.27441
N	-0.84331	-1.42345	0.37529
N	0.84332	1.42345	0.37529
N	-2.25595	0.65156	-0.64837
N	2.25595	-0.65156	-0.64838
C	2.14227	1.30526	0.74336
C	-0.09007	-2.39514	0.91393
H	0.93781	-2.44614	0.58625
C	-2.14227	-1.30526	0.74336
C	-4.28099	1.79176	-1.19682
H	-4.76993	2.59726	-1.72662
C	4.3233	0.13428	0.22999
H	4.88144	0.85282	0.81059
C	-2.90918	1.62738	-1.29157
H	-2.31176	2.293	-1.90398
C	0.57855	3.2924	1.84548
H	-0.06865	4.0587	2.24813
C	2.90917	-1.62738	-1.29158
H	2.31176	-2.293	-1.90399
C	4.28099	-1.79176	-1.19683
H	4.76993	-2.59726	-1.72663
C	0.09007	2.39514	0.91393
H	-0.93781	2.44614	0.58625
C	2.93931	0.23197	0.10383
C	-2.68604	-2.17415	1.68568
H	-3.71412	-2.06839	1.99602
C	2.68604	2.17415	1.68568
H	3.71412	2.06839	1.99602
C	-2.93931	-0.23198	0.10383
C	4.99731	-0.8901	-0.42113
H	6.0724	-0.97352	-0.33147
C	1.90342	3.17536	2.24067
H	2.32462	3.85198	2.97252
C	-4.99731	0.8901	-0.42113
H	-6.0724	0.97351	-0.33147
C	-0.57855	-3.29239	1.84548
H	0.06865	-4.0587	2.24813

C	-4.8597	0.94284	-0.86926
H	-5.93754	1.01973	-0.9252
C	4.85971	-0.94284	-0.86926
H	5.93754	-1.01972	-0.9252

E = -3032.554006

Ge	0	-1.59209	-0.69704
Cl	0.00001	-2.34161	1.39029
N	-1.30299	0.01573	-0.27102
N	1.30299	0.01574	-0.27102
C	-2.63232	-0.12135	-0.29424
H	-3.01641	-1.10902	-0.51589
C	-3.4761	0.94597	-0.04051
H	-4.54698	0.80283	-0.05952
C	-2.91274	2.18518	0.23448
H	-3.54309	3.04135	0.43462
C	-1.5311	2.32167	0.2528
H	-1.08407	3.28112	0.46615
C	-0.73709	1.21028	-0.00137
C	0.73708	1.21028	-0.00137
C	1.53109	2.32168	0.2528
H	1.08405	3.28113	0.46615
C	2.91273	2.1852	0.23448
H	3.54307	3.04136	0.43463
C	3.4761	0.94599	-0.04052
H	4.54698	0.80285	-0.05952
C	2.63231	-0.12134	-0.29424
H	3.01642	-1.10901	-0.51589

C	-4.3233	-0.13428	0.22998
H	-4.88144	-0.85283	0.81059
C	-1.90341	-3.17536	2.24067
H	-2.32462	-3.85198	2.97252

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