Electronic Supplementary Information for Dalton Transactions

Supplementary Material

$[(\eta\text{-}C_5H_4R)Fe(CO)_2X]$, X = Cl, Br, I, NO_3, CO_2Me and $[(\eta\text{-}C_5H_4R)Fe(CO)_3]^+$, R = $(CH_2)_nCO_2Me$ (n = 0 - 2), and $CO_2CH_2CH_2OH$: a new group of CO releasing molecules

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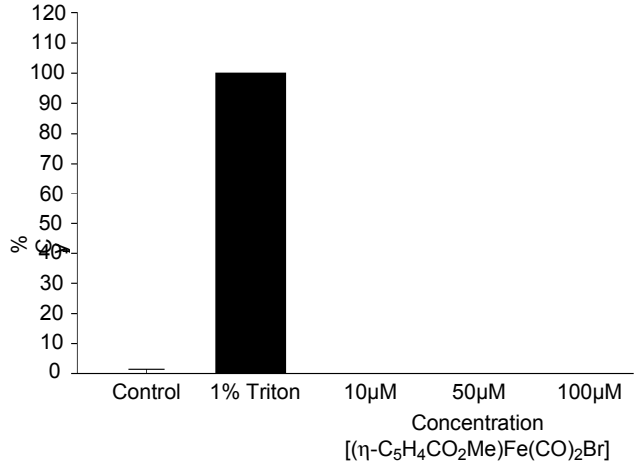

Fig S1. The cytotoxicity of 10, 50 and 100 μM $[(\eta\text{-}C_5H_4CO_2Me)Fe(CO)_2Br]$ in murine RAW264.7 macrophages treated for 24 hours was tested using the LDH release assay. 1% Triton was used to produce the total release of LDH from the cells to provide a reference for 100% toxicity.

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Table S1. The *cis/trans* ratio of $[(\eta\text{-}C_5H_4R)Fe(CO)_2]_2$, R = H,[39] CO_2Me, $CO_2CH_2CH_2OH$, in various solvents determined from the intensity of ν(CO) bands.

Solvent	R = H	R = CO_2Me	R = $CO_2CH_2CH_2OH$
Benzene	50/50	48/52	
$CHCl_3$	55/45	57/43	60/40
Et_2O	56/44	56/44	
CH_2Cl_2	60/40	67/33	67/33
$MeNO_2$		71/29	70/30
THF	71/29	78/22	80/20
Acetone		76/24	80/20
MeCN	81/19	79/21	78/22
DMSO	85/15	81/19	82/18

Table S2 X-ray crystal data for [(η-$C_5H_4CO_2R$)$Fe(CO)_2]_2$, R = Me, CH_2CH_2OH, [(η-$C_5H_4CO_2Me$)$Fe(CO)_2X$], X = Cl, Br, I, NO_3, CO_2Me, and [(η-$C_5H_4CO_2Me$)$Fe(CO)_3$][$FeCl_4$].

Compound	[(η-$C_5H_4CO_2Me$) $Fe(CO)_2]_2$	[(η-$C_5H_4CO_2Me$) $Fe(CO)_2Cl$]	[(η-$C_5H_4CO_2Me$) $Fe(CO)_2Br$]	[(η-$C_5H_4CO_2Me$) $Fe(CO)_2I$]
Empirical formula	C18 H14 Fe2 O8	C9 H7 Cl Fe O4	C9 H7 Br Fe O4	C9 H7 Fe I O4
Formula weight	469.99	270.45	314.91	361.90
Temperature	150(2) K	150(2) K	150(2) K	150(2) K
Wavelength	0.71073 Å	0.71073 Å	0.71073 Å	0.71073 Å
Crystal system	Triclinic	Triclinic	Monoclinic	Monoclinic
Space group	P-1	P-1	$P2_1/n$	$P2_1/n$
Unit cell dimensions	a = 6.525(3) Å b = 6.757(3) Å c = 10.099(5) Å α= 87.774(8)°. β= 86.467(8)° γ = 73.748(7)°.	a = 6.6386(8) Å b = 12.6126(16) Å c = 13.5047(17) Å α= 67.996(2)° β= 83.392(2)° γ = 77.062(2)°	a = 6.8607(8) Å b = 18.920(2) Å c = 8.0278(9) Å α= 90° β= 90.881(7)° γ = 90°	a = 7.0434(18) Å b = 8.121(2) Å c = 19.607(5) α= 90° β= 100.262(4)° γ = 90°
Volume	426.5(3) $Å^3$	1021.2(2) $Å^3$	1041.9(2) $Å^3$	1103.6(5) $Å^3$
Z	1	4	4	4
Density (calculated)	1.830 Mg/m^3	1.759 Mg/m^3	2.008 Mg/m^3	2.178 Mg/m^3
Absorption coefficient	1.748 mm^{-1}	1.726 mm^{-1}	5.270 mm^{-1}	4.153 mm^{-1}
F(000)	238	544	616	688
Crystal size	0.32 x 0.20 x 0.04 mm^3	0.50 x 0.20 x 0.10 mm^3	0.39 x 0.21 x 0.18 mm^3	0.39 x 0.29 x 0.22 mm^3
Theta range for data collection	2.02 to 24.99°.	1.63 to 27.55°	2.15 to 27.46°	2.11 to 27.55°
Index ranges	-7<=h<=7, -8<=k<=8, -11<=l<=11	-8<=h<=8, -16<=k<=16, -17<=l<=17	-8<=h<=8, -24<=k<=24, -10<=l<=10	-9<=h<=8, -10<=k<=10, -25<=l<=25
Reflections collected	3012	11391	11597	11922
Independent reflections	1472 [R(int) = 0.0428]	4526 [R(int) = 0.0257]	2361 [R(int) = 0.1916]	2509 [R(int) = 0.0566]
Completeness to	theta = 24.99°, 98.4%	theta = 25.00°, 99.7%	theta = 27.46°, 99.1%	theta = 25.00°, 100.0%
Absorption correction	Semi-empirical from equivalents	Semi-empirical from equivalents	Semi-empirical from equivalents	Semi-empirical from equivalents
Max. and min. transmission	0.9334 and 0.6047	0.8463 and 0.4791	0.4505 and 0.2330	0.4619 and 0.2942
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data / restraints / parameters	/1472 / 0 / 127	4526 / 0 / 273	2361 / 0 / 138	2509 / 0 / 137
Goodness-of-fit on F^2	1.296	1.041	0.929	1.001
Final R indices [I>2sigma(I)]	R1 = 0.0677, wR2 = 0.1856	R1=0.0277, wR2 = 0.0705	R1=0.0562, wR2 = 0.1215	R1=0.0368, wR2 = 0.0924
R indices (all data)	R1 = 0.0778, wR2 = 0.1893	R1=0.0354, wR2 = 0.0732	R1=0.0964, wR2 = 0.1381	R1=0.0530, wR2 = 0.1004
Extinction coefficient			0.0030(9)	
Largest diff. peak and hole	1.280 and -0.708 e.$Å^{-3}$	0.439 and -0.271 e.$Å^{-3}$	1.189 and -0.863 e.$Å^{-3}$	1.376 and -0.657 e.$Å^{-3}$
Weighting scheme	$1/[\sigma^2(Fo^2)+(0.0380*P)^2+5.2504*P]$	$1/[\sigma^2(Fo^2)+(0.0416*P)^2+0.0477*P]$	$1/[\sigma^2(Fo^2)+(0.057500*P)^2+0.00*P]$	$1/[\sigma^2(Fo^2)+(0.058000*P)^2+0.00*P]$
CCDC Reference Number	640137	640139	640140	640141

[a] $P=(Fo^2+2*Fc^2)/3$

Table S2 Continued.

Compound	[(η-$C_5H_4CO_2Me$) $Fe(CO)_2(NO_3)$]	[(η-$C_5H_4CO_2Me$) $Fe(CO)_2(CO_2Me)$]	[(η-$C_5H_4CO_2Me$) $Fe(CO)_3$][$FeCl_4$]	[(η-$C_5H_4CO_2CH_2$ $CH_2OH)Fe(CO)_2]_2$
Empirical formula	C9 H7 Fe N O7	C11 H10 Fe O6	C10 H7 Cl4 Fe2 O5	C20 H18 Fe2 O10
Formula weight	297.01	294.04	460.66	530.04
Temperature	150(2) K	150(2) K	150(2) K	150(2) K
Wavelength	0.71073 Å	0.71073 Å	0.71073 Å	0.71073 Å
Crystal system	Monoclinic	Monoclinic	Monoclinic	Triclinic
Space group	$P2_1/n$	$P2_1/c$	$P2_1/n$	P-1
Unit cell dimensions	a = 7.037(2) Å	a = 6.5711(14) Å	a = 7.0283(6) Å	a = 7.283(4) Å
	b = 8.134(2) Å	b = 12.667(3) Å	b = 23.800(2) Å	b = 8.083(4) Å
	c = 19.058(6) Å	c = 14.092(3) Å	c = 9.6737(8)	c = 17.632(8) Å
	α= 90°	α= 90°	α= 90°	α= 103.139(5)°
	β= 96.053(18)°	β= 97.571(4)°	β= 90.931(4)°	β= 97.091(5)°
	γ = 90°	γ = 90°	γ = 90°	γ = 98.965(5)°
Volume	1084.8(5) $Å^3$	1162.7(4) $Å^3$	1617.9(2) $Å^3$	984.7(9) $Å^3$
Z	4	4	4	2
Density (calculated)	1.819 Mg/m^3	1.680 Mg/m^3	1.891 Mg/m^3	1.788 Mg/m^3
Absorption coefficient	1.418 mm^{-1}	1.313 mm^{-1}	2.466 mm^{-1}	1.533 mm^{-1}
F(000)	600	600	908	540
Crystal size	0.21 x 0.11 x 0.05 mm^3	0.42 x 0.21 x 0.21 mm^3	0.24 x 0.18 x 0.15 mm^3	0.21 x 0.19 x 0.08 mm^3
Theta range for data collection	2.73 to 24.99°	2.17 to 27.52°	1.71 to 27.85°	2.41 to 26.99°
Index ranges	-8<=h<=8,	-8<=h<=8,	-9<=h<=9,	-9<=h<=8,
	-9<=k<=9,	-16<=k<=16,	-31<=k<=31,	-9<=k<=10,
	-22<=l<=22	-18<=l<=18	-12<=l<=12	-22<=l<=22
Reflections collected	10058	12813	31667	9874
Independent reflections	1878 [R(int) = 0.0383]	2638 [R(int) = 0.0362]	3830 [R(int) = 0.0220]	3875 [R(int) = 0.0154]
Completeness to	theta = 24.99°	theta = 27.52°	theta = 25.00°	theta = 25.00°
	98.0%	98.0%	100.0%	99.5%
Absorption correction	Semi-empirical from equivalents	Semi-empirical from equivalents	Semi-empirical from equivalents	Semi-empirical from equivalents
Max. and min. transmission	0.9325 and 0.7550	0.7700 and 0.6085	0.7086 and 0.5891	0.8872 and 0.7391
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data / restraints / parameters	1878 / 0 / 164	2638 / 0 / 165	3830 / 0 / 190	3875 / 8 / 302
Goodness-of-fit on F^2	1.168	1.058	1.061	1.067
Final R indices [I>2sigma(I)]	R1 = 0.0360, wR2 = 0.1077	R1 = 0.0299, wR2 = 0.0762	R1 = 0.0192, wR2 = 0.0528	R1 = 0.0236, wR2 = 0.0597
R indices (all data)	R1 = 0.0396, wR2 = 0.1131	R1 = 0.0387, wR2 = 0.0802	R1 = 0.0203, wR2 = 0.0534	R1 = 0.0255, wR2 = 0.0611
Largest diff. peak and hole	0.647 and -0.642 $e.Å^{-3}$	0.669 and -0.243 $e.Å^{-3}$	0.548 and -0.481 $e.Å^{-3}$	0.594 and -0.525 $e.Å^{-3}$
Weighting Scheme[a]	$1/[\sigma^2(Fo^2) + (0.0822*P)^2 + 0.00*P]$	$1/[\sigma^2(Fo^2) + (0.0498*P)^2 + 0.0932*P]$	$1/[\sigma^2(Fo^2) + (0.0106*P)^2 + 0.00*P]$	$1/[\sigma^2(Fo^2) + (0.0285*P)^2 + 0.7338*P]$
CCDC Reference Number	640142	640143	640144	640138

[a] $P=(Fo^2+ 2 * Fc^2)/3$.