Supporting Information for

Location of CO_2 During its Uptake by the Flexible Porous Metal-Organic Framework MIL-53(Fe): a High Resolution Powder X-ray Diffraction Study

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1. Crystal Structure of INT MIL-53(Fe)[0.22CO_2]

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data_
_chemical_name_mineral ??
_cell_length_a  6.87219(24)
_cell_length_b  11.10220(36)
_cell_length_c  13.92736(35)
_cell_angle_alpha 108.3144(21)
_cell_angle_beta  92.6033(31)
_cell_angle_gamma 112.2990(36)
_cell_volume 916.797(59)
_symmetry_space_group_name_H-M P-1
loop_
_symmetry_equiv_pos_as_xyz
        '-x, -y, -z'
        'x, y, z'
loop_
_atom_site_label
_atom_site_type_symbol
_atom_site_symmetry_multiplicity
_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
_atom_site_occupancy
_atom_site_B_iso_or_equiv
Fe1 Fe+2   2 0.53339(73) 0.49152(38) 0.28969(31) 1 0.248(44)
Fe2 Fe+2   2 0.02963(74) 0.49657(37) 0.29048(31) 1 0.248(44)
O1 O-2   2 -0.2353(22) 0.4723(10) 0.2046(10) 1 1.817(92)
O2 O-2   2 0.2895(21) 0.4929(11) 0.38923(86) 1 1.817(92)
O11 O-2   2 -0.05338919 0.3304538 0.5946922 1 1.817(92)
O12 O-2   2 0.2639689 0.374427 0.5521712 1 1.817(92)
O21 O-2   2 0.5016198 0.3302498 0.3109738 1 1.817(92)
O22 O-2   2 0.8411071 0.3742858 0.3511123 1 1.817(92)
O23 O-2   2 0.2178023 -0.3923198 0.2075258 1 1.817(92)
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O24 O-2 2 0.5589242 -0.3519237 0.2276706 1 1.817(92)
O31 O-2 2 0.02199237 -0.324293 -0.1577821 1 1.817(92)
O32 O-2 2 -0.3175553 -0.363439 -0.1620928 1 1.817(92)

C11 C 2 0.04138396 0.138526 0.5288622 1 1.08(12)
C12 C 2 -0.1600063 0.04288101 0.5238116 1 1.08(12)
C13 C 2 -0.2013902 -0.09564497 0.4949495 1 1.08(12)
C17 C 2 0.08611244 0.2882472 0.5600569 1 1.08(12)

H12 H 2 -0.2744633 0.07355502 0.5408448 1 4
H13 H 2 -0.3454504 -0.1640625 0.4913367 1 4

C21 C 2 0.5854198 0.1322798 0.2965913 1 1.08(12)
C22 C 2 0.3712499 0.0364697 0.2606446 1 1.08(12)
C23 C 2 0.3156934 -0.1057371 0.2383739 1 1.08(12)
C24 C 2 0.4743069 -0.1521337 0.25205 1 1.08(12)
C25 C 2 0.6884768 -0.05632366 0.2879967 1 1.08(12)
C26 C 2 0.7440333 0.08588309 0.3102673 1 1.08(12)
C27 C 2 0.646156 0.2877447 0.3209382 1 1.08(12)
C28 C 2 0.4135708 -0.3075987 0.227703 1 1.08(12)

H22 H 2 0.2601824 0.06895854 0.251068 1 4
H23 H 2 0.165723 -0.1728272 0.2132025 1 4
H25 H 2 0.7995443 -0.08881251 0.2975732 1 4
H26 H 2 0.8940037 0.1529732 0.3354387 1 4

C31 C 2 -0.05826205 -0.1355674 -0.06305448 1 1.08(12)
C32 C 2 0.1536077 -0.04908804 -0.02394462 1 1.08(12)
C33 C 2 0.2118698 0.08647931 0.03910986 1 1.08(12)
C37 C 2 -0.1207768 -0.2810301 -0.1307115 1 1.08(12)

H32 H 2 0.2634877 -0.08420212 -0.04107289 1 4
H33 H 2 0.3634263 0.1483404 0.06708626 1 4

O3 O 2 0.633023 0.4595029 0.005087555 0.8808(60) 4
C3 C 1 0.5 0.5 0 0.8808(60) 4

Crystal Structure of NP MIL-53(Fe)[0.63CO_2]

data_
_chemical_name_mineral ?anhydre?
_cell_length_a 20.85952(15)
_cell_length_b 8.25079(10)
_cell_length_c 6.87385(10)
_cell_angle_alpha 90
_cell_angle_beta 113.73099(75)

_cell_angle_gamma 90
_cell_volume 1083.008(23)
_symmetry_space_group_name_H-M C12/c1
loop_
_symmetry_equiv_pos_as_xyz
'-x, -y, -z'
'-x, y, -z+1/2'
'-x+1/2, -y+1/2, -z'
'-x+1/2, y+1/2, -z+1/2'
'x, -y, z+1/2'
'x, y, z'
'x+1/2, -y+1/2, z+1/2'
'x+1/2, y+1/2, z'
loop_
_atom_site_label
_atom_site_type_symbol
_atom_site_symmetry_multiplicity
_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
_atom_site_occupancy
_atom_site_B_iso_or_equiv
Fe Fe+3 4 0 0 0 1 0.839(29)
O1 O-2 4 0 -0.13280(50) 0.75 1 2.341(59)
O11 O-2 8 0.4360981 0.3220945 0.3194872 1 2.341(59)
O12 O-2 8 0.4179841 0.3575982 -0.01577772 1 2.341(59)
C11 C 8 0.3195132 0.2852772 0.0596241 1 2.989(91)
C12 C 8 0.2950384 0.2423956 0.2098749 1 2.989(91)
C13 C 8 0.2255252 0.2071184 0.1502508 1 2.989(91)
C17 C 8 0.3948215 0.3234952 0.1242188 1 2.989(91)
H12 H 8 0.3272557 0.236956 0.3600044 1 4
H13 H 8 0.2080177 0.176444 0.2577295 1 4
C2 C 8 -0.009857748 -0.4802838 0.8385022 0.3128(11) 4
O2 O-2 8 0.04492282 -0.5077538 0.9741424 0.3128(11) 4
O3 O-2 8 -0.06463832 -0.4528139 0.7028621 0.3128(11) 4

Crystal Structure of LP MIL-53(Fe)[2.72CO_2]

data_
_chemical_name_mineral ?hkl_Phase?
_cell_length_a 16.56794(24)
_cell_length_b 13.63839(28)
_cell_length_c 6.917599(53)
_cell_angle_alpha 90
_cell_angle_beta 90
_cell_angle_gamma 90
_cell_volume 1563.101(40)
_symmetry_space_group_name_H-M Imcm
loop_

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_symmetry_equiv_pos_as_xyz
        '-x, -y, -z'
        '-x, -y, z'
        '-x, y+1/2, -z+1/2'
        '-x, y+1/2, z+1/2'
        '-x+1/2, -y+1/2, -z+1/2'
        '-x+1/2, -y+1/2, z+1/2'
        '-x+1/2, y, -z'
        '-x+1/2, y, z'
        'x, -y+1/2, -z+1/2'
        'x, -y+1/2, z+1/2'
        'x, y, -z'
        'x, y, z'
        'x+1/2, -y, -z'
        'x+1/2, -y, z'
        'x+1/2, y+1/2, -z+1/2'
        'x+1/2, y+1/2, z+1/2'
loop_
_atom_site_label
_atom_site_type_symbol
_atom_site_symmetry_multiplicity
_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
_atom_site_occupancy
_atom_site_B_iso_or_equiv
Fe Fe+3   4 0.75 0.25 0.25 1 0.035(40)
O1 O-2   4 0.75 0.17438(44) 0 1 0.372(71)
O11 O-2  16 -0.1648964 0.164283 0.6589934 1.00 0.372(71)
C11 C   8 -0.06394417 0.06370631 0.5 1 1.30(10)
C12 C  16 -0.03197209 0.03185315 0.6715299 1 1.30(10)
C17 C   8 -0.135261 0.1347579 0.5 1 1.30(10)
H12 H  16 -0.05484031 0.05463631 0.7942177 1 4
C2 C   8 0.75 -0.4563318 0.2598212 0.5 4
O2 O   8 0.75 -0.4563318 0.4289551 0.5 4
O3 O   8 0.75 -0.4563318 0.09068738 0.5 4
C4 C  16 -0.6016403 -0.7716419 0.8807642 0.4296(13) 4
O4 O  16 -0.5343043 -0.7963097 0.8960025 0.4296(13) 4
O5 O  16 -0.6689762 -0.7469742 0.865526 0.4296(13) 4
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