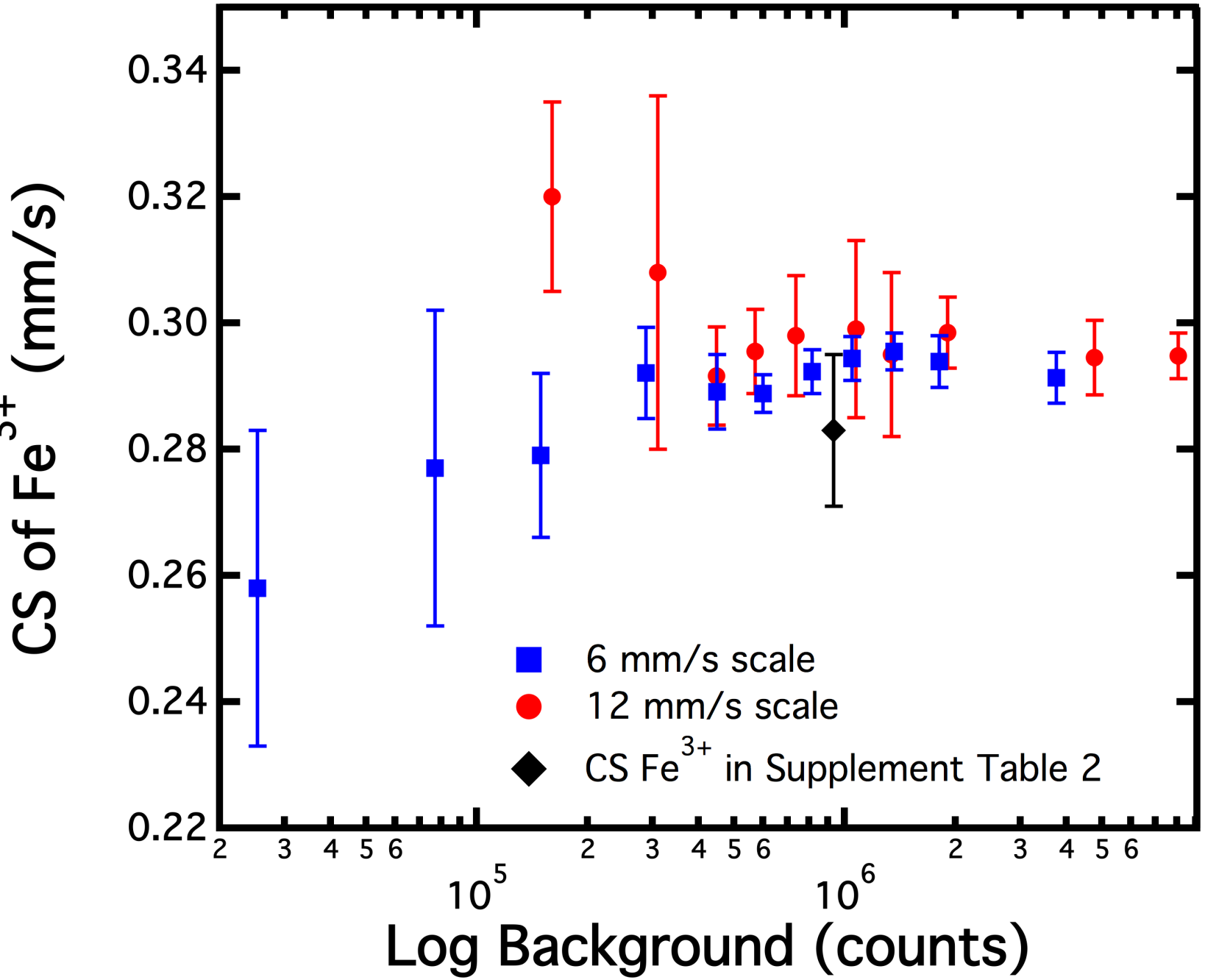
Supplement Materials:

Supplement Fig. 1: Effect of variable Mössbauer collection time and velocity scale on Center Shifts (CS) for (a) ferric and (b) ferrous iron for sample VF3. The blue squares refer to CS obtained from Mössbauer spectra collected with 6 mm/s scale. The red dots show CS obtained from Mössbauer spectra collected with 12 mm/s scale. The black diamond refers to the room temperature data point collected with the standard methodology applied during data collection at variable temperature, as shown in Supplement Table 2. All uncertainties in this figure are in 2σ.

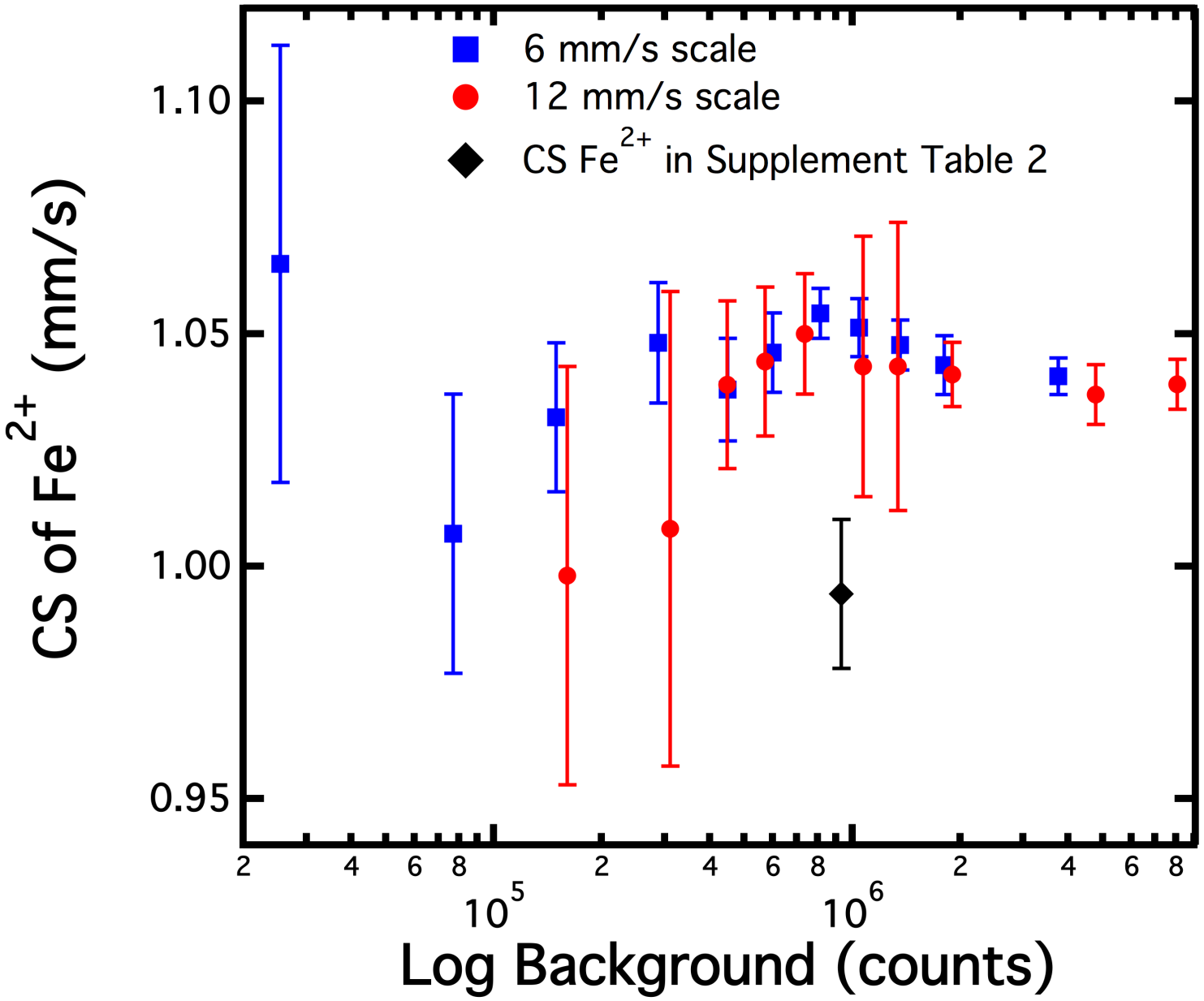
Supplement Fig. 2: Effect of variable Mössbauer collection time and scale on $Fe^{3+}/\Sigma Fe$ for sample VF3, with data points as described in Supplement Fig. 1.

Supplement Fig. 3: Hysteresis between applied magnetic field and sample magnetization determined for powdered sugar.

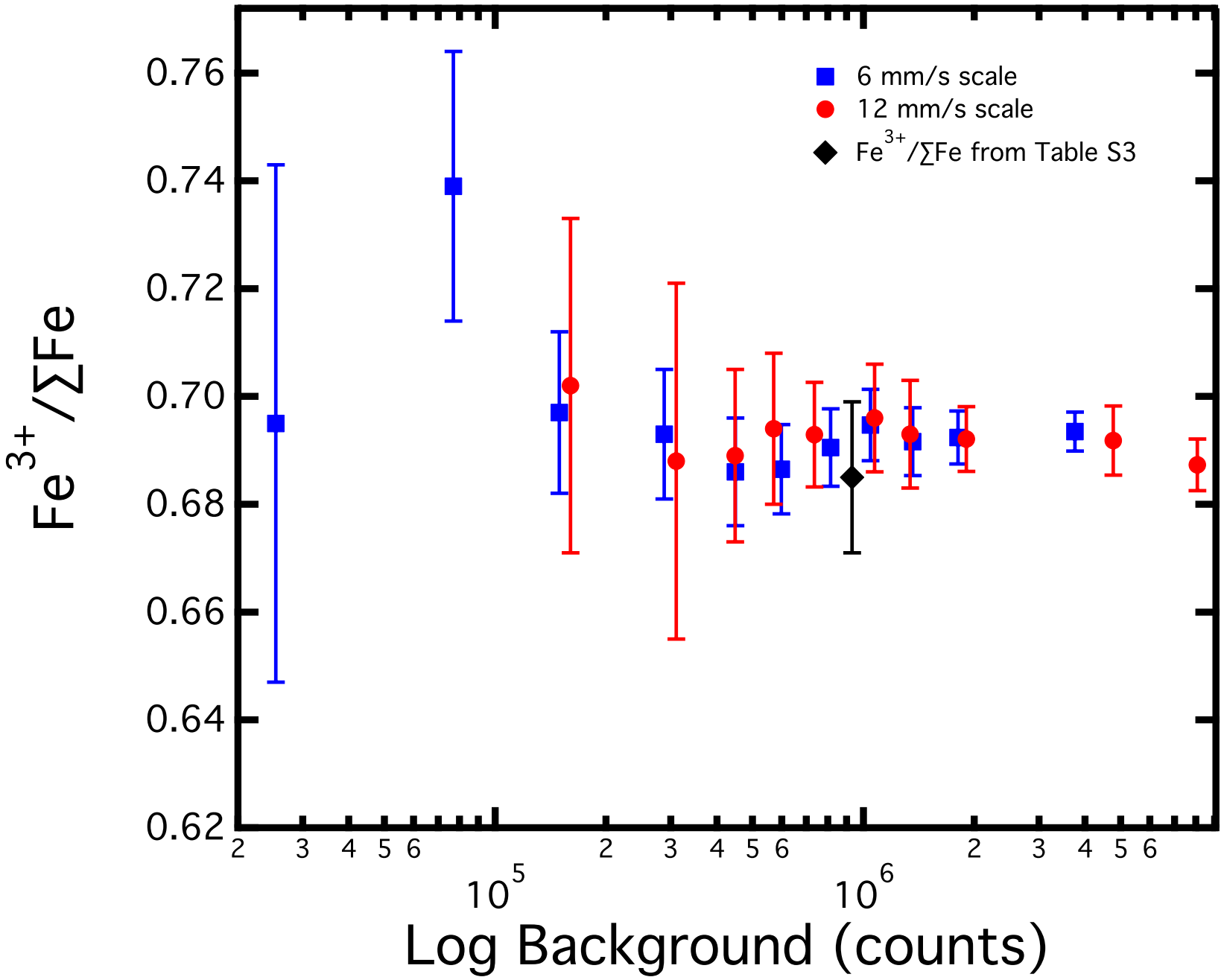
Supplement Figure 1a:



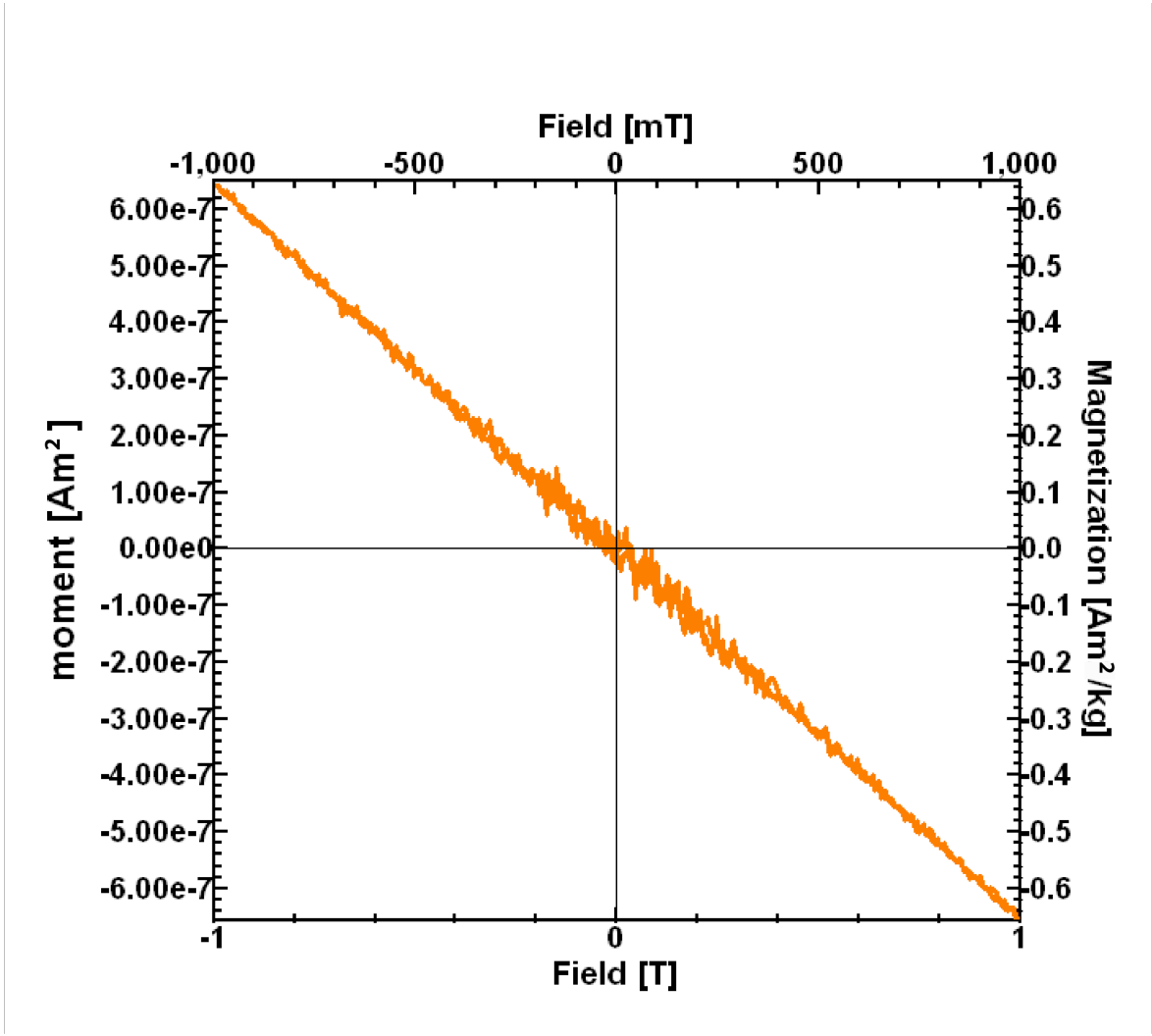
Supplement Figure 1b:



Supplement Figure 2:



Supplement Figure 3:



Supplement Table 1: Parameters of VF3 Mössbauer spectra collected with different scales and different duration at room temperature.

Time	χ^2	L HWHM	CS (Fe^{2+})	δ_{CS} (Fe^{2+})	QS (Fe^{2+})	δ_{QS} (Fe^{2+})	ρ (Fe^{2+})	CS (Fe^{3+})	δ_{CS} (Fe^{3+})	QS (Fe^{3+})	δ_{QS} (Fe^{3+})
6 mm/s scale											
0.188	0.877	0.175(61)	1.057(84)	0.04(12)	2.01(18)	0.33(33)	-0.82(87)	0.256(36)	0.002(95)	1.243(83)	0.382(61)
0.5	1.097	0.2030(94)	0.979(55)	0.15(10)	1.98(11)	0.13(16)	0.43(99)	0.290(35)	0.022(95)	1.195(56)	0.563(69)
1	0.949	0.113(29)	1.017(56)	0.236(28)	1.88(13)	0.293(84)	0.23(29)	0.295(24)	0.000(51)	1.207(50)	0.578(14)
2	1.109	0.140(20)	1.047(28)	0.212(30)	1.825(53)	0.262(95)	0.31(25)	0.304(15)	0.000(51)	1.198(28)	0.512(21)
3	1.057	0.160(15)	1.020(15)	0.171(12)	1.836(24)	0.341(23)	0.432(84)	0.293(70)	0.000(38)	1.157(13)	0.478(16)
4	1.128	0.122(16)	1.0321(86)	0.223(11)	1.854(29)	0.206(39)	0.400(79)	0.2944(79)	0.000(43)	1.182(11)	0.531(12)
5.5	0.939	0.168(15)	1.0394(94)	0.1635(81)	1.852(19)	0.327(16)	0.421(79)	0.291(57)	0.000(38)	1.1681(87)	0.470(15)
7	0.991	0.150(21)	1.0314(97)	0.106(22)	1.875(28)	0.436(22)	0.400(81)	0.2933(63)	0.000(42)	1.155(10)	0.505(10)
9	1.147	0.177(17)	1.034(10)	0.220(11)	1.858(22)	0.194(34)	0.40(14)	0.3014(83)	0.000(41)	1.1616(75)	0.467(13)
12	1.228	0.177(10)	1.0264(61)	0.2039(84)	1.852(16)	0.214(26)	0.479(72)	0.2973(46)	0.000(33)	1.1655(78)	0.4591(75)
25	1.136	0.1558(94)	1.0303(55)	0.2316(47)	1.834(13)	0.189(14)	0.394(52)	0.2945(27)	0.001(27)	1.1567(39)	0.4882(91)
12 mm/s scale											
1	0.494	0.317(35)	0.998(45)	0.276(71)	1.991(69)	0.142(99)	-0.59(85)	0.320(15)	0.002(71)	1.108(28)	0.354(41)
2	0.517	0.250(40)	1.008(51)	0.239(53)	1.90(12)	0.33(16)	-0.10(62)	0.308(28)	0.050(67)	1.143(50)	0.399(46)
3	0.578	0.234(43)	1.039(18)	0.176(51)	1.861(39)	0.20(14)	0.40*	0.2916(78)	0.031(53)	1.191(16)	0.389(40)
4	0.542	0.299(42)	1.044(16)	0.185(16)	1.861(34)	0.220(99)	0.40*	0.2955(67)	0.073(49)	1.185(16)	0.382(36)
5	0.569	0.232(17)	1.050(13)	0.199(17)	1.861(32)	0.115(76)	0.25(40)	0.298(95)	0.000(40)	1.185(12)	0.421(13)
7	0.552	0.227(18)	1.043(28)	0.240(48)	1.851(62)	0.116(96)	0.40(58)	0.299(14)	0.000(52)	1.174(36)	0.439(17)
9	0.549	0.2311(99)	1.043(31)	0.217(25)	1.853(76)	0.132(70)	0.62(42)	0.295(13)	0.001(41)	1.180(31)	0.425(11)
12.5	0.556	0.228(10)	1.0412(69)	0.069(15)	1.851(17)	0.432(26)	0.40(15)	0.2985(56)	0.000(35)	1.1758(90)	0.4275(99)
31.5	0.658	0.232(12)	1.0369(64)	0.211(16)	1.860(19)	0.130(22)	0.63(13)	0.2945(59)	0.000(35)	1.1754(90)	0.427(10)
53.5	0.887	0.2392(71)	1.0391(54)	0.208(12)	1.854(15)	0.150(17)	0.623(99)	0.2948(36)	0.000(27)	1.1731(75)	0.4102(90)

Notes: The uncertainty notation is such that, for example, 0.175(61) is equivalent to 0.175±0.061.
And uncertainties are in two sigma range standard deviation (2σ)
Time: Mössbauer spectra collection time in hours; χ^2: Chi square;
L HWHM: Lorentzian half width at half maximum in mm/s; CS: Center Shift in mm/s;
δ_{CS}: Gaussian width of CS in mm/s; QS: Quadrupole splitting in mm/s;
δ_{QS}: Gaussian width of QS in mm/s; ρ: correlation of δ_{CS} and δ_{QS};
∗: for the VF3 Mössbauer spectra collected at 12 mm/s scale with 3 and 4 hours, ρ was fixed at 0.40.

Supplement Table 1 continue:

Time	Background	RA (Fe^{2+})	AA (Fe^{2+})	RA (Fe^{3+})	AA (Fe^{3+})
6 mm/s scale					
0.188	25358(15)	32.6(20)	866(66)	67.4(20)	1788(90)
0.5	76996(47)	27.1(36)	2150(340)	72.90(36)	5790(530)
1	149297(42)	28.3(13)	4170(220)	71.7(13)	10550(410)
2	287826(45)	30.2(13)	8660(480)	69.8(13)	19990(550)
3	449139(77)	29.4(11)	13110(640)	70.6(11)	31460(700)
4	599028(98)	29.6(11)	17070(820)	70.4(11)	40600(850)
5.5	814281(79)	29.38(75)	23790(790)	70.62(75)	57180(800)
7	1045800(130)	27.87(93)	28300(1000)	72.13(93)	73400(2100)
9	1361180(160)	28.74(86)	38900(1400)	71.26(86)	96600(2100)
12	1808490(160)	29.03(52)	52800(1100)	70.97(52)	129000(1700)
25	3757280(260)	29.05(43)	107600(2000)	70.95(43)	263000(2500)
12 mm/s scale					
1	160183(27)	29.8(31)	4700(640)	70.2(31)	11090(700)
2	310318(58)	31.2(33)	9100(1200)	68.8(33)	20100(1400)
3	448449(83)	31.1(16)	14410(810)	68.9(16)	31900(1400)
4	570725(97)	30.6(14)	17910(960)	69.4(14)	40500(1700)
5	736184(80)	30.71(97)	23270(920)	69.29(97)	52500(1200)
7	1073312(96)	30.4(10)	30800(1300)	69.6(10)	70600(1500)
9	1340091(92)	30.7(10)	39000(1700)	69.3(10)	88100(1800)
12.5	1903489(83)	30.79(60)	55200(1400)	69.21(60)	124000(1600)
31.5	4773590(190)	30.82(64)	139800(3600)	69.18(64)	313900(4500)
53.5	8072960(210)	31.27(48)	240900(4900)	68.73(48)	529400(5100)

Notes: The uncertainty notation is such that, for example, 25358(15) is equivalent to 25358±15.
And uncertainties are in two sigma range standard deviation (2σ)
T: Temperature in K; Background: in counts;
AA: Absorption area in counts·mm/s; RA: Relative sub-spectra areas;

Supplement Table 2: Mössbauer fitting parameters

T	χ^2	L HWHM	CS (Fe^{2+})	δ_{CS} (Fe^{2+})	QS (Fe^{2+})	δ_{QS} (Fe^{2+})	ρ (Fe^{2+})	CS (Fe^{3+})	δ_{CS} (Fe^{3+})	QS (Fe^{3+})	δ_{QS} (Fe^{3+})
M544											
47	0.783	0.204(15)	1.223(21)	0.169(20)	2.239(48)	0.357(39)	0.41(17)	0.439(21)	0.105(16)	1.213(37)	0.384(18)
57	0.777	0.211(18)	1.228(20)	0.210(20)	2.230(44)	0.262(52)	0.40(17)	0.438(20)	0.076(24)	1.215(33)	0.386(19)
67	0.822	0.222(19)	1.232(12)	0.184(13)	2.249(33)	0.317(35)	0.40(11)	0.439(11)	0.115(21)	1.229(16)	0.398(24)
97	0.911	0.201(21)	1.221(10)	0.144(17)	2.210(20)	0.422(26)	0.41(10)	0.425(10)	0.106(18)	1.227(16)	0.398(19)
117	0.711	0.202(21)	1.206(12)	0.231(18)	2.213(24)	0.218(34)	0.41(17)	0.428(13)	0.110(22)	1.195(26)	0.393(25)
147	0.67	0.211(20)	1.179(27)	0.228(21)	2.175(62)	0.277(41)	0.40(22)	0.414(23)	0.055(44)	1.189(43)	0.421(23)
177	0.742	0.200(23)	1.180(20)	0.163(20)	2.138(39)	0.441(32)	0.40(15)	0.394(13)	0.097(23)	1.203(27)	0.402(22)
212	0.731	0.256(28)	1.150(21)	0.275(23)	2.114(81)	0.220(89)	0.40(27)	0.385(28)	0.128(39)	1.195(39)	0.491(40)
293	0.762	0.186(10)	1.080(11)	0.272(10)	2.013(34)	0.156(27)	0.38(13)	0.334(12)	0.108(15)	1.166(17)	0.494(13)
VF3											
47	0.541	0.262(14)	1.150(29)	0.219(25)	2.034(56)	0.242(73)	0.44(29)	0.398(18)	0.090(17)	1.257(33)	0.346(18)
57	0.608	0.225(16)	1.160(14)	0.230(15)	2.023(41)	0.246(42)	0.40(18)	0.390(11)	0.090(26)	1.250(12)	0.386(24)
67	0.464	0.239(15)	1.168(13)	0.186(17)	2.050(27)	0.374(34)	0.45(15)	0.3945(78)	0.085(17)	1.237(14)	0.412(14)
77	0.535	0.260(18)	1.156(16)	0.157(18)	2.019(28)	0.323(35)	0.44(13)	0.394(12)	0.089(22)	1.225(12)	0.328(24)
97	0.605	0.234(16)	1.144(28)	0.241(21)	2.018(51)	0.179(64)	0.40(22)	0.396(14)	0.001(45)	1.227(27)	0.426(14)
117	0.664	0.222(21)	1.131(13)	0.236(20)	1.998(33)	0.155(46)	0.41(25)	0.384(10)	0.062(31)	1.217(16)	0.410(19)
147	0.698	0.245(24)	1.120(16)	0.194(19)	1.961(36)	0.265(46)	0.41(18)	0.367(10)	0.086(28)	1.209(15)	0.340(28)
177	0.701	0.250*	1.096(26)	0.171(23)	1.927(42)	0.349(44)	0.5(19)	0.352(11)	0.061(24)	1.209(19)	0.358(17)
212	0.625	0.256(22)	1.080(24)	0.197(42)	1.894(42)	0.189(97)	0.49(41)	0.341(15)	0.063(38)	1.198(24)	0.339(30)
293	0.556	0.232(19)	0.994(16)	0.225(23)	1.754(37)	0.155(60)	0.49(28)	0.283(12)	0.087(23)	1.148(18)	0.405(20)

Notes: The uncertainty notation is such that, for example, 0.204(15) is equivalent to 0.204±0.015.
And uncertainties are in two sigma range standard deviation (2σ)
T: Temperature in K; χ^2: Chi square;
L HWHM: Lorentzian half width at half maximum in mm/s; CS: Center Shift in mm/s;
δ_{CS}: Gaussian width of CS in mm/s; QS: Quadrupole splitting in mm/s;
δ_{QS}: Gaussian width of QS in mm/s; ρ: correlation of δ_{CS} and δ_{QS};
∗: for the VF3 Mössbauer spectra collected at 177 K, the L HWHM was fixed at 0.250 mm/s.

Supplement Table 3: Absorption areas, and calculated recoilless fractions for Mössbauer spectra

T (K)	Background	RA (Fe^{2+})	AA (Fe^{2+})	RA (Fe^{3+})	AA (Fe^{3+})
			M544		
47	1303330(150)	43.3(11)	89500(3400)	56.7(11)	117000(3100)
57	1318270(110)	44.0(12)	92200(3700)	56.0(12)	117100(3000)
67	1469490(130)	41.5(11)	97000(3600)	58.5(11)	137000(3200)
97	2576030(190)	42.62(75)	168700(3700)	57.38(75)	227100(4800)
117	1135410(110)	42.83(91)	73100(2000)	57.17(91)	97500(2400)
147	1471380(130)	43.43(78)	93900(2300)	56.57(78)	122300(2500)
177	1230347(100)	42.9(12)	74600(2800)	57.1(12)	99300(2900)
212	1218430(150)	41.2(21)	69900(5200)	58.8(21)	99600(4500)
293	5764070(260)	39.99(83)	278600(7800)	60.01(83)	418000(8300)
			VF3		
47	541460(80)	36.9(14)	44900(2400)	63.1(14)	76800(1800)
57	532837(83)	35.8(12)	42500(2000)	64.2(12)	76200(1800)
67	434524(60)	34.7(10)	33900(1400)	65.3(10)	63600(1300)
77	511304(75)	34.05(88)	38800(1300)	65.95(88)	75100(1500)
97	533547(57)	34.31(47)	40160(710)	65.69(47)	76890(810)
117	506043(90)	33.62(96)	35600(1300)	66.38(96)	70200(1500)
147	472994(88)	34.0(16)	32900(2100)	66.0(16)	63800(2000)
177	491374(68)	34.5(16)	33800(2200)	65.5(16)	59800(2200)
212	470929(62)	32.9(12)	29400(1300)	67.1(12)	60100(1800)
293	931880(88)	31.5(14)	49000(2800)	68.5(14)	106400(2800)

Notes: The uncertainty notation is such that, for example, 1303330(150) is equivalent to 1303330±150.
And uncertainties are in two sigma range standard deviation (2σ)
T: Temperature in K; Background: in counts;
AA: Absorption area in counts·mm/s; RA: Relative sub-spectra areas;

Supplement Table 3 continue: Absorption areas, and calculated recoilless fractions for Mössbauer spectra

T (K)	$f^{RM}(Fe^{2+})$	$f^{RM}(Fe^{3+})$	C^{RM}	$f^{CSM}(Fe^{2+})$	f^{CSM}(Fe3+)	C^{CSM}
			M544			
47	0.880(14)	0.905(12)	1.028(21)	0.880(14)	0.931(17)	1.058(26)
57	0.873(16)	0.901(12)	1.032(23)	0.873(16)	0.929(18)	1.064(28)
67	0.865(18)	0.896(14)	1.036(26)	0.865(18)	0.927(20)	1.072(32)
97	0.838(24)	0.879(18)	1.049(36)	0.838(24)	0.919(25)	1.097(43)
117	0.818(28)	0.866(20)	1.059(44)	0.818(27)	0.913(29)	1.115(51)
147	0.788(34)	0.846(26)	1.074(55)	0.788(33)	0.902(35)	1.145(65)
177	0.757(38)	0.824(30)	1.089(67)	0.757(38)	0.890(41)	1.176(80)
212	0.722(44)	0.799(34)	1.107(82)	0.722(43)	0.876(47)	1.213(98)
293	0.645(54)	0.742(44)	1.151(118)	0.645(54)	0.842(63)	1.305(146)
			VF3			
47	0.860(17)	0.898(10)	1.044(23)	0.818(160)	0.925(14)	1.131(224)
57	0.850(19)	0.893(11)	1.051(27)	0.802(180)	0.923(15)	1.150(259)
67	0.840(21)	0.888(12)	1.057(31)	0.786(196)	0.920(16)	1.170(293)
77	0.828(24)	0.882(14)	1.065(35)	0.769(210)	0.917(17)	1.192(327)
97	0.804(29)	0.869(16)	1.080(44)	0.736(235)	0.910(20)	1.238(399)
117	0.779(34)	0.854(19)	1.097(54)	0.702(257)	0.903(24)	1.285(468)
147	0.740(40)	0.831(23)	1.122(69)	0.654(277)	0.890(29)	1.360(577)
177	0.703(46)	0.807(27)	1.148(85)	0.609(292)	0.876(34)	1.437(691)
212	0.660(52)	0.779(31)	1.180(104)	0.561(304)	0.859(40)	1.531(831)
293	0.570(62)	0.716(39)	1.256(153)	0.465(312)	0.820(52)	1.762(1188)

Notes: The uncertainty notation is such that, for example, 0.880(14) is equivalent to 0.880±0.014.
And uncertainties are in two sigma range standard deviation (2σ)
T: Temperature in K;
f: recoilless fraction; C: $f(Fe^{3+})/f(Fe^{2+})$;
RM: relative method; CSM: center shift method;

Supplement Table 4: Mossbauer parameters of VF3 for fit with nanophase oxide

	nanophase	Fe_3O_4	$MgFe_2O_4$
	$\chi 2$	0.557	0.554
	L HWHM	0.230(22)	0.224(28)
paramagnetic sites	CS (Fe^{2+})	0.998(26)	1.006(38)
	δ_{CS} (Fe^{2+})	0.232(35)	0.245(53)
	QS (Fe^{2+})	1.775(58)	1.736(11)
	δ_{QS} (Fe^{2+})	0.15(10)	0.15(21)
	P (Fe^{2+})	0.42(50)	0.26(56)
	RA (Fe^{2+})	31.6(11)	31.6(17)
	CS (Fe^{3+})	0.283(16)	0.285(18)
	δ_{CS} (Fe^{3+})	0.089(30)	0.094(32)
	QS (Fe^{3+})	1.151(30)	1.157(49)
	δ_{QS} (Fe^{3+})	0.406(26)	0.411(31)
	RA (Fe^{3+})	68.0(11)	67.2(17)
magnetic site	RA	0.438(12)	1.182(40)

Notes: The uncertainty notation is such that, for example, 0.998(26) is equivalent to 0.998±0.026.

And uncertainties are in two sigma range standard deviation (2σ)

L HWHM: Lorentzian half width at half maximum in mm/s;

CS: Center Shift in mm/s;

δ_{CS}:Gaussion width of CS in mm/s;

QS:Quadrupole splitting in mm/s;

δ_{QS}: Gaussion width of CS in mm/s;

ρ: corellation of δ_{CS} and δ_{QS};

RA:Relative sub-spectral areas