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Precision and interlaboratory reproducibility of measurements of the Mössbauer effect in minerals

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Abstract

Although the technique of Mössbauer spectroscopy is now 25-years old and widely used, little empirical work has been done to determine its accuracy in measurements on minerals. To solve this deficiency, two mineral standards (a grunerite and an almandine/andradite garnet mix) have been selected. Precision of the technique was measured through five different sets of experiments seeking to analyze the reproducibility of measurements on a single sample mount, on several identical mounts of the same sample, and on a set of mounts with different sample concentrations, run times, and background counts. The two mineral standards were analyzed by other scientists at seven different laboratories; their data were also fit by the MIT program. The standard deviation of multiple measurements on the MIT apparatus is better than 0.016 mm/sec for isomer shift, 0.060 mm/sec or better for quadrupole splitting, and 1.02% on individual peak area data. The standard deviation of interlaboratory measurements on the same minerals is slightly better because only ideal run conditions were used: 0.006 mm/sec for isomer shift, 0.023 mm/sec for quadrupole splitting, and 1.44% on individual peak area data. Probable errors on different aliquots of the same sample are approximately ±0.02 mm/sec for isomer shift and quadrupole splittings, and ±1.5% on area data for well-resolved peaks.

Introduction

Since 1967, over 814 papers have been published in the geological literature which apply the Mössbauer effect in ^{57}Fe to interpretations of mineral crystal chemistry (Fig. 1). Numerous other papers have made reference to Mössbauer measurements for Fe^{3+}/Fe^{2+} determinations or structural Fe site occupancy information, to the extent that the technique has become one of the many commonly used analytical tools available to geochemists and mineralogists.

However, the technique of Mössbauer spectroscopy is still relatively young; Rudolph Mössbauer published his first papers only 26-years ago (Mössbauer, 1958). In the first 10–15 years after Mössbauer's discovery, Mössbauer spectrometers were literally home-built from scratch in chemistry, physics, and mineralogy laboratories around the world, with a wide array of geometries, standards, and electronic configurations. Because the experimental apparatus and methodology for Mössbauer work were customized for each lab, there was little consistency in the type of source used or the method by which spectral data were processed. By the 1970's, commercial Mössbauer apparatus became widely available, but many workers continued to maintain and update their original equipment. Today each Mössbauer laboratory has its own distinctive experimental apparatus, computing facility, and philosophy for recording, measuring, and reporting its results (for example, Mitrofanov et al., 1977; Graham et al., 1977; LeFever, 1979; and Fultz and Morris, 1978).

Over the years there have been some attempts to standardize the type of calibration procedures (Herber, 1971) and the method of reporting results (Zuckerman et al., 1972); these have been received with varying degrees of success. The predominant trend has been for each lab (and subsequent generations of graduate students and colleagues) to develop its own philosophy on optimization of experimental technique and curve-fitting. A few attempts at interlaboratory standardization (e.g., Minai and Tominaga, 1982) or comparison of Fe^{3+}/Fe^{2+} ratios against wet chemistry (Whipple, 1973 and 1974; Bancroft et al., 1977) have given inconsistent results, although agreement between different Mössbauer labs is consistently better than between Mössbauer and wet chemical labs.

Fortunately, several workers in the field have devoted great effort toward a statistical evaluation of the technique. The literature prescribes the optimal sample concentration and thickness (Hafemeister and Shera, 1966; Ure and Flinn, 1971; Shenoy *et* al., 1974), the relative merits of fitting techniques (Lin and Preston, 1974),

Table 3. 24 Hours, Counts and Concentration Varying

Number hours	Baseline counts per channel	Total Fe conc. (mg/cm^2)	Peak Positions (mm/sec)				Widths at Half Peak Height (mm/sec)			
			1	2	3	4	1	2	3	4
24	1661501	40	-0.302	0.308	1.928	2.603	0.332	0.416	0.277	0.313
24.3	1775161	30	-0.310	0.300	1.933	2.619	0.346	0.428	0.297	0.327
24.75	2661504	20	-0.302	0.308	1.928	2.603	0.332	0.416	0.277	0.313
26.5	2716282	15	-0.279	0.343	1.931	2.605	0.312	0.402	0.264	0.294
26	3728779	7	-0.280	0.349	1.922	2.602	0.287	0.382	0.243	0.279
26.25	3785614	5	-0.273	0.353	1.938	2.607	0.294	0.394	0.259	0.280
26	4208489	3.5	-0.275	0.352	1.928	2.606	0.292	0.381	0.244	0.285
25.5	4529096	2.5	-0.280	0.355	1.927	2.605	0.292	0.384	0.254	0.291
26	5369985	1	-0.278	0.358	1.933	2.609	0.278	0.405	0.282	0.285
Variance			0.0002	0.0006	0.0000	0.0000	0.0006	0.0003	0.0003	0.0003
Standard Deviation			0.0140	0.0236	0.0046	0.0053	0.0241	0.0169	0.0183	0.0172
Mean			-0.2866	0.3362	1.9298	2.6066	0.3072	0.4009	0.2663	0.2963

Table 3. 24 Hours, Counts and Concentration Varying (CONTINUED)

% Areas				Isomer Shift (mm/sec)		Quadrupole Splitting (mm/sec)		% Transmitted	χ^2	Misfit
1	2	3	4	1-4	2-3	1-4	2-3			
32.27	24.47	14.46	28.80	1.151	1.118	2.905	1.620	93.91	759	0.00101
33.24	23.29	14.24	29.23	1.155	1.117	2.929	1.633	92.29	989	0.00111
32.26	24.48	14.46	28.80	1.151	1.118	2.905	1.620	90.96	471	-0.00026
35.34	24.19	12.89	27.58	1.163	1.137	2.884	1.588	-	875	0.00068
35.30	23.93	12.21	28.56	1.161	1.136	2.882	1.573	94.72	780	0.00090
35.00	24.08	12.68	28.24	1.167	1.146	2.880	1.585	97.02	553	0.00047
35.01	23.45	11.99	29.56	1.166	1.140	2.881	1.576	97.53	472	-0.00054
36.21	24.07	12.12	27.59	1.163	1.141	2.885	1.572	97.96	402	-0.00272
32.46	26.94	12.25	28.35	1.166	1.146	2.887	1.575	99.20	340	-0.02199
2.4034	1.1274	1.0439	0.4500	0.0000	0.0001	0.0003	0.0006			
1.5503	1.0618	1.0217	0.6708	0.0064	0.0122	0.0165	0.0240			
34.1211	24.3222	13.0111	28.5233	1.1603	1.1332	2.8931	1.5936			

Table 4. 1 Million Counts, Time and Concentration Varying

Number hours	Baseline counts per channel	Total Fe conc. (mg/cm^2)	Peak Positions (mm/sec)				Widths at Half Peak Height (mm/sec)			
			1	2	3	4	1	2	3	4
18	1145444	40	-0.251	0.315	1.849	2.556	0.360	0.325	0.305	0.341
16.5	1198750	30	-0.234	0.335	1.832	2.529	0.335	0.313	0.266	0.327
10.5	1101536	20	-0.242	0.337	1.863	2.550	0.325	0.310	0.259	0.308
9.75	1116850	15	-0.237	0.339	1.848	2.536	0.309	0.295	0.258	0.294
8.5	1072936	10	-0.229	0.346	1.847	2.530	0.308	0.290	0.257	0.295
7.5	1073238	7	-0.180	0.385	1.875	2.566	0.316	0.320	0.256	0.313
6.67	1042950	5	-0.242	0.321	1.856	2.547	0.282	0.311	0.250	0.304
6.5	1036633	3.5	-0.223	0.340	1.840	2.529	0.279	0.332	0.257	0.295
7.0	1123864	2.5	-0.232	0.330	1.842	2.533	0.298	0.311	0.254	0.295
6.8	1104674	1	-0.267	0.291	1.878	2.581	0.248	0.309	0.277	0.291
Variance			0.0005	0.0006	0.0002	0.0003	0.0010	0.0002	0.0003	0.0003
Standard Deviation			0.0226	0.0241	0.0150	0.0177	0.0316	0.0126	0.0162	0.0165
Mean			0.2337	0.3339	1.8530	2.5457	0.3060	0.3116	0.2639	0.3063

Table 4. 1 Million Counts, Time and Concentration Varying (CONTINUED)

% Areas				Isomer Shift (mm/sec)		Quadrupole Splitting (mm/sec)		% Transmitted	χ^2	Misfit
1	2	3	4	1-4	2-3	1-4	2-3			
34.83	19.15	15.39	30.63	1.153	1.082	2.807	1.534	93.23	684	0.00084
35.70	18.60	14.13	31.56	1.148	1.084	2.763	1.497	91.53	785	0.00086
36.62	18.63	13.77	30.98	1.154	1.100	2.792	1.526	90.59	705	0.00058
38.22	18.36	13.72	29.70	1.150	1.094	2.773	1.509	91.91	632	0.00054
39.26	17.86	13.11	29.76	1.151	1.097	2.759	1.501	93.80	545	0.00032
38.18	18.44	12.61	30.76	1.193	1.130	2.746	1.490	94.87	517	0.00008
36.47	18.85	13.08	31.60	1.153	1.089	2.789	1.535	96.57	406	-0.00332
35.55	20.43	12.59	31.43	1.153	1.090	2.752	1.500	97.31	416	-0.00592
38.57	18.66	12.63	30.14	1.151	1.086	2.765	1.512	97.38	372	-0.00737
31.49	21.28	13.69	33.54	1.157	1.085	2.848	1.587	98.83	403	-0.04891
5.2366	1.0796	0.7582	1.2744	0.0002	0.0002	0.0009	0.0008			
2.2884	1.0390	0.8708	1.1289	0.0131	0.0140	0.0308	0.0285			
36.4890	19.0260	13.4720	31.0100	1.1563	1.0937	2.7794	1.5191			

Table 5. 7mg Fe/cm^2 with Time Varying

Number hours	Baseline counts per channel	Total Fe conc. (mg/cm^2)	Peak Positions (mm/sec)				Widths at Half Peak Height (mm/sec)			
			1	2	3	4	1	2	3	4
3	293152	7	-0.265	0.283	1.836	2.553	0.303	0.277	0.306	0.295
7	663667	7	-0.253	0.289	1.820	2.532	0.301	0.285	0.285	0.301
10	897661	7	-0.243	0.297	1.840	2.553	0.294	0.275	0.275	0.294
12	1075768	7	-0.244	0.298	1.845	2.545	0.296	0.267	0.267	0.296
15	1540192	7	-0.262	0.286	1.846	2.554	0.293	0.271	0.271	0.293
18	1820108	7	-0.254	0.295	1.842	2.553	0.296	0.279	0.279	0.296
21	1986456	7	-0.268	0.281	1.840	2.545	0.298	0.277	0.277	0.298
24	2455543	7	-0.261	0.281	1.837	2.547	0.301	0.278	0.278	0.301
28	2749269	7	-0.263	0.285	1.838	2.555	0.299	0.276	0.276	0.299
33	3356315	7	-0.264	0.281	1.838	2.549	0.295	0.280	0.271	0.307
39	3888139	7	-0.255	0.287	1.831	2.539	0.296	0.272	0.272	0.296
48	4627081	7	-0.257	0.285	1.835	2.544	0.296	0.279	0.279	0.296
60	5971105	7	-0.257	0.292	1.830	2.535	0.298	0.292	0.276	0.307
Variance			0.0001	0.0000	0.0000	0.0001	0.0000	0.0000	0.0001	0.0000
Standard Deviation			0.0077	0.0061	0.0069	0.0073	0.0030	0.0063	0.0096	0.0045
Mean			-0.2574	0.2877	1.8368	2.5465	0.2974	0.2775	0.2778	0.2984

Table 5. 7mg Fe/cm^2 with Time Varying (CONTINUED)

% Areas				Isomer Shift (mm/sec)		Quadrupole Splitting (mm/sec)		% Transmitted	χ^2	Misfit
1	2	3	4	1-4	2-3	1-4	2-3			
38.08	16.71	14.36	30.85	1.144	1.060	2.818	1.553	92.72	495	-0.00034
37.20	17.27	14.00	31.53	1.140	1.055	2.785	1.531	93.46	503	-0.00008
37.73	17.08	13.82	31.37	1.155	1.069	2.796	1.544	92.92	542	0.00023
37.96	16.46	13.84	31.75	1.150	1.071	2.789	1.547	92.76	545	0.00022
37.97	16.76	13.89	31.38	1.146	1.066	2.816	1.561	93.40	592	0.00043
37.36	16.90	14.08	31.66	1.150	1.068	2.807	1.547	93.50	570	0.00026
37.58	16.93	13.83	31.66	1.139	1.061	2.813	1.559	93.45	553	0.00017
37.79	16.59	13.86	31.76	1.143	1.059	2.808	1.556	93.60	707	0.00064
37.66	16.69	14.02	31.63	1.146	1.061	2.819	1.553	93.47	640	0.00036
37.41	16.77	13.75	32.08	1.143	1.060	2.813	1.557	93.53	745	0.00055
37.58	16.76	14.00	31.65	1.142	1.059	2.794	1.544	93.51	807	0.00060
37.31	16.89	14.24	31.56	1.144	1.060	2.801	1.550	93.56	767	0.00043
37.07	17.36	13.77	31.80	1.139	1.061	2.792	1.538	93.55	947	0.00057
0.0974	0.0655	0.0338	0.0832	0.0000	0.0000	0.0001	0.0001			
0.3120	0.2559	0.1838	0.2885	0.0047	0.0047	0.0117	0.0087			
37.5923	16.8592	13.9585	31.5908	1.1447	1.0623	2.8039	1.5492			

Table 6. Repeated Runs of Same Sample, Same Mount

Number hours	Baseline counts per channel	Total Fe conc. (mg/cm^2)	Peak Positions (mm/sec)				Widths at Half Peak Height (mm/sec)			
			1	2	3	4	1	2	3	4
12	1155249	7	-0.269	0.279	1.840	2.544	0.307	0.301	0.295	0.316
12	1145712	7	-0.270	0.276	1.853	2.551	0.295	0.289	0.277	0.305
12	1090119	7	-0.266	0.278	1.835	2.534	0.301	0.295	0.284	0.312
12	1075779	7	-0.244	0.297	1.844	2.544	0.289	0.278	0.255	0.304
12	1138450	7	-0.266	0.278	1.828	2.531	0.292	0.280	0.267	0.307
12	1091482	7	-0.264	0.285	1.828	2.538	0.293	0.286	0.273	0.302
12	1068833	7	-0.267	0.277	1.827	2.537	0.284	0.294	0.272	0.302
12	960189	7	-0.264	0.278	1.832	2.544	0.292	0.323	0.273	0.317
12	949604	7	-0.262	0.286	1.837	2.547	0.283	0.320	0.263	0.318
12	985877	7	-0.259	0.287	1.833	2.549	0.289	0.293	0.279	0.303
Variance			0.0001	0.0000	0.0001	0.0000	0.0001	0.0002	0.0001	0.0000
Standard Deviation			0.0074	0.0066	0.0082	0.0065	0.0073	0.0152	0.0111	0.0065
Mean			-0.2631	0.2821	1.8357	2.5419	0.2925	0.2959	0.2738	0.3086

Table 6. Repeated Runs of Same Sample, Same Mount (CONTINUED)

% Areas				Isomer Shift (mm/sec)		Quadrupole Splitting (mm/sec)		% Transmitted	χ^2	Misfit
1	2	3	4	1-4	2-3	1-4	2-3			
36.92	17.43	13.90	31.74	1.138	1.060	2.813	1.561	88.71	551	0.00028
37.18	17.33	13.93	31.55	1.141	1.065	2.821	1.577	91.78	543	0.00023
37.15	17.18	13.82	31.86	1.134	1.057	2.800	1.557	93.39	505	-0.00003
37.34	16.95	13.40	32.31	1.150	1.071	2.788	1.547	92.76	528	0.00011
37.10	17.09	13.63	32.17	1.133	1.053	2.797	1.550	93.36	511	0.00001
37.03	17.37	13.56	32.03	1.137	1.057	2.802	1.543	90.99	491	0.00013
36.28	18.04	13.49	32.18	1.135	1.052	2.804	1.550	93.46	531	0.00015
35.36	19.07	13.34	32.22	1.140	1.055	2.808	1.554	93.85	580	0.00061
34.99	19.44	13.15	32.41	1.143	1.062	2.809	1.551	93.81	538	0.00025
36.00	18.23	13.77	32.00	1.145	1.060	2.808	1.546	93.57	592	0.00064
0.6972	0.7440	0.0668	0.0713	0.0000	0.0000	0.0001	0.0001			
0.8350	0.8626	0.2584	0.2670	0.0053	0.0057	0.0090	0.0098			
36.5350	17.8130	13.5990	32.0470	1.1396	1.0592	2.8050	1.5536			

Table 7. 10 Aliquots of the Same Sample; Identical Run Conditions

Number hours	Baseline counts per channel	Total Fe conc. (mg/cm^2)	Peak Positions (mm/sec)				Widths at Half Peak Height (mm/sec)			
			1	2	3	4	1	2	3	4
12	1063038	7	-0.256	0.296	1.885	2.615	0.303	0.307	0.294	0.328
12	1054196	7	-0.250	0.289	1.855	2.573	0.228	0.429	0.449	0.247
12	962361	7	-0.249	0.288	1.846	2.569	0.296	0.273	0.283	0.313
12	996971	7	-0.269	0.284	1.878	2.616	0.308	0.296	0.299	0.328
12	980563	7	-0.270	0.279	1.864	2.602	0.308	0.292	0.298	0.334
12	756641	7	-0.285	0.274	1.911	2.651	0.301	0.308	0.298	0.332
12	1046152	7	-0.270	0.278	1.892	2.639	0.309	0.315	0.306	0.338
12	859754	7	-0.257	0.300	1.906	2.650	0.321	0.363	0.328	0.383
12	890532	7	-0.225	0.313	1.811	2.522	0.306	0.297	0.283	0.351
12	1075779	7	-0.244	0.297	1.844	2.544	0.289	0.278	0.255	0.304
Variance			0.0003	0.0001	0.0010	0.0020	0.0015	0.0022	0.0032	0.0012
Standard Deviation			0.0169	0.0119	0.0312	0.0447	0.0391	0.0468	0.0563	0.0350
Mean			-0.2575	0.2898	1.8692	2.5981	0.3050	0.3158	0.3193	0.3258

Table 7. 10 Aliquots of the Same Sample; Identical Run Conditions (CONTINUED)

% Areas				Isomer Shift (mm/sec)		Quadrupole Splitting (mm/sec)		% Transmitted	χ^2	Misfit
1	2	3	4	1-4	2-3	1-4	2-3			
34.88	17.46	14.43	33.22	1.180	1.091	2.871	1.589	94.42	506	-0.00003
35.39	16.06	13.70	34.84	1.162	1.072	2.823	1.566	94.68	641	0.00133
36.26	15.99	14.90	32.85	1.160	1.067	2.818	1.558	93.93	474	-0.00033
35.15	16.40	14.67	33.78	1.174	1.081	2.885	1.594	94.63	536	0.00027
34.48	16.91	14.98	33.63	1.166	1.072	2.872	1.585	95.47	495	-0.00022
34.40	18.02	14.15	33.42	1.183	1.093	2.936	1.637	94.61	430	-0.00112
33.49	17.58	14.90	34.03	1.185	1.085	2.909	1.614	95.42	517	0.00010
32.95	19.18	14.00	33.87	1.197	1.103	2.907	1.606	96.10	501	-0.00019
34.69	17.07	13.34	34.90	1.149	1.062	2.747	1.498	95.21	507	-0.00004
37.34	16.95	13.40	32.31	1.150	1.071	2.788	1.547	92.76	528	0.00011
1.5963	0.9285	0.3903	0.6500	0.0003	0.0002	0.0036	0.0015			
1.2634	0.9636	0.6247	0.8062	0.0158	0.0131	0.0598	0.0393			
34.9030	17.1620	14.2470	33.6850	1.1706	1.0797	2.8556	1.5794			

Table 12. Mossbauer Data on Sample R (All unconstrained Fits)

Lab No.	Temp. (°K)	Baseline counts per channel	Peak Positions (mm/sec) 1	2	3	4	Widths at Half Peak Height (mm/sec) 1	2	3	4
1	300	374600	-0.233	0.308	1.853	2.564	0.281	0.285	0.273	0.299
2	295	3847859	-0.236	0.289	1.841	2.551	0.265	0.237	0.247	0.284
2	295	3491639	-0.240	0.297	1.836	2.552	0.261	0.245	0.253	0.283
3	295	1365000	-0.239	0.303	1.848	2.559	0.266	0.269	0.251	0.291
4	295	1141242	-0.248	0.295	1.833	2.549	0.296	0.322	0.325	0.318
5	297	4072459(402)	-0.205(10)	0.321(10)	1.836(8)	2.530(5)	0.299(5)	0.281(5)	0.272(5)	0.321(5)
6	298	1075779	-0.244	0.297	1.844	2.544	0.289	0.278	0.255	0.304
7	298	746459	-0.243	0.302	1.836	2.545	0.304	0.276	0.228	0.286
7	298	2350842	-0.241	0.302	1.846	2.558	0.300	0.267	0.262	0.275
7	298	362213	-0.241	0.301	1.835	2.545	0.305	0.276	0.231	0.280
7	298	1160999	-0.240	0.302	1.844	2.560	0.305	0.269	0.258	0.272
Variance*			0.0002	0.0001	0.0000	0.0001	0.0003	0.0006	0.0008	0.0003
Standard Deviation			0.0142	0.0096	0.0070	0.0109	0.0160	0.0241	0.0276	0.0164
Mean			-0.2354	0.3027	1.8418	2.5499	0.2854	0.2783	0.2674	0.2992
2	77	4040139	-0.287	0.414	1.962	2.833	0.280	0.268	0.249	0.305

* All statistics were computed using one value (averaged, if necessary) from each lab.

Table 12. (continued)

Lab No.	% Area (or X_{eff})				Isomer Shift (mm/sec)		Quadrupole Splitting (mm/sec)		Misfit	χ^2	Degrees of Freedom
	1	2	3	4	1-4	2-3	1-4	2-3			
1	35.65	16.81	13.31	34.22	1.166	1.080	2.797	1.545	0.014	523.5	487
2	35.4	13.5	14.0	37.1	1.158(1)	1.069(1)	2.787(1)	1.544(2)	0.086±0.006	1336	487
2	37.6	15.2	12.6	34.5	1.156(1)	1.067(1)	2.792(1)	1.539(2)	0.048±0.007	751	487
3	34.7	15.8	13.5	36.0	1.158	1.074	2.798	1.545	0.00087	1008	497
4	34.24**	15.76**	15.76**	34.24**	1.151	1.064	2.796	1.537	-	979.5	-
5	35.1(4)	16.6(4)	13.3(4)	35.0(4)	1.163±.01	1.079±.015	2.735±.01	1.515±.01	0.100%	381	241
6	37.34	16.95	13.40	32.31	1.150	1.071	2.788	1.547	0.00011	528	509
7	30.75**	13.84**	13.84**	30.75**	1.151	1.069	2.787	1.534	-	243	237
7	33.00**	17.00**	17.00**	33.00**	1.159	1.074	2.799	1.544	-	289	237
7	34.81**	15.19**	15.19**	34.81**	1.152	1.068	2.786	1.534	-	520	493
7	33.45**	16.55**	16.55**	33.45**	1.160	1.073	2.800	1.542	-	456	493
	2.0819	0.8100	1.3132	1.8765	0.0000	0.0000	0.0005	0.0001			
	1.4429	0.9000	1.1459	1.3698	0.0058	0.0057	0.0225	0.0109			
	35.2186	15.9886	14.0314	34.3671	1.1573	1.0724	2.7852	1.5385			
2	37.1	17.8	12.4	32.8	1.273(1)	1.188(1)	3.120(2)	1.548(2)	0.084±0.009	969	487

** These workers gave their % areas in terms of area per doublet; these values were halved for purposes of this table (probably a poor assumption, but necessary to enable compilation of statistics).

Table 13. Mossbauer Data on Sample A (All unconstrained Fits)

Lab No.	Temp. (°K)	Baseline counts per channel	Peak Positions (mm/sec) 1	2	3	4	Widths at Half Peak Height (mm/sec) 1	2	3	4
1	300	454800	-0.470	0.137	0.686	3.061	0.292	0.276	0.282	0.263
2	293	4962630	-0.466	0.122	0.669	3.051	0.269	0.246	0.244	0.246
2	293	1519572	-0.468	0.123	0.674	3.052	0.280	0.235	0.266	0.250
3	295	1245000	-0.475	0.132	0.685	3.061	0.278	0.241	0.254	0.257
4	296	993278	-0.484	0.123	0.682	3.049	0.306	0.282	0.310	0.282
5	297	4266211(615)	-0.478(10)	0.123(10)	0.669(10)	3.043(5)	0.313(5)	0.289(5)	0.283(5)	0.284(5)
6	298	1805976	-0.473	0.126	0.685	3.056	0.349	0.330	0.367	0.334
7	298	2275546	-0.477	0.127	0.673	3.041	0.262	0.266	0.262	0.280
7	298	2900293	-0.476	0.126	0.677	3.044	0.262	0.266	0.269	0.280
Variance*			0.0000	0.0000	0.0001	0.0001	0.0009	0.0009	0.0016	0.0007
Standard Deviation			0.0055	0.0055	0.0071	0.0078	0.0293	0.0308	0.0399	0.0272
Mean			-0.4748	0.1271	0.6791	0.0520	0.2964	0.2749	0.2881	0.2791
2	77	3652275	-0.403	0.260	0.802	3.272	0.288	0.312	0.266	0.269

* All statistics were computed using one value (averaged, if necessary) from each lab.

Table 13. (continued)

Lab No.	% Area (or X_{eff})				Isomer Shift (mm/sec)		Quadrupole Splitting (mm/sec)		Misfit	χ^2	Degrees of Freedom
	1	2	3	4	1-4	2-3	1-4	2-3			
1	40.94	10.11	9.91	39.05	1.296	0.412	3.531	0.550	0.00465	512	487
2	41.7	8.8	8.8	40.7	1.293(1)	0.396(1)	3.517(1)	0.547(3)	0.013±0.004	586	487
2	42.8	8.1	9.2	39.9	1.292(1)	0.399(1)	3.520(1)	0.551(3)	0.017±0.004	633	487
3	42.7	7.6	7.9	41.9	1.291	0.407	3.536	0.553	0.00022	637	497
4	40.04**	9.96**	9.96**	40.04**	1.282	0.403	3.534	0.562	-	765	-
5	40.8(4)	10.7(6)	10.3(4)	38.2(4)	1.283±.01	0.396±.015	3.521±.01	0.546±.005	0.125%	705	241
6	41.17	8.65	9.64	40.53	1.291	0.405	3.528	0.559	0.0018	293	499
7	40.37**	9.64**	9.64**	40.37**	1.282	0.400	3.517	0.546	-	247	237
7	40.51**	9.49**	9.49**	40.51**	1.284	0.401	3.520	0.551	-	516	493
	0.9172	0.9751	0.6410	1.3817	0.0000	0.0000	0.0001	0.0000			
	0.9577	0.9875	0.8006	1.1755	0.0056	0.0056	0.0074	0.0059			
	41.1914	9.3750	9.4679	40.0657	1.2884	0.4030	3.5267	0.5525			
2	41.5	10.5	7.7	40.2	1.435(1)	0.531(2)	3.675(1)	0.542(4)	0.034±0.005	764	487

** These workers gave their % areas in terms of area per doublet; these values were halved for purposes of this table (probably a poor assumption, but necessary to enable compilation of statistics).