# Documentation Sheet Industry Reference Material – 241 Lot G Butyl Rubber (IRM prepared according to ASTM D4678)

#### Introduction

IRM 241 is the designation for a special butyl rubber used to evaluate the operation of Mooney Viscometers according to the procedures as described in D1646. See page 15 of this Documentation Sheet for additional background on this IRM.

#### Production of IRM241 Lot G

The ASTM D11.20 IRM Task Group requested that the production of IRM241 Lot G contain approximately 1200 bales. The producer made 1258 bales that were each placed in a box and numbered consecutively from 2000 to 3257. The production took place on one day and involved the use of two baling machines. The output of the two balers was intermingled and no attempt was made to isolate material by baler or to do any analysis by baler. The sampling plan during production was to take a sample from every third bale. This frequency of testing was selected to give a better estimate of lot uniformity. Of the 1258 bales/boxes, 22 were rejected during inspection, leaving a net production of 1236 bales/boxes.

During the production of IRM241 Lot G, the producer's sampling plan was to sample every third bale. Unfortunately, some of the samples were not collected so there is no data for that period. All the collected samples were tested. Besides testing and reporting the ML (1+8) at 125°C data, the producer also reported the ML (1+4) at 125°C data on every third bale. In addition, the producer agreed to test and report the ML (1+4) at 100°C and ML (1+8) at 100°C data on every sixth bale.

The producer's data on all the testing was evaluated to find a bale with properties at or near the mid-point values. The bale in box 3182 was selected because the test value for three of the four properties was at the mid-point value and the fourth only differed from the mid-point value by 0.1 Mooney units. This bale was used to provide the sample material for the Interlaboratory Test Program (ITP) to establish the Accepted Reference Values (AR or ARV).

#### **Properties for IRM241 Lot G**

The testing conducted as part of the IRM241 Lot G evaluation using ASTM D4678, generated an accepted reference value, AR-value, as defined below and 2 and 3 sigma limits on this value or on individual daily values as obtained by any laboratory using IRM241 Lot G. The 2 and 3 sigma limits apply to a single measurement of Mooney viscosity for the four conditions of test: ML (1+4) or ML (1+8) at either 100°C or 125°C. Two times the 2 or 3 sigma limit equals the total 4 or 6 sigma range, respectively. All the calculations were performed by the chairman of ASTM subcommittee D11.16.

'Accepted Reference Value' or AR-value; this is the average, for various Mooney viscosity (Mooney unit) values as listed below, obtained in an interlaboratory test program (ITP) for a large group of typical laboratories using samples taken from the mid-lot bale. See page 15 for

more details on the ITP. To better aid the user in establishing laboratory control limits, Subcommittee D11.16 requested that an AR value be determined for sublots of the IRM instead of just one AR value for the entire lot. This data was used to select a subset of the production material that met the desired uniformity requirements. Because of the care and testing frequency during production and the testing performed, lot G may be the most uniform, and is certainly the best documented, of any of the lots of the IRM241 material.

During the production of past IRM241 lots, the producer sampled at a set frequency, which has been different from lot to lot. The samples were tested and only the ML (1+8) at 125°C values reported. A bale at the mid-point value was selected and used in the ITP to determine the AR values and the testing control limits. During the ITP data on four values, ML (1+4) and ML (1+8) each at 100°C and 125°C, was collected and evaluated. The mean of the data from the ITP for each of the four properties was used to calculate offset values from the producer's ML (1+8) at 125°C data. Thus, the ML (1+8) at 125°C AR values were **corrected** by the offset while the ML (1+4) at 100°C, ML (1+8) at 100°C, and ML (1+4) at 125°C AR values were **calculated**, assuming the offset of ML (1+8) at 125°C applied to the other three properties.

Due to the extensive amount of sampling and testing done by the producer for Lot G, it is possible to determine the offset for each of the four properties and use this information to prepare corrected AR values for each property instead calculating the AR value for three of the properties based on an assumption concerning the magnitude of the offset. This helps to remove one source of variation from the AR value tables.

See Tables 2A to 2H for the AR values by box number.

'Within Typical Laboratory' 2 and 3 sigma value; this is the within laboratory +/- 2 and +/- 3 standard deviation (Sr) value (for single measurements) on the IRM241 Lot G AR-values, as obtained from the same group of typical ITP laboratories. (See Table 1.)

'Between Typical Laboratory' 2 and 3 sigma value; this is the between laboratory +/- 2 and +/- 3 standard deviation (SR) value (for single measurements) on the IRM241 Lot G AR-values, as obtained from the same group of typical ITP laboratories. (See Table 1.)

Special consideration for bias: The ITP used data from eleven companies and sixteen instruments. The statistical analysis of the data showed there was no statistically significant differences in the mean values between laboratories although a few values were declared to be outliers and were removed from the final data analysis. Three of the four properties had one mean outlier, all from the same instrument. All four properties had one outlier for variability, three of which were from the same instrument. No instrument was an outlier for mean and variability. An analysis for bias indicated that there may be bias within a laboratory and between laboratories. No investigation on the cause of the observed bias was made. A laboratory that did not participate in the ITP may find that it cannot maintain control within the control limits due to factors unique to that laboratory causing bias in its values or increased variation. The laboratory should conduct an investigation to identify the causes of the bias and variation to eliminate them so that it is aligned with the ITP data. The eleven companies that participated in the ITP could use the data submitted for the ITP to establish the bias for each of the sixteen instruments and adjust the AR value and control limits accordingly to maintain the instrument in control.

However, that does not address the need to eliminate the bias to bring the instrument into alignment with the ITP. Participation in a multi-laboratory precision study may help to identify the unique sources of bias and variation.

Table 1							
IRM 241 Lot G	Wit	hin Labora	tory	Betwe	Between Laboratories		
Test	Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR	
ML (1 + 4) @ 100°C	0.30	0.61	0.91	0.67	1.33	2.00	
ML (1 + 8) @ 100°C	0.33	0.66	0.98	0.78	1.56	2.35	
ML (1+4) @ 125°C	0.57	1.14	1.71	1.26	2.52	3.78	
ML (1 + 8) @ 125°C	0.39	0.78	1.16	1.13	2.26	3.38	

Box Number	2000	2003	2006	2009	2012	2015	2018	2021
ML (1+4) @ 100°C	75.5		75.3		75.9		75.7	
ML (1+8) @ 100°C	74.5		74.4		74.7		74.9	
ML (1+4) @ 125°C	55.2	54.7	54.4	54.5	55.2	55.3	54.6	54.3
ML (1+8) @ 125°C	51.2	50.8	51.1	50.7	51.0	51.1	51.2	51.0
Box Number	2024	2027	2030	2033	2036	2039	2042	2045
ML (1+4) @ 100°C	76.1		75.8		75.6		75.9	
ML (1+8) @ 100°C	74.8		74.8		74.8		75.0	
ML (1+4) @ 125°C	55.4	54.6	55.9	54.5	54.7	54.8	54.0	55.5
ML (1+8) @ 125°C	51.0	50.9	51.5	50.8	50.9	50.8	50.4	51.4
					2000	2052	2056	2072*
Box Number	2048	2051	2054	2057	2060	2063	2066	2072*
ML (1+4) @ 100°C	75.5		75.9		75.5		75.6	75.5
ML (1+8) @ 100°C	74.4	54.0	74.8	FF 2	74.5	55.0	74.7	74.7
ML (1+4) @ 125°C	55.2	54.9	55.4	55.3	54.6	55.8	54.9	54.6
ML (1+8) @ 125°C	51.0	50.9	51.4	51.1	50.9	51.3	51.1	50.8
					2000	2005*	*2069 ren	T
Box Number	2078*	2081	2084	2087	2090	2096*	2099	2105*
ML (1+4) @ 100°C	75.6	-	76.0		76.2	75.4		
ML (1+8) @ 100°C	74.7		74.9	540	75.0	74.7	F4.2	F4.2
ML (1+4) @ 125°C	54.6	55.3	54.4	54.2	54.4	55.2	54.3	54.2
ML (1+8) @ 125°C	50.7	51.0	50.9	50.6	50.7	51.7	50.5	50.4
	*2075 ren	1		0400*	*2093 removed		*2103 removed	
Box Number	2108	2111	2117*	2120*	2123*	2135*	2138	2141
ML (1+4) @ 100°C	76.0						76.2	
ML (1+8) @ 100°C	75.2						75.3	56.0
VIL (1+4) @ 125°C	54.3	54.3	54.7			55.0	55.2	56.0
ML (1+8) @ 125°C	50.6	50.7	50.9	100 .	L	51.2	51.4	51.8
	*2114 ren			123 not sa			29, & 2132	
Box Number	2144	2147	2150	2153	2156	2159	2162	2168*
ML (1+4) @ 100°C	75.8		76.2		76.0		76.1	75.8
VIL (1+8) @ 100°C	75.0		75.2	FF 0	75.4		75.2	75.2
ML (1+4) @ 125°C	55.5	54.5	55.3	55.3	55.2	55.7	55.5	54.7
VIL (1+8) @ 125°C	51.5	51.1	51.1	51.2	51.1	51.6	51.2	50.7
							*2165 ren	T
Box Number	2171	2174	2177	2180	2183	2186	2189	2192
ML (1+4) @ 100°C		75.9		75.8		76.0		75.7
ИL (1+8) @ 100°C		75.1		74.8		75.2		74.9
ML (1+4) @ 125°C	55.1	55.3	54.9	55.7	54.9	55.1	55.7	55.1
ML (1+8) @ 125°C	51.3	51.6	51.1	51.1	51.0	51.0	51.3	51.1

Box Number	2195	2198	2201	2204	2207	2210	2213	2216
ML (1+4) @ 100°C		76.0		76.0		75.6		76.0
ML (1+8) @ 100°C		75.3		75.5		74.8		75.2
ML (1+4) @ 125°C	56.3	54.8	54.6	54.7	54.3	55.0	55.7	54.9
ML (1+8) @ 125°C	51.9	50.9	51.0	51.1	51.2	51.5	51.8	51.4
Box Number	2219	2222	2225	2228	2231	2234	2237	2240
ML (1+4) @ 100°C		76.2		75.7		76.5		76.3
ML (1+8) @ 100°C		75.2		74.9		75.2		75.2
ML (1+4) @ 125°C	54.8	55.2	54.9	55.6	55.2	55.3	55.8	55.2
ML (1+8) @ 125°C	51.5	51.7	51.5	51.4	51.4	51.8	52.3	51.2
Box Number	2243	2246	2249	2252	2255	2258	2261	2264
ML (1+4) @ 100°C		75.9		75.8		75.8		76.0
ML (1+8) @ 100°C		75.3		74.9		75.2		75.3
ML (1+4) @ 125°C	55.1	54.9	54.9	54.9	55.1	55.0	55.0	55.9
ML (1+8) @ 125°C	51.3	51.0	51.2	51.0	51.0	51.1	51.0	51.5
Day Number	2267	2270	2273	2276	2279	2282	2285	2288
Box Number	2267		22/3	75.9	22/3	75.9	2203	76.3
ML (1+4) @ 100°C		75.7		75.7		75.9		75.2
ML (1+8) @ 100°C	55.3	75.2	55.8	56.1	55.6	55.4	55.5	56.0
ML (1+4) @ 125°C		55.2	51.6	51.5	51.9	51.6	51.4	51.4
ML (1+8) @ 125°C	51.3	51.2	31.0	31.3	31.9	31.0	31.4	31.4
Box Number	2291	2294	2297	2300	2303	2306	2309	2312
ML (1+4) @ 100°C		75.8		76.2		76.0		76.0
ML (1+8) @ 100°C		75.4		75.3		75.2		75.3
ML (1+4) @ 125°C	56.3	54.7	56.0	56.2	55.2	54.8	55.2	55.9
ML (1+8) @ 125°C	51.5	51.0	51.3	52.0	51.4	51.1	51.1	51.3
Box Number	2315	2318	2321	2324	2327	2330	2333	2336
ML (1+4) @ 100°C		76.0		76.1		76.0		76.2
ML (1+8) @ 100°C		75.0		74.9		74.8		75.2
ML (1+4) @ 125°C	55.0	54.2	55.4	55.0	55.1	55.0	54.7	54.6
ML (1+8) @ 125°C	51.6	51.1	51.6	52.3	51.7	51.5	51.5	51.5
Box Number	2339	2342	2345	2348	2351	2354	2357	2360
ML (1+4) @ 100°C		76.4		75.8		75.9		75.6
ML (1+8) @ 100°C		75.6		75.0		75.3		75.0
ML (1+4) @ 125°C	54.8	54.7	54.7	55.2	55.2	55.1	54.8	54.4
ML (1+8) @ 125°C	51.3	51.5	51.5	51.8	52.0	51.3	51.4	51.1
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Table 2C, Boxes 2								
Box Number	2363	2366	2369	2372	2375	2378	2381	2384
ML (1+4) @ 100°C		76.0		76.0		76.0		76.2
ML (1+8) @ 100°C		75.5		75.1		75.4		75.3
ML (1+4) @ 125°C	54.7	54.5	54.6	54.8	54.6	54.6	55.3	55.6
ML (1+8) @ 125°C	51.2	51.1	51.2	51.4	51.4	51.3	51.3	51.6
Box Number	2387	2390	2393	2396	2399	2402	2405	2408
ML (1+4) @ 100°C		76.6		76.4		76.7		76.7
ML (1+8) @ 100°C		75.7		75.9		75.4		75.6
ML (1+4) @ 125°C	54.6	55.0	55.0	54.5	55.6	54.7	55.0	56.0
ML (1+8) @ 125°C	51.3	51.3	51.3	51.0	51.4	51.2	51.2	51.8
Box Number	2411	2414	2417	2420	2423	2426	2429	2432
ML (1+4) @ 100°C		76.6		76.4		76.5		76.5
ML (1+8) @ 100°C		75.7		75.6		75.5		75.6
ML (1+4) @ 125°C	56.1	55.6	55.5	55.4	55.5	54.8	55.3	55.1
ML (1+8) @ 125°C	51.9	51.7	51.5	51.4	51.5	51.1	51.3	51.3
WE (1.0) @ 125 C	02.0	<b>J</b>	52.0					
Box Number	2435	2438	2441	2444	2447	2450	2453	2456
ML (1+4) @ 100°C		76.6		76.1		76.3		76.5
ML (1+8) @ 100°C		75.6		75.3		75.1		75.8
ML (1+4) @ 125°C	54.5	55.1	56.6	55.0	55.1	55.3	54.9	55.3
ML (1+8) @ 125°C	51.4	51.3	51.8	51.1	51.4	51.2	51.6	51.7
Box Number	2459	2462	2465	2468	2471	2474	2477	2483
ML (1+4) @ 100°C		76.4		76.0		76.2		
ML (1+8) @ 100°C		75.3		75.3		75.6		
ML (1+4) @ 125°C	55.5	55.3	56.1	55.2	55.6	55.6	55.5	55.9
ML (1+8) @ 125°C	51.7	51.8	52.0	51.4	51.6	51.6	51.3	51.5
							*2480 ren	noved
Box Number	2486	2489	2492	2495	2498	2501	2504	2507
ML (1+4) @ 100°C	76.3		76.2		76.5		76.2	
ML (1+8) @ 100°C	75.7		75.4		75.6		75.3	
ML (1+4) @ 125°C	55.5	55.3	54.7	55.5	55.2	55.0	54.9	55.0
ML (1+8) @ 125°C	51.2	51.3	51.4	51.1	51.2	51.2	51.4	51.1
Box Number	2510	2513	2516	2519	2522	2525	2528	2531
ML (1+4) @ 100°C	76.2		76.1		75.9		76.2	
ML (1+8) @ 100°C	75.1		75.4		75.2		75.3	
ML (1+4) @ 125°C	54.9	55.6	55.6	55.6	55.7	55.9	55.5	55.3
	51.4	51.5	51.8	51.4	52.0	51.8	51.7	51.7

Box Number	2534	2537	2540	2543	2546	2549	2552	255
ML (1+4) @ 100°C	76.0		76.3		76.0		76.1	
ML (1+8) @ 100°C	75.2		75.5		75.3		75.4	
ML (1+4) @ 125°C	55.6	55.5	55.5	54.9	55.9	54.9	55.0	55.7
ML (1+8) @ 125°C	51.6	51.6	52.0	51.0	51.3	51.2	51.1	51.6
Box Number	2558	2561	2564	2567	2570	2573	2576	2579
ML (1+4) @ 100°C	76.4		76.2		75.8		75.8	
ML (1+8) @ 100°C	75.6		75.2		75.1		75.0	
ML (1+4) @ 125°C	55.5	55.3	56.0	55.5	56.0	55.7	56.1	56.1
ML (1+8) @ 125°C	51.7	51.2	51.8	51.8	51.9	52.2	52.4	52.2
Box Number	2582	2585	2588	2591	2594	2597	2600	2603
ML (1+4) @ 100°C	75.6		76.0		75.8		75.9	
ML (1+8) @ 100°C	75.0		75.1		75.2		75.1	
ML (1+4) @ 125°C	55.9	56.5	55.2	55.9	55.3	55.3	54.8	54.8
ML (1+8) @ 125°C	52.0	52.7	51.7	52.1	51.5	51.6	51.5	51.3
Box Number	2606	2609	2612	2615	2618	2624*	2627	2630
ML (1+4) @ 100°C	75.9		76.0		75.9	75.9		76.6
ML (1+8) @ 100°C	75.0		74.9		75.0	75.0		75.6
ML (1+4) @ 125°C	54.8	55.1	55.3	54.8	54.5	56.3	54.8	54.6
ML (1+8) @ 125°C	51.4	51.4	51.6	51.5	51.0	51.7	51.3	51.2
						*2621 ren	noved	
Box Number	2633	2636	2639	2642	2645	2648	2651	2654
ML (1+4) @ 100°C		76.3		76.8		76.6		76.3
ML (1+8) @ 100°C		75.6		75.5		75.7		75.2
ML (1+4) @ 125°C	56.0	55.7	57.0	56.4	54.8	56.5	55.6	55.8
VIL (1+8) @ 125°C	52.1	51.7	52.1	52.0	51.3	52.1	51.6	51.3
Box Number	2657	2660	2663	2666	2669	2672	2678*	2681
ML (1+4) @ 100°C		76.4		76.3		76.2	76.5	
ML (1+8) @ 100°C		75.4		75.3		75.2	75.3	
ML (1+4) @ 125°C	55.6	55.4	55.5	55.1	55.1	55.3	55.0	55.1
ML (1+8) @ 125°C	51.5	51.5	51.4	51.2	51.5	51.2	51.1	51.0
							*2675 rem	
Box Number	2684	2687	2690	2693	2696	2699	2702	2705
ИL (1+4) @ 100°C	76.3		76.0		76.4		76.0	
ИL (1+8) @ 100°C	75.4		75.1		75.5		75.0	
ИL (1+4) @ 125°C	54.6	54.9	55.2	55.3	55.3	55.3	55.5	55.7
ИL (1+8) @ 125°C	50.9	50.9	51.2	51.4	51.7	51.6	51.4	51.7

<b>Box Number</b>	2708	2711	2714	2717	2720	2723	2726	2729
ML (1+4) @ 100°C	75.9		75.9		76.1		76.0	
ML (1+8) @ 100°C	75.2		75.0		75.2		75.3	
ML (1+4) @ 125°C	55.8	55.8	55.6	55.5	55.4	55.4	55.7	55.2
ML (1+8) @ 125°C	51.6	51.8	51.7	51.7	51.7	51.5	51.6	51.4
Box Number	2732	2735	2738	2741	2744	2747	2750	2753
ML (1+4) @ 100°C	76.3		76.2		76.2		76.1	
ML (1+8) @ 100°C	75.3		75.4		75.2		75.1	
ML (1+4) @ 125°C	55.8	54.8	55.6	55.8	55.7	55.5	55.3	55.8
ML (1+8) @ 125°C	51.5	51.3	51.8	51.6	51.7	51.7	51.6	51.8
					2762	0774	2774	227-
Box Number	2756	2759	2762	2765	2768	2771	2774	2777
ML (1+4) @ 100°C	75.9		76.2		76.1		76.2	
ML (1+8) @ 100°C	75.2	55.1	75.2	F	75.1	E4.0	75.2	FF A
ML (1+4) @ 125°C	56.0	55.4	55.3	55.4	55.1	54.9	55.7	55.4
ML (1+8) @ 125°C	51.8	51.3	51.6	51.3	51.1	51.3	51.9	51.6
Box Number	2780	2786	2792*	2795	2798	2801	2804	2807
ML (1+4) @ 100°C	76.1	76.0	76.1	2/33	76.3	2001	76.0	2007
ML (1+8) @ 100°C	75.1	75.1	75.3		75.3		75.4	
ML (1+4) @ 125°C	55.8	55.8	55.5	55.2	55.2	55.4	55.6	55.8
ML (1+4) @ 125°C	51.6	52.0	51.5	51.2	51.4	52.0	51.6	52.2
IVIL (1+6) @ 125 C	31.0	32.0	*2789 rem		31.4	32.0	31.0	32.2
Box Number	2810	2813	2816	2819	2822	2825	2828	2831
ML (1+4) @ 100°C	76.2	2013	75.9	2013	75.9		76.9	
ML (1+8) @ 100°C	75.4		75.2		75.5		75.6	
ML (1+4) @ 125°C	55.5	56.3	56.3	55.7	56.1	55.9	56.3	55.7
ML (1+8) @ 125°C	51.6	52.1	52.1	51.6	51.7	51.6	52.0	51.8
WE (1.0) @ 123 C	31.0	J2.1	02.2	51.0	02.17		02.0	
Box Number	2837*	2840	2843	2846	2849	2852	2855	2858
ML (1+4) @ 100°C		76.5		76.3		76.4		76.1
ML (1+8) @ 100°C		75.5		75.4		75.4		75.6
ML (1+4) @ 125°C	55.3	55.2	55.6	55.0	55.7	55.1	56.4	55.4
ML (1+8) @ 125°C	51.5	51.1	51.5	51.3	51.4	51.3	51.9	51.5
	*2834 rem							
Box Number	2861	2864	2867	2870	2873	2876	2879	2882
ML (1+4) @ 100°C		76.7		76.2		76.5		76.1
ML (1+8) @ 100°C		75.6		75.5		75.6		75.3
VIL (1+4) @ 125°C	55.6	55.0	56.0	55.4	54.9	55.0	55.0	55.2
ML (1+8) @ 125°C	51.5	51.4	52.1	51.6	51.2	51.4	51.3	51.6

Box Number	2885	2888	2891	2894	2897	2900	2903	2906
ML (1+4) @ 100°C		76.1		76.3		76.3		76.2
ML (1+8) @ 100°C		75.2		75.2		75.3		75.3
ML (1+4) @ 125°C	56.1	55.0	54.8	55.8	55.1	54.8	55.1	55.2
ML (1+8) @ 125°C	52.0	51.4	51.2	51.7	51.1	50.9	50.9	51.3
Box Number	2909	2912	2918*	2921	2924	2927	2930	2933
ML (1+4) @ 100°C		76.4	76.5		76.3		76.4	
ML (1+8) @ 100°C		75.7	75.4		75.5		75.2	
ML (1+4) @ 125°C	55.0	54.9	55.5	55.7	55.6	55.7	54.7	55.6
ML (1+8) @ 125°C	51.0	51.0	51.4	51.3	51.7	51.5	51.1	51.4
			*2915 rem	oved				
Box Number	2936	2939	2942	2945	2948	2951	2954	2957
ML (1+4) @ 100°C	76.1		76.0		76.3		76.5	
ML (1+8) @ 100°C	75.3		75.6		75.5		75.5	
ML (1+4) @ 125°C	55.5	55.1	54.9	55.1	54.6	55.4	55.1	55.6
ML (1+8) @ 125°C	51.2	51.0	51.1	51.2	51.1	51.1	51.0	51.2
Box Number	2960	2963	2966	2969	2972	2975	2978	2981
ML (1+4) @ 100°C	76.3	2505	76.2	2505	75.9		76.1	
ML (1+8) @ 100°C	75.5		75.2		75.0		75.3	
ML (1+4) @ 125°C	55.6	55.5	55.5	54.7	55.6	55.3	54.9	54.9
ML (1+8) @ 125°C	51.3	51.5	51.5	51.2	51.3	51.2	51.2	51.0
Day Number	2984	2007	2000	2002	2996	2999	3002	3005
Box Number	75.8	2987	<b>2990</b> 76.0	2993	76.4	2555	75.9	3003
ML (1+4) @ 100°C ML (1+8) @ 100°C	75.3		75.4		75.3		75.2	
ML (1+4) @ 125°C	54.8	54.7	54.6	54.7	54.3	54.4	55.6	55.2
ML (1+4) @ 125°C	50.8	50.8	50.7	50.8	50.8	50.7	51.1	51.5
VIL (1+8) @ 125 C	30.0	30.8	30.7	30.6	30.8	30.7	31.1	31.3
Box Number	3008	3011	3014	3017	3020	3023	3026	3029
VIL (1+4) @ 100°C	76.0		76.0		75.7		75.8	
ML (1+8) @ 100°C	75.0		75.2		75.1		75.2	
ML (1+4) @ 125°C	54.8	54.9	54.5	55.0	54.5	54.7	54.8	54.7
ML (1+8) @ 125°C	51.1	51.0	50.9	51.1	50.7	51.0	50.9	51.1
Box Number	3032	3035	3041*	3044	3047	3056*	3059	3062
ML (1+4) @ 100°C	76.0			76.5		76.7		76.7
VIL (1+8) @ 100°C	75.1			75.7		75.8		75.5
VIL (1+4) @ 125°C	55.4	55.8	55.3	55.7	55.5	55.3	55.1	55.3
VIL (1+4) @ 125°C	51.6	51.5	51.3	51.5	51.3	51.3	51.4	51.2
, , , , , , , ,			*3038 rem		+	053 remov		

Box Number	3065	3068	3071	3074	3077	3080	3086*	3089
ML (1+4) @ 100°C		76.4		76.1		76.2	76.1	
ML (1+8) @ 100°C		75.6		75.3		75.3	75.1	
ML (1+4) @ 125°C	55.6	55.6	54.3	54.3	54.7	54.1	55.0	56.0
ML (1+8) @ 125°C	51.6	51.3	51.0	50.8	51.0	50.6	51.0	51.8
							*3083 rem	noved
Box Number	3092	3095	3098	3101	3104	3107	3110	3113
ML (1+4) @ 100°C	76.0		76.2		76.3		76.1	
ML (1+8) @ 100°C	75.2		75.5		75.1		75.2	
ML (1+4) @ 125°C	55.3	54.8	55.4	55.7	54.4	54.6	54.8	55.6
ML (1+8) @ 125°C	51.5	51.6	51.4	51.5	51.1	51.2	51.2	51.7
Box Number	3116	3119	3122	3125	3128	3134*	3137	3140
ML (1+4) @ 100°C	76.0		75.7		76.1	76.3		76.0
ML (1+8) @ 100°C	75.2		74.9		75.2	75.2		75.3
ML (1+4) @ 125°C	54.4	56.2	54.5	54.6	56.2	56.1	55.6	55.9
ML (1+8) @ 125°C	51.1	51.6	50.9	51.1	52.0	52.0	51.7	51.7
						*3131 rer	noved	
Box Number	3143	3146	3149	3152	3155	3158	3164*	3176*
ML (1+4) @ 100°C		76.0		76.3		75.9	76.1	75.9
ML (1+8) @ 100°C		75.1		75.2		75.1	75.3	75.0
ML (1+4) @ 125°C	56.0	55.6	55.6	55.3	55.0	55.0	55.2	55.2
ML (1+8) @ 125°C	51.9	51.8	51.6	51.3	51.3	51.3	51.2	51.2
			*3161 rem	oved	*3167, 31	70, & 3173	not sample	T
Box Number	3179	3182*	3185	3188	3191	3194	3197	3203*
ML (1+4) @ 100°C		76.1		76.0		76.1		
ML (1+8) @ 100°C		75.1		75.2		75.2		
ML (1+4) @ 125°C	55.9	55.2	55.7	55.7	55.4	55.3	55.5	54.9
ML (1+8) @ 125°C	51.5	51.4	51.7	51.7	51.3	51.4	51.4	51.1
		*Used fo	ARV ITP				*3200 ren	
Box Number	3206	3209	3212	3215	3218	3221	3224	3227
ML (1+4) @ 100°C	76.1		76.2		76.1		76.2	
ML (1+8) @ 100°C	75.0		75.3		75.0		75.4	
ML (1+4) @ 125°C	55.0	55.0	55.3	56.3	54.7	55.2	55.1	55.9
ML (1+8) @ 125°C	51.4	51.2	51.2	51.8	51.0	51.4	51.1	51.7
						1		
Box Number	3230	3233	3236	3239	3242	3245	3248	3251
ML (1+4) @ 100°C	76.5		75.9		76.2		76.1	
ML (1+8) @ 100°C	75.5		75.0		75.1		75.4	
ML (1+4) @ 125°C	55.4	54.9	55.6	55.6	55.9	55.2	54.8	54.9
ML (1+8) @ 125°C	51.2	50.9	51.7	51.6	51.2	51.4	51.0	50.9

Table 2H, Boxes	3254 to 3	257						
Box Number	3254	3257			MIENE WE		Mark Too	
ML (1+4) @ 100°C	76.1			ALCOHOL:		B707 -> 7152		
ML (1+8) @ 100°C	75.0				F1 - 1/5 H	17 2 3		
ML (1+4) @ 125°C	55.1	55.3		Militar Vacce				V.C.
ML (1+8) @ 125°C	51.2	51.2	11.3					1111
					1945	自己なみ前		Ballalia Ka

#### **Determining control limits examples:**

#### Example 1, Exact box number given in table:

For this example, the user wants to establish the 2 sigma control limits for his (or her) laboratory for the ML (1 + 4) @ 125°C test. The laboratory received box 2027 from IRM241 Lot G.

Step 1: Find the AR value for box 2027 for the ML (1 + 4) @ 125°C test.

From Tables 2A to 2H, find the table whose range of boxes includes box number 2027. Box 2027 is found in Table 2A. Box 2027 is one of the boxes listed in the table. Read down the column under box number 2027 to the cell that intersects the ML (1 + 4) @ 125°C test row to find the AR value of 54.6.

Step 2: Find the standard deviation value to use for the 2 sigma control limits for the ML (1 + 4) @ 125°C test.

From Table 1, in the "Within Laboratory" columns, find the "2 x Sr" column. Read down the "2 x Sr" column to the cell that intersects the ML (1 + 4) @ 125°C test row to find the 2 sigma value of 1.14.

Step 3: Calculate the 2 sigma control limits for the ML (1 + 4) @ 125°C test.

The Lower Control Limit (LCL) = AR - 2 sigma = 54.6 - 1.14 = 53.46

The Upper Control Limit (UCL) = AR + 2 sigma = 54.6 + 1.14 = 55.74

The 2 sigma control limits for the ML (1 + 4) @ 125°C test for box 2027 are 53.46 to 55.74.

In a similar manner, the control limits for the other possible choices of sigma limits, within laboratory or between laboratory, and tests can be calculated.

#### Example 2, Exact box number NOT given in table but bracketed without missing boxes:

For this example, the user wants to establish the 2 sigma control limits for his (or her) laboratory for the ML (1 + 8) @ 100°C test. The laboratory received box 2400 from IRM241 Lot G.

Step 1: Determine the AR value for box 2400 for the ML (1 + 8) @ 100°C test.

While a sample was taken about every third box, this was not always completed. To determine the value for a box whose values are not in the table, it will be necessary to interpolate from the values that are given.

From Tables 2A to 2H, find the table whose range of boxes includes box 2400. This is found in Table 2C. In the table, find the box numbers that bracket box number 2400. Box 2400 is bracketed by the columns for box 2399 and box 2402. However, there are no 100°C values shown for box 2399. Therefore, it is necessary to go to the next previous box with values, which is box 2396. Read down the column under box number 2396 to the cell that intersects the ML (1 + 8) @ 100°C test row to find the AR value of 75.9. Read down the column under box number 2402 to the cell that intersects the ML (1 + 8)@ 100°C test row to find the AR value of 75.4. The absolute difference between box 2396 and box 2402 test values is 0.5 (75.9 – 75.4). Starting with box 2396, six boxes are counted to get to box 2402. The absolute difference between the boxes' test values must be apportioned across the six boxes. Therefore, 0.5/6 = 0.083 is to be apportioned to each of the six boxes. The determined AR value for box 2400 must be between the known AR values for the two bracketing boxes. Since the test value for box 2396 is higher than that of box 2402, the determined AR value for box 2400 will be lower than box 2396 and higher than box 2402. Box 2400 is closer by count to box 2402 so the AR for box 2400 will be determined as an offset from box 2402. The count difference between box 2402 and box 2400 is 2 boxes. Therefore, the AR for box 2400 can be determined as 75.4 + (0.083 \* 2) = 75.567, which rounds to the nearest 0.1 Mooney unit to 75.6. The determined AR value for box 2400 is 75.6.

Step 2: Find the standard deviation value to use for the 2 sigma control limits for the ML (1 + 8) @ 100°C test.

From Table 1, in the "Within Laboratory" columns, find the " $2 \times Sr$ " column. Read down the " $2 \times Sr$ " column to the cell that intersects the ML (1 + 8) @ 100°C test row to find the 2 sigma value of 0.66.

Step 3: Calculate the 2 sigma control limits for the ML (1 + 8) @ 100°C test.

The Lower Control Limit (LCL) = AR - 2 sigma = 75.6 - 0.66 = 74.94

The Upper Control Limit (UCL) = AR + 2 sigma = 75.6 + 0.66 = 76.26

The 2 sigma control limits for the ML (1 + 8) @  $100^{\circ}$ C test for box 2400 are 74.94 to 76.26.

In a similar manner, the control limits for the other possible choices of sigma limits, within laboratory or between laboratory, and tests can be calculated.

### Example 3, Exact box number NOT given in table and bracket includes missing box(es):

For this example, the user wants to establish the 3 sigma control limits for his (or her) laboratory for the ML (1 + 8) @ 125°C test. The laboratory received box 2620 from IRM241 Lot G.

Step 1: Determine the AR value for box 2620 for the ML (1 + 8) @  $125^{\circ}$ C test.

While a sample was taken about every third box, some boxes were removed from the lot. To determine the value for a box whose values are not in the table, it will be necessary to interpolate from the values that are given.

From Tables 2A to 2H, find the table whose range of boxes includes box 2620. This is found in Table 2D. In the table, find the box numbers that bracket box number 2620. Box 2620 is bracketed by the columns for box 2618 and box 2624. There is a note on box 2624 stating that box 2621 was removed. Values are given for both box 2618 and box 2624. Read down the column under box number 2618 to the cell that intersects the ML (1 +8) @ 125°C test row to find the AR value of 51.0. Read down the column under box number 2624 to the cell that intersects the ML (1 + 8) @ 125°C test row to find the AR value of 51.7. The absolute difference between box 2618 and box 2624 test values is 0.7 (51.0 - 51.7). Starting with box 2618, normally six boxes would be counted to get to box 2624. Since box 2621 has been removed, there are only five boxes in the lot. However, the removed box was in the lot at the time of testing and should be included in the count for interpolation. Therefore, the absolute difference between the boxes' test values must be apportioned across the six boxes. (There are a few cases where more than one consecutive box has been removed. The same logic must be used to determine the number of boxes to be used for interpolation.) Therefore, 0.7/6 = 0.117 is to be apportioned to each of the six boxes. The determined AR value for box 2620 must be between the known AR values for the two bracketing boxes. Since the test value for box 2618 is lower than that of box 2624, the determined AR value for box 2620 will be higher than box 2618 and lower than box 2624. Box 2620 is closer by count to box 2618 so the AR for box 2620 will be determined as an offset from box 2618. The count difference between box 2618 and box 2620 is 2 boxes. Therefore, the AR for box 2620 can be determined as 51.0 + (0.117 \* 2) = 51.234, which rounds to the nearest 0.1 Mooney unit to 51.2. The determined AR value for box 2620 is 51.2.

Step 2: Find the standard deviation value to use for the 3 sigma control limits for the ML (1 + 8) @ 125°C test.

From Table 1, in the "Within Laboratory" columns, find the "3 x Sr" column. Read down the "3 x Sr" column to the cell that intersects the ML (1 + 8) @  $125^{\circ}$ C test row to find the 3 sigma value of 1.16.

Step 3: Calculate the 3 sigma control limits for the ML (1 + 8) @ 125°C test.

The Lower Control Limit (LCL) = AR - 3 sigma = 51.2 - 1.16 = 50.04

The Upper Control Limit (UCL) = AR + 3 sigma = 51.2 + 1.16 = 52.36

The 3 sigma control limits for the ML (1 + 8) @ 125°C test for box 2620 are 50.04 to 52.36.

In a similar manner, the control limits for the other possible choices of sigma limits, within laboratory or between laboratory, and tests can be calculated.

#### Other possible cases:

Because some bales were not sampled, it is possible to have a box number appear in the tables without any associated data. The techniques of example 2 can be used to get an interpolated value for these boxes.

There are some cases in the tables where bales were not sampled and adjacent bales were removed. The techniques of examples 2 and 3 can be combined to get an interpolated value for these boxes.

## Background and Interlaboratory Test Program Details: IRM241 Lot G Butyl Rubber

Background - Industry Reference Materials (IRMs), used for a number of test methods under the jurisdiction of ASTM Committee D11, are prepared according to D4678, "Preparation, Testing, Acceptance and Documentation for Industry Reference Materials (IRM)". Testing of the IRM lot produces four types of 'lot properties' for each type of test conducted as part of the IRM evaluation. This lot is a typical NB or non-blended IRM that requires an evaluation for lot uniformity or homogeneity as well as accepted reference or AR-value using D4678.

Evaluation of the IRM Lot - This seventh lot of Butyl rubber IRM, designated as IRM241 Lot G, was produced by ExxonMobil Chemical Co. at their Baytown, TX facility in September 2017 and packaged into boxes. Following testing and evaluation for uniformity by ExxonMobil, the lot was analyzed by the D11.16 chairman to determine lot uniformity or homogeneity in October 2017. A lot of 1236 bales was found to be uniform in properties using the D4678 Annex A3 procedure.

Interlaboratory Test Program (ITP) - Eleven companies using a total of sixteen Mooney instruments each tested IRM241 Lot G per D1646, determining values for four properties: ML (1+4) at 100°C, ML (1+8) at 100°C, ML (1+4) at 125°C, and ML (1+8) at 125°C. Each property was determined by two technicians once on each of two different days for a total of four measurements for each instrument. The testing was performed in the range of the first week of December 2017 to the first week of March 2018. The data was analyzed per D4678, annex A4.

<u>Using IRM241 Lot G</u> - When cutting samples from the bale it is recommended that rubber in the outer layers of the bale (12 mm or one-half inch from surface) not be used for testing. Samples from this outer layer frequently give Mooney values that are less than interior samples by approximately 0.3 Mooney units.

It is strongly recommended that laboratories determine if they are operating in an "in control" manner, by the use of the +/- 2 sigma limits. Despite rigorous analysis of the ITP data for the AR-value(s) and associated standard deviation(s), the group of laboratories in this (and any) ITP do not represent a typical "in statistical control system" to which the usual 6 sigma limits are applied. All the assignable causes of variation that are typically eliminated to attain 'statistical control' have not and cannot be, eliminated for the AR testing.

<u>Supplementary Information</u> - Store this IRM in a cool, normal humidity location. It is expected to remain stable for a period of ten years. A full 'Research Report' documenting all the analysis for IRM241 Lot G will be on file at ASTM headquarters (expected after June 2018), 100 Barr Harbor Dr., W. Conshohocken, PA 19428, USA; Phone 610-832-9500, Fax 610-832-9555.

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