

Documentation Sheet

Industry Reference Material – 241 Lot H 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 16 of this Documentation Sheet for additional background on this IRM.

Production of IRM241 Lot H

The ASTM D11.20 IRM Task Group requested that the production of IRM241 Lot H contain 1425 bales. The producer made 1497 bales that were each placed in a box and numbered consecutively from 2000 to 3496. The production took place on one day. From this production, 1425 bales were selected to be Lot H. 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.

During the production of IRM241 Lot H, the producer's sampling plan was to sample every third bale. 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 2510 was selected because the test values for three of the four properties were at the mid-point value and the fourth only differed from the mid-point value by 0.2 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 H

The testing conducted as part of the IRM241 Lot H 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 H. 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 H 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 H, 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.

While the testing plan was to test every third and sixth bale, the actual testing pattern did not always follow that plan. In a few cases, the testing interval between bales was greater or fewer than three or six. There are several instances where consecutive bales were tested. The test results for any bale that had some testing performed for it are included in Table 2. For any particular bale that was not tested, it will be necessary to interpolate using the data for the bales on either side of that particular bale to get the reference value for that particular bale.

See Tables 2A to 2I 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 H 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 H AR-values, as obtained from the same group of typical ITP laboratories. (See Table 1.)

Special consideration for bias: The ITP used data from seven companies and fourteen 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 seven companies that participated in the ITP could use the data submitted for the ITP to establish the bias for each of the fourteen 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 H	Within Laboratory			Between Laboratories		
Test	Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR
ML (1 + 4) @ 100°C	0.27	0.54	0.81	0.70	1.40	2.09
ML (1 + 8) @ 100°C	0.29	0.58	0.87	0.91	1.82	2.73
ML (1 + 4) @ 125°C	0.74	1.49	2.23	1.39	2.78	4.17
ML (1 + 8) @ 125°C	0.51	1.01	1.52	1.13	2.26	3.40

Table 2A, Boxes 2000 to 2149								
Box Number	2001	2002	2003	2004	2007	2008	2011	2014
ML (1+4) @ 100°C		76.7			76.4		76.6	
ML (1+8) @ 100°C		75.3			75.0		75.1	
ML (1+4) @ 125°C	56.9	57.1	57.1	54.7	56.1	54.5	55.4	55.4
ML (1+8) @ 125°C	51.2	51.2	51.2	51.2	51.1	50.4	51.2	50.6
Box Number	2016	2019	2022	2025	2028	2029	2030	2033
ML (1+4) @ 100°C	76.6		76.8		76.8		76.4	
ML (1+8) @ 100°C	75.2		75.5		75.4		74.9	
ML (1+4) @ 125°C	55.2	56.1	54.5	54.5	55.4	55.4	55.2	54.7
ML (1+8) @ 125°C	50.5	51.1	50.3	50.4	51.2	50.6	50.5	51.0
Box Number	2036	2039	2042	2045	2048	2051	2054	2055
ML (1+4) @ 100°C	76.5		76.8		76.9		77.1	
ML (1+8) @ 100°C	75.4		75.7		75.6		75.7	
ML (1+4) @ 125°C	54.3	55.0	55.0	55.1	54.3	55.0	54.0	53.7
ML (1+8) @ 125°C	50.3	50.3	50.5	50.4	50.1	50.4	50.1	49.9
Box Number	2056	2062	2065	2068	2071	2074	2077	2080
ML (1+4) @ 100°C	77.0	76.7	76.7		77.1		77.0	
ML (1+8) @ 100°C	75.6	75.5	75.5		75.8		75.8	
ML (1+4) @ 125°C	54.6	55.2	54.3	54.4	53.9	55.5	54.1	53.9
ML (1+8) @ 125°C	50.3	50.0	50.4	50.4	50.4	50.2	50.2	50.0
Box Number	2083	2086	2089	2090	2092	2095	2098	2101
ML (1+4) @ 100°C	76.9	77.0			76.7		76.9	
ML (1+8) @ 100°C	75.9	75.6			75.5		75.5	
ML (1+4) @ 125°C	54.3	55.3	55.3		54.3	53.8	55.2	54.2
ML (1+8) @ 125°C	50.2	50.9	50.9	50.5	50.1	50.3	50.5	50.2
Box Number	2104	2107	2110	2113	2116	2119	2122	2125
ML (1+4) @ 100°C	77.1		76.8		76.7		76.5	
ML (1+8) @ 100°C	75.7		75.4		75.4		75.3	
ML (1+4) @ 125°C	55.8	53.8	54.7	53.8	54.1	53.7	53.5	54.1
ML (1+8) @ 125°C	51.2	50.0	50.4	50.0	50.3	50.1	50.0	50.0
Box Number	2128	2131	2134	2137	2140	2143	2146	2149
ML (1+4) @ 100°C	76.7		77.0		76.6		76.4	
ML (1+8) @ 100°C	75.4		75.5		75.3		75.2	
ML (1+4) @ 125°C	54.1	53.6	54.9	53.7	55.8	54.5	55.8	54.1
ML (1+8) @ 125°C	50.3	50.0	50.3	49.9	50.8	50.3	50.7	50.2

Table 2B, Boxes 2150 to 2291								
Box Number	2152	2155	2158	2161	2164	2166	2169	2172
ML (1+4) @ 100°C	76.7		76.8		76.8		76.7	76.9
ML (1+8) @ 100°C	75.4		75.5		75.4		75.4	75.7
ML (1+4) @ 125°C	54.9	54.2	57.1	54.6	54.5	55.9	54.8	55.4
ML (1+8) @ 125°C	50.8	50.4	51.3	50.7	50.7	50.8	50.6	50.8
Box Number	2175	2177	2180	2183	2186	2187	2188	2190
ML (1+4) @ 100°C		76.5		76.5		76.8		76.5
ML (1+8) @ 100°C		75.2		75.1		75.3		75.3
ML (1+4) @ 125°C	54.0	55.7	54.3	54.2	54.3	54.2	54.2	54.2
ML (1+8) @ 125°C	50.3	50.9	50.8	50.6	50.5	50.7	50.6	50.6
Box Number	2193	2196	2199	2202	2203	2206	2209	2210
ML (1+4) @ 100°C		76.8		76.9		76.8		76.8
ML (1+8) @ 100°C		75.4		75.5		75.6		75.4
ML (1+4) @ 125°C	54.1	54.2	55.5	54.7	54.6	54.6	55.9	55.3
ML (1+8) @ 125°C	50.5	50.5	50.7	50.8	50.9	50.8	51.0	50.7
Box Number	2213	2216	2219	2222	2225	2228	2231	2233
ML (1+4) @ 100°C		76.8		77.1		76.8		76.8
ML (1+8) @ 100°C		75.5		75.5		75.5		75.3
ML (1+4) @ 125°C	55.5	54.3	54.4	54.5	55.0	54.4	54.0	54.3
ML (1+8) @ 125°C	50.9	50.5	50.6	50.6	50.8	50.5	50.6	50.6
Box Number	2236	2239	2242	2245	2248	2249	2250	2252
ML (1+4) @ 100°C		76.5		76.6		76.7		76.7
ML (1+8) @ 100°C		75.1		75.2		75.3		75.3
ML (1+4) @ 125°C	54.2	53.8	53.8	55.9	54.5	55.5	54.7	54.4
ML (1+8) @ 125°C	50.6	50.3	50.3	51.0	50.6	50.9	50.7	50.8
Box Number	2253	2254	2255	2256	2259	2263	2266	2269
ML (1+4) @ 100°C		76.8		76.7		76.7		76.8
ML (1+8) @ 100°C		75.4		75.3		75.5		75.4
ML (1+4) @ 125°C	54.3	54.5	54.1	56.1	55.0	54.8	54.3	54.7
ML (1+8) @ 125°C	50.8	50.8	50.4	51.0	51.1	51.1	50.8	50.9
Box Number	2272	2273	2276	2279	2282	2285	2288	2291
ML (1+4) @ 100°C		76.6		76.6		76.0		76.7
ML (1+8) @ 100°C		75.4		75.1		74.6		75.4
ML (1+4) @ 125°C	54.3	55.8	54.9	54.0	54.0	53.3	55.3	54.2
ML (1+8) @ 125°C	50.8	51.1	50.2	50.5	50.6	49.8	51.1	50.7

Table 2C, Boxes 2292 to 2453								
Box Number	2297	2300	2303	2305	2308	2311	2315	2318
ML (1+4) @ 100°C	76.5		76.6		76.5		76.6	
ML (1+8) @ 100°C	75.3		75.2		75.2		75.3	
ML (1+4) @ 125°C	56.4	55.0	55.4	54.7	54.6	55.1	55.4	54.7
ML (1+8) @ 125°C	51.3	50.6	51.1	50.9	50.9	51.1	51.1	51.0
Box Number	2319	2322	2325	2328	2331	2334	2336	2339
ML (1+4) @ 100°C	76.6		76.4		76.6		76.2	
ML (1+8) @ 100°C	75.2		75.1		75.1		74.8	
ML (1+4) @ 125°C	55.0	54.6	55.6	55.3	54.9	55.4	55.3	55.7
ML (1+8) @ 125°C	51.0	50.9	51.1	51.2	51.0	51.2	50.8	51.2
Box Number	2342	2345	2348	2350	2353	2356	2359	2362
ML (1+4) @ 100°C	76.1		76.2		76.7		76.4	
ML (1+8) @ 100°C	74.7		75.0		75.3		74.9	
ML (1+4) @ 125°C	54.6	54.7	55.0	54.7	55.0	54.4	54.5	54.9
ML (1+8) @ 125°C	51.2	50.7	51.1	51.1	51.1	50.6	50.9	51.0
Box Number	2365	2368	2371	2373	2376	2379	2382	2385
ML (1+4) @ 100°C	76.4		76.3		76.4		76.3	
ML (1+8) @ 100°C	75.0		75.1		75.2		75.1	
ML (1+4) @ 125°C	55.7	54.4	54.4	55.8	56.8	56.8	55.2	55.4
ML (1+8) @ 125°C	50.9	50.9	50.8	51.5	51.3	51.7	51.4	51.5
Box Number	2388	2390	2394	2397	2400	2403	2406	2409
ML (1+4) @ 100°C	76.4		76.7		77.1		76.8	
ML (1+8) @ 100°C	75.3		75.4		75.5		75.3	
ML (1+4) @ 125°C	56.1	55.5	55.5	55.1	54.9	55.1	55.6	54.9
ML (1+8) @ 125°C	51.5	51.5	51.4	51.4	51.3	51.4	51.4	51.2
Box Number	2412	2413	2414	2417	2420	2423	2426	2429
ML (1+4) @ 100°C	76.7		76.8		76.6		76.7	
ML (1+8) @ 100°C	75.3		75.3		75.3		75.3	
ML (1+4) @ 125°C	55.2	56.2	55.2	54.9	54.8	56.8	56.4	54.8
ML (1+8) @ 125°C	51.3	51.5	51.3	51.2	51.0	51.5	51.4	51.1
Box Number	2432	2435	2438	2441	2444	2447	2450	2453
ML (1+4) @ 100°C	76.7		76.7	76.6	76.5		76.4	
ML (1+8) @ 100°C	75.3		75.3	75.4	75.2		75.2	
ML (1+4) @ 125°C	55.3	55.0	55.7	55.1	56.0	55.0	54.7	55.2
ML (1+8) @ 125°C	51.2	51.2	51.3	51.1	51.3	51.1	51.0	50.9

Table 2D, Boxes 2454 to 2613								
Box Number	2456	2459	2462	2465	2468	2471	2474	2477
ML (1+4) @ 100°C	76.5		76.4		76.2		76.3	
ML (1+8) @ 100°C	75.2		75.1		75.0		75.0	75.3
ML (1+4) @ 125°C	57.4	56.0	55.3	57.1	56.0	56.6	55.1	55.4
ML (1+8) @ 125°C	51.8	51.7	51.4	51.8	51.6	51.7	51.3	51.2
Box Number	2480	2483	2486	2489	2492	2495	2498	2501
ML (1+4) @ 100°C	76.5		76.6		76.3		76.5	
ML (1+8) @ 100°C	75.3		75.1		75.0		75.2	
ML (1+4) @ 125°C	56.5	55.1	57.1	55.7	56.5	55.6	56.1	55.3
ML (1+8) @ 125°C	51.7	51.4	51.8	51.6	51.9	51.8	51.8	51.5
Box Number	2504	2507	2510*	2513	2516	2519	2522	2525
ML (1+4) @ 100°C	77.0		76.7		76.6		76.5	
ML (1+8) @ 100°C	75.4		75.5		75.5		75.2	
ML (1+4) @ 125°C	55.6	55.1	55.9	55.4	55.3	55.4	57.0	55.7
ML (1+8) @ 125°C	51.5	51.4	51.6	51.5	51.6	51.5	51.8	51.7
			*Used for ARV ITP					
Box Number	2528	2531	2534	2537	2538	2539	2542	2545
ML (1+4) @ 100°C	76.8		76.8		76.6		76.6	
ML (1+8) @ 100°C	75.6		75.4		75.2		75.4	
ML (1+4) @ 125°C	55.6	55.4	55.8	56.0	56.1	55.2	55.4	55.7
ML (1+8) @ 125°C	51.7	51.6	51.7	51.7	51.7	51.5	51.6	51.6
Box Number	2548	2551	2554	2557	2560	2563	2567	2569
ML (1+4) @ 100°C	77.5		76.9		76.7		76.6	
ML (1+8) @ 100°C	76.0		75.6		75.6		75.4	
ML (1+4) @ 125°C	56.4	55.5	55.5	56.6	57.6	55.5	55.8	55.5
ML (1+8) @ 125°C	51.8	51.6	51.6	51.9	52.1	51.6	51.9	51.7
Box Number	2572	2575	2578	2581	2584	2587	2590	2593
ML (1+4) @ 100°C	76.6		76.9		76.8		76.8	
ML (1+8) @ 100°C	75.4		75.6		75.7		75.5	
ML (1+4) @ 125°C	55.9	55.0	55.6	55.8	55.9	55.5	56.4	55.3
ML (1+8) @ 125°C	51.8	51.0	51.8	51.9	51.8	51.7	51.9	51.7
Box Number	2594	2595	2598	2601	2604	2607	2610	2613
ML (1+4) @ 100°C	76.6			76.6	76.8		77.2	
ML (1+8) @ 100°C	75.4			75.5	75.6		76.1	
ML (1+4) @ 125°C	55.4	55.7	54.9	57.1	55.1	56.2	56.1	55.2
ML (1+8) @ 125°C	51.7	51.7	51.0	51.5	51.1	51.9	51.9	51.6

Table 2E, Boxes 2614 to 2768								
Box Number	2616	2619	2622	2625	2628	2631	2634	2637
ML (1+4) @ 100°C	76.9		77.2		77.2		77.2	
ML (1+8) @ 100°C	75.6		75.7		75.8		75.8	
ML (1+4) @ 125°C	55.7	56.5	57.4	56.9	56.8	55.8	55.7	57.6
ML (1+8) @ 125°C	51.8	51.8	52.2	52.2	52.2	51.9	51.9	52.4
Box Number	2640	2643	2646	2649	2652	2655	2658	2661
ML (1+4) @ 100°C	76.8		76.8		76.9		77.0	
ML (1+8) @ 100°C	75.7		75.6		75.7		75.7	
ML (1+4) @ 125°C	56.4	56.4	55.0	55.1	55.1	57.3	57.1	55.5
ML (1+8) @ 125°C	52.1	51.6	51.4	51.3	51.4	51.8	51.8	51.4
Box Number	2664	2667	2670	2673	2676	2679	2682	2685
ML (1+4) @ 100°C	76.8		76.8		76.9		77.0	
ML (1+8) @ 100°C	75.7		75.5		75.7		75.7	
ML (1+4) @ 125°C	54.9	55.3	55.6	55.3	55.2	55.2	58.0	55.8
ML (1+8) @ 125°C	51.2	51.4	51.4	51.3	51.3	51.4	52.3	51.9
Box Number	2688	2690	2693	2696	2699	2702	2708	2711
ML (1+4) @ 100°C	76.9		77.4		76.8			76.9
ML (1+8) @ 100°C	75.6		75.8		75.7			75.7
ML (1+4) @ 125°C	55.8	56.4	56.4	56.3	56.1	55.9	55.4	55.7
ML (1+8) @ 125°C	51.8	51.9	52.0	51.8	51.8	51.9	51.7	51.8
Box Number	2714	2717	2720	2723	2726	2728	2729	2730
ML (1+4) @ 100°C		76.8		77.5	77.0		76.8	
ML (1+8) @ 100°C		75.5		76.2	75.6		75.7	
ML (1+4) @ 125°C	58.2	56.5	57.1	58.0	55.6	56.2	56.4	56.1
ML (1+8) @ 125°C	52.4	52.0	52.1	52.3	51.8	52.2	52.2	52.1
Box Number	2733	2734	2735	2736	2737	2738	2741	2744
ML (1+4) @ 100°C	76.8		77.0		76.3		77.0	
ML (1+8) @ 100°C	75.5		75.6		75.0		75.6	
ML (1+4) @ 125°C	55.6	57.2	55.8	54.9	55.2	54.8	55.7	55.9
ML (1+8) @ 125°C	51.9	52.3	52.2	51.4	51.6	51.4	52.1	52.1
Box Number	2747	2750	2753	2756	2759	2762	2765	2768
ML (1+4) @ 100°C	76.5		77.1		76.9		76.7	
ML (1+8) @ 100°C	75.3		75.7		75.6		75.5	
ML (1+4) @ 125°C	55.5	55.2	55.8	55.4	56.4	55.5	55.2	55.5
ML (1+8) @ 125°C	51.9	51.9	52.1	51.8	52.2	52.2	51.8	51.8

Table 2F, Boxes 2769 to 2934								
Box Number	2771	2774	2776	2779	2782	2785	2788	2791
ML (1+4) @ 100°C	77.0		76.7		76.7		77.0	
ML (1+8) @ 100°C	75.6		75.5		75.6		75.6	
ML (1+4) @ 125°C	55.5	55.6	55.3	55.2	55.7	54.5	55.7	58.1
ML (1+8) @ 125°C	51.8	51.9	51.9	51.8	51.9	51.3	52.0	52.5
Box Number	2794	2797	2800	2803	2806	2809	2812	2815
ML (1+4) @ 100°C	76.8		76.9		76.6		77.0	
ML (1+8) @ 100°C	75.6		75.5		75.1		75.8	
ML (1+4) @ 125°C	56.0	55.6	58.0	58.0	56.9	55.7	57.5	55.3
ML (1+8) @ 125°C	52.3	52.1	52.6	52.7	51.8	52.1	52.4	51.7
Box Number	2818	2821	2824	2827	2830	2833	2836	2838
ML (1+4) @ 100°C	76.4		77.1		76.4		76.4	
ML (1+8) @ 100°C	75.1		75.7		75.1		75.2	
ML (1+4) @ 125°C	55.2	56.2	56.2	56.6	55.7	55.8	55.7	57.0
ML (1+8) @ 125°C	51.6	51.8	52.4	52.3	51.7	52.1	51.6	52.4
Box Number	2841	2844	2847	2850	2853	2856	2859	2862
ML (1+4) @ 100°C	76.9		77.0		76.3		77.2	
ML (1+8) @ 100°C	75.6		75.9		75.1		75.7	
ML (1+4) @ 125°C	56.5	56.6	55.0	55.3	56.5	58.4	56.4	56.2
ML (1+8) @ 125°C	52.4	51.8	51.5	51.6	52.2	52.8	52.4	52.4
Box Number	2865	2868	2871	2874	2877	2880	2883	2886
ML (1+4) @ 100°C	76.9		77.0		77.4		76.9	
ML (1+8) @ 100°C	75.7		75.8		76.0		75.7	
ML (1+4) @ 125°C	56.0	56.4	56.0	56.1	56.1	56.7	59.9	56.7
ML (1+8) @ 125°C	52.3	52.4	52.2	52.2	52.2	52.4	53.2	52.7
Box Number	2889	2892	2895	2898	2901	2904	2907	2910
ML (1+4) @ 100°C	77.0		76.9		77.2		77.2	
ML (1+8) @ 100°C	75.7		75.7		75.8		75.9	
ML (1+4) @ 125°C	57.7	56.7	57.1	56.8	56.3	58.4	58.9	57.1
ML (1+8) @ 125°C	52.8	52.7	52.6	52.6	52.5	52.9	53.0	52.6
Box Number	2913	2916	2919	2922	2925	2928	2931	2934
ML (1+4) @ 100°C	77.0		77.2		77.1		77.2	
ML (1+8) @ 100°C	75.8		75.8		75.8		76.0	
ML (1+4) @ 125°C	57.5	57.0	57.0	56.8	56.6	56.8	56.7	56.7
ML (1+8) @ 125°C	52.8	52.8	52.7	52.7	52.6	52.7	52.5	52.6

Table 2G, Boxes 2935 to 3101								
Box Number	2937	2940	2943	2946	2949	2952	2955	2958
ML (1+4) @ 100°C	77.2		77.3		77.0		77.2	
ML (1+8) @ 100°C	75.8		75.8		75.8		75.9	
ML (1+4) @ 125°C	56.4	57.0	56.5	58.9	56.8	56.6	56.5	56.8
ML (1+8) @ 125°C	52.3	52.6	52.6	52.8	52.5	52.5	52.5	52.6
Box Number	2961	2964	2967	2970	2973	2976	2979	2982
ML (1+4) @ 100°C	77.2		77.2		77.1		77.4	
ML (1+8) @ 100°C	75.9		75.8		75.8		75.9	
ML (1+4) @ 125°C	57.2	56.7	56.8	56.5	56.1	56.7	56.6	56.6
ML (1+8) @ 125°C	52.9	52.3	52.7	52.4	52.4	52.8	52.6	52.6
Box Number	2985	2987	2990	2993	2996	2999	3002	3005
ML (1+4) @ 100°C	76.8		77.0		77.3		77.2	
ML (1+8) @ 100°C	75.7		75.7		75.8		75.9	
ML (1+4) @ 125°C	56.5	56.3	57.4	56.8	56.2	56.2	57.5	56.5
ML (1+8) @ 125°C	52.6	52.5	52.7	52.6	52.5	52.5	52.6	52.5
Box Number	3008	3011	3014	3017	3020	3023	3026	3029
ML (1+4) @ 100°C	77.1		77.3		77.2		77.0	
ML (1+8) @ 100°C	75.9		75.9		75.9		75.9	
ML (1+4) @ 125°C	56.9	56.9	58.0	59.1	56.7	56.9	56.3	56.3
ML (1+8) @ 125°C	52.6	52.4	52.7	52.9	52.6	52.5	52.4	52.2
Box Number	3032	3035	3038	3041	3044	3047	3050	3053
ML (1+4) @ 100°C	77.1		77.3		76.9		77.0	
ML (1+8) @ 100°C	75.7		75.8		75.9		75.8	
ML (1+4) @ 125°C	56.4	57.0	56.2	55.8	58.7	56.8	56.6	56.0
ML (1+8) @ 125°C	52.4	52.4	52.2	52.1	53.0	52.6	52.4	52.3
Box Number	3056	3059	3062	3065	3068	3071	3074	3077
ML (1+4) @ 100°C	77.0		76.9		77.1		77.2	
ML (1+8) @ 100°C	75.8		75.8		75.7		75.9	
ML (1+4) @ 125°C	56.7	57.2	58.1	58.0	56.2	58.5	56.8	56.1
ML (1+8) @ 125°C	52.4	52.5	52.5	52.5	52.3	52.7	52.4	52.3
Box Number	3080	3083	3086	3089	3092	3095	3098	3101
ML (1+4) @ 100°C	77.0		77.2		76.9		76.9	
ML (1+8) @ 100°C	75.7		75.9		75.8		75.7	
ML (1+4) @ 125°C	56.3	56.3	56.1	55.9	57.6	56.6	56.1	56.4
ML (1+8) @ 125°C	52.3	52.0	52.1	52.0	52.7	51.9	52.4	52.0

Table 2H, Boxes 3102 to 3269								
Box Number	3104	3107	3110	3113	3116	3119	3122	3125
ML (1+4) @ 100°C	77.0		77.8		76.8		77.0	
ML (1+8) @ 100°C	75.9		76.4		75.6		75.6	
ML (1+4) @ 125°C	56.2	56.7	56.2	55.8	56.2	55.5	57.0	56.1
ML (1+8) @ 125°C	51.8	52.4	51.9	51.7	52.3	51.9	52.4	52.1
Box Number	3128	3131	3134	3137	3140	3143	3146	3149
ML (1+4) @ 100°C	77.1		76.8		77.0		76.9	
ML (1+8) @ 100°C	75.7		75.4		75.6		75.6	
ML (1+4) @ 125°C	56.2	56.0	55.3	55.5	58.2	55.2	55.5	55.0
ML (1+8) @ 125°C	52.2	52.2	51.8	51.8	52.7	51.8	51.9	51.6
Box Number	3152	3155	3158	3161	3164	3167	3170	3173
ML (1+4) @ 100°C	76.9		76.9		76.9		76.8	
ML (1+8) @ 100°C	75.7		75.6		75.6		75.7	
ML (1+4) @ 125°C	56.7	57.1	56.6	56.9	56.3	56.1	55.9	56.3
ML (1+8) @ 125°C	52.6	52.7	52.4	52.4	52.4	52.2	52.2	52.2
Box Number	3176	3179	3182	3185	3188	3191	3194	3197
ML (1+4) @ 100°C	76.9		76.9		76.9		77.0	
ML (1+8) @ 100°C	75.5		75.7		75.7		75.6	
ML (1+4) @ 125°C	56.0	56.2	56.6	55.5	55.7	55.0	56.5	57.8
ML (1+8) @ 125°C	52.1	52.2	52.3	51.9	52.1	51.7	52.3	52.5
Box Number	3200	3203	3206	3209	3212	3215	3218	3221
ML (1+4) @ 100°C	77.0		76.8		77.0		76.7	
ML (1+8) @ 100°C	75.6		75.6		75.7		75.5	
ML (1+4) @ 125°C	55.8	55.9	55.8	55.7	56.5	56.8	55.6	55.7
ML (1+8) @ 125°C	52.0	52.1	52.1	52.0	52.3	52.1	51.9	51.9
Box Number	3224	3227	3230	3233	3236	3239	3242	3245
ML (1+4) @ 100°C	77.0		76.9		76.8		76.9	
ML (1+8) @ 100°C	75.8		75.6		75.6		75.6	
ML (1+4) @ 125°C	55.6	55.6	55.9	56.8	55.5	56.4	56.8	55.6
ML (1+8) @ 125°C	51.9	52.0	51.9	52.0	51.9	51.9	52.3	51.9
Box Number	3248	3251	3254	3257	3260	3263	3266	3269
ML (1+4) @ 100°C	76.7		76.9		76.8		77.2	
ML (1+8) @ 100°C	75.6		75.6		75.5		75.9	
ML (1+4) @ 125°C	58.2	56.6	56.5	56.0	56.7	55.6	55.5	57.7
ML (1+8) @ 125°C	52.4	52.2	52.1	52.1	52.1	51.9	51.9	52.3

Table 21, Boxes 3270 to 3427								
Box Number	3272	3275	3278	3281	3284	3287	3290	3293
ML (1+4) @ 100°C	77.1		76.9		76.7		76.9	
ML (1+8) @ 100°C	76.0		75.6		75.6		75.5	
ML (1+4) @ 125°C	55.7	55.6	56.1	56.4	55.6	56.5	55.7	56.7
ML (1+8) @ 125°C	51.8	51.7	52.0	51.9	51.7	51.8	51.8	51.9
Box Number	3296	3299	3302	3305	3308	3311	3314	3317
ML (1+4) @ 100°C	76.8		76.9		76.7		76.8	
ML (1+8) @ 100°C	75.6		75.6		75.5		75.4	
ML (1+4) @ 125°C	56.0	55.9	55.3	55.1	57.3	56.5	55.9	55.6
ML (1+8) @ 125°C	51.9	51.8	51.5	51.5	52.4	52.1	52.0	51.9
Box Number	3320	3323	3326	3329	3332	3335	3338	3341
ML (1+4) @ 100°C	76.9		76.7		76.9		76.9	
ML (1+8) @ 100°C	75.5		75.3		75.5		75.6	
ML (1+4) @ 125°C	55.6	55.4	56.1	55.5	55.4	55.8	56.4	56.5
ML (1+8) @ 125°C	51.9	51.8	52.0	51.9	51.8	51.9	51.7	51.8
Box Number	3344	3346	3349	3352	3355	3358	3361	3364
ML (1+4) @ 100°C	76.9		76.8		77.2		76.6	
ML (1+8) @ 100°C	75.6		75.5		75.5		75.4	
ML (1+4) @ 125°C	55.3	57.7	57.1	55.4	55.3	56.6	56.8	56.6
ML (1+8) @ 125°C	51.7	52.0	52.2	51.8	51.7	52.1	52.1	52.0
Box Number	3367	3370	3373	3376	3379	3382	3385	3388
ML (1+4) @ 100°C	77.0		77.0		77.0		76.9	
ML (1+8) @ 100°C	75.6		75.5		75.6		75.6	
ML (1+4) @ 125°C	55.7	55.7	55.7	57.6	55.9	55.8	55.7	55.5
ML (1+8) @ 125°C	51.9	51.7	51.7	52.1	51.8	51.7	51.8	51.6
Box Number	3391	3394	3397	3400	3403	3406	3409	3412
ML (1+4) @ 100°C	77.3		76.9		77.3		77.1	
ML (1+8) @ 100°C	75.8		75.8		76.1		75.8	
ML (1+4) @ 125°C	57.0	55.5	55.7	55.8	55.5	55.1	54.9	56.8
ML (1+8) @ 125°C	52.0	51.7	51.7	51.7	51.7	51.6	51.5	52.0
Box Number	3415	3418	3421	3424	3427			
ML (1+4) @ 100°C	76.8		77.0		76.9			
ML (1+8) @ 100°C	75.7		75.8		75.9			
ML (1+4) @ 125°C	55.6	55.5	55.9	55.5	55.5			
ML (1+8) @ 125°C	51.7	51.8	51.9	51.6	51.7			

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 2028 from IRM241 Lot H.

Step 1: Find the AR value for box 2028 for the ML (1 + 4) @ 125°C test.

From Tables 2A to 2I, find the table whose range of boxes includes box number 2028. Box 2028 is found in Table 2A. Box 2028 is one of the boxes listed in the table. Read down the column under box number 2028 to the cell that intersects the ML (1 + 4) @ 125°C test row to find the AR value of 55.4.

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 1.49.

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

The Lower Control Limit (LCL) = $AR - 2 \text{ sigma} = 55.4 - 1.49 = 53.91$

The Upper Control Limit (UCL) = $AR + 2 \text{ sigma} = 55.4 + 1.49 = 56.89$

The 2 sigma control limits for the ML (1 + 4) @ 125°C test for box 2028 are 53.91 to 56.89.

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 given in table but value for test of interest is not given and is bracketed by boxes with values for the test of interest:

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 2403 from IRM241 Lot H.

Step 1: Determine the AR value for box 2403 for the ML (1 + 8) @ 100°C test.

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 2I, find the table that includes box 2403. This is found in Table 2C. In the table, find the box numbers that bracket box number 2403. Box 2403 is bracketed by the columns for box 2400 and box 2406. Read down the column under box number 2400 to the cell that intersects the ML (1 + 8) @ 100°C test row to find the AR value of 75.5.

Read down the column under box number 2406 to the cell that intersects the ML (1 + 8) @ 100°C test row to find the AR value of 75.3. The absolute difference between box 2400 and box 2406 test values is 0.2 (75.5 – 75.3). Starting with box 2400, six boxes are counted to get to box 2406. The absolute difference between the boxes’ test values must be apportioned across the six boxes. Therefore, $0.2/6 = 0.033$ is to be apportioned to each of the six boxes. The determined AR value for box 2403 must be between the known AR values for the two bracketing boxes. Since the test value for box 2400 is higher than that of box 2406, the determined AR value for box 2403 will be lower than box 2400 and higher than box 2406. Box 2403 is halfway between boxes 2400 and 2406 (2400 to 2403 is 3 boxes and 2403 to 2406 is 3 boxes). The AR for box 2403 can be determined as an offset from box 2400 or 2406. Using box 2400 for the calculation, the count difference between box 2403 and box 2400 is 3 boxes. Therefore, the AR for box 2403 can be determined as $75.5 - (0.033 * 3) = 75.401$, which rounds to the nearest 0.1 Mooney unit to 75.4. The determined AR value for box 2403 is 75.4. A similar calculation could have been used for box 2406 except the adjustment would have been added to the value (75.3) for box 2406. Either calculation will give the same AR value to 1 decimal place.

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 x Sr” column. Read down the “2 x Sr” column to the cell that intersects the ML (1 + 8) @ 100°C test row to find the 2 sigma value of 0.58.

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

$$\text{The Lower Control Limit (LCL)} = \text{AR} - 2 \text{ sigma} = 75.4 - 0.58 = 74.82$$

$$\text{The Upper Control Limit (UCL)} = \text{AR} + 2 \text{ sigma} = 75.4 + 0.58 = 75.98$$

The 2 sigma control limits for the ML (1 + 8) @ 100°C test for box 2400 are 74.82 to 75.98.

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 is bracketed by boxes with values for the test of interest:

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 H.

Step 1: Determine the AR value for box 2620 for the ML (1 + 8) @ 125°C test.

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 2I, find the table whose range of boxes includes box 2620. This is found in Table 2E. In the table, find the box numbers that bracket box number 2620. Box

2620 is bracketed by the columns for box 2619 and box 2622. Values are given for both box 2619 and box 2622. Read down the column under box number 2619 to the cell that intersects the ML (1 + 8) @ 125°C test row to find the AR value of 51.8. Read down the column under box number 2622 to the cell that intersects the ML (1 + 8) @ 125°C test row to find the AR value of 52.2. The absolute difference between box 2619 and box 2622 test values is 0.4 (51.8 – 52.2). Starting with box 2619, three boxes are counted to get to box 2622. The absolute difference between the boxes' test values must be apportioned across the three boxes. Therefore, $0.4/3 = 0.133$ is to be apportioned to each of the three 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 2619 is lower than that of box 2622, the determined AR value for box 2620 will be higher than box 2619 and lower than box 2622. Box 2620 is closer by count to box 2619 so the AR for box 2620 will be determined as an offset from box 2619. The count difference between box 2619 and box 2620 is 1 box. Therefore, the AR for box 2620 can be determined as $51.8 + (0.133 * 1) = 51.933$, which rounds to the nearest 0.1 Mooney unit to 51.9. The determined AR value for box 2620 is 51.9. A similar calculation could have been used for box 2622 except the adjustment would have been subtracted from the value (52.2) for box 2622 and the box count between 2620 and 2622 is 2. Either calculation will give the same AR value to 1 decimal place.

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°C test row to find the 3 sigma value of 1.52.

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

The Lower Control Limit (LCL) = AR – 3 sigma = 51.9 – 1.52 = 50.38

The Upper Control Limit (UCL) = AR + 3 sigma = 51.9 + 1.52 = 53.42

The 3 sigma control limits for the ML (1 + 8) @ 125°C test for box 2620 are 50.38 to 53.42.

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.

Background and Interlaboratory Test Program Details: IRM241 Lot H 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 eighth lot of Butyl rubber IRM, designated as IRM241 Lot H, was produced by ExxonMobil Chemical Co. at their Baytown, TX facility in June 2022 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 July 2022. A lot of 1425 bales was found to be uniform in properties using the D4678 Annex A3 procedure.

Interlaboratory Test Program (ITP) - Seven companies using a total of fourteen Mooney instruments each tested IRM241 Lot H 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 September to early November 2022. The data was analyzed per D4678, annex A4. The material was accepted as being sufficiently uniform and suitable for use as an IRM at the December 2022 meeting of the ASTM D11.20.02 IRM task group.

Using IRM241 Lot H - 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.

Supplementary Information - 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 H will be on file at ASTM headquarters (expected after June 2023), 100 Barr Harbor Dr., W. Conshohocken, PA 19428, USA; Phone 610-832-9500, Fax 610-832-9555.