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BDX-613-1880 (Rev.)

SHELF LIFE AGING OF DC-302
SILICONE MOLDING COMPOUND

By J. W. McFarland

Published August 1978

Topical Report

Prepared for the United States Department of Energy
Under Contract Number EY-76-C-04-0613.

**Kansas City
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Topical Report
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BDX-613-1880 (Rev.), UNCLASSIFIED Topical Report,
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DC-302 silicone molding compound (Dow Corning Corporation) was packaged 10 different ways, stored at three different temperatures, and tested for spiral flow periodically for 8 months. The material was very stable at 5°C and fairly stable at 24°C. It was sensitive to heat, however, and was unstable at 55°C. Samples that were stored for 12 months at 5°C showed no significant change in physical properties.

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SUMMARY

A shelf life study of DC-302 silicone molding compound (Dow Corning Corporation) was conducted to determine what effect the type of packaging and the storage temperature have on spiral flow. In addition, material that was stored at 5°C was tested periodically over a 12-month period for tensile, flexural, and impact strengths.

Spiral flow tests determined that DC-302 was very sensitive to temperatures above 24°C. Material that was stored at 55°C in an open container had a spiral flow of 368.3 mm at the beginning and 76.2 mm after 58 days. However, material that was stored at 55°C in a sealed, vapor barrier bag--but not evacuated--had a spiral flow of 76.2 mm after only 14 days. Apparently, if the evolved vapors cannot escape, they will attack the DC-302.

Four nonevacuated samples were stored at 24°C under the following conditions: polyethylene bag, closed fiber drum, open fiber drum, and a sealed vapor barrier bag. One sample was evacuated at 399 Pa and stored at 24°C in a sealed vapor barrier bag. All four of the nonevacuated samples suffered an initial drop in spiral flow and then stabilized. After 8 months, all four of them had the same spiral flow of 266.7 mm, compared to 368.3 mm at the beginning. The material that was evacuated at 399 Pa and then stored at 24°C had exactly the same spiral flow (368.3 mm) at the end of 8 months as it did at the beginning. The evacuation step apparently removed some of the catalyst.

DC-302 samples were stored at 5°C in a polyethylene bag, a closed fiber drum, and a sealed, evacuated, vapor barrier bag. All three samples had a spiral flow of 368.3 mm at the beginning. Of these, the material stored in the fiber drum was the only one to show a drop in spiral flow, and it was only a small drop. The sample stored in the polyethylene bag ran exactly the same at the end as it did at the beginning, and the sample that had been evacuated showed an increase in spiral flow. Again the evacuation seemed to pull out some of the catalyst.

For 12 months DC-302 that was stored at 5°C was tested for tensile strength, flexural strength, and impact strength. During this time the tensile strength decreased from 33.44 to 32.54 MPa, the flexural strength increased from 77.91 to 85.49 MPa, and the impact strength decreased from 2.37 to 1.99 J/cm. These variations are not considered significant, and it was concluded that the physical properties did not change significantly during the 12-month period.

DISCUSSION

SCOPE AND PURPOSE

The purpose of this project was to investigate the effect that packaging and storage conditions have on the shelf life of Dow Corning's DC-302 silicone molding compound. In this study the material was packaged in open drums, closed drums, polyethylene bags, and vapor barrier bags at temperatures of 5°, 24°, and 55°C.

A series of mechanical tests was included in the scope. The material was tested periodically for tensile, flexural, and impact strengths. These tests were continued for approximately 12 months.

PRIOR WORK

No prior work has been done on the shelf life of DC-302.

ACTIVITY

Spiral Flow Characteristics

DC-302 (lot number BJ 076030) was packaged under 10 different conditions which are listed in Table 1 as Conditions A through I and K. When it was seen that Condition G would run out of material while the spiral flow was still quite high, another test was started which duplicated Condition G but which was labeled Condition J.

As seen in Table 1, the material was packaged in open drums, closed drums, polyethylene bags, and vapor barrier bags at temperatures of 5°, 24°, and 55°C. Each type of package was not necessarily represented at each temperature. The vapor barrier bag is a bag that can be heat sealed. It consists of an inside layer of polyethylene a middle layer of aluminum sheet and an outside layer of canvas.

Spiral flow tests were run periodically for 8 months. All of the tests could not be completed on the same day because of manpower and equipment limitations. However, at the end of the designated time period, the tests were completed as quickly as possible--usually within 3 days. The spiral flow was run in the Mesa mold. Testing was continued until the flow was 76 mm or until material ran out.

Table 1. Storage Conditions for DC-302

Storage Condition	Type of Bag and Storage	Temperature (°C)
A	Closed polyethylene bag	5
B	Closed polyethylene bag	24
C	Closed fiber drum	24
D	Sealed, vapor barrier bag	55
E	Open fiber drum	24
F	Sealed, vapor barrier bag	24
G	Open cardboard carton	55
H	Sealed, vapor barrier bag. Bag was evacuated before sealing.	24
I	Sealed, vapor barrier bag. Bag was evacuated before sealing.	5
J	Open cardboard carton (Repeat of G)	55
K	Closed fiber drum	5

Results of the spiral flow tests (Table 2) show that DC-302 is very sensitive to temperatures above 24°C (room temperature) and that it is very stable at temperatures below 24°C. At room temperature all four of the materials that were not evacuated suffered an initial drop of 50 to 75 mm and then stabilized. The evacuated material stored at room temperature (Condition H) had a higher spiral flow after 8 months than it did at the beginning.

In Figure 1 the results are grouped according to the temperatures at which the materials were stored. Figure 1 shows the spiral flow of DC-302 stored at 5°C. Material stored in a polyethylene bag (Condition A) was exactly the same after 6 months as it was at the beginning. This test was discontinued at this time because there was no more material left.

Table 2. Spiral Flow of DC-302

Time (Days)	Spiral Flow (mm)	Time (Days)	Spiral Flow (mm)	Time (Days)	Spiral Flow (mm)
Condition A		Condition B		Condition C	
0	368.3	0	368.3	0	368.3
15	381.0	11	292.1	11	330.2
22	393.7	15	292.1	15	330.2
32	393.7	18	279.4	18	342.9
39	393.7	25	292.1	22	330.2
84	368.3	30	279.4	25	317.5
115	381.0	37	292.1	29	330.2
154	393.7	44	279.4	35	317.5
190	368.3	84	266.7	42	304.8
		115	279.4	85	292.1
		162	279.4	116	304.8
		190	254.0	163	292.1
		238	266.7	191	292.1
				238	266.7
Condition D		Condition E		Condition F	
0	368.3	0	368.3	0	368.3
1	279.4	9	292.1	10	304.8
2	215.9	11	279.4	14	292.1
5	152.4	14	279.4	17	292.1
6	127.0	16	279.4	21	304.8
7	127.0	18	279.4	24	292.1
8	114.3	21	292.1	30	292.1
9	101.6	25	292.1	37	304.8
12	88.9	29	266.7	44	292.1
13	88.9	35	279.4	85	279.4
14	76.2	42	279.4	116	292.1
		85	266.7	163	266.7

Table 2 Continued. Spiral Flow of DC-302

Time (Days)	Spiral Flow (mm)	Time (Days)	Spiral Flow (mm)	Time (Days)	Spiral Flow (mm)
Condition D		Condition E		Condition F	
		116	292.1	191	254.0
		163	279.4	239	266.7
		191	266.7		
		239	266.7		
Condition G		Condition H		Condition I	
0	368.3	0	368.3	0	368.3
2	266.7	10	368.3	9	368.3
5	266.7	14	368.3	16	381.0
7	254.0	17	381.0	23	393.7
9	241.3	21	342.9	32	381.0
12	228.6	24	368.3	39	393.7
16	215.9	30	381.0	85	381.0
		37	381.0	116	381.0
		44	368.3	164	355.6
		85	355.6	191	368.3
		116	355.6	240	381.0
		164	342.9		
		191	330.2		
		239	368.3		
Condition J		Condition K			
0	368.3	0	368.3		
4	266.7	35	330.2		
7	254.0	42	317.5		
11	241.3	86	304.8		
14	228.6	116	330.2		
18	266.7	164	317.5		
21	254.0	192	317.5		

Table 2 Continued. Spiral Flow of DC-302

Time (Days)	Spiral Flow (mm)	Time (Days)	Spiral Flow (mm)	Time (Days)	Spiral Flow (mm)
Condition J		Condition K			
25	241.3	240	342.9		
28	203.2				
30	215.9				
34	215.9				
41	266.7				
48	215.9				
58	76.2				
79	50.8				

DC-302 stored at 5°C in the closed fiber drum (Condition K) dropped from 368.3 mm to 342.9 mm after 8 months. This drop of 25.4 mm is not a large drop. Material that had been evacuated for 1 hour at 399 Pa and stored at 5°C (Condition I) had a spiral flow of 381 millimeters, compared to 368.3 millimeters at the beginning which is an increase of 12.7 millimeters.

Figure 1 also shows material stored at 24°C (room temperature) and packaged five different ways. The following packaging methods for Conditions B, C, E, and F were used: closed polyethylene bag, closed fiber drum, open fiber drum, and sealed vapor barrier bag--not evacuated. In Condition H the material was evacuated for 1 hour at 399 Pa and then stored at 24°C in a sealed vapor barrier bag. All of the nonevacuated samples (Conditions B, C, E, and F) had a spiral flow of 266.7 mm after 8 months. This is a drop of 101.6 mm and is not considered to be excessive for material stored at room temperature for 8 months. Moreover, most of the drop took place within the first 45 days.

The sample that was evacuated at 399 Pa and then sealed in a vapor barrier bag and stored at room temperature had exactly the same spiral flow (368.3 mm) at the end of the 8 months as it did at the beginning. This is Condition H and is shown as the top line. Apparently, the evacuation step removed part of the curing agent from the DC-302.

Figure 1. Shelf Life of Material Stored at Three Different Temperatures

Figure 1 Continued. Shelf Life of Material Stored at Three Different Temperatures

Two samples stored at 55°C are shown in Figure 1. Condition D was material sealed in a vapor barrier bag but not evacuated; Condition J material was stored in an open container. The 55°C condition shows an interesting phenomenon. Material stored in a sealed bag at 55°C had a spiral flow of 76.2 millimeters after 14 days, but material that was left open at 55°C lasted for 58 days before the spiral flow dropped to 76.2 mm. It appears that when DC-302 is heated, something evolves that will attack the DC-302 itself if the DC-302 and the evolved product remain in contact with each other.

Physical Properties

DC-302 stored for 12 months at 5°C (lot number BJ 016007) was tested periodically for tensile, flexural, and impact strengths. The test methods and test results are shown in Table 3. For the first 7 months the material was tested every month; after that it was tested on the ninth month and the twelfth month. This was done because the supply of material was running low, and it was desired to run the study a full 12 months.

Table 3. Physical Properties of DC-302

Time (Months)	Tensile Strength ASTM D 651 (MPa)	Flexural Strength ASTM D 790 (MPa)	Impact Strength ASTM D 256 (J/cm)
0	33.44	77.91	2.37
1	37.99	73.64	1.83
2	32.68	83.29	2.90
3	33.78	70.19	2.30
4	33.51	78.60	2.32
5	35.78	69.77	2.08
6	33.92	82.60	1.96
7	35.23	74.81	2.07
9	30.61	91.98	1.99
12	32.54	85.49	---

In 12 months the tensile strength decreased from 33.44 to 32.54 MPa, the flexural strength increased from 77.91 to 85.49 MPa, and the impact strength decreased from 2.37 to 1.99 J/cm. As none of these changes is considered significant, it is concluded that the physical characteristics of the material changed very little over a period of 12 months.

ACCOMPLISHMENTS

It has been shown that the shelf life of DC-302 is quite good when the material is stored in a cold box at 5°C. It was also shown that the material is fairly stable at room temperature, but not as stable as it is at 5°C.

We have determined that DC-302 is very sensitive to heat. The spiral flow of material stored in an open container at 55°C decreased from 368.3 to 76.2 mm in 58 days. However, nonevacuated material sealed in a vapor barrier bag decreased from 368.3 to 76.2 mm in only 14 days. Apparently, when nonevacuated DC-302 is heated in a sealed container, the evolved products, which cannot escape, will attack the DC-302.

It was found that material stored at 5°C for 12 months showed very little change in tensile, flexural, or impact strengths.

The tensile and impact strengths decreased slightly while the flexural strength increased slightly, these changes are not considered to be significant.

The analysis indicates that, for at least 12 months, the physical properties of DC-302 change very little with time when stored at 5°C.

FUTURE WORK

No more work is planned on the shelf life of DC-302. The shelf life at three different temperatures has been studied and defined. This information will be sufficient unless there is a change in the formulation.

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SHELF LIFE AGING OF DC-302 SILICONE MOLDING COMPOUND, J. W. McFarland, UNCLAS Topical, August 1978.

DC-302 silicone molding compound (Dow Corning Corporation) was packaged 10 different ways, stored at three different temperatures, and tested for spiral flow periodically for 8 months. The material was very stable at 5°C and fairly stable at 24°C. It was sensitive to heat, however, and was unstable at 55°C. Samples that were stored for 12 months at 5°C showed no significant change in physical properties.

PLASTICS: Silicone Shelf Life

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