

Particle Shedding and Metal Extraction from Furon® Q Valves

Furon Q valves (QV2-812NC) were tested by an independent lab¹ to verify that they do not contaminate process chemicals with particles or metal ions. Four Q valves were tested for passive particle shedding during initial flush up and for particle shedding during cycling using the protocol of a major North American OEM of semiconductor process equipment. The DyconE^x SM dynamic extraction procedure² was used to measure the type and rate of metal ion extraction from a second set of four valves.

Particle Shedding-Test Method

Tests were conducted in a Class 100 cleanroom. The test stand, equipped with a PMS Liquistat 100 Optical Particle Counter, was flushed until background levels were ≤ 0.05 particles ($\geq 0.10 \mu$ diameter) for four or more hours before each test.

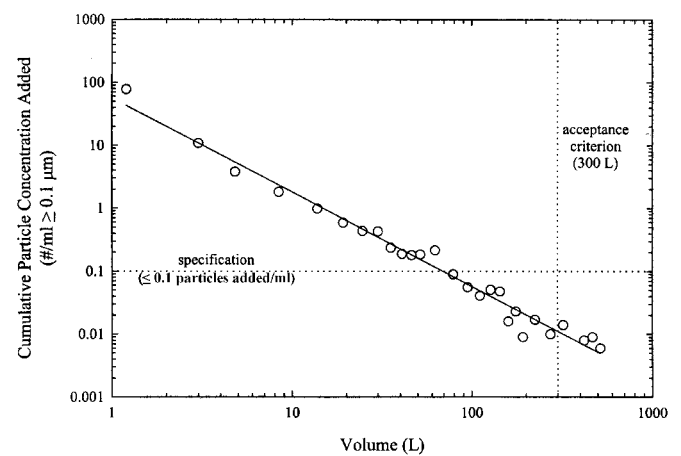
Individual valves were installed in a test system, opened, and flushed with ultrapure water at 600 ml/min. Pressure downstream of the valves was maintained at 30 psig during the tests. The concentration of particles downstream of the valves was monitored continuously.

After all the valves had been tested, the system was modified to accommodate two valves in parallel. The paired valves were cycled in an overlapping open/close cycle so that the 250 ml/min flow through the system was maintained. The test continued for 10,000 cycles.

Results

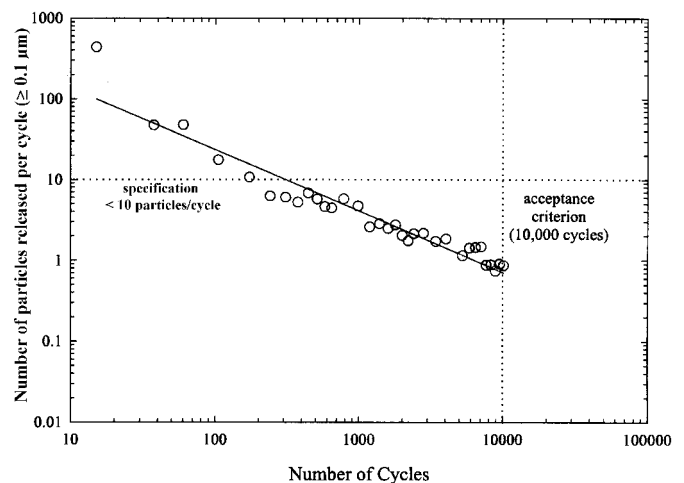
The average performance of the four valves during the flush up test is shown in Figure 1. Reference lines show the concentration specification, < 0.1 particle/ml added (horizontal line), and the acceptance criterion, < 300 liters (vertical line), by which the specification must be met. Linear regression of the averaged values is shown. The valves added < 0.1 particle/ml within 69 liters of flushing, significantly fewer than the criterion of ≤ 300 liters.

Figure 1: Particle shedding during flush up tests



Data from the cycle test are shown in Figure 2. The specification of ≤ 10 particles/cycles (horizontal line) and the acceptance criterion $< 10,000$ cycles (vertical line) are included. On average, the Q valves reached the specification after only 308 cycles.

Figure 2: Particles released during the cycle tests



Metal Extraction

Test method: A dynamic extraction system was filled with 0.97 liters of 35% baseline grade HCl. Four valves were installed in the circulation loop. HCl was circulated through the system at 1 liter/min for 12 days. A sample was withdrawn before flow through the valves was initiated to determine background levels of trace metals.

An acid sample was taken after 40 min to determine the mass of metal extracted from the wetted surface of the valves. Additional samples, taken at 2 hrs, 8 hrs, 2 days, and 12 days, were used to determine the masses and rates of metal extraction from the valves over time. All samples were analyzed for 37 metals by inductively coupled plasmas mass spectroscopy (ICP-MS) or graphite furnace atomic absorption (GFAA) spectroscopy

Results

Metal extraction data were normalized to the wetted area of a single valve. Table 1 shows the average mass of surface metal that was extracted per valve. Iron accounted for 70% of the contamination extracted. The total mass extracted was 1.55 µg/valve. This is less than the specification of 2.15 µg/valve set by a major manufacturer of chemical delivery systems.

Table 1: Masses of individual metals extracted from the surface of a valve by 35% HCl

<i>Element</i>	<i>Mass Extracted (µg/valve)</i>
<i>Fe</i>	<i>1.09</i>
<i>Al</i>	<i>0.09</i>
<i>Cr</i>	<i>0.09</i>
<i>Cu</i>	<i>0.08</i>
<i>Ni</i>	<i>0.07</i>
<i>Total of 32 other metals</i>	<i>0.13</i>

Figure 3 shows the total mass of all 37 metals extracted over time. The data were normalized to wetted surface area. The extraction rate at 7 days calculated from these data was 0.14 ng/cm²-day. This extraction rate was well below the 0.5 ng/cm²-day specification set by a major chemical distribution system manufacturer for delivery system components.

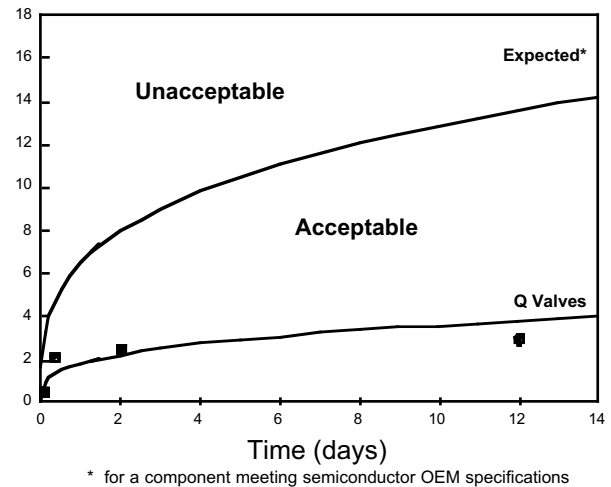


PERFORMANCE PLASTICS

Saint-Gobain Performance Plastics - USA
7301 Orangetown Avenue
Garden Grove, CA 92841-1411
Tel: (714) 630-5818 Fax: (714) 688-2614
USA Toll Free: (800) 833-5661

Saint-Gobain Performance Plastics-France
41 Boulevard des Bouvets
Nanterre F-92000, France
Tel: 33 / (0) 1 55 68 59 59 Fax: 33 / (0) 1 55 68 59 68

Figure 3: Cumulative total metal extraction during a 12-day dynamic extraction test



Summary

Furon Q valves were tested for particle cleanliness according to the protocol of a leading North American OEM manufacturer. During a passive flush up test in ultrapure water, the valves reached the acceptance criterion of < 0.1 particle/ml added in 69 liters, considerably less than the required ≤ 300 liters. During cycling, the valves averaged < 10 particles/cycle shed after 308 cycles, far fewer than the requirement of ≤ 10,000 cycles.

The valves were also tested for extraction of 37 metals by 35% HCl. The surface metal contamination of a single valve was 1.55 µg/valve, less than the specification of 2.15 µg/valve set by a major chemical distribution system manufacturer. The calculated extraction rate at 7 days was 0.14 ng/cm²-day. This rate is far less than the specification for metal extraction rate, 0.5 ng/cm²-day.

References

Grant DC, T Lemke and D Carrieri, "Specification and verification of metallic extractables in fluid handling components by dynamic extraction," in Proceedings of the Semicon West Workshop on Contamination in Liquid Chemical Distribution Systems," July, 1997.

Grant DC, T Lemke, G Duepner, D Wilkes, and N Powell, "Measurement of Inorganic Contaminant Extraction from Fluid Handling Components by Dynamic Extraction," J of the IES, 39(2): 29-37, 1996.

¹ CT Associates, Inc., 10777 Hampshire Ave. S., Bloomington, MN 55438.

² The Dycon^{ExSM} procedure is patented by BOC Edwards Chemical Management Division (US patent No. 5,641,895).

The data provided here were obtained under defined test conditions. The tests were designed to mimic use or worst case conditions. However, Saint-Gobain Performance Plastics makes no specific claims about the performance of the valves in other chemicals or systems.