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Outgassing Measurements on RTV S691 Silicone Adhesive

Prepared for:

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ASTM E 1559
Outgassing/Deposition Kinetics Test

Test Method
The material sample is placed in a temperature-controlled effusion cell in a vacuum chamber. Outgassing flux leaving the effusion cell orifice impinges on four QCMs which are controlled at selected temperatures. One of the QCMs is at 80 K to collect essentially all the impinging species. The total mass loss (TML) and outgassing rate from the sample are determined as functions of time from the mass deposited on this QCM and the sample-to-QCM view factor. The percent of outgassing species which are condensable on higher temperature surfaces is referred to as Volatile Condensable Material (VCM) and is measured as a function of time from the mass collected on the warmer QCMs, which are temperature-controlled appropriately. The QCMs and effusion cell are surrounded by liquid nitrogen shrouds to ensure that the molecular flux impinging on the QCMs is due only to the sample in the effusion cell.

After the isothermal outgassing test, a QCM thermogravimetric analysis (QTGA) is performed on the collected outgassed species. The QCMs are heated at a controlled rate from their base temperatures to 398 K in order to volatilize the collected species. During this QCM heat-up the mass remaining on the QCMs is measured as a function of time and temperature.

In general, the species condensed on the QCMs have different evaporation characteristics (volatilities) and hence will leave a QCM surface at different temperatures during QTGA. Therefore, QTGA data are characterized by temperature regimes in which the deposit mass remaining on the QCM decreases due to evaporation of a particular species, separated by temperature regimes in which no species evaporate. The number of temperature regimes in which species are evaporating from the QCM indicates the number of major groups of species that were present in the outgassing flux. The relative amount of a given species in the outgassing flux can be estimated from the ratio of the mass loss associated with the evaporation of that species to the total deposit mass on the QCM. QTGA also provides an effective means for cleaning the QCM surfaces before subsequent outgassing tests.

The species outgassed from the sample during the isothermal test, and evaporating from the QCM during the QTGA also are monitored using a mass spectrometer. While the QCMs provide quantitative outgassing and deposition data, the mass spectrometer records the intensities of mass peaks which aid in the identification of the outgassed species.

Test Parameters
Outgassing testing was performed using the following chamber and test parameters.

- Chamber pressures were 10^{-10} to 10^{-8} torr
- View factor from a QCM to the sample was 415.02 cm^2
- Sensitivity of each of the four QCMs was 4.43×10^{-9} $g/cm^2/Hz$

References
° ASTM E 1559, "Standard Test Method for Contamination Outgassing Characteristics of Spacecraft Materials."

° J.W. Garrett, A.P.M. Glassford, and J. M. Steakley, "ASTM E1559 Method for Measuring Material Outgassing/Deposition Kinetics", Journal of the IEST, pp. 19-28, Jan/Feb 1995

° A.P.M.Glassford and J.W.Garrett, "Characterization of Contamination Generation Characteristics of Satellite Materials", Final Report WRDC-TR-89-4114, Jun 82 - Aug 89

ASTM E 1559
Outgassing/Deposition Kinetics Test

Test Sample:
RTV S691 Silicone Adhesive.

Material Description:
The material consisted of a single piece of reddish-brown RTV S691 silicone adhesive manufactured by Wacker Chemical.

Material Packaging:
The material was wrapped in foil then placed in a clear plastic zip-lock. The bag was labeled "RTV-S691". Also a label was placed inside the zip-lock along with the sample. The sample was labeled "Pretest, Feb./3/2006, Outgassing Rate Test, Sample: RTV S691, Sample Mass: 5458.6 mg, (1 pc.), Sample Temperature: 60°C, QCMs Temperature, QCM 1: -183°C, QCM 2: -40°C, QCM 3: 0°C, QCM 4: 20°C, Heating Duration: 144 hours".

Sample Description:
The submitted material was a disc with an irregular edge (several small pieces were torn from the edge). It appears that the sample was mixed then cured in the bottom of a small beaker. The test sample consisted of a 1.711 inch by 1.277 inch by nominally 0.070 inch thick rectangle of RTV that was cut from the center section of the supplied disc of material. The sample area listed below includes one face and none of the edges. After the six day test the sample was removed from the chamber and reweighed.

Material Supplier:
The sample was supplied by Norihiro Sakurai in the Utilization & Engineering Department of JAMSS in Japan.

Sample Preconditioning:
The sample was tested with no additional preconditioning.

Sample Area:	14.1 cm^2			
Initial Mass of Sample:	3.78130 gram			
Final Mass of Sample:	3.77873 gram			
Sample Temperature:	60 °C			
Test Duration:	144 hr			
QCM Temperatures:	80 K	233 K	273 K	293 K
Isothermal Data File:	FEB1406A			
QTGA Data File:	FEB2006Q			

Isothermal Test - QCM Data:
QCM data from the end of the outgassing test are summarized in Table 1.

Table 1

		(μg/cm^2)		(%)		(% of TML)
80 K	TML =	184.39	=	0.0687	=	100.0
233 K	VCM =	16.65	=	0.0062	=	9.0
273 K	VCM =	1.78	=	0.0007	=	1.0
293 K	VCM =	0.66	=	0.0002	=	0.4

Total outgassing rate data for the sample were calculated by differentiating the data obtained from the 80 K QCM. Figures showing these total outgassing rate data as a function of test time are attached. These outgassing rates are for species condensable at 80 K and so would not include certain gases such as nitrogen and oxygen.

QCM Thermogravitmetric Analysis - QCM Data:
The QTGA test data can be used to determine the relative amounts of the species outgassed. As the temperature of the 80 K QCM is increased during QTGA, the collected species will evaporate from the QCM in order of their relative volatilities. The attached QTGA data are plotted as evaporation rate from the QCM as a function of QCM temperature.

Mass Spectrometer Data:
Data from the in situ mass spectrometer are sometimes used to help identify the outgassed species. Identification of outgassed species is not within the normal scope of work for this testing and is not ordinarily pursued because of the analysis time required. However, observations on some of the species that are contributing to the outgassing flux from the sample have been noted below. Species identifications are based on engineering and chemistry experience and have not been confirmed by comparison with standards.

Species observed in the outgassing flux are listed below.

Species	Relative Abundance	Characteristic Ions (m/z)
methane*	major	14, 15, 16
benzene	minor	39, 50, 51,52, 77, 78
methylene chloride	trace	49, 51, 84, 86, 88
water	major	18
triethyl phosphate	minor	45, 81, 99, 109, 127, 155
hydrocarbons	trace	41, 43, 55, 57, 69, 71
polymethyl cyclosiloxanes	moderate	59, 73, 147, 221, 267, 281, 327, 355
polyaromatic unidentified species	trace	91, 156, 253, 327
phenyl silicones	moderate	73, 135, 197

Methane, benzene, water, polymethyl cyclosiloxanes, and the phenyl silicones were still present in the outgassing flux at the end of the outgassing test.

*It should be noted that methane is not condensable on an 80 K quartz crystal microbalance (QCM) so its quantitative amount will not appear in Table 1 of the report. However, the mass spectrometer signal intensity indicates that the sample outgassed a greater amount of methane than water.

Attachments:

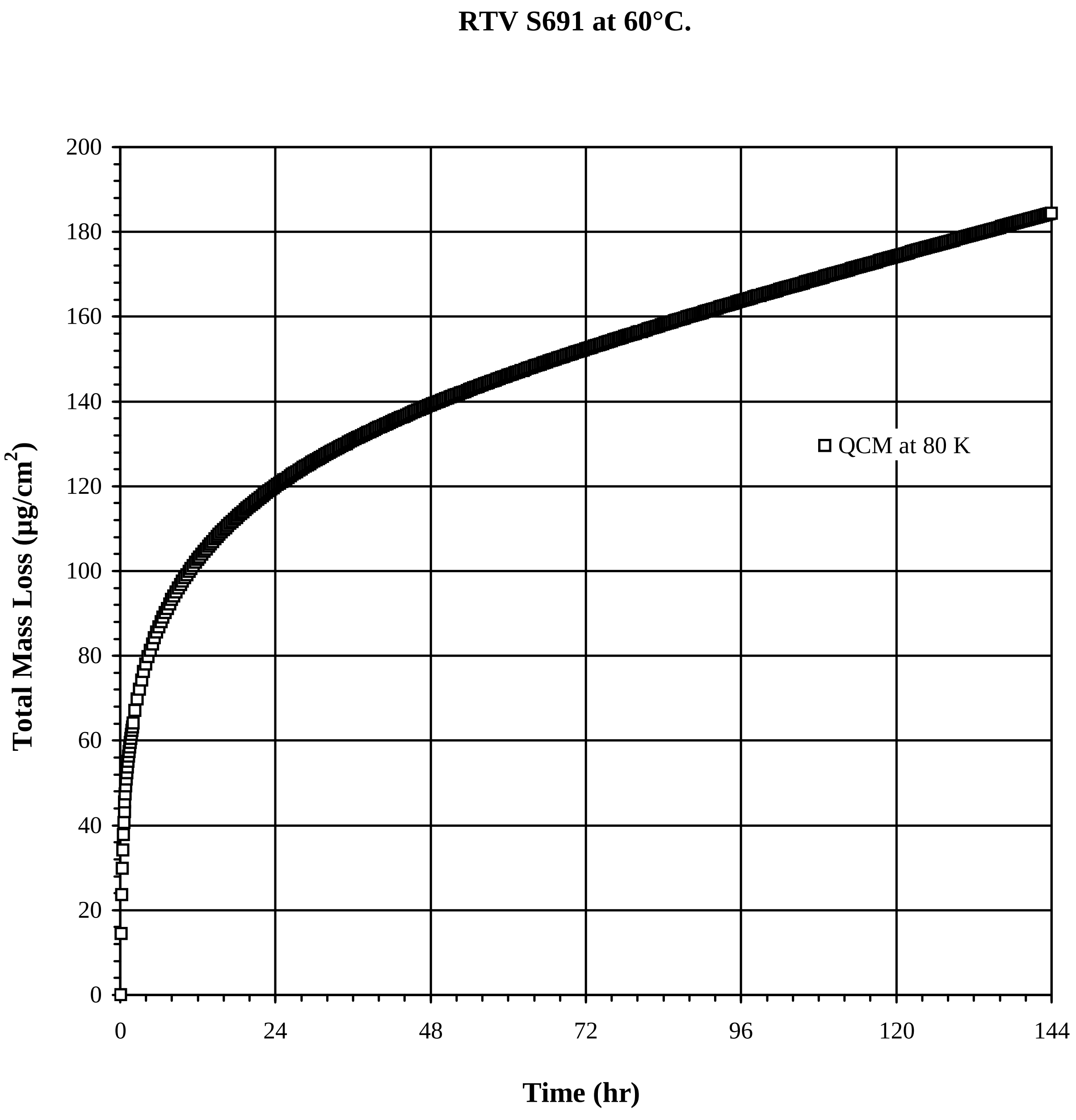
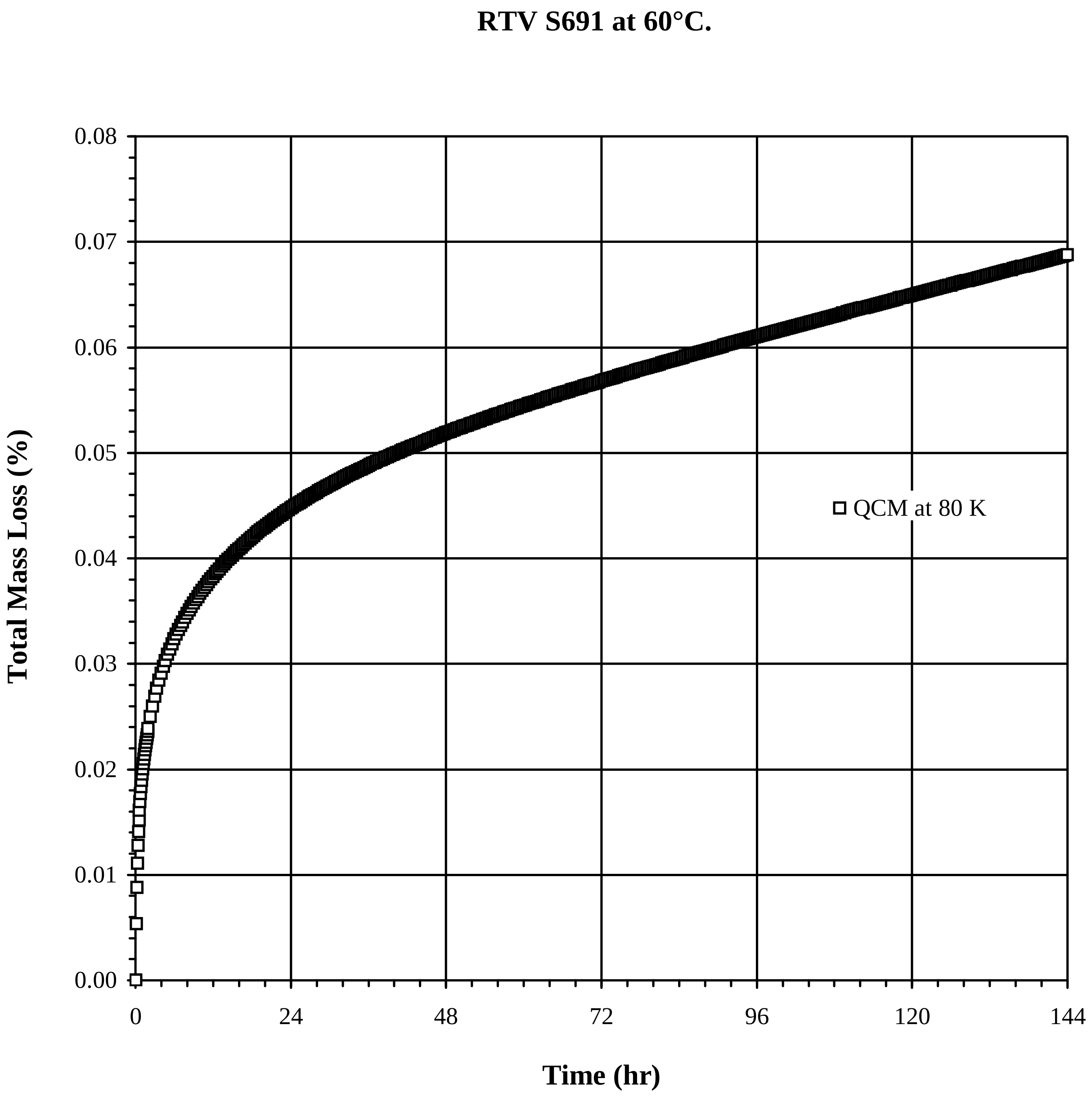
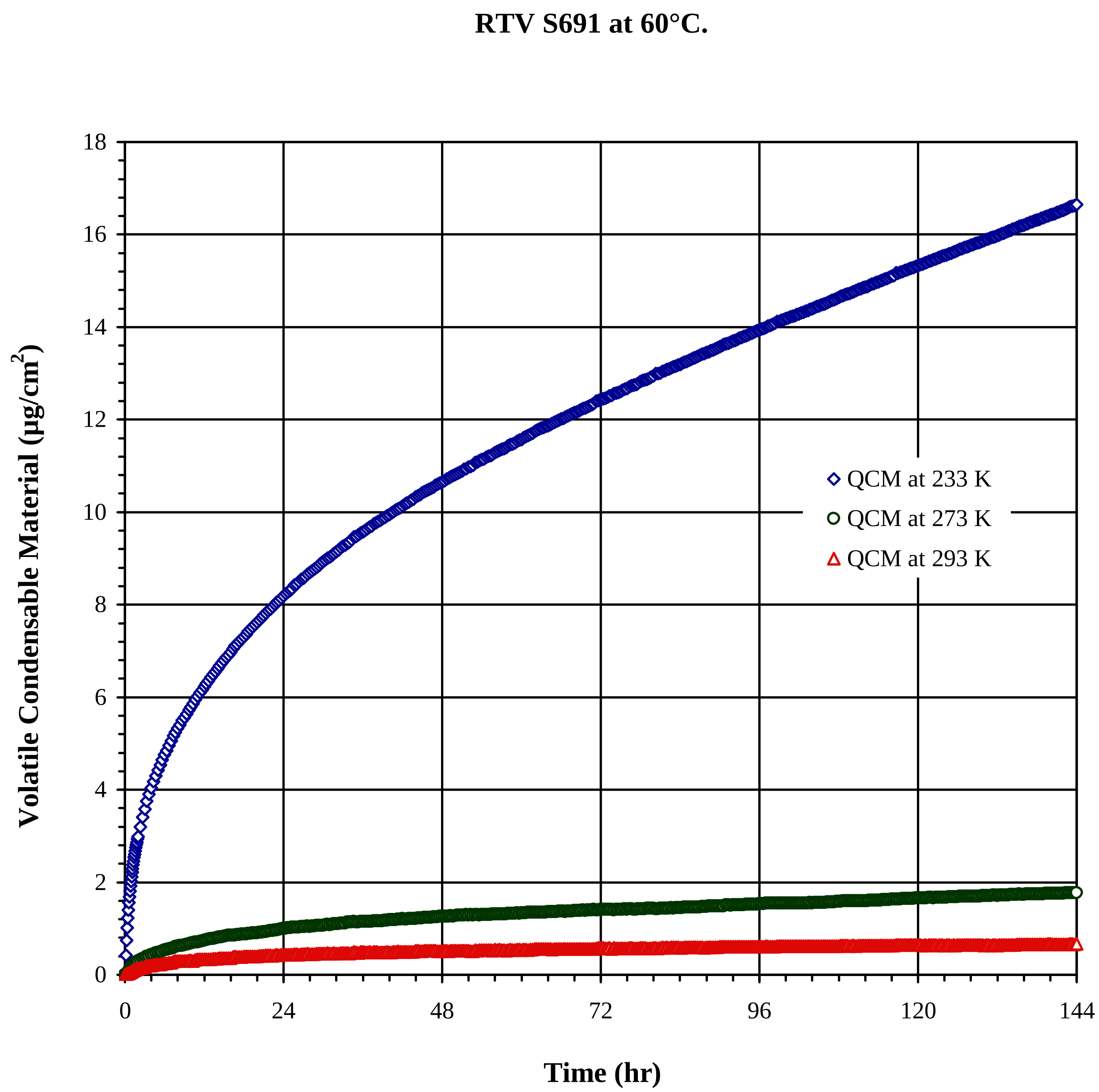

RTV S691 at 60°C.
Total Mass Loss (μg/cm2)
200
180
160
140
120
100
80
60
40
20
0
QCM at 80 K
0
24
48
72
96
120
144
Time (hr)

Fig. 1(a)



RTV S691 at 60°C.
Total Mass Loss (%)
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0.00
0
24
48
72
96
120
144
Time (hr)
QCM at 80 K

Fig. 1(b)



RTV S691 at 60°C.
Volatile Condensable Material (µg/cm2)
Time (hr)
QCM at 233 K
QCM at 273 K
QCM at 293 K
0
2
4
6
8
10
12
14
16
18
0
24
48
72
96
120
144

Fig. 2(a)

RTV S691 at 60°C.

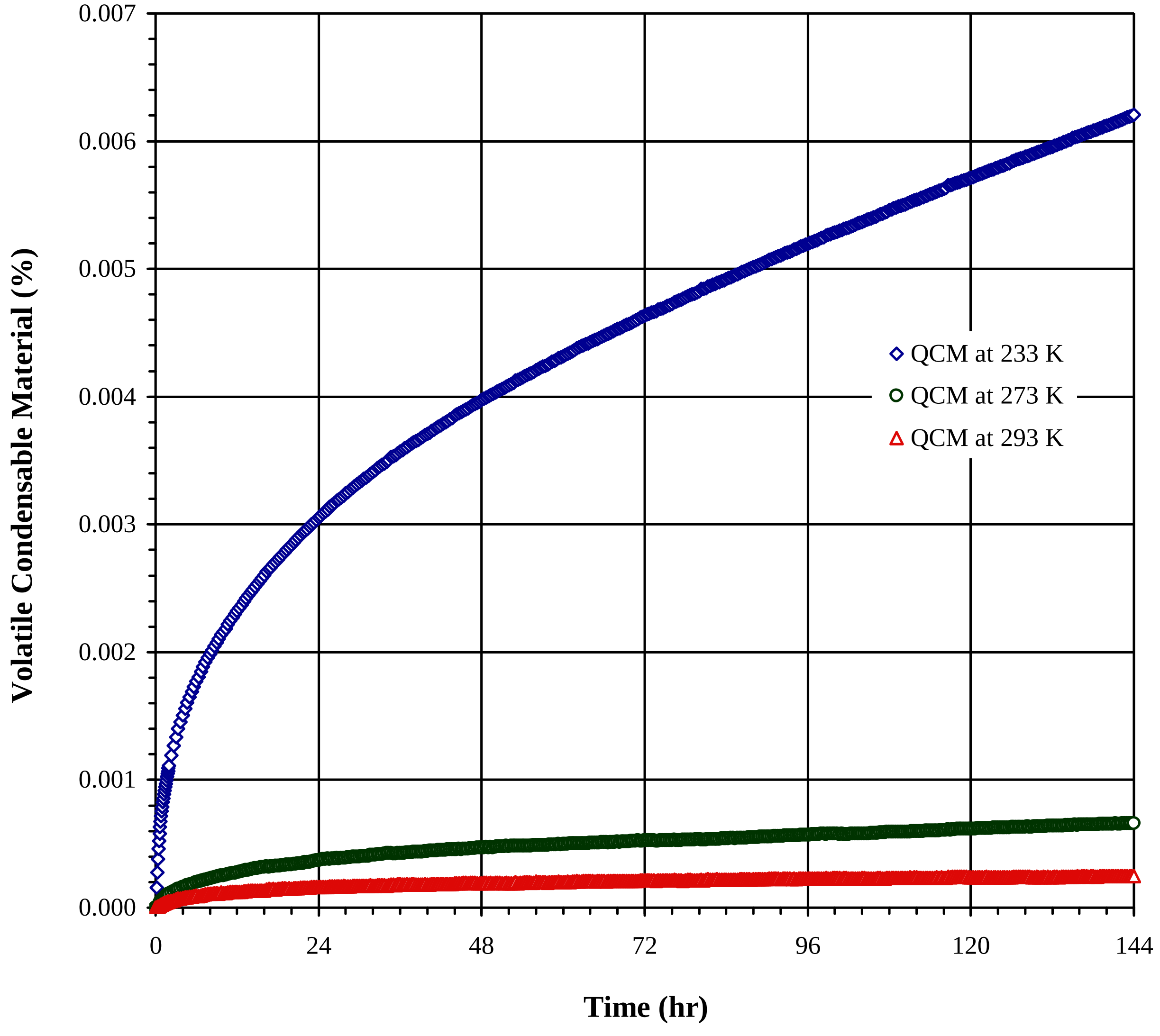


Fig. 2(b)

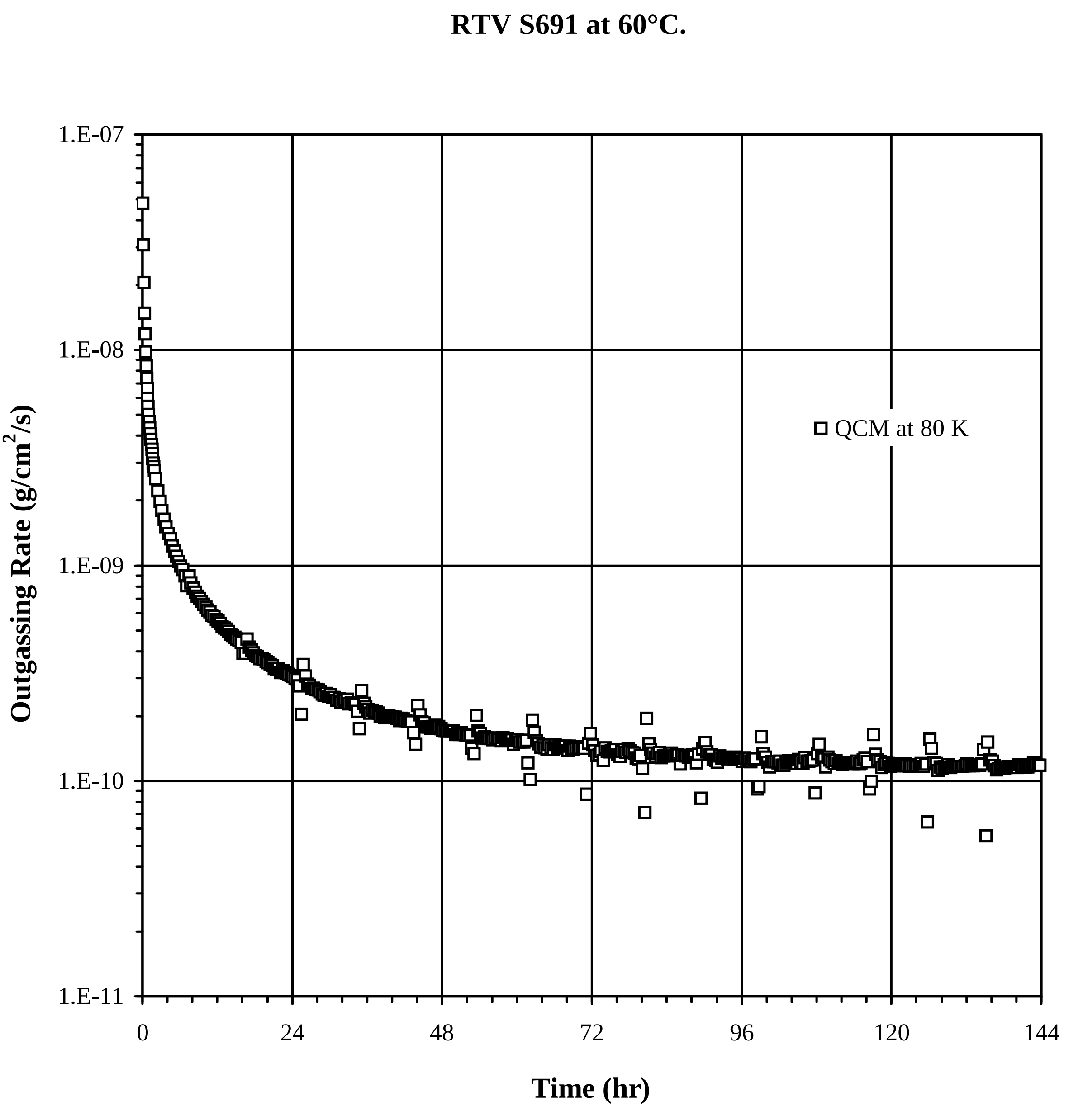

RTV S691 at 60°C.
1.E-07
1.E-08
1.E-09
1.E-10
1.E-11
Outgassing Rate (g/cm2/s)
QCM at 80 K
0
24
48
72
96
120
144
Time (hr)

Fig. 3(a)

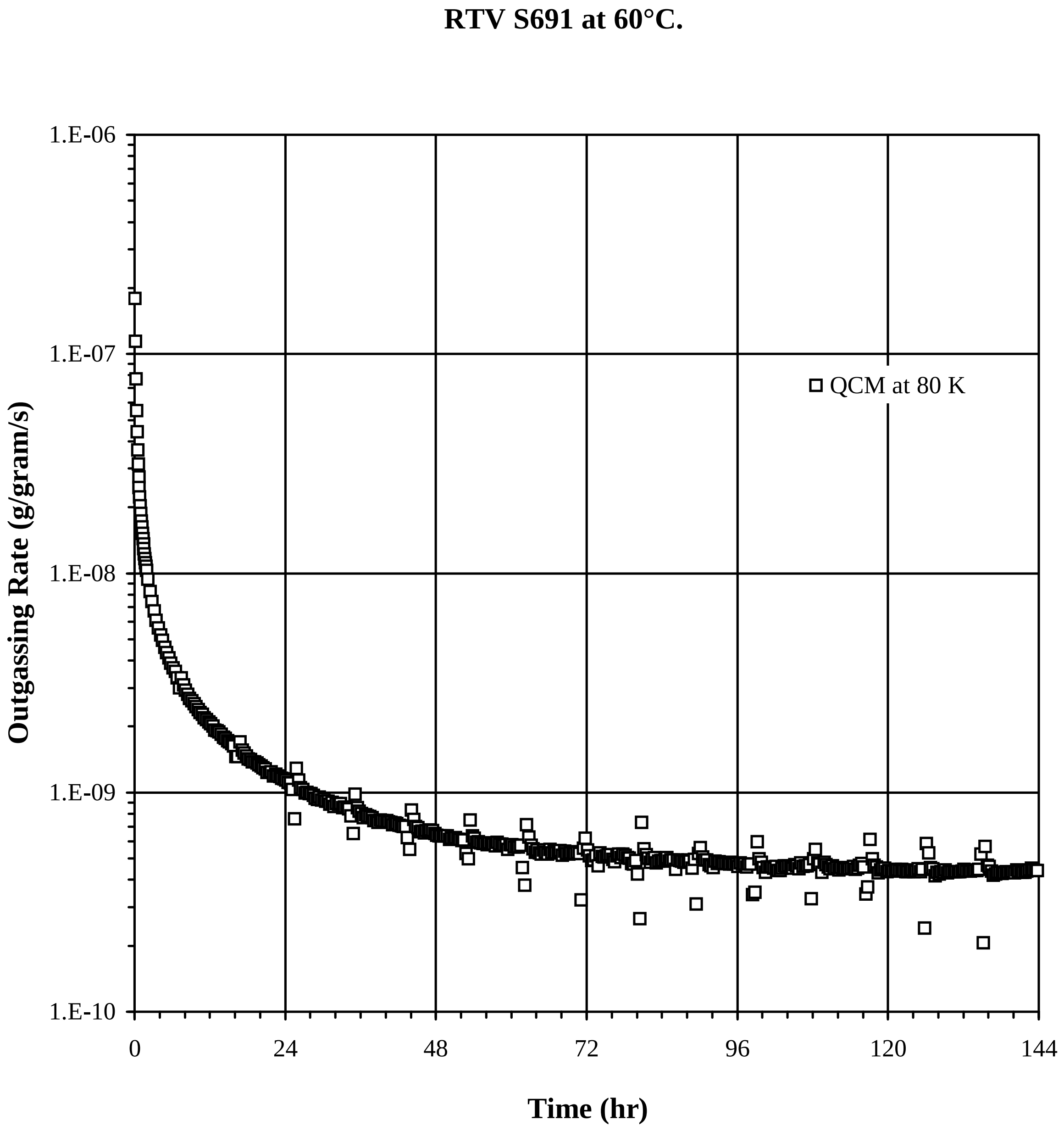

RTV S691 at 60°C.
1.E-06
1.E-07
1.E-08
1.E-09
1.E-10
Outgassing Rate (g/gram/s)
QCM at 80 K
0
24
48
72
96
120
144
Time (hr)

Fig. 3(b)

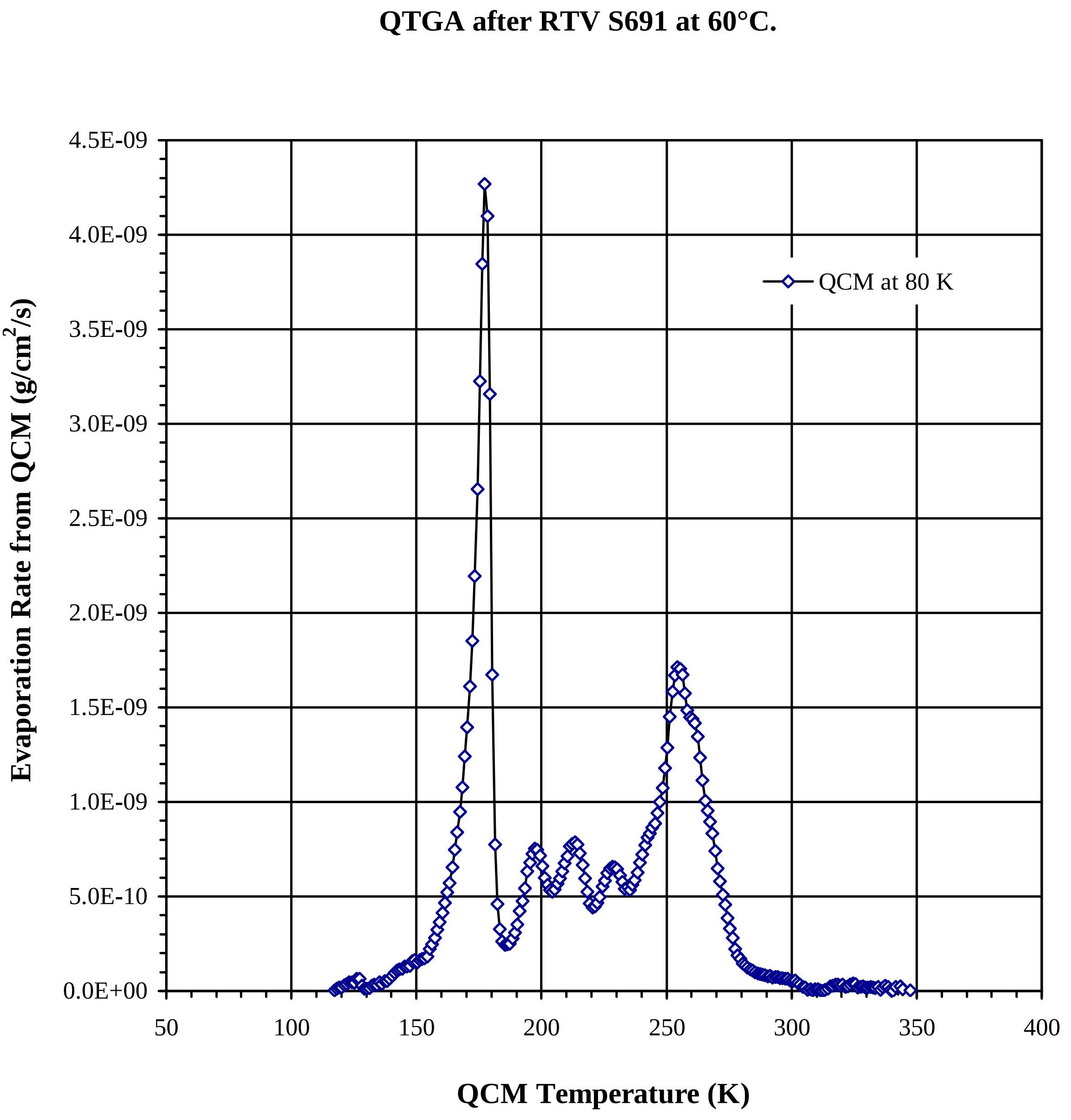

QTGA after RTV S691 at 60°C.
Evaporation Rate from QCM (g/cm2/s)
4.5E-09
4.0E-09
3.5E-09
3.0E-09
2.5E-09
2.0E-09
1.5E-09
1.0E-09
5.0E-10
0.0E+00
50
100
150
200
250
300
350
400
QCM Temperature (K)
QCM at 80 K

Fig. 4(a)

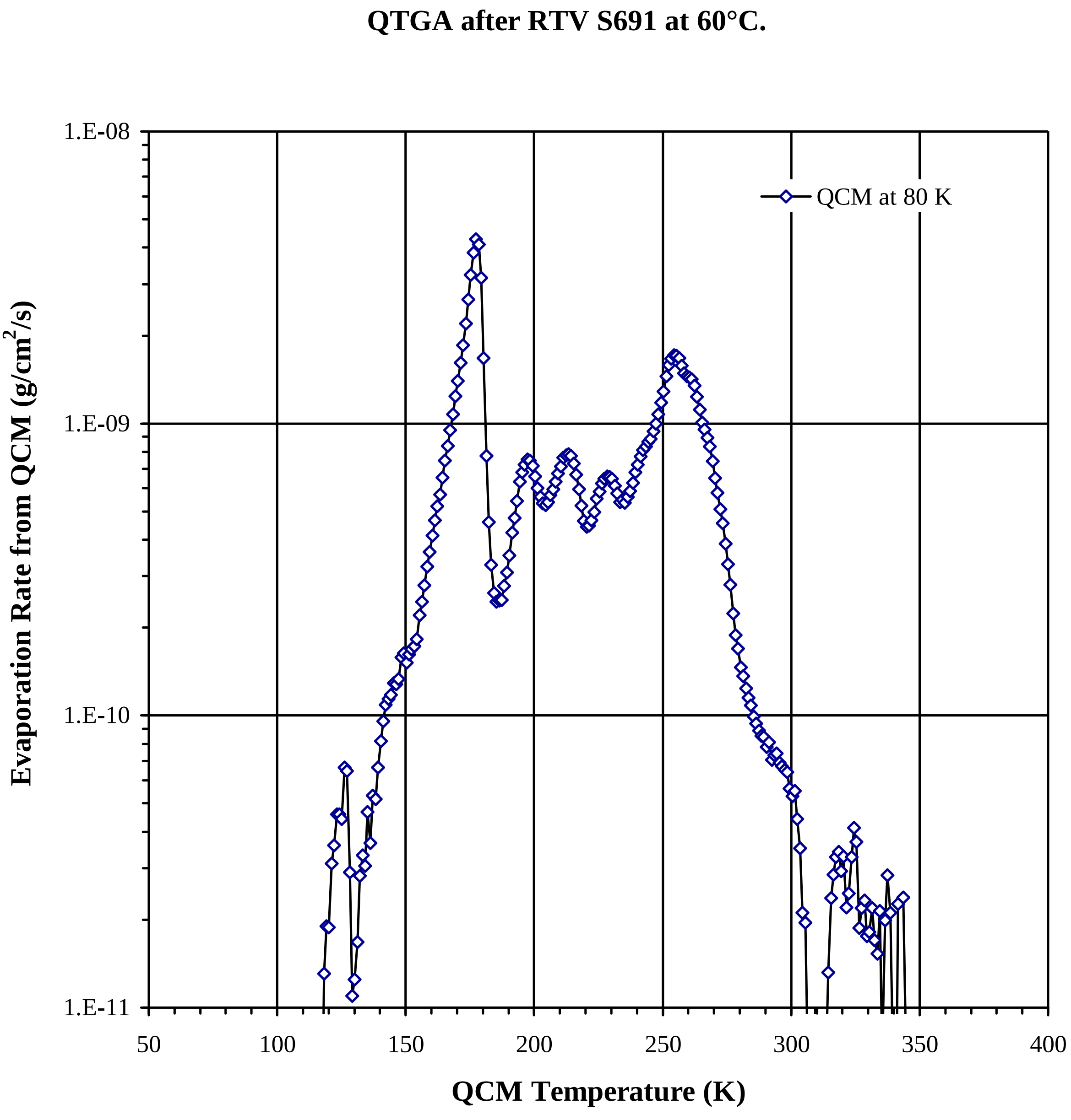

QTGA after RTV S691 at 60°C.
QCM at 80 K
1.E-08
1.E-09
1.E-10
1.E-11
Evaporation Rate from QCM (g/cm2/s)
50
100
150
200
250
300
350
400
QCM Temperature (K)

Fig. 4(b)