

OSI

Outgassing Services International
2224-H Old Middlefield Way, Mountain View, CA 94043-2421
Phone (650) 960-1390 Fax (650) 960-1388

garrett@osilab.com

Outgassing Measurements on Hysol EA-9309NA Epoxy

Prepared for:

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Prepared by:

*Jeff Garrett
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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 cm²
- Sensitivity of each of the four QCMs was 4.43×10^{-9} g/cm²/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:

Hysol EA-9309NA epoxy

Material Description:

The material consisted of 1 rectangular piece of aluminum foil with the EA-9309NA epoxy applied to one side. The epoxy is manufactured by Hysol and is light brown/tan.

Material Packaging:

The epoxy sample was wrapped in aluminum foil then single-bagged in a heat-sealed clear plastic "MagicCut" bag.

Sample Description:

The epoxy sample was 1.35 inches by 0.90 inches by 0.030 inches thick and weighed 0.55984 g. The initial mass of the aluminum foil substrate was 0.035765 g giving a net initial sample weight of 0.52408 g. The epoxy sample was placed in the effusion cell and tested while still attached to the aluminum foil substrate. The sample area listed below includes only the one exposed face of the epoxy.

Material Supplier:

The sample was supplied by Jim Tokunaga in the Utilization & Engineering Department of JAMSS in Japan.

Sample Preconditioning:

The sample was tested with no additional preconditioning.

Sample Area:	7.8 cm ²
Mass of Sample:	0.52408 grams
Sample Temperature:	74 °C
Test Duration:	144 hr
QCM Temperatures:	80 K 233 K 263 K 298 K

Isothermal Data File:	JUL2702G
QTGA Data File:	AUG0202R

Isothermal Test - QCM Data:

QCM data from the end of the outgassing test are summarized in Table 1.

Table 1

	($\mu\text{g}/\text{cm}^2$)	(%)	(% of TML)
80 K TML =	854.34	= 1.2779	= 100.0
233 K VCM =	25.07	= 0.0375	= 2.9
263 K VCM =	4.38	= 0.0066	= 0.5
298 K VCM =	1.54	= 0.0023	= 0.2

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 Thermogravimetric 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.

<u>Species</u>	<u>Relative Abundance</u>	<u>Characteristic Ions (m/z)</u>
water	major	18
unidentified species possibly amines and/or glycols	moderate	30, 45, 56, 58, 74, 86, 89, 103, 120 133, 164
phthalates (plasticizers)	minor	149
ethyldiphenylphosphine oxide	minor	51, 77, 183, 201, 202, 229

Water was still present in the outgassing flux at the end of the 144-hour test.

Attachments:

- Figs. 1(a-b). Total Mass Loss from the Sample as a Function of Test Time.
(Species Condensable on the 80 K QCM)
- Figs. 2(a-b). Volatile Condensable Material from the Sample on the Warmer QCMs
as a Function of Test Time.
- Figs. 3(a-b). Total Outgassing Rate for the Sample as a Function of Test Time.
(Species Condensable on the 80 K QCM)
- Figs. 4(a-b). QTGA Data: Evaporation Rate from the 80 K QCM of the Collected
Outgassed Material as a Function of QCM Temperature.

Hysol EA-9309NA Epoxy at 74°C.

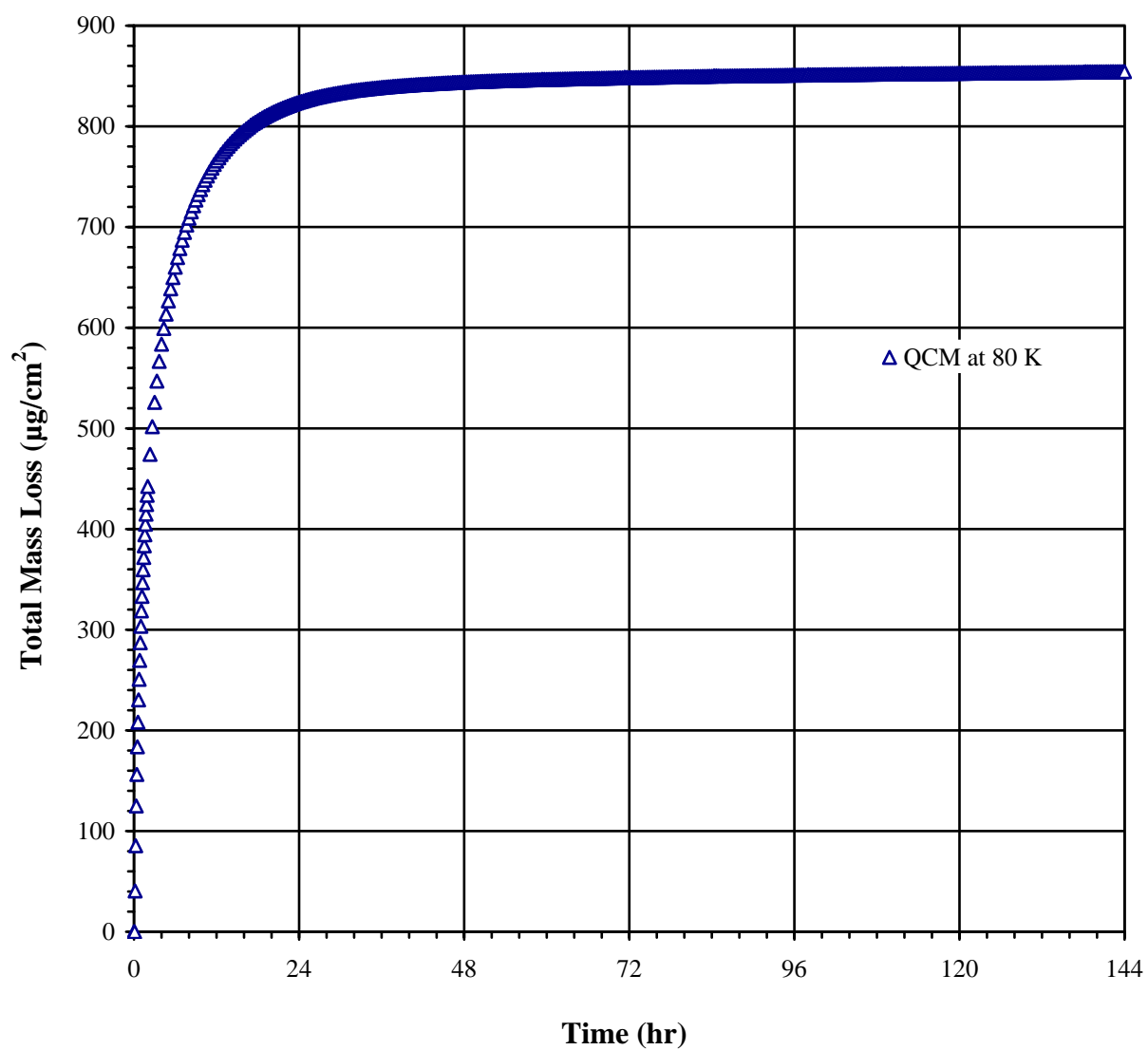


Fig. 1(a)

Hysol EA-9309NA Epoxy at 74°C.

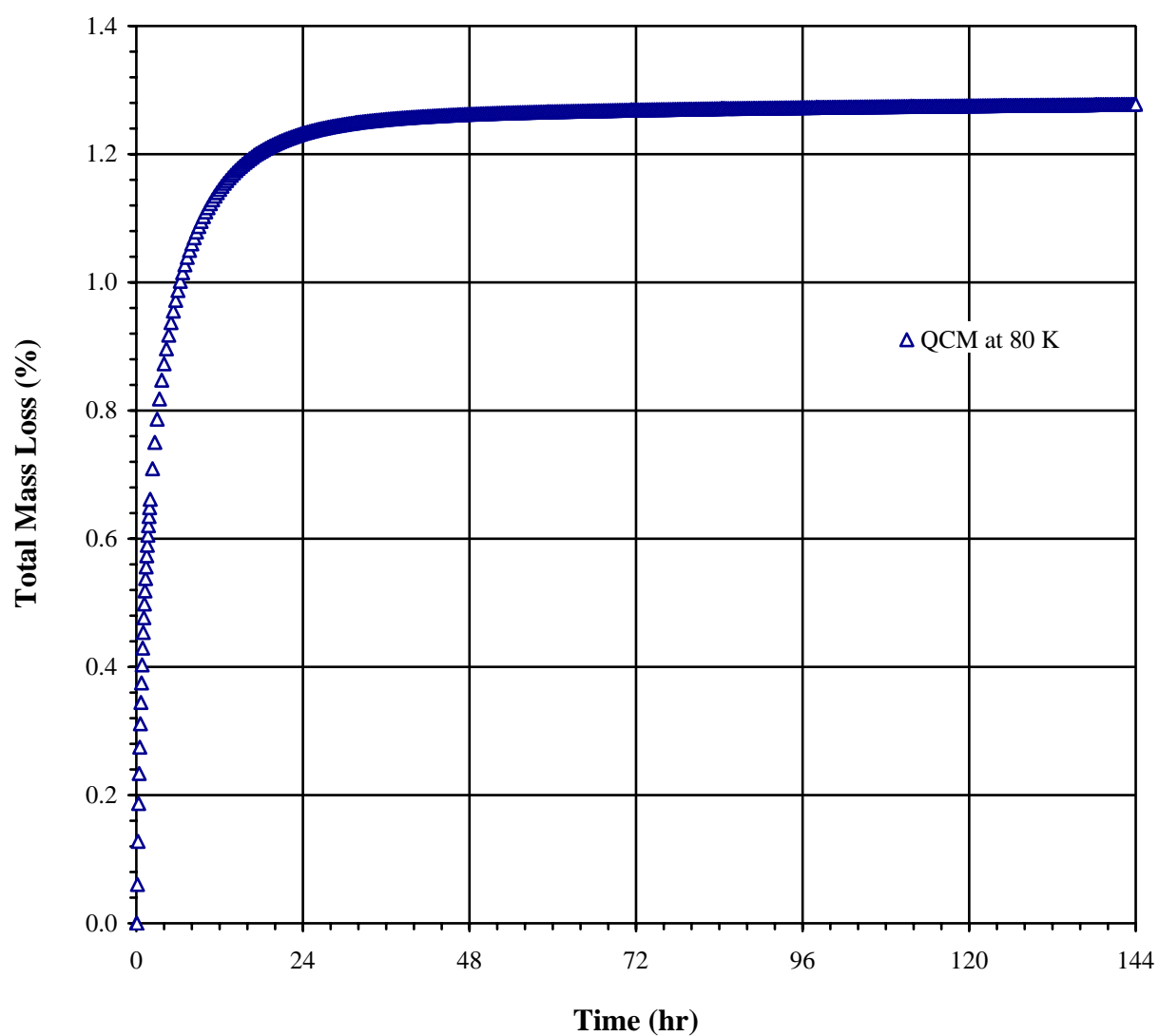


Fig. 1(b)

Hysol EA-9309NA Epoxy at 74°C.

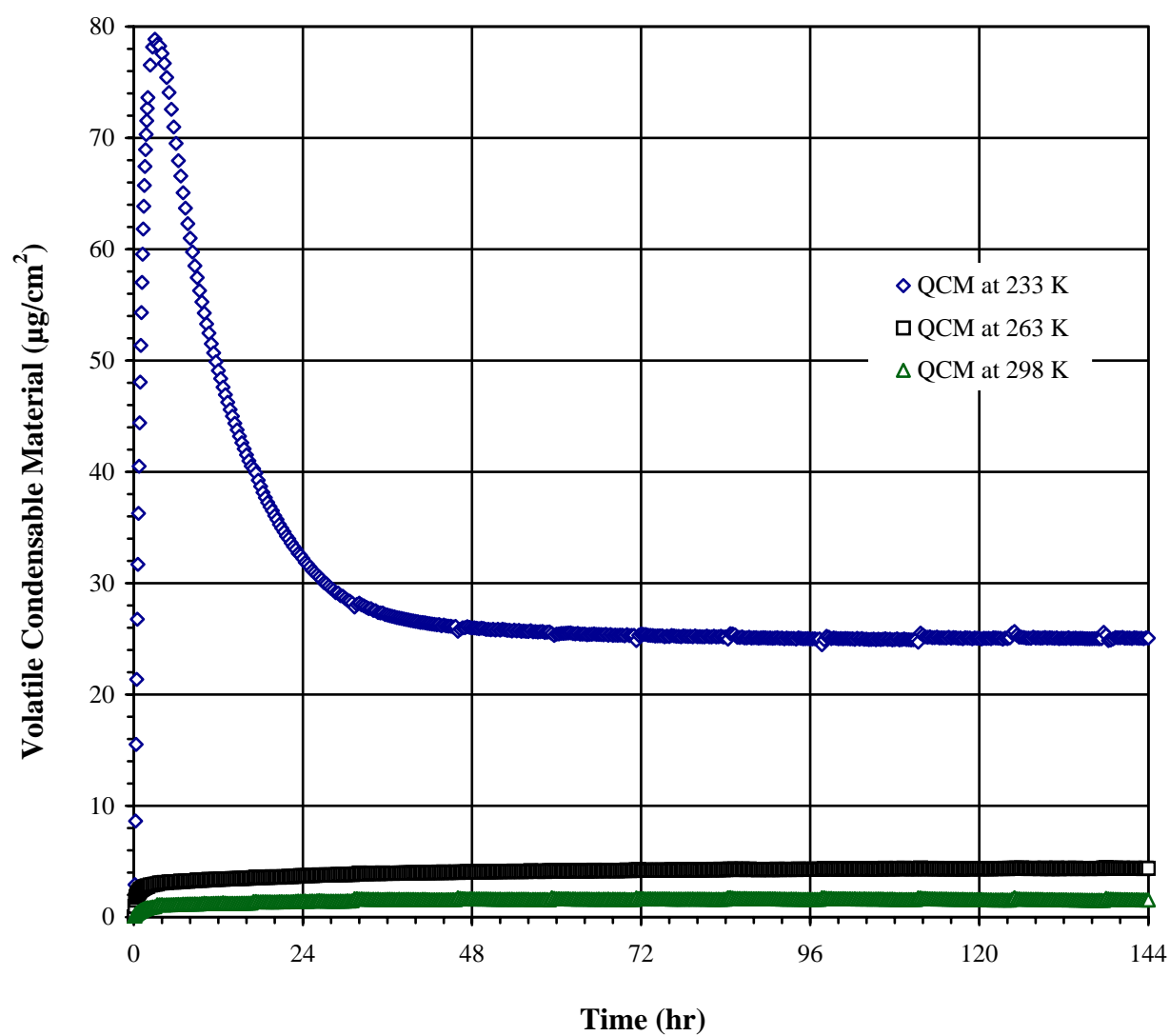


Fig. 2(a)

Hysol EA-9309NA Epoxy at 74°C.

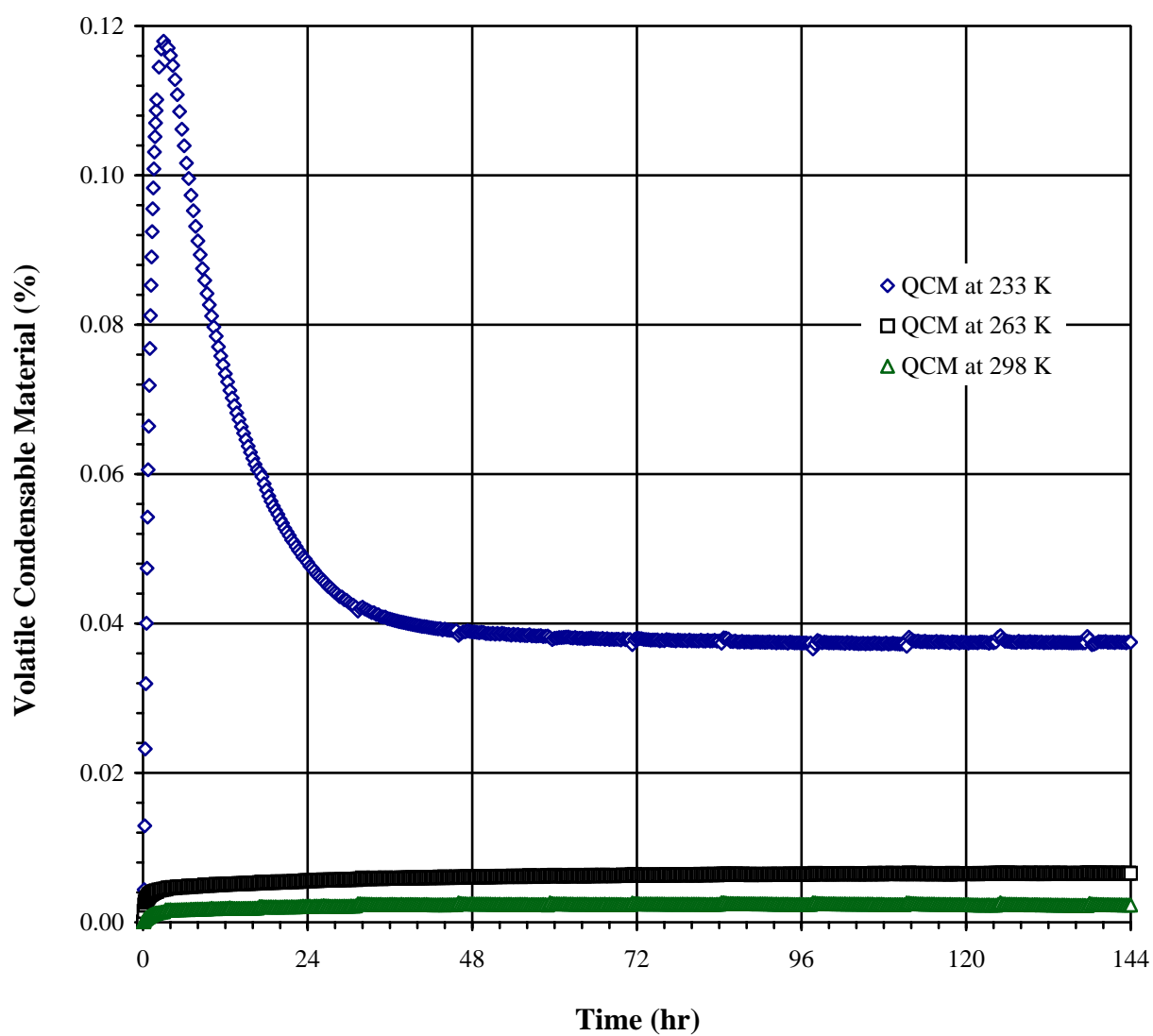


Fig. 2(b)

Hysol EA-9309NA Epoxy at 74°C.

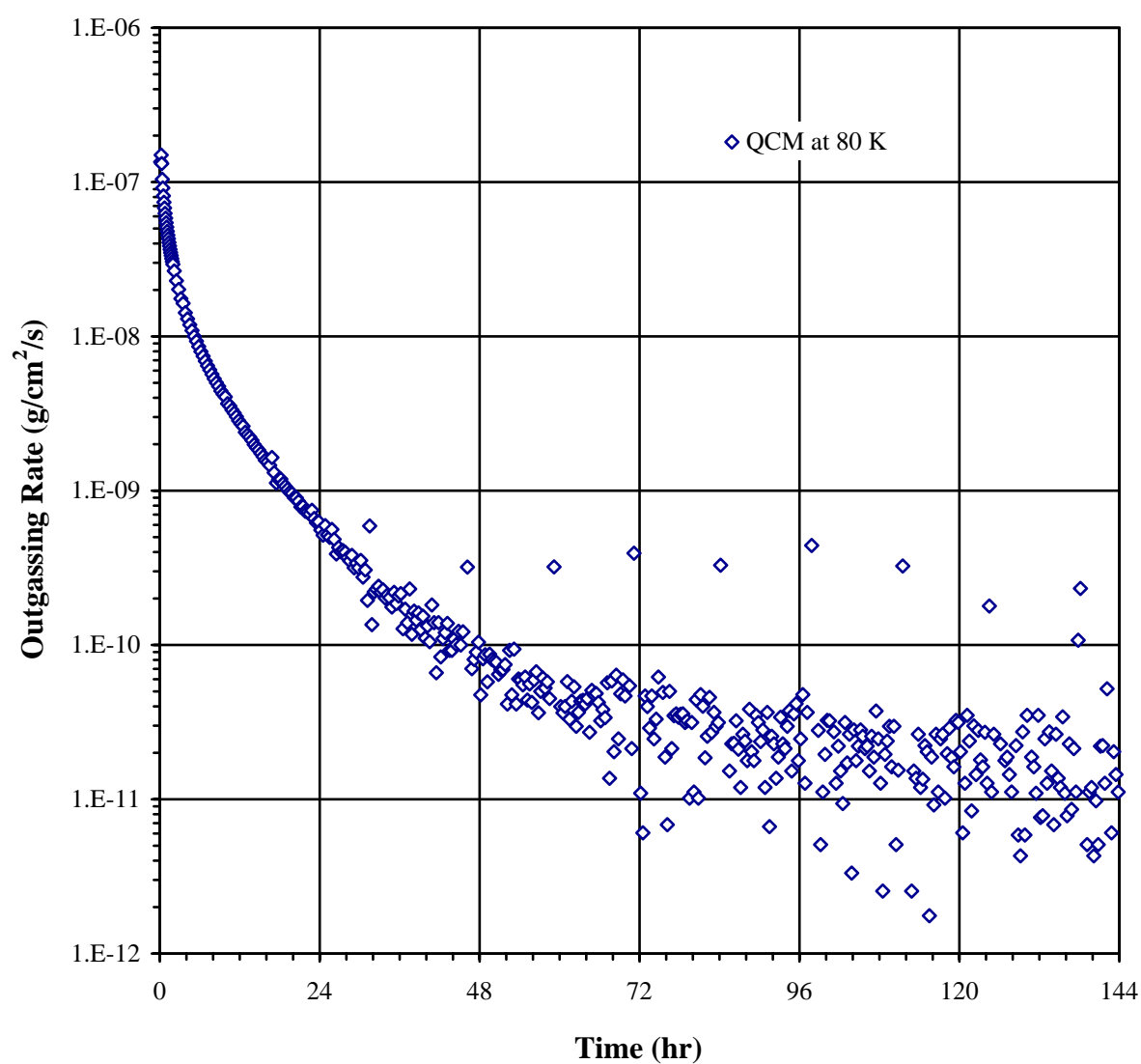


Fig. 3(a)

Hysol EA-9309NA Epoxy at 74°C.

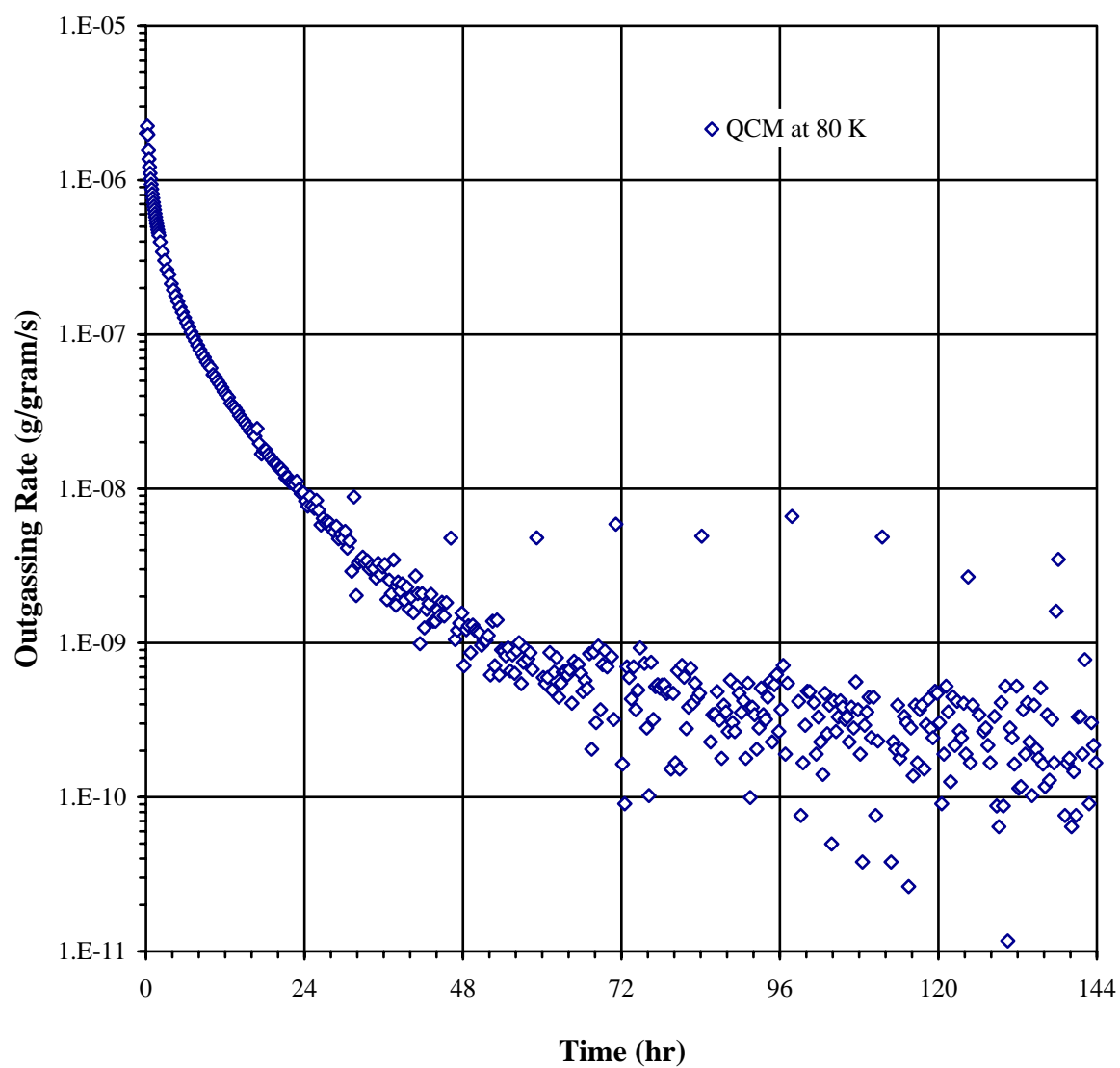


Fig. 3(b)

QTGA after Hysol EA-9309NA Epoxy at 74°C.

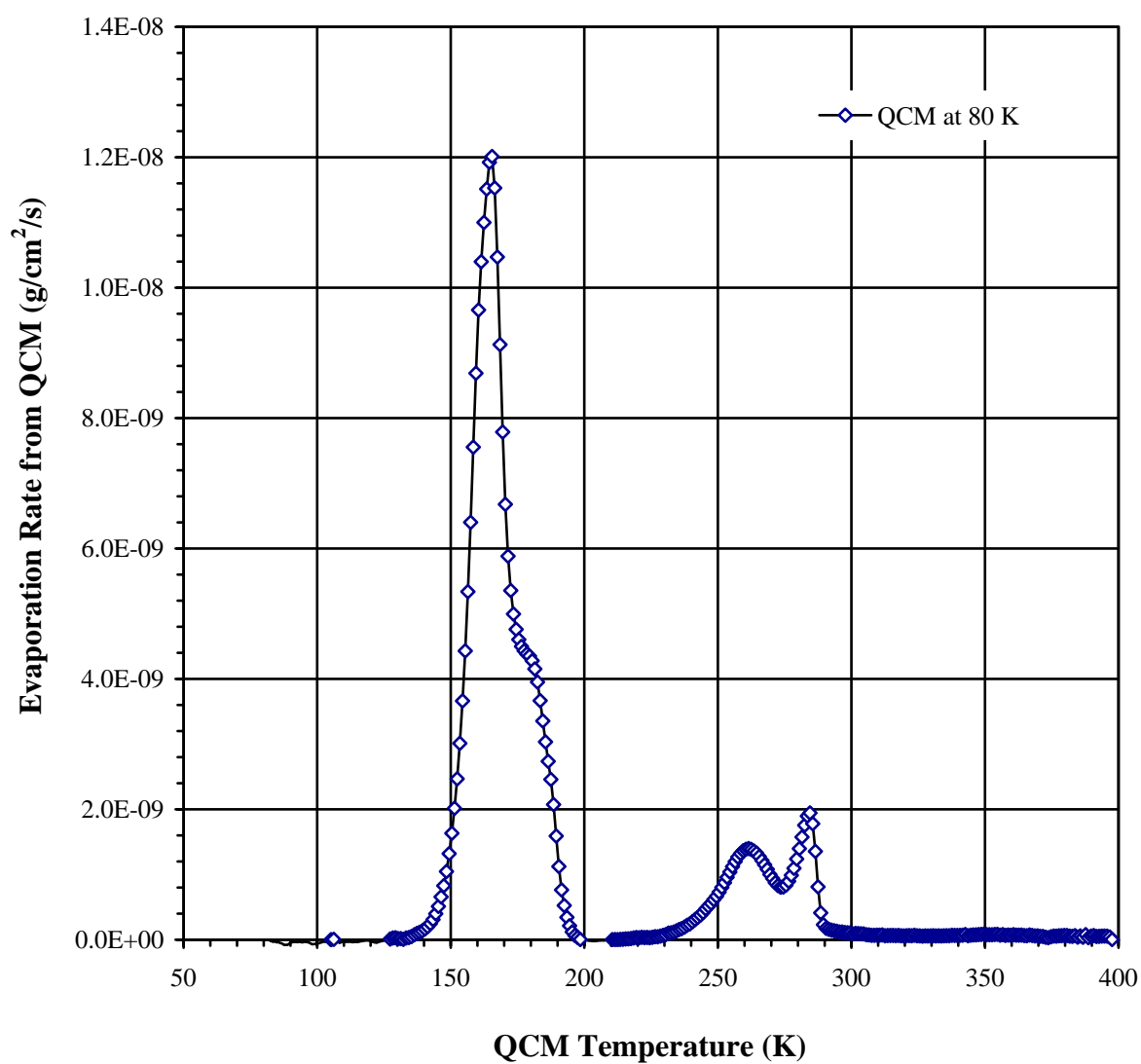


Fig. 4(a)

QTGA after Hysol EA-9309NA Epoxy at 74°C.

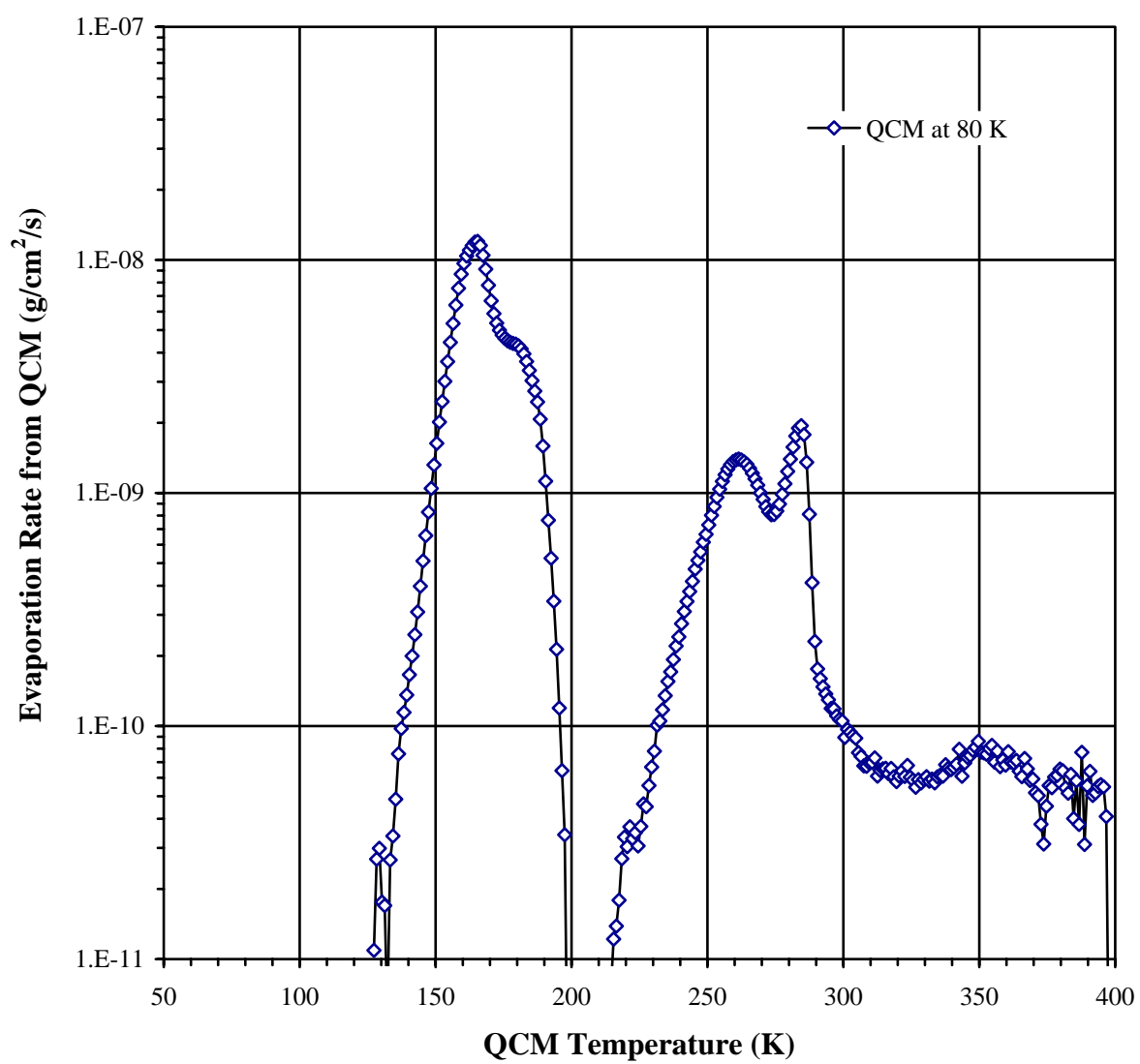


Fig. 4(b)