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Outgassing Measurements on Nextel AF-62

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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

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Test Sample:

Nextel AF-62

Material Description:

The material consisted of 2 rectangular pieces of white woven cloth manufactured by 3M.

Material Packaging:

The material sample was double-bagged in heat-sealed clear plastic "MagicCut" bags.

Sample Description:

One piece of the Nextel was 1.810 inches by 1.740 inches by 0.050 inches thick and weighed 1.43496 g. The second piece of Nextel was 1.645 inches by 1.585 inches by 0.050 inches thick and weighed 1.19737 g. Both pieces of Nextel were placed in the effusion cell and tested together as one sample. The sample area listed below includes one side of the cloth pieces and no edges.

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: 37.1 cm²

Mass of Sample: 2.63233 grams

Sample Temperature: 125 °C

Test Duration: 144 hr

QCM Temperatures: 80 K 233 K 263 K 298 K

Isothermal Data File: JUN2002G

QTGA Data File: JUN2602R

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 =	107.95	=	0.1523	=	100.0
233 K VCM =	94.27	=	0.1330	=	87.3
263 K VCM =	90.27	=	0.1274	=	83.6
298 K VCM =	48.61	=	0.0686	=	45.0

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	minor	18
glycerin	moderate	15, 29, 31, 43, 45, 61
glycol	minor	27, 29, 31, 43, 45, 73, 87, 89, 103, 129
glycol	major	45, 59, 72, 89, 133, 177, 239, 283
glycol	major	45, 59, 73, 89, 133, 177, 221, 265, 327, 371

The glycol species were still present in the outgassing flux at the end of the 144-hour test.

Attachments:

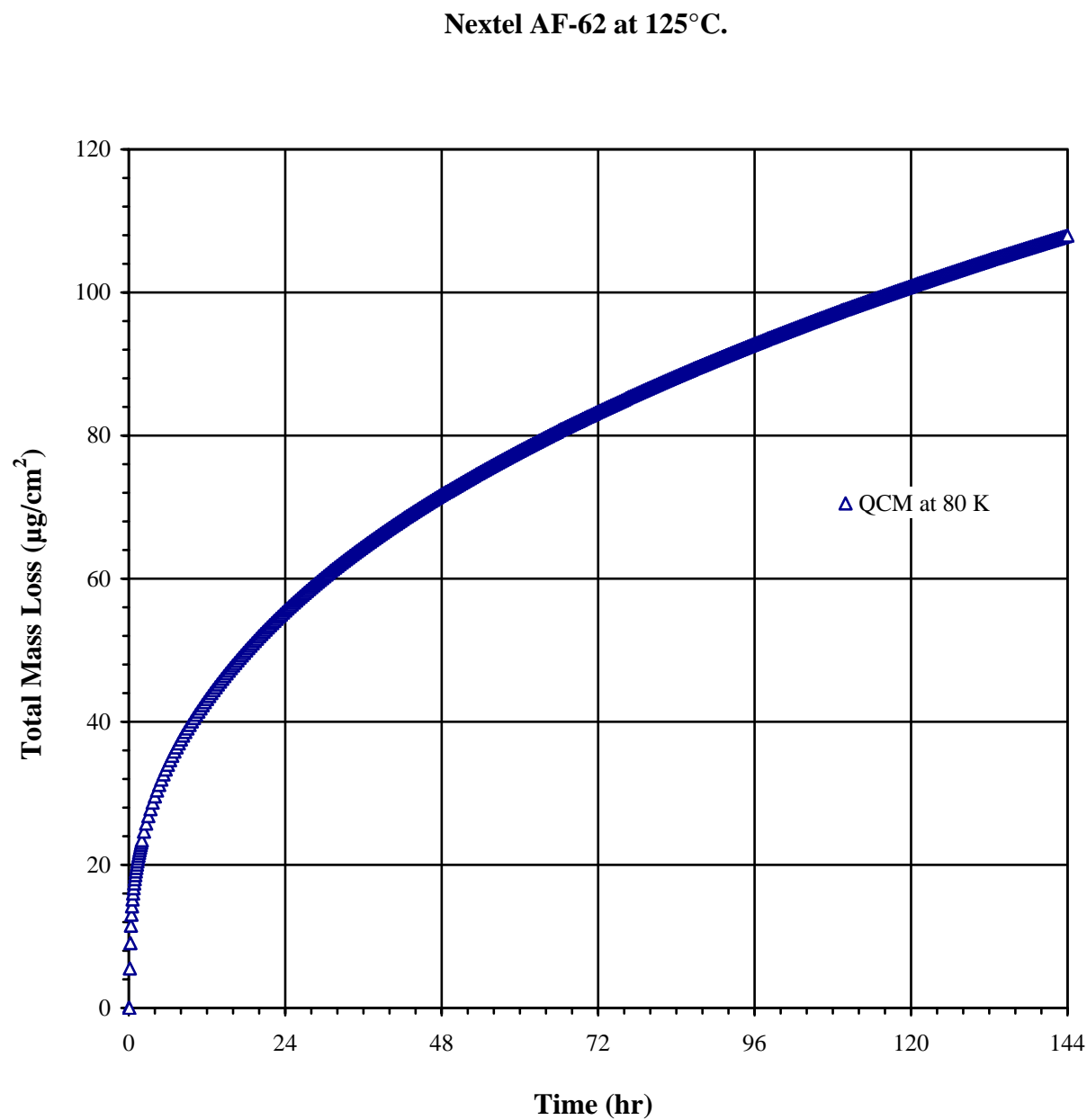


Fig. 1(a)

Nextel AF-62 at 125°C.

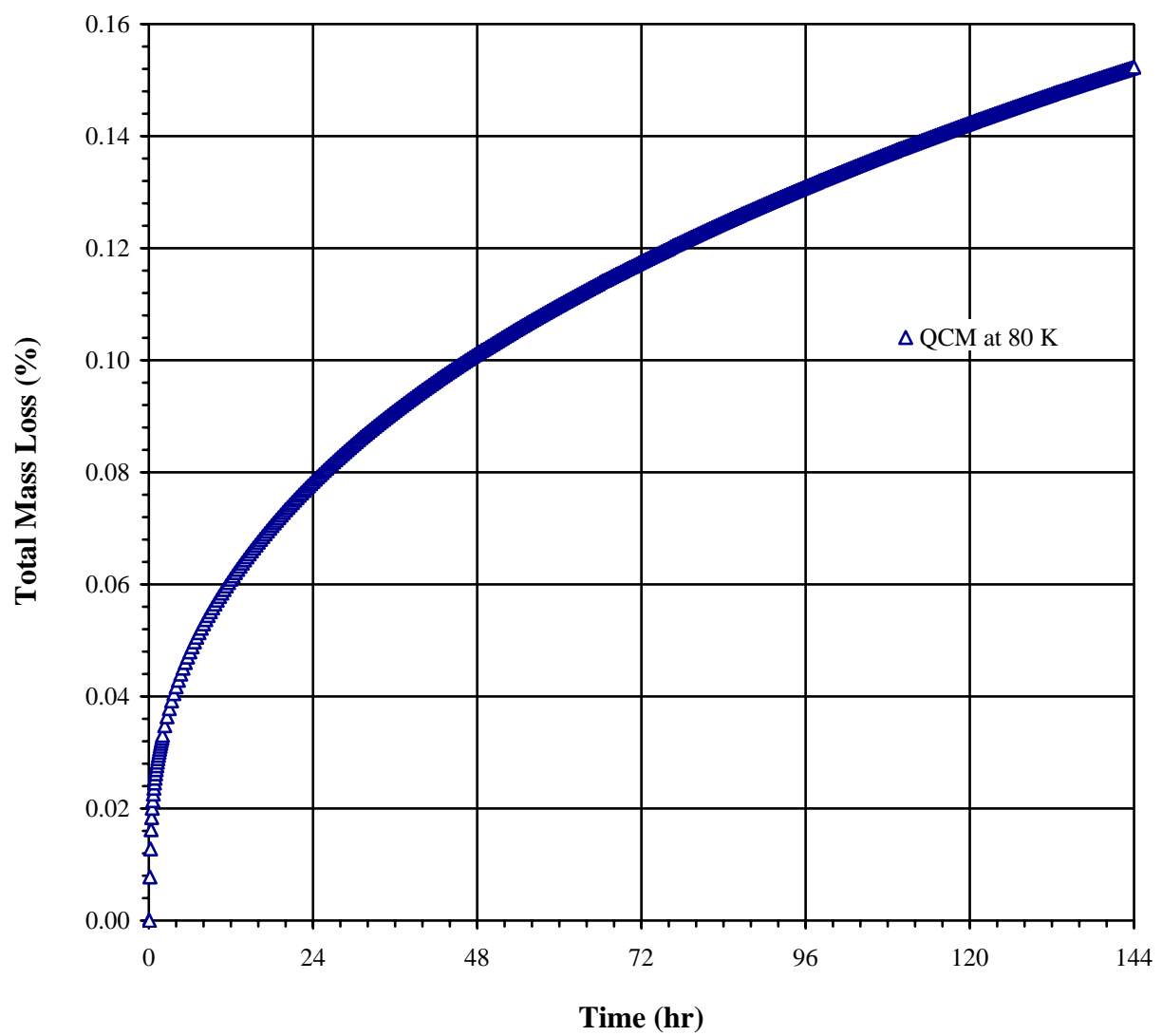


Fig. 1(b)

Nextel AF-62 at 125°C.

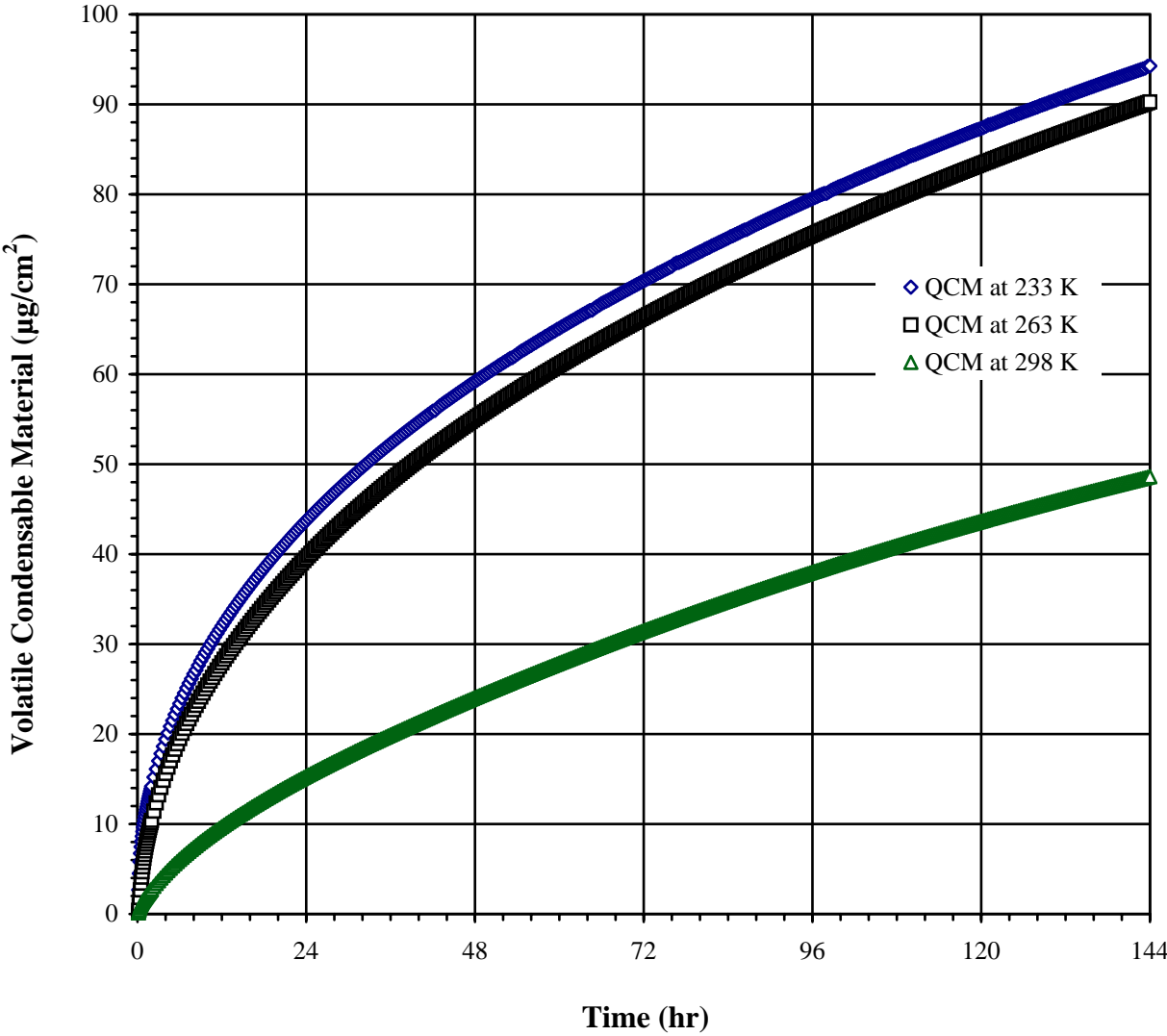


Fig. 2(a)

Nextel AF-62 at 125°C.

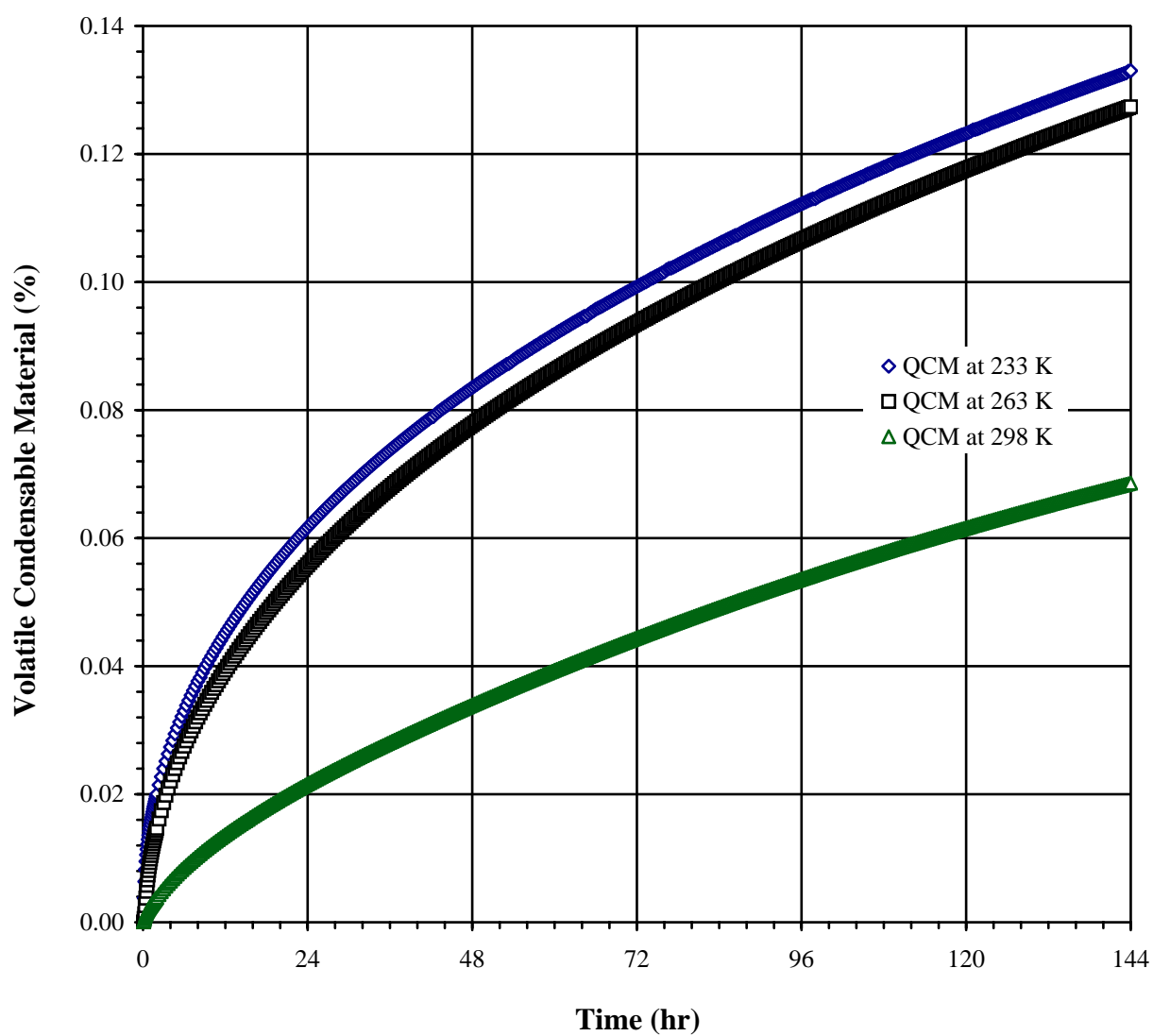


Fig. 2(b)

Nextel AF-62 at 125°C.

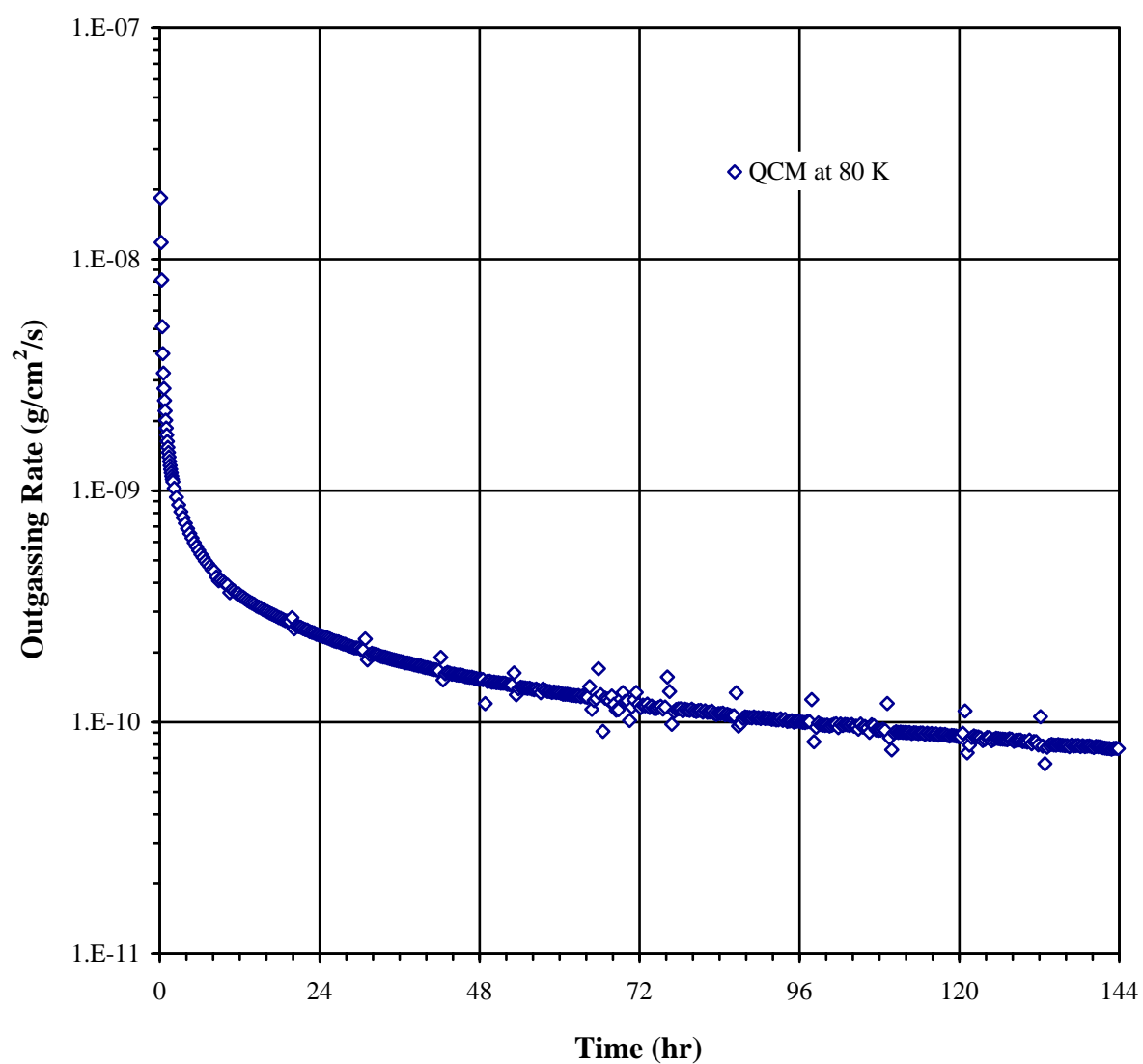


Fig. 3(a)

Nextel AF-62 at 125°C.

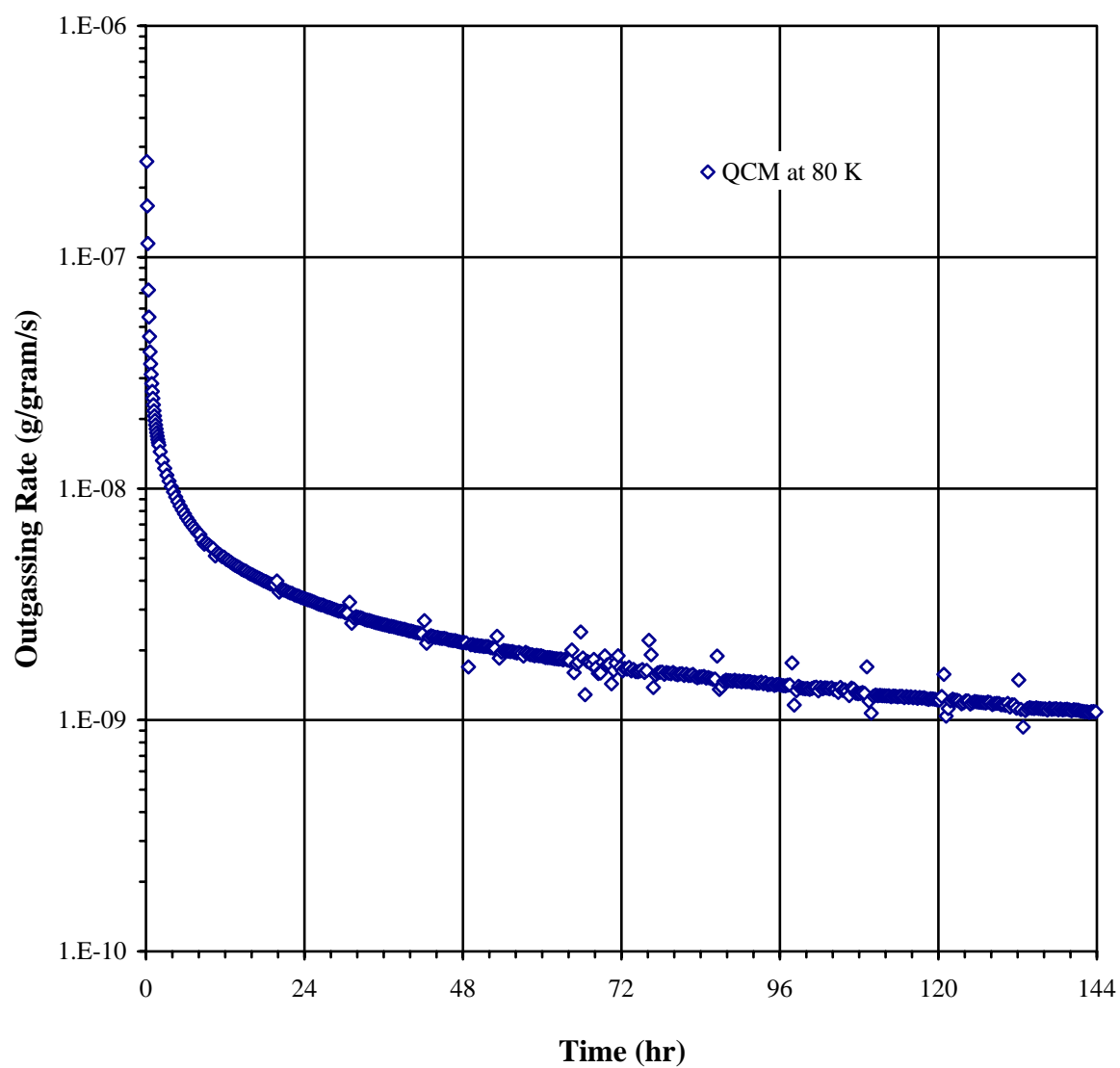


Fig. 3(b)

QTGA after Nextel AF-62 at 125°C.

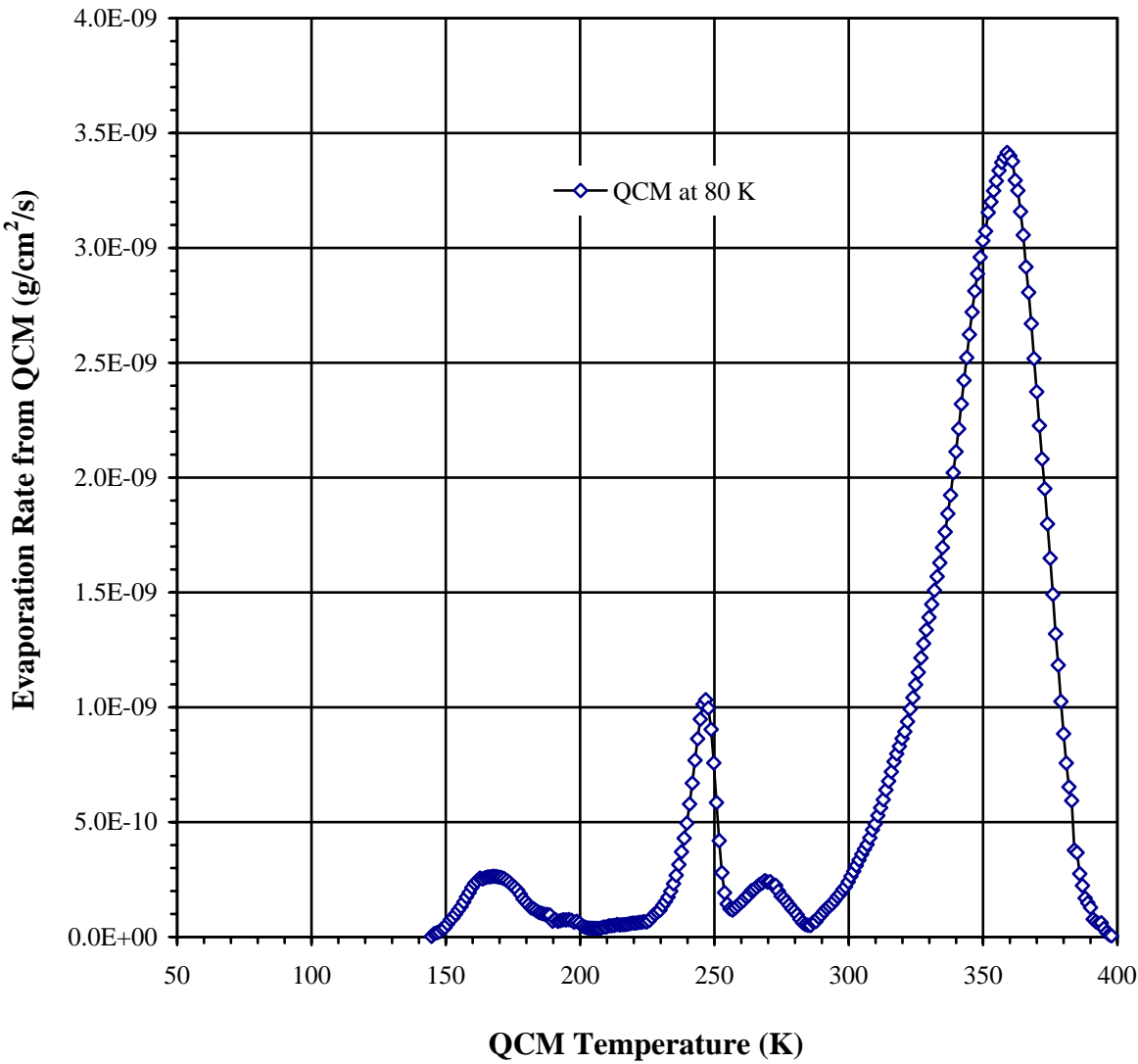


Fig. 4(a)

QTGA after Nextel AF-62 at 125°C.

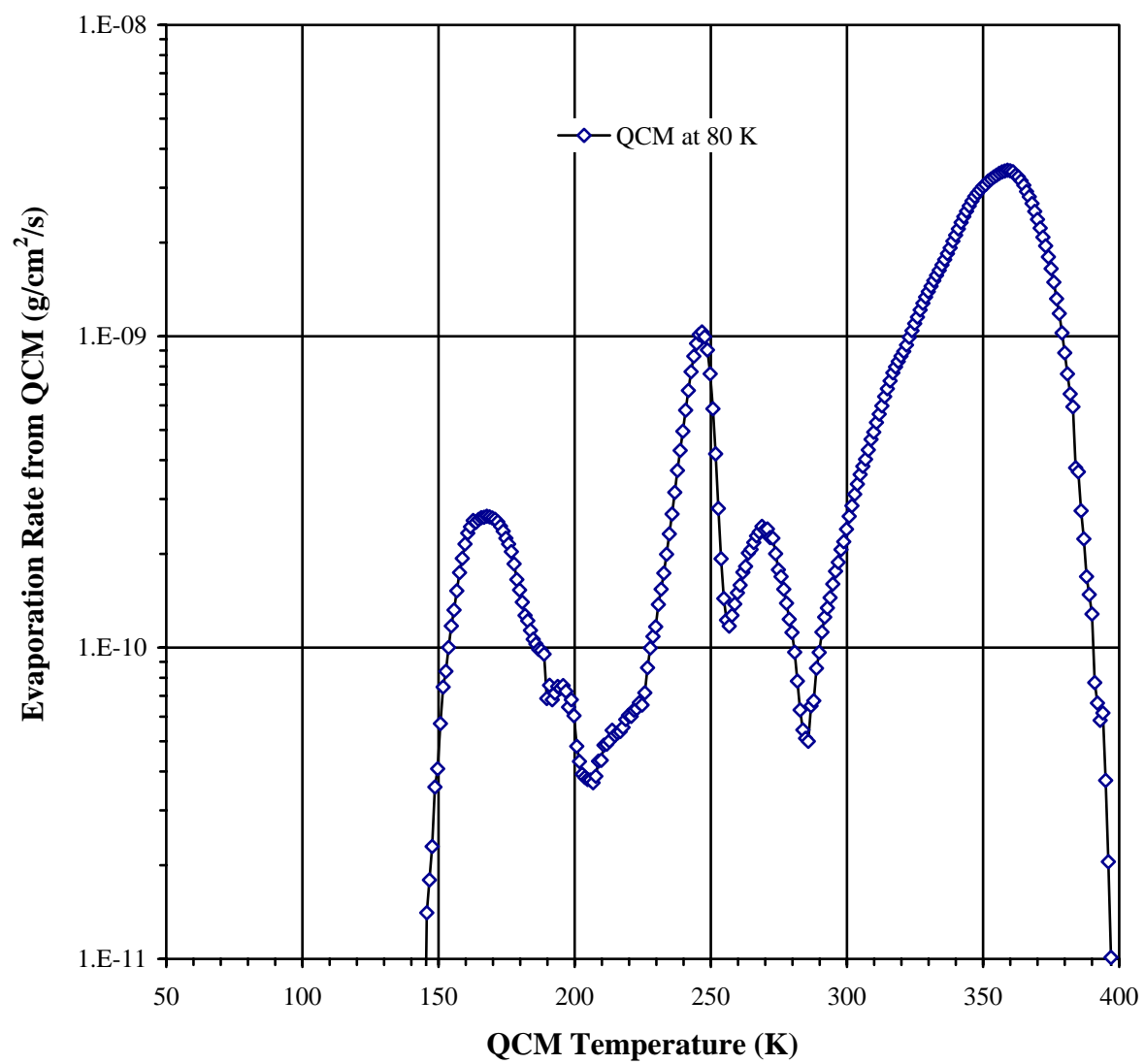


Fig. 4(b)