INVESTIGATION OF THE DETERMINATION OF RESPIRABLE QUARTZ ON FILTER MEDIA USING FOURIER TRANSFORM INFRARED SPECTROPHOTOMETRY

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ABSTRACT

The use of Fourier transform infrared spectrophotometry for the determination of quartz in respirable dust directly on filter media was investigated. Fifteen different 25-mm filters which might be used in a respirable dust sampler were examined. With the best of these, the limit of detection in practical operating conditions (three times the standard deviation of the blank) was estimated to be 3 micrograms with linearity in excess of 1 mg. The spatial inhomogeneity of the filter background appears to be a limiting factor to the sensitivity.

Procedures were devised to correct for the filter background and the absorbance of relatively non-structured interferences, such as coal dust. In addition, two methods to correct for the interference of the overlapping absorbance bands of other minerals (kaolinite, cristobalite) with those of quartz were also studied. Scaled spectral subtraction of reference spectra of the interferences provided results that did not differ significantly from those which were obtained by a more traditional approach. Both methods overcorrected for the presence of the interference.

The absorbance signal per unit weight of quartz was observed to be influenced by particle size within the respirable range.

INTRODUCTION

An estimated 2.3 million workers are potentially exposed to dusts containing alpha-quartz and other polymorphs of crystalline silica, such as cristobalite or tridymite. Occupations and industries in which exposures can occur include mining, foundry work, glass and clay manufacturing, cement production, pottery making, and abrasive blasting operations.[1]

Since the inhalation of these minerals poses a significant risk for the development of respiratory disease, limits for safe exposure have been defined by a number of groups concerned with occupational safety and health. The NIOSH Recommended Exposure Limit (REL) for respirable quartz is an eight-hour time-weighted average of 0.05 mg/m^3.[2] On the other hand, OSHA has defined a permissible exposure limit (PEL) for respirable quartz in terms of the concentration of respirable dust with an adjustment for the quartz content of the dust: $10/(\%SiO_2 + 2)$ mg/m^3.[3] If the dust is entirely composed of quartz, then the PEL is equivalent to a concentration of 0.1 mg/m^3 or 2 X REL. The standards or recommendations for cristobalite or tridymite are half of those for quartz.

Sampling and analytical methods for the determination of respirable quartz which have been published by NIOSH recommend that the respirable portion of airborne dust be selected by means of a 10-mm nylon cyclone and collected upon 37-mm filters. Subsequently, the filters are destroyed and the collected dust is transferred to an appropriate matrix for analysis by X-ray powder diffractometry (NIOSH Method 7500)[4] or dispersive infrared spectrophotometry (NIOSH Method 7602).[5] While detection limits for the

X-ray or the infrared methods have been estimated to be 5 micrograms based on the behavior of calibration standards, the actual performance of the entire sampling and analytical method is worse.

A collaborative test of the X-ray method indicated that if a single laboratory conducted the sampling and analysis it could produce values within twenty-five percent of the "true" value at the ninety-five percent confidence limit for quartz deposits in a range of 50 to 200 micrograms which were taken from a test atmosphere.[6] However, when the results of all laboratories were considered and personal sampling pumps were used, large errors were encountered. One possible source of this problem is leaks in the cyclone sampler that are introduced during the assembly of the sampler prior to sampling.

In response, the ruggedness of the 10-mm nylon cyclone sampler was evaluated.[7] The results of controlled leak experiments indicated that only major leaks in the adapter collar of the cyclone had a significant impact on the mass of material collected by the sampling device onto properly sealed cassettes. Therefore, improvements to these methods would have to be found elsewhere.

A method in which the collection filter is analyzed without additional sample preparation (a direct-on-filter method) would eliminate errors due to the transfer step and be more efficient. However, in a recent comparison of a direct-on-filter X-ray diffraction method with NIOSH Method 7500, it was concluded that equivalent results could only be obtained if the direct-on-filter samples were referred to standards which had been prepared by deposition of a quartz aerosol.[8] This procedure is awkward and requires

specialized equipment. It would be preferable to employ a method for which calibration standards could be produced by filtration of a quartz suspension. Transmission infrared spectrophotometry offers this possibility. In addition, this approach is faster, cheaper and simpler than an X-ray method. Finally, there is the opportunity for enhanced sensitivity because of an improvement to the technology for making infrared absorption measurements, Fourier transform infrared spectrometry (FT-IR).

The major infrared bands of the principal polymorphs of crystalline silica include:

Polymorph	Primary (cm^{-1})	Secondary (cm^{-1})
α-quartz	780; 799	695
cristobalite	621	796
tridymite	567	789

These are most suited for analytical purposes and provide the lowest limits of detection. Other spectral bands of quartz, such as 470 cm^{-1}, 516 cm^{-1}, 1100 cm^{-1}, and 1175 cm^{-1}, also appear in silicates and are not as suitable for quantitation. The IR spectra of α-quartz, cristobalite, tridymite, and amorphous SiO_2 are available elsewhere.[9]

A number of direct-on-filter methods for quartz which employ dispersive IR for analysis have been described.[9-14] In a comparison of an X-ray method with an infrared method, it was pointed out that while the two methods had similar sensitivities, the infrared method exhibited greater precision. It was, however, more susceptible to interferences from other mineral dusts.[13] Although some investigators noted that an uneven deposit of quartz is obtained on the collection filter when sampling through a BCIRA

25-mm cyclone elutriator[12], others observed that a direct-on-filter method that employed 25-mm cassettes and nylon cyclones for collection did not suffer from errors due to an inhomogeneous distribution of the collected dust.[14]

There are a number of advantages that can be gained from the application of FT-IR rather than dispersive IR. In an FT-IR the IR beam is split into two beams and sent to two mirrors: one fixed and the other movable. The beams are recombined after a path difference, resulting in constructive and destructive interference of the various wavelengths. All wavelengths are passed by the optics simultaneously, there are no gratings or slits in these instruments. The absorbance spectrum from this modulated beam contains information in the time domain which is then Fourier transformed to yield information in the more common form of absorbance versus wavelength.

The Jacquinot, or throughput, advantage is a result of the FT-IR instrument not having slits that limit the optical throughput of the radiation and hence more light reaches the detector. Since an FT-IR uses interference phenomena to define the absorbance spectrum, information is collected on all spectral wavelengths simultaneously: the Fellgett, or multiplex, advantage. Further, dispersive instruments are prone to errors arising from stray light, whereas FT-IR instruments are unaffected by stray light. The use of a reference laser beam to index the position of the moving mirror in an FT-IR allows very accurate and reproducible wavelength determinations of the spectra to be made. Hence, successive spectra can be precisely added together over an extended time to allow for improvements in the signal-to-noise ratio. The combination of the above advantages produces an increase in sensitivity. Also,

the accurate determination of wavelength allows more precise application of spectral subtraction as a means for correcting for the presence of interferences.

One researcher has reported on efforts to develop FT-IR based direct-on-filter methods for quartz analysis.[15] In that study, calibration curves of samples prepared from KBr pellet samples and those of samples prepared by collection of a quartz aerosol (and analyzed directly on the collection media) were examined. All were found to be linear to one milligram. However, the limits of detection (LOD) were relatively poor; the LOD based on one curve was in excess of one hundred micrograms. The results of the study were inconclusive, but quartz weights appeared to be overestimated by about 20% after spectral subtraction of interferents (cristobalite or kaolinite).

This report describes the determination of respirable quartz on filter media by means of FT-IR. A variety of different filter types were assessed. Several means of correcting for the presence of expected interferences, both broad-band and structured, were investigated. Finally, the influence of particle size upon the measurement was studied briefly. The overall objective was to provide a basis for the further development of a direct-on-filter sampling and analytical method for crystalline silica by means of FT-IR.

EXPERIMENTAL SECTION

A. Instrumentation

The analytical instrument used was a Nicolet 60-SX Fourier Transform

Infrared Spectrophotometer with a potassium bromide (KBr) beamsplitter and triglycine sulfate (TGS) detector. Spectra were collected at two wavenumber resolution, 640 co-added scans (unless noted otherwise), and full (6.9 mm) aperture. Mirror velocity for all scans was set at 0.813 cm/sec. The detector gain was optimized for each sample by maximizing (without saturating the analog-to-digital converter) the centerburst of the interferogram. Filters were mounted in a Barnes Analytical magnetic filter holder. This holder, when placed in the instrument, resulted in the IR beam being slightly eccentric from the filter center.

The quartz content of all starting materials was assessed using a Philips Model 3720 X-ray powder diffractometer, which was equipped with theta-compensating slits and a graphite monochromator. Copper K-alpha radiation was employed and all samples were spun during analysis by means of a sample spinner (Philips Model PW 1780/10) attached to the goniometer. The analysis conditions were those used in NIOSH Method 7500.[4]

B. Materials

The filters were obtained commercially from the following vendors: Gelman Sciences, Ann Arbor, MI (VM-1, DM-450, DM-800, Teflo, Zefluor, GLA-5000, GN-6, HT-450, FP-450, GA-6S, Nylaflo); Nuclepore Corporation, Pleasanton, CA (Nuclepore PVC, Nuclepore PE); Millipore Corporation, Bedford, MA (AAWP); and Mine Safety Appliance Corporation, Pittsburg, PA (FWSB).

National Institute for Standards and Technology (formerly the National Bureau of Standards) Standard Reference Material (SRM) 1878 was the alpha-quartz used in this study. This material is defined as a respirable

quartz standard having a mass median equivalent spherical diameter, obtained from sedimentation analysis, of 1.62 micrometers and a phase purity of 95.5%.[16] Ninety-five percent of the material, by mass, is in the range 0.33 to 5.0 micrometers.

The cristobalite was prepared by the Illinois Institute of Technology Research Institute (IITRI) as part of NIOSH contract 210-75-0043. The synthetic material is provided as a respirable cristobalite standard with a mass median aerodynamic diameter of 6.2 micrometers.[17] The material contains no alpha-quartz and less than two percent impurities.

The kaolinite used in this study was obtained commercially (Fisher Scientific Company, Fair Lawn, N. J., P/N K-5, lot 762146).

Coal dust from Alabama was employed as a broad band absorber. The material was ball milled and, then, freezer milled at liquid nitrogen temperatures. Then, the dust was suspended in isopropanol and wet sieved to below 10 micrometers.

The interferences were examined by an independent technique, X-ray powder diffraction, to verify that they contained no quartz. Kaolinite was found to have a peak near the primary quartz line but no other lines indicative of quartz. The kaolinite spectra was a close match to the Joint Center for Powder Diffraction Standards (JCPDS) pattern number 29-1488, kaolinite.

Samples of coal dust were ashed for 6 hours using a low-temperature plasma asher (Model PM1640/PM118, Branson International Plasma Corp., Hayward, CA) at 300 W and 2 torr of oxygen. The ash represented about 6.3%, by weight, of the starting material. The ash was suspended in isopropanol and aliquots, which represented about 1.6 milligrams of ash, were filtered through silver membrane

filters. The samples were analyzed for quartz content by Method 7500. No quartz was found in these samples.

Sized quartz fractions were prepared by aerosolizing SRM 1878 using a TSI Model 3200 fluidized bed aerosol generator (Thermo-Systems, Inc., St. Paul, MN) and sampling with an Andersen Mark II impactor (Andersen Sampler Inc., Atlanta, GA) at 28.3 liters per minute. This separated the quartz aerosol into seven stages Five stages contained sufficient material to allow for further study. The nominal size range (in micrometers) of each was: 0.65 to 1.1, 1.1 to 2.1, 2.1 to 3.3, 3.3 to 4.7, and 4.7 to 5.8. The collected material was washed with toluene to remove any traces of Resolve® oil (used to prevent particle "bounce" during collection) and dried overnight at 110° C.

The sized quartz fractions were analyzed with a TSI Model 3300 Aerodynamic Particle Sizer [APS] (Thermo-Systems, Inc., St. Paul, MN). The instrument indicated that the fractions had different particle size distributions. Because of difficulties in aerosolizing the samples, accurate particle size distribution parameters could not be obtained. The sizes listed are representative of particles from an Andersen impactor sampling at the given flow rate.[18]

C. Sample preparation

Samples were prepared by filtering aliquots of isopropanol suspensions of material through filters. The suspensions were sonicated prior to the withdrawal of aliquots. The loadings of material deposited onto filters was calculated based upon the nominal concentration of the suspension.

DISCUSSION

This investigation of the determination of alpha-quartz on filter media using FT-IR proceeded in the following steps:

A. Selection of suitable filters

B. Examination of purge effects

C. Calibration curve formation and estimation of limit of detection

D. Examination of interference effects

E. Examination of particle size effects

Each of these steps will be discussed individually below.

A. Selection of Suitable Filters

The initial step was the selection of filters which could be used for on-filter determinations of quartz. The fifteen commercially-available filter varieties investigated were: polyvinyl chloride [PVC] (Nuclepore PVC, VM-1, GLA-5000, and MSA FWSB), PVC co-polymer (DM-450 and DM-800), mixed cellulose ester [MCE] (AAWP and GN-6), polytetrafluoroethylene [PTFE] (Teflo and Zefluor), nylon (Nylaflo), polyester (Nuclepore PE), polysulfone (HT-450 and GA-6S), and polyvinylidene difluoride (FP-450).

A representative spectrum of each filter type is included in Appendix A.

Samples of filters were examined to determine if the filter transmitted in the spectral range to be used for quantitation of quartz (560 - 800 cm^{-1}). Ideal filters would have a low constant absorbance in this range. It has been noted that quantitative analysis is most reliable if the measured absorbance is less than 0.7 A.U. and, in fact, a more stringent criterion of 0.5 A.U. has

been suggested to insure that Beer's Law is obeyed.[19] The following filters failed this criterion and were thus dropped from consideration: Zeflour, FP-450, HT-450, Nuclepore PVC, AAWP, GN-6, Nylaflo, and GA-6S. The VM-1 and MSA FWSB varieties of PVC filters were found to have, not unexpectedly, the same general spectra as the other PVC filters, but had higher absorbances. Therefore, only the two types of PVC filters with the lowest absorbances were examined further.

For those which met the absorbance criterion, the variation of absorbance between filters and within-filter were examined as a measure of the homogeneity of the filter surface. Filters of a given type were preweighed and sorted into groups by weight, which translated into roughly low, medium, and high weight groups. For each filter type, three filters were examined from each of the three different weight ranges. Each filter was mounted in the filter holder, scanned three times (64 co-added scans), rotated about 120 degrees and rescanned three times, and repeated for a total of eighty-one scans per filter type. Rotation of the filter allowed for the estimation of spatial homogeneity.

The absorbance at each prospective analytical wavelength for quartz was determined from these spectra. Absorbances for typical filter samples of known weight are given in Table I.

An index of the variability across the filter surface was determined by normalizing each absorbance value to the weight of the filter and evaluating the standard deviation of this value for the each weight-matched set of filters. The relative standard deviations (R.S.D.) did not vary with weight. These relative standard deviations were pooled to serve as an estimate of the

uniformity of filter thickness. The variation for Teflo filters was found to be too large and thus it was eliminated from further consideration.

This left only the "DM" series filters, GLA-5000, and Nuclepore PE for further study. The DM-450 and DM-800 filters represented the best filter in terms of lowest (absolute) absorbance and lowest relative standard deviation of absorbance of all filter types examined. DM Metricel filters, both the 0.8-micrometer pore size (DM-800) and the 0.45-micrometer pore size (DM-450), have greater than 99% filtration efficiency to 0.03 micrometers and thus are suitable for aerosol sampling.[20] Considerations of backpressure and capacity will determine which of the DM-series filters would be best for general use as sampling media. DM-800 would probably be best choice as it has a larger pore size and thus would have a lower pressure drop across the filter. It has been adopted for use in a number of direct-on-filter IR methods.[10,12,13] The DM-450 filters were found to have the best analytical performance.

Plots of measured absorbance at 798 cm^{-1}, 779 cm^{-1}, and 695 cm^{-1} of DM-450 filters versus weight are given as Figures 1, 2, and 3. The correlations are linear but there is significant overlap of the absorbance measurements across different weights, especially in the 695 cm^{-1} plot.

In an attempt to discern the cause of this variation, the data were examined to find intra- and inter-filter variation for the weight matched filters. The relative standard deviation for individual DM-450 filters (within-filter) ranged from 1.29 to 2.37 percent at 798 cm^{-1}, with similar values at 779 cm^{-1} and 695 cm^{-1}. This is in agreement with earlier work which estimated such variation to be within two percent.[13] Two percent

variation would amount to 3.4 micrograms for the DM-450 filter in the table. The relative standard deviations of DM-450 filters (between-filter variation) is, within experimental error, the same as that observed for within-filter variations. Similar results were found for DM-800 and Nuclepore PE filters.

Samples of DM-800 filters were scanned for 1280 scans to increase their signal-to-noise relative to the earlier measurements of 64 scans, and hence reduce any instrumental contribution of error. The variation, both within-filter and between filter, did not decrease. Thus, the conclusion is that differences in the filter absorbances are due to filter inhomogeneity rather than due to instrumental effects. This lack of filter homogeneity places a limit on the performance of direct-on-filter IR methods.

B. Examination of Purge Effects-

It was observed that the first run of a filter at a given orientation had a slightly higher absorbance than subsequent runs. This prompted a study of the effect of purging the instrument compartment with nitrogen prior to analysis.

Spectra of DM-800 filters were examined prior to purge and following a purge of nitrogen. Samples were placed in the instrument and immediately scanned. The sample, which was not moved, then was rescanned after a period of purge with nitrogen. The sample holder was removed, keeping the sample in the same nominal position relative to the holder, and allowed to equilibrate with the room environment. The sample holder was then reinserted and the procedure replicated. The absorbance of the unpurged filter was as much as 2% greater than that of the purged filter, which would amount to 4.9 micrograms

of quartz. The initial absorbance for the unpurged filters was not consistent, possibly due to a non-uniform equilibration environment.

This procedure when repeated with dry air purge gave equivalent results. The filters were found to give the same absorbance after purging when either air or nitrogen was used.

The experiments were repeated, but with the IR beam blocked during purge. Again, the absorbances did not vary, which eliminates heating of the filter by the IR beam as a parameter.

The experiments point out that purging results in better precision in determining the absorbance of the filters. Further, purging with dry air for twenty minutes was found to be sufficient to eliminate the effect. Finally, this effect was independent of the IR beam.

The effect of CO_2 on the wavelength window of interest was examined by purging the FT-IR with dry carbon dioxide [5.01% v/v in N_2] for about one hour. A sample filter used in an earlier section was then scanned with this CO_2-purged instrument. The absorbance values were then compared (the sample had not been moved from the earlier scan) to earlier runs of nitrogen purged spectra.

The absorbance at 695 cm^{-1} was greatly affected; it increased by as much as 0.1424 A.U. which is equivalent to 1.2 milligrams of quartz. This would amount to 6 micrograms at typical CO_2 levels of 0.03%. The bands at 798 cm^{-1} and 779 cm^{-1} were not affected at this level of carbon dioxide. The value of 5.01% carbon dioxide is far larger than one would encounter in a normal environment, and, hence, quartz determinations at the 798 cm^{-1} and 779 cm^{-1} analytical wavelengths are rugged with respect to the presence of

carbon dioxide.

C. Preparation of calibration standards

After the filter had been selected, the linearity of response for the analyte was determined and the limit of detection (LOD) estimated. Three suspensions of quartz in isopropanol were prepared, at 10, 20, and 50 milligrams per liter, and standards were prepared by filtering aliquots through preweighed filters. Triplicate samples were prepared at the following levels: 20, 30, 40, 50, 80, 100, 200, 250, 400, and 1000 micrograms. A Millipore chimney with a deposition area of 349 mm^2 was used, which enables approximate comparison with a directly-deposited sample collected with a nylon cyclone and a 25-mm cassette, which has an area of deposition of 394 mm^2. With full aperture, 10.7 percent of the area of the sample is interrogated by the IR beam.

Three methods of quartz calibration were examined. Each is discussed individually below.

1. Calibration by Peak height after subtraction of estimated filter contribution

In this approach, the absorbance of the sample on a preweighed filter was determined at the analytical wavelength and the estimated absorbance of the filter at the same wavelength was subtracted from it to produce the net quartz absorbance. Filter absorbance estimates were obtained from the plots of absorbance versus weight determined in the earlier discussion on filter selection. The curve of net quartz absorbance, using the 798 cm^{-1} and 779

cm^{-1} analytical wavelengths, versus amount of quartz was found to be linear to one milligram (see Figures 4 and 5). A similar plot of peak heights at 695 cm^{-1} exhibited nonlinearity at about 500 micrograms (see Figure 6).

2. Calibration by Peak height after spectral subtraction of filter contribution

The subtraction of the entire spectrum of the filter background allows the analyst to identify interferents in a manner that the correction at a single wavelength does not.

The spectrum used for subtraction can be based on either (1) prescanning the collection filter prior to use or (2) a "representative" filter spectrum. From the earlier filter selection section, it was found that a weight-matched filter would be no worse an estimator of filter absorbance than the given filter itself, due to filter inhomogeneity. For that reason, as well as its ease of use, we opted for the subtraction of a representative filter based on weight.

The DM-450 spectra collected for the earlier filter selection section were normalized by weight and two filter spectra from each weight group were co-added to form the "representative filter spectrum" used for the rest of the study. Scaled subtraction of the filter component was performed by multiplying the "representative filter spectrum" by the weight of the given collection filter, and subtracting this absorbance spectrum from that of the filter of interest. The efficacy of this method was then checked on blank filters at a number of weights. The correction to these filters resulted in a flat baseline. This served as a check to verify that the absorbance per weight behavior is independent of wavelength. However, the subtraction did

result in an offset from zero. The magnitude of the offset ranged from -0.0034 to 0.0018 at 798 cm^{-1}, -0.0036 to 0.0017 at 779 cm^{-1}, and -0.0077 to 0.0026 at 695 cm^{-1}. This translates to between four and six micrograms of quartz for the maxima observed. The effect of this offset is to introduce additional error in the determination of the analyte, and hence, it needs to be eliminated.

A baseline-correction routine was adopted to alleviate this error due to the offset. In the baseline correction routine points are established on both sides of the absorption band and a baseline is drawn between them. The peak height (absorbance) at the analytical wavelength is then determined from this baseline.

The points at which to draw the baseline were determined from examination of quartz spectra and their derivatives. The baseline points were defined by the minima around the bands of the absorbance spectrum of quartz used for quantitation. The baseline points were checked against those of the derivative of the spectra. Points equal to zero in the first derivative of the spectra which do not correspond to maxima or inflection points of the absorbance spectra represent minima of the absorbance band.

For the 798 cm-1, 779 cm-1 quartz doublet, slight band broadening was observed. The absorbance band at the one milligram loading level was not fully confined within the bounds chosen, notably 831 cm^{-1} and 750 cm^{-1}. However, the error in using these points for determination at that level was found to be -1.5% (at 798 cm^{-1}) and -2.9% (at 779 cm^{-1}). One milligram is sufficiently above the REL for a full shift sample that these errors would not affect the conclusions drawn from determinations at this level.

The 695 cm^{-1} peak was found to have a much larger problem with band broadening. Due to the proximity of the CO_2 absorption band, the quartz absorption band at this wavelength does not reach the (normal) baseline. The result is an apparent shift in the minimum (inflection point of the curve) which is a function of the loading. The points chosen for the baseline of the 695 cm-1 band, 707 cm^{-1} and 682 cm^{-1}, were found to be best for quartz loadings up to 200 µg. The curve was only examined from twenty to two-hundred micrograms at this wavelength.

The baseline-corrected calibration curves were plotted and the limits of detection computed (see Figures 7, 8, and 9). Again, the calibration curves were found to be linear to one milligram for the 798 cm^{-1} and 779 cm^{-1} curves and linear to about 500 micrograms for the 695 cm^{-1} curve. The slope of the curves, with a restricted range of samples from 20 microgram to 200 microgram, was 0.44 A.U./ milligram at 798 cm^{-1}, 0.39 A.U./ milligram at 779 cm^{-1}, and 0.10 A.U./ milligram at 695 cm^{-1}.

The limit of detection defined as three times the standard deviation of a blank was estimated to be 2.9 micrograms at 798 cm^{-1} and 5.3 micrograms at 779 cm^{-1}, based on the standard deviation determined from the baseline-corrected values from twenty blank filters. An analogous instrumental LOD, at 798 cm^{-1}, from ten repetitive scans of a single (unmoved) filter was determined to be 0.40 micrograms.

3. Calibration by integrated absorbance after spectral subtraction of filter contribution

The integrated absorbance of the quartz doublet at 798 cm-1, 779 cm-1 has

been proposed as a measure of the quartz content of samples which is less sensitive to particle size.[21] Toward this end, the integrated absorbance of the baseline-corrected intensity was investigated for use in quartz determination.

The absorbance spectra of the quartz-loaded samples were corrected for filter contribution by spectral subtraction. Then, a baseline was drawn as described above and the integrated absorbance above the baseline determined for the samples. The resultant baseline-corrected integrated absorbance calibration curve is given as Figure 10. The slope of the curve, with a restricted range of samples from 20 microgram to 200 microgram, was 12.9 A.U.-cm^{-1}/ milligram. Although the sensitivity, defined as absorbance per microgram, for the curves of integrated absorbance and background-corrected peak absorbance at 798 cm^{-1} are comparable, the integrated absorbance measurement is more prone to errors from the filter subtraction routine as well as interferents which may be present.

D. Examination of interference effects

The interferences of coal dust, kaolinite, and cristobalite were investigated by co-depositing them with known amounts of quartz onto preweighed filters. The coal dust served as a broad band absorber. It has a broad ill-defined absorbance band which varies in an approximately linear fashion across the analytical wavelengths under study. Kaolinite and cristobalite, on the other hand, possess distinct absorption bands which overlap one or more of those of quartz, as noted in NIOSH Method 7602.[5] The study investigated the effects of the interferents cristobalite or

kaolinite either alone or at ten percent in a coal dust matrix. Quartz estimates were based on the absorbance at 798 cm^{-1} because of superior analytical performance at that wavelength.

Initially, calibration curves of the interferents were prepared to examine their linearity of response. Isopropanol suspensions of each interferent were prepared. Calibration samples of kaolinite were prepared, in duplicate, at load levels of 50, 100, 250, 500 and 1000 micrograms. Cristobalite samples were prepared, in triplicate, at lower levels of about 40, 80, 120, and 160 micrograms.

An absorption band of the interferent was identified which did not overlap any bands in the quartz spectrum. This unique line was then used as an indicator of the amount of the interferent in the sample. The spectra from the calibration samples were used to establish a curve of baseline-corrected absorbance at the 798 cm^{-1} analytical line for quartz versus baseline-corrected absorbance at the unique interferent line (see Figures 11 and 12). The analytical line chosen for kaolinite was 913 cm^{-1}, with a baseline drawn from 954 cm^{-1} to 827 cm^{-1}. The cristobalite analytical line was taken to be 621 cm^{-1}, with its baseline drawn from 636 cm^{-1} to 600 cm^{-1}. These correction curves were found to be linear in the range of forty to two hundred micrograms. Representative spectra of the interferents were prepared by co-adding two spectra at each of the loadings.

The coal dust spectrum was examined along side that of a quartz spectrum (Figure 13). A slight negative curvature in the coal spectrum was found at the quartz analytical lines. Correction of the baseline to minimize the effect of this small bias was not possible. Expanding the baseline beyond the

earlier points would increase the amount of bias of the coal dust due to the presence of a sharp drop in the coal dust spectrum beyond the upper set point. Narrowing the width of the baseline would not change the magnitude of the coal dust bias but would result in a smaller absorbance for quartz and hence a larger relative error due to the presence of coal dust.

An experimental design was developed that would discern whether the values obtained in the study were within 20% of the true value with 95% confidence. A randomized complete block with three factors- quartz, coal dust, and either kaolinite or cristobalite- was employed for the study. Triplicate replication of the design provided sufficient power to estimate the effect of the interferences.

Isopropanol suspensions of the interferents were prepared so as to deliver, when present, one milligram of coal dust and 100 micrograms of either kaolinite or cristobalite. The quartz loadings in the samples were set at either fifty or two-hundred micrograms, if present. To insure thorough mixing and uniform deposits, aliquots of the interferents and quartz were delivered to large round-bottom test tubes. The samples was sonicated, then stirred using a vortex mixer, prior to filtration through a preweighed DM-450 filter. The prepared samples were run randomly in order to randomize any instrumental drift.

The two means employed earlier in the calibration curve experiments -- simple estimation of absorbance contribution and spectral subtraction -- were used to correct for the interferents contribution, using the correction curves and "representative spectra" above. In all cases, the filter spectrum was spectrally subtracted prior to further analysis.

An illustration of the filter and interference correction procedures can be seen in Figure 14. Curve A represents the samples total absorbance, which includes filter, quartz, coal dust, and cristobalite. Curve B is the result of subtraction of the filter's absorbance contribution. Subtraction of the filter's absorbance contribution allows the identification of interferents, in this case, cristobalite. Spectral subtraction of the cristobalite yields Curve C as the resultant spectrum. The spike at 667 cm^{-1} is due to differences in the carbon dioxide levels between the spectra. The baseline-corrected absorbance is then calculated from this resultant spectrum.

For ease of discussion, the influence of kaolinite in the presence and absence of coal dust upon the recovery of quartz will be presented first. Then, cristobalite's influence on quartz recovery will be discussed.

1. Interference study of alpha-quartz, coal dust, and/or kaolinite

Quartz recoveries in the absence of interferents are seen to be quantitative, at least 97%, at both levels (Table II). The presence of the broad band absorber results in negative bias of seven to eight percent. Some of the bias could be traced to that of the quartz estimates of -1.3 microgram in the samples containing only coal dust.

The kaolinite interferent study (Table II) shows a definite bias for both levels of quartz, whether in the presence of the additional coal dust or not. The magnitude of the bias does appear to be level dependent. One case, that of kaolinite-only low-level quartz samples, results in excess of twenty percent error in quartz estimates. No significant difference in the quartz estimates was observed between values where the interferent was spectrally

subtracted and those where the interferent contribution was estimated from the (correction) calibration curve. The estimates of quartz for samples containing both coal dust and kaolinite as interferents are slightly higher than those obtained from filters containing only coal dust presumably due to errors in the interference correction.

2. Interference study of alpha-quartz, coal dust, and/or cristobalite

Similar results are seen in the cristobalite interferent study (Table III). Whereas no groups are in excess of twenty percent error, the low-level quartz in the presence of cristobalite has a negative bias of about twelve to seventeen percent. The estimates of quartz for filters containing only this interferent is even larger than that found for kaolinite. As with the kaolinite study, similar results were obtained with both methods of interference correction.

This study limited itself to two interferents being present in any given sample. It is likely that a number of compounds would be present, all of which would need to be confirmed as to identity, and corrected accordingly. Despite knowledge of identity and level of interferent, some samples of quartz were underestimated in excess of twenty percent.

E. Examination of particle size effects

An investigation of particle size effect on quartz IR absorbances was performed to verify the results of other investigators. Isopropanol suspensions of the sized quartz fractions were prepared and aliquots dispersed onto preweighed filters. Duplicate samples were prepared for each stage at

loadings of 50, 100, and 200 micrograms. Spectra were collected as described in the Experimental Section. The baseline-corrected absorbances at 798 cm^{-1} and 779 cm^{-1} were normalized to the weight of the sized-quartz deposit, and are given as Tables IV and V. The integrated absorbance across these bands was also obtained, the weight-normalized values are given as Table VI.

The data indicate a definite variation in the absorbance per unit weight as a function of particle size. The magnitude of the difference between fractions was as high as 30%. This behavior agrees with that noted by other researchers.[12] Due to lack of material, the very small particle sizes (less than 0.65 micrometers) investigated by others could not be examined.

The integrated absorbance of the doublet (Table VI) was also found to vary as a function of the particle size. The variation of this intensity, however, is larger than that for baseline corrected peak heights. This indicates that any correction for particle size mismatch between sample and standard would best be performed by peak height ratios rather than by integrated absorbance. It should also be noted that the integrated absorbance would be more prone to error from interferences.

While the differences in absorbance per unit weight of sized quartz below four micrometers were fairly close, samples with particle sizes larger than four micrometers appear to be significantly different from the parent reference material. The intensities of these materials dropped to below 30% of the response of SRM 1878. This would result in a serious underestimate of the quartz present for samples with particle distributions skewed toward the larger end of the respirable range.

The use of peak height ratio of the quartz 798, 779 cm^{-1} doublet has

been proposed for correction of particle size mismatch between the reference standard and the sample.[14] Toward this end, the peak height ratios of the sized quartz were examined to determine if any trend could be established. The peak ratios of the baseline-corrected absorbances of the 798/779 cm^{-1} doublet were found to increase with decreasing particle size, in the range studied. The peak ratios ranged from 1.0 for samples of the stage containing particles 4.7 to 5.8 micrometers, to 1.3 for samples of the stage containing particles 0.65 to 1.1 micrometers. No dependence of load level on peak ratio was found. The correlation of the peak ratio with particle size and its immunity to the amount of quartz on the filter suggest that this is a promising approach for correction of particle size mismatch between samples and standards.

SUMMARY AND CONCLUSIONS

This investigation explored some factors which would affect the performance of a direct-on-filter method for quartz by FT-IR. Test samples were limited to those prepared by liquid filtration rather than collection of an aerosol. The use of FT-IR to analyze for quartz on 25-mm filters does not offer any substantial improvement in analytical performance over existing dispersive IR methods. Also, this approach is more vulnerable to the presence of interferences since no sample pretreatment is used before analysis and procedures to correct for the presence of expected interferences were not totally successful.

While the direct analysis of the collection media (a direct-on-filter

method) will eliminate errors due to the work up of the sample, it is possible that uneveness in the deposit of the aerosol of the collection filter would mitigate this advantage. This aspect was not investigated here.

Even with the best filter from an analytical perspective (DM-450), variations in the absorbance of the filter due to spatial inhomogeneity restrict the limit of detection to about three micrograms of quartz (three times the standard deviation of the blank). This LOD represents a twofold improvement in the value which has been reported for Method 7602.

Any potential problems due to carbon dioxide or water vapor could be easily controlled. Purging the sample compartment for 20 minutes was found to be sufficient to yield reproducible spectra.

Direct-on-filter IR methods are inherently more susceptible to interferences than KBr-pellet IR methods, because they do not have an ashing step which would eliminate interferents such as coal dust. Methods used to correct for interferents were found to overcorrect the net absorbance attributed to quartz which resulted in calculated quartz values being underestimated by as much as twenty percent. This bias was found whether the interferences were corrected by spectral subtraction or by simple estimation based on calibration curves.

A dependence of absorbance on particle size was observed.

SUGGESTIONS FOR FUTURE WORK

Two approaches might serve to extend the limit of detection of an IR method for quartz. In the first case, the sample could be ashed and the resultant material redeposited onto a preweighed filter, but with a smaller

deposition area (10-mm). This allows an enhancement by about a factor of six in the density of material on the filter. In addition, it would eliminate coal dust as an interferent. However, filter inhomogeneity would still be a limitation to such a method and the prospect for errors during sample preparation would be introduced.

The second approach would be to simply extend the present KBr-pellet dispersive IR methods to FT-IR. Potassium bromide is transparent in the analytical infrared region and thus does not interfere with quartz determinations. An FT-IR method where the samples are analyzed in 13-mm KBr pellets would allow about a factor of four enhancement in quartz density, would eliminate coal dust as an interferent, and would remove filter homogeneity as a factor in the analysis. The limiting factors would then be transfer of sampled dust and the homogeneity of the pressed pellets, which are more difficult to control operator-dependent factors.

Multicomponent analysis by least squares might provide a better mechanism for correction of interferences,[22] including that due to the filter background. However, caution must be taken that all materials present in the matrix be determined since minor, unsuspected bands on the wings of overlapping peaks can result in errors in the calculated values.[23]

DISCLAIMER

Mention of commercial names or products does not constitute endorsement by the National Institute for Occupational Safety and Health.

ACKNOWLEDGEMENT

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Table I

Infrared Absorbance of Selected Filters

Filter Name	Filter Type[a]	Representative Weight (mg)	Absorbance at Selected Quartz Bands[b] 798 cm^{-1}	779 cm^{-1}	695 cm^{-1}
Nuclepore PE	PE	4.90	0.157 ± .003	0.116 ± .004	0.057 ± .005
Teflo	PTFE	4.50	0.063 ± .010	0.074 ± .011	0.157 ± .021
DM-450	PVC/A	12.60	0.074 ± .001	0.078 ± .001	0.163 ± .003
DM-800	PVC/A	14.13	0.108 ± .003	0.111 ± .003	0.205 ± .005
GLA-5000	PVC	5.31	0.112 ± .006	0.105 ± .006	0.222 ± .005

a. PTFE = polytetrafluoroethylene; PVC = polyvinylchloride; PVC/A = polyvinylchloride-acrylonitrile co-polymer; PE = polyester.

b. The numbers below the absorbances are estimates of the standard deviation of the absorbance for the representative filter.

Table II

Percentage Recovery of Quartz
in Presence of Coal Dust and/or Kaolinite
Using Scaled Spectral Subtraction[a]

Amount of Quartz (mg)	COAL	INTERFERENCES[b]			
		–	–	+	+
	KAOLINITE	–	+	–	+
0		N/A	-4.4[c] ± 1.0	-1.3[c] ± 0.6	-3.2[c] ± 0.8
0.0498		99.9 ± 1.0	80.8 ± 2.9	93.4 ± 0.8	86.4 ± 3.5
0.199		97.7 ± 1.1	94.9 ± 5.5	89.9 ± 4.4	85.7 ± 7.9

(a) The numbers below the recoveries are the standard deviations for the set with the interferences in the amount shown.

(b) The amounts of interferences were, when present, 1000 micrograms for coal and 100 micrograms for kaolinite. [+, interferent present; –, interferent absent]

(c) These numbers are the negative bias (in micrograms) introduced into the method by the correction for the interference in the amount shown.

Table III

Percentage Recovery of Quartz
in Presence of Coal Dust and/or Cristobalite
using Scaled Spectral Subtraction[a]

Amount of Quartz (mg)	COAL	CRISTOBALITE	INTERFERENCES[b] – –	– +	+ –	+ +
0			N/A	-6.8[c] ± 2.0	-1.3[c] ± 0.6	-1.4[c] ± 2.9
0.0498			99.9 ± 1.0	87.9 ± 2.3	93.4 ± 0.8	97.2 ± 1.5
0.199			97.7 ± 1.1	94.2 ± 1.7	89.9 ± 4.4	93.7 ± 0.7

(a) The numbers below the recoveries are the standard deviations for the set with the interferences in the amount shown.

(b) The amounts of interferences were, when present, 1000 micrograms for coal and 100 micrograms for kaolinite. [+, interferent present; -, interferent absent]

(c) These numbers are the negative bias (in micrograms) introduced into the method by the correction for the interference in the amount shown.

Table IV

Sized Quartz
Background-Corrected Peak Absorbance at 798 cm^{-1}/ Weight (A.U./mg)

Loading	Stage 2 (4.7-5.8μm)	Stage 3 (3.3-4.7μm)	Stage 5 (1.1-2.1μm)	Stage 6 (.65-1.1μm)
50 μg	.349	.503	.530	.442
100 μg	.324	.488	.487	.442
200 μg	.300	.464	.477	.412

Table V

Sized Quartz
Background-Corrected Peak Absorbance at 779 cm^{-1}/ Weight (A.U./mg)

Loading	Stage 2 (4.7-5.8μm)	Stage 3 (3.3-4.7μm)	Stage 5 (1.1-2.1μm)	Stage 6 (.65-1.1μm)
50 μg	.335	.454	.411	.327
100 μg	.323	.434	.378	.336
200 μg	.298	.416	.375	.330

Table VI

Sized Quartz
Background-Corrected Integrated Absorbance of 798/ 779 cm^{-1} Doublet
(A.U. cm^{-1}/mg)

Loading	Stage 2 (4.7-5.8μm)	Stage 3 (3.3-4.7μm)	Stage 5 (1.1-2.1μm)	Stage 6 (.65-1.1μm)
50 μg	13.2	16.6	15.5	13.0
100 μg	11.9	15.2	13.5	12.5
200 μg	10.6	14.3	13.0	11.2

FIGURE CAPTIONS

Figure 1--Plot of measured absorbance of DM-450 filters at 798 cm^{-1} versus weight

Figure 2--Plot of measured absorbance of DM-450 filters at 779 cm^{-1} versus weight

Figure 3--Plot of measured absorbance of DM-450 filters at 695 cm^{-1} versus weight

Figure 4--Net quartz absorbance at 798 cm^{-1} after subtraction of estimated filter contribution

Figure 5--Net quartz absorbance at 779 cm^{-1} after subtraction of estimated filter contribution

Figure 6--Net quartz absorbance at 695 cm^{-1} after subtraction of estimated filter contribution

Figure 7--Baseline-corrected quartz absorbance at 798 cm^{-1} (after spectral subtraction of filter spectrum) versus weight.

Figure 8--Baseline-corrected quartz absorbance at 779 cm^{-1} (after spectral subtraction of filter spectrum) versus weight.

Figure 9--Baseline-corrected quartz absorbance at 695 cm^{-1} (after spectral subtraction of filter spectrum) versus weight.

Figure 10--Baseline-corrected integrated absorbance of quartz 798/ 779 cm^{-1} doublet (after spectral subtraction of filter spectrum) versus weight.

Figure 11--Kaolinite correction curve: baseline-corrected absorbance of kaolinite at 913 cm^{-1} versus baseline-corrected absorbance of kaolinite at 798 cm^{-1}

Figure 12--Cristobalite correction curve: baseline-corrected absorbance of cristobalite at 798 cm^{-1} versus baseline-corrected absorbance of cristobalite at 621 cm^{-1}

Figure 13--Coal dust absorption spectrum and absorption spectrum of 80 µg quartz

Figure 14--Illustration of spectral subtraction correction routine- Spectrum of quartz, coal dust, and cristobalite

Curve A- Total absorbance of sample

Curve B- Net absorbance after spectral subtraction of filter component

Curve C- Net absorbance after spectral subtraction of filter and cristobalite components

Figure 1

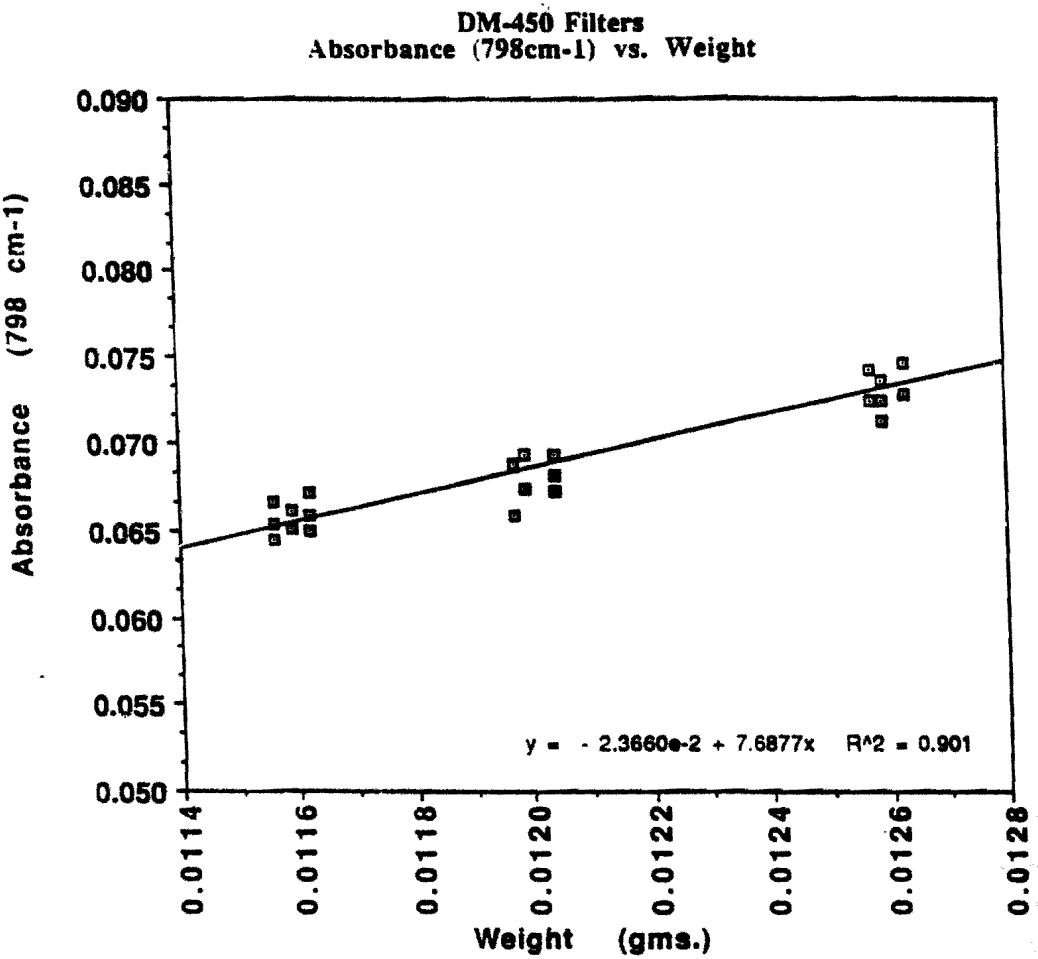

DM-450 Filters
Absorbance (798cm-1) vs. Weight
Absorbance (798 cm-1)
0.090
0.085
0.080
0.075
0.070
0.065
0.060
0.055
0.050
y = - 2.3660e-2 + 7.6877x R^2 = 0.901
0.0114
0.0116
0.0118
0.0120
0.0122
0.0124
0.0126
0.0128
Weight (gms.)

Figure 2

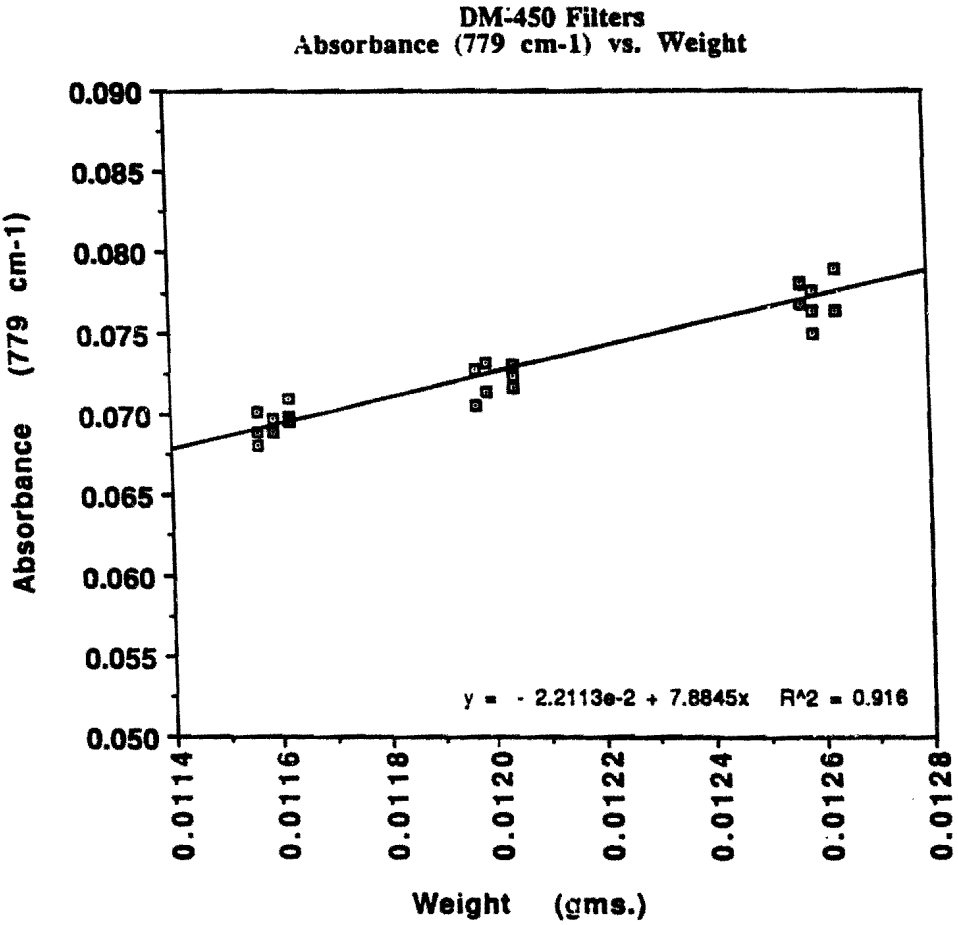
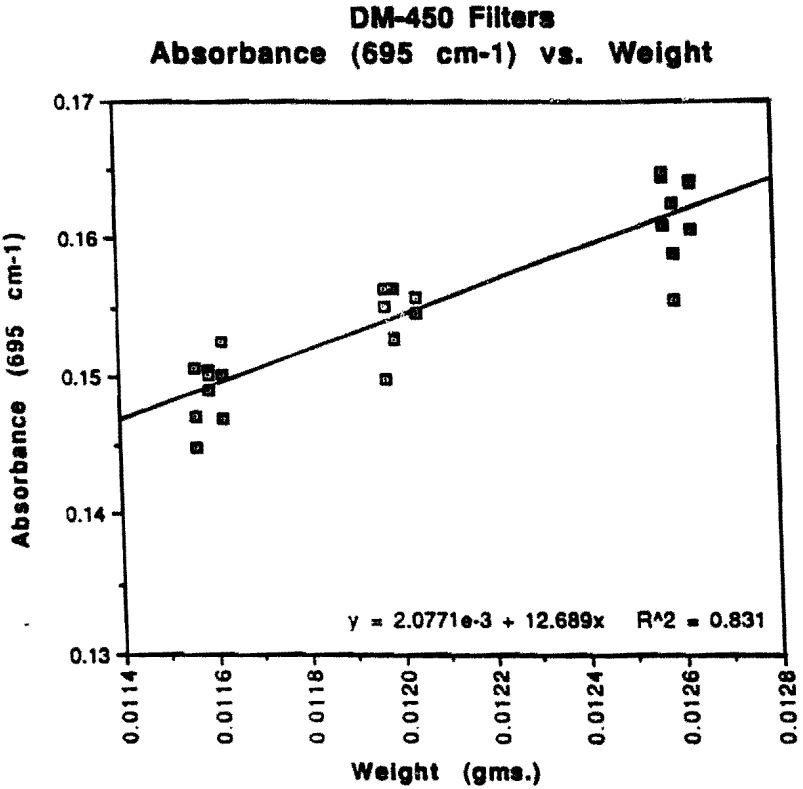

DM-450 Filters
Absorbance (779 cm-1) vs. Weight
Absorbance (779 cm-1)
0.090
0.085
0.080
0.075
0.070
0.065
0.060
0.055
0.050
y = - 2.2113e-2 + 7.8845x R^2 = 0.916
0.0114
0.0116
0.0118
0.0120
0.0122
0.0124
0.0126
0.0128
Weight (gms.)

Figure 3


DM-450 Filters
Absorbance (695 cm-1) vs. Weight
Absorbance (695 cm-1)
0.17
0.16
0.15
0.14
0.13
y = 2.0771e-3 + 12.689x R^2 = 0.831
0.0114
0.0116
0.0118
0.0120
0.0122
0.0124
0.0126
0.0128
Weight (gms.)

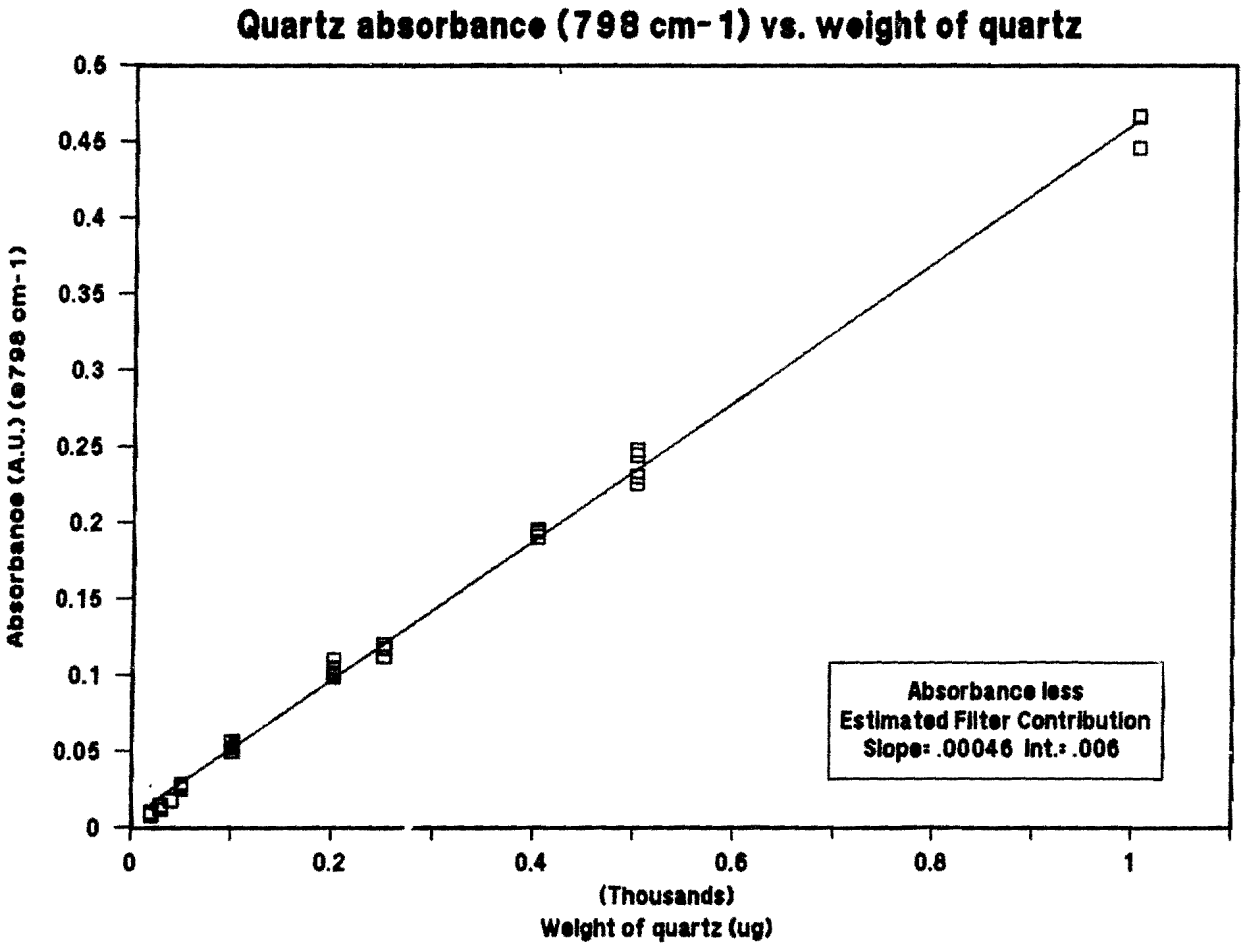

Quartz absorbance (798 cm-1) vs. weight of quartz
Absorbance (A.U.) (@798 cm-1)
0.6
0.45
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
0
0.2
0.4
0.6
0.8
1
(Thousands)
Weight of quartz (ug)
Absorbance less
Estimated Filter Contribution
Slope= .00046 Int.= .006

Figure 4

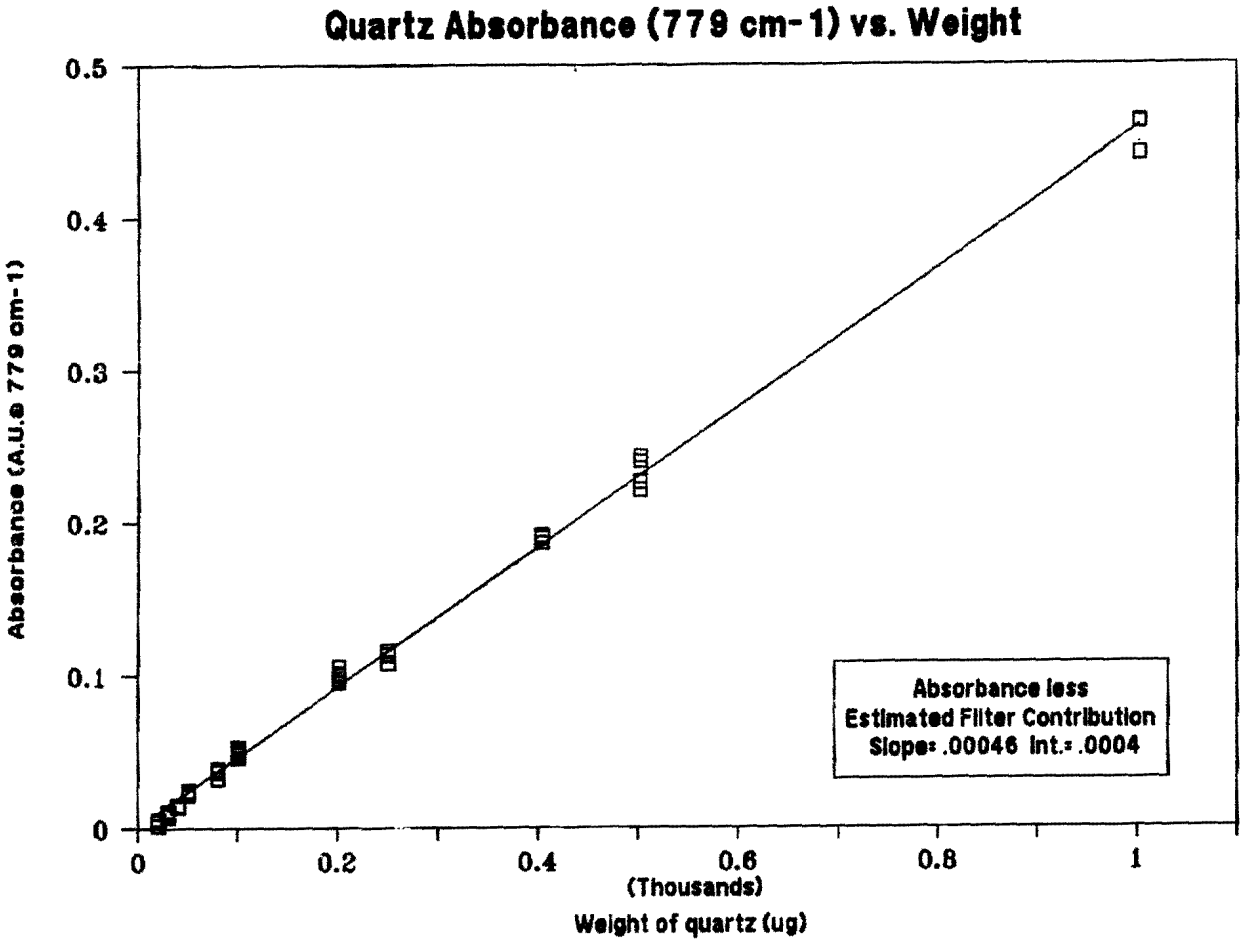

Quartz Absorbance (779 cm-1) vs. Weight
Absorbance (A.U.@ 779 cm-1)
0.5
0.4
0.3
0.2
0.1
0
0
0.2
0.4
0.6
0.8
1
(Thousands)
Weight of quartz (ug)
Absorbance less
Estimated Filter Contribution
Slope= .00046 Int.= .0004

Figure 5

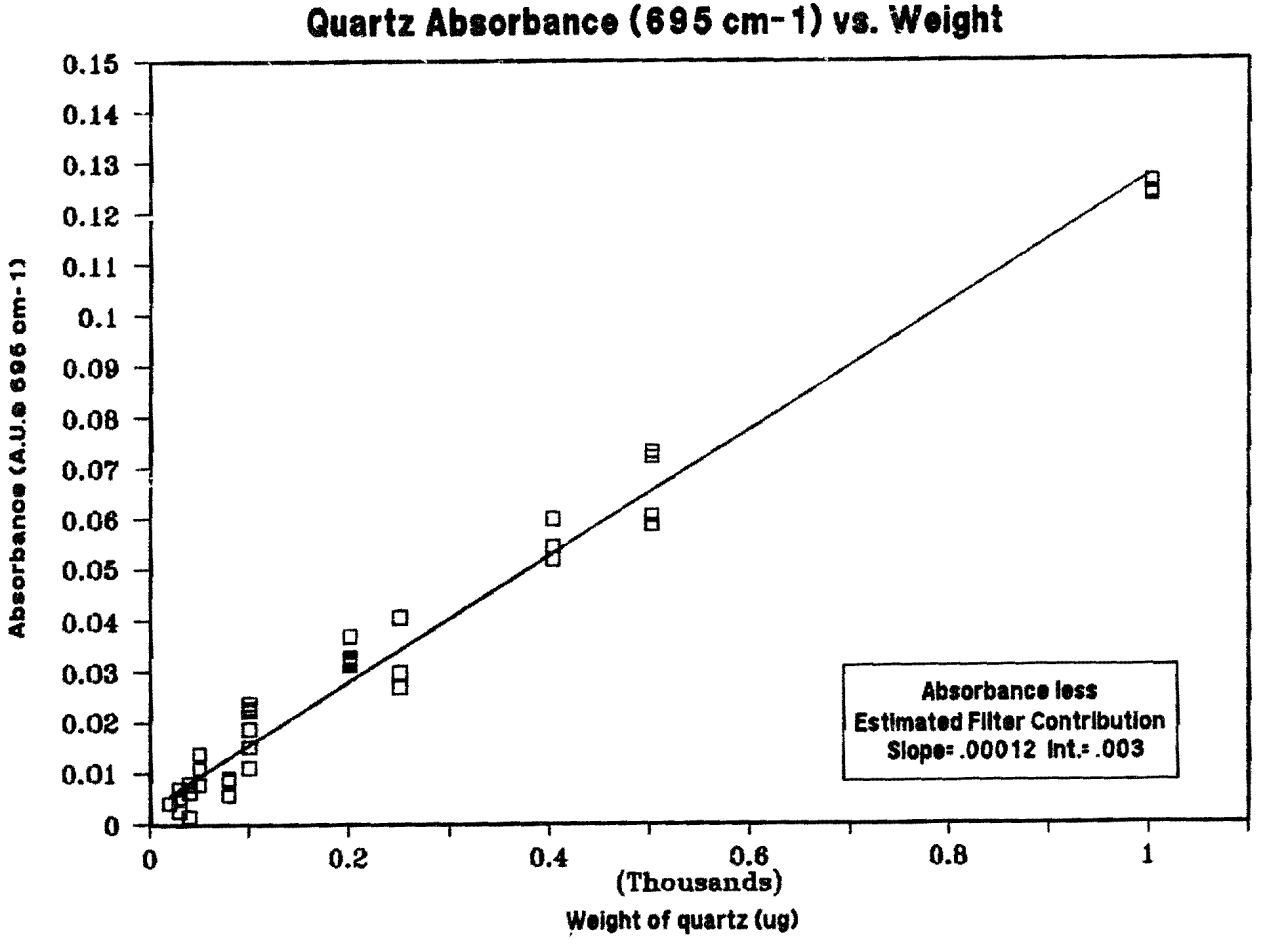
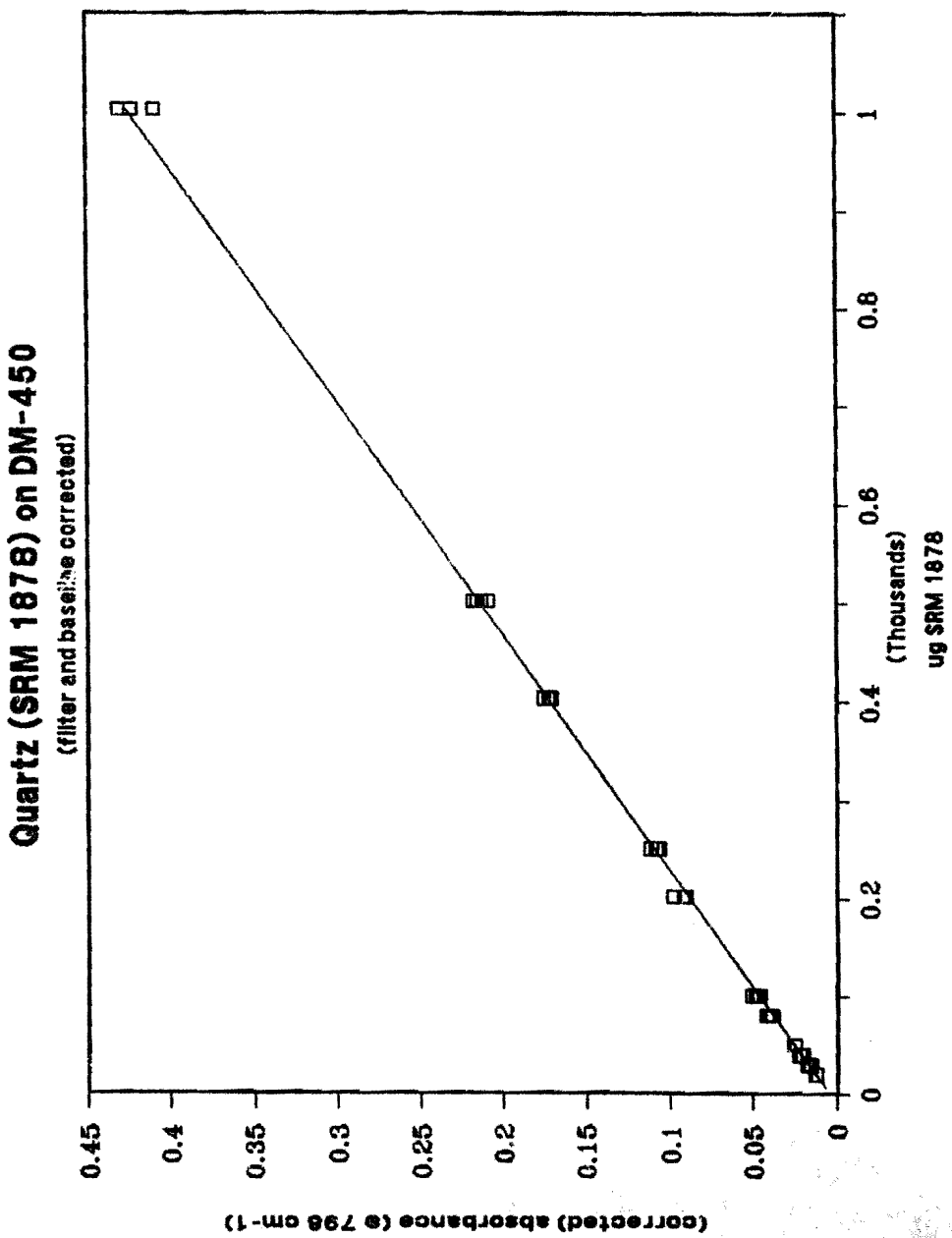

Quartz Absorbance (695 cm-1) vs. Weight
Absorbance (A.U.@ 695 cm-1)
0.15
0.14
0.13
0.12
0.11
0.1
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
0
0.2
0.4
0.6
0.8
1
(Thousands)
Weight of quartz (ug)
Absorbance less
Estimated Filter Contribution
Slope= .00012 Int.= .003

Figure 6

Figure 7


Quartz (SRM 1878) on DM-450
(filter and baseline corrected)
(corrected) absorbance (@ 798 cm-1)
0.45
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
0
0.2
0.4
0.6
0.8
1
(Thousands)
ug SRM 1878

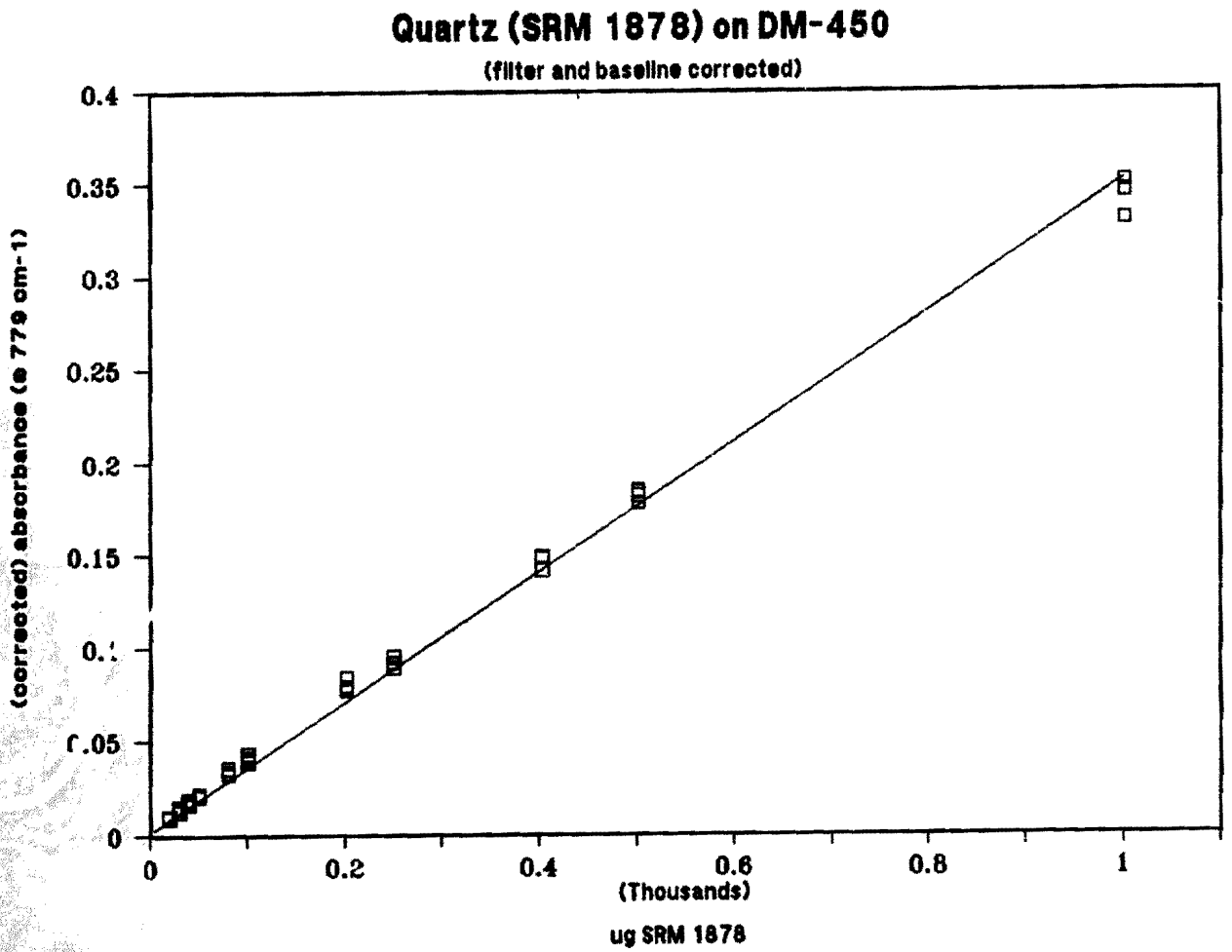

Quartz (SRM 1878) on DM-450
(filter and baseline corrected)
(corrected) absorbance (@ 779 cm-1)
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
0
0.2
0.4
0.6
0.8
1
(Thousands)
ug SRM 1878

Figure 8

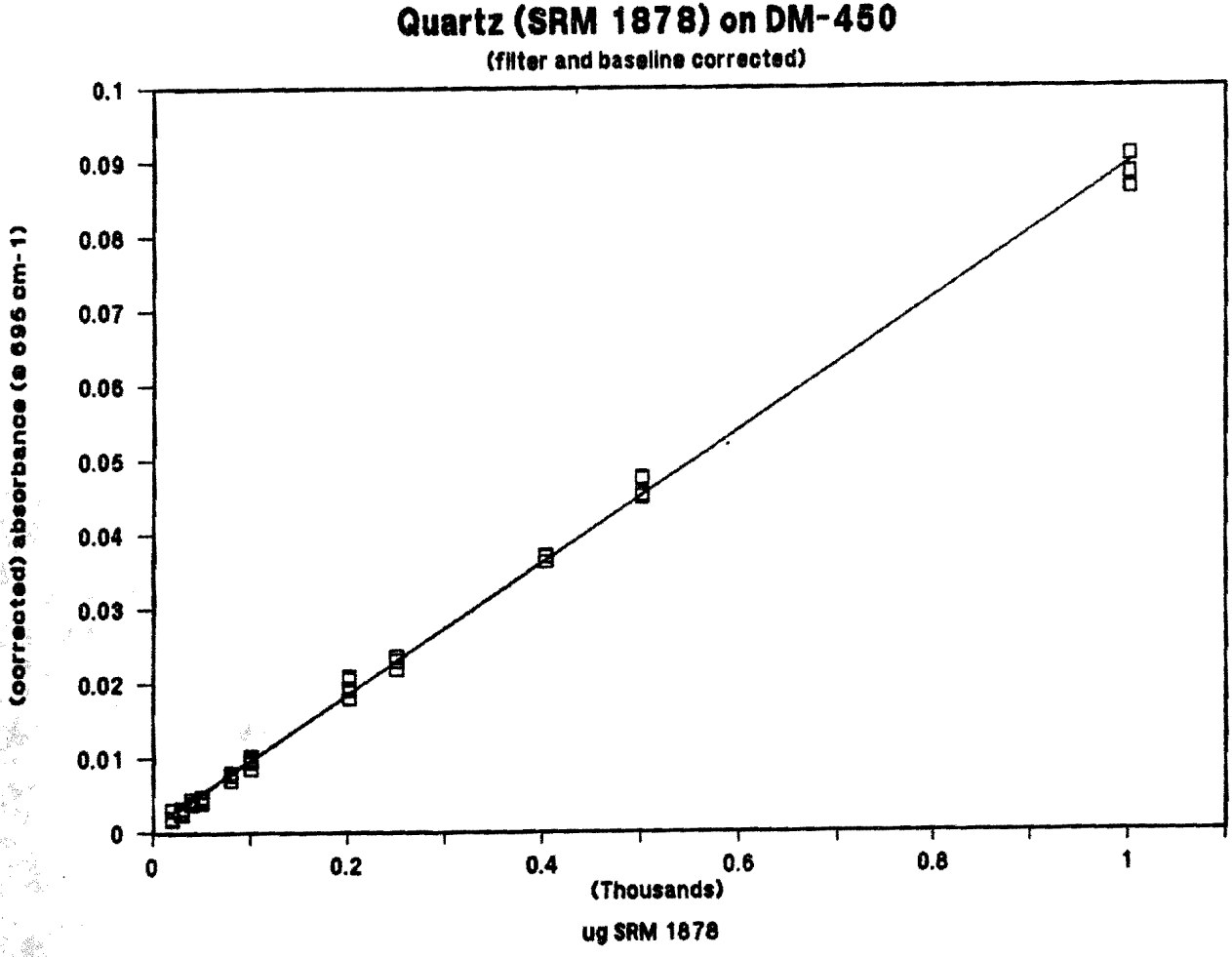

Quartz (SRM 1878) on DM-450
(filter and baseline corrected)
(corrected) absorbance (@ 695 cm-1)
0.1
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
0
0.2
0.4
0.6
0.8
1
(Thousands)
ug SRM 1878

Figure 9

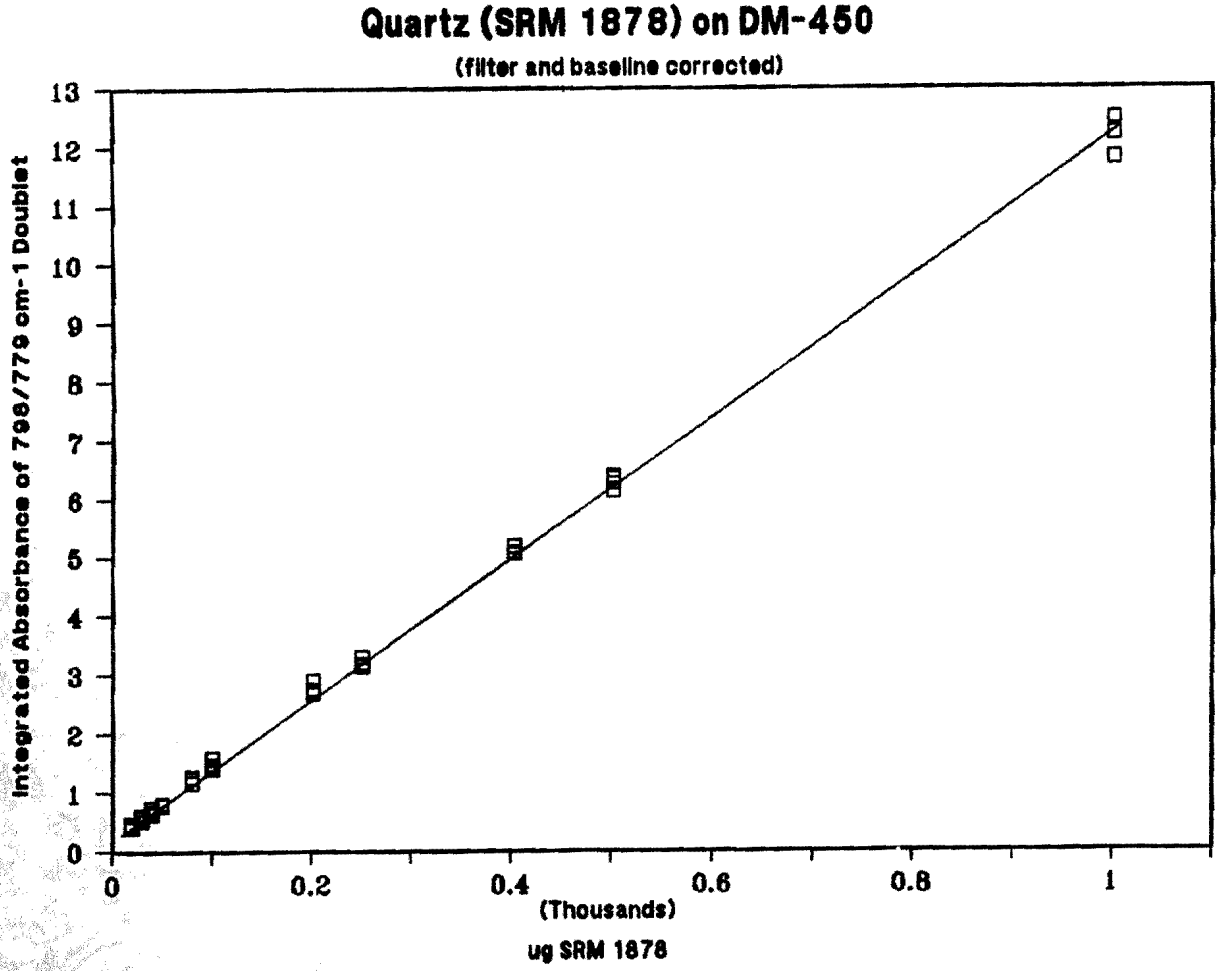

Quartz (SRM 1878) on DM-450
(filter and baseline corrected)
Integrated Absorbance of 796/779 cm-1 Doublet
13
12
11
10
9
8
7
6
5
4
3
2
1
0
0
0.2
0.4
0.6
0.8
1
(Thousands)
ug SRM 1878

Figure 10

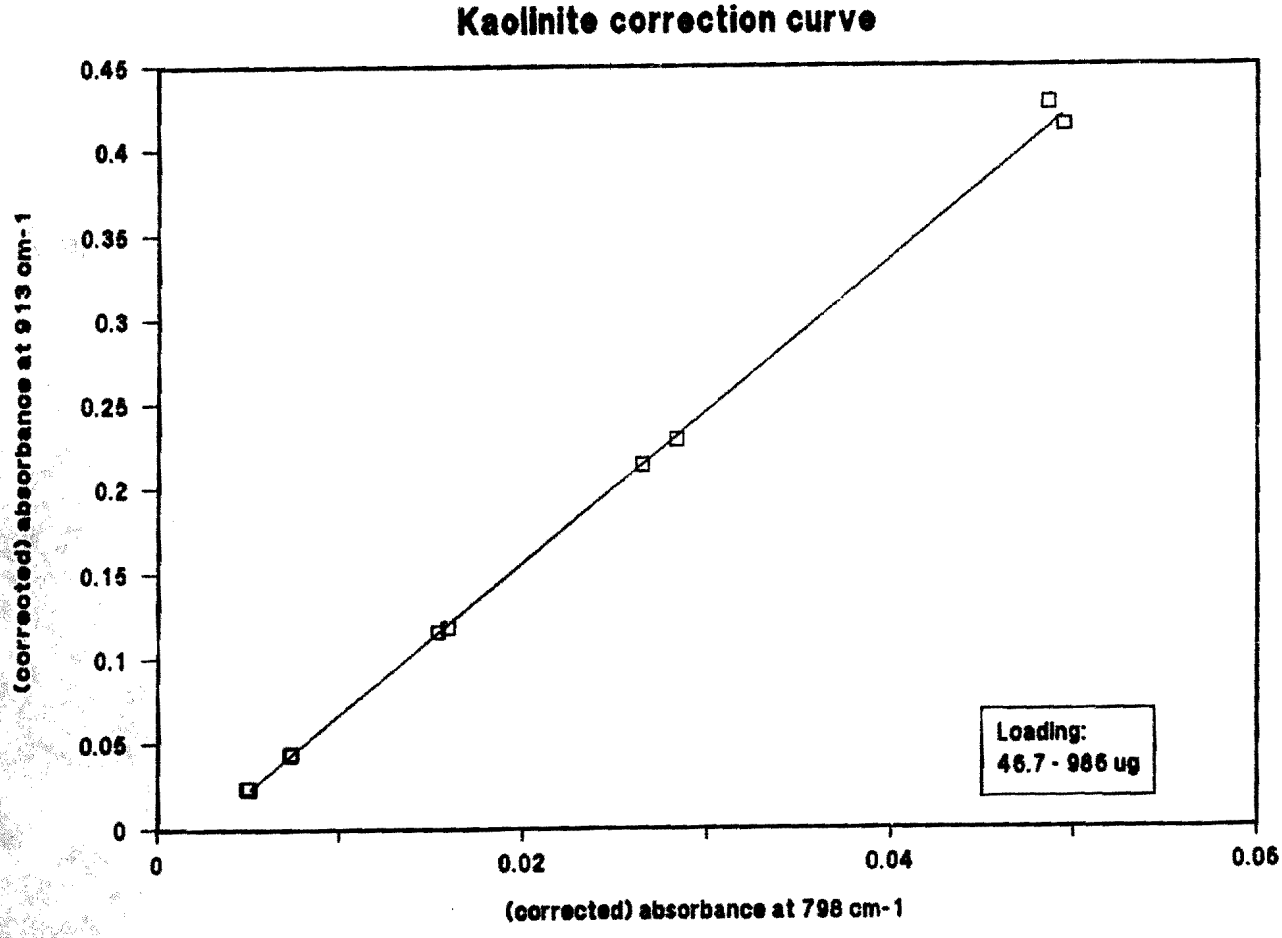

Kaolinite correction curve
0.45
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
(corrected) absorbance at 913 cm-1
0
0.02
0.04
0.06
(corrected) absorbance at 798 cm-1
Loading:
46.7 - 986 ug

Figure 11

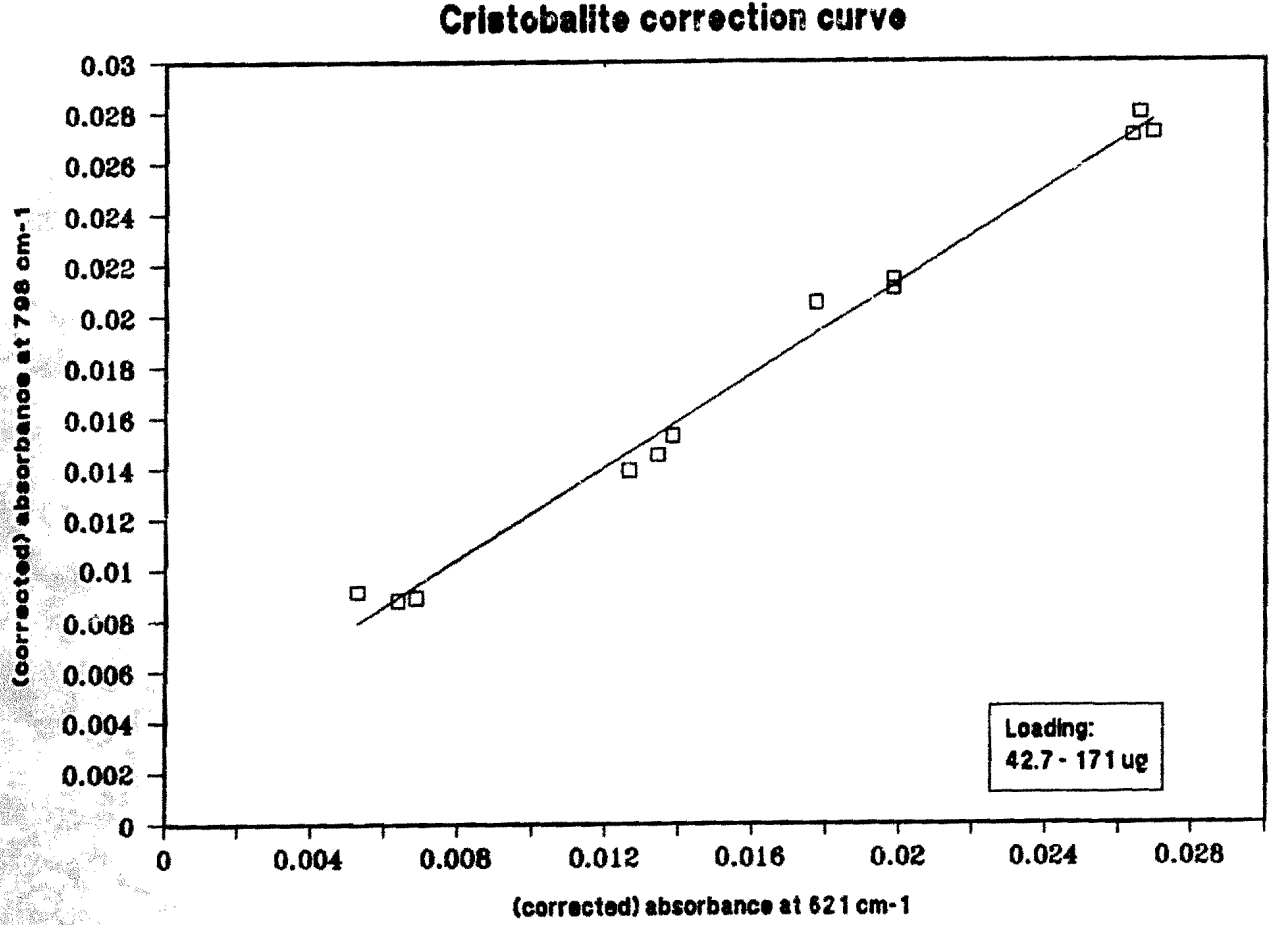

Cristobalite correction curve
(corrected) absorbance at 798 cm-1
0.03
0.028
0.026
0.024
0.022
0.02
0.018
0.016
0.014
0.012
0.01
0.008
0.006
0.004
0.002
0
0
0.004
0.008
0.012
0.016
0.02
0.024
0.028
(corrected) absorbance at 621 cm-1
Loading:
42.7 - 171 ug

Figure 12

Figure 13

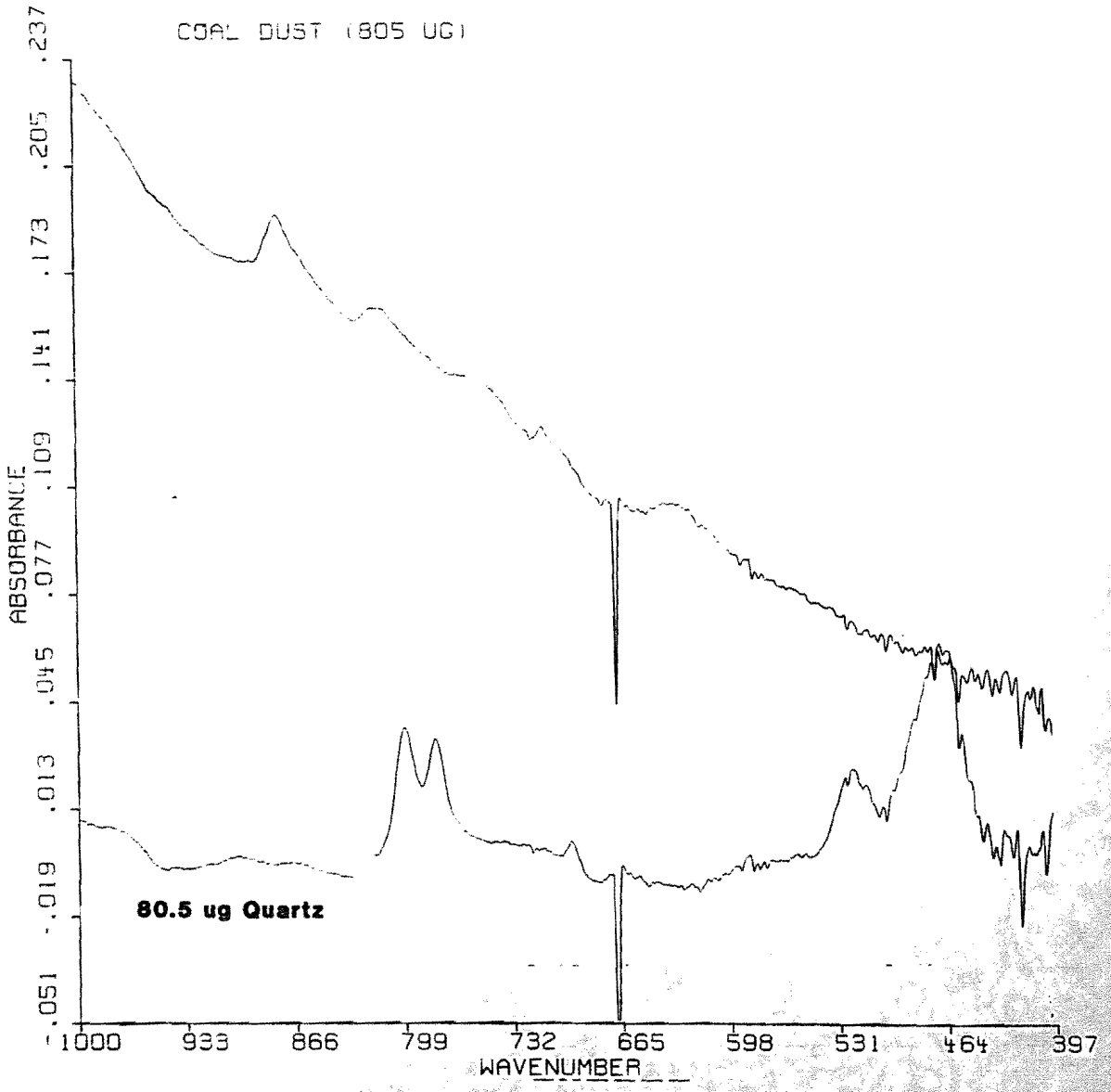
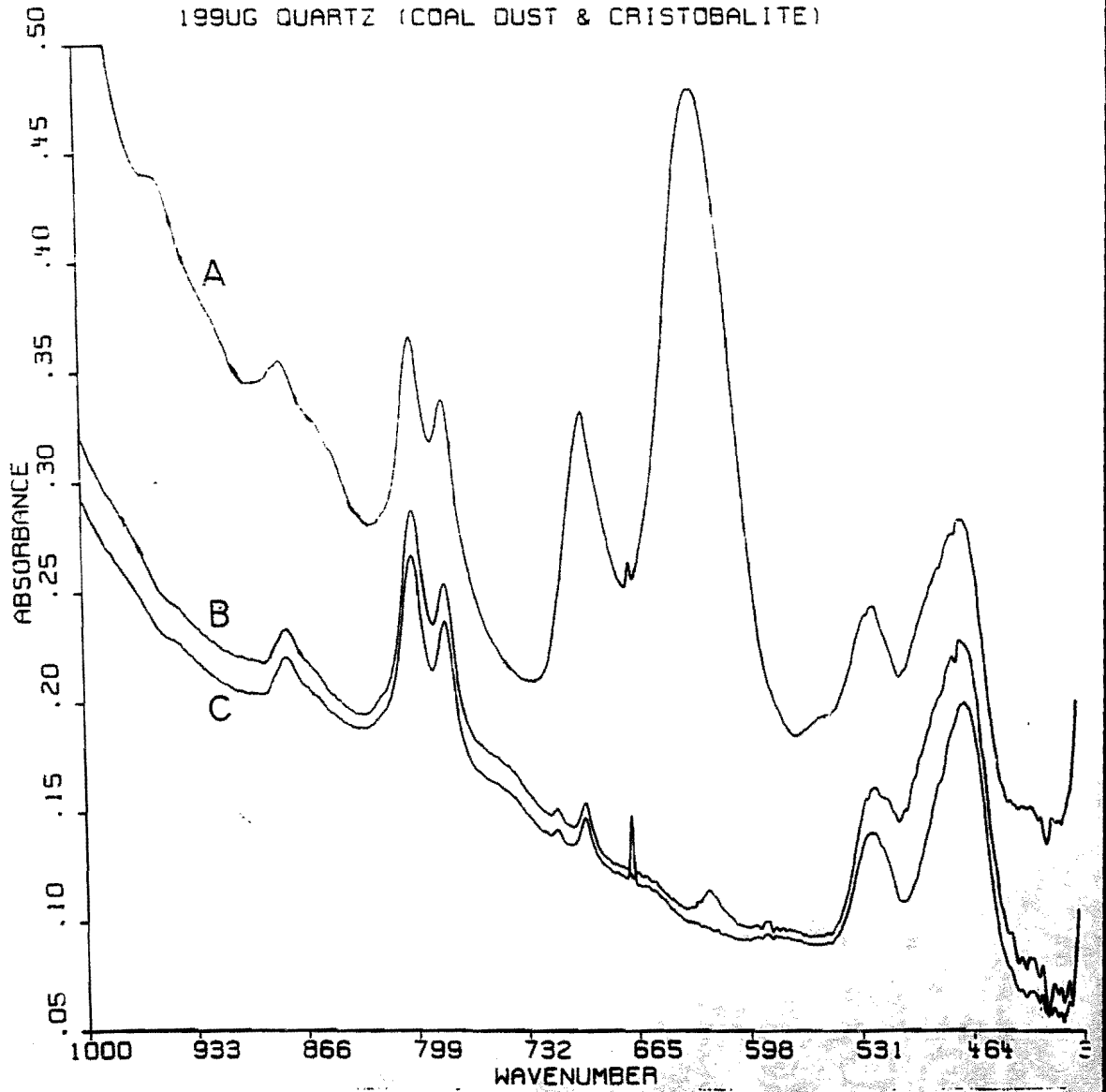
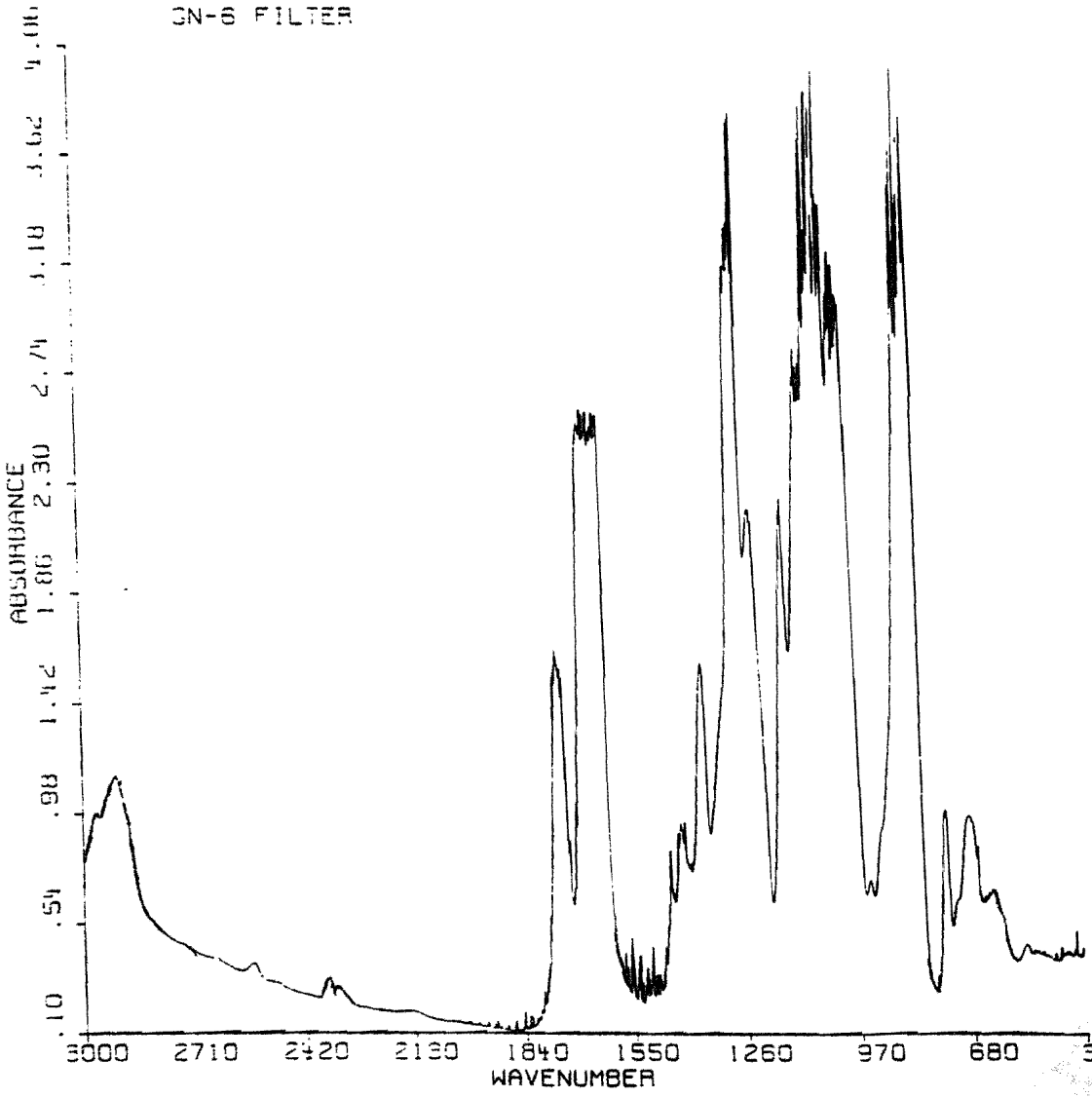

COAL DUST (805 UG)
ABSORBANCE
.237
.205
.173
.141
.109
.077
.045
.013
-.019
-.051
80.5 ug Quartz
1000
933
866
799
732
665
598
531
464
397
WAVENUMBER

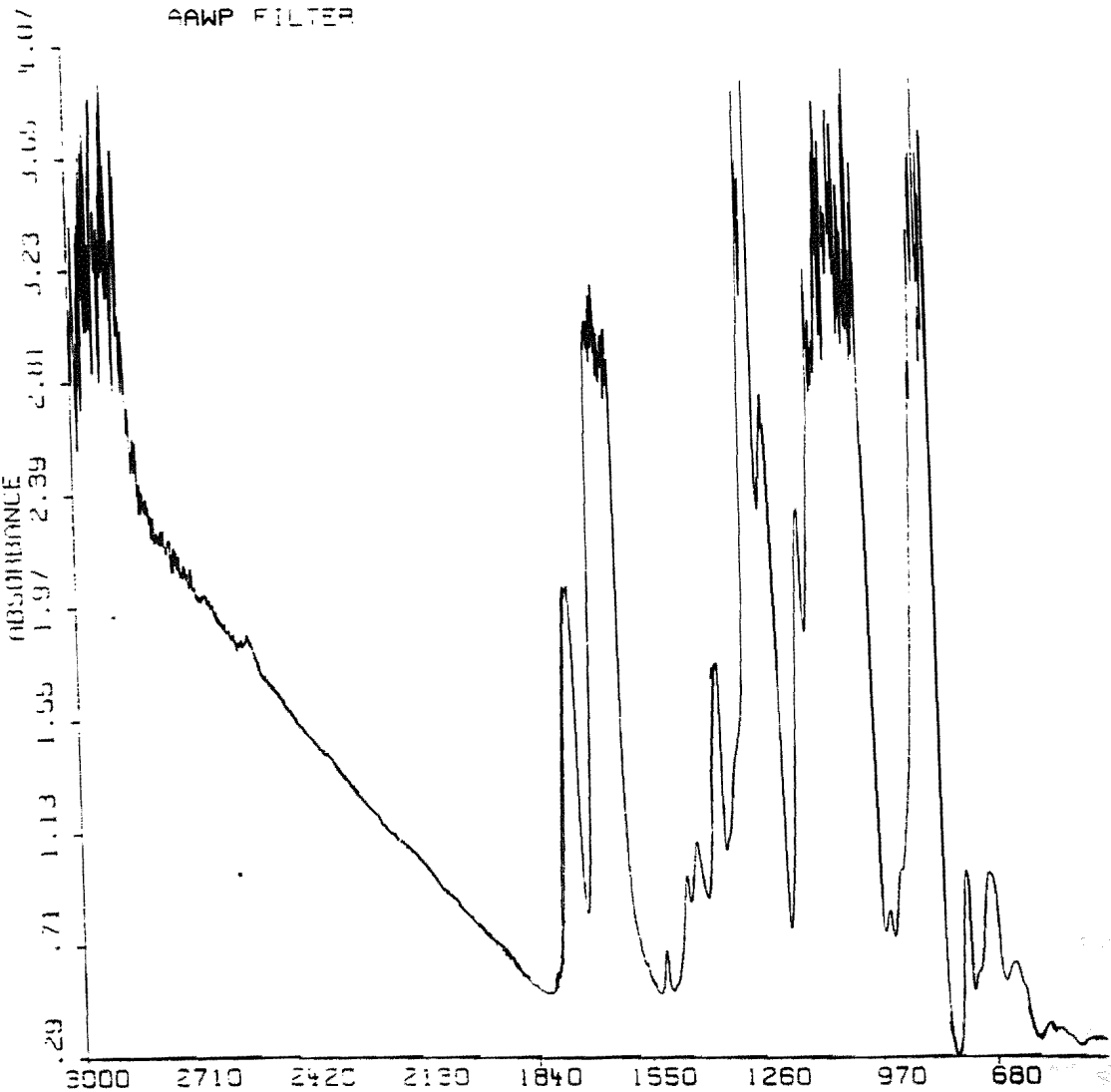
Figure 14

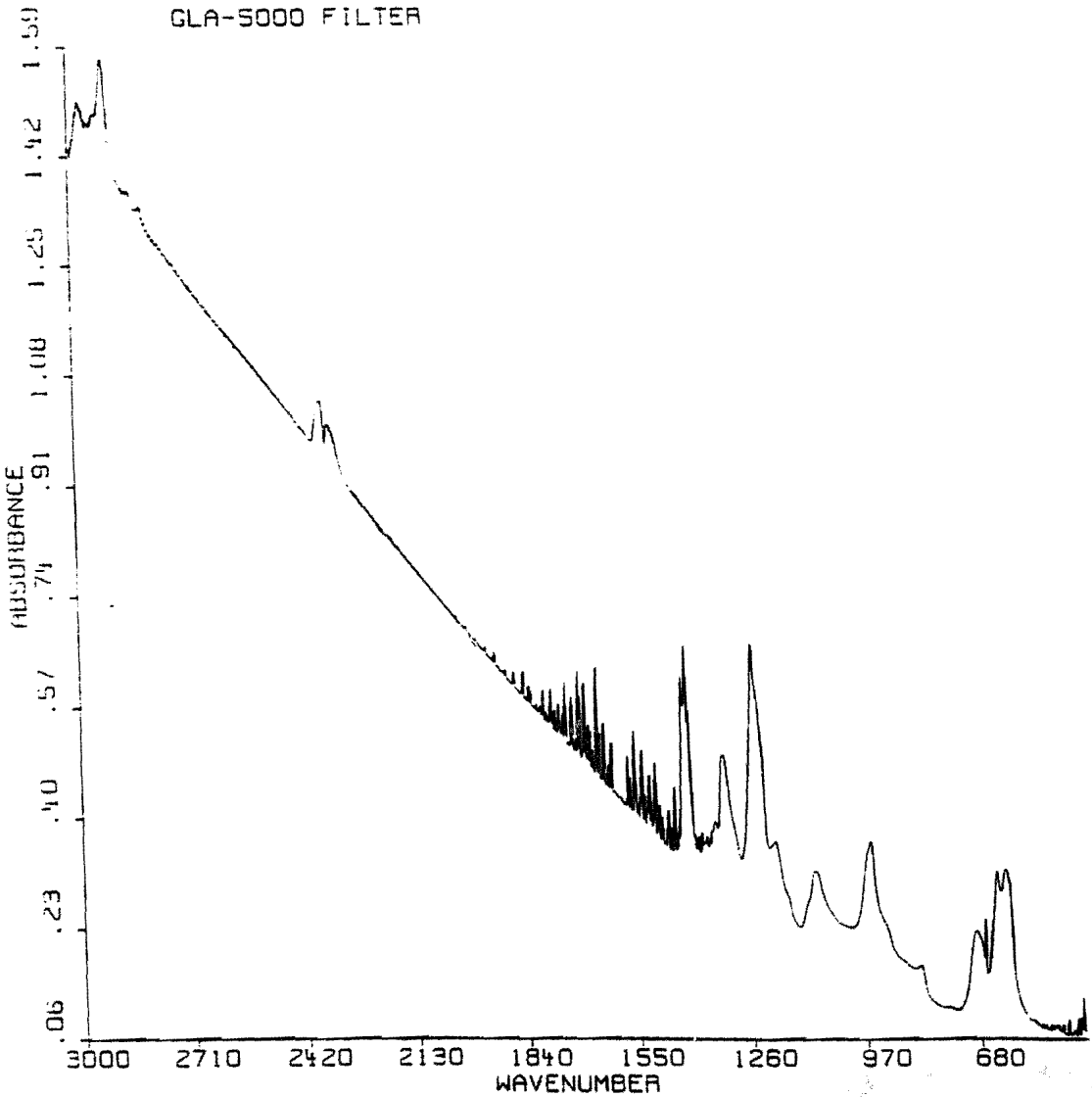

199UG QUARTZ (COAL DUST & CRISTOBALITE)
ABSORBANCE
.50
.45
.40
.35
.30
.25
.20
.15
.10
.05
A
B
C
1000
933
866
799
732
665
598
531
464
WAVENUMBER

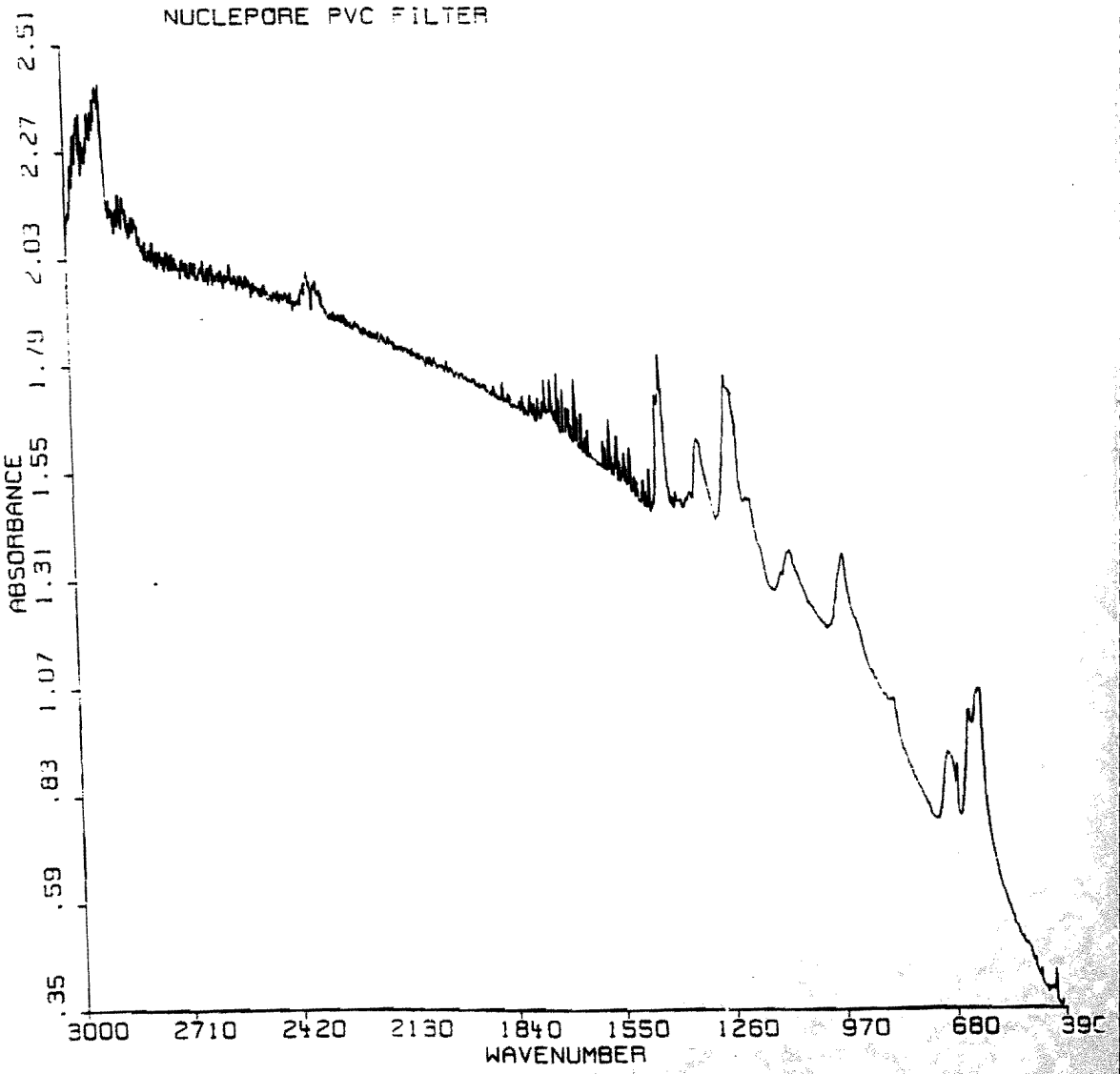
APPENDIX A

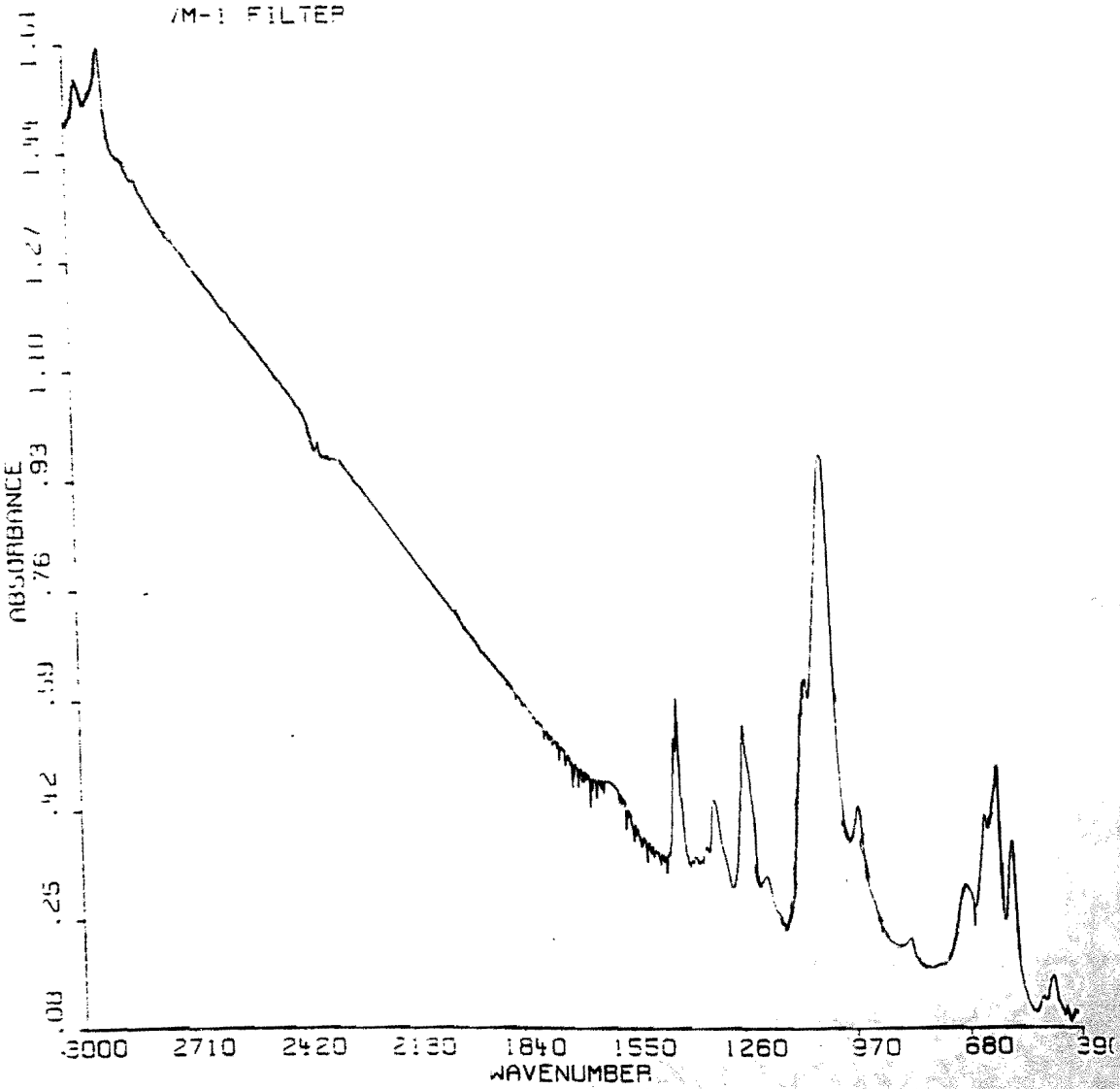
REPRESENTATIVE SPECTRA OF FILTERS

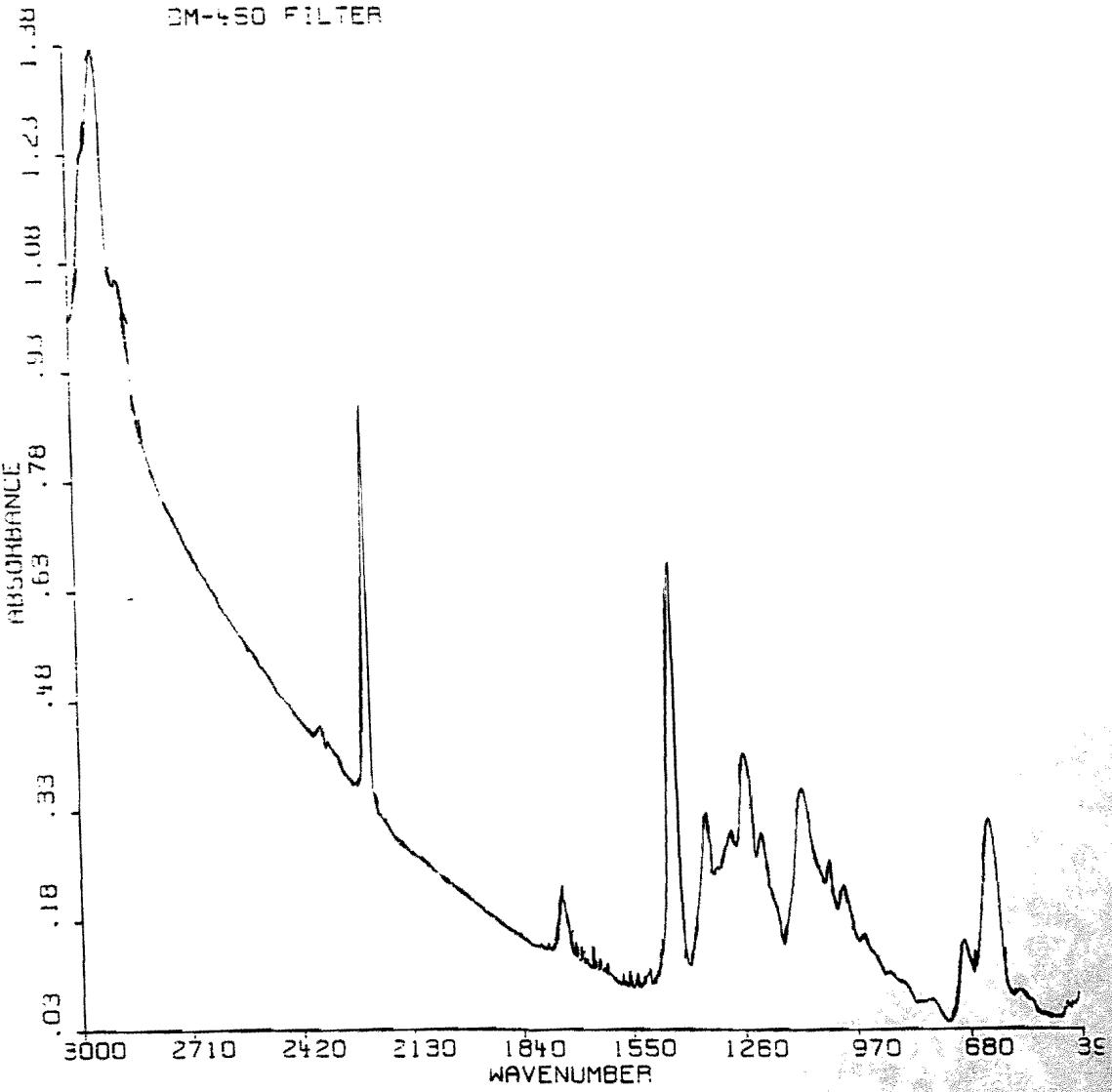

CN-6 FILTER
ABSORBANCE
.10
.54
.98
1.42
1.86
2.30
2.74
3.18
3.62
4.06
3000
2710
2420
2130
1840
1550
1260
970
680
WAVENUMBER

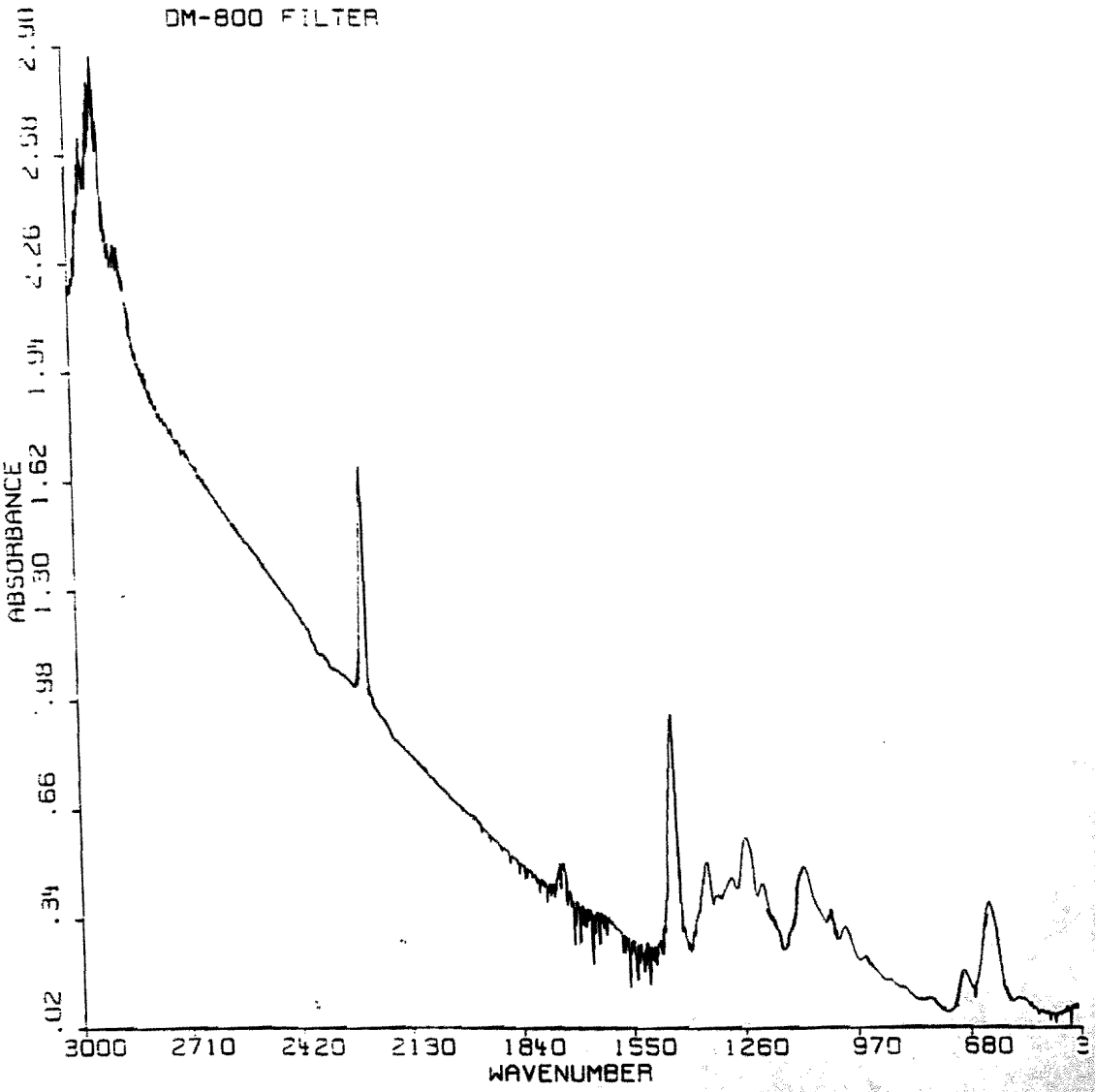

AAWP FILTER
ABSORBANCE
.29
.71
1.13
1.55
1.97
2.39
2.81
3.23
4.07
3000
2710
2420
2130
1840
1550
1260
970
680
WAVENUMBER

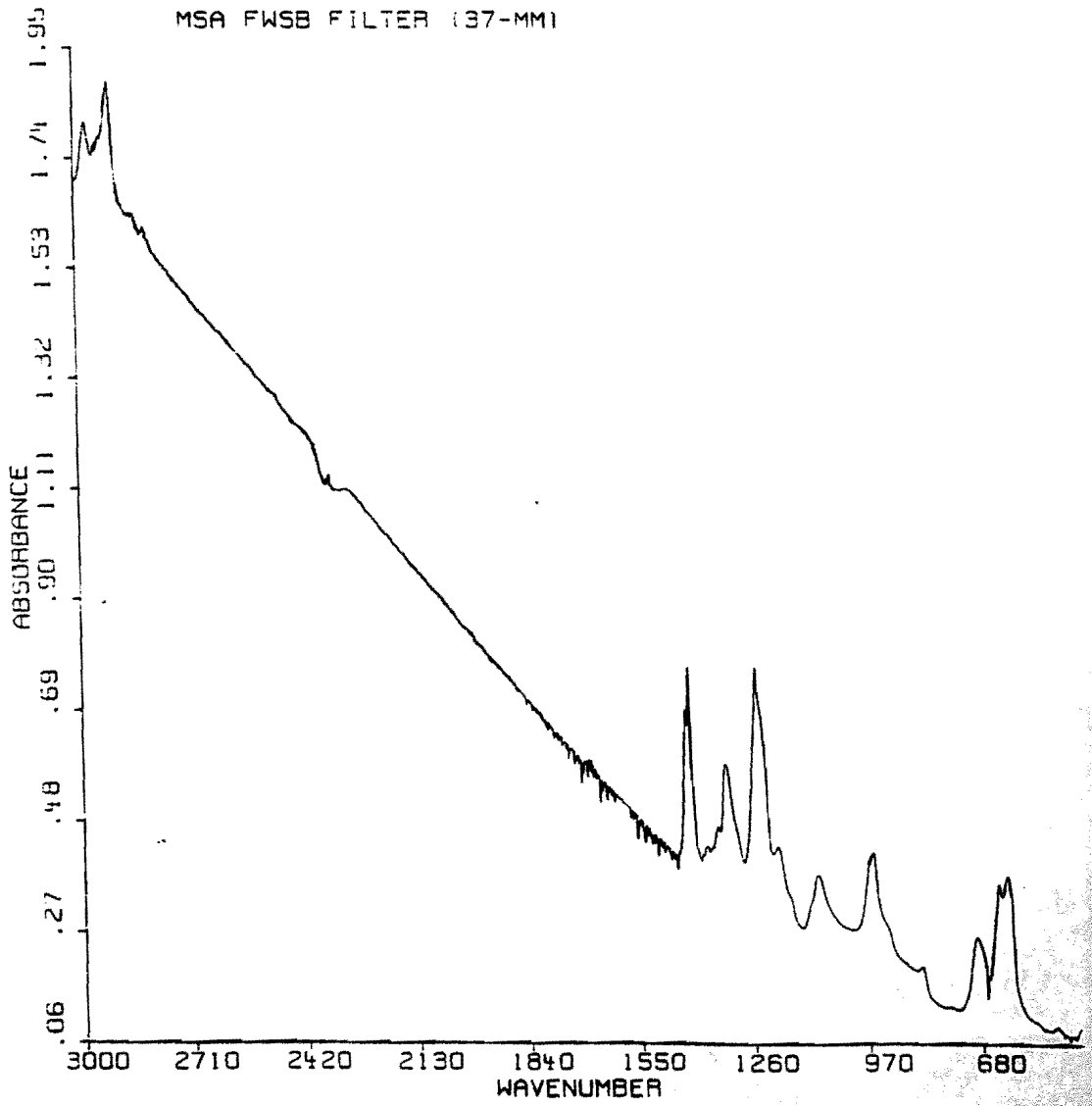

GLA-5000 FILTER
ABSORBANCE
1.59
1.42
1.25
1.08
.91
.74
.57
.40
.23
.06
3000
2710
2420
2130
1840
1550
1260
970
680
WAVENUMBER

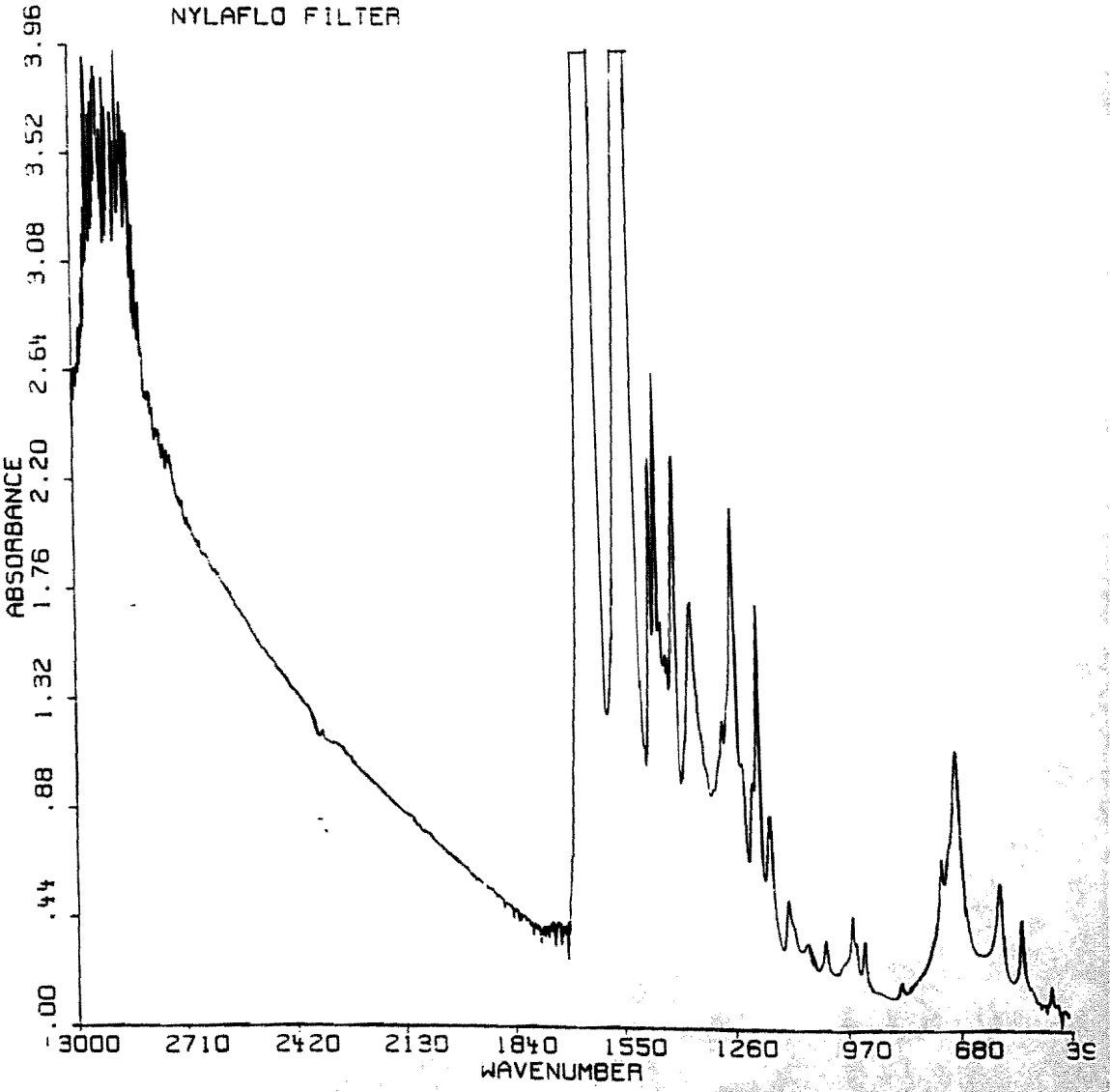

NUCLEPORE PVC FILTER
ABSORBANCE
2.51
2.27
2.03
1.79
1.55
1.31
1.07
.83
.59
.35
3000
2710
2420
2130
1840
1550
1260
970
680
390
WAVENUMBER

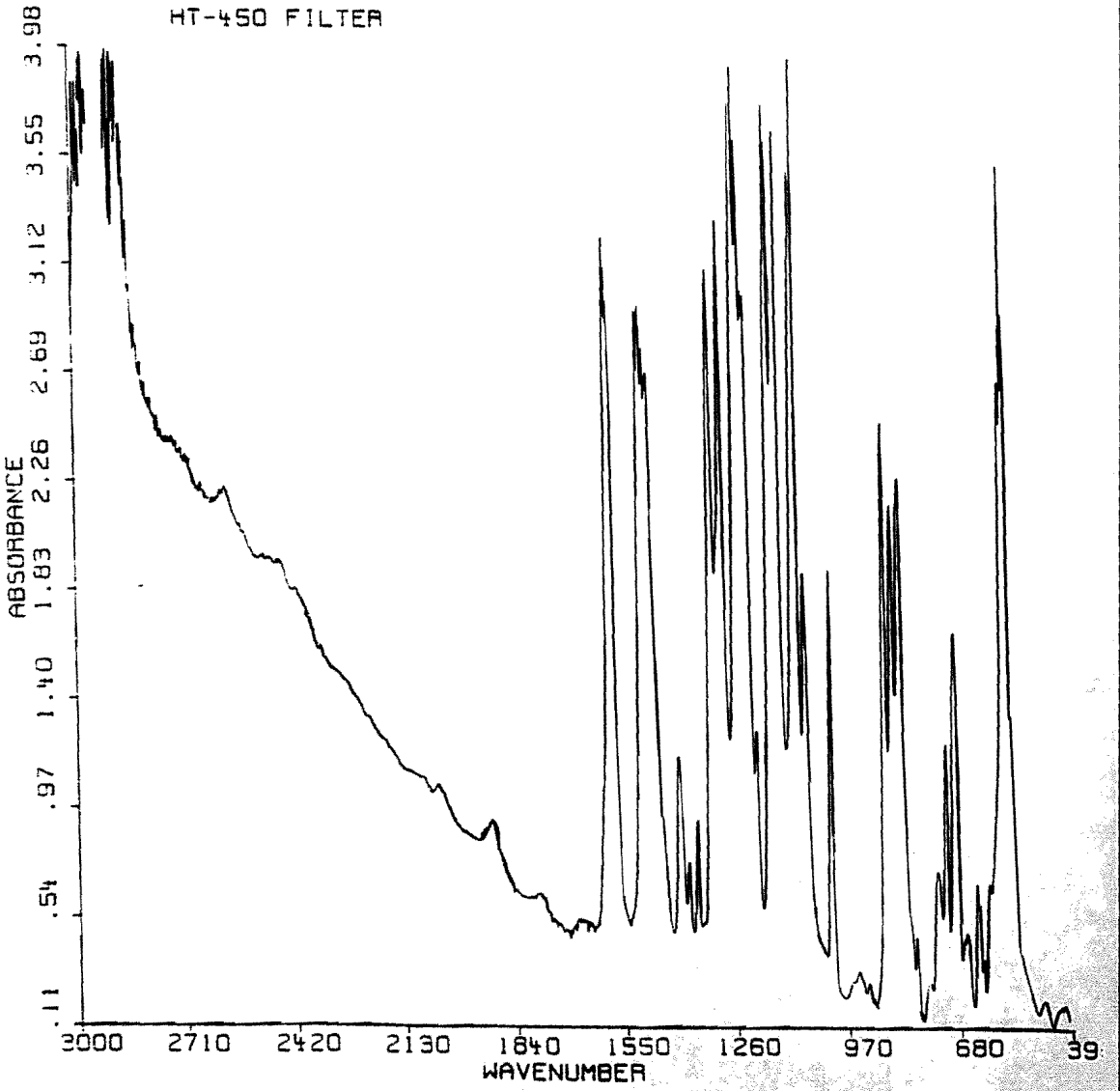

/M-1 FILTER
ABSORBANCE
.08
.25
.42
.59
.76
.93
1.10
1.27
1.44
1.61
3000
2710
2420
2130
1840
1550
1260
970
680
390
WAVENUMBER

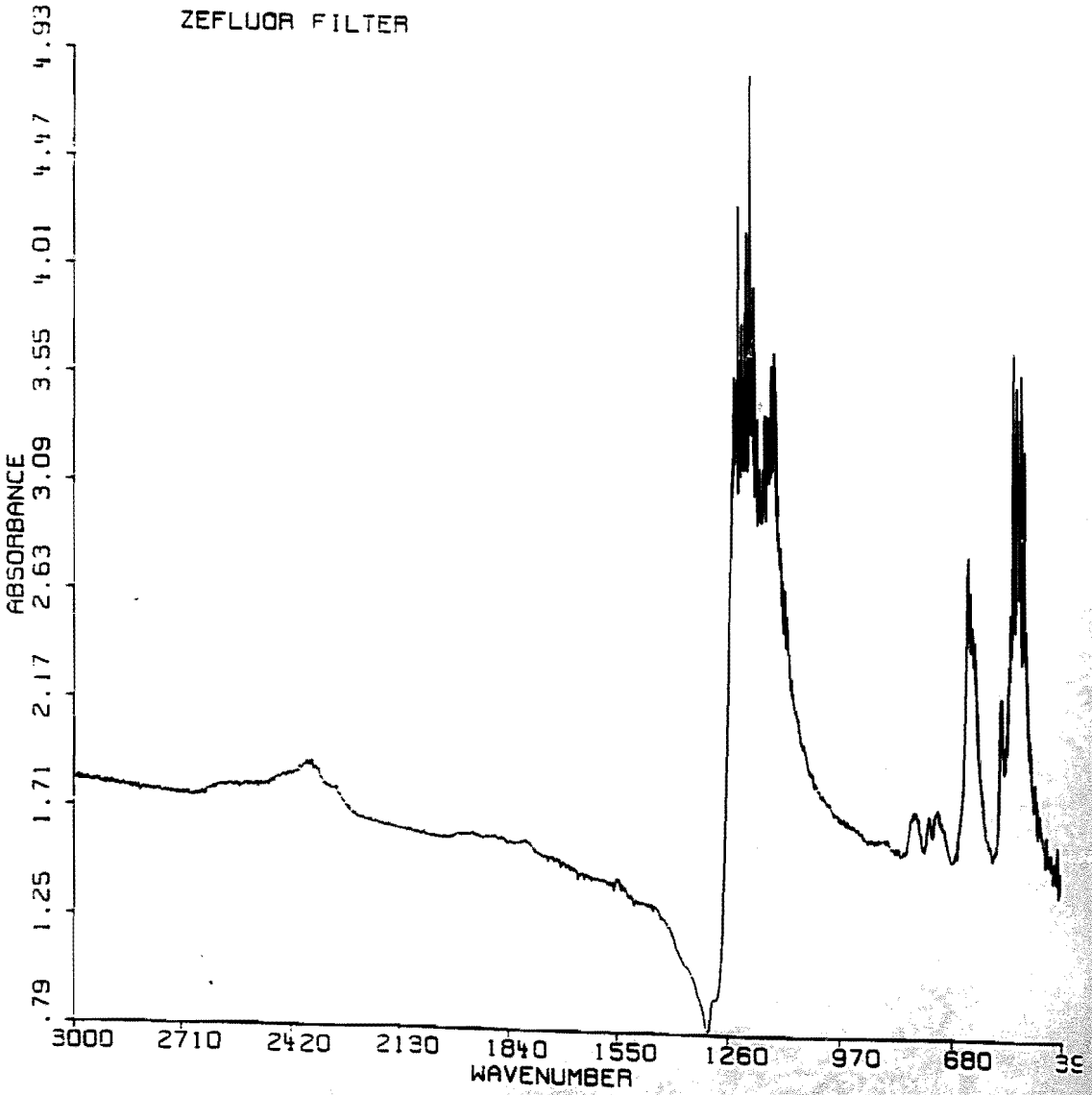

DM-450 FILTER
ABSORBANCE
.03
.18
.33
.48
.63
.78
3000
2710
2420
2130
1840
1550
1260
970
680
WAVENUMBER

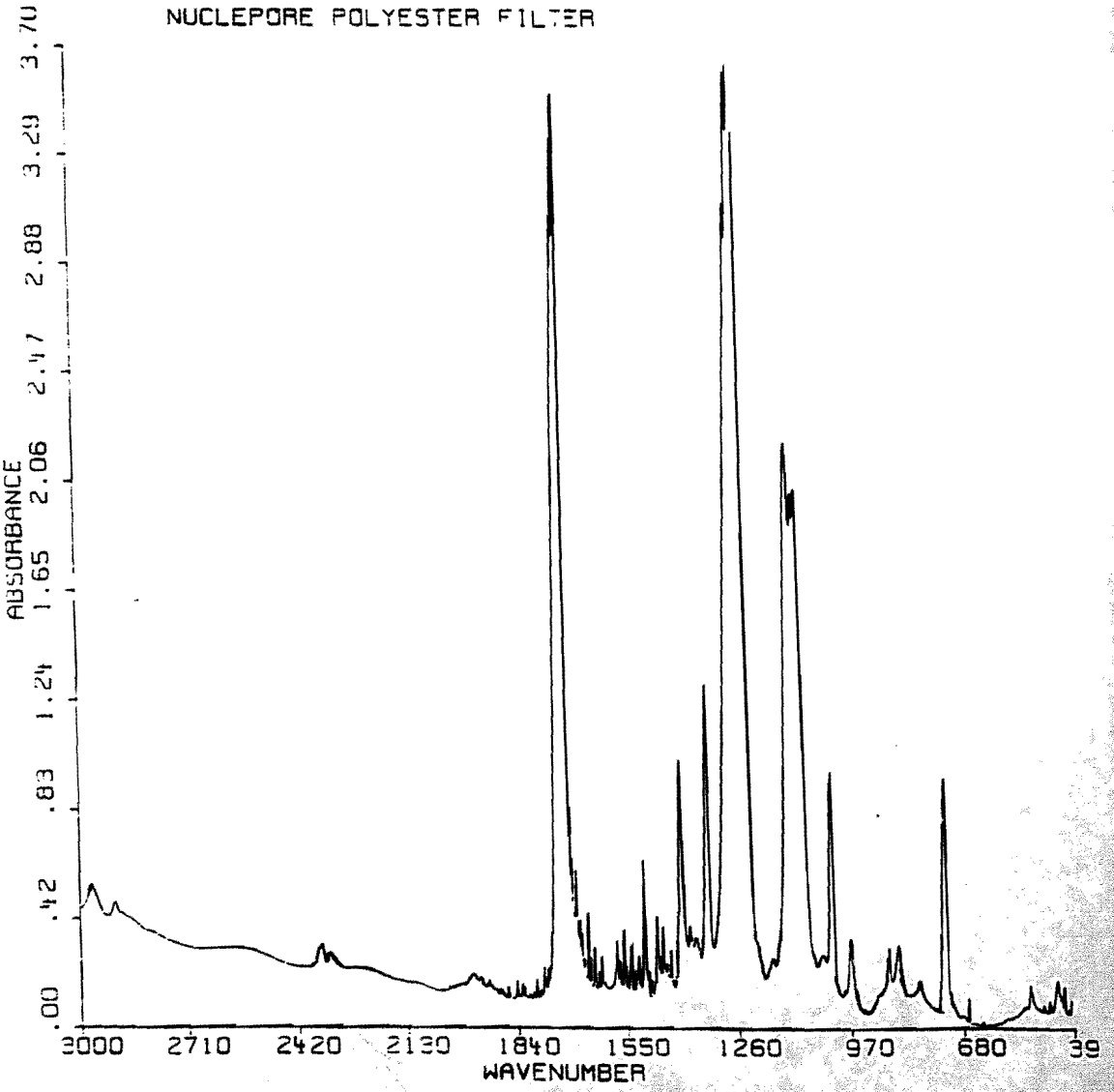

DM-800 FILTER
ABSORBANCE
.02
.34
.66
.98
1.30
1.62
1.94
2.26
2.58
2.90
3000
2710
2420
2130
1840
1550
1260
970
680
WAVENUMBER

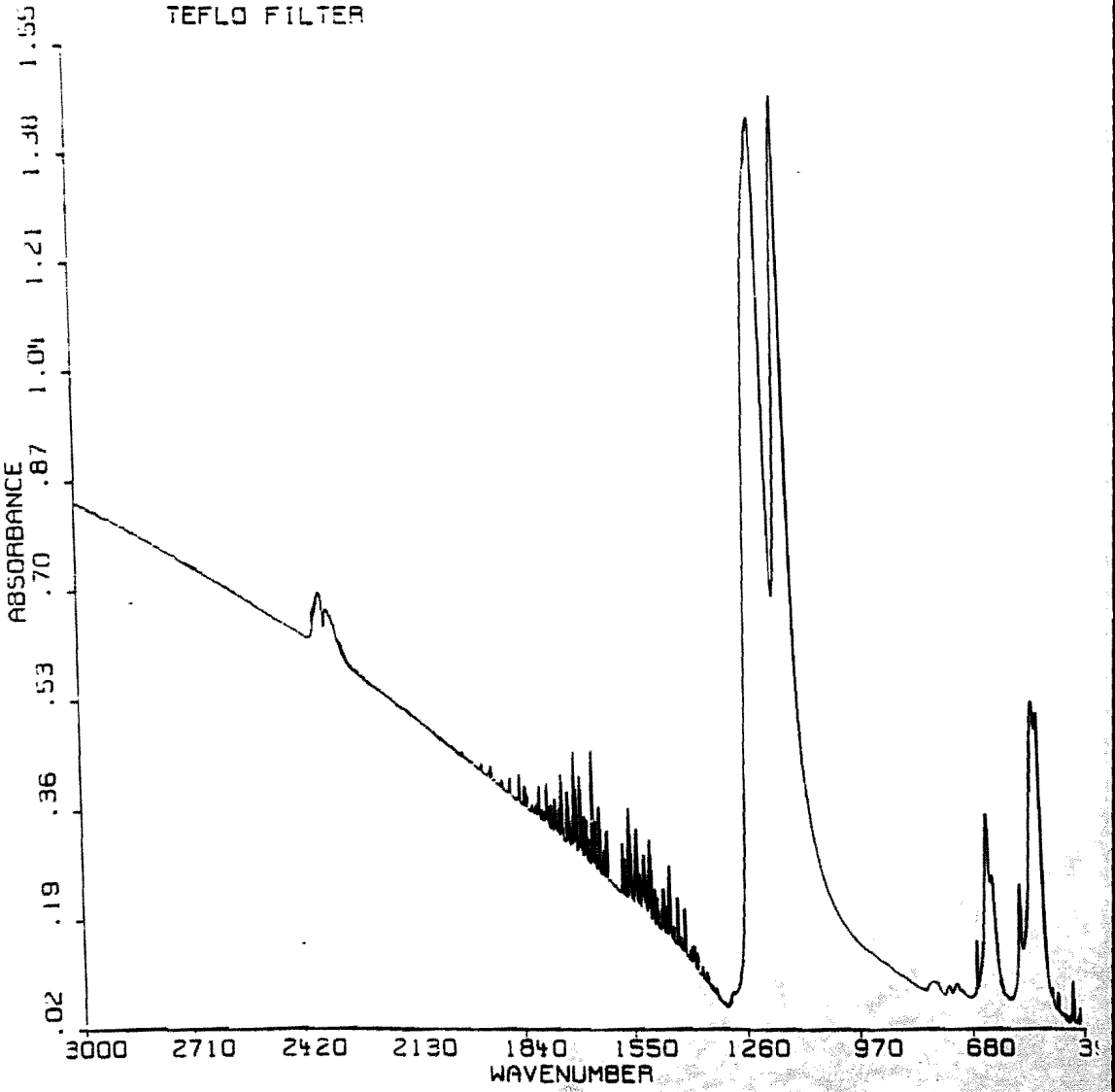

MSA FWSB FILTER (37-MM)
ABSORBANCE
1.95
1.74
1.53
1.32
1.11
.90
.69
.48
.27
.06
3000
2710
2420
2130
1840
1550
1260
970
680
WAVENUMBER

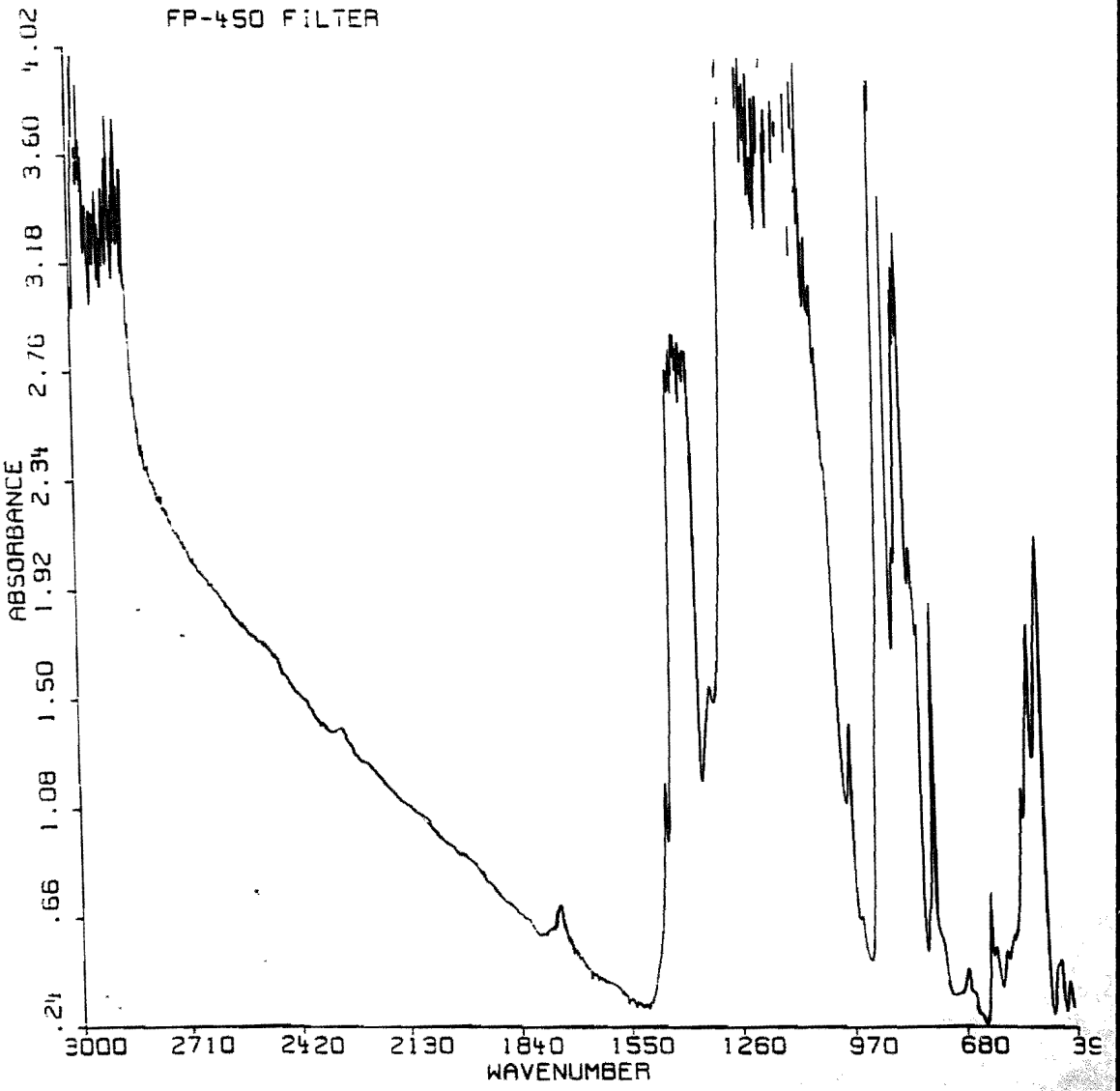

NYLAFLO FILTER
ABSORBANCE
3.96
3.52
3.08
2.64
2.20
1.76
1.32
.88
.44
.00
3000
2710
2420
2130
1840
1550
1260
970
680
WAVENUMBER

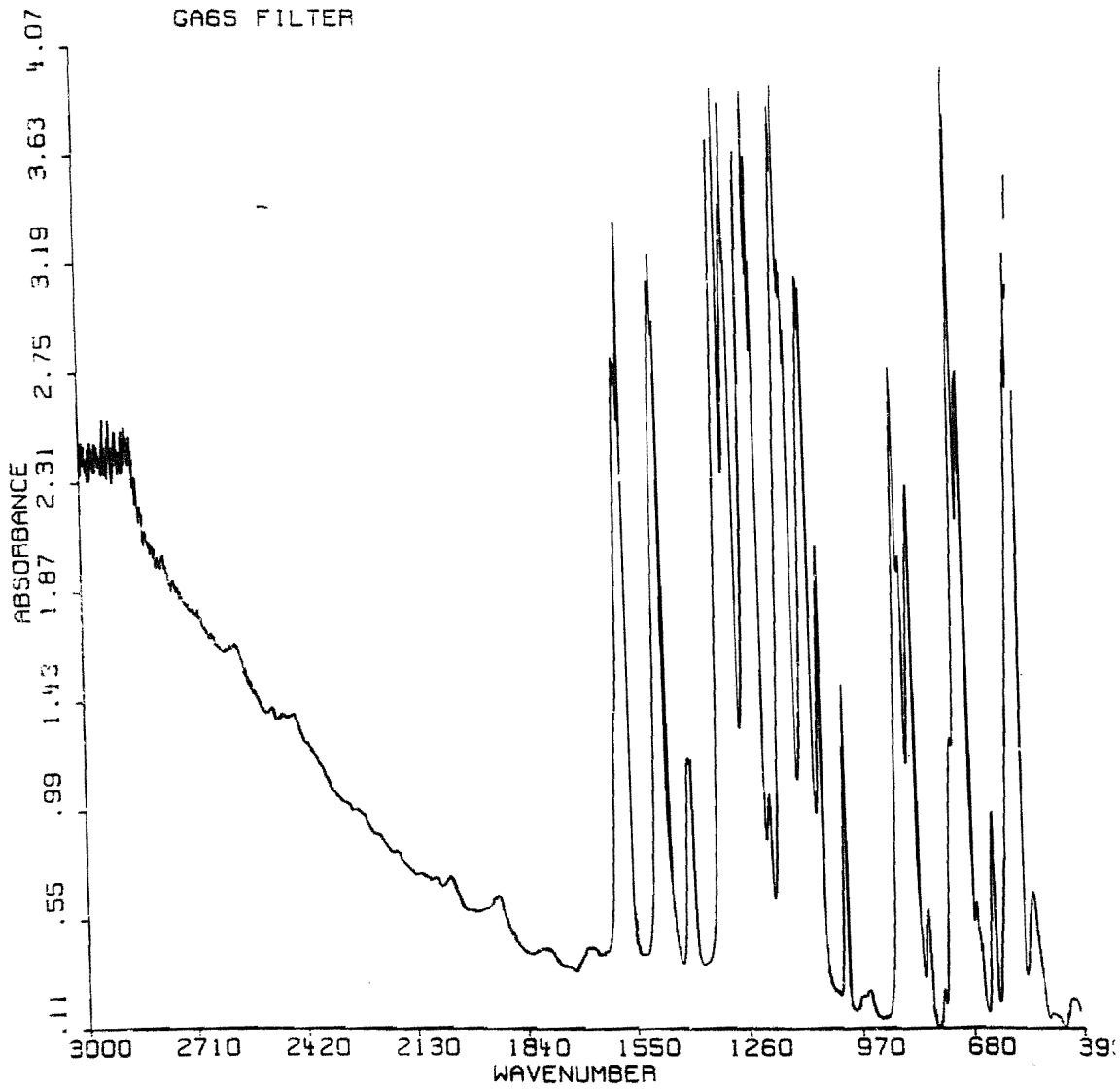

HT-450 FILTER
ABSORBANCE
3.98
3.55
3.12
2.69
2.26
1.83
1.40
.97
.54
.11
3000
2710
2420
2130
1840
1550
1260
970
680
39
WAVENUMBER


ZEFLUOR FILTER
ABSORBANCE
.79
1.25
1.71
2.17
2.63
3.09
3.55
4.01
4.47
4.93
3000
2710
2420
2130
1840
1550
1260
970
680
WAVENUMBER


NUCLEPORE POLYESTER FILTER
ABSORBANCE
.00
.42
.83
1.24
1.65
2.06
2.47
2.88
3.29
3.70
3000
2710
2420
2130
1840
1550
1260
970
680
39
WAVENUMBER


TEFLO FILTER
ABSORBANCE
1.55
1.38
1.21
1.04
.87
.70
.53
.36
.19
.02
3000
2710
2420
2130
1840
1550
1260
970
680
WAVENUMBER


FP-450 FILTER
ABSORBANCE
4.02
3.60
3.18
2.76
2.34
1.92
1.50
1.08
.66
.21
3000
2710
2420
2130
1840
1550
1260
970
680
WAVENUMBER


GASS FILTER
ABSORBANCE
.11
.55
.99
1.43
1.87
2.31
2.75
3.19
3.63
4.07
3000
2710
2420
2130
1840
1550
1260
970
680
WAVENUMBER