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Gas-phase UV spectroscopy of anthracene, xanthone, pyrene, 1-bromopyrene and 1,2,4-trichlorobenzene at elevated temperatures

Andreas Thöny, Michel J. Rossi *

Institute of Environmental Sciences/Laboratory of Air and Soil Pollution Studies (IGE/LPAS), Swiss Federal Institute of Technology-Lausanne (EPFL), CH-1015 Lausanne, Switzerland

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Abstract

We present quantitative UV-absorption spectra in the wavelength range 200 to 400 nm and the temperature range 150 to 600 °C for anthracene, xanthone, pyrene, 1-bromopyrene and 1,2,4-trichlorobenzene obtained at 1 atm of air in a slowly flowing gas mixture. As a general trend, the spectra shift to the red with increasing temperature and decrease in peak intensity thus leading to a loss of the characteristic structure of the spectrum. The absorption integrated over a particular band generally stays constant or slightly decreases with increasing temperature. © 1997 Elsevier Science S.A.

Keywords: Hot UV absorption spectroscopy; Anthracene; 1-Bromopyrene; Pyrene; 1,2,4-Trichlorobenzene; Xanthone

1. Introduction

General gas-phase spectroscopy in the UV wavelength range is of fundamental interest in many applications, certain of which require quantitative knowledge of the absorption cross section or the molar absorption coefficient as well as their temperature dependence. UV absorption spectra at temperatures of up to 1800 °C are needed for example to confirm or exclude reaction channels in the thermal decomposition of ethylbenzene in shock waves [1] or to study the reactions between CH_3 and NO in order to determine the reaction rate constant for the OH forming channel [2]. Other recent applications require the knowledge of the absorption coefficient at elevated temperatures of polycyclic and substituted aromatic hydrocarbons for predictive purposes or to interpret data. Examples for the need to know are the studies on UV photon-assisted thermal destruction of toxic organic compounds as reported by Graham et al. [3] and by Thöny et al. [4] and the design and evaluation of a laser-induced fluorescence/continuous emission monitoring system for dioxins and furans [5]. The principal aim of our investigations is to improve the efficiency of the thermal oxidation of toxic waste using an alternative technique by adding UV photons from a laser or a lamp source to a thermal reactor [4,6]. Within this feasibility study, we needed spectroscopic data at elevated temperatures in order to perform quantitative theoretical predictions on photon-assisted destruction. Due to the lack of spectral data for large UV-wavelength and temperature ranges, we decided to measure the spectra of model compounds at elevated temperature in this auxiliary study. In the photon-assisted thermal destruction study we selected model organic compounds each representing a large family of compounds. Therefore, we present spectra of this rather uncommon set of different compounds in one paper.

There is a limited temperature range over which gas-phase spectroscopic measurements may be performed. The lower temperature limit is given by the vapour pressure of the usually low vapour pressure sample, the upper by the temperature at which thermal decomposition of the molecules sets in at a given gas-phase residence time. Nevertheless, to our knowledge, we are the first to present absolute absorption coefficients of the title molecules up to such elevated temperatures.

2. Experimental

In order to perform absorption measurements at elevated temperatures, we used a basic configuration using an absorbing sample between a lamp and a photodetector (Fig. 1). We use a deuterium lamp (Zeiss, H30DS) as a light source because we are basically interested in the wavelength range between 200 and 400 nm. The UV light is detected using a 1024×256 pixel CCD camera (Princeton Instruments, TE/CCD-1024EMUV/1) after wavelength separation in a small

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* Corresponding author.

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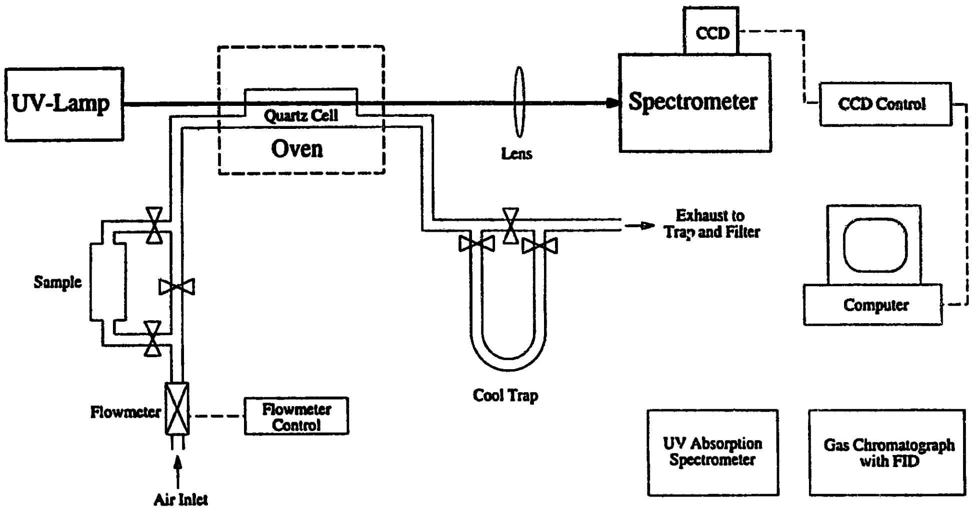

Fig. 1. Experimental setup for the measurement of UV-absorption spectra at elevated temperatures with the quartz cell placed in an oven between a deuterium lamp and a spectrometer equipped with a CCD camera.

grating spectrometer (Acton, SP150, 15 cm focal length). With its grating of 1200 grooves mm^{-1} blazed at 300 nm we are able to image a wavelength range of about 125 nm at once onto the CCD detector. In addition, we use the feature of the CCD/spectrometer software (CSMA 2.4a) to assemble adjacent spectra to one spectrum of extended wavelength range. This is achieved by collecting overlapping spectra at several grating positions obtained by turning the grating with the aid of a stepper motor to a given position. It is possible to record the wavelength range from 185 to 420 nm by just changing the grating position once. The recorded CCD data are then transferred to a personal computer using the CCD controller and are evaluated or stored onto files.

The spectral resolution of our configuration was determined using the 253.7 nm line of a low pressure Hg calibration lamp and is an order of magnitude higher than the FWHM of the narrow peak of pyrene at 322.4 nm (see below). We have estimated the accuracy of the wavelength to be ±0.25 nm and the errors of the molar absorption coefficients to be ±15%. The main contribution to errors of the latter is due to the analytical procedure in determining the gas-phase concentration.

The absorption cell is an all-quartz tube with a length of 50 cm and an internal diameter of 35 mm placed inside a tubular oven (Heraeus, Ro 4/50). It is connected to a heated gas supply system. The vapours of anthracene (Aldrich, 98+%), xanthone (Fluka, >97%) pyrene (Aldrich, 99+%), 1-bromopyrene (Aldrich, 99%) and 1,2,4-trichlorobenzene (Fluka, >99%) are entrained in an inlet stream of filtered compressed air which flows through the sample vessel at a known flow rate (Fig. 1). It contains either a solid sample coated on Raschig rings in order to increase the sample surface for faster evaporation or a liquid sample as for example 1,2,4-trichlorobenzene. Due to the generally low volatility of the investigated compounds used in this study, the sample is heated in order to increase its vapour pressure and therefore to obtain sizable optical densities. The temperature of the sample vessel is kept constant by coiled heating wires that are connected to a PID controller. For 1,2,4-trichlorobenzene, the sample vessel was cooled down using a temperature stabilized bath. The vapour concentration of these organic compounds may be estimated by the sample's vapour pressure at the temperature of the sample vessel, its rate of evaporation and the flow of compressed air. The flow rate can be set at the entrance of the gas-inlet system using a mass flowmeter to values up to 6 dm^3 per min at ambient temperature. The setting of the flow rate determines the residence time (t_R) of the molecules in the absorption cell, typically a few to tens of seconds. In order to avoid condensation of the sample vapour on any cold spot of the internal surface, we heat the transfer lines and the oven to a temperature at least 30 °C higher than the sample vessel. Therefore, we are restricted to measurements at temperatures higher than 100 °C for 1,2,4-trichlorobenzene, 200 °C for 1-bromopyrene and 150 °C for the remainder of the compounds. All the temperatures are measured using type K thermocouples. In order to avoid a concentration gradient within the absorption cell the sample gas preheated to 100 to 200 °C was admitted to the absorption cell through a 10 cm long section of the inlet system located inside the oven in order to heat the gas to oven temperature before its absorption was measured.

The dependence of the optical density $D(\lambda)$ on the wavelength was determined using three measurements, namely an intensity spectrum $I_0(\lambda)$ of the source across the cell at one atmosphere of compressed air (without sample), an intensity spectrum $I(\lambda)$ in the presence of a given vapour concentration of the absorbing species and finally a background spectrum BG of the spectrometer and CCD without light. $D(\lambda)$ is then calculated using Beer–Lambert's law allowing for proper background subtraction:

$$D(\lambda) = {}^{10}\log\left(\frac{I_0(\lambda) - BG}{I(\lambda) - BG}\right) \qquad (1)$$

From the measured $D(\lambda)$ we can easily deduce the molar absorption coefficient ϵ:

$$\epsilon(\lambda)=\frac{D(\lambda)}{cL} \qquad (2)$$

where L is the optical path length and c the gas-phase concentration of the sample measured as discussed below. The molar absorption coefficient $\epsilon(\lambda)$ was corrected for the dependence of the concentration on temperature using the law of ideal gases as the constant air–mass flow set at the mass-flow controller is measured at ambient temperature. The time between the measurement of the two spectra $I_0(\lambda)$ and $I(\lambda)$ is given by the time to operate the shut-off valves and the time of the sample to reach steady state concentration in the heated absorption cell. This total time is about one minute for a gas flow of 2 dm^3 per min that involves a sample gas flow during about 4 × the gas-residence time in the absorption cell. During that time, the lamp is expected to be stable and small signal fluctuations caused by the turbulences of heated axial transfer lines are averaged by coadding 50 spectra to an averaged spectrum $I_0(\lambda)$ or $I(\lambda)$ used in Eq. (1).

In order to measure the sample concentration c, we collect the sample during one minute in a trap cooled to about −78 °C downstream of the absorption cell. Subsequently, we dissolve the trap's content in hexane (UV quality) and determine the spectrum of that solution with a common dual beam UV spectrometer (Uvikon 860) using a 1 cm quartz cuvette. The mass of the sample may now be calculated by measuring the optical density and using liquid–phase spectrophotometric data [7,8]. We also compare these masses with the results that were taken from the same sample solution but obtained from calibrated chromatograms measured using a GC (Varian 3400) equipped with a FID detector.

This method of sample collection works well up to an oven temperature at which pure thermal destruction of the sample begins, usually in the range between 550 to 650 °C. For higher temperatures, we observe progressive destruction of the sample by thermal decomposition. The spectra taken in the gas-phase absorption cell as well as in the quartz cuvette of the UV spectrometer are superpositions of the compound's spectrum with the spectra of its decomposition products.

We also considered to determine the concentration in the absorption cell by measuring the temperature of the sample vessel followed by a calculation of the vapour pressure. But this was abandoned as it is not straightforward to measure the temperature in the sample cell of our flow experiment. Furthermore, no exact vapour pressure data were available at these rather low temperatures. We therefore decided to undertake absolute measurements of the mass of the vapour phase throughout the cell by integrating the mass flow of the vapour at the exit of the absorption cell.

3. Results and discussion

In this chapter we present the quantitative absorption spectra of anthracene, xanthone, pyrene, 1-bromopyrene and 1,2,4-trichlorobenzene as a function of temperature in the range from 100 to 250 °C as a lower limit to 550 to 650 °C, the temperature at which thermal decomposition sets in.

For anthracene and the pyrenes we use the Platt nomenclature [9] to describe the type of absorption bands.

We have selected the sample concentration in the absorption cell so as to obtain a maximum optical density of approximately 1.1. If there was an additional band of interest having a peak optical density of an order of magnitude lower, we performed a second series of measurements at a correspondingly higher sample concentration as shown for anthracene and 1,2,4-trichlorobenzene below.

With increasing temperature, we observe the progressive disappearance of the structure of the absorption bands and a redshift in the absorption spectrum whose extent however may be different for different bands of the same molecule. The absorption peaks are in general getting weaker but broaden towards the red with increasing temperature. The values of the absorption coefficients and the corresponding wavelengths of some selected absorption maxima are shown in Table 1 for the five model compounds at selected temperatures. The absorption coefficients are only listed if a relative maximum could be determined and identified. An example for which this was not the case was 1,2,4-trichlorobenzene at 600 °C where no such maximum could be determined (see below).

The absorption spectrum of anthracene at four temperatures between 150 to 550 °C is shown in Fig. 2 for the ^1B_b and in Fig. 3 for the much weaker ^1L_a band, respectively. The spectrum is a superposition of two measurements performed at 3.0 μM and 105 nM measured relative to ambient temperature. As an exception to the usually observed intensity decrease with increasing temperature, we note that the absorption spectrum of the ^1L_a band at 550 °C is slightly more intense than the spectrum at 450 °C (Fig. 3). This effect can not be attributed to thermal decomposition at our experimental conditions. It may be due to the redshift of the neighbouring ^1B_b band which contributes intensity to the ^1L_a band.

If we consider the integrated molar absorption coefficient over the ^1B_b band we estimate that this value is approximately constant or even slightly decreasing with increasing temperature as can be verified in Fig. 2.

A first comparison of the two bands measured in this work at 150 °C may be made with the gas-phase spectra of Ferguson et al. [10] recorded at 40 °C. The peak wavelengths and the shape of the spectra are in very good agreement. However, we determine absorption coefficients that are about a factor of 2 higher for the ^1B_b band and about a factor of 7 higher for the ^1L_a band. However, if we take into account the correction factors for the oscillator strengths proposed by Lyons and Morris [11] for the ^1B_b band it follows that the absorption coefficient for this band is still lower by 20% compared to the result of this work. A further comparison of the ^1B_b absorption band at 150 °C of Fig. 2 and the vapour spectrum published by Lyons and Morris [11] may be made. The shape and the wavelengths are in very good agreement, except that

Table 1
Dependence of the peak wavelengths and corresponding molar absorption coefficients on temperature for some important absorption features of five selected compounds

Compound	Temperature (°C)	λ (nm)	ϵ^a	λ (nm)	ϵ^a	λ (nm)	ϵ^a	λ (nm)	ϵ^a	λ (nm)	ϵ^a
Anthracene	150	362.4	9369	344.7	9487	328.6	6400	236.7	157 175		
	300	363.6	4594	345.4	5698	329.4	4329	237.1	146 014		
	450	364.7	3420	346.3	4709	330.5	3847	237.6	128 148		
	550	364.9	3594	346.7	5051	331.8	4249	238.3	115 491		
Xanthone	150	326.4	9334	223.8	36 957						
	300	327.0	6764	224.4	40 228						
	450	327.3	5925	224.9	40 153						
	600	327.3	5385	226.2	38 904						
Pyrene	150	322.4	53 889	308.6	27 142	265.2	33 791	255.1	19 120	231.9	74 834
	300	323.6	38 332	309.7	23 217	266.1	29 503	255.9	18 747	232.7	71 026
	450	323.9	35 776	310.0	22 871	266.3	29 926	256.3	20 187	232.9	70 694
	600	325.7	24 065	312.9	18 160	267.8	24 058	259.0	18 880	234.3	58 467
1-Bromopyrene	250	332.2	33 643	317.3	21 181	270.3	24 546	260.0	15 599	235.8	46 349
	350	333.0	29 725	318.2	20 819	270.9	23 272	260.8	16 024	236.3	44 150
	500	334.1	22 830	319.7	16 916	271.9	18 544	262.1	13 701	237.0	39 256
	650	335.3	21 149	321.9	16 915	272.7	18 389			237.4	40 345
1,2,4-Trichlorobenzene	100	285.6	621	276.9	633						
	300	286.4	498	278.7	528						
	450	286.4	487	280.0	504						

[a] In (dm^3 mol^{-1} cm^{-1}).

our spectra present less structure, most probably because we performed our measurements at a higher temperature. Nevertheless, the molar absorption coefficient for anthracene measured in this work is a factor of 1.43 higher than the data published by Lyons and Morris. Another comparison of our spectra may be made with the gas-phase spectra of the ^1L_a band at two different temperatures published by Härdtl and Scharmann [12]. This comparison gives a similar dependence of the band shapes on temperature as our spectrum. Finally, our spectra of the ^1B_b band at 150 and 300 °C may be compared with the spectra presented in a review by Dygdala and Stefanski [13]. We find a generally good agreement with all the reviewed spectra. The position of the peak maxima measured in this work correspond better to the ones obtained by Dygdala and Stefanski rather than to the data of two other authors that they reviewed, namely of Härdtl and Scharmann [12] and of Ware and Cunningham [14]. The spectral shape for wavelengths longer than approximately

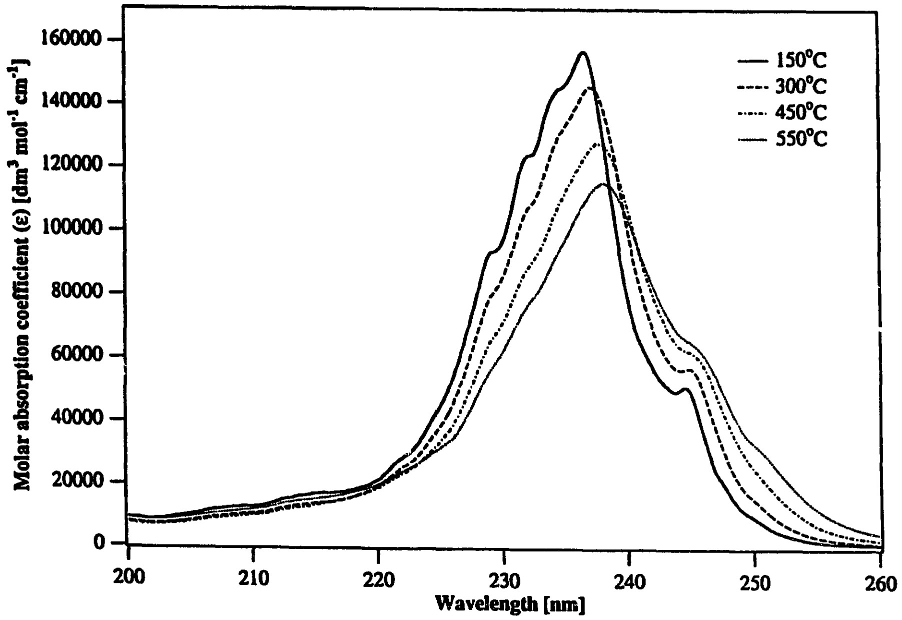

Fig. 2. Dependence of the absorption spectrum as a function of temperature of anthracene (^1B_b band). ——— 150 °C; - - - 300 °C; – · – 450 °C; · · · 550 °C.

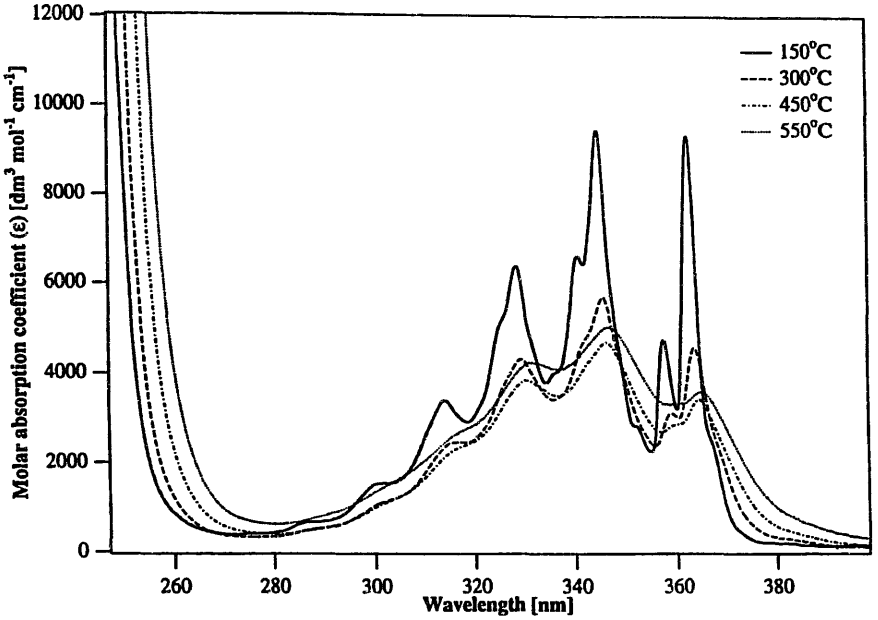

Fig. 3. Dependence of the absorption spectrum as a function of temperature of anthracene (^1L_a band). ——— 150 °C; - - - 300 °C; – · · – 450 °C; · · · 550 °C.

335 nm from our work is also in good agreement with Dygdala and Stefanski's spectrum, but our absorption coefficients are slightly higher. For wavelengths smaller than approximately 335 nm our measurements agree well with the data of other authors [12,14] presented in that review. Nevertheless, we have to mention that in the spectrum of Dygdala and Stefanski [13] at wavelengths smaller than 335 nm the values of the absorption coefficient become increasingly smaller with decreasing wavelengths. Moreover, a small additional peak appears in that wavelength region that seems extraneous to anthracene.

Fig. 4 shows the absorption spectrum of xanthone at four temperatures in the range of 150 to 600 °C recorded at a concentration of 1.09 μM measured relative to ambient temperature. We observe again a disappearance of the structure, a significant redshift and a general decrease of absorption intensity. The absorption intensity of the ($\pi\pi^*$) band at 223 nm is increasing slightly which is most probably due to the redshift of another band located below 200 nm. For xanthone there does not seem to be any previous gas-phase spectroscopic data. Yet, it is well known from the literature that the absorption bands of the gas-phase spectrum are similar but shifted by several nm towards shorter wavelength when comparing the gas-phase absorption spectra with spectra in solution [15,16]. The amount of the shift depends on the compound, the absorption band and the solvent used. The comparison of our gas-phase spectrum with a solution spectrum [7] shows good agreement.

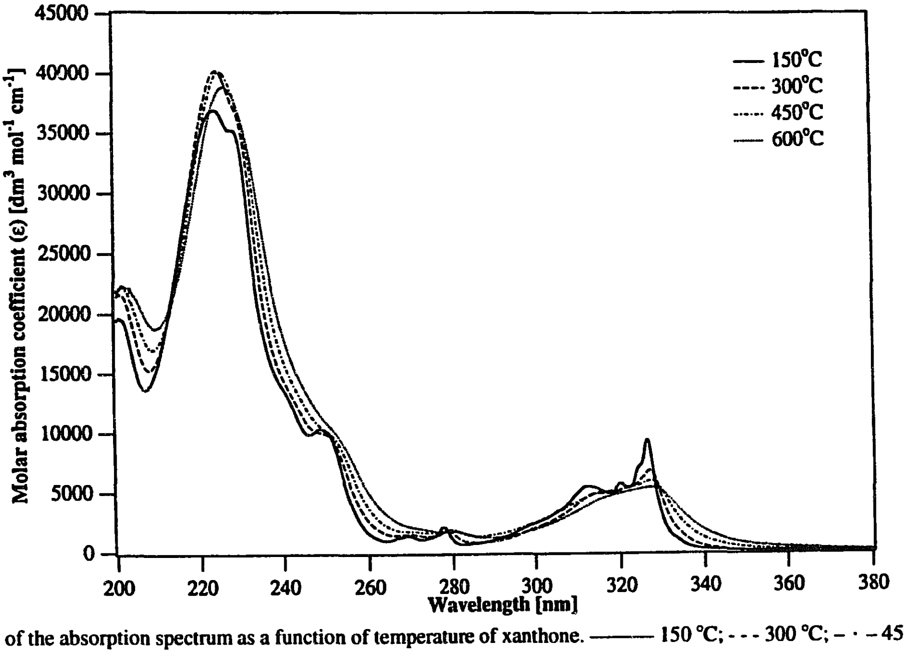

Fig. 4. Dependence of the absorption spectrum as a function of temperature of xanthone. ——— 150 °C; - - - 300 °C; – · · – 450 °C; · · · 600 °C.

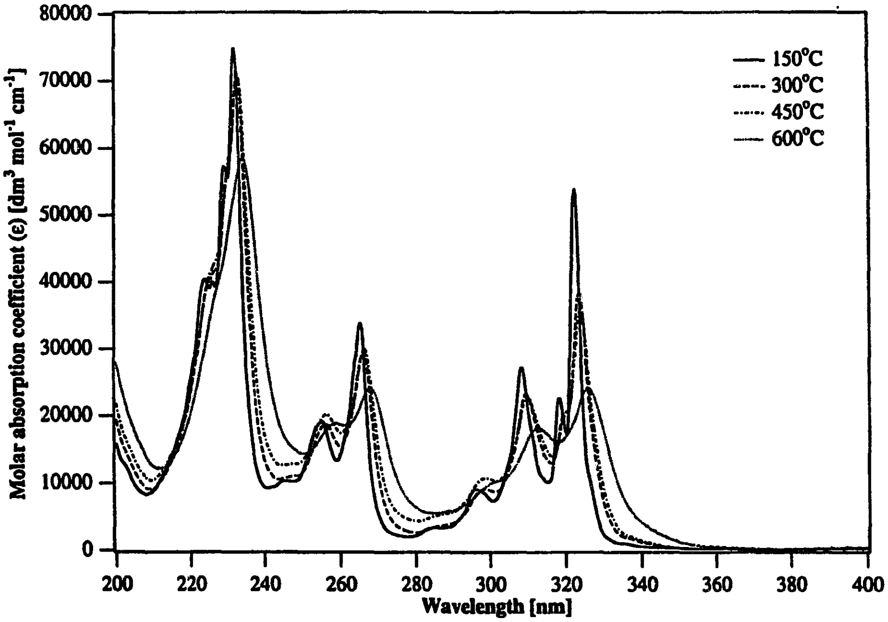

Fig. 5. Dependence of the absorption spectrum as a function of temperature of pyrene. ——— 150 °C; - - - 300 °C; – · – 450 °C; · · · 600 °C.

In Figs. 5 and 6, we present the absorption spectrum of pyrene and 1-bromopyrene, respectively. Pyrene was recorded at a concentration of 432 nM and 1-bromopyrene at 736 nM measured relative to ambient temperature. They show a very similar and well structured spectrum having a strong absorption over a large wavelength range. The heavy Br atom causes a shift of the spectrum of 1-bromopyrene of a few nm towards longer wavelengths compared to normal pyrene as shown in Baliah and Pillay [17] for solution spectra. The absorption spectrum of 1-bromopyrene is slightly less intense than the one for pyrene. The absorption at the peaks decrease with increasing temperature while the absorption between the peaks and bands increases. The latter may be due to the redshift of neighbouring peaks or bands at shorter wavelengths.

The positions of the peak maxima of our pyrene measurements are in good agreement with the data on the ^1L_a and ^1B_b bands from Ferguson et al. [10], but we note that our values for the absorption coefficients are a factor of 2 higher than theirs. However, if we apply a similar correction factor on the published data as for anthracene the agreement is improving significantly and the deviations are smaller than 25%.

In the case of 1-bromopyrene, we observed at the given concentration a nearly constant new absorption with a broad peak near 360 nm in the wavelength range between 200 and 560 nm at low temperatures. It most probably extends further

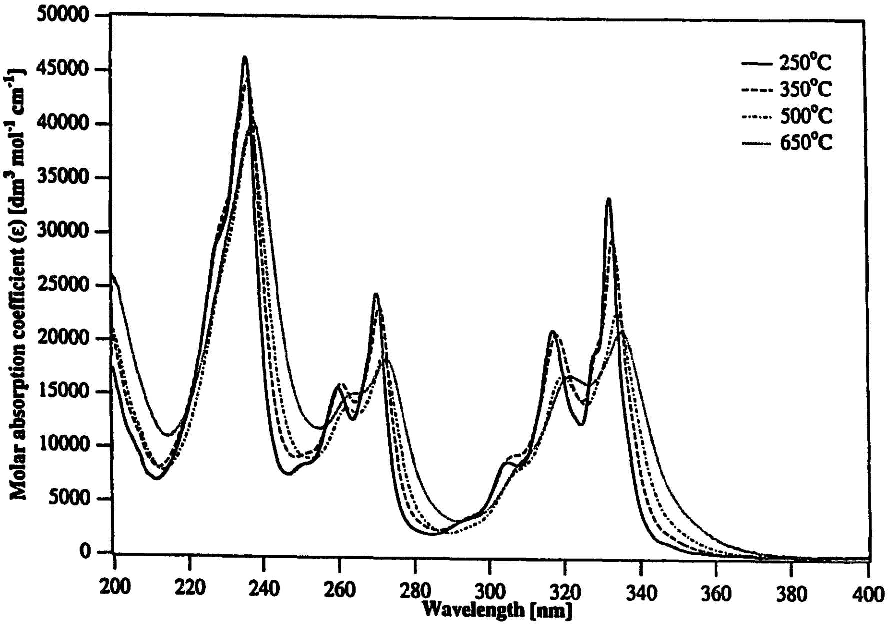

Fig. 6. Dependence of the absorption spectrum as a function of temperature of 1-bromopyrene. ——— 250 °C; - - - 350 °C; – · – 500 °C; · · · 650 °C.

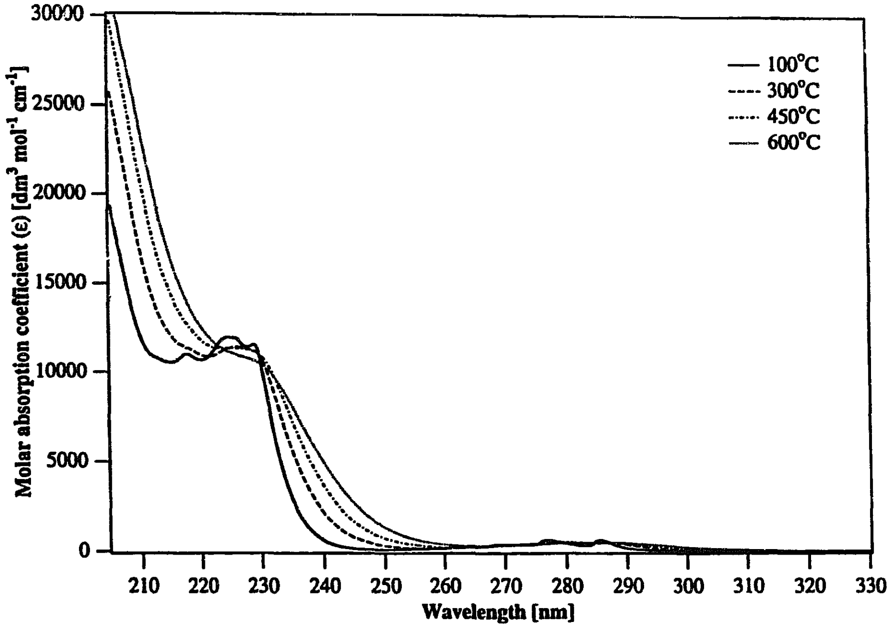

Fig. 7. Dependence of the absorption spectrum as a function of temperature of 1,2,4-trichlorobenzene. ——— 100 °C; - - - 300 °C; – · – 450 °C; · · · 600 °C.

towards longer wavelengths. This absorption decreases with increasing temperature and vanishes at temperatures above 175 °C. We attribute this broad and structureless absorption to dimers having a finite stability at low temperatures.

The spectrum of 1,2,4-trichlorobenzene has a strong absorption in the short UV wavelength range with a peak well below 200 nm and a weak band centred at 280 nm as shown in Figs. 7 and 8. The spectrum is a superposition of spectra recorded at a concentration of 20.9 and 1.21 μM measured relative to ambient temperature. The liquid-phase reference spectrum for this compound has been taken from the work of Conrad-Billroth [8]. A large redshift with increasing temperature is easily discovered in Fig. 8 where we observe for example an increase of the absorption coefficient at 248 nm by a factor of 20 at 600 °C compared to 100 °C.

The wavelengths in our spectrum at 100 °C are displaced towards the red by a few tenths of nm compared to literature data [15,18]. This may be explained by differences in recording temperature. The comparison of the spectral shape leads to good agreement with Scharping et al. [15], but we point out that our data are less resolved due to the higher temperature at which our spectra were taken. In addition, the value of the absorption coefficients we measure are larger by a factor of 1.55 and 4.6 at the peak of 225 and 285 nm, respec-

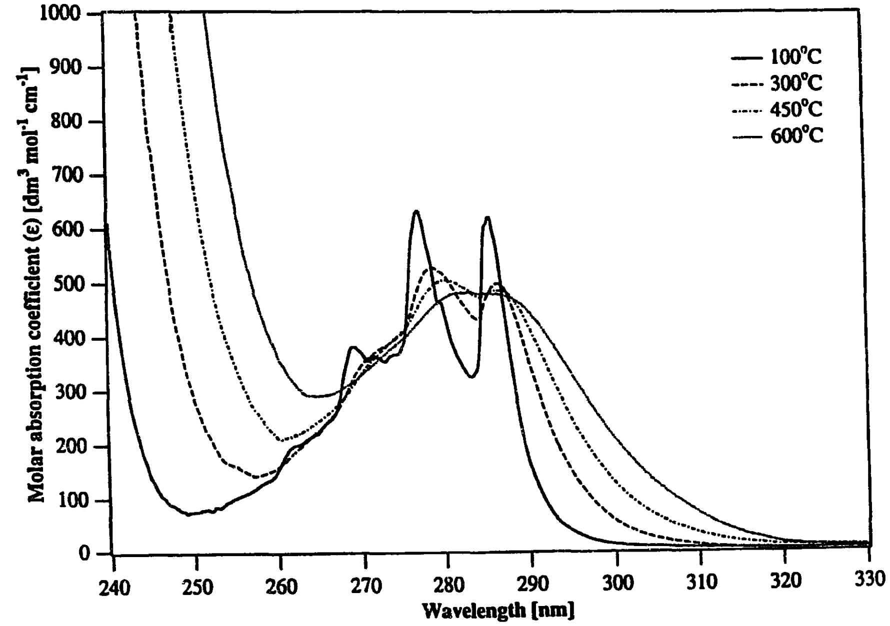

Fig. 8. Enlarged view of the dependence of the absorption spectrum as a function of temperature of 1,2,4-trichlorobenzene (weak ¹L$_b$ band). ——— 100 °C; - - - 300 °C; – · – 450 °C; · · · 600 °C.

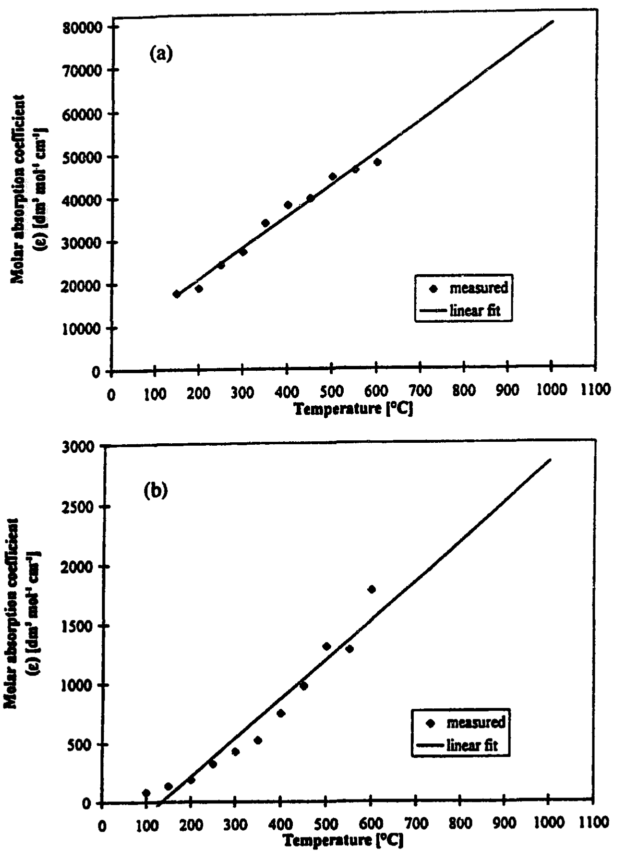

Fig. 9. Dependence of the molar absorption coefficient on temperature at 248 nm. The line represents a linear fit calculated from the measured points and extrapolated to higher temperatures for (a) anthracene and (b) 1,2,4-trichlorobenzene. ◆ measured; ——— linear fit.

tively. Our measured absorption coefficients are close to the ones obtained from the liquid-phase spectrum of the literature [8]. If we take these literature data as a reference for the peak near 225 nm we obtain good agreement with our gas phase measurements.

We summarize from our experiments that the integrated molar absorption coefficient stays roughly constant or even slightly decreases with increasing temperature for all the compounds investigated in this work. This is at variance with the results published by Berman et al. [19] in which they report on an unexpected increase of the integrated absorption intensity as the temperature increases. The increases of intensity of a band with temperature we observe in our work is slight, e.g., for anthracene and xanthone, and may be explained by the redshift of a more intense band located at shorter wavelengths.

Finally, for the purpose of predicting the photon-assisted destruction of these compounds we linearly extrapolated the absorption coefficients for some wavelengths from the measured data to the temperature range between 600 and 1000 °C using a linear regression fit. We believe that such a procedure leads to a good approximation of the true absorption coefficient as we do not expect any dramatic change of the absorption coefficient at those wavelengths and temperatures. Two examples of extrapolated data are presented in Fig. 9 for anthracene and 1,2,4-trichlorobenzene at 248 nm. The absorption coefficients for anthracene (Fig. 9a) begin to increase continuously from an already large value due to the redshift as shown in Fig. 2, while those for 1,2,4-trichlorobenzene (Fig. 9b) start at a low value to increase continuously due to the redshift shown in Fig. 7. It may also be possible to apply a parabolic fit in order to obtain predictions at higher temperatures. These calculated absorption coefficients will be used later in order to determine the quantum yields at temperatures at which photon-assisted decomposition takes place [20].

4. Conclusion

We present the temperature dependence of the UV absorption spectrum at wavelengths between 200 and 400 nm for anthracene, xanthone, pyrene, 1-bromopyrene and 1,2,4-trichlorobenzene in the temperature range from 150 to 600 °C. In general, the absorption peak is decreasing in intensity, the band is broadening and shifting to the red with increasing temperature. Therefore, the absorption coefficients at a certain wavelength may either increase or decrease with increasing temperature, depending on the wavelength and temperature interval. The integrated absorption over a band generally stays constant or slightly decreases with increasing temperature. In several cases, we observe an increase in intensity which may be attributed to the increase in optical density due to the redshift of a neighbouring band at a smaller wavelength. We discuss our results in relation to the few published gas-phase literature data that were obtained in the low temperature range compared to our measurements. We generally find good agreement with these literature spectra both in terms of peak position and intensity. Using our measured UV-spectra taken at elevated temperatures we are able to predict absorption coefficients at still higher temperatures.

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