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Absorption Spectra and Electronic Properties of Alkali Metal Doped C_{60}

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Absorption Spectra and Electronic Properties of Alkali Metal Doped C_{60}

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Abstract: Changes in optical absorption spectra of ~500 Å thick films of C_{60} during doping with alkali metals (A=Li,Na,K, and Rb) were recorded. The stoichiometry of the potassium-doped C_{60} films was monitored by simultaneous *in situ* measurements of electrical conductivity . The spectral changes indicate that the molecular solid behavior of C_{60} bulk is preserved in the A_6C_{60} phase. Assignments for the C_{60} and K_6C_{60} absorption spectra based on molecular orbitals of C_{60} are discussed.

Recent discoveries of metallic conductivity in alkali metal doped C_{60} [1] and superconductivity in K_3C_{60} [2] ($T_c = 19$ K) as well as Rb_3C_{60} [3] ($T_c = 28$ K) have increased interest in the electronic and structural properties of these materials. Upon exposure to alkali vapor the conductivity of alkali metal (A) doped C_{60} first increases, then decreases [1]. The conductivity increase has been explained in terms of electron transfer to the empty three-fold degenerate $2t_{1u}$ LUMO [4] of C_{60} leading to a maximum conductivity ascribed to an A_3C_{60} phase with a half filled $2t_{1u}$ orbital. In the fully doped insulating A_6C_{60} phase the $2t_{1u}$ orbital is fully occupied. Photoemission studies [5,6,7] confirm that the HOMO of K_6C_{60} is derived from the LUMO of C_{60}. Doping with alkali metals induces a phase transition after half of the $2t_{1u}$ orbital is filled, as tentatively indicated in the photoemission study of Wertheim et al. [5] It was found later [8] that, upon alkali doping, a face centered cubic (fcc) lattice of C_{60} transforms into a body centered cubic lattice (bcc) capable of accommodating six alkali metal atoms per C_{60} molecule.

The changes in electronic structure of C_{60} induced by doping with alkali metals have been investigated by several different techniques. These include PES [5,6,7], IPES and XPS [9], and EELS [10] studies of potassium-doped C_{60}. To the best of our knowledge there have been no reports yet on optical spectroscopy of the alkali metal-doped C_{60} systems. Here we present our observed changes in absorption spectra of potassium-doped C_{60} in the 220 nm $< \lambda <$ 2.1 μm region, and Li, Na, and Rb doped C_{60} in the 220 nm $< \lambda <$ 800 nm region.

Based upon the changes in the electronic spectra of K-doped C_{60}, the assignment of the electronic states involved in the optical transitions of the C_{60} and K_6C_{60} phases is proposed and compared with existing data in the literature.

C_{60} was synthesized using a simple benchtop reactor [11] and purified by liquid phase chromatography [12]. A few milligrams of C_{60} were sublimed from a quartz crucible, heated to 450 $^{\circ}$C at a pressure of 5×10^{-6} Torr, onto 1" x 1" fused silica plates to produce films with thicknesses between 300 and 600 Å as determined from absorbance measurements. Raman spectroscopy indicated that the films contained less than 4% C_{70}. For the simultaneous absorbance/resistivity measurements that were performed on the K_xC_{60} samples, a pair of thin gold pads was deposited on the quartz plate prior to the C_{60} deposition. Simple two probe resistivity measurements were carried out using a laboratory Fluke-25 multimeter with lowest readout corresponding to 1 nS. No attempt to estimate specific resistance was made since our goal was a simple correlation of the absorption spectra with change in conductivity.

The vacuum chamber originally used to study reactions of alkali metals with zeolites [13] was adapted for this experiment as shown in Fig. 1. Absorption spectra in the region between $220<\lambda<800$ nm were collected using a commercial HP 8452A UV-VIS spectrophotometer whose optical cavity was modified to provide a double pass of the UV-VIS light through the sample. Due to the considerable optical losses in the far-UV region the measurements were restricted to $\lambda>220$ nm. Commercial SAES-

Getters alkali sources were powered using a dc current stabilized power supply. Prior to deposition, the alkali metal flux was adjusted using the feed-back from a quadrupole mass-spectrometer (UTI 100C) placed in line of sight with the alkali source. The fused silica plate containing the C_{60} film was attached to the end of a metal rod and vacuum sealed by an Ultratorr fitting. Using such an arrangement the sample plate could be rotated and translated in the direction perpendicular to the optical axis. Successive alkali metal deposition intervals followed by quick absorbance measurements were achieved by a simple 90^{o} rotation of the sample. The HP spectrometer equipped with a diode array detector provided 2 nm spectral resolution with an integration time of 5 s. The spectra were corrected for the absorption of the fused silica plate. The IR spectrum of the K_6C_{60} phase in the 0.85 $\mu m < \lambda < 2.1$ μm region was recorded with a Cary-14 digital spectrophotometer modified in such a way as to include the vacuum chamber with doped sample inside its optical path.

The change in the K_xC_{60} resistivity with deposition time is plotted in Fig 2. For every experimental point in this figure the absorption spectrum of the K_xC_{60} film was recorded in the manner described above. For the sake of clarity a few of the recorded spectra, which correspond in time to the filled circles on the conductivity curve, are shown in Fig. 3.

Given the film thickness of ~ 450 Å and the deposition time of 73 minutes required to form the insulating K_6C_{60} phase, the potassium flux in our experiment was estimated to be on the order of $5x10^{13}$ atoms$\cdot$cm$^{-2}\cdot$s^{-1}. In a series of experiments with up to four times higher deposition rates, we obtained essentially identical

spectral/conductivity behavior as presented in Figs. 2 and 3. This suggests high potassium diffusion rates through thin films of A_xC_{60} which is in agreement with recent observations [9,14,15].

Owing to the exceptional stability of the alkali source one may attempt to associate deposition time with the predominant K_xC_{60} stoichiometry. This requires that the sticking coefficient of the alkali metal remains unchanged and that the potassium diffusion rate through the sample is much greater than the deposition rate. While the latter requirement appears to be met, the most recent studies indicate certain nonlinearities in the sticking coefficient of potassium on C_{60} films [9]. Therefore, direct correlation between the observed spectra and K_xC_{60} stoichiometry using the deposition time is strictly correct only for x=0 and x=6 while intermediate concentrations should be regarded as approximate. Deviation of the resistivity curve in Fig. 2 from the Gausian shape could be a consequence of both the change in the sticking coefficient and/or the sign of an onset of the phase transition expected to take place after half of the doping is completed.

Among the spectroscopic studies of potassium-doped C_{60} the best comparison with our data can be made with the XPS [9] and EELS [10] spectra. While there is substantial agreement in the 0.6 - 4 eV region, our absorption spectra show more details in the 4-6 eV region. The reason is that the 216 nm and 264 nm bands [16], as well as the strong 6.4 eV plasmon band, of C_{60} [10] broaden in the EELS and XPS spectrum to the point where no individual transitions can be recognized (for comparison see Fig 3. in ref [9]).

Starting from the high energy end of the spectrum in Fig. 3, we can clearly follow in time a slowly increasing red shift of the 216 nm band of C_{60}. The intensity does not change significantly with doping and the shift appears to be almost completed at half the deposition time. The λ_{max} of this band was found at 225 nm in the K_6C_{60} spectrum. The strongest UV band of C_{60}, at 264 nm, broadens in the initial doping stages although its maximum can be clearly followed through all of the intermediate spectra. The band appears to be only slightly red shifted with λ_{max} found at ~ 267 nm in the K_6C_{60} spectrum. The maximum intensity of this band in the K_6C_{60} spectrum is slightly less than the band at 225 nm. The first new feature that appears in the K_6C_{60} spectrum is the band at 302 nm. It is difficult to say from our data whether or not this band evolves from a splitting of the strong 264 nm band of C_{60}. In any case, its maximum gains in intensity beyond the half doping point but does not significantly shift.

The 339 nm absorption band of C_{60} shows rather different behavior from the bands described above. As can be seen in Fig. 3, the intensity of this band rapidly decreases with doping so that it can not longer be recognized in spectra taken past the half-doping point. A weak absorption plateau in the C_{60} spectrum in the $420<\lambda<500$ nm region vanishes at a rate similar to the 339 nm band. Disappearance of the 339 nm band is followed by appearance of two new absorption features: a broad (~ 1 eV FWHM) asymmetric band in the visible with λ_{max} at ~ 425 nm and an additional band in the near IR with λ_{max} at~ 1100 nm (see Fig. 4c). Both of these new bands continuously grow with alkali

deposition to reach maximum intensity at the end of the doping cycle. The broad and asymmetric shape of the 425 nm band indicates that it may consist of more than one optical transition. Even though the two absorption maxima at 1.1 μm (1.1 eV) and 425 nm (2.9 eV) nm measured in the absorption spectrum of K_6C_{60} differ slightly from the peak energies in the EELS spectrum [10], it appears that the same transitions were recorded in both experiments.

While no definite assignment exists for the 216 nm band of C_{60}, the strongest UV 264 nm band has been tentatively attributed to the $1h_u$-> $3h_g$ transition by Sochmen et al. [10]. They also attribute the C_{60} 339 nm band to the $1h_u$->$1t_{1g}$ transition in agreement with theoretical calculations based on the CNDO/S [17] method. However, another optically allowed [4] $2h_g$ -> $2t_{1u}$ transition is also predicted in the same energy region [17,18].

The two new bands which appear in the EELS spectrum of potassium-doped C_{60} [10] at 1.3 and 2.75 eV were attributed, respectively, to the $2t_{1u}$->$1t_{1g}$ and the $1h_u$->$1t_{1g}$ transitions of K_6C_{60} with the energy shifts from C_{60} to K_6C_{60} being considered a consequence of nonrigid behavior of the C_{60} π levels as a function of dopant concentration [10]. The relatively large (3.66 - 2.75 = 0.91 eV) energy shift of the $1h_u$->$1t_{1g}$ transition in the EELS spectrum was supported by the shifts of the 1s - π_i^* transitions in potassium doped C_{60} [10]. The largest shift was found for the 1s - π_2^* (π_2^* = $1t_{1g}$) transition.

Based upon changes in the absorption spectra with doping and the following theoretical considerations, we offer an alternative spectral assignment, specifically for the 339 nm band

of C_{60} and the 425 nm band of K_6C_{60}. The transfer of electrons from alkali metal atoms into the empty $2t_{1u}$ LUMO of C_{60} is expected to strongly affect optical transitions which involve electronic states derived from this orbital. Thus, the $2h_g$ -> $2t_{1u}$ transition should lose its intensity as the $2t_{1u}$ orbital is filled with electrons to the point where it is completely extinct in the fully doped K_6C_{60}. In addition, the two lowest energy electric dipole allowed transitions originating from the newly occupied $2t_{1u}$ orbital should occur: $2t_{1u}$ -> $1t_{1g}$ and $2t_{1u}$ -> $3h_g$ [19]. These are expected to gain in intensity with doping since the population of the $2t_{1u}$ orbital increases from C_{60} to K_6C_{60}.

The above suggest that the 339 nm (3.66 eV) band of C_{60} should be assigned to the $2h_g$-> $2t_{1u}$, rather than to the $1h_u$->$1t_{1g}$, transition. If $2h_g$->$2t_{1u}$ were the stronger of the two transitions, then the calculated 18:1 [18] ratio of oscillator strengths for these two transitions would be consistent with our analysis. Our analysis is also consistent with assigning the two absorption bands of K_6C_{60} occurring at 425 nm (2.92 eV) and 1.1 μm (1.1 eV) as due to excitation of an electron from the $2t_{1u}$ orbital to the $3h_g$ and the $1t_{1g}$ orbitals respectively. Again, if the $1h_u$->$1t_{1g}$ transition is weak it is possible that both our assignment and the previous one could be valid.

There is little doubt that the $2t_{1u}$->$1t_{1g}$ assignment of the 1.1 μm band in K_6C_{60} is correct. This transition was found at a similar energy in the XPS [9] study and in the absorption spectrum of the C_{60} - radical anion [20]. The LDA calculations of K_6C_{60} predict an indirect band gap at Eg=0.48 eV and, from the

band diagram, it appears that the direct $\Delta k=0$ transition is just above the calculated band gap [21]. Although the true origin of the electric dipole allowed $2t_{1u}$->$1t_{1g}$ transition of K_6C_{60} is not known, we found the long wavelength onset of the absorption in this excitonic band at ~2 μm (0.62 eV). One should be careful, however, about comparing this experimental result with band gap calculations [22].

Changes in the UV-VIS absorption spectra with doping were also recorded for Li, Na and Rb doped C_{60} films but no simultaneous conductivity measurements were made in these experiments. We find similar overall spectral behavior for all alkali metal doped C_{60} samples with some variations in the positions, splitting, and relative intensities of the absorption bands. These differences can be readily noticed in Fig. 4 where the spectra of the fully doped A_6C_{60} (A=Na, K, Rb) phases are compared. Changes in the spectrum of the Li doped C_{60} sample could only be monitored in the initial doping stages since continuous absorption due to the reaction of Li with the fused silica substrate eventually masks the spectrum. Up to the point where the spectral changes could no longer be followed (~ Li_3C_{60}), the changes in the Li_xC_{60} spectrum were found to be similar to the rest of the alkali doped samples.

In an early study on alkali metal-doped C_{60} [8], performed under thermodynamic equilibrium, only the fcc phase of C_{60} and the bcc phase of A_6C_{60} (A=K, Cs) were found in the X-ray diffraction spectra. The existence of the stable fcc A_3C_{60} (A_3=K_3,Rb_3,Rb_2Cs) phases has since been documented [24]. In fact, X-ray photoelectron studies of C_{60}/A_xC_{60} (A=K,Cs)

heterostructures [14] indicate that the C_{60}/A_3C_{60} heterostructure is more stable than that of C_{60}/A_6C_{60}. Quite recently, a new K_4C_{60} phase with the body centered tetragonal (bct) lattice has also been identified [24].

We took advantage of our experimental set up to study the stability of the K_3C_{60} stoichiometry and its possible disproportionation to more stable C_{60} and K_6C_{60} phases [8]. A thin film of K_3C_{60} stoichiometry with the characteristic spectrum shown in Fig 3 (t_d= 37 min) was heated for 24h using collimated light from a 300 W xenon lamp and the spectrum remained unchanged. In addition, normalized absorption spectra of C_{60} and K_6C_{60} from Fig. 3 were averaged and upon comparing to the spectrum of K_3C_{60} some obvious differences were seen. Finally, we also found that all K_xC_{60} spectra in the $0<x<3$ region can be well reproduced by addition of appropriate intensity normalized spectra of the C_{60} and K_3C_{60} phases. This analysis excludes the possibility that disproportionation into C_{60} and K_6C_{60} took place during the experiment and supports the postulated[14] nonexistence of solid solutions of K_xC_{60} in the $0<x<3$ region. Similar modeling to the above, based on the K_3C_{60} and K_6C_{60} spectra, was not successful in the $3<x<6$ region.

In conclusion, we have introduced a simple experimental technique to monitor changes in the absorption spectra of thin films of solid C_{60} during doping with alkali metals. This technique can be easily extended to other heterostructures. The UV-VIS and near IR spectra of alkali metal (A=Li, Na, K, Rb) doped C_{60} molecules were investigated with simultaneous conductivity

measurements being used to monitor the K_xC_{60} stoichiometry. All investigated A_xC_{60} systems show similar spectral behavior with alkali doping, although the absorption spectrum of Na_6C_{60} is noticeably different from that of K_6C_{60} and Rb_6C_{60}. Based upon the changes in the absorption spectra induced by electron transfer from potassium atoms to the C_{60} molecule we propose alternative assignments of the 339 nm band of C_{60} and the 425 nm band of K_6C_{60} to those previously proposed. Annealing of the K_3C_{60} phase showed no signs of disproportionation, supporting higher stability with respect to the other phases. Analysis of the K_xC_{60} spectra in the $0<x<3$ region indicates the existence of a simple mixture of the C_{60} and the K_3C_{60} phases but the same cannot be said for the K_3C_{60} and K_6C_{60} phases in the $3<x<6$ region.

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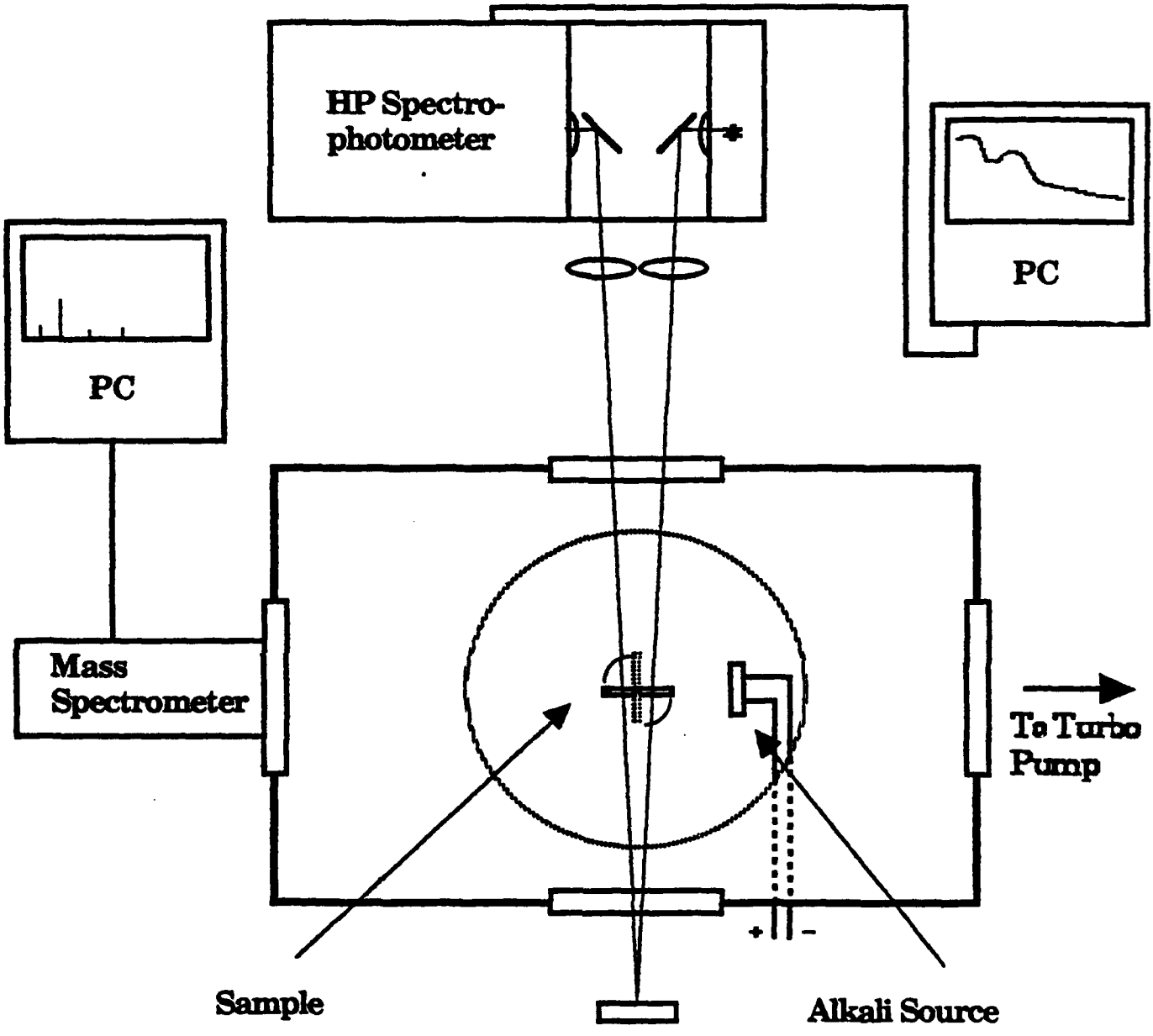
Figure captions:

Fig.1. Experimental apparatus.

Fig.2. Two probe conductivity measurements of the ~ 450 Å potassium doped C_{60} film with electrode separation of ~1". Solid curve is a least squares fit to a Gausian function using slightly more than the half experimental points in the region to the left of the maximum. Experimental points assigned by filled circles correlate to the spectra shown in Fig.3.

Fig 3. Change in the absorption spectrum of an ~ 450 Å C_{60} film deposited on the quartz substrate during doping with potassium. Absorption spectra taken after 0, 37 and 74.5 min correspond to the C_{60}, K_3C_{60} and K_6C_{60} samples respectively.

Fig 4. Absorption spectra of A_6C_{60} (A=Na, K, Rb).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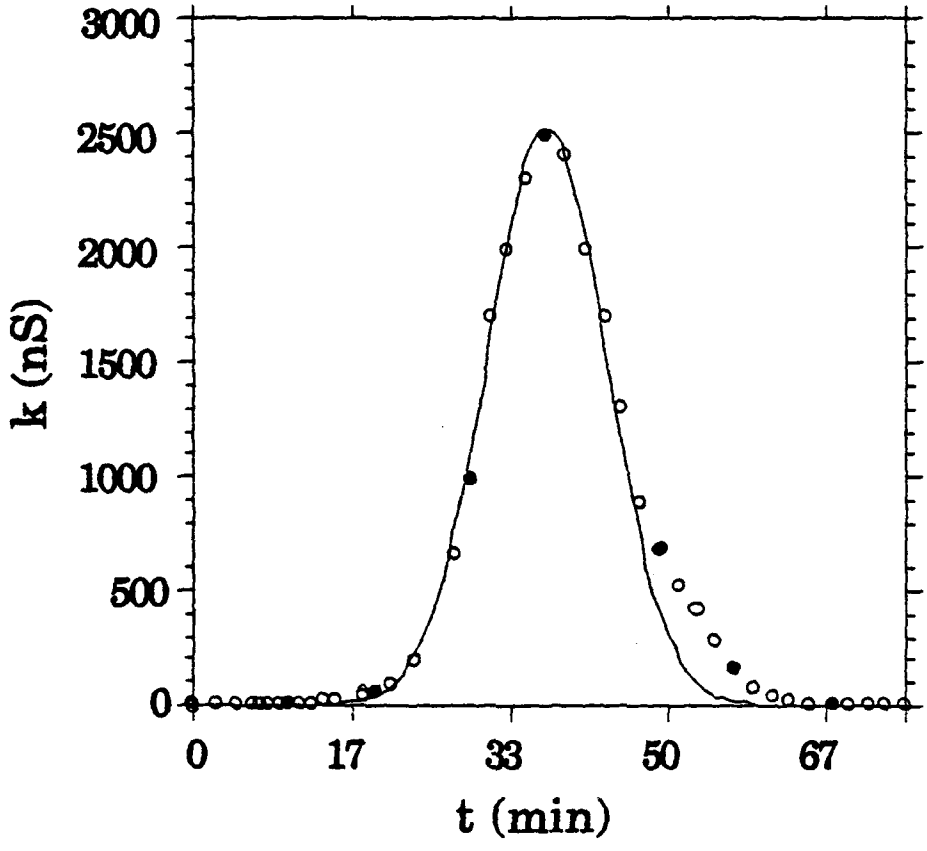
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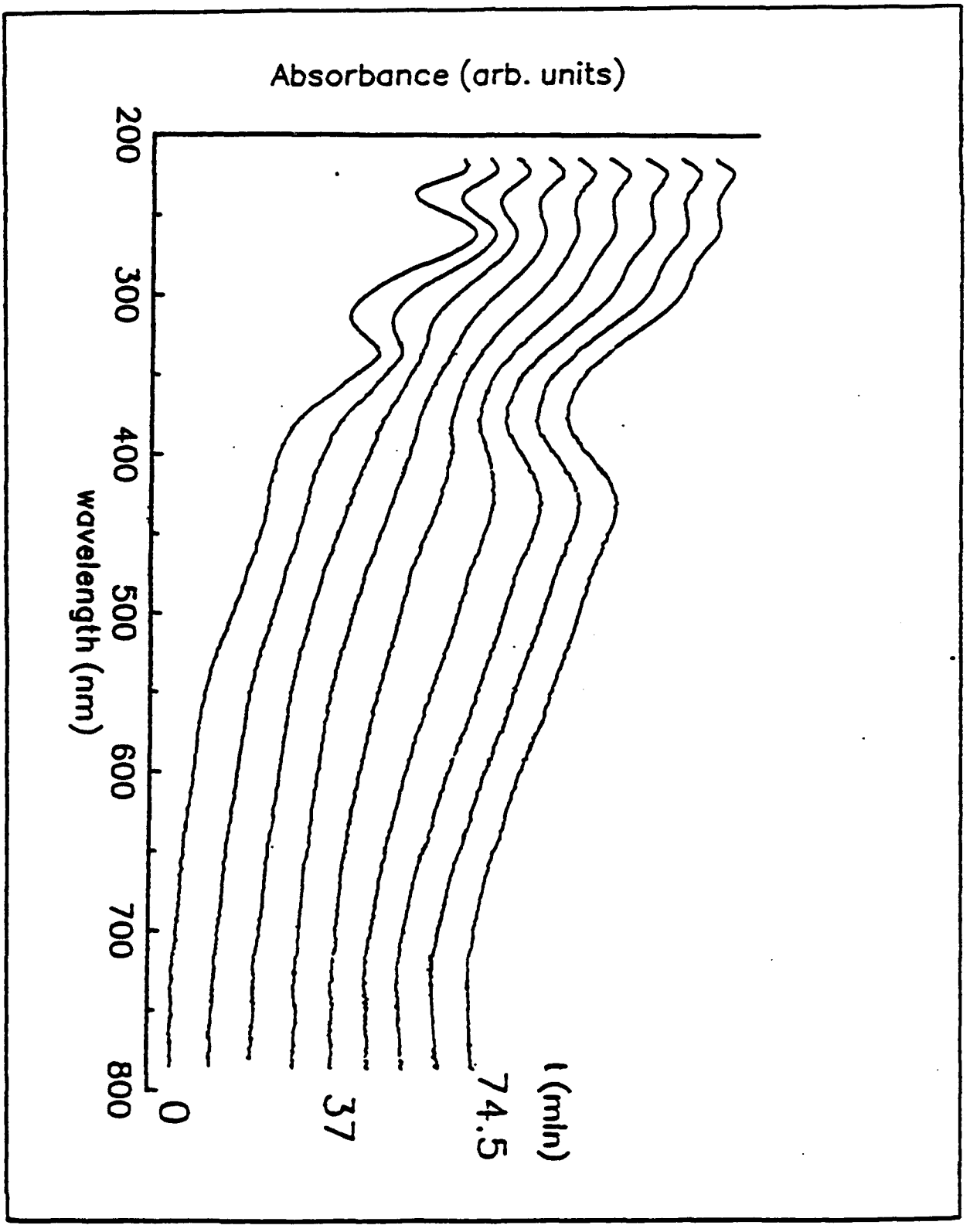
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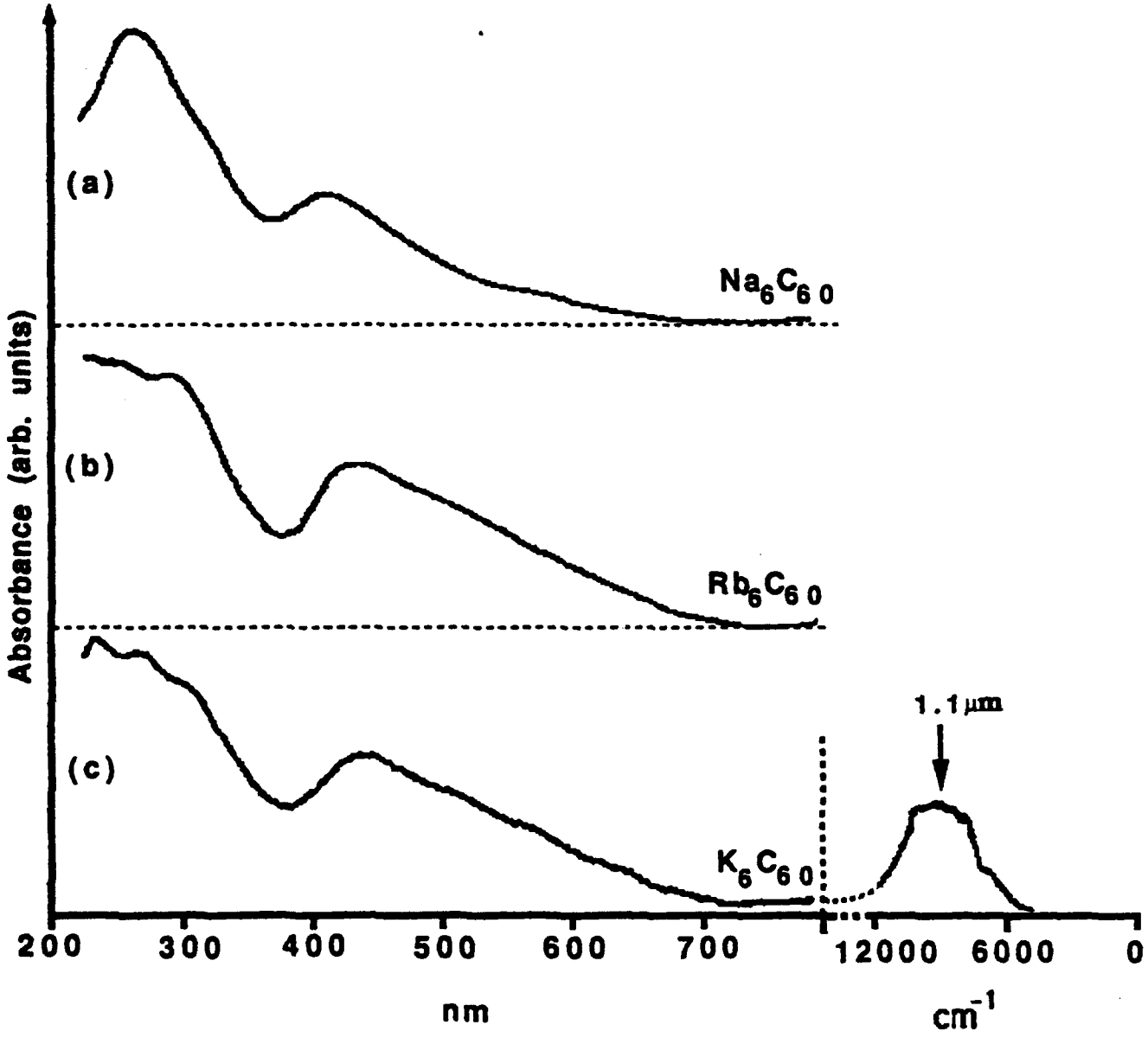
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6000
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