

# Laser flash photolysis system with vidicon digitizer and computer data analysis\*

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Laser spectroscopy system for investigation of dynamics of fast processes and molecules with lifetimes  $10^{-3}$ – $10^{-9}$  s has been described. The application of an interface module coupling the optical unit with a microcomputer through an analog-digital converter allowed full automation of data acquisition and collection in a short time of the accurate results of the lifetimes and other photophysical properties measured. The operation of the system is illustrated by some examples.

## 1. Introduction

To obtain a complete set of data describing the properties of molecules in excited states a series of spectral (SP), photophysical (PP) and photochemical (PH) measurements, both stationary and in particular dynamic were performed.

The aim of the measurements was to obtain quantitative information about the energy and lifetime of the investigated molecule in the excited states as well as about the quantum yield ( $\Phi$ ) and rate constants of all deactivation processes from these states.

Owing to the development of laser technology, many possibilities have been opened up for SP, PP and PH investigations. On one hand such properties of laser radiation as monochromaticity, coherence and high intensity enabled measurements of PP and PH processes with  $\Phi$  as small as  $10^{-6}$ – $10^{-7}$ . On the other hand, very short duration of laser pulses allowed us to investigate the processes as fast as  $10^{-3}$ – $10^{-13}$  s [1], [2].

Laser flash photolysis system LFP [1]–[3] has been most widely applied in dynamic investigations. This system may be used to investigate the properties of molecules in electronic excited states by measuring the decay dynamics of emission processes ( $E$ ), which may be realized for the compounds of  $\Phi_E > 10^{-5}$  as well as the transient absorption processes which may be measured for all compounds. LFP

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system was described for the first time by Porter and Windsor and their coworkers [2], [3].

The modified system of this kind constructed in our laboratory [4] has been successfully applied for investigation of SP, PP and PH properties of osazones, and in particular, the mechanism of photoisomerization process and structural changes of phenylosazone D-glucose [5]–[7].

The essential disadvantage of the LFP system was the method of detection and the analysis of the obtained results. At first the decay curves recorded on the oscilloscope screen were photographed. Then to determine the lifetimes screen were photographed. Then to determine the lifetimes of the molecules in the excited states or the time duration of the processes studied, a time consuming and inaccurate analysis of the oscillograms obtained was required. To avoid that, a system called a transient digitizer was introduced. When used in the LFP system it enables a transformation of an analogous signal into a digital one with fairly high accuracy, and equipped with a computer it allows to obtain the results within a short time [8], [9]. However, the normal transient digitizer is very expensive and is produced neither in Poland nor in any of the Eastern countries.

Therefore, we concentrated on digitization of the curve stored on the oscilloscope screen by applying an industrial TV camera. Thus, 256 sweep lines were used to code each point along the time scale by 8-bits binary number, which resulted in a raster of the size  $256 \times 256$ . The interfacing board to the optical system was built in such a way that it transformed the TV-camera signal into numerical data which then was transmitted with a TV standard speed to the memory of a computer equipped with a parallel module.

In order to reach a full automation of the process of the measured data acquisition, 11 programmes were worked out, which form a system called Data Acquisition System (DAS).

## 2. Laser flash photolysis system — construction and operation

A schematic diagram of the apparatus is presented in Fig. 1. The optical unit of the system presents a modified version in comparison to that described in [4]. The electronic part of the LFP, consisting primarily of an analog to digital converter and the computer system for storing the curves and analysing the studied processes is described for the first time in this paper.

### 2.1. Optical unit

The source of the exciting light is a Q-switch ruby laser with a resonator modulated passively by cryptocyanine in methanol. A ruby rod of 150 mm in length and 10–15 mm in diameter pumped optically by two xenon-flash lamps VQX 1515 (Verret Quartz, France) is placed in a head cooled with distilled water of room temperature. The optical resonator is formed by two dielectric mirrors of transmissions 0% and 60–80%, respectively (for  $\lambda = 694$  nm). In Q-switch regime the

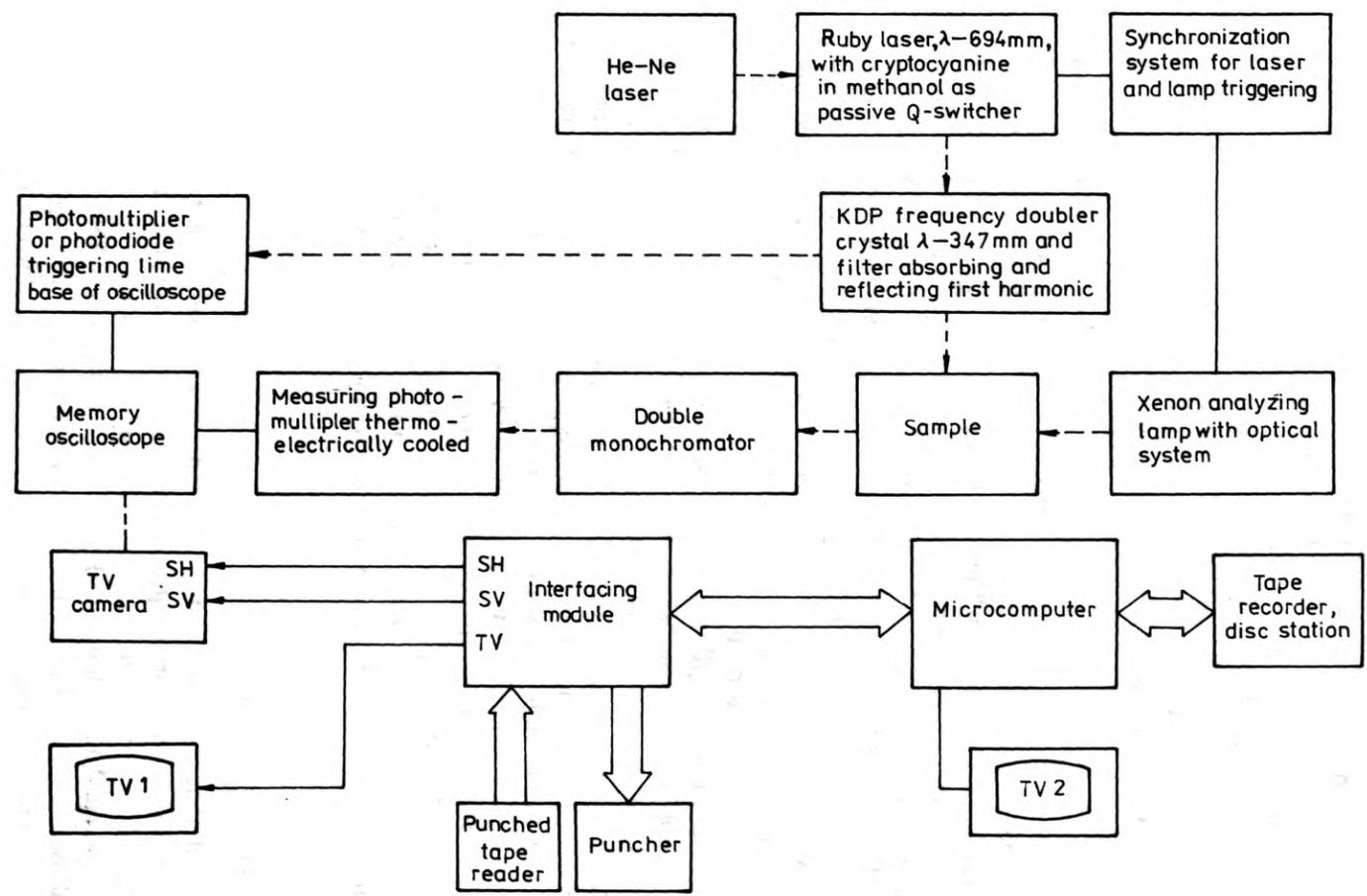


Fig. 1. General block diagram of the laser flash photolysis system: - - - - - optical line, ——— electric line (SH — horizontal signal, SV — vertical signal)

laser produces the pulses of energy about 0.5 J and a half-lifetime of 15 ns (mean power 30 MW). Through a filter absorbing the radiation of  $\lambda = 694$  nm, the incident beam is directed onto the KDP crystal which acts as a frequency doubler. Another filter placed behind the crystal absorbs and partially reflects the first harmonics and transmits the second ( $\lambda = 347$  nm). The beam of the wavelength 347 nm and the energy in the pulse  $\sim 20$  mJ focused by the cylindrical lens falls onto the studied sample. Part of the radiation is directed through a beam splitter to a photoelectric energy meter the signal of which is registered on the recorder.

The analysing beam from a xenon pulse lamp of flash time 1–15 ms falls also on the sample at the normal to the direction of the exciting laser beam. This flash lamp is filled with xenon under a relatively high pressure which provides radiation of approximately the same intensity and a broad spectral range from 200–1000 nm. The total pulse lamp energy reaches 50–3000 J, but only a part of it in the form of a parallel beam shaped by the system of diaphragms and lenses, falls on the first millimetre of the laser-irradiated sample perpendicular to the laser radiation [5], [7], [10], [11]. Having passed through the sample studied, the analysis beam is focused by a quartz cylindrical lens on a slit of the double quartz or flint monochromator.

The release detection system with a characteristic rise-time  $\leq 2$  ns consists of photomultipliers 1P-28 (RCA) or R446, R928 and R106 (Hamamatsu) as its essential elements. The signal from the detection system is observed on the oscilloscope screen. The time base of the oscilloscope is released by a signal from a photodiode or a photomultiplier. High frequency oscilloscope was optically coupled with the system of computer registration and analysis.

When the transient-absorption signals of the investigated sample are from 458 to 515 nm (six probing wavelengths) or from 568 to 676 nm (three probing wavelengths), we may use an argon laser, ILA-120 (C. Zeiss Jena) or a krypton laser ILK-120 (C. Zeiss Jena), as an analysing one. It is particularly convenient since such a laser emits continuous radiation of high intensity and small cross-section of the beam, which provides a high sensitivity of the detection system. A synchronous system of release of the analysing lamp and ruby laser is applied.

The apparatus presented in Fig. 1 although usually used for absorption measurements, may also be applied for investigation of the dynamics of the emission processes. In this case the analyzing lamp is turned off and the detection system measures directly the decay of the emission signal of the sample excited by the laser pulse [10], [11].

## 2.2 Data processing system

A general block diagram of the system recording very fast signals is shown in Fig. 2. The TV camera is mounted on an optical bench and is connected to the oscilloscope in such a way that its horizontal lines are perpendicular to the vertical lines on the oscilloscope screen. This is shown in Fig. 3 where  $y(t)$  indicates the signal recorded on the oscilloscope screen, and  $V(t)$  the video camera signal. The digital form of the signal  $V(t)$  is obtained by the analog to digital converter (A/C) which is a part of the

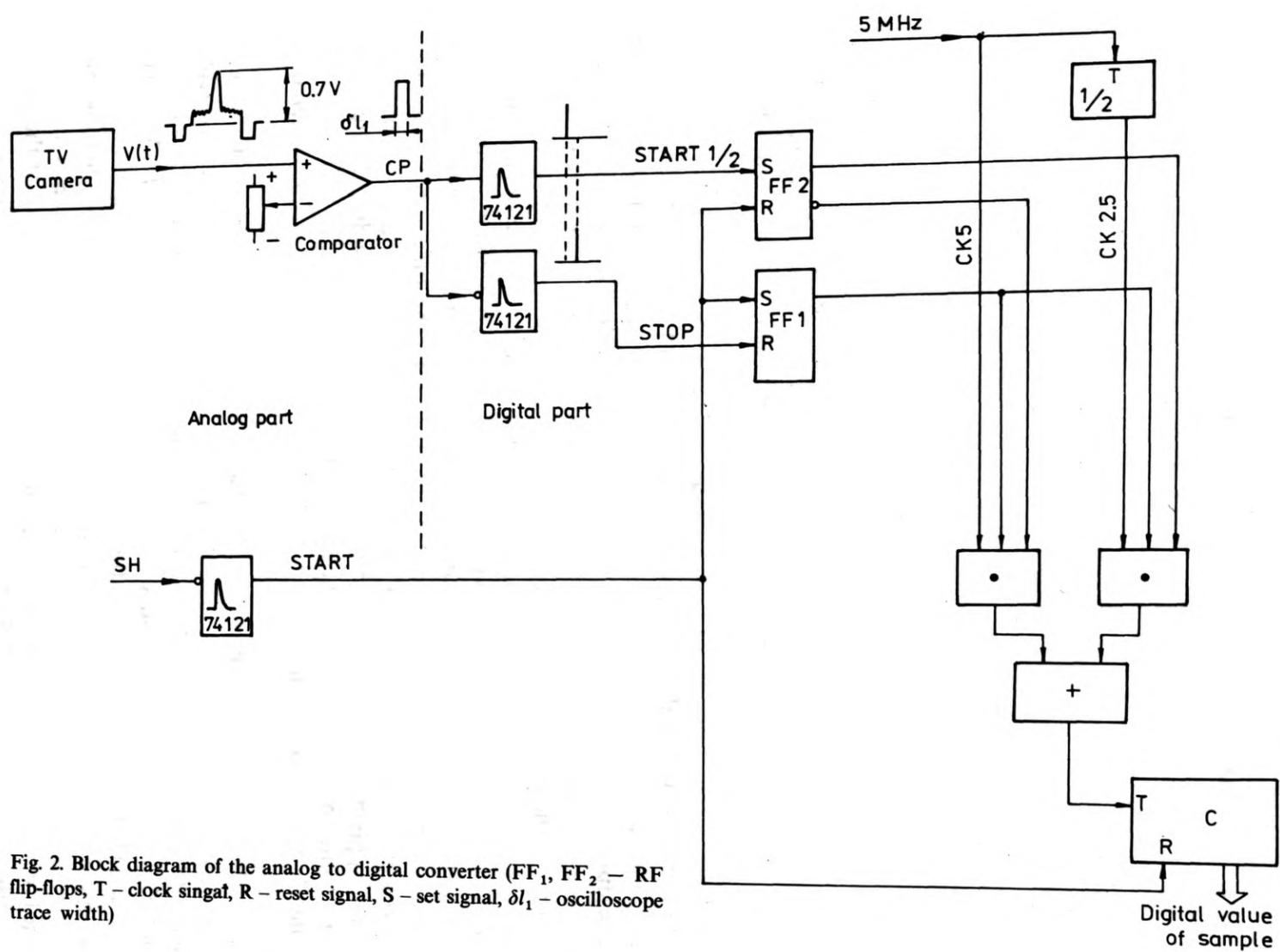


Fig. 2. Block diagram of the analog to digital converter (FF<sub>1</sub>, FF<sub>2</sub> – RF flip-flops, T – clock signal, R – reset signal, S – set signal,  $\delta l_1$  – oscilloscope trace width)

interfacing module. The latter is equipped with parallel interface which connects it with a ZX-81 microcomputer (at the beginning stage) or IBM-XT (presently). To improve the reliability of the ZX-81 computer, when applied, it was directly connected with the measuring system, equipped with a professional key board and

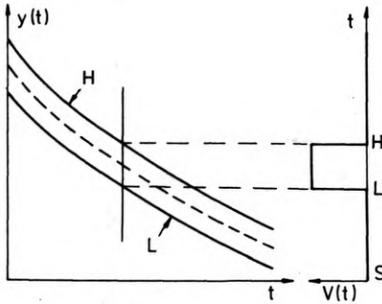


Fig. 3. Oscilloscope trace width and video signal level

enclosed in a Faraday box. The system has two TV-sets. The first one enables the user to control visually the signal recorded on the oscilloscope screen, while the second one serves as a monitor and is used by an operator communicating with the DAS system.

In order to explain the operation of the system we will refer to Fig. 3. The ideal trace (dotted line) of the signal visible on the oscilloscope screen is always in midposition between two edges, the lower  $L$  and upper  $H$ . The explanation of this phenomenon turns out to be very simple, namely it points to a heterogeneous structure of the luminophore and to the parasitic signals which when superimposed with the signal measured determine a definite thickness of the trace. As follows, the digital value of the sample's signal  $y(t)$  for a given value  $t$  corresponds to the distance measured from the camera's base (time axis  $t$ ) to the trace of the measured signal in Fig. 3. In this system 256 camera lines are analysed. The time required for the analysis of a single line is  $64 \mu\text{s}$  thus the complete analysis of the entire screen takes 20 ms. Applying the above-mentioned method for measuring the digital value of a single point, we may measure all the 256 points along the time axis ( $t$ ). The value of a single point is recorded in the 8-bit counter and the whole curve in  $1/4 \text{ K}$  memory RAM.

The hardware system operation is explained by a block diagram of A/C converter (Fig. 2) as well as time diagrams which are presented in Fig. 4. The camera horizontal signal (SH) starts the clock 5 MHz (CK5). The video signal  $V(t)$  which corresponds to the brightness of each point on the oscilloscope screen varies from 0.0 V which corresponds to the dark point to about 0.7 V for the point at the bright line. This voltage is the threshold value for the comparator. The 5 MHz signal controls the 8-bit counter C and it is stopped when the system of the comparator detects the trace of a bright line (lower edge) of the signal observed on the oscilloscope. At the same time the signal of CK 2.5 (2.5 MHz) starts the C counter, which counts till the comparator detects the upper edge  $H$  of the trace. Signal D indicates the data valid

of the sample's value which is stored in the counter C. It is worth noticing that  $\delta_1$  and  $\delta_2$  correspond to the width of the curve trace on the oscilloscope. The sequence of signals is repeated 256 times, and the points obtained in results form a digital

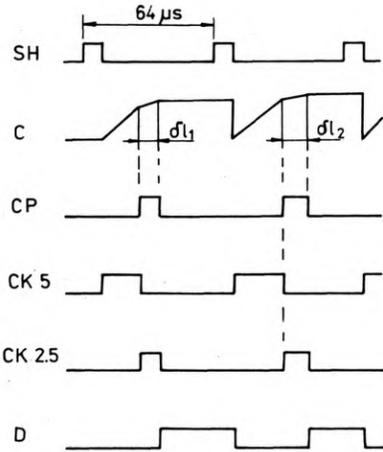


Fig. 4. Timing diagram of vidicon digitizer, for explanation see the text, ( $\delta l_1$ ,  $\delta l_2$  - oscilloscope trace widths)

representation of the measured signal. As the analysis reveals, the digital value of the sample corresponds to the distance (amplitude)

$$D = L - S + \frac{1}{2}(H - L). \tag{1}$$

The last expression determines the distance on the oscilloscope screen and the value of the counter that corresponds to it. The operation idea of a similar hardware system can be found in [13], [14].

### 2.3. Software

As Figure 1 shows, the system described has been provided with the following off-line equipment: disk station, cassette tape recorder, paper-tape reader and puncher. Since the cassette recorder is a part of the standard computer system ZX-81, thus the original software of ZX-81 required further developments in order to operate on new equipment.

In case of using IBM-XT computer equipped with a disk station all the records can be stored on a floppy disk. In the DAS the sets of results of the measurement stored in RAM memory can be called by symbolic names defined by a user furthermore, those names become formal parameters of the DAS procedures. In this system 11 commands were defined. The procedure's names are as follows: Run, Plot, Copy, Save, Load, New, Rand, Clear, Print, Input and List. Additional information provided by the system include, e.g., the list of names of measurement records, error signalling, etc. Further, the DAS allows to fulfill some other functions such as cancelling false records, error corrections, etc.

As it is presented the DAS appears to be a specialized operating system. The set

of binary routines residing in 2.5 kB of EPROM memory. It should be emphasized that the system may store up to 124 records.

Two exemplifying programmes including some comments and description are given in the Appendix.

### 3. Results of testing measurements

The possibilities of the described LFP with vidicon digitizer and computer data analysis system were illustrated on the basis of several examples. Figure 5 shows a transient absorption decay for a triplet  $\rightarrow$  triplet transition,  $T_1 \rightarrow T_n$  for the deoxygenated anthracene solution ( $c = 1 \cdot 10^{-3}$ ) in EtOH. The obtained  $\tau_{T_1} = 108 \mu\text{s}$  is in agreement with the literature data [2], [14]. As to Fig. 6, it presents the

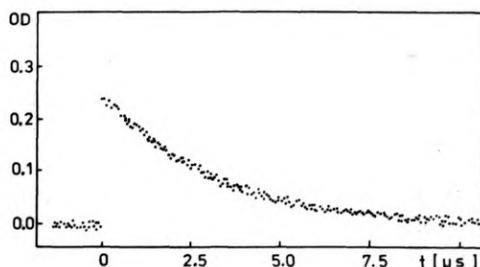
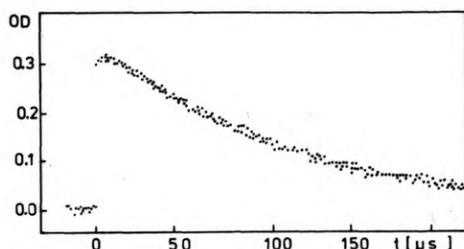


Fig. 5. Experimental trace for the decay of the transient absorption  $T_1 \rightarrow T_n$  signal of anthracene ( $c \sim 1 \cdot 10^{-3}$  M) in deoxygenated 95% EtOH at room temperature,  $\lambda = 421 \text{ nm}$  (OD – optical density)

Fig. 6. Experimental trace for the decay of the transient absorption  $T_1 \rightarrow T_n$  signal of chrysenes ( $c \sim 1 \cdot 10^{-4}$  M) in deoxygenated cyclohexane solution at room temperature,  $\lambda = 570 \text{ nm}$

absorption decay signal  $T_1 \rightarrow T_n$  for deoxygenated chrysenes solution ( $c = 1 \cdot 10^{-4}$  M) in EtOH, and in this case the measured  $\tau_{T_1} = 2.7 \mu\text{s}$  agrees with the literature data, too [14]. In both cases the error of lifetime measurements is smaller than 1%.

In the LFP system described here, investigations of SP and PP properties of aromatic thioketones in  $T_1$  state have been carried out [15].

### 4. Application of computer vidicon digitizer and data analysis system

The application of the computer system of vidicon digitizer and data analysis (VDDA) enables to obtain more accurate results of the lifetimes and absorption values. For example, the previous method of lifetime measurement gives an error of 10–20%, whereas the new method usually gives 0.5–3%.

The error in the calculated lifetime was found by regression and its value was assumed to be three times standard deviation ( $3\sigma$ ). This value depends on the

amplitude (distance)  $D$ , of the measured signal, the range of fitting and the width of the oscilloscope line,  $B = H - L$ . This is illustrated by an example of calculation of the error in the measured lifetime (Fig. 7). As follows from these calculations this error does not exceed 1%, even for disadvantageous conditions of measurements. This conclusion was drawn from the analysis of the experimental curve distribution

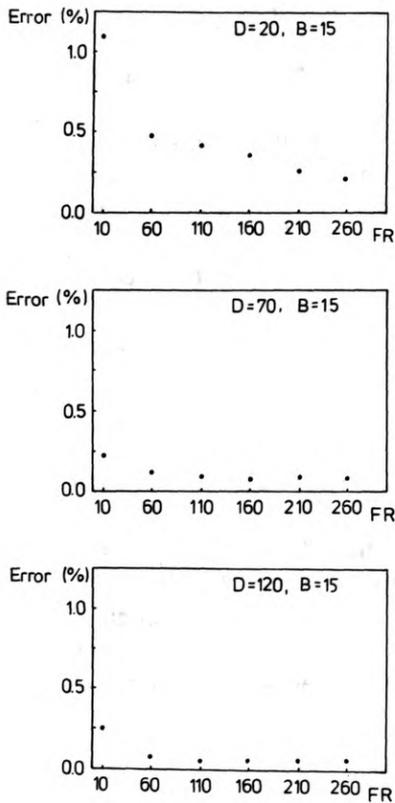


Fig. 7. Error in the measured lifetime (three standard deviation) versus the amplitude of the signal ( $D$ ), the fitting range ( $FR$ ), and the oscilloscope line width ( $B$ )

performed for as many as 256 points as well as from the observations of the signal on the appropriately chosen time base. The total error in lifetime measurements includes the systematic errors related to the signal distortions in the oscilloscope and in the camera. This systematic error can reach at most 2%. Thus, the total error should not exceed 3% and it can be reduced even to 0.5% by programmable elimination of the errors originating from the oscilloscope and camera induced distortions of the signal as well as by choosing appropriate conditions of measurements.

With such a small error it is possible to state more easily and with greater precision whether the investigated decay is one-, two-, or multi-exponential. For the two-exponential decays we may determine the lifetimes as well as the contribution of both processes.

Having obtained as a result of VDDA application a precisely measured and analysed shape of the laser exciting pulse and of the signal measured, we may carry

out an accurate deconvolution. It allows to study the processes that occur in the time comparable to or even shorter than that of the laser exciting pulse. Also the decay paths of the excited molecules could be determined even if three various short-living species were present in the solution, contributing to produce the observed emission and absorption signals [16], [17]. To make it possible the three independent processes studied should have significantly different decay rates and the contribution of each of them to the total decay process should not be less than 5%. We may also obtain a more accurate value of quantum yield of the transition between the singlet ( $S_1$ ) and triplet ( $T_1$ ) excited states as well as molar absorption coefficients for the transitions from these states to higher excited levels.

The application of VDDA also provides an independent, considerably faster (several times) and easier analysis of lifetime results. At present the analysis of one set of data takes 1 min, whereas the traditional method of oscillogram analysis and calculation of results involved nearly 60 min.

Additionally, the operation of the whole LFP system is fully controlled by the computer.

## 5. Essential parameters of LFP

Ruby laser: pulse duration 10–15 ns, pulse energy 0.5 J for  $\lambda = 694$  nm (I harmonic) and 0.05 J for  $\lambda = 347$  nm (II harmonic).

Analysing lamp: flash-time 1–15 ms, emission range 200–1000 nm. Optical detection system: spectral range 200–950 nm, time measuring range  $10^{-3}$ – $10^{-9}$  s.

System of digital recording of results: transition of an analog signal from oscilloscope screen into digital data the analysis time of the investigated run equals to about 1 min with the time measuring error of the investigated process about 0.5 to 3%.

## 6. Fundamental applications of LFP system

1. Measurements of absorption and emission spectra of short lifetime excited singlet  $S_1 \rightarrow S_n$  and triplet  $T_1 \rightarrow T_n$  transitions (where  $n = 2, 3, 4, \dots$ ) of noninteracting molecules, charge-transfer complexes, exciplexes, excimers and ion-radicals appearing in result of laser excitation.

2. Measurements of lifetimes of the above mentioned species, in the range  $10^{-3}$ – $10^{-9}$  s.

3. Determination of the quantum yield of intersystem-crossing  $S_1 \rightarrow T_n$  (where  $n = 1, 2, 3, \dots$ ) as well as the rate constant of this process.

4. Determination of the electronically excited level responsible for the course of the examined PP and PH processes.

5. Studies of the inter- and intramolecular processes of energy transfer.

6. Studies of the mechanism of PH processes taking place in result of the structural and conformational changes and bond dissociation.

7. Studies of the mechanism of vibrational relaxation in the electronic excited state.

## Appendix

Below we are going to present two subroutines in the DAS system. The first is the COPY \* directive that enables to rewrite the measurement results recorded under the name given by the argument into the Table C (256) declared in the programme REGRESJA (REGRESSION). After the keys COPY and NEWLINE have been pressed the DAS system asks for the buffer's name by printing BUFFER NAME: " ". When a given name is not in the memory the system goes out of the COPY

### Programme I

9853	17	11	)	LD DE, 10149	
9854	165	A5	9		
9855	39	27	B		
9856	1	01		LD BC, 5	
9857	5	05			
9858	0	00			
9859	205	CD	?	CALL 2923	Printing of the text COPY
9860	107	6B	?		
9861	11	0B	"		
9862	205	CD	?	CALL 8382	NEWLINE key control
9863	190	BE	g		
9864	32	20	4		
9865	205	CD	?	CALL 8807	Subroutine searching a name in a vocabulary
9866	103	67	?		
9867	34	22	6		
9868	58	3A	U	LD A, (65514)	
9869	234	EA	?		
9870	255	FF	?		
9871	6	06		LD B,2	
9872	2	02			
9873	184	B8	s	CP B	
9874	202	CA	?	JP Z, 8313	Jump to the level? when the RUBOUT key is pressed
9875	121	79	?		
9876	32	20	4		
9877	62	3E	Y	LD A, 7	
9878	7	07			
9879	50	32	M	LD (65514), A	
9880	234	EA	?		
9881	255	FF	?		
9882	201	C9	?	RET	Jump to REGRESJA programme

directions and prints "?". When the name is found in the vocabulary the system goes automatically to the programme REGRESJA which is written in BASIC. This programme calculates the lifetimes after some parameters, which will not be discussed here in detail, have been introduced.

The second interesting subroutine is the module coworking directly with the equipment. It is made to record 256 sample values of the signal analysed in the

#### Programme II

9883	205	CD	?	CALL 743	Fast mode is set
9884	231	E7	?		
9885	2	02			
9886	62	3E	Y	LD A, 152	
9887	152	98	\		
9888	211	D3	?	OUT 191, A	8255 is set to work
9889	191	BF	z		
9890	1	01		LD BC, 167	Zeroing of the B register and set of address of the C register
9891	167	A7	b		
9892	0	00			
9893	125	7D	?	LD A, L	
9894	211	D3	?	OUT 175, A	Sending of the number of the sample to the B buffer
9895	175	AF	j		
9896	62	3E	Y	LD A, 1	
9897	1	01			
9898	211	D3	?	OUT 183, A	Writing the sample number in the register
9899	183	B7	r		
9900	219	DB	?	IN A, 183	
9901	183	B7	r		
9902	203	CB	?	BIT 4, A	The loop verifying if the sample is ready
9903	103	67	?		
9904	40	28	C	JR Z, 9900	
9905	250	FA	?		
9906	237	ED	?	INI	The value of the sample is written under the address in HL pair
9907	162	A2	6		
9908	62	3E	Y	LD A, 0	
9909	0	00			
9910	211	D3	?	OUT 183, A	Reset the signal which shows that the sample is ready
9911	183	B7	r		
9912	32	20	4	JR NZ, 9893	
9913	235	EB	?		
9914	62	3E	Y	LD A, 0	
9915	0	00			
9916	211	D3	?	OUT 175, A	Set a marker into $\Phi$ position
9917	175	AF	j		
9918	205	CD	?	CALL 3883	SLOW mode is set
9919	43	2B	F		
9920	15	0F	?		
9921	42	2A	E	LD HL, (65521)	Set a new value of the HL registers
9922	241	F1	?		
9923	255	FF	?		
9924	201	C9	?	RET	Return from the subroutine

operating memory of the microcomputer. The analog-to-digital converter is joined with a microcomputer through the system 8255 working in  $\Phi$  mode. The port A works as an eight-bit input buffer, the port B is an output buffer whereas in the port C four older bites are declared as inputs and four younger bites as outputs. At the beginning of the subroutine a microcomputer is switched to the FAST mode and can work at the maximum speed since in this mode it does not cooperate with a TV set. Then, the 8255 system is set to  $\Phi$  mode. The microcomputer sends the number of the sample whose value is to be read, to the buffer B. This number is  $\Phi$  or any other integer number from the range 0–255 (instruction OUT 175, A). The instruction OUT 183 A sets the flip-flop and makes that the number of the sample is written into the register. Then the 4th bit of the C latch is analysed. If it is set to 1 the of the sample whose number has been given earlier, appears at the buffer A. This value is written under the address memorized in the pair of HL registers (instruction INI). The signal which shows that the sample is ready is reset by the instruction OUT 183, A. After the 256 values of the analysed course have been recorded the marker of the sample is set to  $\Phi$  and the mode of work of a microcomputer is changed to SLOW. The state of HL pair of registers is verified. The 256 samples are counted by the register B. The initial value of B is set to  $\Phi$  by the instruction LD BC OO A7 and then decremented by the instruction INI. The time required to write a single sample in the memory is of about 36  $\mu$ s that is shorter than the 64  $\mu$ s of a single TV line duration.

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### **Система лазерного импульсного фотолиза с видиконным преобразителем из аналоговой формы в цифровую с машинным расчётом**

Описана система лазерной спектроскопии применяемая для исследования динамики быстрых процессов и времён жизни молекул, находящихся в короткоживущих состояниях в пределе  $10^{-3}$ – $10^{-9}$  с. Снабжение измерительной установки модулем интерфейса, связывающего оптическую часть системы через блок аналого-цифрового преобразователя с микрокомпьютером, позволило полностью автоматизировать получение измерительных данных и быстрое вычисление точных значений времён жизни и других фотофизических величин. Работы системы иллюстрированы на нескольких примерах.