



**LASER BASED ANALYTICAL SPECTROSCOPY OF URANIUM**

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## ABSTRACT

Analytical spectroscopy of uranium has been studied using a XeCl excimer laser, using the fluorescence emission of U(VI) ions doped in a solid solution of sodium fluoride (NaF) and sodium chloride (NaCl) in 3:2 proportion. An electronic circuitry involving time-gating of the photomultiplier tube and facility to integrate the analytical signal over ten laser pulses has been developed to enable laser operation and signal detection with high S/N ratio. The matrix enhanced U(VI) fluorescence emission is free from chemical and spectral interferences due to the concomitant presence of ten metallic elements generally associated with uranium. The digital signal output is highly precise and does not saturate upto 5 ppm uranium concentration. X-ray diffraction patterns obtained for uranium doped compounds at 2.5% and 10% dopant concentrations are broadly similar to that of  $\text{Na}_2\text{U}_2\text{O}_7$ . The detailed studies have, however, revealed fine structure for individual peaks, thereby, revealing the formation of sodium fluoro-uranate complex which is responsible for the enhanced intensity of fluorescence emission.

## LASER BASED ANALYTICAL SPECTROSCOPY OF URANIUM

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### 1. INTRODUCTION

Extensive work has been reported in the literature about spectro-fluorometric determination of uranium using its intense green fluorescence and also matrix-enhanced emission due to site-selective excitation in suitable matrices.<sup>(1-4)</sup> Prominent among the matrices chosen for such an emission are  $\text{CaF}_2$  and mixture of  $\text{Na}_2\text{CO}_3$ ,  $\text{K}_2\text{CO}_3$  and  $\text{NaF}$ . While analyte uranium does not enter  $\text{CaF}_2$  lattice quantitatively<sup>(3)</sup> thereby introducing an uncertainty in its determination, fusion mixture type of the matrix<sup>(4)</sup> does not enhance uranium fluorescence signal appreciably. Detailed studies have, therefore, been carried out to find a suitable host to enhance uranium emission. This has led to the choice of a mixture of  $\text{NaF}$  and  $\text{NaCl}$  in 3:2 proportion leading to determination of uranium at sub-ppb level. The fluorescence emission gets appreciably enhanced on using a high powered suitable laser such as  $\text{XeCl}$  excimer laser in the present case but the intense laser radiation and the associated high proportion of the scattered light affects the detection system considerably. This results in a very poor S/N ratio at low amounts of uranium. An electronic circuitry has, therefore, been specially developed here to (i) enable gating of photomultiplier tube which would result in a better S/N ratio by totally avoiding the

source radiation and also to (ii) allow effectively a single pulse operation of the laser so that the life of one fill of laser gases can be enhanced appreciably. A method based on these innovations has enabled highly precise determination of uranium at sub-ppb concentration levels. With a view to increasing the applicability of the method, possible interferences in the spectral emission have also been examined. In the next section, the design, fabrication and performance features of the electronic circuitry are described, followed by the experimental procedure adopted for chemical preparation of the compounds and the fluorescence intensity measurements on the samples.

## 2. DESIGN, FABRICATION AND PERFORMANCE OF THE ELECTRONIC CIRCUITRY

A 10 W XeCl excimer laser (Lambda Physik Model MSC 53) is used as the spectral source of excitation. A 0.25 M scanning monochromator with an RCA photomultiplier tube (RCA 4523) as the detector serves as the monitor for the fluorescence emission. A light - tight sample chamber fitted with a right angled quartz prism and a condensing quartz lens is placed in the path of the laser radiation. The chamber is equipped to hold the sample tube in a reproducible manner. The chamber has two vertical slits of 1 mm width placed at right angles to each other while the light cone is restricted to a width of 100 microns at the monochromator stage. The block diagram of the experimental set-up is shown in Fig. 1.

## 2.1 Details of electronic circuitry

The block diagram of the electronic circuitry is shown in Fig. 2. The system consists of the following units:

- 1) Intensity measurement unit (NIM module)
- 2) P.M. tube gating unit GU-175 (NIM module)
- 3) Detection assembly (Gated PMT. and bleeder)
- 4) HV unit for P.M. tube
- 5) Triple Scaler unit (NIM module)
- 6) Laser unit and high voltage trigger unit and
- 7) Bin and Power Supply LV 236

Of these, the first three items are designed and fabricated here while the remaining ones are standard units adapted for this work.

### 2.1.1 Intensity Measurement Unit:

The unit is built in double width modular form. It consists of three sections: a) Trigger Generator and Driver, b) Voltage Follower and Gain Amplifier and c) V/F Converter.

a) Trigger Generator and Driver : The circuit diagram of the Trigger Generator and Driver is given in Fig. 3. It consists of four monostables, a clock and a driver stage. All monostables are designed by using IC 74121 while IC 555 is used as a clock of frequency 1 Hz. <sup>(5)</sup> Mono-I (1-10 sec) serves as a master clock for the system. The timing of this Mono-I can be externally adjusted by using helipot P1 mounted on front panel of the module and indicated by the repetition rate. Thus one can vary the repetition rate of the system from 1 to 10. By pressing the "PRESS TO START" push button mounted on the front panel of the module, the Mono-I gets triggered. The



output from pin 6 (Q) of IC1 is connected to pin 4 (RESET) of clock (IC2). The combination of R3, R2 and C2 gives the frequency of 1 Hz. Mono-II (IC3) forms the desired pulse width for the laser trigger. By varying trimpot TR1, the pulse width varies from 0.1 - 2.5  $\mu$ sec. For the present work, pulse width of 1  $\mu$ sec has been fixed. Mono-II gets triggered from the O/P pin 3 of IC2. The O/P of Mono-II (pin 6) is connected to the base of transistor Q1 of driver stage -1. Transistors Q1, Q2 and Q3 along with R4, R5, R6 and R7 form the driver stage - 1. Transistors Q1 and Q2 form an inverter while Q3 is used as emitter follower. A pulse with width of 1  $\mu$ sec and height of 18 V is generated by the driver stage - 1 to drive the trigger input stage of the laser unit and also to provide input to the counter/display channel 2 of a triple scaler. Mono-III (0.1  $\mu$ sec - 8  $\mu$ sec) provides the desired delay before gating the P.M. tube ON. The O/P from Q of Mono-II triggers the Mono-III (IC4). The desired delay is obtained by varying trimpot TR2. Mono-IV (0 - 1 msec) forms the ON time for the PMT. The Mono-IV gets triggered from the O/P pin1 (Q) of Mono-III. The desired ON time of PMT, is obtained by varying trimpot TR3. The O/P from Q of the Mono-IV is connected to the base of transistor Q4. Transistor Q4, R10 and R11 form the driver stage - 2 which drives the PMT. Gating unit. The LM7805 provides the 5 V power supply for the various IC's in the circuit.

b) Voltage Follower and Gain Amplifier : Fig. 4 gives the circuit diagram of the voltage follower and amplifier. Operational Amplifiers (IC 741) are used as A1 and A2 of the

circuit. The output of PMT is connected to the non-inverting input of A1 which acts as a voltage follower and buffered stage to the input to gain amplifier A2. The gain of A2 can be varied in steps of 1, 5 and 10 through rotary switch (RSW1) which is connected in the feedback circuit of A2.

c) V/F Converter : V/F converters provide precision conversion of analog voltage into a train of pulses whose frequency is a direct linear function of the analog voltage over a nominal range of 0 to +10 volts. The converters provide TTL compatible output pulses with nominal width equal to 1/3 the period of full scale frequency of the device (1 MHz at 10 V). Teledyne Philbrick 4705-01 is used as the V/F converter module in this system.<sup>(6)</sup> The O/P from the gain amplifier A2 of the previous section is connected to the input  $V_{in}$  of the V/F converter. Calibration of the V/F converter is done with a standard voltage source, a scaler and an oscilloscope. The O/P of V/F converter (pin 4) is connected to the counter/display channel - 1 of the triple scaler as shown in Fig. 4.

#### 2.1.2 P.M. Tube Gating Unit GU 175

For the measurement of fluorescence intensity, the technique of photomultiplier tube gating has been used. Here, the PM tube is gated ON for a short time (0 - 1 msec) after a desired delay (0.1  $\mu$ sec - 8  $\mu$ sec) with respect to the laser excitation flash so that the intense excitation flash is not "seen" by the PM Tube.

There are several ways to gate a photomultiplier tube,<sup>(7)</sup> some of which are:

- 1) Pulsing overall high voltage supply,
- 2) Pulsing groups of dynodes,
- 3) Pulsing the photocathode,
- 4) Pulsing the first dynode,
- 5) Pulsing a focusing or gating electrode, if present.

In the present setup, the technique based on pulsing of dynodes has been used. Gating of two dynodes of PM tube has been carried out by employing transistorized circuitry as shown in Fig. 5. The laser pulse is very intense as compared to the weak fluorescence generated from the sample. If the laser pulse is also shined on the PM tube, it causes "fatigue" of the PM tube and hence the initial part of the decay trace will not truly represent the fluorescence. In the present system, however, the PM tube is gated ON only after the initial flash is over, thus totally eliminating the errors due to its "fatigue".

The gating unit is similar to the one developed earlier in the laboratory <sup>(8)</sup>. It consists of a normally conducting transistor, Q1 (EC491) whose base is connected to the variable delay output of trigger generator, Mono-IV from driver stage 2 through coupling capacitor  $C_x$ . The collector of transistor Q1 provides the two gate pulses through isolating diodes D5 and D6. The gates have the same duration as the input pulses and are of positive polarity with amplitude of 100 V.

### 2.1.3 Detection Assembly

The detection assembly (Gated PM tube and bleeder) is enclosed in a cylindrical housing and mounted appropriately

to receive the fluorescence signal from the sample. It consists of a fine variable slit, a photomultiplier tube (RCA 4523) and its dynode resistor chain as shown in Fig. 5. Dynode D1 and shield S of PM tube are connected together. The dynode chain is so arranged that D1 and S are at cathode potential and D7 is at the same potential as D6, thus preventing electron multiplication from taking place and hence keeping the PM tube in the cut off condition normally. But as soon as the gate input pulses from the PM tube gating unit GU175 are applied, all dynodes obtain voltages in the proper sequence and the PM tube is gated ON. The PM tube detects any light input only during the ON time and generates a negative output at its anode. To avoid capacitive coupling, the PMT. cathode is raised to negative high voltage and the O/P is directly taken from its anode.

#### 2.1.4 HV Unit type HV-4800/E

A standard unit supplied by ECIL has been used to supply HV to the PM tube. It is a low noise, highly stable source of high voltage which can be varied from 10 to 3000 V, either manually in the "INT" mode or automatically in the "EXT" mode of operation. The polarity of output voltage can be changed either to positive or negative by a front panel rotary switch. Its current capacity is 10 mA.

#### 2.1.5 Triple Scaler

A standard commercially available triple scaler is incorporated in the system for counting purposes. It contains three channels of six-digit scalers. Channel 1 is used to count

the V/F converter output pulses and Channel 2 is used to count the laser trigger pulses.

#### 2.1.6 Laser Unit and HV Trigger Unit

The system, consisting of 1) Laser head, 2) power supply, 3) vacuum pump with gas filter and 4) HV trigger unit is a stand-alone unit by itself. Its specifications are as follows:

- a) Wavelength : 308 nm
- b) Pulse energy : 50 mJ
- c) Maximum power : 10 w
- d) Maximum repetition rate : 200 Hz
- e) HV for all gases and repetition rates : 16 kV

#### 2.1.7 Bin and Power Supply LV236

A standard NIM Bin in which modular units described above are plugged in, has an attached power supply unit with the voltages,  $\pm 12$  V / 2 A,  $\pm 24$  V / 1 A and  $\pm 6$  V / 5 A available for use by the modules.

### 3. EXPERIMENTAL

#### 3.1 Preparation of Standards and samples

The following matrices were initially examined to study their suitability in these experiments:

- (i)  $\text{Na}_2\text{CO}_3 + \text{K}_2\text{CO}_3 + \text{NaF}$  in 9:9:2 proportion
- (ii) NaF
- (iii) NaF+LiF in 5:2 proportion
- (iv) NaF+NaCl in 3:2 proportion

Uranium at 5 ppm concentration was mixed with each of these matrices and the fluorescence yield was studied. The matrix combination of NaF and NaCl as found to be

optimum for the fluorescence studies. This was, therefore, used in further studies. The method for the preparation of standards is given below:

High purity NaF and NaCl in 3:2 proportion were thoroughly ground together using a pestle and mortar after which the mixture was heated in platinum crucible at  $800^{\circ}\text{C}$  on a burner flame in open air. On melting, the analyte aliquot in 3M  $\text{HNO}_3$  was added. The melt was further heated for 5 minutes before it was poured hot into the mortar. On cooling, the sample was ground to form a fine powder. A series of standards with varying concentrations of uranium in the range of 0-5 ppm was prepared. An additional sample at 0.1 ppm uranium concentration but mixed with ten metallic impurities, viz., Ag, Ca, Cd, Co, Cu, K, Mg, Mn, Pb and Zn each at 0.1 ppm concentration was also prepared to study interference effect, if any, in uranium determination. In addition, the following samples were also prepared for structural investigations of the fluorescing species at high uranium concentrations:

(i)  $\text{NaF} + \text{NaCl} + \text{UO}_2(\text{NO}_3)_2$ : A sample containing 2.5% by weight of uranium was prepared using the same procedure as described above for smaller proportions of uranium.

(ii)  $\text{Na}_2\text{U}_2\text{O}_7$ : This was prepared by mixing  $\text{UO}_2(\text{NO}_3)_2$  and NaOH in solution form. The precipitate was dried and further heated in the furnace at  $800^{\circ}\text{C}$  for an hour.

(iii)  $\text{NaUO}_3 \cdot \text{Cl}$ : Part of the sodium diuranate as prepared earlier was mixed with NaCl at 2.5% uranium concentration and the mixture was taken through melting stage (heating it

in a furnace in air at  $1000^{\circ}$  for ten minutes) and homogenised thereafter.

(iv)  $\text{NaUO}_3 \cdot \text{F}$ :  $\text{Na}_2\text{U}_2\text{O}_7$  was mixed with NaF at 2.5% and 10% uranium concentrations and each of the two mixtures was taken through melting stage.

### 3.2 Fluorescence Intensity Measurements

Using 308 nm laser radiation as the excitation source, the fluorescence spectrum for uranium was scanned in the spectral range 400-700 nm. The monochromator was then set on the most intense fluorescence group appearing around 550 nm. A weighed amount of each of the powder standards was transferred to identically fabricated quartz tubes of 6 mm I.D. (wall thickness of 1 mm). The tubes were successively transferred and placed in the sample holder. Care was taken to ensure that the sample tubes would go in reproducible manner in the sample holder and there would not be any change in the optical signal other than that due to varying amounts of uranium in the standards / samples. The U(VI) ion fluorescence was monitored on the triple scaler unit for each of the standards prepared earlier. The scatter components for the blank sample holder and also for the matrix alone were evaluated to know the net fluorescence signal due to uranium. The compounds prepared with uranium concentrations varying in the range 0.0001-5 ppm were placed in the sample holder space and the signal averaged over ten repetition cycles was noted in each case. With a fresh fill of the gases for XeCl laser, the relevant intensities obtained for these concentrations are tabulated in Table 1.

Detailed X-ray diffraction studies were carried out using DIANO X-ray Diffraction spectrometer with Cu-K radiation to identify the complex formed with uranium at macro-scale.

#### 4. RESULTS AND DISCUSSION

Uranyl ions are known to fluoresce strongly with their characteristic green emission around 550 nm, thus providing means for uranium determination at low concentration levels in certain compounds. The fluorescence yield, however, depends upon, among other instrumental parameters, the host lattice and hence the literature is abound with the use of several such lattices.

As mentioned earlier, initial trials with  $\text{CaF}_2$  did not prove to be successful, while fusion mixture with NaF did not give intense U(VI) fluorescence. Detailed studies were, therefore, carried out to obtain a suitable host lattice for uranium determination at low concentration levels. It was seen that uranium would fluoresce intensely when homogenised with NaF alone but the melting point for NaF, being  $990^\circ\text{C}$  it was not feasible to use commonly available laboratory furnace for such studies. Further, the mixture was found to solidify immediately on cooling and stick to the walls of the platinum crucible. Therefore a suitable additive was searched for which would make the eutectic melt at relatively low temperature and still achieve strong fluorescence yield as with NaF alone. It was seen that a mixture of NaF and NaCl in 3:2 proportion as also a mixture of NaF and LiF in 5:2 proportion would provide a combination which would melt at



about 700°C. The U(VI) fluorescence signal for NaF-NaCl combination was, however, found to be highly intense as compared to that with NaF-LiF mixture.

The use of a laser certainly improves the fluorescence signal and commercial units equipped with N<sub>2</sub> laser for uranium determination are known. The present work includes use of a 10 W XeCl laser equipped with the electronic circuitry described in detail in Section II. The salient features of the electronic set-up are: (i) elimination of the scatter component of the light signal due to high intensity incident laser pulse at the detection stage, (ii) preventing "fatigue" of the PMT, (iii) digital output for the fluorescence signal, thus eliminating use of a chart recorder and accompanying problems such as slow pen response, saturation of signal, etc. and (iv) efficient operation with pulse repetition rate of 1 per sec and facility to integrate the signal for ten repetition cycles. In addition, facilities normally available with other scaling units could also be used successfully.

As seen from Table 1, counts for 0.0001 ppm uranium concentration are significantly higher than those for the sample blank. The fluorescence signal did not show any saturation effect upto 5 ppm. The precision in the signal measurements is better than 1% as judged from the repetitive measurements on a sample containing 0.1 ppm uranium. The fluorescence intensity for the sample with uranium concentration at 0.1 ppm and mixed with concomitant impurities did not show appreciable change, thereby, showing that the method does not suffer from chemical interferences. The

spectral interference, if any, due to overlap of the absorption contours of these impurities with that of the uranium emission does not appear to be significant. It was interesting to note that the highest intensity fluorescence peak observed at 550 nm did not show any wavelength shift when studied at ppb and percentage concentrations of uranium. In order to identify the complex formed at high uranium concentrations, the sample prepared with 2.5% uranium concentration was investigated for its X-ray diffraction pattern. The pattern showed structure similar to that of  $\text{Na}_2\text{U}_2\text{O}_7$ <sup>(9)</sup>. A freshly prepared  $\text{Na}_2\text{U}_2\text{O}_7$ , however, did not exhibit any fluorescence emission. Hence the same compound was mixed successively with NaCl and NaF at higher uranium percentages and the X-ray patterns for these compounds were studied. These studies showed marked difference in X-ray diffraction pattern over the one obtained for pure  $\text{Na}_2\text{U}_2\text{O}_7$  in that the individual peaks showed fine structure, changes in intensity etc. A slow scan showed that additional peaks with  $2\theta$  values close to the peaks observed for  $\text{Na}_2\text{U}_2\text{O}_7$  would appear for the samples prepared with NaCl and NaF. The  $d$  values calculated from the basic data and intensities for various peaks observed for  $\text{Na}_2\text{U}_2\text{O}_7$  alone, those observed for  $\text{Na}_2\text{U}_2\text{O}_7$  mixed with NaF at 2.5% and 10% uranium concentrations and the data on 2.5% uranyl nitrate doped in the mixture of NaF + NaCl are given in Table 2. Based on the literature data,<sup>(10)</sup> on the formation of chloro-complexes of uranium, the complex species formed with NaCl and NaF have been identified as  $\text{NaUO}_3.\text{Cl}$

and  $\text{NaUO}_3\cdot\text{F}$  respectively. The X-ray diffraction patterns for  $\text{Na}_2\text{U}_2\text{O}_7$  and  $\text{NaUO}_3\cdot\text{F}$  have been shown in Fig. 6 which clearly shows the fine structure observed for oxyfluoro complexes and the relative changes in intensity as a function of uranium concentration. It is interesting to note that  $\text{NaUO}_3\cdot\text{Cl}$  does not exhibit any fluorescence while  $\text{NaUO}_3\cdot\text{F}$  showed very high fluorescence intensity. From X ray diffraction studies, it is concluded that at high uranium concentrations sodium fluoro-uranate complex species account for high fluorescence yield and they exhibit X-ray diffraction pattern which is very close to that of  $\text{Na}_2\text{U}_2\text{O}_7$ . This is also supported by literature data on the X-ray structural studies of oxychloro compounds.<sup>(10)</sup> The fluorescence intensity is likely to enhance on cooling the sample to 77K and on compaction of the powder as a high density pellet and may result in further improvements in the analytical performance of the method. The method described here is suitable for low level detection of uranium as required in environmental monitoring of radioactivity.

In conclusion, a mixture of NaF and NaCl in 3:2 proportion has proved to be an ideal host matrix for intense uranium (VI) fluorescence. A time gated electronic circuitry for detection and measurement of the fluorescence signal has helped in improving S/N ratio and precision of determination in the laser-based analytical spectroscopy of uranium.

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TABLE 1.

Relative Intensities For Uranium Fluorescence at 550 nm

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U Concentration (ppm)	Counts
Blank Tube	134±6
Sample Blank	430±13
0.0001	650±18
0.001	1173±27
0.005	1335±20
0.01	1516±27
0.05	1905±32
0.1	2310±24
0.5	2942±20
1.0	4555±40
5.0	9763±40

---

TABLE 2.  
 $d(A^0)$  Values For Different Compounds

Na <sub>2</sub> U <sub>2</sub> O <sub>7</sub> (SDU)		2.5% SDU in NaF		10% SDU in NaF		2.5% UO <sub>2</sub> (NO <sub>3</sub> ) <sub>2</sub> in NaF+NaCl	
I/I <sub>0</sub>	$d(A^0)$	I/I <sub>0</sub>	$d(A^0)$	I/I <sub>0</sub>	$d(A^0)$	I/I <sub>0</sub>	$d(A^0)$
100	5.95	100	5.96	100	5.98	100	5.96
50	3.36	15	3.385	10	3.386	20	3.377
		45	3.347	15	3.345	40	3.344
						35	3.255
70	3.194	50	3.187	15	3.188	40	3.186
25	2.969	70	2.985	100	2.993	60	2.975
25	2.715	20	2.706	7	2.738	18	2.732
		35	2.673	10	2.713	20	2.700
				<5	2.679		
15	2.470	15	2.472	5	2.471	15	2.467
		<5	2.318	<5	2.318		
10	2.040	8	2.046	5	2.045	5	2.045
25	1.983	45	1.989	65	1.994	10	1.959
20	1.870	15	1.879	5	1.877	12	1.881
		<5	1.865	<5	1.866	15	1.868
10	1.693	5	1.697	<5	1.697	10	1.696
5	1.697	<5	1.673	<5	1.672		
12	1.645						
		<5	1.639	<5	1.639		
5	1.595	<5	1.592	<5	1.594		

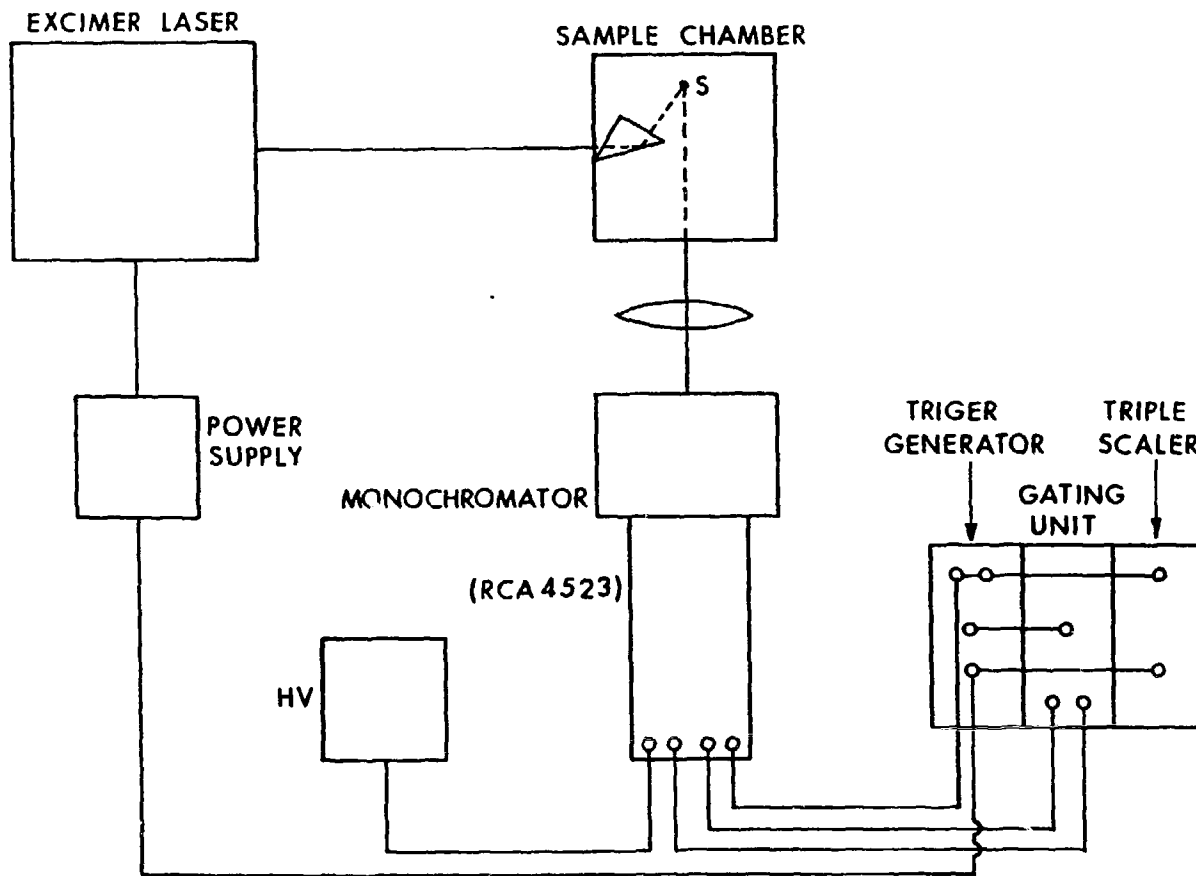


FIG.-1. EXPERIMENTAL SET-UP FOR FLUORESCENCE STUDIES.

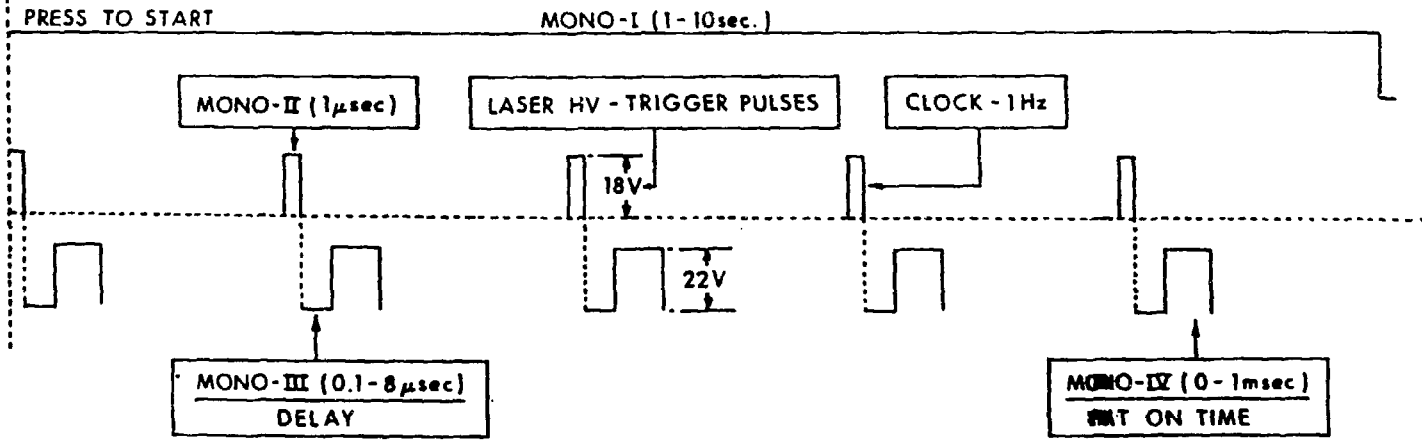
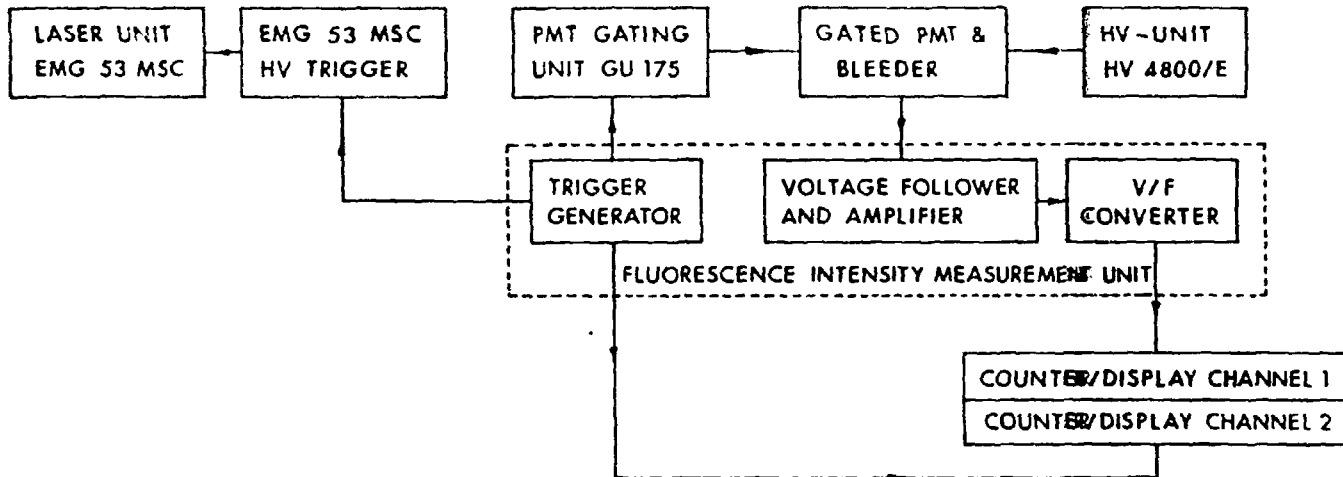
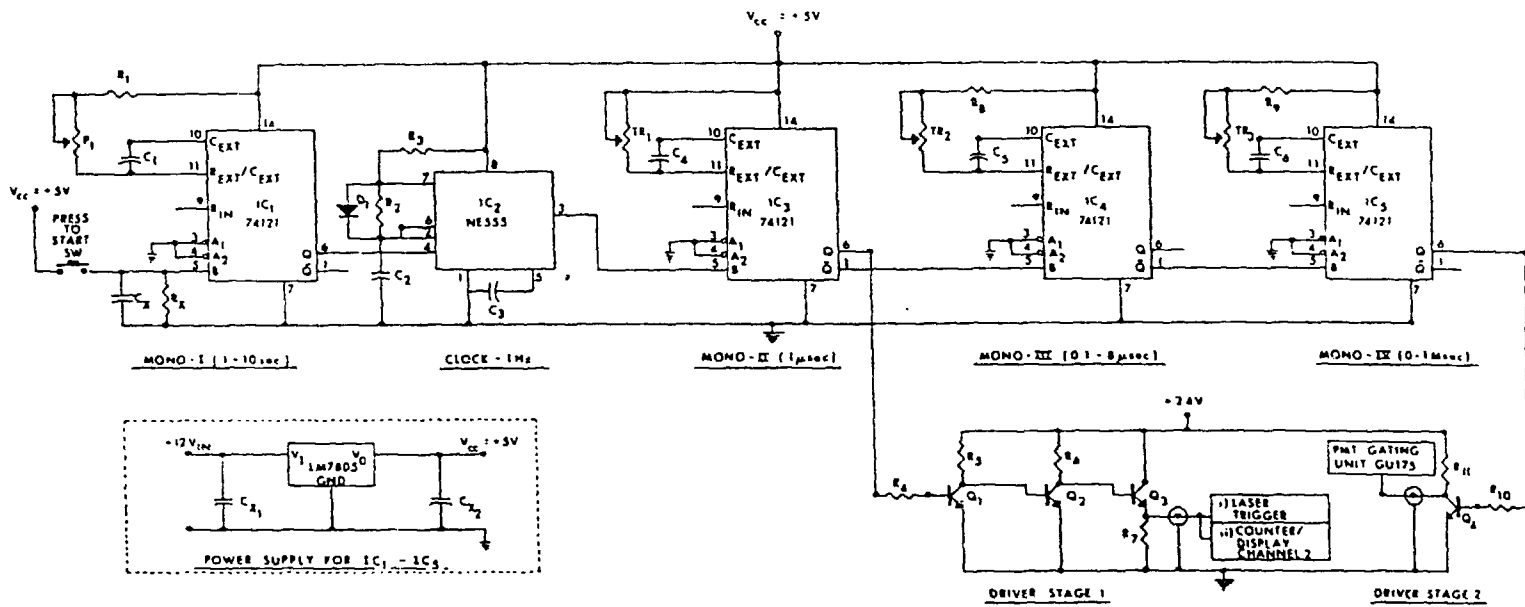


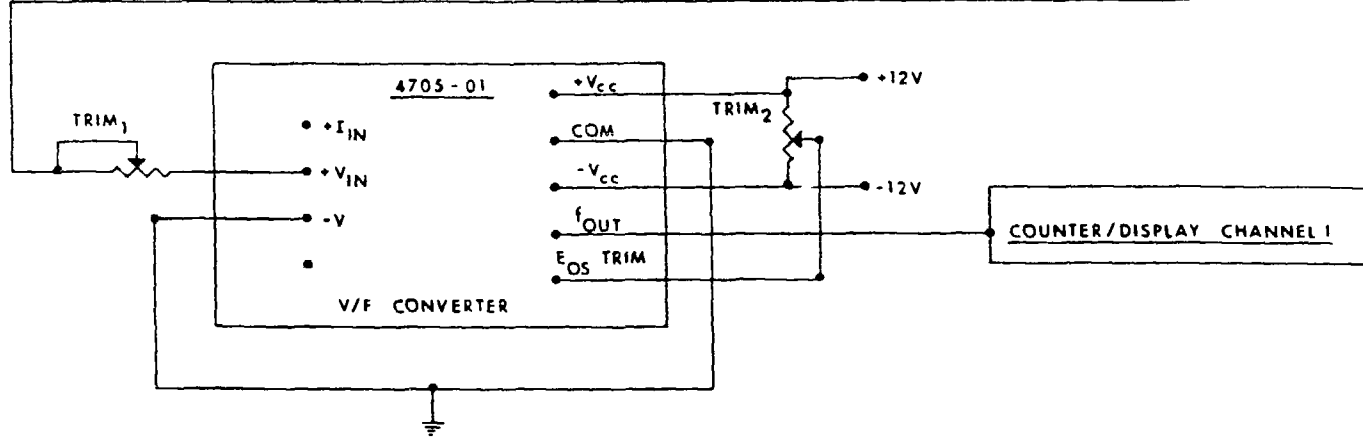
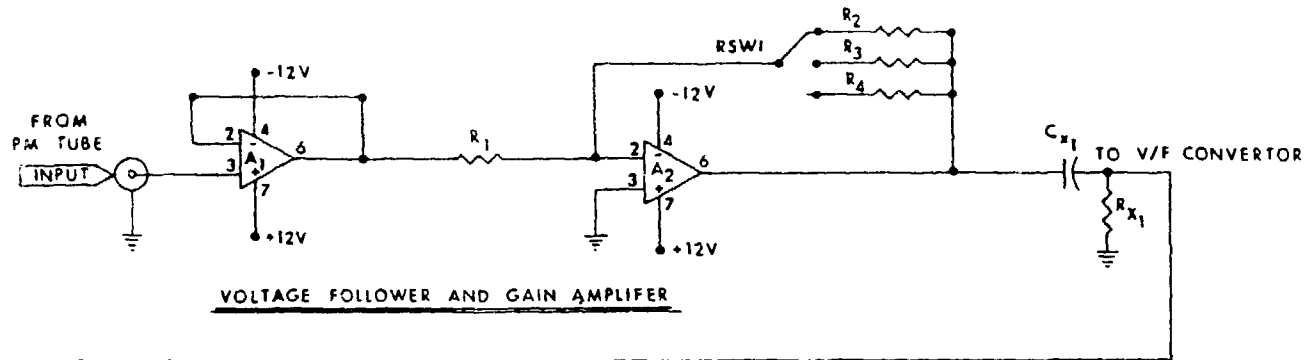
FIG.-2. BLOCK AND TIMING DIAGRAM OF THE ELECTRONICS SYSTEMS USED FOR FLUORESCENCE INTENSITY MEASUREMENT.





RESISTANCE		CAPACITOR		TRANSISTOR	TRIMPOT
R <sub>1</sub> : 9.5K	R <sub>7</sub> : 200 E	C <sub>1</sub> : 150 μf	C <sub>7</sub> : 0.22 μf/30V	Q <sub>1</sub> - Q <sub>4</sub> : 2M2219	TR <sub>1</sub> : 5E
R <sub>2</sub> : 100 K	R <sub>8</sub> : 150 E	C <sub>2</sub> : 10 μf	C <sub>8</sub> : 0.01 μf/30V		TR <sub>2</sub> : 10E
R <sub>3</sub> : 44 K	R <sub>9</sub> : 150 E	C <sub>3</sub> : 0.01 μf	C <sub>9</sub> : 0.01 μf/100V	MEL POT	TR <sub>3</sub> : 10E
R <sub>4</sub> : 10 K	R <sub>10</sub> : 1.0K	C <sub>4</sub> : 1000 pf		P <sub>1</sub> : 100Ω	
R <sub>5</sub> : 4.7K	R <sub>11</sub> : 560 E	C <sub>5</sub> : 1000 pf			
R <sub>6</sub> : 1.0	R <sub>12</sub> : 470 E	C <sub>6</sub> : 150 μf			

FIG.-3. CIRCUIT DIAGRAM FOR TRIGGER GENERATOR AND DRIVER.



AMPLIFIER  
 $A_1, A_2$  : LM741  
CAPACITOR  
 $C_{x1}$  :  $0.22 \mu\text{F}/\text{LV}$

RESISTANCE  
 $R_1, R_2$  = 1 K  
 $R_3$  = 5 K  
 $R_4$  = 10 K  
 $R_{x1}$  = 1.5 ME

TRIM  
 $\text{TRIM}_1$  = 200 E  
 $\text{TRIM}_2$  = 50K $\Omega$   
RSWI : ROTARY SWITCH

FIG.-4. CIRCUIT DIAGRAM FOR (i) VOLTAGE FOLLOWER & GAIN AMPLIFIER AND (ii) V/F CONVERTOR.

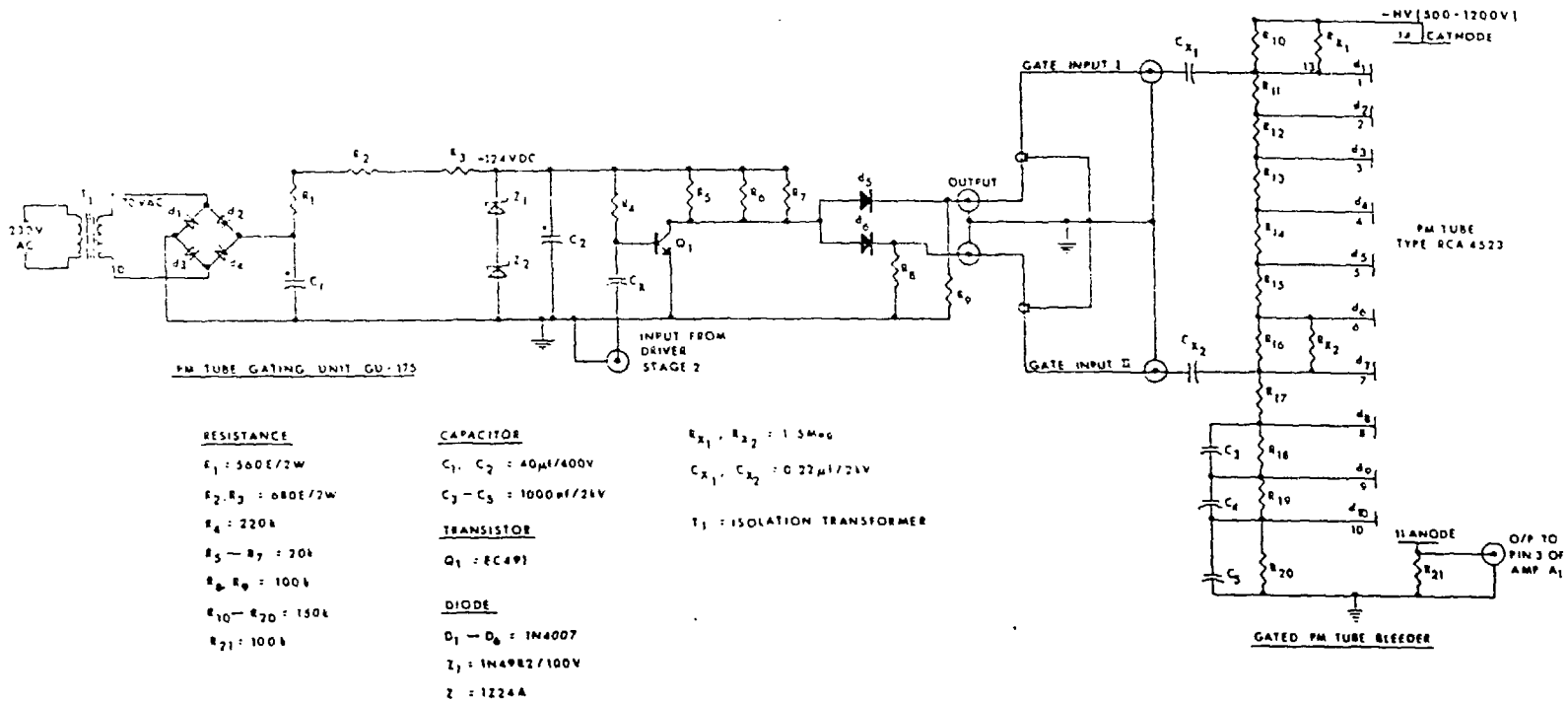


FIG.-5. CIRCUIT DIAGRAM FOR (i) PM TUBE GATING UNIT GU175 AND (ii) DETECTION ASSEMBLY.

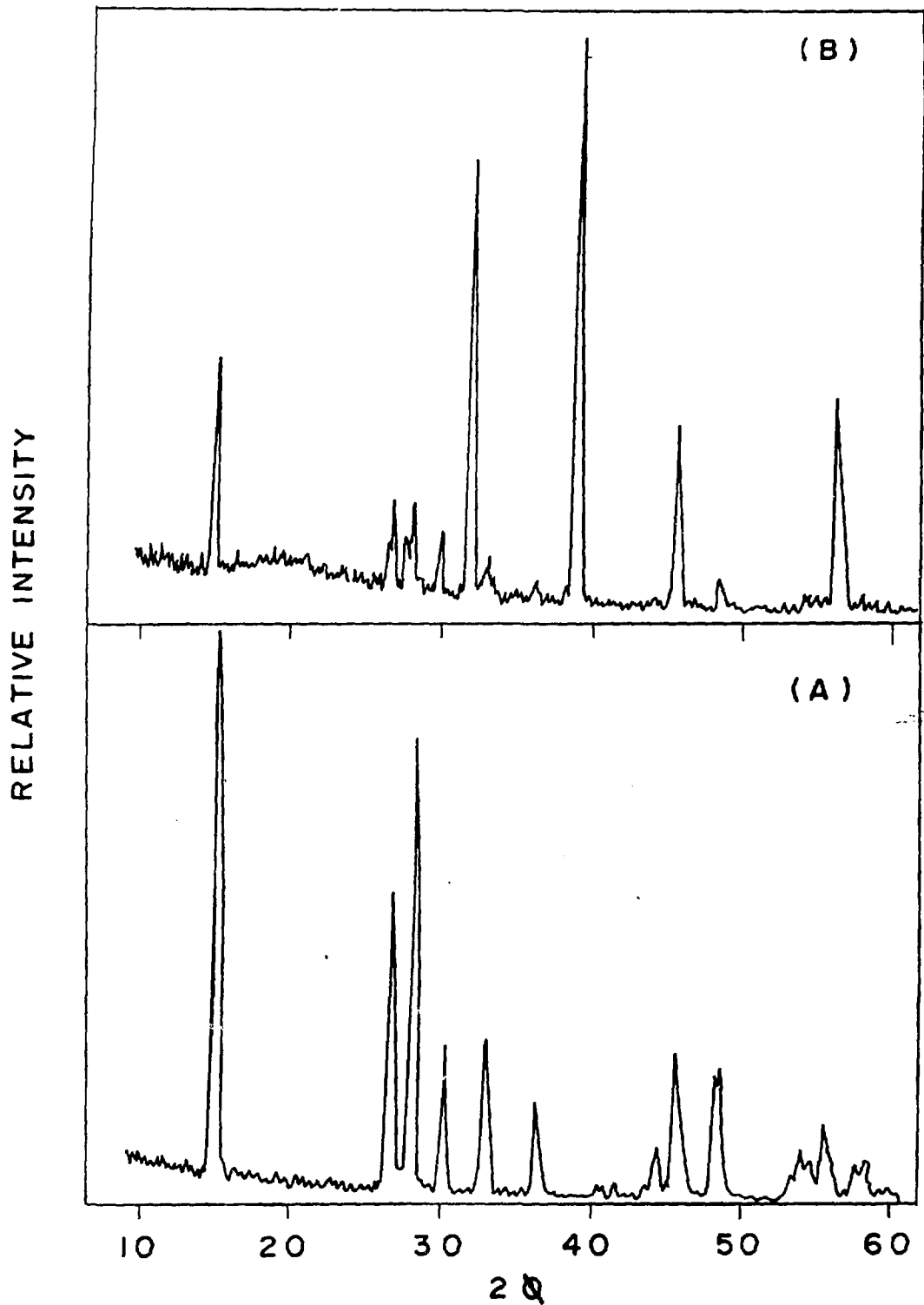


FIG.-6. X-RAY DIFFRACTION PATTERN OBSERVED FOR  
(A)  $\text{Na}_2\text{U}_2\text{O}_7$  AND (B)  $\text{Na}_2\text{U}_2\text{O}_7$  DOPED IN  
NAF + NaCl (3:2) MIXTURE AT 2.5%  
U CONCENTRATION

