CHARACTERIZATION METHODS OF NUCLEAR FUEL MATERIALS.

L.C. Paula Reino, O.W.R. Bustillos, S. Rainho Teixeira, A.R. Lordello, K. Imakuma and C. Rodrigues. Área de Processos Especiais - Instituto de Pesquisas Energéticas e Nucleares - São Paulo, Brazil.

I. INTRODUCTION

Achievement of high quality standard in the design and development of fuel rods and assemblies of modern nuclear plants requires especial manufacturing techniques as well as rigorous testing procedures after the fabrication of fuel elements. In most thermal reactors the UO_2 pellet has been the source of a large number of defects arising from chemical and mechanical effects.

In our laboratory we are presently engaged in organising quality control and quality assurance activities for PWR fuel productions. As a part of these activities, the laboratory has developed various chemical and physical methods to be employed for fuel characterization of UO_2 pellets.

The techniques developed are the determination of total residual gases by vacuum fusion method, determination of impurity elements by optical spectrograph and characterization methods by X-ray diffraction. The development and the implementation of these techniques under the general scheme of caracterization and quality control is the major theme of this paper.

II. DIRECT SPECTROGRAPHIC DETERMINATION OF IMPURITIES IN URANIUM TETRAFLUORIDE

The presence of certain micro constituents, mailny elements with high neutron absorption cross section, inhibit the proper performance of the fuels. Some of these elements are Boron, Cadmium and Silver.

The majority of the spectrographic techniques used for the impurity determination of UF_4 involves a conversion of UF_4 to U_3O_8 by pyrohydrolise[1]. So the time of analysis becomes rather long due to slow reaction of UF_4 to U_3O_8. Also there is a possibility of losses of various elements (like B and Si due to the volatility of their fluorides). For this reason it is necessary to develop a direct method of analyses.

II.1. STUDY OF SPECTROCHEMICAL CARRIERS

II.1.1. CARRIERS YIELDING LOW VOLATILE FLUORIDES

The efficiency of several substances used as spectrochemical carriers in the volatilization of residual elements in a matrix of UF_4 has been tested. The main idea, consisted in reacting the fluorine in UF_4 with a metal (carrier) during the discharge inside the electrode cavity, a fluoride compound with low volatility has been obtained as a subproduct from the reaction. This procedure makes it possible to avoid the presence of UF_4 inside the discharge region. This is interesting because its spectrum would interfere with the other chemical elements spectra otherwise.

Several carriers in the proportion of 25% relative to the UF_4 matrix have been tested (Table I).

TABLE I - LOW VOLATILE FLUORIDES

CARRIERS	FLUORIDES	BOILING POINT OF THE FLUORIDES (^oC)
-	UF_4	960
CaO	CaF_2	1360
Li_2CO_3	LiF	1676
CdO	CdF_2	1758
$BaCO_3$	BaF_2	2137
Mg^o	MgF_2	2239
La_2O_3	LaF_3	2330

The results were obtained through a visual comparison of the uranium spectra with the trace elements spectra in the sample.

The efficiency of the carrier in the suppression of the volatilization of the uranium as well as its action on the microconstituentes volatilization were tested. The carriers showing the best effi

ciency on the suppression of the UF_4 volatilization are MgO.

II.1.2. TESTS WITH MIXTURES OF CARRIERS

These tests were made adding a salt of an alcaline metal in the sample besides the carrier MgO.

The alkaline salts, like NaCl, stabilize the discharge and decrease the temperature of the electric arc as a consequence the entrance of the uranium in the discarge regions becames very dificult.

The following mixtures were studied with:

15% MgO - 15% NaCl - 70% UF_4

20% MgO - 10% NaCl - 70% UF_4

25% MgO - 5% NaCl - 70% UF_4

The mixtures 10% NaCl, 20% MgO and 70% UF_4 give the best conditions with respect to the low background, diminishes the entrance of uranium in the arc and increases the volatilization of the impurities in the UF_4. Even when an adequate carrier was used, ejection of uranium particles could be observed and more material was ejected than in the case when U_3O_8 was used as the matrix. To eliminate this problem, a series of tests (unpublished results) were taken. It consisted in dropping a gelatine solution on the electrode, over the already perforated UF_4 pellets. The best results (absence of sample ejection and an improved impurity detection) were obtained with the addition of one drop of gelatine solution at 0,5% (Photo 1).

II.2. EQUIPMENT AND EXPERIMENTAL CONDITIONS

a) Emission spectrographer: Jarrell-Ash Co., Ebert 3,4 m, supplied with a diffraction reticule of 15000 lines/in performing a linear reciprocal dispersion of 2,47 Å/mm on second order spectrum.

b) Excitation source: standard model varisource Jarrell-Ash Co.

c) Microphotometer: Digital comparer - Jarrell-Ash Co.

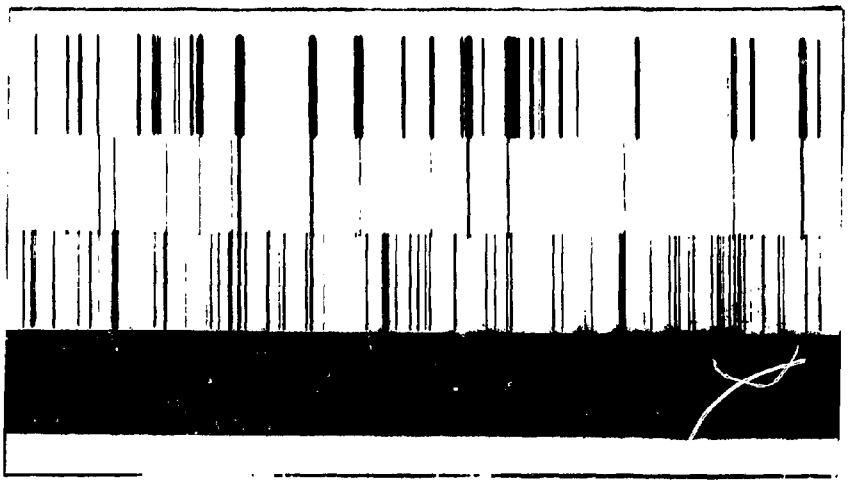


PHOTO 1 - UF_4 SPECTRA WITH AND WITHOUT UTILIZATION OF CARRIERS AND GELATINE SOLUTION.

A - Fe spectrum.

B - Use of MgO and NaCl carriers with addition of drop of gelatine solution.

C - Use of MgO and NaCl carriers.

D - UF_4 sample without addition of carriers and drop of gelatine solution.

The Photo 1 shows:

1) - The hight intensity of the C spectrum shows the high volatility of UF_4.

2) - The C spectrum shows the suppression of the UF_4 volatility when the MgO and NaCl are used.

3) - The B spectrum shows the great reduction the background and the interferences produced by the uranium spectrum as drops of gelatine solution are added. An improved impurity detection in the sample could be observed too.

d) Glass dispositive : Kodak Spectrum Analysis N° 1 (SA-1).

e) Electrodes : Anode - AGKSP 9066
Cathode - SPKL 4236
Support - AGKSP 9068

f) Diffraction lattice position : 10.00 to envolve an wave lengths range from 2[illegible] Å to 3500 Å (2nd order spectrum).

g) Continuous current: 10A, d.c. arc.

h) Slit width : 10μ

i) Preburn : 5" - Fe and Ni.
[illegible]" - Other elements.

j) Exposition time : 25" - Fe and Ni.
30" - Other elements.

k) Anode load : 100 mg

l) Analytical gap : 4 mm

Table 2 shows the analytical lines, concentration range and the precision of the method.

II.3. ANALYSIS OF THE PELLETS BY X-RAY DIFFRACTION

In order to determine the reactions occuring into the cavity of the electrode at high temperatures, an analysis of the pellet after the excitation was made by X-Ray Diffraction.

The powder diagram (Debye-Scherrer) obtained was compared to a standard UO_2 diagram. It made possible the identification of several lines and the characterization of the sample as being UO_2. From the diagram the other interplanar distances (d) were calculated. The comparison of these values with those of the ASTM[2] file made possible the identification of the lines as being the magnesium fluoride (MgF_2). It proves the initial idea of formation of a low volatility fluoride.

TABLE 2 - ANALYTICAL LINES, CONCENTRATION RANGE AND PRECISION

ELEMENT	WAVELENGTH (A^{o})	CONCENTRATION RANGE ug ELEMENT/g UF_4	COEFICIENT OF VARIATION (%)
Ag	3382,89	0,25 - 5,0	20,0
Al	3082,16	12 - 110	16,2
B	2497,73	0,9 - 50	10,8
Cd	2288,02	0,25 - 12,5	15,0
Cr	2677,13	30 - 120	19,0
Fe	2599,39	9 - 200	13,4
Mm	2605,69	7 - 106	16,3
Mo	3132,59	2 - 200	24,0
Ni	3050,82	10 - 400	18,0
Pb	2833,06	1 - 100	10,6
Si	2506,90	17 - 213	12,2
Sn	2429,49	2 - 200	9,8
V	3183,98	2 - 100	15,3
Zn	3345,02	30 - 400	15,2

III. RESIDUAL GASES PRESENT IN UO_2 PELLETS

Quantitative analysis of residual gases present in nuclear fuel elements are of primary importance in the evaluation of fuel element performance. Trace amounts of residual gases can significantly alter the mechanical properties of the fuel elements. Further, the amount of gases present as well as produced in the fuel element determines the internal pressure, wich may eventually lead to the mechanical failure and consequent release of fission gases into the primary coolant circuit.

Mass spectrometers are generally employed for the quantitative analysis of the gases in the fuel elements because of their high sensivity of detection and the ability for the positive idenfitication of each gas.Total amount of residual gases released from UO_2 pellets has been determined in this laboratory using a combination of vacuum fusion apparatus and a Mcleod gauge. Presently we are engaged in the identification and the quantitative analisis of individual residual gases using a quadrupole mass spectrometer.

The high temperature gas extraction and analisis apparatus was desinged and assembled for sequential analisis of up to four uranium dioxide pellets[3]. The system consists of three major units (Figure I), namely, outgassing unit, transfer unit and analytical unit. The whole system is evacuated to a final pressure of less than 10^{-5} Torr using a mercury diffusion pump and a mechanical pump.

The outgassing unit consists of a pellet loading and unloading arm with an externally operated magnet feed, a quartz furnace glass tube heated with induction coil from a "Politron" model 1-5 (450 KHz - 10 Kw)induction furnance.

The transfer unit, consisting of two glass difusion pumps and a cold trap cooled to a temperature of $-96^{\circ}C$ is capable of transfering the liberated gas into the analytical unit.

The analytical unit consists of a Toepler pump and calibrated Mcleod gauge.

A weighed pellet is transfered into the outgassing unit for subsequent dropping into a platinum-rhodium or graphite crucible wich is heated inductively up to $1700^{\circ}C$ during 20 minutes. The released gases are immediately transfered from the outgassing unit to the analytical unit passing through

a cold trap (at -98°C) to remove water vapor. The gases are transfered to previously calibrated volumetric bulb where the total pressure and temperature are determined. For accurate measurement of the low gas pressure a Mc Leod is used. A measurement of the blank of the whole system is carried out before each run. For this purpose the crucible, without pellets, is heated up to 1700°C during 20 minutes and the total pressure of the released gases is measured.

An estimate of the gas content in the pellets at STP condition is obtained from the measured volume, pressure and temperature of the gas mixture by applying ideal gases equation. The lower detection limit of this technique is 0,002 cm^3/g UO_2 (STP).

Analysis of one lot of uranium pellets by this method indicated a mean gas content of 0,02 cm^3/g UO_2 at temperature of 1700°C. This result represents a good performance in the fabrication of UO_2 pellets. Those pellets were fabricated by Centro de Metalurgia Nuclear (CMN) of the Instituto de Pesquisas Energéticas e Nucleares (IPEN).

Quantitative and qualitative gas analysis can be performed using a Quadrupole Mass Spectrometry[4]. The experimental unit is built up of the following parts (Figure 2): sample introduction system and Quadrupole Mass Spectrometer (including associated electronics and recording units).

The sample introduction system consists of a 1,5 litre gas expansion bulb coupled to a cold trap, leak valve and a thermocouple gauge. The bulb can be evacuated and filled with gas at desired pressure from the container. The gas from the bulb enters the vacuum inlet of the ion source through a bakcable leak valve, that can operate from atmospheric pressure to below 10^{-11} torr. The minimun leak that can be achieved is 1×10^{-9} torr litre/sec. The cold trap is used to remove condensible gases during the analysis of non-condensible gases. The whole system can be baked.

Varian model V6A-100 quadrupole mass spectrometer was used for the analysis. The spectrometer consists of an eletron impact source, a four rod quadrupole system and a faraday colletor. The out put from the collector can be displayed on an oscilloscope, as well as recorded on a potentiometer chart recorder. The total pressure in the ion source region is measured by the repeller ion current produced in the Bayard - Alpert type electron impact source.

A weighted least squares procedure has been utilized to analyse the

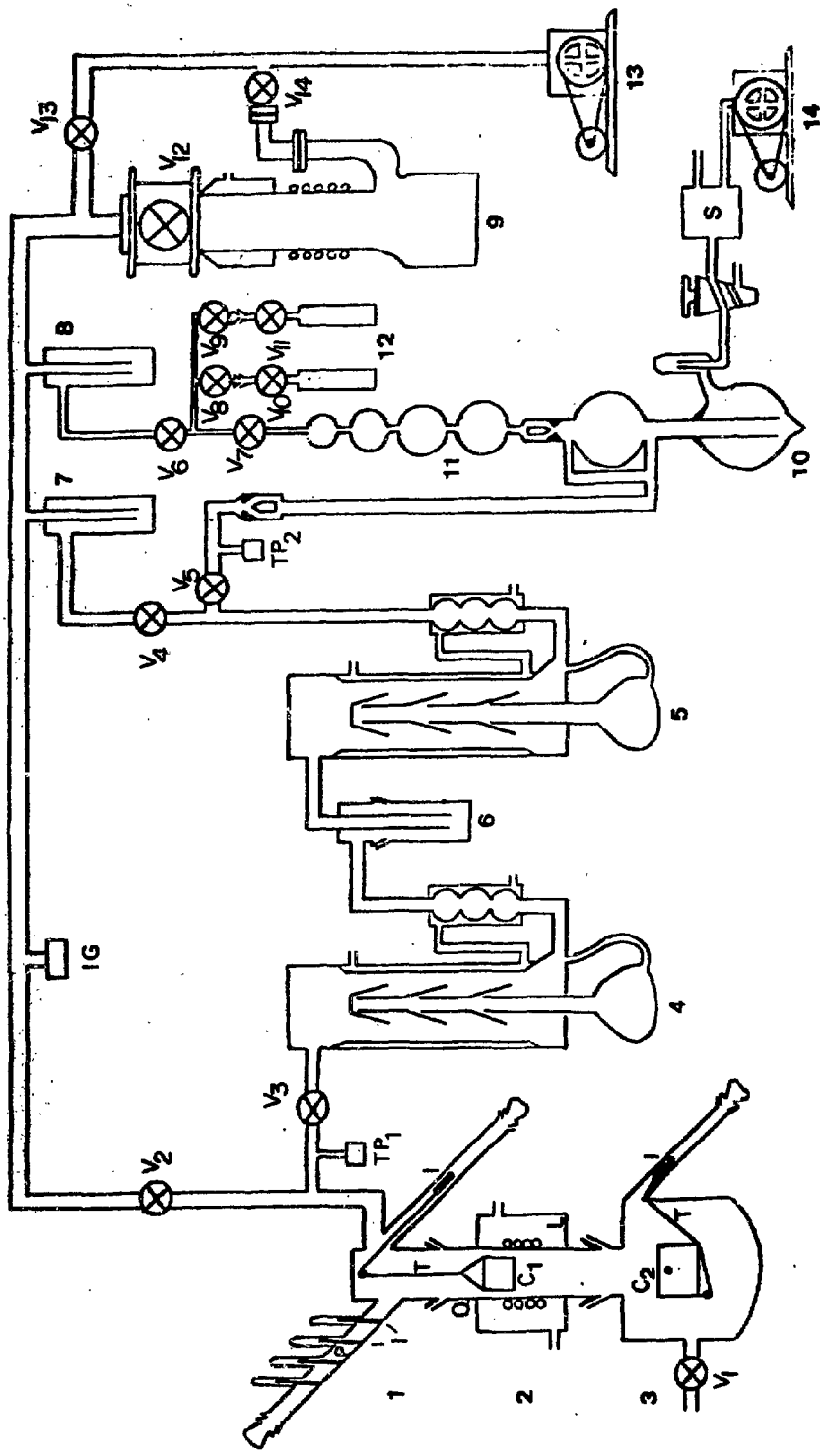


FIGURE 1 : Vacuum extraction system. (details on the next page).

VACUUM EXTRATION SYSTEM

1	= Pellets loading
2	= Introduction furnace
3	= Pellets unloading
4 & 5	= Glass difusion pumps
6	= Cold trap (- 9[illegible] °C)
7 & 8	= Cold trap (liquid Nitrogen
9	= Metalic difusion pump
10	= Toepler pump
11	= McLeod gauge
12	= Conteiners
13 & 14	= Mechanical pumps
I	= Magnets
P	= Pellets place
T	= Tungsterium wire
C_1	= Graphite crucible
L	= Watter bucket
C_2	= Glass crucible
TP_1 & TP_2	= Termocouples
IG	= Ion gange
S	= Solenoid valve
V_1 - V_{11}	= Stopcock valves
V_{12} - V_{14}	= Stainless Steel bellows valves
Q	= Quartz tuvs

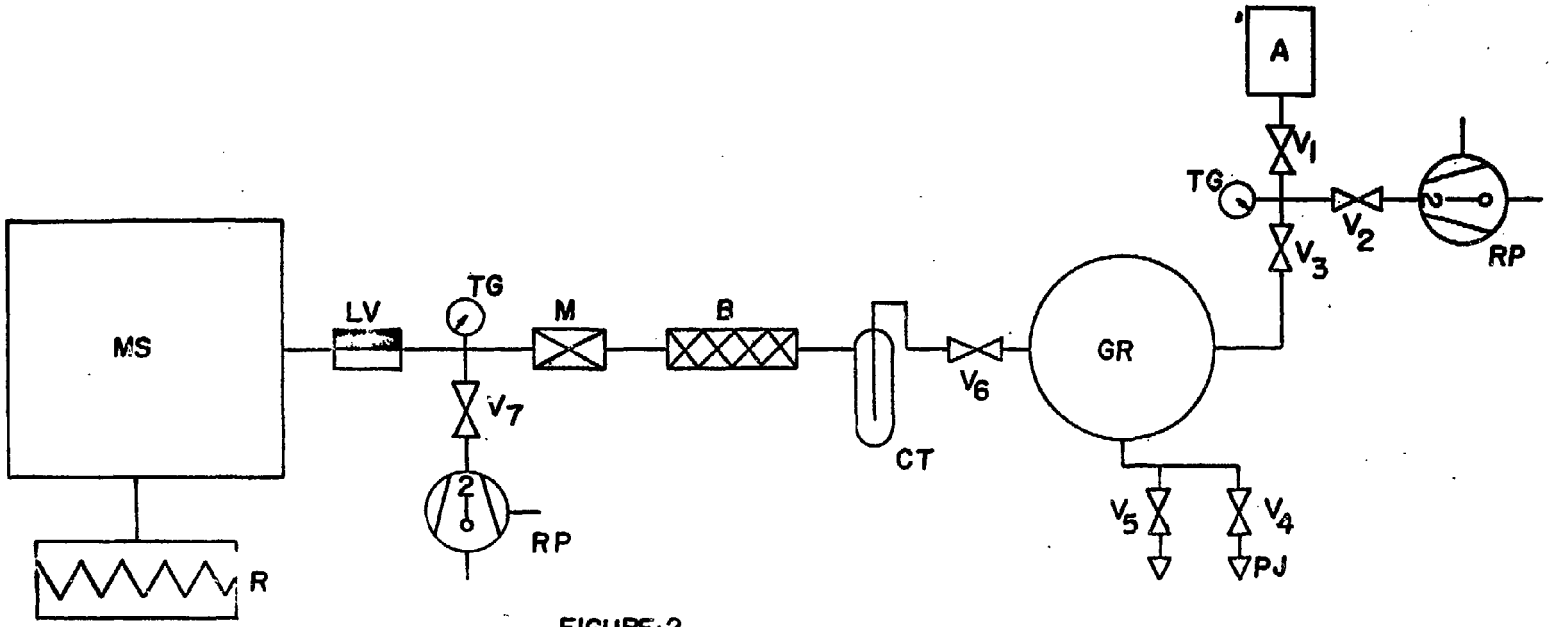


FIGURE:2

A= UNIT FOR STANDARD GAS PREPATION

V_{1-7} = STRAIGHT – THRONG STOPCOCK

TG= TERMOCOUPLE GAUGE

R=RECORDE (SERVAGE· GOERZ)

MS=MASS SPECTOMETER VGA-100

LV=LEAK VALVE (VARIAN MODEL Nº 9515100)

CT= LIQUID NITROGEN COLD TRAP

GR= GAS RESERVOIR (1.5 LITER)

PJ=PYREX JOINT 14/35 FOR SAMPLE INTRODUCTION

M= MINI VALVE BAKEABLE

B=STAINLESS STEEL BELLOW

RP= ROTARY PUMP TWO STAGE

TABLE 3: QUANTITATIVE AND QUALITATIVE ANALYSIS OF THE RESIDUAL GASES PRESENTS IN UO_2 PELLETS

PELLETS Nº	TEMPERATURE OF GAS EXTRACTION (°C)	TOTAL VOLUME (CM^3/g UO_2)	QUANTITATIVE ANALYSIS (ppm)			
			H_2	CO	N_2	CO_2
1	1100	0,007	1	3	1	1
2	1200	0,006	3	4	2	1
3	1300	0,008	3	6	1	1
4	1400	0,008	3	4	1	1
5	1500	0,014	7	1	1	1
6	1600	0,017	11	1	3	2
7	1700	0,029	22	2	3	1

mass spectra of gas sample in order to determine the species and quantities of gases in the sample. This procedure needs a precalibration with various types of gas mixtures (H_2, N_2, CO and CO_2). The least squares solution reproduces the original spectrum to within 10%. Analysis of mass spectra of gas samples should be done by a weighted least squares procedure on a computer because of the speed, reproducibility and accuracy obtained with it.

The realeased gases from UO_2 pellets were studied in function of temperature by mass spectrometric technic and by weighted least squares method. The results shows (Table 3) that hydrogen is one of the highest contributor in the total gas volume released by the UO_2 pellets.

IV. X-RAY DIFFRACTION

The uranium oxides are now widely used as nuclear fuel. The ceramic materials choice for nuclear fuel is mainly due to their favourable properties at high temperatures. These high temperature properties strongly influence the performance of a nuclear power reactor.

One of the important fuel property is related to its nonstoichiometry due to the propensity of the ceramic fuel materials to become nonstoichiometric at high temperatures with their properties tending to modify significantly with the extend of deviation from stoichiometry[5].

The X-ray diffraction is one of the potential physical method to perform the oxygen metal ratio (O/M) analysis in UO_2 based fuels by associating the lattice parameter to its O/M value. However, the establishment of a workable method of performing such measurements must be provided by a well defined annealing and analysing conditions[10].

Some relevant results of high temperature X-rays diffraction studies being conducted in UO_{2+X} sintered samples, as well as the estimate O/M ratio by means of accurate lattice constants will be presented here.

IV.1. EXPERIMENTAL

IV.1.1. SAMPLE PREPARATION

The sample was prepared by Centro de Metalurgia Nuc

er-IPEN from DUA calcination. The oxide was treated with hydrogen at 750°C for 30 min. After compactation, the sample was sinterized in hydrogen at 1700°C for 1:30 hour. The sinterized pellet was cut to yield a 15 x 24 x 1,5 mm plate. The plate surface was polished and positioned in the platinum sample holder. Spectroscopic analysis showed the following impurities in uranium weight ppm: Si, 82; Al, 200; Ni, 6; Cr, 12; Fe, 20; Mn, 3.6; Cu, 0.9; Mg, 2.4; Cd, 0.1.

IV.1.2. HIGH TEMPERATURE X-RAYS DIFFRACTION

X-Ray diffraction data were obtained on a diffractometer (Rigaku Denki Co.) equiped with a platinum resistance furnace (model A.4).

Three high-temperature X-ray diffraction experimental sets were performed in three UO_{2+X} plates.

Series 1) - The (511), (531) and (600) Kbeta reflections were measured by step scanning method, at intervals of 0.02°, for succesive isothermal treatments at 165°C, 350°C, 460°C, 620°C, 800°C, 810°C and 950°C. An argon gas flux was kept at 180 cc/min during the experiments.

Series 2) - Under argon gas atmosphere, the uranium dioxide sample was submitted to a 230°C isothermal treatment. The succesive transformation from UO_{2+X} to U_4O_9 was observed by measuring (200), (220), and (311) Kbeta reflections, by using step scanning device at intervals of 0,02°; the counting time was 50 minutes for each angle.

Series 3) - Using a mixture 80% argon plus 20% oxigen atmosphere, the transformation from UO_{2+X} to U_3O_8 was observed by measuring (220) and (311) Kbeta reflection without step scanning device. The sample was submitted to isothermal treatments at 280°C.

X-ray diffraction pattern of each sample was taken, before and after the thermal treatment, by Guinier-Hagg camera at room temperature, in each of three sets experiments.

IV.2. RESULTS AND DISCUSSION

IV.2.1. SERIES 1.

The lattice constants of the sample 1, derived from observed peak positions of the (511), (531) and (600) Kbeta reflections, are show in the Table 4.

TABLE 4 - LATTICE CONSTANTS OF UO_{2+X} AT DIFFERENT TEMPERATURES

TEMPERATURE ^{o}C	LATTICE CONSTANTS OF SAMPLE 1 IN Å		
	511	531	600
25	5.467	5.463	5.466
165	5.472	5.471	5.471
350	5.480	5.480	5.480
460	5.486	5.487	5.486
625	5.496	5.539	5.537
810	5.509	5.509	5.509

The lattice constants found for $UO_{2.00}$, $UO_{2.05}$ and $UO_{2.10}$ by Gronvold[6], together with the values for sample 1 are listed in the Table 5.

From the experimental data, lattice constants at different temperatures, shown in Table 4, the constant linear expansion coefficient of UO_{2+X} was derived:

$$\alpha = 10.5 \times 10^{-6}/^{o}C.$$

The above result agree very well with those obtainec from Gronvold[6] in the same range of temperature, $450^{o}C$ to $950^{o}C$:

$$\alpha = 10.8 \times 10^{-6}/^{o}C \quad \text{for } UO_{2.00}$$

and

$$\alpha = 10.5 \times 10^{-6}/^{o}C \quad \text{for } UO_{2.05}$$

The agreements of present results with respect to the Gronvold's can be well visualized in the Table 5 and the Figure 3.

TABLE 5 : LATTICE CONSTANTS OF $UO_{2.00}$, $UO_{2.05}$, $UO_{2.10}$ AND SAMPLE 1 AT DIFFRENT TEMPERATURES. IN (Å)

TEMPERATURE	GRONVOLD			SAMPLE 1
	$UO_{2.00}$	$UO_{2.05}$	$UO_{2.10}$	
20	5.4704	-	5.4696	-
25	-	-	-	5.467
138	-	-	5.4769	-
165	-	-	-	5.472
250	5.4839	-	-	-
260	-	-	5.4841	-
350	-	-	-	5.480
397	-	-	5.4896	-
456	-	5.4907	-	-
460	-	-	-	5.486
520	-	5.4943	-	-
522	5.4988	-	-	-
536	-	-	5.4936	-
599	-	-	5.4936	-
607	-	5.4980	-	-
625	-	-	-	5.496
661	5.5087	-	-	-
724	-	5.5052	-	-
778	5.5153	-	-	-
785	-	-	5.5038	-
810	-	-	-	5.509
946	5.5246	-	-	-
951	-	5.5194	-	-
969	-	-	5.5148	-

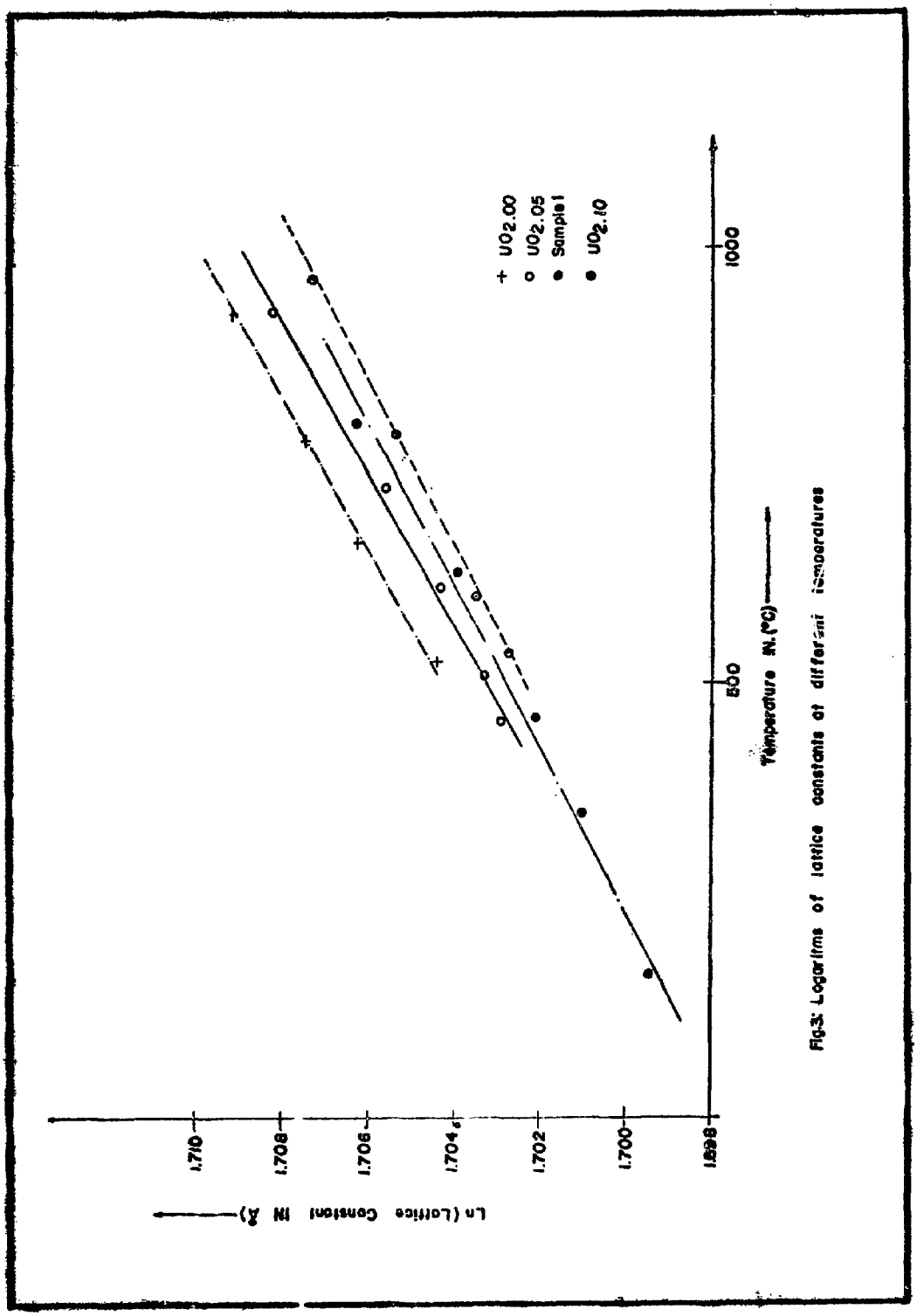


Fig.3: Logarithms of lattice constants at different temperatures

The Figure 3 shows that our experimental data lies amongst Gronvold's $UO_{2.05}$ and $UO_{2.10}$ data, leaving us to the conclusion that the sample 1 probably has O/M ratio of the order 2.07.

Young et al[7] published the values of lattice constants of UO_{2+X} in the interval of X from 0.001 to 0.217 at room temperature. The Young's data were compared with the lattice constant obtained from Guinier pattern, a = 5.4655 ± 0.0002 Å, yielding the O/M ratio value as predicted before:

$$O/M = 2.060 \pm 0.005$$

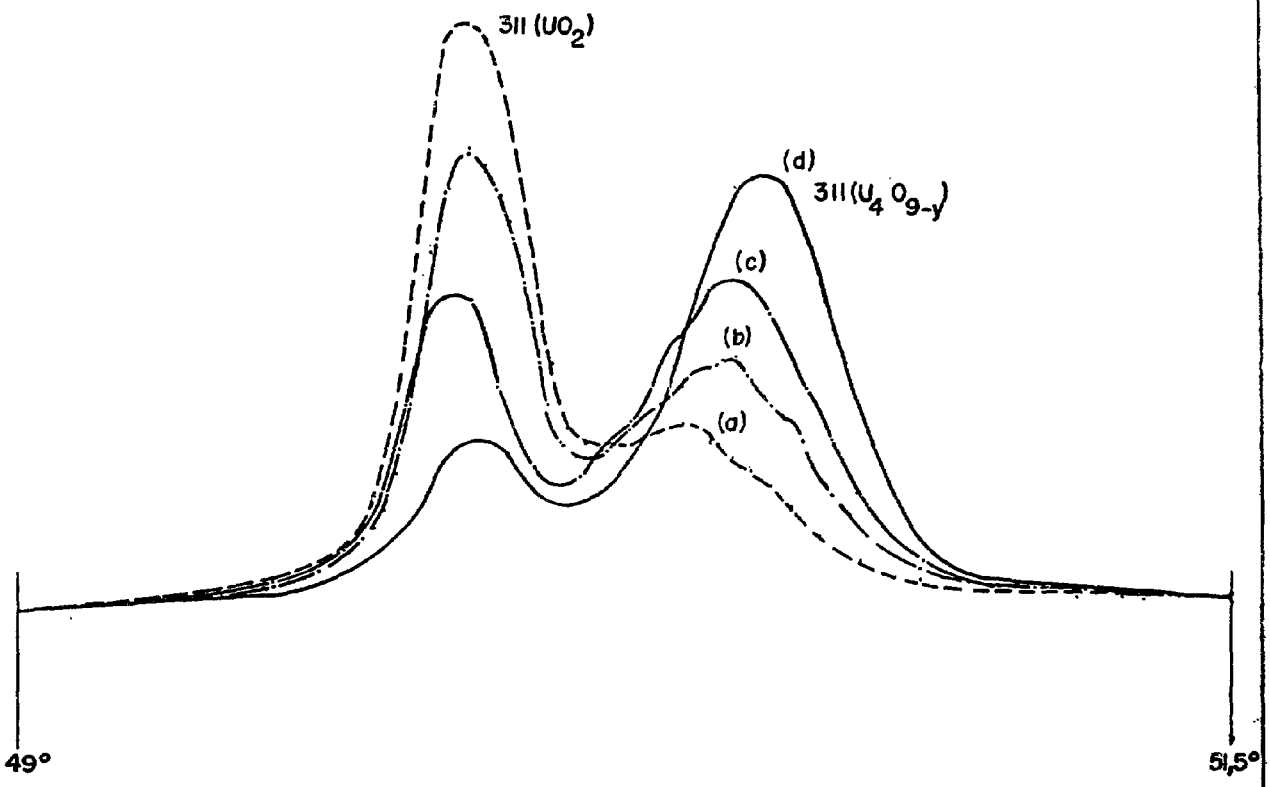
IV.2.2. SERIES 2

The sample was prepared under the same conditions described in the previous series and afterwards it was reduced in H_2 at 500°C for 5:30 hours. The lattice constants obtained from Guinier pattern, a = 5.4712 ± 0.0004 A, has indicated that the sample 2 presented the O/M ratio practically iqual to 2.00.

In this series it was observed structurel transformation $UO_2 - U_4O_{9-y}$ in argon with approximately 18 ppm oxygen at 235 ± 5°C . The evolution of Kbeta (311) reflection profiles can be seen in the figure 4. The evolution of U_4O_{9-y} phase can be observed through its (311) reflection. The figure 5 presents the diffraction diagram of sample 2 after heat treatment for 290:00 hours. The presence of the UO_2 is still noted in the higher reflections whereas the (200), (111) and (220) peaks do not shows any evidence of this phase.

The initiel and final state of the sample 2 represented by Kbeta (311) reflection, got at room temperature, are also in the figure 6.

The time dependence of the intensities ratio $I_{(U_4O_{9-y})}/I_{(UO_2)}$ is found in figure 7. The figure shows a more rapid transformation through (200) than (311) reflections, On the other side, the complete X-ray diagram, figure 5, discloses that degree of transformation effectively is not the same in each chrystallographic direction, For instance the increase of (111) and (200) U_4O_{9-y} reflections have happened more rapidly then (220) and (222) peak; possibly the transformation from UO_2 to



a- After 19:40 hours

b- After 46:00 hours

c- After 116:13 hours

d- After 260:05 hours

Figure4: (311) K-beta profiles for sample 2 as a function of amnealing times.

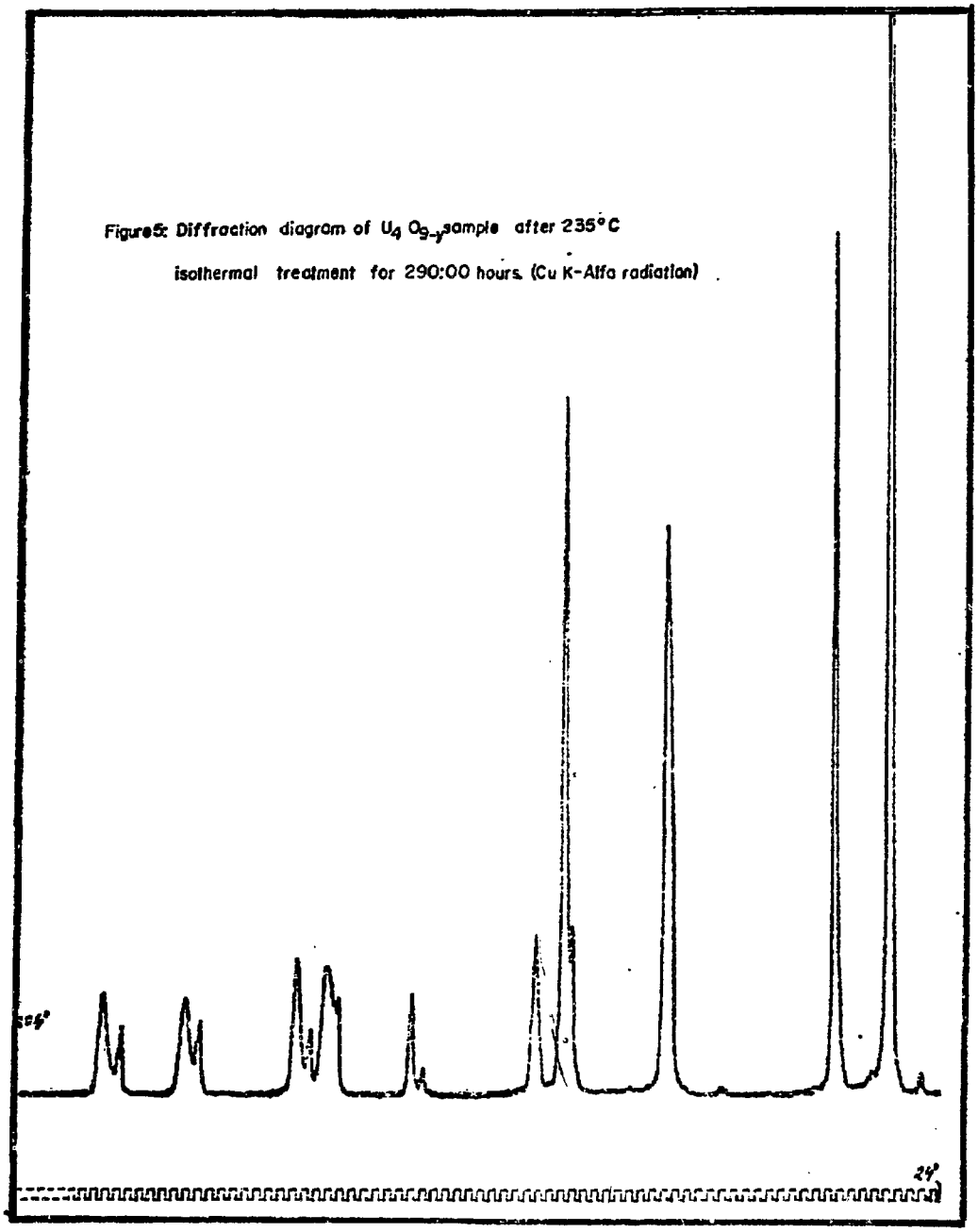


Figure5: Diffraction diagram of U_4O_{9-y} sample after 235°C isothermal treatment for 290:00 hours. (Cu K-Alfa radiation)

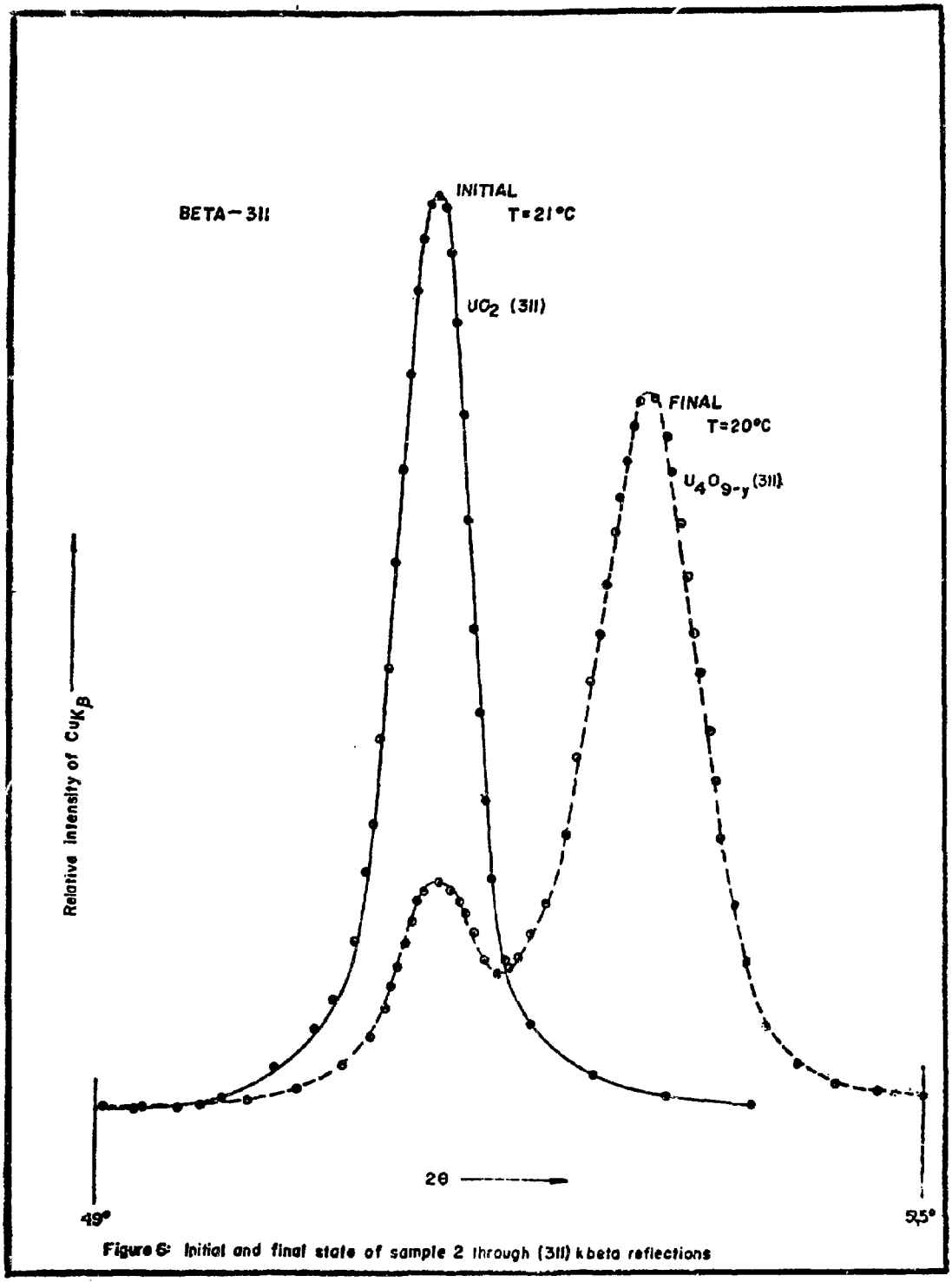


Figure 6: Initial and final state of sample 2 through (311) kbeta reflections

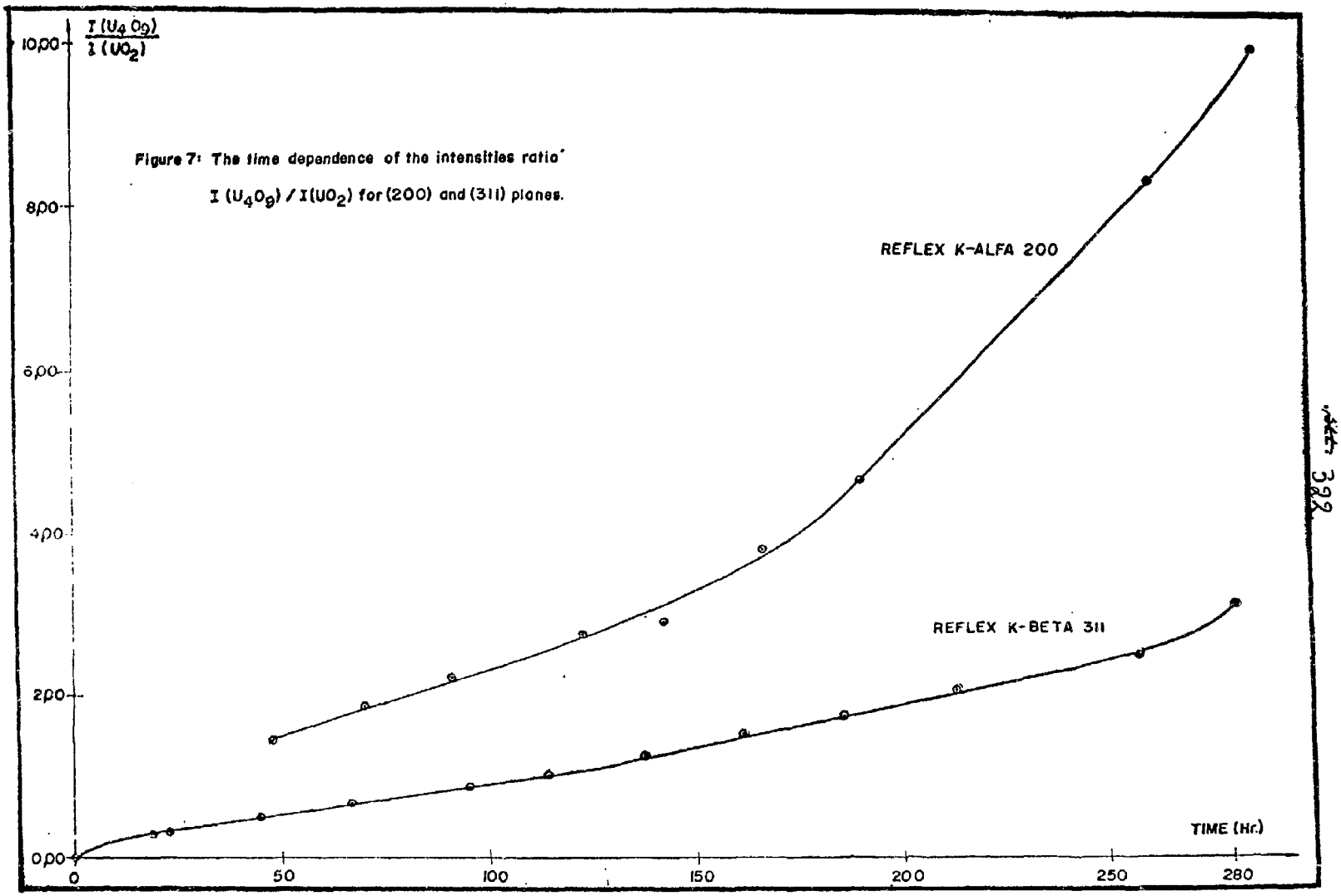


Figure 7: The time dependence of the intensities ratio $I(U_4O_9) / I(UO_2)$ for (200) and (311) planes.

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U_4O_{9-y} may have been completed in that directions.

It was not yet possible to explain the behaviours discussed before and observed in figures 5 and 7. The occupation mechanism of the UO_2-U_4O_9 transformation when the UO_2 interstitial sites are preferencialy occupied by new incoming oxygen atoms, as discussed by Willis (8) for example, may probably explain those behaviours.

IV.2.3. SERIES 3

The structural transformation at 280°C by means of Kbeta (220) and (311) reflections, under atmosphere of 80% Ar. - 20% O_2 , was observed in a sample recognized as $UO_{2.09}$. Both reflections were observed by common scanning method, without use of step scanning devise, where (220) and (311) Kbeta reflex profiles were registered on the recorder as shown in the figures 8 and 9 respectively.

The figure 8 shows the progress of Kbeta (220) reflection from $UO_{2.09}$ to tetragonal (220) doublet reflection, The figure 9 shows the U_4O_9 and UO_2 (311) reflex profiles transforming to the tetragonal doublet reflections.

From the figures 8 and 9 it can be observed that the sample 3 transformation occurs as follows:

- -the intensities of UO_2 reflection decreases,
- -the intensities of U_4O_9 phase increases,
- -when the UO_2 reflections becames very weak, the new peacks appears forming the U_3O_7 doublets,
- -the doublets gaps increases as the annealing time increases,
- -it is not possible to precise the exact beginning position of the tetragonal phase.

The indexed X-ray diffraction diagram of the final U_3O_7 phase is presented in the figure 10. The lattice parameters were determined with the help of Guinier pattern as:

$a = 5.401 \pm 0.004$ Å,

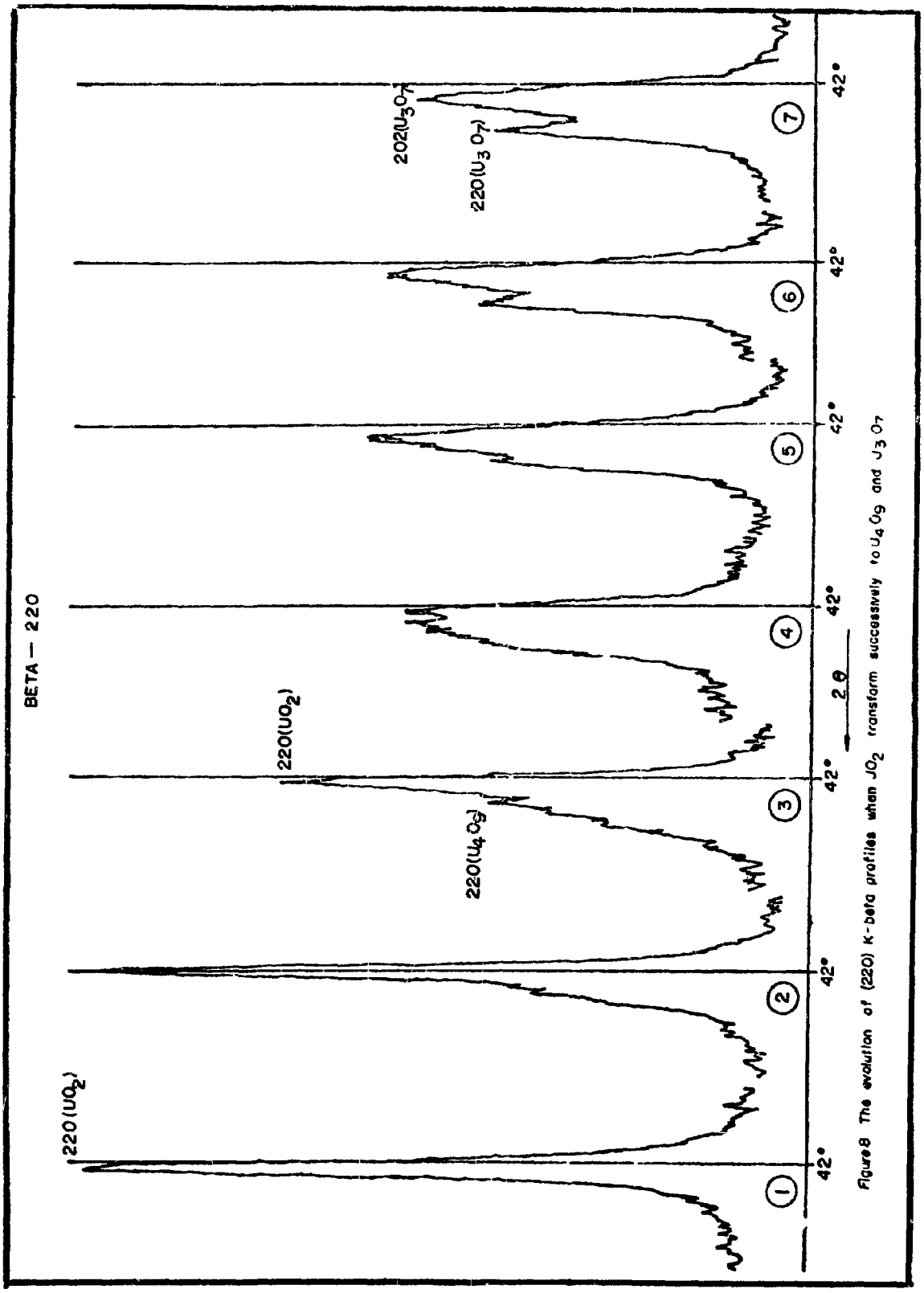

Figure 8 The evolution of (220) K-beta profiles when UO_2 transform successively to U_4O_9 and U_3O_7

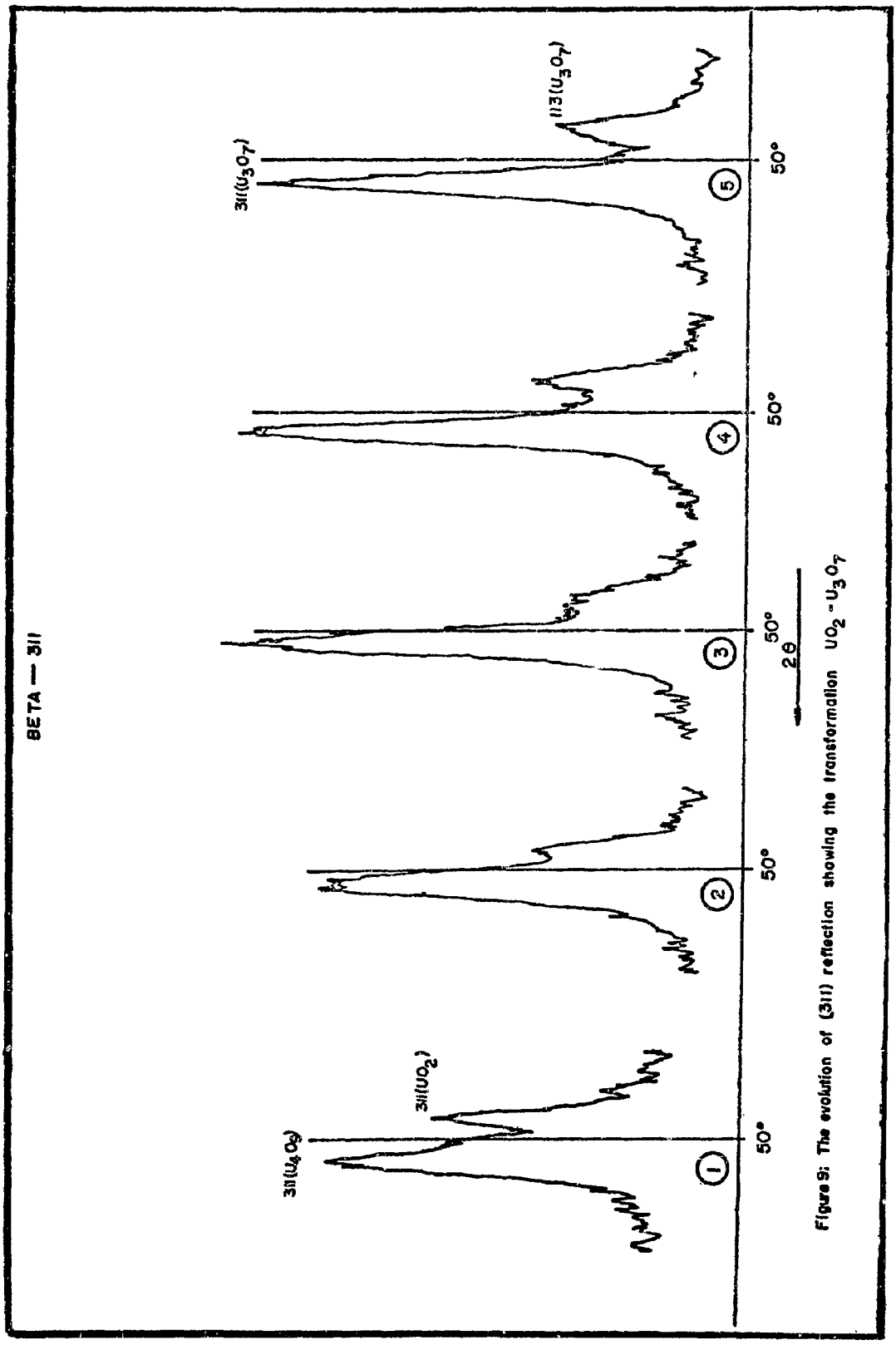

Figure 9: The evolution of (311) reflection showing the transformation $UO_2 - U_3O_7$

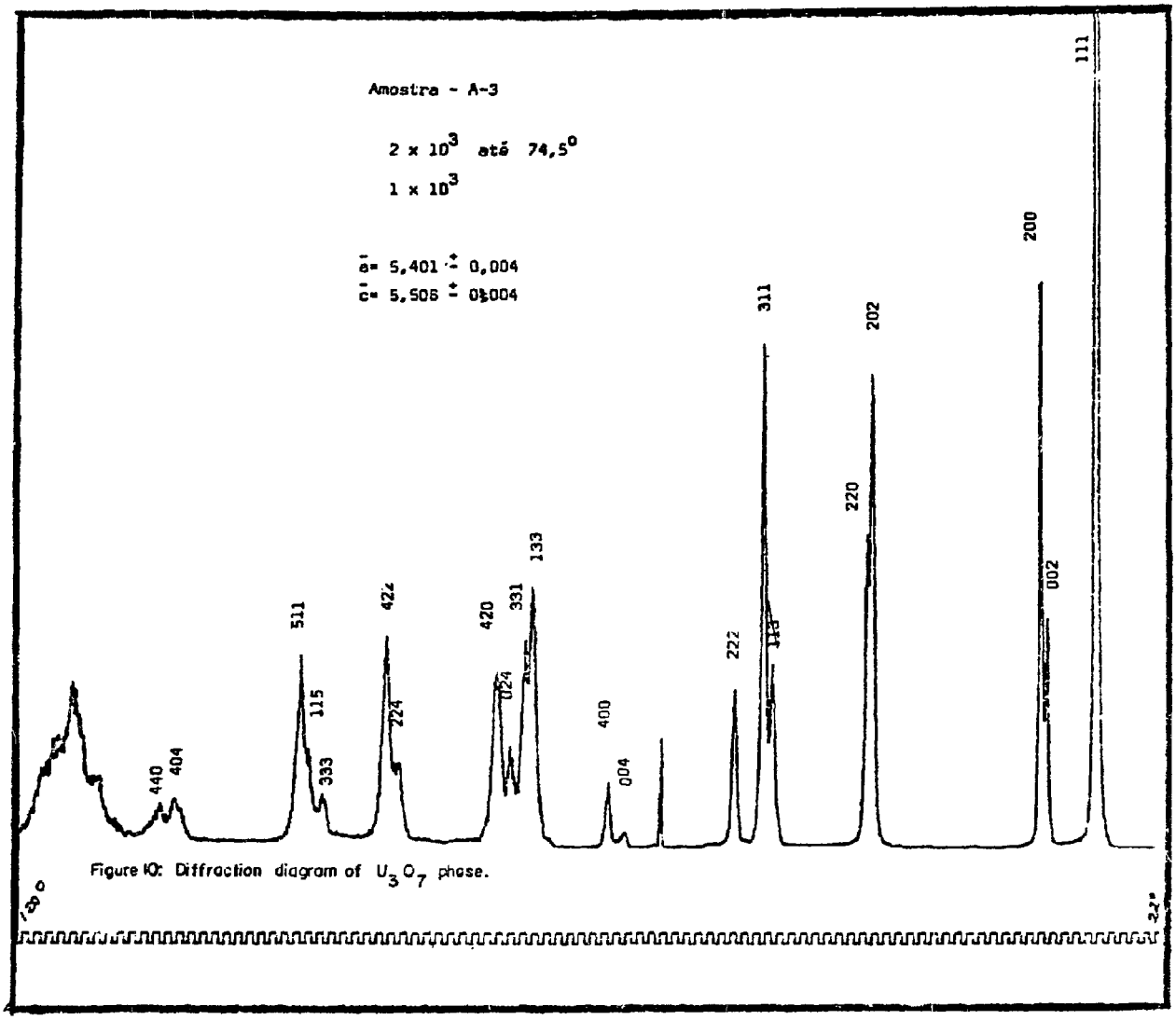

Figure 10: Diffraction diagram of U_3O_7 phase.

$c = 5.506 \pm 0.004$ A,

and was identified as a $UO_{2.30}$ in comparison with the Aronson's results(9).

V. GENERAL CONCLUSIONS

It was outlined in this paper the main activities in the field of Characterization and Control of Nuclear Materials carried out in the "Area de Preqcessos Especiais" IPEN Brasil.

A direct spectrographic method using the fractional destillation technique with carries has been developed for the determination of impubities in UF_4 such as Fe, Ni and Cr, the main contaminants in the UF_4 production. An original technique was employed to avoid the projection of the particles, from the electrode.

The total volume of residual gases from UO_2 pellets was successfully measured with the specified accuracy for UO_2 pellets fabrication. A mass spectrometry apparatus is now employed for quantitative analyses of extracted gases.

Routinely applicable methode of performing O/M analysis by precise lattice parameter measurements on UO_2 pellets is being developed. The recommended list points related to the annealing and analysis conditions are being developed as a indispensable support to permit a successful conclusion of the proposed method.

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