

X-Ray Analysis of Samples from LH84-2

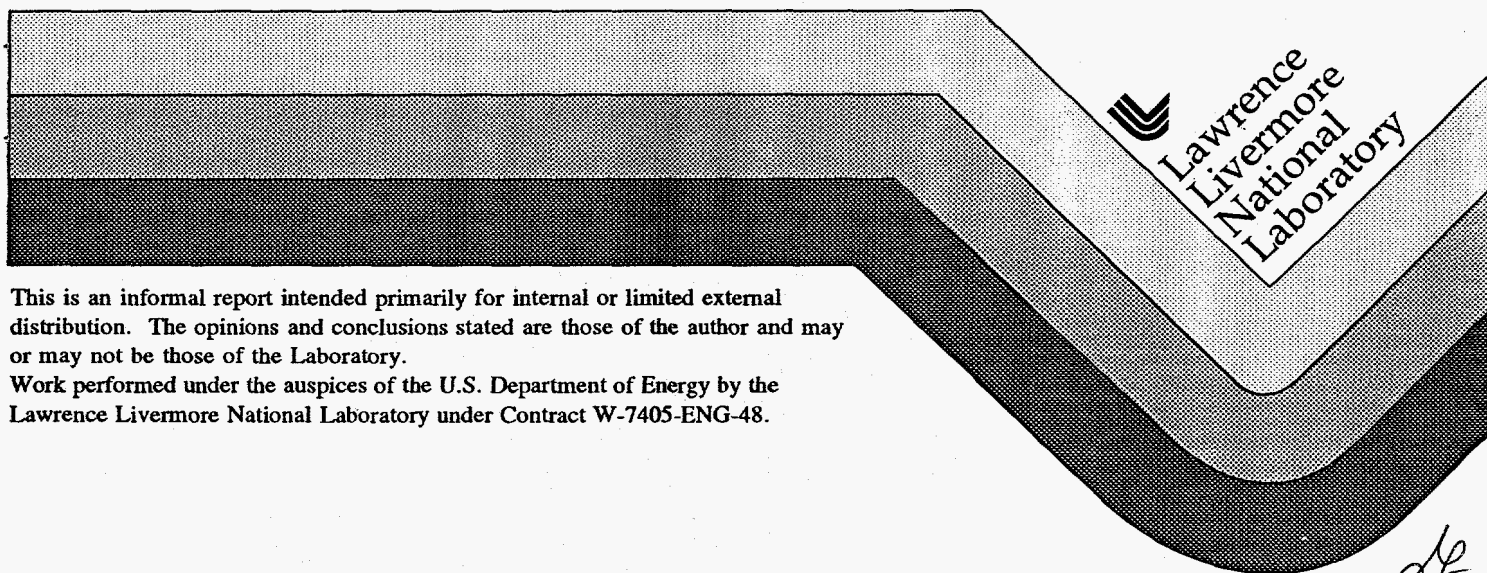
P. L. Wallace
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 by author 5 of R2722 of COCE-82-27 series A.
 by R. June Barron 3/22/95
 August 4, 1982
 1 by [Signature] 3-29-95
 (Signature of [Name])

TO: L. W. Hrubesh
 FROM: P. L. Wallace/D. F. Del Giudice
 SUBJECT: X-Ray Analysis of Samples From LH84-2

<u>Your Sample Nos.</u>	<u>X-Ray Analysis Nos.</u>
BG-4-6 (Vanadium)	G00100
BG-4-7 (Scale)	G00104

INTRODUCTION


Each of these samples was analyzed using automated, scanning x-ray diffractometry. The blue vanadium surface was run in the as-received condition, while a new method of sample preparation was used for the scale. This new method involved (1) grinding the sample in a conventional fashion, (2) mixing the sample with collodion to form a castable slurry, (3) pouring and spreading the mixture on a taut, clean sheet of plastic film, and (4) then covering the resultant sample with a second plastic film layer to form a sandwich-type assembly. Only a few milligrams of sample are needed for this procedure, and the resultant data is much more accurate than that obtained by the previously-used Debye-Scherrer technique.

RESULTS

Figure 1 shows the diffraction spectrum for the blue vanadium surface. The phase analysis for this sample finds vanadium as the major constituent and minor constituents of V₂C and a surface contaminant, PuO₂. Although the source of carbon in this case could be either the carbon susceptor in the RF furnace or the added CO₂, the finding of V₂C does indicate that carbonaceous materials will react with the vanadium.

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In Figure 2, the spectrum for the ground-up scale is presented. In this pattern, the major phases are δ -Pu and PuO₂ with secondary amounts of α -U and PuO. At this time, we have no explanation of why metallic phases (δ -Pu and α -U) are present in the scale. Although this analysis doesn't indicate it, some earlier preliminary work seemed to say the zeta PuU phase might also be present. This spectrum doesn't confirm the earlier work.




Peter L. Wallace

Plutonium Technology

cc: 1/A J. Bergin
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0227Z/rml/PU8



100 7-1-82 DIF CN2 BLUE VANADIUM BG4-6
 INITIAL 2THETA= 24.000 DELTA 2THETA= 0.040 TIME INTERVAL= 4.00SEC
 ISM= 0 IBKG= 0 0 TGKAB= TGDSP=CU ND2= 9 SENS= 1.0 NUM= 1 IPF=5
 D(1)= 3.1057 D(2)= 2.6950 D(3)= 2.4813 D(4)= 2.3700 D(5)= 2.0599
 D(6)= 1.9185

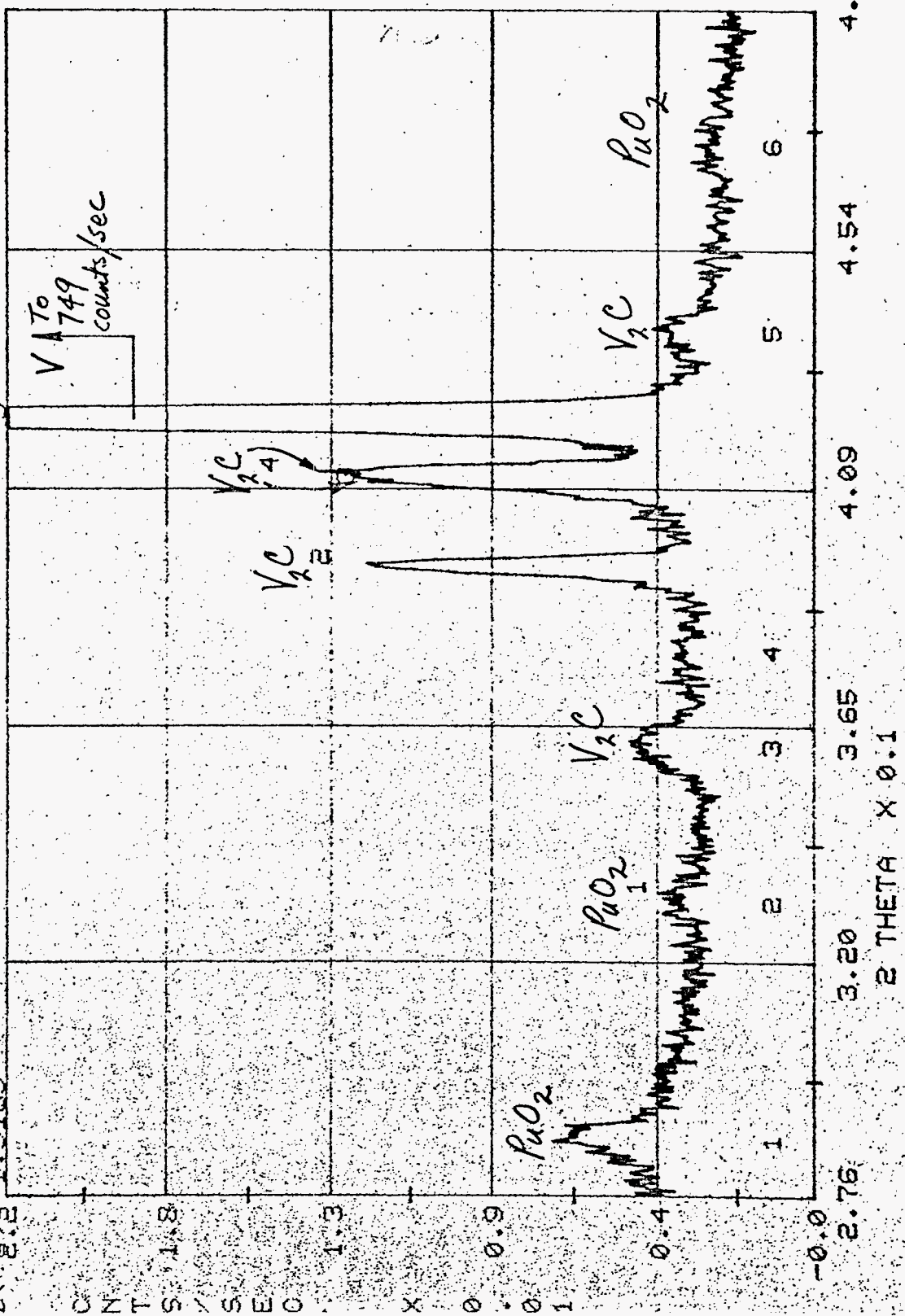


Figure 1: Diffraction spectrum obtained from a blue surface on vanadium from Test LH84-2. This spectrum provides confirmation of the reaction of vanadium w/carbon in this test. The sample designation is BG4-5.

104 7-14-82 DIF CN2 BG4-7
 INITIAL 2THETA= 24.000 DELTA 2THETA= 0.040 TIME INTERVAL= 4.00SEC
 ISM= 0 IBKG= 3.1 TGKA2= TGDSP=CU ND2= 9 SENS= 0.5 NUM= 1 IPF=5
 D(1)= 3.3415 D(2)= 2.9963 D(3)= 2.8970 D(4)= 2.3978 D(5)= 1.9408
 D(6)= 1.6413 D(7)= 1.5585

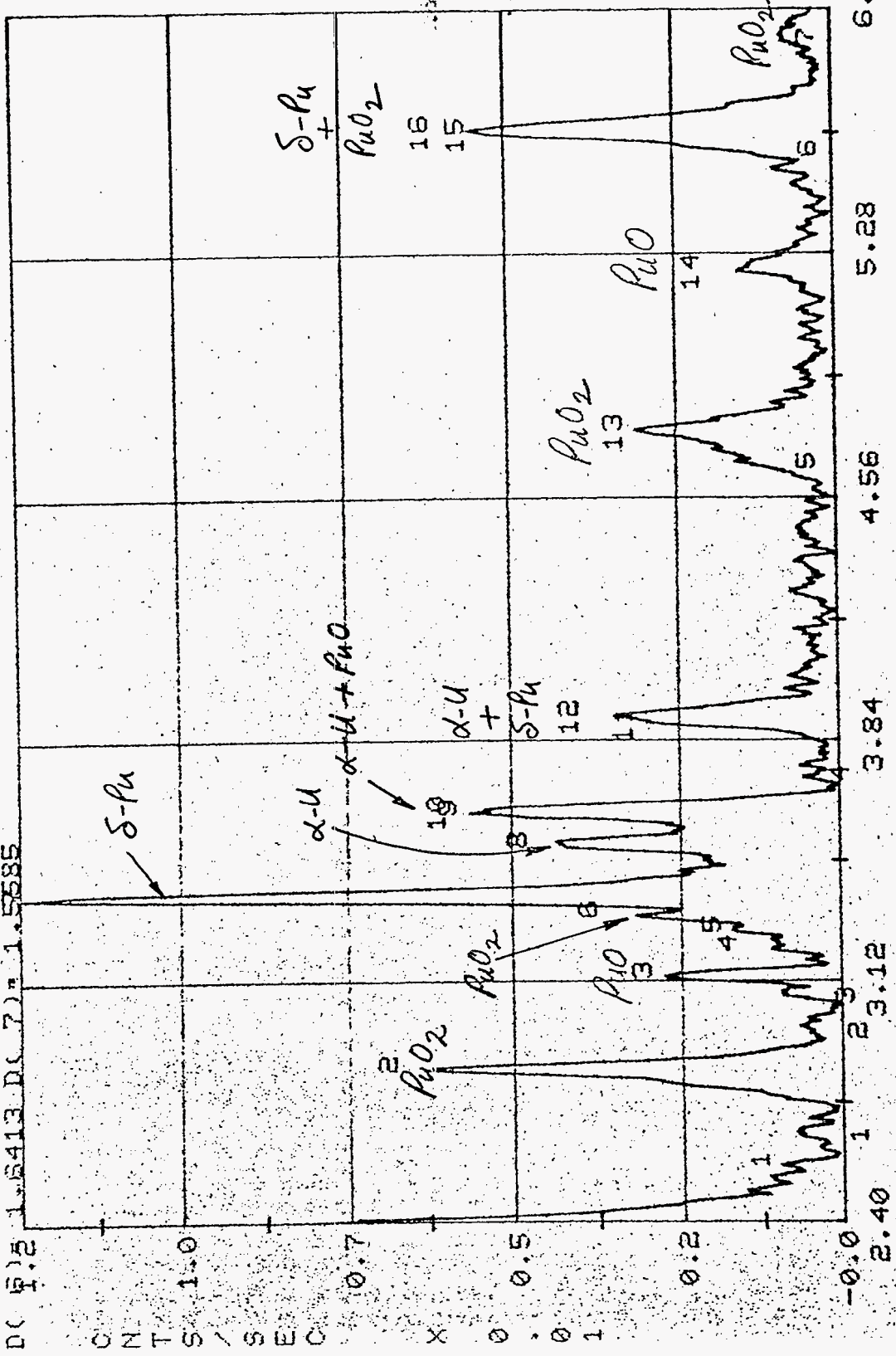


Figure 2: Diffraction spectrum for ground-un scale (designated BG4-7). Beside the expected oxides, two metallic phases ($\delta\text{-Pu}$ and $\alpha\text{-U}$) were found in this material.