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METALLURGY AND CERAMICS

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UNITED STATES ATOMIC ENERGY COMMISSION

FIFTH QUARTERLY REPORT ON THE REDUCTION OF URANIUM OXIDES

Period Covered: October 1, 1953 through December 31, 1953

Sidney Johnson

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Beverly, Massachusetts

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UNCLASSIFIED**ABSTRACT**

During the sixth quarterly period the development of evaluation techniques for more precise determination of the purity of uranium hydride powders, and their suitability for powder metallurgical processes, has been concluded. The evaluation procedures now available are: (1) densities of hot-pressed uranium pellets processed from uranium hydride powder; (2) direct oxygen determinations by the reaction of uranium hydride powders with bromine trifluoride; and (3) uranium oxide assays by dissolution of uranium hydride in a measured excess of ceric acid sulfate solution. Utilization of these methods now makes it possible for the first time to evaluate the reductive processes with sufficient accuracy to determine optimum conditions for the variables involved.

New furnacing equipment of increased capacity has been placed in operation during this period, and several reduction runs to produce well over one pound of uranium hydride have been successfully furnaced.

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INTRODUCTION

Until recently the study of the reduction of uranium oxides was at an impasse because no adequate means of evaluation of the reduction product were available. Thus three of the four experimental techniques employed gave products which were, within the experimental error of the analyses employed, 100% pure. Since the uranium assays were accurate to no better than $\pm 0.3\%$, however, an analysis of $98.4 \pm 0.3\%$ uranium on a sample of a mixture of uranium hydride and uranium dioxide could indicate an uranium dioxide content as low as 0.43% or as high as 6.09% . Since the maximum limit of uranium dioxide allowable by the A. E. C. is approximately 1% , it was evident that more precise methods of evaluation had to be found before the suitability of the reduction processes could be ascertained. Further, changes in purity resulting from a systematic study of the variables of the reaction (temperature, type and amount of excess of reducing agent, particle size of reactants, reagents for and method of leaching, etc.) were masked by the error of analysis, and it was not possible to ascertain the optimum conditions for the reaction.

It was therefore necessary to suspend the study of the variables of the reaction until such a time as more precise evaluation procedures became available. These procedures have now been satisfactorily worked out, and it remains to apply them to the study of the reduction reaction.

DISCUSSION OF RESULTS

1. Reductions

During the past quarterly period the size of the reduction runs has been scaled up in two steps so that a one-kilogram charge has now been successfully furnaceed. Runs 63 through 65 were reductions of 0.75 mole of -325 mesh uranium dioxide with a 50% excess of -40 mesh calcium hydride. These reductions utilized the maximum capacity of the furnace equipment used for all previous reduction runs. Runs 66 and 67 were carried out in newly acquired equipment of increased capacity. Each charge was one kilogram in weight and consisted of 2.46 moles of -325 mesh uranium dioxide and a 50% excess of -40 mesh calcium hydride. In all external characteristics, and in the chemical activity observed, the product from the charge of increased size appeared identical to that obtained previously in smaller runs except that the hue varied from black at the front of the charge to the usual grayish appearance at the rear. This was due to the fact that a temperature gradient of approximately one hundred and fifty degrees existed within the charge. This gradient is a consequence of the facts that the furnace tube is necessarily short in order that it may be handled through the introduction

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chamber of the dry box, and that one end is maintained close to room temperature. There is no reason to doubt that, were the furnacing equipment free of these limitations and a uniform temperature maintained throughout, the nature of the entire product would be identical to that obtained in the rear portion of the charge, and to the products obtained previously on the small-scale reduction runs.

Because of the temperature gradient which existed in Runs 66 and 67, the furnace products from these runs were divided into three sections (front, middle and back), and each section leached separately. A summary of reduction runs made during this period is given in Table I. The data are self-explanatory, but the variation of oxide content with reduction temperature in Runs 66-68 is particularly noteworthy. While the tabulated temperature values for the middle and back portions are nearly the same in Runs 66 and 67, they are of course only mean values taken near the center of each section, and the higher oxide content of the middle portions is without question a consequence of the fact that a portion of the middle section did not attain a sufficiently high temperature to undergo complete reduction. The criticality of the temperature is very probably a function of the vapor pressure of calcium within the charge.

The oxygen analyses given are for the product leached by the standard method. Results obtained by varying the leach conditions are given elsewhere. The analyses by ceric acid sulfate are lower than the true values, but offer a basis of comparison of the various reduction runs. This method of analysis is discussed in a later section of this report.

2. Evaluation of Reduction Product

Three evaluation procedures have been worked out so that they may now be utilized for the study of reduction and leaching processes. The development of each method is recounted below, together with brief statements as to the advantages and limitations of each method.

a. Density of Hot-Pressed Uranium Pellets

The degassing and compacting of uranium hydride powders is a technique for producing massive uranium with a crystalline structure of random orientation which was developed at Sylvania Electric Bayside Laboratories by the group under the direction of Dr. H. H. Hausner. In order to make use of this technique to evaluate the product from small scale laboratory reduction runs, it was necessary to modify the procedure and equipment so that a pellet could be obtained from 25-40 grams of uranium hydride. Accordingly, equipment has been designed, constructed and tested at Metal Hydrides Incorporated which makes it possible to pro-

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6 TABLE I
SUMMARY OF REDUCTION RUNS

Run	Grams UO ₂ (Mesh Size)	% xs CaH ₂ (Mesh Size)	Temp. (°C)	Time at Temp. (hrs.)	% O [by Ce(HSO ₄) ₄]	% O [by BrF ₃]
59 I-32	110 (-200)	50	950	11	0.44	
60 I-33	110 (-200)	50	950	11	0.05	
61 I-34	110 (-325)	50	950	11	0.18	
62 I-35	110 (-325)	50	950	11	-0.08	
63 I-36	202 (-325)	50 (-40)	950	11	0.14	
64 I-37	202 (-325)	50 (-40)	950	11	0.20	
65 I-38	202 (-325)	50 (-40)	950	11	0.07	
66 I-39	669 (-325)	50 (-40)	F 795 M 940 B 940	11	0.61 0.14 -0.11	
67 I-40	669 (-325)	50 (-40)	F 755 M 950 B 945	11	-0.06 -0.32	0.30
68 I-41	271 (-325)	50 (-40)	F 890 B 968	11	-0.10 -0.31	0.53
69 I-42	271 (-325)	50 (-40)	F 900 B 978	11	-0.04 +0.04	

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duce uranium pellets from this quantity of uranium hydride. Development of this process was delayed both by lack of knowledge of the Sylvania process, and by inability to obtain materials.

The initial hot-press die unit was designed so that the entire operation could be carried out in a dry box. However, the limitations imposed on the design of the die unit by the stipulation that it was required to be manipulated and hot-pressed in the dry box were such that its performance in operation was unsatisfactory. Only two pellets (Nos. 1 and 2) were produced from this unit. (See NYO-3795.) When it was found to be unnecessary to carry out the actual hot-pressing in the dry box, an improved die unit was designed. The die body, plunger, and bottom pin were fabricated from Rex M-2, because Haldi #2 tool steel had proven unsatisfactory in the original die unit, and Rexalloy, the preferred material of construction, could be obtained only after a prohibitive time delay. The Rex M-2 plunger barreled out under the duress of the pressing operation, and a plunger of Electrite Ultra Cobalt* was tried. This also barreled out under the conditions employed. While all of these materials proved unsatisfactory, it should be pointed out that they were inadvertently used at temperatures far in excess of those actually required. These excessive temperatures were a consequence of holding the temperature at 600°C as measured by a chromel-alumel thermocouple situated in a thermowell within the bottom pin. That the actual temperature of the pellet was considerably greater than the measured temperature was suggested by the following facts: (1) the carbon content of the uranium pellet was consistently higher than that of the uranium hydride powder from which it was pressed; (2) the plungers used (both Rex M-2 and Electrite Ultra Cobalt) were chemically attacked by the uranium; (3) the plungers were barreled out under the duress of the hot pressing operation to a much greater extent than expected for the supposed operating conditions; (4) there was considerable heat loss through the bottom of the die unit; and (5) of the first seven pellets produced, the only one which gave a satisfactory density (No. 3) was pressed with diminishing heat after the heating element burned out.

In order to obtain better temperature control and to establish the relationship between the temperature at the thermowell and the temperature of the charge, a thermowell was drilled into the wall of the die body so that the thermocouple junction would be

* A high cobalt high-speed steel. Approximate analysis:

W	18.75%	C	0.85%
Co	12.0	Mo	0.8
Cr	4.1	Mn	0.25
V	1.9	Si	0.25

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at the level of the pellet being pressed and within 1/4" of the bore. An old plunger was drilled out so that a thermocouple would be placed through the plunger and into a powder charge. A determination of the temperature gradient between the thermocouple well and the metal powder charge was made (titanium powder was used for this purpose), and the temperature of the charge was found to be in satisfactory agreement with the temperature recorded by the thermocouple in the thermowell, a gradient of only 10-20°C being observed. Thus an accurate measurement of the temperature of the actual powder charge is obtained with the thermowell in the wall of the die body. That this was not the case previously, when the thermowell was situated in the bottom pin, is shown by the fact that, employing the same heating unit, a Variac setting of 65 volts sufficed to maintain a temperature of 600-620°C in the die body at the level of the pellet, whereas 100 volts had been required to maintain the same temperature with the bottom pin.

A Rexalloy die unit with thermowell in the die body is now in operation and uranium metal of density 18.85 g.-cm.³ has been hydrided, degassed, and hot-pressed to a density of 18.83 g./cm.³. Thus it has been shown that the small-scale hot-pressing operation itself is not introducing additional impurities to the powder.

The new die unit consists of the Rexalloy die body, plunger, and bottom pin, and in operation a degassed 1/8" wall AUC-grade carbon sleeve is placed between the die body and plunger. The heater for the unit is a 1500-watt helix resistance coil, insulated with Sauereisen, firecrete, and an alundum outer shell, all encased in a cylinder of stainless steel. Electrical leads are made with silicone gaskets compressed by tightening the connecting nut. The thermocouple is introduced through the O-ring gasket. The minimum charge for this die unit is approximately 25 grams of uranium hydride powder.

A complete summary of all pellets produced during 1953 is given in Table II. The significance of the densities reported is better related to the development of the method rather than to the purity of the uranium hydride powders used. The densities of the last two pellets are the only ones to be considered representative of the powders.

The theoretical densities listed in Table II were calculated according to the equation:

$$\rho = \frac{19.05 (\%U) + 160.78 (\% O)}{\% U + 14.656 (\% O)} .$$

This equation was derived with the assumptions that all the oxygen is present as uranium dioxide and that uranium and uranium

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TABLE II

SUMMARY OF HOT-PRESSED URANIUM PELLETS PRODUCED AT METAL HYDRIDES INCORPORATED

Pellet No.	Starting Material		Pressing Operations			Final Product		
	U _H ₃ From	% O*	Temp. (°C)	Time (min.)	Pressure (t.s.i.)	% O	(theoretical)	(actual)
1	SF U P _U = 18.85		>600	30	30			18.58
2	Run 43	0.59 (D)	>600	25	20			16.60
3	SF U P _U = 18.85		~600	120	30	0.01 (V)	19.04	18.80
4	Run 55 (73%) Run 56 (27%)	0.66 (C)	≥600	180	30	0.50 (V)	18.50	17.88
5	Run 56	0.35 (C)	≥600	30	30	0.65 (V)	18.34	17.82
6	Run 57	0.22 (C)	≥600	30	30			18.06
7	Run 61	0.18 (C)	600	100	30	0.52 (B)	18.48	18.13
8	SF U P _U = 18.85		600	225	30	0.09 (B)	18.94	18.83
9	Run 63 (60%) Run 65 (40%)	0.13 (C)	600	190	30	0.66 (B)	18.33	17.39

- * (B) By bromine trifluoride method (includes nitrogen)
 (C) By ceric acid sulfate method (low results)
 (D) By difference
 (V) By vacuum fusion method

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dioxide are the only constituents present. It was further assumed that the density of the mixture of uranium and uranium dioxide is equal to the weighted average of the densities of the component parts, the contribution of each constituent being determined by the fraction of the total volume which it occupies. (This assumption has the tacit implication that the two phases are completely immiscible--i.e., there is no solid solution of one phase in the other; or, if such solution does occur, the partial molal volumes of the constituents are equal to the molal volumes of the pure substances.)

It is known of course that other impurities also lower the density of uranium metal, and it is to be expected that actual densities will always be less than a calculated density which does not account for all impurities present. An empirical linear relation has been reported (A. R. Kaufmann, Report CT-685, May 20, 1943, p. 15) between the density of uranium metal and the carbon content. However, from the values in Table II and those subsequently obtained, it is highly improbable that the discrepancy between actual and calculated values can be explained solely on the basis of impurities other than the oxide. The oxide appears to have a detrimental effect on the compacting or internal cohesive properties of the metal which prevents attainment of theoretical density. A small decrease in the oxide content should, therefore, cause a relatively large increase in density.

As a check on the precision obtained in determining the density of uranium metal, the densities of five samples of SF uranium metal obtained from the A. E. C. New Brunswick Laboratories were determined for comparison with the values reported by the New Brunswick Laboratory. Comparisons are shown in Table III.

TABLE III
COMPARISON OF DENSITY VALUES OF URANIUM METAL

New Brunswick Lab Identification	B1703	B1728*	B1978	B1990	B2110
New Brunswick Lab	18.99	18.91	18.95	18.94	18.96
Metal Hydrides Lab	18.98	18.95	18.92	18.91	18.94
	19.00	18.79	18.95	18.90	18.91
		18.83			
		18.72			

*Very irregular surface; difficult to remove gas pockets

Thus, excluding the sample identified as B1728, the limit of error is approximately ± 0.03 g./cm.³.

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As an analytical method, a density determination suffers from the disadvantages that production of a pellet is cumbersome and time-consuming, and that it fails to distinguish between or identify the impurities present. Its great asset is that it is also an evaluation of the suitability of the uranium hydride for powder metallurgical processes.

b. Direct Oxygen Determination by Displacement
With Bromine Trifluoride

The high-vacuum apparatus necessary for oxygen determinations utilizing bromine trifluoride has been assembled and vacuum-tested. Considerable difficulty was encountered in obtaining zero blanks, due to the dissolved gases which are contained in commercially available bromine trifluoride. Three samples of SF uranium metal of density 18.99 have been run and oxygen values of 0.03, 0.02, and 0.03% obtained.

This method does not distinguish between oxygen and nitrogen. Since oxygen contamination is of primary concern, all of the gas collected is measured and reported as oxygen. The principal advantage of this method is that it gives a direct assay of the principal impurity, and greater accuracy can therefore be expected than from any method in which the oxygen content is obtained by difference. A separate topical report on this method will be prepared.

c. Uranium Oxide Assay by Direct Solution in
Ceric Acid Sulfate

This method has been shown to give excellent precision. However, other impurities, as carbon and/or nitrogen, cause low results and in some cases result in negative values. Preliminary results indicate that oxygen values obtained by the ceric acid sulfate method are approximately 0.5 to 0.7% lower than corresponding oxygen values obtained by the bromine trifluoride method. This, of course, depends on the nature and extent of impurities present, which tend to make values obtained by the ceric acid sulfate method low, and results obtained by the bromine trifluoride method high. It is planned to determine the effect of carbon on the two methods. While the oxygen values obtained by the ceric acid sulfate method are low, it does give excellent precision and offers a valuable analytical tool for comparative purposes. This method is a modification of an uranium analysis first employed by Mr. Petretic of the A. E. C. New Brunswick Laboratories. The handling procedure has been modified to eliminate possible sources of error peculiar to handling pyrophoric powders, and a complete description is, therefore, given below.

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Materials

Ceric acid sulfate, G. F. Smith Co.
Sulfuric acid, C. P., J. T. Baker Co.
Ferrous ammonium sulfate, Mallinckrodt, A R grade
1,10 o-phenanthroline ferrous sulfate indicator (0.1 M)
(Ferroin)

Procedure

Standardization of $\text{Ce}(\text{HSO}_4)_4$: A 25.00 ml. portion of approximately 0.1 N ceric acid sulfate is refluxed with 25 ml. of 96% sulfuric acid for 0.5 hour, cooled to room temperature and titrated against ferrous ammonium sulfate to obtain the normality ratio of the two solutions. Approximately two grams of MS-ST U_3O_8 are weighed by difference into a reflux flask. After pipetting in 100.00 ml. of ceric acid sulfate, the solution is refluxed for 0.5 hour (longer, if necessary for complete dissolution). The condenser is washed down and removed, the solution is cooled to room temperature in a water bath, and the excess ceric acid sulfate titrated with ferrous ammonium sulfate using seven drops of ferroin as indicator. The normality of the ceric acid sulfate is then calculated from the known content of tetravalent uranium in U_3O_8 and the previously determined normality ratio of the ceric acid sulfate to ferrous ammonium sulfate.

Determination of uranium oxides: A 0.2-0.5 gram sample of uranium hydride is weighed out in the dry box into a 3-ml. glass vial which is placed in a 500-ml. Florence flask fitted with a dropping funnel by means of a gas-tight 3 24/40 joint. The flask and contents are removed from the dry box and 100.00 ml. of ceric acid sulfate are pipetted into and through the dropping funnel, so that the sample is immersed in ceric acid sulfate solution without exposure to air. The dropping funnel is washed down with 75 ml. of 1:1 sulfuric acid. The analysis is carried out in exactly the same manner as the standardization of the ceric acid sulfate with X_3O_8 and ferrous ammonium sulfate. If the assumption is made that the sample consists only of uranium hydride and uranium dioxide, then the per cent oxygen may be calculated by the equation:

$$\% \text{ O} = 16.87 - \frac{0.6778 \text{ NV}}{\text{S}},$$

where N and V signify the normality and volume (in milliliters), respectively, of the ceric acid sulfate titrant, and S, the sample weight (in grams). The precision is $\pm 0.1\%$.

3. Leach Studies

Now that analytical methods are available which permit meaningful research on the sources and extent of contamination of the reduction product, a study has been initiated to determine the

effect of varying leach conditions on the purity of the product. This study necessarily must precede a study of the reduction conditions, for by preparing high-purity uranium hydride from the elements, the leach study can be made completely independent of the reduction procedure. On the other hand, the analytical techniques developed are such that the reduction can be evaluated only after the product has been leached. It should be borne in mind that due to possible differences in particle size, etc., uranium hydride prepared from the elements may have slightly different properties and exhibit somewhat different behavior during leaching than uranium hydride prepared by reducing uranium dioxide. In the lack of any contrary evidence, however, it seems reasonable to assume that a given leach procedure will be equally satisfactory for uranium hydride from either source.

While this study is only in its preliminary stages, considerable valuable information has already been obtained concerning the following: (1) effect of adding the furnace charge to water with subsequent addition of acetic acid, as compared to the direct addition of the furnace charge to 10% acetic acid solution; (2) effect of suspending charge in an inert diluent and then adding 10% acetic acid; (3) effect of 1% nitric acid wash; (4) effect of contact time of uranium hydride powder with leach solution; (5) relative merits of filtration vs. centrifugation; and (6) oxide content of material which settles rapidly as compared to suspended fine material which requires centrifuging. Each point is briefly discussed below, while a summary of data to date is given in Table IV.

The addition of the furnace charge to the acid leach is accompanied by the evolution of considerable heat, which contributes to the hazard of the operation. It has been found that adding the charge to water and subsequently adding acetic acid to the cooled solution is equally effective. This is a more desirable procedure because the heat of neutralization of the calcium oxide is not evolved until the charge is suspended in water, thus minimizing the danger of local overheating. The leaches described for Run 66 were, for the sake of uniformity, all made by adding the charge to 10% acetic acid, but henceforth all furnace charges will be suspended in water before the acid is added.

Due to the evolution of heat mentioned above, a small amount of time was spent seeking an inert diluent in which the charge could be suspended and to which the acid could be added. Four leaches (66F3, -4, -5, and -6) were made in which the furnace product was mixed with "Nujol", a highly refined, high boiling, saturated hydrocarbon white oil, to form a slurry, and the slurry then added to aqueous acid. The Nujol served its purpose of moderating the vigor of the initial reaction very well, but exhibited a tendency to hold the charge in an emulsion and render separation very difficult. The high oxygen values for

Considerable interest has been shown in the possibility of using nitric acid as a leaching medium. Leaches 66B5 and -6 were carried out in the standard way except that a wash with 1% nitric acid was included. The oxygen content was not changed appreciably by this treatment. Further work will be done with the object of eliminating acetic acid from the leaching procedure if this is practical.

No suitable filtration media have been found for removing uranium hydride from an aqueous suspension. Limited success was achieved with a sintered steel Grade H filter (Micro Metallic Company, Glen Cove, New York), but the process is too slow with large quantities of uranium hydride.

In leaches 66M3 and -4 it was demonstrated that uranium hydride which settled rapidly and from which the mother liquor is readily decanted has less oxide contamination than the fine suspended material which requires centrifugation. This was not unexpected, as the fine particles undoubtedly have a much greater surface area for a given weight of material, and are therefore more readily oxidized.

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TABLE IV SUMMARY OF LEACH STUDIES

Leach No.	Purpose	% O Ce(HSO ₄) ₄	Method of Addition	Method of Separation	Washes		Time (min.)	
					No.	Kind	Addition	Contact
66F1)	Standard)	0.66)	Standard)	Decant)	1	10% HOAc)	18	93
66F2)		0.56)	(Powder to)		2	H ₂ O)		
			10% HOAc))		3	MeOH)	18	93
66F3)	Nujol)	1.84)	Nujol slurry,)	Centrifuge)	1	10% HOAc)	85	24h
66F4)		1.91)	added to 10% HOAc)		2	H ₂ O)		
					4	MeOH)	0.2	24h
66F5)		2.48)	Nujol slurry added)	1 Decant - 2 Cent.)	2	iPrNH ₂)	0.5	66h
			to H ₂ O, HOAc added)		2	H ₂ O)		
66F6)		2.66)			3	MeOH)	0.5	66h
66F4r)	Hexane)	1.78)	Hexane syringed)	Decant)			1	85
66F6r)		2.70)	into vial to cover)		3	n-Hexane)	1	85
			powder)					
66M1)	Standard)	0.17)	Standard)	Decant)	1	10% HOAc)	10	110
66M2)		0.10)			2	H ₂ O)		
					3	MeOH)	10	110
66M3)	Settle)	0.35)	Standard)	Decant)			15	105
		0.84)			1	10% HOAc)		
66M4)	Settle)	0.29)	Standard)	Decant)	2	H ₂ O)		
					3	MeOH)		
		0.38)					16	105
66M5)	Short)	-0.04)	Standard)	Decant)	1	10% HOAc)	8	50
66M6)					2	H ₂ O)		
		0.12)			3	MeOH)	8	50
66M7)	Long)	-0.02)	Standard)	Decant)	1	10% HOAc)	20	150
66M8)					2	H ₂ O)		
		0.11)			3	MeOH)	20	150
66B1)	Standard)	-0.19)	Standard)	Decant)	1	10% HOAc)	2.8	180
66B2)					2	H ₂ O)		
		-0.02)			3	MeOH)	2.8	180
66B3)	Filter)	0.22)	Standard)	Filter)	1	10% HOAc)	15	125
					2	H ₂ O)		
					3	MeOH)		
66B5)	HNO ₃ wash)	-0.14)	Standard)	Decant)	1	10% HOAc)	10	110
66B6)					1	H ₂ O)		
		-0.14)			1	1% HNO ₃)	10	110
					2	H ₂ O)		
					1	CH ₃ OH)		

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